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Coke—Testing methods

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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 K 2151 : 1993** is replaced with this Standard.

This revision has been made based on **ISO 567 : 1995** *Coke—Determination of bulk density in a small container*, **ISO 616 : 1995** *Coke—Determination of shatter indices*, **ISO 728 : 1995** *Coke (nominal top size greater than 20 mm)—Size analysis by sieving*, **ISO 1014 : 1985** *Coke—Determination of true relative density, apparent relative density and porosity* and **ISO 2325 : 1986** *Coke—Size analysis (Nominal top size 20 mm or less)* 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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Coke—Testing methods

Introduction This Japanese Industrial Standard has been prepared based on the second edition of **ISO 567** *Coke—Determination of bulk density in a small container* published in 1995, the second edition of **ISO 616** *Coke—Determination of shatter indices* published in 1995, the third edition of **ISO 728** *Coke (nominal top size greater than 20 mm)—Size analysis by sieving* published in 1995, the second edition of **ISO 1014** *Coke—Determination of true relative density, apparent relative density and porosity* published in 1985 and the third edition of **ISO 2325** *Coke—Size analysis (Nominal top size 20 mm or less)* published in 1986 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 method for the particle-size analysis, the method for the determination of the bulk density, the method for determination of the density and porosity, the method for determination of the shatter indices, the method for determination of the drum indices, the method for the reactivity test and the method for the fusibility-of-ash test.

Remarks : The International Standards corresponding to this Standard is as follows.

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

ISO 567 : 1995 *Coke—Determination of bulk density in a small container* (MOD),

ISO 616 : 1995 *Coke—Determination of shatter indices* (MOD),

ISO 728 : 1995 *Coke (nominal top size greater than 20 mm)—Size analysis by sieving* (MOD),

ISO 1014 : 1985 *Coke—Determination of true relative density, apparent relative density and porosity* (MOD), and

ISO 2325 : 1986 *Coke—Size analysis (Nominal top size 20 mm or less)* (MOD)

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

JIS K 0557 *Water used for industrial water and wastewater analysis*

JIS K 8646 *Dextrin hydrate*

JIS M 0104 *Technical terms used in coal utilization*

JIS M 8810 *Coal and coke—General rules for sampling, analysis and testing*

JIS M 8811 *Coal and coke—Sampling and sample preparation*
JIS M 8812 *Coal and coke—Methods for proximate analysis*
JIS M 8820 *Coal and coke—Determination of total moisture content of a lot*
JIS Z 0701 *Silicagel desiccants for packaging*
JIS Z 8401 *Guide to the rounding of numbers*
JIS Z 8801-1 *Test sieves—Part 1 : Test sieves of metal wire cloth*
JIS Z 8801-2 *Test sieves—Part 2 : Test sieves of perforated metal plate*

3 General matter

3.1 Scale The reciprocal sensibility of a scale, unless otherwise specified, shall be taken as 1/1 000 of weighing capacity. The scale which is near the weighing capacity according to the amount of sample shall be preferably used.

3.2 Handling of sample In accordance with clause 5 of JIS M 8810.

3.3 Rules for rounding of numerical values The calculation of measured value and the report value in each test shall be rounded in accordance with JIS Z 8401.

3.4 Report In accordance with clause 12 of JIS M 8810.

4 Definitions For the purpose of this Standard, the definitions given in JIS M 0104 and the following definitions apply.

- a) **gross sample** a sample which collected all increments taken from the sub-lot or the lot which is not divided
- b) **partial sample** a sample which collected two or more increments taken from the sub-lot or the lot which is not divided in order to measure the total moisture
- c) **increment** a part of the lot sampled in one action of a sampler or a part of the sample taken in accordance with the increment reduction method
- d) **nominal top size** the size of the minimum sieve opening (nominal size) of the test sieve (see square hole, JIS Z 8801-2) of which the plus-sieve residual percentage of a sample becomes not more than 5 %

5 Method for particle-size analysis

5.1 Principle of test A sample is screened with the predetermined sieve, and the residual amount on each sieve and the amount passing through the minimum sieve opening are weighed, and the particle size of the sample is expressed by the mass fraction (%) to the sample.

5.2 Sieve The required sieves selected from the sieves specified in JIS M 8801-1 and JIS Z 8801-2 are determined and used in accordance with the agreement between the parties concerned with delivery.

5.3 Sampling, preparation and drying

5.3.1 Sampling The primary increment of the mass specified in table 5-4 of **JIS M 8811** shall be taken by not less than the number of pieces specified in table 5-1 of **JIS M 8811**.

Remarks : The confirmation of the precision should be performed in accordance with clause 11 of **JIS M 8811**.

5.3.2 Preparation of sample Usually, the whole quantity every increment, every partial sample, without carrying out the reduction of gross sample as it is shall be taken as the particle-size measurement sample. However, when the mass of the sample for particle-size analysis exceeds twice the mass specified in table 5.1, it may be reduced to the mass in table 5.1. That is, in the case of the gross sample, it may be reduced to the mass in table 5.1. In the case of a partial sample and an increment, they may be reduced to m_P and m_1 which are obtained from the formula (1) and the formula (2).

$$n_P m_P = m_G \quad \text{..... (1)}$$

and

$$n_1 m_1 = m_G \quad \text{..... (2)}$$

where, m_P : mass of partial sample after reduction
 n_P : number of partial samples
 m_G : mass of gross sample specified in table 5.1.
 m_1 : mass of increment after reduction
 n_1 : number of pieces of increment

Table 5.1 Minimum mass of gross sample for particle-size analysis

Nominal top size mm	Minimum mass kg
Over 125	2 000
125	1 000
90	500
63	250
45	125
31.5	60
22.4	30
16	15
11.2	8
8	4
5.6	2
4	1

5.3.3 Drying of sample When the moisture of the sample which is taken and prepared in accordance with 5.3.1 and 5.3.2 exceeds 5 % of mass fraction, the sample shall be dried so that the moisture becomes not more than 5 % of mass fraction.

5.4 Procedure The procedure shall be performed as follows:

- a) Arrange the set of sieves in a stack in order of nominal hole sizes, with the smallest size at the bottom. Weigh the test sample, in several portions if necessary.
- b) Place on the top sieve a portion of the test sample small enough that not more than 75 % of the sieve area is covered, and shake to allow most of the undersize to pass. Manually place the remaining coke on the sieve and transfer the oversize to a suitable container. Remove the top sieve and repeat these operations for sieves of nominal hole sizes down to and including 40 mm.
- c) Continue the process described in b) for sieves of nominal hole sizes less than 40 mm, but using manual shaking. Carry out manual shaking by holding the sieve in the hands, or suspending it freely, and moving it horizontally to and fro with a displacement of about 75 mm. Complete fifty such oscillations (each consisting of one movement to and fro) in a period of about 30 s. If the amount of coke remaining on the sieve is then such that it covers more than 75 % of the sieve area, divide it into two or more portions and manually shake each portion separately. Otherwise, transfer the oversize to a suitable container.

Remarks : Mechanical sieving may be carried out if it can be demonstrated that the method is free from bias when compared to the manual sieving method.

- d) If necessary, re-stack the sieves and repeat the steps described in b) and c) for successive portions until the entire test sample has been sized and each size fraction has been collected in a separate container.
- e) Either weigh each container separately with its size fraction and subtract the respective tare mass, or, after weighing the first fraction, successively add the other fractions and note the cumulative mass after each addition.

Remarks : Cumulative weighing is preferable because it reduces the weighing error for the cumulative fractions. Depending on how the results are to be expressed (see 5.5), cumulative weighing should be in descending order of size fractions (cumulative oversize) or in ascending order of size fractions (cumulative undersize).

5.5 Expression of results Calculate the cumulative mass on each sieve starting with the sieve with the largest size of holes, or the cumulative mass passing through each sieve starting with the sieve with the smallest size of holes, from the masses of the individual size fractions.

Calculate the apparent loss, i.e. the difference between the total mass of the test sample before sieving and the total mass of coke collected afterwards. If the loss is greater than 0.4 % of the original mass of the test sample, reject the results and carry out a further test. Otherwise, add the apparent loss to the mass of the smallest size fraction.

Convert each cumulative mass to a percentage of the total mass of the test sample. Report the cumulative masses to the nearest 0.1 kg and the cumulative percentages to the nearest 0.1 %.

Remarks 1 The mean size of the coke may be calculated from the following formula:

$$\frac{B(a-c) + C(b-d) + \dots + J(h-k) + 100j}{200}$$

where,

$a, b, c, d, \dots, h, j, k$ are the hole sizes, in millimetres, of successive sieves;

$A, B, C, D, \dots, H, J, K$ are the cumulative percentage oversizes for each of the sieves.

The sieve with hole size a is the smallest size through which all the coke passes (i.e. $A = 0$ %). The sieve with hole size k is a hypothetical sieve through which no coke will pass (i.e. $k = 0$ mm, $K = 100$ %).

Alternative methods of calculation or graphical methods of determination may lead to slightly different results and the same method should, therefore, be used for comparative tests.

The precision of a determination of mean size can be expressed in terms of the repeatability limit, as follows.

The results of duplicate determinations of mean size, carried out at different times in the same laboratory, by the same operator with the same apparatus, on representative test samples taken from the same lot of coke, should not differ by more than 2.5 mm.

No value for reproducibility can be quoted for determinations carried out in different laboratories, since the transport of coke samples involves the risk of breakage and thus alteration of the size distribution.

2 An example of the calculation of the results of a size analysis of coke, in terms of the cumulative oversize, is given in table 5.2.

Table 5.2 Example of size analysis of coke (cumulative oversize)

Nominal size of hole (round hole) mm	Mass of fraction kg	Cumulative mass kg	Cumulative percentage oversize %
140	4.9	4.9	1.1
125	9.4	14.3	3.2
100	41.3	55.6	12.4
80	113.4	169.0	37.7
71	78.5	247.5	55.2
63	67.7	315.2	70.3
50	51.5	366.7	81.8
40	37.2	403.9	90.1
31.5	17.9	421.8	94.1
20	9.9	431.7	96.3
10	8.1	439.8	98.1
passing 10	7.4 + 1.1 = 8.5	448.3	100.0
Sum	447.2		
Original mass	448.3		
Loss in mass	1.1		
0.4 % of original mass	1.8		
The loss in mass, being less than 0.4 % of the original mass, is added to the fraction of smallest size (< 10 mm).			

6 Method for determination of bulk density

6.1 Principle A weighed container of known volume is filled with coke in accordance with the specified method and the increase in mass is determined. The total moisture of the sample is measured, and the measured value is converted into the mass of anhydrous coke and the bulk density is calculated from the value.

6.2 Sample A sample shall be taken in accordance with **JIS M 8811**. However, when the amount of lump coke of about 25 mm or more in the gross sample is less than 1 t, the sampled number of pieces of increment should be increased so that the amount becomes 1 t or more. It is desirable to screen the gross sample taken in accordance with clause 5 with the sieve of hole size of 25 mm, to collect those which remain on this sieve, and to take one-time sample of 250 kg or more by the increment reduction method, etc.

6.3 Apparatus

- Test container** A test container with internal dimensions of $794 \text{ mm} \pm 2 \text{ mm}$ (volume $0.5 \text{ m}^3 \pm 0.005 \text{ m}^3$) long, wide and deep, each.
- Container for insertion** It shall be a container made of steel, etc. with an internal volume of about 25 L.

6.4 Procedure The procedure shall be performed as follows:

- a) Take about 10 kg at random from every portion of the sample, divide the sample into each 25 or more pieces, and enter these into the container for insertion.
- b) Place the test container on the scale, measure the mass, and take this mass as A kg.
- c) Insert the sample in the container for insertion one by one from a height of about 30 cm above the container staying on the scale. Insert the sample firstly in the central part. During inserting, do not move the sample manually, or do not move the test container.
- d) Stop inserting when the sample is filled to the edge of the test container, and remove any pieces of coke of which more than half of the volume stick out from the horizontal plane of the test container.
- e) Weigh the mass of the test container with the coke staying in, and take this mass as B kg.
- f) Take out the sample, take the sample for total moisture measurement in accordance with **JIS M 8811**, prepare it, and measure the total moisture in accordance with **JIS M 8820**.
- g) For the residual sample from which the sample for total moisture measurement was taken, the particle-size analysis may be performed.

6.5 Calculation of bulk density The bulk density from sample coke mass (C) and anhydrous coke mass (D) shall be calculated to be rounded to two decimal places according to the following formula.

$$\text{Sample coke mass } C = B - A$$

$$\text{Anhydrous coke mass } D = C \times (100 - \text{total moisture}) / 100$$

$$D_b = \frac{D}{0.5 \times 1\,000}$$

where, A : mass of test container (kg)
 B : mass when filling the sample in the test container (kg)
 C : sample coke mass (kg)
 D : anhydrous coke mass (kg)
 D_b : bulk density of coke (kg/m³)

6.6 Expression of results The test shall be performed repeatedly twice in the same laboratory.

When the difference between the measured values of 2 pieces in the same laboratory is less than the tolerance ($n = 2$) in **6.7**, the average value of the measured values of 2 pieces shall be treated according to the following a). However, when not less than the tolerance ($n = 2$), further one-time test shall be additionally performed,

and when the range (the maximum value – the minimum value) of the measured values of 3 pieces is less than the tolerance ($n = 3$), the average value of the measured values of 3 pieces shall be treated according to the following **a)**. When not less than the tolerance ($n = 3$), the median shall be treated according to the following **b)**.

- a) The average value shall be rounded to two decimal places, and expressed as the bulk density.
- b) In the case of the median, it shall be expressed as the bulk density with the two digits after the decimal place being as they are, and that (the median value) shall be described together. Furthermore, the particle-size distribution shall be also described together.

Remarks : The cylindrical container may be used as the test container. In this case, the container made of steel plate of $914 \text{ mm} \pm 2 \text{ mm}$ in inside diameter, $761 \text{ mm} \pm 2 \text{ mm}$ high and 6 mm thick shall be used, and the use of the cylindrical container shall be described.

6.7 Repeatability limit The repeatability limit of the bulk density of the repetitive test result in the same laboratory should be within the range in table 6.1.

Table 6.1 Repeatability limit

Coke particle size mm	$n=2$	$n=3$
25 min.	0.02	0.02

7 Method for determination of density and porosity

7.1 Principle of test After drying the sample and measuring mass, immerse it in a water bath, measure the mass of water which was removed and the mass of hydrous coke are measured, and calculate the apparent density from these values.

For a true density, measure it with a pycnometer using the finely broken sample. A true density here is for calculating porosity. For the porosity, obtain a pore volume from an apparent density and a true density, and express it by the fraction (%) to the sample volume.

7.2 Method for determination of apparent density

7.2.1 Sample A sample shall be taken in accordance with **JIS M 8811**. However, when the amount of lump coke of about 25 mm or more in the gross sample is less than 40 kg, the sampled number of pieces of increment should be increased so that the amount becomes 40 kg or more. The gross sample taken in accordance with clause 5 shall be screened with the sieve of hole size of 25 mm, those which remain in this sieve shall be collected, and the one-time sample of 10 kg or more shall be taken by the increment reduction method, etc.

7.2.2 Apparatus The apparatus shall be as follows:

- a) **Water tank** The water tank made of steel plate of 2 mm or more thick with inside dimensions of 330 mm high, 560 mm long, 280 mm or more wide shall be used. The one short tube of 13 mm in inside diameter shall be attached as a spout of water at a distance of about 65 mm downward from the upper end.
- b) **Cage** A cage made of metallic mesh with hole size of about 13 mm attached with a cover and two long handles shall be capable of containing about 10 kg of the sample, and furthermore entering thoroughly under the spout of water tank.
- c) **Receiver** Capable of receiving 10 L of water.
- d) **Plate** A plate made of steel plate with a size of about 380 mm long, wide each, and 75 mm high.
- e) **Dryer** The dryer should be equipped with a thermostat and a blower.

7.2.3 Procedure The procedure shall be performed as follows:

- a) Dry the sample at $150\text{ }^{\circ}\text{C} \pm 20\text{ }^{\circ}\text{C}$ until the mass will become fixed, and after drying measure the mass after wiping powder dust well, and take this mass as A kg.
- b) Place the water tank almost horizontally, put a cork stopper in the spout, pour the water of room temperature to the level above the spout after putting an empty cage, and shake the cage several times.
- c) Remove the cork stopper after the water surface stands still, let the excessive water run off. After leaving for 1 min from the time when water begins to trickle after water finished mostly running off, put the cork stopper again.
- d) Take out the cage from the water tank. At this time shake off the adhered water well in the water tank.
- e) Put the sample in the cage, put down calmly in the water tank after covering. Immerse the cage in the water tank for 15 min shaking it sometimes in order to remove air bubbles adhered on the surface of coke. At this time take care so that the water tank does not move.
- f) Remove the cork stopper after the water surface stands still, and let the excessive water run in the receiver of mass known. After leaving for 1 min from the time when water begins to trickle after water finished mostly running off, put the cork stopper again.
- g) Take out the cage from the water tank, and after leaving it for 1 min while water trickles, take out the sample from the cage, transfer it to the plate of mass known, and weigh the mass of the hydrous coke. Take this mass as B kg.
- h) Measure the mass of water removed in the receiver. Take this mass as C kg.

7.2.4 Calculation of apparent density The apparent density shall be calculated to be rounded to two decimal places according to the following formula.

$$D_a = \frac{A}{B + C - A}$$

- where, D_a : apparent density (kg/m³)
 A : anhydrous sample mass (kg)
 B : hydrous sample mass (kg)
 C : mass of the water removed in the receiver (kg)

7.2.5 Expression of results The test shall be performed repeatedly twice in the same laboratory.

When the difference between the measured values of 2 pieces in the same laboratory is less than the tolerance ($n = 2$) in 7.2.6, the average value of the measured values of 2 pieces shall be treated according to the following a). However, when not less than the tolerance ($n = 2$), further one-time test shall be additionally performed, and when the range (the maximum value – the minimum value) of the measured values of 3 pieces is less than the tolerance ($n = 3$), the average value of the measured values 3 pieces shall be treated according to the following a). When not less than the tolerance ($n = 3$), the median value shall be treated according to the following b).

- a) The average value shall be rounded to two decimal places, and expressed as the apparent density.
- b) In the case of the median value, it shall be expressed as the apparent density with the two digits after the decimal place being as they are, and that (median value) shall be described together.

Information : **JIS K 2151** : 1993 had also prescribed the following two methods in addition.

Volume method The method for determination by measuring the water volume removed by the sample.

- a) **Sample** It is the same as 7.2.1.
- b) **Apparatus** See figure 7.1.
- c) **Procedure** Perform the same procedure as that in 7.2.3, but read the graduation of the water level tube when the sample encased in the cage is immersed in the water tank. Take this as E . Take out the sample from the cage, and immerse the cage in the water tank again, and read the graduation of the water level tube. Take this as F .
- d) **Calculation of measured value** The measured value shall be calculated to be rounded to two decimal places according to the following formula.

$$D_a = \frac{D}{G \times (E - F)}$$

- where, D_a : apparent density (kg/m³)
 D : anhydrous sample mass (kg)

E : water level with the sample encased in the cage immersed in the water tank (mm)

F : water level with only the cage immersed in the water tank (mm)

G : mass of water which indicates one graduation (kg)

- e) The number of times of tests, tolerance, report value are the same as those in 7.2.5 to 7.2.6. When the measurement is performed with this method, that (volume method) shall be described together.

Small-quantity method The method used when the amount of sample is small.

- a) **Sample** Cut out one cube of a side of about 20 mm from the centre section of the lump coke, and take this as the sample.

The number of pieces of sample shall be at least three or more.

- b) **Procedure** The procedure shall be performed as follows:

- 1) Add water of A2 or A3 specified in **JIS K 0557** in a beaker (capacity of 500 ml) in such a degree that the sample is immersed in water, and boil the sample for about 4 h to remove air in coke.
- 2) After cooling, transfer the sample in a beaker (capacity of 100 ml) with water kept at 15 °C.
- 3) Bind the sample with the hair, etc. which were made clean, hang at the hook of balance pan, put in almost central part of the beaker, weigh mass in water, and take this mass *H* g.
- 4) Next, pull up coke from water calmly, remove water drop-let, put it into the weighing bottle of mass known, measure the mass of hydrous coke, and take this mass as *K* g.
- 5) Take out the sample from the weighing bottle, put it in an evaporating dish, heat until the mass becomes fixed at 150 °C ± 20 °C, put it in a desiccator, and after cooling weigh the anhydrous coke. Take this mass as *L* g.

- c) **Calculation of measured value** The measured value shall be calculated to be rounded to two decimal places according to the following formula.

$$D_a = \frac{L}{K - H}$$

where, *D_a* : apparent density (g/cm³)

H : mass in water of sample (g)

K : hydrous sample mass (g)

L : anhydrous sample mass (g)

- d) **Number of times of tests** The test shall be repeatedly performed in the same laboratory three times or more.
- e) **Report value** The average value of the measured values of 3 pieces or more shall be rounded to two decimal places, and expressed as the apparent density.

Remarks : When the measurement is performed by this method, that (small-quantity method) shall be described together.

7.2.6 Repeatability limit The repeatability limit of the apparent density of the repetitive test result in the same laboratory shall be 0.03 in the case of $n = 2$, 0.04 in the case of $n = 3$.

7.3 Method for determination of true density

7.3.1 Sample Take the sample in accordance with **JIS M 8811**, take the sample of about 10 g by performing the breaking reduction, break this to a size of 212 μm or less, and take one sample of about 2 g by the increment reduction method.

Remarks : When the porosity is obtained by measuring the apparent density and true density, the sample used for the determination of apparent density shall be used for the determination of true density by performing the breaking reduction.

7.3.2 Apparatus The apparatus shall be as follows:

- a) **Pycnometer** A pycnometer with a capacity of volume of 50 ml, to which the thin tube with a marked line shall be attached, and of which the mouth shall have a size capable of inserting a mercury thermometer (see figure 7.2). Since it is influenced by heat, at the beginning it should be stabilized by heating at about 170 °C.
- b) **Dryer** A suitable dryer capable of controlling at $150\text{ °C} \pm 10\text{ °C}$ shall be used.

7.3.3 Procedure The procedure shall be performed as follows:

- a) Dry the sample at $150\text{ °C} \pm 10\text{ °C}$ for 1 h, and after cooling weigh the mass. Take this mass as M g.
- b) Put the sample into the pycnometer calmly, then pour water⁽¹⁾ of A3 specified in **JIS K 0557** of half the amount of sample, and boil calmly and carefully on the sound plate for 1 h. In the meantime, supplement water of A3 sometimes, and wash out the sample adhered to the wall of pycnometer.
- c) After boiling, put down the pycnometer from the sound plate, and after filling the pycnometer by adding water of A3 specified in **JIS K 0557**, cool to room temperature.
- d) After adding the distilled water of room temperature to the upper edge of the mouth of pycnometer, attach a thin tube by inserting it into the mouth of