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**Coal and coke—Methods for
proximate analysis**

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee, as the result of proposal for revision of Japanese Industrial Standard submitted by Center for Coal Utilization, Japan (CCUJ)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14. Consequently **JIS M 8812 : 1993** is replaced with this Standard.

This revision has been made based on **ISO 562 : 1998** *Hard coal and coke—Determination of volatile matter*, **ISO 687 : 1974** *Coke—Determination of moisture in the analysis sample* and **ISO 5071-1 : 1997** *Brown coals and lignites—Determination of the volatile matter in the analysis sample—Part 1 : Two-furnace method* for the purposes of making it easier to compare this Standard with International Standards; to prepare Japanese Industrial Standard conforming with International Standards; and to propose a draft of an International Standard which is based on Japanese Industrial Standard.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

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Coal and coke—Methods for proximate analysis

Introduction This Japanese Industrial Standard has been prepared based on **ISO 562 : 1998** *Hard coal and coke—Determination of volatile matter*, **ISO 687 : 1974** *Coke—Determination of moisture in the analysis sample* and **ISO 5071-1 : 1997** *Brown coals and lignites—Determination of the volatile matter in the analysis sample—Part 1 : Two-furnace method* with some modifications of the technical contents.

Portions given sidelines or dotted underlines are the matters modified from the original International Standards. A list of modifications with the explanations is given in annex (informative).

1 Scope This Standard specifies the methods for the proximate analysis of coal and coke.

- Remarks 1 The proximate analysis in this Standard covers the determination of moisture content in the analysis sample (hereafter referred to as “moisture content”), ash content, volatile matter content, and fixed carbon in the sample.
- 2 This Standard may be applied to the analysis of charcoal and briquets.
- 3 The International Standards corresponding to this Standard are as follows.

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standards and **JIS** are **IDT** (identical), **MOD** (modified), and **NEQ** (not equivalent) according to **ISO/IEC Guide 21**.

ISO 562 : 1998 *Hard coal and coke—Determination of volatile matter* (MOD)

ISO 687 : 1974 *Coke—Determination of moisture in the analysis sample* (MOD)

ISO 5071-1 : 1997 *Brown coals and lignites—Determination of the volatile matter in the analysis sample—Part 1 : Two-furnace method* (MOD)

2 Normative references The standards listed in attached table 1 contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards (including amendments) shall be applied.

3 Definitions For the purpose of this Standard, the definitions given in **JIS M 8810**, **JIS Z 8402-1** to **JIS Z 8402-4** and **JIS Z 8402-6** apply.

4 General requirement

4.1 Sample The air dried sample prepared in accordance with 9.7.3.4 of JIS M 8811 shall be used (see 4.4.1).

Remarks : Since the moisture content of the air dried sample stored in a sealed container changes little for a period of about seven days, it is not necessary to determine the moisture content in the air dried sample every time for analysis of the ash content and the volatile matter content within the above period. However, coal with low coalification has comparatively much moisture content, and since a moisture content value may be changed according to the storage condition, it is necessary to give enough attention to handling.

4.2 Measuring instrument

4.2.1 Balance and weight The chemical balance and weight of grade 1 specified in 5.2 of JIS M 8810 shall be used for weighing mass. The balance whose reciprocal sensibility is less than 0.001 g may be used.

4.2.2 Mercury thermometer The mercury thermometer which was approved in accordance with the Measurement Law and, of which the correction, if necessary, was further carried out shall be used.

4.2.3 Thermoelectric thermometer The thermoelectric thermometer shall consist of the following instruments.

- a) **Indicating instrument** The class 0.5 moving coil type indicator specified in JIS C 1601 shall be used.
- b) **Thermocouple** One of the following two thermocouples specified in JIS C 1602 shall be used. R (or S) class 0.25 thermocouple (0.5 mm in diameter, 500 mm or 1 000 mm long). K [old symbol (CA)] class 0.4 or class 0.75 thermocouple [2.3 mm or 3.20 mm in diameter⁽¹⁾, 500 mm or 1 000 mm long].

Note ⁽¹⁾ The thermocouple of 1.60 mm diameter may be used, but in this case it shall be verified every month.
- c) **Protection tube for thermocouple** One of the following two protection tubes specified in JIS R 1401 shall be used. Porcelain protection tube (PT2). Quartz glass protection tube (QT).
- d) **Compensating lead wires** The compensating lead wires specified in JIS C 1610 shall be used.
- e) **Reference junction apparatus** The freezing point type reference apparatus specified in JIS Z 8705 shall be used. The apparatus with a reference junction compensating circuit shall not be used.

Though a room temperature type reference junction apparatus may be used, the temperature of reference junction shall be measured by using other thermometer and corrected, in this case.

Remarks : When the temperature is measured with a thermoelectric thermometer, the grade B of measuring systems in **JIS Z 8704** shall be applied.

4.3 Reagent and water The reagents and water to be used shall be those specified in **JIS M 8810** unless otherwise specified in each item.

4.4 Expression of analytical result

4.4.1 Basis of measured value and report value The expression of basis shall be as follows:

- a) **For coal** Four components; moisture content, ash content, volatile matter content and fixed carbon shall be expressed by the air dried basis, or three components; ash content, volatile matter content and fixed carbon shall be expressed by anhydrous basis, apart from the above-mentioned. Basis (air dried, anhydrous) shall be appended to the measured values and report. The calculation of analytical values on anhydrous basis shall be made in accordance with **6.5 a) 2)**, **7.2.5 a) 2)** and **8 a) 2)**.
- b) **For coke** Three components; ash content, volatile matter content and fixed carbon shall be expressed on the anhydrous basis. The calculation of analytical values shall be made in accordance with **6.5 b)**, **7.2.5 b)** and **8 b)**.

Remarks 1 When it is necessary to convert each basis into other basis, an abbreviated symbol of the converted basis shall be appended. The conversion method and abbreviated symbol shall be in accordance with clause **8** and clause **9** of **JIS M 8810**.

- 2 For coal, application of either air dried basis or anhydrous basis shall be subjected to the agreement between the purchaser and the supplier, and the name of basis adopted to measurement item shall be appended.

4.4.2 Expression of results The expression of the results shall be in accordance with clause **8** of **JIS M 8810**. The air dried basis shall be used in this case.

4.4.3 Tolerance application method on analytical precision

- a) **Tolerance within the same laboratory** The tolerances (measured values) within the same laboratory shall be specified in the subclause for the tolerance for each method. The tolerance application method shall be in accordance with clause **10** of **JIS M 8810**.
- b) **Tolerance between different laboratories** The tolerance between different laboratories (anhydrous basis) shall be as given in table 1.

Table 1 Tolerance⁽²⁾

Unit: mass percentage (%)

Analysis item	Division		Tolerance between different laboratories (report value)
Ash content	10.0 or under		0.4
	10.1 to 20.0		0.6
	20.1 or over		0.8
Volatile matter content	Heating loss ⁽³⁾	50.0 or under	1.4
		50.1 or over	1.7
Volatile matter content	High temperature carbonization coke		0.5

Notes ⁽²⁾ For coal, the tolerances shall be applied to the values converted to the same water content (air dried basis), for coke, the tolerances shall be applied to anhydrous basis.

⁽³⁾ This tolerance shall be applied to coal. However, coke other than high temperature carbonization coke shall be included.

Remarks 1 For moisture content, tolerance is not specified since it depends on the humidity in a laboratory.

2 The tolerance within the same laboratory (measured values) shall be specified in the subclause for the tolerance for each method.

3 The tolerance between different laboratories shall be applied to the difference of report values between different laboratories with regard to the same analytical samples. The same analytical samples in this case refer to the samples prepared in accordance with 9.7.3.4 of JIS M 8811.

4 JIS M 8810 shall apply to all items other than those specified in this Standard.

5 Determination of moisture content

5.1 Classification and application of method

- Measuring method of loss on drying in air** This method shall be applied to coal with high coalification (bituminous coal, anthracite, etc.) and coke.
- Measuring method of loss on drying in helium gas flow** This method shall be applied to coal with low coalification (brown coal, and others).
- Measuring method of loss on drying in nitrogen gas flow** This method shall be applied to coal with high coalification (bituminous coal, anthracite, etc.).
- Azeotropic distillation method (direct volumetric method)** This method shall be applied to coal.

5.2 Measuring method of loss on drying in air

5.2.1 Summary When the sample of coal (coal with high coalification) is dried by heating for 1 h at 107 °C, and that of coke for 4 h at 200 °C, the mass percentage (%) of the loss on drying to the sample shall be taken as moisture content.

5.2.2 Apparatus and instrument The apparatus and instruments shall be as follows.

- a) **Thermometer** In accordance with 4.2.2.
 - b) **Drying apparatus**⁽⁴⁾ The drying apparatus shall be provided with the following conditions (see attached figure 1).
 - 1) The atmosphere in the drying chamber can be well-ventilated.
 - 2) The temperature measured at a bottom of the sample container inserted can be maintained at the specified heating temperature⁽⁴⁾.
 - 3) The temperature can be regained within 10 min after insertion of the sample.
 - c) **Drying container** The flat-bottom weighing bottle (nominal dimensions 40 mm × 20 mm) specified in attached figure 57 of **JIS R 3503** (see attached figure 2).
- Note ⁽⁴⁾ The electric thermostat with the automatic temperature regulator should be used.

It should be capable of regulating and maintaining the temperature at 107 °C ± 2 °C for coal, and at 200 °C ± 10 °C for coke.

5.2.3 Test portion The sample shall be weighed by about 1 g to the nearest 0.1 mg.

5.2.4 Procedure The procedure shall be as follows:

- a) **For coal**
 - 1) Raise the temperature of drying apparatus and adjust the temperature to 107 °C ± 2 °C in the drying chamber.
 - 2) Weigh out the sample into a drying container whose mass is known, level off the surface and cover with a lid.
 - 3) Take off the lid of the drying container and insert the container into a drying chamber together with the lid.
 - 4) Heat for 1 h after inserting the sample.
 - 5) Cover the drying container with the lid, take out from the drying chamber and transfer it into a desiccator to cool.
 - 6) Weigh the mass to the nearest 0.1 mg immediately after cooling⁽⁵⁾.

Note ⁽⁵⁾ The cooling time should be within 20 min.
- b) **For coke**
 - 1) Raise the temperature of drying apparatus and adjust the temperature to 200 °C ± 10 °C in the drying chamber.

- 2) Weigh out the sample into the drying container whose mass is known, level off the surface and cover with a lid.
- 3) Take off the lid of the drying container and insert it into a drying chamber together with the lid.
- 4) Heat for 4 h after inserting the sample.
- 5) Cover the drying container with the lid, take out from the drying chamber and transfer it into a desiccator to cool.
- 6) Weigh the mass to the nearest 0.1 mg immediately after cooling⁽⁵⁾.

5.2.5 Calculation of measured value The moisture content shall be measured to three decimal places according to the following formula and rounded off to two decimal places in accordance with **JIS Z 8401**.

$$M_s = \frac{m_1 - m_2}{m_0} \times 100$$

where, M_s : moisture content in the sample [mass percentage (%)]

m_1 : mass of the container and the test portion before drying (g)

m_2 : mass of the container and the test portion after drying (g)

m_0 : mass of the test portion weighed out (g)

5.2.6 Number of measurements This operation shall be repeated two times in the same laboratory. If the difference between two measured values exceeds the tolerance, comply with **JIS M 8810**. The operation may be repeated not less than two times at the same time by the use of a different drying apparatus but it shall not be repeated simultaneously by the use of the same drying apparatus.

5.2.7 Tolerance The tolerance on moisture content measured with the method of the loss on drying in air shall be as given in table 2.

Table 2 Tolerance on moisture content within the same laboratory

Unit: mass percentage (%)	
Moisture content	Tolerance (measured value)
Not more than 5.0	0.20
5.1 to 10.0	0.30
10.1 to 16.0	0.40
Not less than 16.1	0.50

5.2.8 Expression of report value If the difference between two measured values is within the tolerance, the moisture content shall be reported as the average of those values, rounded off to one decimal place.

5.3 Measuring method of loss on drying in helium gas flow

5.3.1 Summary When the sample (coal with low coalification) is dried by heating for 1 h at 107 °C in helium gas flow, the mass percentage (%) of the loss on drying to the sample shall be taken as moisture content.

5.3.2 Reagent The reagents shall be as follows:

- a) **Helium gas** The purity shall be not less than 99.995 %.
- b) **Toluene** As specified in **JIS K 8680**.

5.3.3 Apparatus and instrument The apparatus and instrument shall be as follows:

- a) **Drying apparatus** (see attached figure 3 and attached figure 4) The apparatus prepared by remodeling the Abderhalden's apparatus capable of blowing helium gas at a rate of 150 ml/min through an innermost part of the drying chamber and exhausting the waste gas containing moisture through a front side of the chamber.
- b) **Drying container** (see attached figure 5) The flat-bottom weighing bottle (nominal dimensions 40 mm × 20 mm) specified in **JIS R 3503**.

5.3.4 Test portion The sample shall be weighed out by about 1 g to the nearest 0.1 mg.

5.3.5 Procedure The procedure shall be as follows:

- a) After boiling toluene in the flask which is connected with the drying chamber of the Abderhalden's apparatus and cooling the vapour of it to condense in a condenser, adjust the fall down rate of the liquid drops to two or three drops per second. Furthermore, the exterior of double tube shall be kept warm by a ribbon heater so as not to condense the steam in the double tube.
- b) Weigh the sample into the drying container whose mass is known, and level off the surface of it.
- c) Take off the lid of the drying container and place it, together with the lid, on a bed for weighing bottle of the drying apparatus and insert into a drying chamber.
- d) Blow helium gas at a rate of 150 ml/min through an innermost part of the drying chamber and exhaust the waste gas containing moisture through a front side of the chamber.
- e) Heat for 1 h after inserting the sample.
- f) Take out the drying container from the drying chamber and transfer it into a desiccator after covering with the lid to cool.
- g) Weigh the mass to the nearest 0.1 mg immediately after cooling⁽⁵⁾.

5.3.6 Calculation of measured value In accordance with the measuring method of the loss on drying in air in 5.2.5.

5.3.7 Number of measurements In accordance with the measuring method of the loss on drying in air in 5.2.6.

5.3.8 Tolerance In accordance with the measuring method of the loss on drying in air in 5.2.7 (see table 2).

5.3.9 Expression of report value In accordance with the measuring method of the loss on drying in air in 5.2.8.

Remarks : When the moisture content is measured in accordance with this method, the abbreviated symbol (HM) shall be appended.

5.4 Measuring method of loss on drying in nitrogen gas flow

5.4.1 Summary The sample (coal with high coalification) shall be heated at 107 °C in nitrogen gas flow, and maintained at this temperature until constant in mass. The percentage moisture content shall be calculated from the loss in mass of the coal.

5.4.2 Reagent The reagent shall be as follows:

a) **Nitrogen gas** Class 1 of high purity nitrogen specified in **JIS K 1107**.

5.4.3 Apparatus and instrument The apparatus and instrument shall be as follows:

- a) **Drying apparatus** (see attached figure 6 and attached figure 7) This shall be capable of controlling the temperature at $107\text{ °C} \pm 2\text{ °C}$. Furthermore, this shall be structured so that the volume of nitrogen of 15 times the volume of the furnace can be flowed for 1 h. The actual volume should be as small as possible.
- b) **Drying container** This shall be made of thin glass or corrosion-resistant metal, and shall have a lid capable of making airtight. For size, the coal layer shall not exceed 0.20 g/cm^2 .

5.4.4 Test portion The sample shall be weighed by about 1 g to the nearest 0.1 mg.

5.4.5 Procedure The procedure shall be as follows:

- a) Weigh the sample into the drying container whose mass is known, level off the surface of it and cover with a lid.
- b) Take off the lid of the drying container, and insert the drying container into a drying chamber together with the lid.
- c) Heat for 1 h after inserting the sample. At this time, flow nitrogen gas of 15 times the volume of the drying apparatus per 1 h. Furthermore, do not open the door of the drying apparatus during drying.
- d) Take out the drying container from the drying chamber and immediately cover with the lid.
- e) Transfer it into a desiccator and cool to room temperature.

f) Weigh the mass to the nearest 0.1 mg immediately after cooling.

- Remarks
- 1 When taking out the drying container from the drying chamber, if the volume of the drying apparatus is large enough, cover the drying container with the lid before taking it out.
 - 2 When using a corrosion-resistant metal container, at the time of cooling, after cooling for 10 min on a metal plate, transfer it to a desiccator to cool. However, when using the cooling container capable of flowing nitrogen gas, it may be transferred directly to the cooling container.
 - 3 When there is a possibility that the drying is not enough, heat again for 30 min until the mass variability does not exceed 1 mg.

5.4.6 Calculation of measured value In accordance with the measuring method of the loss on drying in air in **5.2.5**.

5.4.7 Number of measurements In accordance with the measuring method of the loss on drying in air in **5.2.6**.

5.4.8 Tolerance In accordance with the measuring method of the loss on drying in air in **5.2.7** (see table 2).

5.4.9 Expression of report value In accordance with the measuring method of the loss on drying in air in **5.2.8**.

Remarks : When the moisture content is measured in accordance with this method, the abbreviated symbol (NM) shall be appended.

5.5 Azeotropic distillation method (direct volumetric method)

5.5.1 Summary When the sample is heated under the reflux conditions with toluene or xylene, the mass percentage (%) of the distilled water to the sample shall be taken as moisture content.

5.5.2 Reagents The reagents shall be as follows:

WARNING : Care shall be taken to ensure that the following reagents are handled in the well-ventilated place with protective gloves worn.

- a) **Toluene** As specified in **JIS K 8680**.
- b) **Xylene** As specified in **JIS K 8271**.

5.5.3 Apparatus and instrument The apparatus and instrument shall be as follows:

- a) **Distillation apparatus** The apparatus shown in the attached figure 8 is appropriate, but the apparatus specified in **JIS K 2275** or **JIS K 2435** may be used.

Whichever apparatus is used, it shall be calibrated by distilling the prepared solvent as follows:

- Take several amounts of water with whole pipettes of various volumes which cover the expected range of moisture content in the analytical sample.
- Add the series of water into each solvent whose amount is specified.

The working curve shall be plotted by indicating the volume of the added water in millimetres against the scale reading of water recovered in the receiver. In order to correct the reading amount of water in analysis, this working curve should be used.

- b) **Receiver** One whose volume is 5 ml, 10 ml, 20 ml or 30 ml graduated with 0.1 ml shall be used.

The receiver shall be constituted so that the moisture generated by the azeotropic distillation and condensed by a condenser is returned into the receiver and toluene or xylene is refluxed from the receiver to the distilling flask.

Remarks : It is important that the receiver and the condenser shall be cleansed thoroughly before use. For this reason, it is necessary to wash them with suitable detergent for removing organic solvent, fats and oil which adhered to the container.

5.5.4 Test portion The sample shall be weighed by about 50 g to the nearest 0.01 g. If the moisture content is expected to exceed 20 %, the sample weighed shall be about 25 g.

5.5.5 Procedure The procedure shall be as follows:

- a) **Analytical sample** Weigh the sample and transfer it into a dried distilling flask. Add 200 ml of toluene or xylene into the flask in such a way that the sample attached to the neck or the side wall of the flask is rinsed off.
- b) **Measurement** Fill the receiver with the same toluene or xylene as poured into the distilling flask. Assemble the apparatus after putting two or three small glass tube pieces⁽⁶⁾ in the distilling flask to avoid bumping. Pass the cooling water to the condenser and heat the flask gradually so that the content in the flask begins to boil after about 15 min. Then, adjust the heating rate so that two or three drops of condensate fall down per second. Continue the distillation until toluene or xylene is refluxed and the amount of water recovered in the receiver is kept constant. Rinse off water drops attached to the inside wall of the condenser and the upper wall of the receiver with the same solvent by using a spray tube. Continue distillation for sufficient time until water returned in the distilling flask is recovered into the receiver. Read the volume of water recovered in the receiver when cloudiness of the distillates becomes the same state as that when the scale correction of the receiver was performed.

Note ⁽⁶⁾ Small pieces with a sharp cut of about 5 mm in diameter and about 5 mm long shall be used.

5.5.6 Calculation of measured value The moisture content shall be measured to three decimal places according to the following formula and rounded off to two decimal places in accordance with JIS Z 8401.

$$M_s = \frac{V \times \rho_{aq}}{m_0} \times 100$$