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Natural rubber (NR) — Evaluation
procedure

ICS 83.060

Descriptors : natural rubber, volatile matter determination, ash determination,
gums, determination of content, physical testing, chemical analysis
and testing

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law:

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Natural rubber (NR) — Evaluation procedure

Introduction This Standard is Japanese Industrial Standard established based on the corresponding International Standards as shown in Remarks 2 in 1 Scope without any change of technical contents, except that the test methods of volatile matter, ash content, dirt content, acetone extract and manganese content specified in the old edition of Japanese Industrial Standard are supplemented as the specified test items.

1 Scope This Standard specifies the chemical and physical test methods for natural rubber (NR). However, to the test method for latex, this Standard shall not apply.

Remarks 1 The normative references to this Standard are as follows.

- JIS H 2107 *Zinc metal*
- JIS K 0121 *General rules for atomic absorption spectrochemical analysis*
- JIS K 1410 *Zinc oxide*
- JIS K 3331 *Hardened oils and fatty acids for industrial use*
- JIS K 6200 *Glossary of terms used in rubber industry*
- JIS K 6202 *Organic vulcanization accelerators for rubber*
- JIS K 6222 *Sulfur powder for rubber*
- JIS K 6250 *General rules of physical testing methods for rubber, vulcanized or thermoplastic*
- JIS K 6251 *Tensile testing methods for vulcanized rubber*
- JIS K 6299 *Rubber test mixes—Preparation, mixing and vulcanization—Equipment and procedures*
- JIS K 6300 *Physical testing methods for unvulcanized rubber*
- JIS P 3801 *Filter paper (for chemical analysis)*
- JIS R 3503 *Glass apparatus for chemical analysis*
- JIS R 3505 *Volumetric glassware*
- JIS Z 8401 *Rules for rounding off of numerical values*
- JIS Z 8801 *Test sieves*

2 The corresponding International Standards to this Standard are as follows.

- ISO 37:1994 *Rubber, vulcanized or thermoplastic—Determination of tensile stress-strain properties*
- ISO 247:1990 *Rubber—Determination of ash*
- ISO 248:1991 *Rubbers, raw—Determination of volatile-matter content*

- ISO 249:1987 *Rubber, raw natural—Determination of dirt content*
- ISO 289-1:1994 *Rubber, unvulcanized—Determinations using a shearing-disc viscometer—Part 1: Determination of Mooney viscosity*
- ISO 471:1995 *Rubber—Temperatures, humidities and times for conditioning and testing*
- ISO 1407:1992 *Rubber—Determination of solvent extract*
- ISO 1656:1988 *Rubber, raw natural, and rubber latex, natural—Determination of nitrogen content*
- ISO 1658:1994 *Natural rubber (NR)—Evaluation procedure*
- ISO 1795:1992 *Rubber, raw, natural and synthetic—Sampling and further preparative procedures*
- ISO 2007:1991 *Rubber, unvulcanized—Determination of plasticity—Rapid-plastimeter method*
- ISO 2393:1994 *Rubber test mixes—Preparation, mixing and vulcanization—Equipment and procedures*
- ISO 2930:1995 *Rubber, raw natural—Determination of plasticity retention index (PRI)*
- ISO 3417:1991 *Rubber—Measurement of vulcanization characteristics with the oscillating disc curemeter*
- ISO 4660:1991 *Rubber, raw natural—Colour index test*
- ISO 6101-3 :1988 *Rubber—Determination of metal content by atomic absorption spectrometry—Part 3: Determination of copper content*
- ISO 6101-4 :1988 *Rubber—Determination of metal content by atomic absorption spectrometry—Part 4: Determination of manganese content*
- ISO 6502:1991 *Rubber—Measurement of vulcanization characteristics with rotorless curemeters*
- ISO 7780:1987 *Rubbers and rubber latices—Determination of manganese content—Sodium periodate photometric methods*

- 3 The units and numerical values given in { } in this Standard are based on the traditional units and are appended for informative reference.

2 Definitions For the purposes of this Standard, the definitions given in **JIS K 6200** and the following definitions apply.

- (1) **lot** The lot herein means unit group⁽¹⁾ of the bales classified by some common features capable of being clearly divided.

Note ⁽¹⁾ Unless there is any clear sign of being different in nature, all the bales with the same mark may be considered to be one lot.

- (2) **primary sample** The primary sample herein means unit subgroup of the bales selected as the representative of one lot.
- (3) **secondary sample** The secondary sample herein means the rubber sampled from the bale of the primary sample for representing the bale.
- (4) **test piece** The test piece herein means the rubber sampled from unit secondary sample or some secondary samples to be served for the test.

3 Test items The test items specified in this Standard shall be as follows.

(1) Chemical test

- (a) Volatile matter
- (b) Ash content
- (c) Dirt content
- (d) Rubber hydrocarbon
- (e) Acetone extract
- (f) Copper content
- (g) Manganese content
- (h) Nitrogen content

(2) Physical test

- (a) Colour index
- (b) Plasticity (P_0)
- (c) Plasticity retention index (PRI)
- (d) Mooney viscosity
- (e) Vulcanization test with the oscillating disc curemeter
- (f) Tensile test

4 Sampling method

4.1 Method for selecting primary sample Although the more numbers of bale for primary sample represent the lot so much accurately, the amount to be selected is actually limited. Therefore the number of bale to be selected shall be subjected to the agreement between the parties concerned with delivery. If applicable, the statistical sampling inspection method shall be adopted.

For the bale to be a primary sample, the test shall be carried out on every bale and the results shall be recorded.

4.2 Sampling method of secondary sample The secondary sample should be taken from primary sample according to the following method. After removing wrapping material such as polyethylene film, protective material and adhered matter on surface from a bale, cut the bale vertically at the maximum area of surface. At this time, any lubricant shall not be used. Cut off a secondary sample from cut bale at the central portion.

If not being cut off from the central portion of bale, a secondary sample may be cut off from an appropriate portion of bale.

In any case, the sampling mass of secondary sample shall be 600 g to 1 500 g as corresponding to the test item to be carried out. For chip or powder type of primary sample, the sampling from package by the similar method may be allowed.

When the secondary sample to be taken is not served to the test immediately, store it in air-tight container not exceeding twice the sample volume or wrap it doubly with aluminium foils.

4.3 Homogenization of secondary sample The homogenization of secondary sample shall be carried out by either Method A or Method B as follows.

- (1) **Method A** Pass a sample of 600 g to 1 000 g as being rounded 10 times between the test mixing rolls specified in 6, of which the surface temperature of roll is $70^{\circ}\text{C} \pm 5^{\circ}\text{C}$ and the opening of rolls is $1.30 \text{ mm} \pm 0.15 \text{ mm}$.
- (2) **Method B** Pass a sample of 360 g to 400 g as being rounded 6 times between the test mixing rolls specified in 6, of which the surface temperature of roll is room temperature as being cooled with water and the opening of rolls is $1.65 \text{ mm} \pm 0.15 \text{ mm}$.

5 Reagent and glassware used in chemical test For the reagent to be used in analysis, the most excellent grade specified in JIS or the like shall be used. For the glassware for chemical analysis, the glassware specified in JIS R 3503 and JIS R 3505 shall be used.

Silica gel shall be contained in the desiccator used in this Standard.

6 Test mixing roll For a test mixing roll (hereafter referred to as "mixing roll") and an intensive mixer, the mixer specified in 4.1 and 4.2 of JIS K 6299 shall be used in principle. The using of other mixing equipment shall be subjected to the agreement between the parties concerned with delivery.

7 Chemical test method

7.1 Volatile matter Weigh the mass (mass: m_1) taken from secondary sample according to 4.2 to 0.1 g, homogenize it according to 4.3 and then weigh the mass (mass: m_2) again to 0.1 g. Carry out the weighing of mass after cooled to room temperature as it is.

Take approximately 10 g of test piece from homogenized secondary sample and weigh the mass (mass: m_3) to 1 mg. Adjust the roll so that the roll temperature is $70^{\circ}\text{C} \pm 5^{\circ}\text{C}$ and the opening of rolls makes a sheet not more than 2 mm in thickness, and pass the test piece between rolls twice. If this sheeting is impossible, cut it into small pieces of not more than 2 mm in maximum dimension. Put the test piece into a circulating type of oven with ventilator at $105^{\circ}\text{C} \pm 5^{\circ}\text{C}$ and dry it for 1 h. At this time, arrange the test piece so that its surface can be sufficiently touched the hot air. After cooling the test piece in desiccator as it is, weigh it again. Repeat drying and cooling as it is until the mass becomes not so as to decrease more than 1 mg even if heated for more 30 min (final mass: m_4). The drying temperature in this case shall be subjected to the agreement between the parties concerned with delivery.

Calculate the volatile matter according to the following formula and obtain the value to two places of decimals according to **JIS Z 8401**.

$$V = \left(\frac{m_1 - m_2}{m_1} + \frac{m_3 - m_4}{m_3} \right) \times 100$$

where, V : volatile matter (%)

m_1 : mass of secondary sample before homogenized (g)

m_2 : mass of secondary sample after homogenized (g)

m_3 : mass of test piece taken from secondary sample (g)

m_4 : mass of test piece after dried in oven (g)

7.2 Ash content The ash content shall be measured in accordance with either of the following method, Method A or Method B. The applied method shall be recorded in the test report.

- (1) **Method A** Heat a porcelain crucible (hereafter referred to as "crucible") of 50 ml in capacity in muffle furnace⁽²⁾ maintained at $550 \text{ }^{\circ}\text{C} \pm 25 \text{ }^{\circ}\text{C}$ for approximately 30 min. After cooling it in a desiccator to room temperature as it is, weigh its mass (mass: m_6) to 0.1 mg. Take approximately 5 g of test piece from the sample homogenized according to 4.3 and weigh the mass (mass: m_5) to 0.1 mg. Put a test piece into the bottom of crucible laid on the hole of heat-resistant thermal insulation plate⁽³⁾, and heat carefully and gently the crucible with gas burner⁽⁴⁾ so that the test piece does not ignite. When the loss of test piece produces due to spouting out or foaming over, repeat the above-mentioned operation with a new test piece. After the test piece has been completely decomposed to a carbonized lump, strengthen gas burner and heat until it becomes carbon-like residue with almost not containing of volatile decomposed matter. Then transfer the crucible and contents to a muffle furnace maintained at $550 \text{ }^{\circ}\text{C} \pm 25 \text{ }^{\circ}\text{C}$ of temperature and continue to heat until the carbon is completely oxidized and clear ash contents are obtained. After the crucible and contents being taken out and cooled to room temperature in desiccator as they are, weigh the mass to 0.1 mg. After the crucible and contents being heated again in a muffle furnace maintained at $550 \text{ }^{\circ}\text{C} \pm 25 \text{ }^{\circ}\text{C}$ of temperature for approximately 30 min and cooled to room temperature in desiccator as they are, weigh the mass to 0.1 mg. If the mass is different from the mass first measured by over 1 mg, repeat the heating, cooling as they are and reweighing until the difference of mass between the two values in successive weighing becomes under 1 mg. (final mass: m_7)

Calculate the ash content (%) according to the following formula and obtain the value to 2 places of decimals according to **JIS Z 8401**.

$$A = \frac{m_7 - m_6}{m_5} \times 100$$

where, A : ash content (%)

m_5 : mass of test piece (g)

m_6 : mass of crucible (g)

m_7 : mass of crucible and ash (g)

Notes (2) A muffle furnace equipped with flame tunnel and being controllable of air flow in furnace shall be used. (The control may be adjusted by the opening degree of door.) Temperature control apparatus shall be required so as to be maintained at $550\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ of temperature.

(3) Heat-resistant thermal insulation plate, which is 100 mm×100 mm in dimension, approximately 5 mm in thickness and has an open hole corresponding to the crucible at central portion, shall be used.

Approximately $\frac{2}{3}$ parts of crucible shall be stood out underneath the plate.

(4) For gas burner, a Bunsen burner or the one at least equivalent in quality shall be used.

Remarks: When the upper edge of ash in the crucible sticks to the part within 3 mm from the upper edge of the crucible, a test piece shall be newly taken and the measurement shall be carried out again. When rubber is expected to spout out from the crucible in the incineration process, a test piece should be rolled in Class 6 of filter paper of 150 mm in diameter specified in **JIS P 3801** before being incinerated.

(2) **Method B** Heat a porcelain crucible of 50 ml in capacity in muffle furnace⁽²⁾ maintained at $550\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ of temperature for approximately 30 min. After cooling it in a desiccator as it is, weigh its mass (mass: m_6) to 0.1 mg. Take approximately 5 g of test piece from the secondary sample homogenized according to 4.3 and weigh the mass (mass: m_5) to 0.1 mg. Wrap the test piece with an ashless filter paper of approximately 150 mm in diameter specified in **JIS P 3801** and put it into a crucible of which the mass is measured after strongly heated beforehand. Place the crucible in the furnace equipped with 2 or more doors at $550\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ of temperature and close the door and ventilating hole. After approximately 45 min elapsed in the furnace, open the doors and ventilating hole, and continue it until clear ash is obtained by burning of test piece.

After completing of incineration, cool the crucible to room temperature in a desiccator as it is and weigh the mass (mass: m_7). Calculate the ash content (%) according to the formula described in Method A.

7.3 Dirt content Weigh out approximately 30 g of test piece from secondary sample homogenized according to 4.3 and pass it twice between the cold rolls adjusted to $0.5\text{ mm} \pm 0.1\text{ mm}$ in opening of rolls. Weigh out approximately 10 g to 20 g of test piece to 0.1 g (mass: m_8). Cut it so as to be approximately 1 g of one piece and put it in a conical flask of 250 ml or 500 ml in capacity with containing peptizer and solvent⁽⁵⁾ according to either formula rate as shown in Table 1. Heat the flask at $120\text{ }^{\circ}\text{C}$ to $130\text{ }^{\circ}\text{C}$ of temperature until the test piece is completely dissolved. A short air cooler is used in order to prevent solvent from evaporation. Agitate or stir the flask sometimes. When completely dissolved (generally after 2.5 h to 3 h), pour out the hot solution on the sieve (mass: m_9)⁽⁶⁾ which is weighed to 0.1 mg beforehand. Leave the lump of dirt in flask as it is. Repeat to wash with hot solvent⁽⁷⁾ until the test piece containing dirt in flask is completely removed. (Generally, for effective washing, approximately 100 ml of hot solvent should be needed.) At the final time of washing operation, wash out the dirt on a sieve from flask. Wash out the dirt adhered in flask onto the sieve with glass stick. Wash out the gelled test piece which is impassable through sieve according to the following method of either (a) or (b).

- (a) While hot solvent remains on sieve, brush quietly the underside of metal net with a small brush.
- (b) Put the sieve in a beaker with containing toluene to 10 mm in depth and boil it slowly for 1 h in a beaker with lid of watch glass.

Carry out these operations in a hood, if possible. Wash a sieve twice with gas oil or mineral terpene. Dry it at 100 °C of temperature for 30 min in the case of washing with gas oil, or at 100 °C of temperature for 1 h in the case of washing with mineral terpene. When the dirt can not be easily detached from the sieve, treat it with boiling toluene. If gelled rubber remains, repeat this operation again.

Weigh the mass of sieve and dirt to 0.1 mg (mass: m_{10}) after cooled in a desiccator as they are.

Calculate the dirt content (%) according to the following formula and obtain the value to 2 places of decimals according to **JIS Z 8401**.

$$D = \frac{m_{10} - m_9}{m_8} \times 100$$

where, D : dirt content (%)

m_8 : mass of test piece (g)

m_9 : mass of sieve (g)

m_{10} : mass of sieve and dirt (g)

Carry out the test twice and average the two values of measurement results.

Notes ⁽⁵⁾ Mixed xylene having the boiling point of 139 °C to 141 °C or higher aromatic hydrocarbon (mineral terpene) having the boiling point of 155 °C to 196 °C

⁽⁶⁾ The stainless sieve of 45 µm specified in **JIS Z 8801**

⁽⁷⁾ The solvent specified in ⁽⁵⁾ or No. 3 of industrial gasoline (boiling point: 60 °C to 80 °C)

Table 1 Rate of formula for peptizer and solvent

Formula	Kind and quantity of peptizer (g)	Quantity of solvent (ml)
A	2-mercapto-benzothiazole or di(2-benzamidphenyl)disulphide 0.5	200
B	Xylylmercaptane 1.0	50 to 230
C	Tolylmercaptane 1 to 1.5	200

7.4 Rubber hydrocarbon content Cut approximately 0.3 g of test piece homogenized according to 4.3 to small piece or make it to thin sheet and weigh the mass (mass: m_{11}). Extract it with acetone by a suitable method for 8 h or over up to and including 16 h with avoiding light. Dry the rubber extracted for one hour in a furnace at a temperature of 70 °C to 75 °C. Fill water into a receiving flask H to the degree of hiding the terminal of cooler as given in Attached Fig. 1. Mark a symbol showing the surface of liquid on the outside of oxidation reaction flask C when the flask contains 75 ml of solution, and transfer chromate oxidation mixed solution⁽⁸⁾ of 50 ml±1 ml to the oxidation reaction flask. Put the prepared test piece into it with raising the steam introducing tube. After returning the steam introducing tube to the original position, raise the boiling water bath J and dip the oxidation reaction flask into it. Heat the flask at 100 °C for 2 h, and take off the water bath after 2 h.

Thereafter, pass steam into the oxidation reaction flask from a suitable steam generator A. When the volume of solution in the oxidation reaction flask has increased to 75 ml, put a Bunsen burner under the oxidation reaction flask and regulate the flame of it to keep the volume.

Continue to distillate until about 500 ml is collected in the receiving flask H. About 45 min are required to complete this procedure. After completing distillation, wash the cooler and the tube fixed thereto with water, and add the washings to the distillate.

Reduce the temperature of the distillate in receiving flask to 25 °C or lower, and remove the dissolved carbon dioxide by aeration. For this procedure, insert the aeration instrument into the receiving flask and suck at the rate of 1 l per minute through the liquid for 30 min. After suction, remove the tube from the flask, and wash it with water. Collect the washings into the receiving flask, and titrate it with sodium hydroxide solution of 0.1 mol/l{0.1 N} by using phenolphthalein solution⁽⁹⁾ as indicator.

Calculate the rubber hydrocarbon content (%) from the following formula and obtain the value to one place of decimal according to **JIS Z 8401**.

$$C_H = \frac{N \times 0.908}{m_{11}}$$

where, C_H : rubber hydrocarbon content (%)

N : consumed amount of sodium hydroxide solution of 0.1 mol/l{0.1 N} (ml)

m_{11} : mass of test piece (g)

However, for this determination, carry out a blank test concerning the same amount of reagent in the same condition as those of this test, and let the titration amount of sodium hydroxide solution of 0.1 mol/l{0.1 N} be calibrated. Furthermore, let the titration amount of sodium hydroxide solution at the blank test not exceed 0.3 ml.

Notes ⁽⁸⁾ For the chromate oxidation mixed solution herein, dissolve 200 g of chromic anhydride with 500 ml of water, and add 150 ml of sulfuric acid (1.84 g/cm³ in density) to it. Sufficiently mix whole solution.

- (9) For the phenolphthalein solution herein, add 100 ml of ethyl alcohol to 0.2 g of phenolphthalein.

- Remarks 1 For the positions in contact with the oxidation mixed solution in the oxidation reaction flask of 250 ml, in the cooling tube and in the receiving flask of 1 l, the use of connection of rubber or cork shall be avoided.
- 2 The aeration instrument attached to the receiving flask is the unit in which a flask is fixed with two pieces of glass tube by cork or other stopper. One piece of glass tube is extended to the bottom of flask and another piece is so fixed for air as to be introduced from the upper part of flask.

7.5 Acetone extract content The measurement of acetone extract content shall be determined in accordance with the following method of either Method A, Method B or Method C. The applied method shall be recorded in the test report.

- (1) **Method A** Pass a sample homogenized according to 4.3 between a cold rolls with not more than 0.5 mm in opening of rolls and weigh out approximately 3 g to 5 g of test piece to 0.1 mg (mass: m_{12}). Dry a receiving flask in oven at 100 ± 2 °C of temperature, cool it to room temperature in desiccator as it is and weigh the mass to 0.1 mg (mass: m_{13}). Wrap it with filter paper or nylon filter, or put it in a cylindrical filter paper, place it in extracting cup of extraction apparatus⁽¹⁰⁾, pour approximately 50 ml of acetone into receiving flask, assemble the whole apparatus and carry out the extraction operation as heating. Adjust the rate of heating so that the extracting cup is filled with acetone 10 times to 20 times per hour and extract continuously for 16 ± 0.5 h.

After the completion of extraction, eject acetone with a suitable evaporation apparatus or rotary evaporator and dry the receiving flask and its contents for 2 h in oven at 100 ± 2 °C of temperature. Then cool them to room temperature as they are and weigh the mass of receiving flask and contents to 0.1 mg (mass: m_{14}).

Carry out a blank test concerning the same method, apparatus and solvent amount as mentioned above (increased mass: m_{15}). Carry out the test twice and average the two values of measurement result.

Calculate the acetone extract content according to the following formula and obtain the value to one place of decimal according to JIS Z 8401.

$$Ac_A = \frac{m_{14} - m_{13} - m_{15}}{m_{12}} \times 100$$

where, Ac_A : acetone extract content (%)

m_{12} : mass of test piece (g)

m_{13} : mass of empty receiving flask (g)

m_{14} : mass of receiving flask and extract after drying (g)

m_{15} : increased mass of receiving flask at blank test (g)