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(JRMA/JSA)

**Rubber compounding ingredients—
Organic chemicals—Test methods—
Part 1: General**

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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 The Japan Rubber Manufacturers Association (JRMA)/Japanese Standards Association (JSA) with the draft prepared from the association standard 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 6220-1:2001** is replaced with this Standard.

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JIS K 6220 series consists of the following 3 parts under the general title “*Rubber compounding ingredients*”:

JIS K 6220-1 *Rubber compounding ingredients—Organic chemicals—Test methods—Part 1: General*

JIS K 6220-2 *Rubber compounding ingredients—Test methods—Part 2: Organic accelerators and vulcanizing agents*

JIS K 6220-3 *Rubber compounding ingredients—Test methods—Part 3: Anti-degradants*

Rubber compounding ingredients— Organic chemicals—Test methods— Part 1: General

Introduction

This Japanese Industrial Standard has been prepared based on the first edition of **ISO 28641** published in 2010 with some modifications of the technical contents.

The portions given sidelines or dotted underlines are the matters in which the contents of the corresponding International Standard have been modified. A list of modifications with the explanations is given in Annex JD.

1 Scope

This Standard specifies the general test method of compounding ingredients for rubber (hereafter referred to as “rubber compounding ingredients”) such as vulcanizing agents, accelerators and antidegradants. This Standard is not applied to peroxide-based vulcanizing agents.

NOTE : The International Standard corresponding to this Standard and the symbol of degree of correspondence are as follows.

ISO 28641:2010 *Rubber compounding ingredients—Organic chemicals—General test methods* (MOD)

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 21-1**.

WARNING Persons using this Standard should be familiar with normal laboratory practice. This Standard does not purport to address all of the safety problems, if any, associated with its use.

It is the responsibility of the user of this Standard to establish appropriate safety and health practices.

2 Normative references

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

JIS B 7410 *Liquid-in-glass thermometers for testing of petroleum product*

JIS B 7411-1 *Glass thermometers for general—Part 1: General measuring instruments*

NOTE : Corresponding International Standard: ISO 1770 *Solid-stem general purpose thermometers* (MOD)

JIS B 7507 *Vernier, dial and digital callipers*

JIS B 7525-1 *Density hydrometers*

NOTE : Corresponding International Standard: ISO 649-1 *Laboratory glassware—Density hydrometers for general purposes—Part 1: Specification* (MOD)

JIS B 7525-3 *Hydrometer-type relative density meters*

JIS K 0050 *General rules for chemical analysis*

JIS K 5600-1-2 *Testing methods for paints—Part 1: General rules—Section 2: Sampling*

NOTE : Corresponding International Standard: ISO 15528:2000 *Paints, varnishes and raw materials for paints and varnishes—Sampling* (IDT)

JIS K 8474 *Potassium trihydrogen dioxalate dihydrate (Reagent)*

JIS K 8514 *Sodium bromide (Reagent)*

JIS K 8622 *Sodium hydrogen carbonate (Reagent)*

JIS K 8625 *Sodium carbonate (Reagent)*

JIS K 8809 *Potassium hydrogen phthalate (Reagent)*

JIS K 8866 *Sodium tetraborate decahydrate (Reagent)*

JIS K 9007 *Potassium dihydrogen phosphate (Reagent)*

JIS K 9020 *Disodium hydrogenphosphate (Reagent)*

JIS P 3801 *Filter paper (for chemical analysis)*

JIS Z 8401 *Guide to the rounding of numbers*

JIS Z 8801-1 *Test sieves—Part 1: Test sieves of metal wire cloth*

NOTE : Corresponding International Standard: ISO 3310-1 *Test sieves—Technical requirements and testing—Part 1: Test sieves of metal wire cloth* (MOD)

JIS Z 8802 *Methods for determination of pH of aqueous solutions*

ISO 649-2 *Laboratory glassware—Density hydrometers for general purposes—Part 2: Test methods and use*

ISO 6472 *Rubber compounding ingredients—Symbols and abbreviated terms*

3 Abbreviations

The abbreviations of the names of the rubber compounding ingredients used in this Standard are in accordance with **ISO 6472**.

4 Common requirements

4.1 General requirements

The general requirements common to chemical analysis shall be in accordance with **JIS K 0050**.

4.2 Thermometer

The thermometer shall be chosen from those specified in **JIS B 7410** and **JIS B 7411-1** and it shall be calibrated.

4.3 Desiccator

The desiccator containing desiccant.

5 Sampling

Carry out sampling in accordance with **JIS K 5600-1-2**.

6 Drying method of sample

The drying of sample shall be in accordance with the method specified in each test method, if necessary.

6A Characteristics and test method

Each characteristic of rubber compounding ingredient shall be measured in accordance with the test method in Table 1. Unless otherwise specified, the measurement shall be carried out in $n=1$ for the number of repeated tests in each test method, and the multiple times of test may be carried out, if necessary.

Table 1 Each characteristic of rubber compounding ingredient and its test method

Characteristic	Test method	Clause
a) Relative density and density (20 °C)	Hydrometer method (liquid sample) Pyknometer method (liquid sample, solid sample)	7.1.2 7.1.3
b) Loss on heating [% (mass fraction)]	Method A (measurement at 70 °C) Method B (measurement under the condition of temperature determined based on the nature of sample)	7.2.2 7.2.3
c) Sieve residue [% (mass fraction)]	Wet test method	7.3
d) pH of water extract (20 °C)	pH meter method	7.4
e) Melting point (°C)	Visual method Dropping point method (waxy sample) DSC method (automatic method)	7.5.2 7.5.3 7.5.4
f) Solidifying point (°C)	Solidifying point measuring apparatus method	7.6
g) Softening point (°C)	Softening point measuring apparatus method	7.7
h) Bulk density (g/cm ³)	Constant mass method Constant capacity method	7.8.2 7.8.3
i) Ash content [% (mass fraction)]		7.9
j) Refractive index (20 °C)	Refractometer method	7.10

7 Test methods

7.1 Relative density and density

7.1.1 General

Select one of the two test methods for the determination of relative density and density in Table 2, depending the nature and the quantity of the sample.

Table 2 Test method for relative density and density

Test method	Nature of sample	Quantity of sample
Hydrometer method	Liquid	250 ml min.
Pyknometer method	Liquid, solid	50 ml

7.1.2 Hydrometer method

In the hydrometer method, read out the graduation of the hydrometer floating in liquid and obtain density or relative density of the liquid.

7.1.2.1 Instruments and apparatus

The instruments and apparatus shall be as follows.

7.1.2.1.1 Hydrometer, of L50 series specified in **JIS B 7525-1** and capable of measuring density over the range 0.600 g/cm^3 to 2.000 g/cm^3 , or of large-size and small-size 19-piece set of Tables 2 and 3 in **JIS B 7525-3** and capable of measuring relative density over the range 0.700 to 1.850, and having instrumental error known.

The hydrometer graduated with density (15°C) or relative density ($15/4^\circ\text{C}$) and having instrumental error known, may be used.

Example of hydrometer, correction formula of obtaining instrumental error and conversion formula from density (15°C) or relative density ($15/4^\circ\text{C}$) to density (20°C) shall be in accordance with Annex JA.

7.1.2.1.2 Thermometer, of SG-42 or SG44 specified in **JIS B 7410**.

7.1.2.1.3 Cylinder, made of glass, having an inside diameter which is at least 25 mm larger than the maximum diameter of the hydrometer. The height shall be such that, when the hydrometer comes to rest, its base is at least 25 mm above the bottom of cylinder.

7.1.2.1.4 Constant temperature water bath, capable of maintaining the sample in cylinder at a temperature of $20.0^\circ\text{C} \pm 0.5^\circ\text{C}$.

7.1.2.1.5 Stirrer, made of material not corroded by sample and not influencing its nature and capable of stirring well for uniforming the density or relative density of the sample in cylinder.

7.1.2.2 Procedure

The procedure shall be as follows.

- a) Put the sample in the cylinder, avoiding the inclusion of air bubbles. Maintain the cylinder in the constant temperature water bath. Mix the sample up and down with a stirrer. Measure the temperature of the sample with the thermometer, immersing it to the designated mark.
- b) After the temperature of the sample has reached $20\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$, slowly put the conditioned hydrometer into the sample and allow it to come to rest. Then push the hydrometer into the sample by about two-scale divisions and release it. When releasing the hydrometer, turn it slightly so as to be able to stop moving without contacting with the cylinder wall.
- c) When the hydrometer has stopped moving, in the case of using the hydrometer with a scale designated to be read the upper edge, read the scale to half the graduation interval at the upper edge of the meniscus (see Figure JA.2).

In the case of using the hydrometer with a scale designated to be read the horizontal surface, read the scale to half the graduation interval at the lower edge of the meniscus. The details of reading the graduation interval of hydrometer are shown in Figure JA.2.

For measuring density or relative density of a translucent liquid, by using the hydrometer with a scale designated to be read the horizontal surface, read the scale at the point corresponding to the plane of intersection of the sample surface and the stem. Do this by gradually raising the eyes from a level a little below the sample surface (see Figure JA.2) and reading the scale when the elliptical sample surface becomes straight.

For measuring density or relative density of an opaque sample, read the scale at the upper edge of the meniscus of the sample surface and calculate the equivalent lower-edge value by applying a correction in accordance with **ISO 649-2**.

- d) Record the result.

7.1.2.3 Calculation

Obtain the density ($20\text{ }^{\circ}\text{C}$) and relative density ($20/20\text{ }^{\circ}\text{C}$) to three decimal places. If the precision for measurement is not required up to the place of 0.001, the correction of instrumental error may be omitted. For the correction of instrumental error, the instrumental error is subtracted from the indication of hydrometer.

In the case of using the hydrometer of density ($15\text{ }^{\circ}\text{C}$) or relative density ($15/4\text{ }^{\circ}\text{C}$), the correction shall be carried out by the correction formula in Annex JA.

7.1.3 Pyknometer method

7.1.3.1 General

The following two test methods are specified for pyknometer method, depending the type of the sample.

- a) For liquid sample
- b) For solid sample

7.1.3.2 Reagents and apparatus

The reagents and apparatus shall be as follows.

7.1.3.2.1 Water, as specified in 7.1 of JIS K 0050.

7.1.3.2.2 Warden pyknometer, made of glass, with a capacity of about 50 ml and fitted with a stopper and a ground-glass cap as shown in Figure 1.

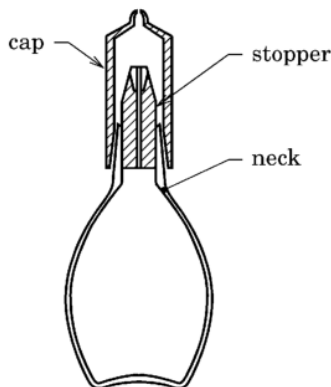


Figure 1 Example of Warden pyknometer

7.1.3.2.3 Constant temperature water bath, capable of maintaining the bath temperature at $20.0\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$.

7.1.3.2.4 Thermometer, of SG44 for pyknometer method specified in JIS B 7410.

7.1.3.2.5 Balance, capable of reading to the nearest 0.5 mg.

7.1.3.3 Test method for relative density and density of liquid sample

7.1.3.3.1 Procedure

The procedure shall be as follows.

- a) Weigh the pyknometer (mass m_0) to the nearest 0.5 mg. After removing the cap and the stopper, fill it with water at a temperature of approximately $20\text{ }^{\circ}\text{C}$. Immerse the pyknometer up to its neck in the constant temperature bath maintained at $20.0\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$.
- b) After immersing in the constant temperature water bath about 1 h, insert the stopper, which has also been brought to the bath temperature. Take the pyknometer out the water bath. Wipe the moisture flushed from the capillary tube.
- c) Thoroughly wipe the external surface with, for instance, a clean dry cloth or paper to remove all moisture and put on the stopper.
- d) Weigh its mass (m_1) to the nearest 0.5 mg.
- e) Dry the pyknometer. Then fill it with the sample at a temperature of approximately $20\text{ }^{\circ}\text{C}$ and immerse it up to its neck in the water bath maintained at $20.0\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$.
- f) After immersing in the constant temperature water bath about 1 h, insert the stopper, which has also been brought to the bath temperature. Take the pyknometer out the water bath. Wipe the top of the stopper to remove the sample flushed from the capillary tube. Thoroughly wipe the external surface with, for instance, a clean dry cloth or paper to remove all moisture and put on the cap.

g) Weigh its mass (m_2) to the nearest 0.5 mg.

7.1.3.3.2 Calculation

Calculate the relative density of liquid sample using the following formula. If the number of test is larger than $n = 2$ (see clause 6A), obtain the average value.

$$d = \frac{m_2 - m_0}{m_1 - m_0}$$

where, d : relative density (20/20 °C)

m_0 : mass of the pyknometer (g)

m_1 : mass of the pyknometer filled with water (g)

m_2 : mass of the pyknometer filled with sample (g)

Calculate the conversion to the density using the following formula.

$$D = 0.998\,20 \times d$$

where, D : density of the sample (20 °C) (g/cm³)

0.998 20 is the density of water at 20 °C (g/cm³)

7.1.3.4 Test method for relative density and density of solid sample

7.1.3.4.1 Procedure

The procedure shall be as follows.

If the sample is soluble in water, select to use another liquid, such as ethanol, toluene, octane.

- Weigh the mass of the pyknometer (m_0) and mass of the pyknometer filled with water (m_1) as described in 7.1.3.3.1 a) to d).
- Dry the pyknometer. Then weigh out about 4 ml of the sample at a temperature of approximately 20 °C and immerse it up to its neck in the water bath maintained at 20.0 °C ± 0.5 °C.
- After immersing in the constant temperature water bath about 1 h, insert the stopper, which has also been brought to the bath temperature. Take the pyknometer out the water bath. Thoroughly wipe the external surface with, for instance, a clean dry cloth or paper to remove all moisture and put on the cap.
- Weigh its mass (m_2) to the nearest 0.5 mg. Fill the pyknometer, after obtaining m_2 , with water at approximately 20 °C. Immerse it up to its neck in the water bath at 20.0 °C ± 0.5 °C.
- After immersing in the constant temperature water bath about 1 h, insert the stopper, which has also been brought to the bath temperature. Take the pyknometer out the water bath. Wipe the top of the stopper to remove the sample flushed from the capillary tube. Thoroughly wipe the external surface with, for instance, a clean dry cloth or paper to remove all moisture and put on the cap.
- Weigh its mass (m_3) to the nearest 0.5 mg.

7.1.3.4.2 Calculation

Calculate the relative density of solid sample using the following formula. If the number of test is larger than $n = 2$ (see clause **6A**), obtain the average value.

$$d = \frac{m_2 - m_0}{m_2 + m_1 - m_0 - m_3} \times D_w$$

where, d : relative density (20/20 °C)

m_0 : mass of the pyknometer (g)

m_1 : mass of the pyknometer filled with water (g)

m_2 : mass of the pyknometer filled with sample (g)

m_3 : mass of pyknometer, sample and water (g)

D_w : relative density of water or the liquid used

Calculate the conversion to the density using the following formula.

$$D = 0.998\,20 \times d$$

where, D : density of the sample (20 °C) (g/cm³)

0.998 20 is the density of water at 20 °C (g/cm³)

7.1.4 Expression of test result

Round the result in accordance with **JIS Z 8401** and express it to three decimal places.

7.1.5 Test report

The test report shall contain at least the following information.

- Record of sample
- Title and number of this Standard
- Test method (hydrometer method or pyknometer method)
- Test temperature (20 °C)
- Mass of the sample (if pyknometer method)
- Laboratory temperature and humidity
- Test results
- Any operation not included in this Standard as well as any unusual features noted during the determination
- Date of the test

7.2 Loss on heating

7.2.1 General

The loss in mass of the sample when heated and dried at a specified temperature is regarded as the loss on heating. There are two methods of measurement as follows.

- a) Method A, in which the loss in mass of the sample when heated at 70 °C is regarded as the loss on heating.
- b) Method B, in which the heating conditions (temperature, time) are determined, and the loss in mass of the sample when heated under these conditions is regarded as the loss on heating.

7.2.2 Method A

7.2.2.1 Instruments and apparatus

The instruments and apparatus shall be as follows.

7.2.2.1.1 Flat weighing bottle

7.2.2.1.2 Desiccator, as specified in 4.3.

7.2.2.1.3 Balance, capable of weighing to the nearest 0.1 mg.

7.2.2.1.4 Drying oven, capable of maintaining a temperature at 70 °C ± 2 °C.

7.2.2.2 Procedure

The procedure shall be as follows.

- a) Dry the clean flat weighing bottle and the stopper in the drying oven at a temperature of (70 ± 2) °C for 30 min. Allow them to cool to reach equilibrium at ordinary temperature in the desiccator. Weigh the flat weighing bottle with the stopper to the nearest 0.1 mg. Record the mass as m_1 .
- b) Take a sample of between 3 and 5 mm in thickness and put it into the flat weighing bottle. Weigh the bottle to the nearest 0.1 mg. Record the mass as m_2 .
- c) Place the flat weighing bottle in the drying oven at a temperature of (70 ± 2) °C, and heat for 3 h. At this time, remove the stopper and place it near the bottle, and heat them at the same time.
- d) After heating, transfer the flat weighing bottle and stopper to the desiccator and allow them to cool to reach equilibrium at ordinary temperature. Weigh the weighing bottle and stopper to the nearest 0.1 mg. Record the mass as m_3 .
- e) Repeat procedures a) to d) to give a second result.

7.2.2.3 Calculation

Calculate the loss on heating from each mass measured using the following formula, and obtain the average value of two results measured.

$$w_v = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where, w_v : loss on heating (%)

m_1 : mass of the flat weighing bottle and stopper (g)

m_2 : mass of the flat weighing bottle, stopper and sample before heating (g)

m_3 : mass of the flat weighing bottle, stopper and sample
after heating (g)

7.2.3 Method B

7.2.3.1 Instruments and apparatus

The instruments and apparatus shall be as follows.

7.2.3.1.1 Flat weighing bottle

7.2.3.1.2 Desiccator, as specified in 4.3.

7.2.3.1.3 Balance, capable of weighing to the nearest 0.1 mg.

7.2.3.1.4 Drying oven, capable of maintaining a temperature selected from the range 35 °C to 110 °C within ± 2 °C in precision.

7.2.3.2 Procedure

The procedure shall be as follows.

- a) Dry the clean flat weighing bottle and the stopper in the drying oven at a temperature of (70 ± 2) °C for 30 min. Allow them to cool to ordinary temperature in the desiccator. Weigh the flat weighing bottle with the stopper to the nearest 0.1 mg. Record the mass as m_1 .
- b) Take a sample of between 3 mm and 5 mm in thickness and put it into the flat weighing bottle. Weigh the bottle with sample and stopper to the nearest 0.1 mg. Record the mass as m_2 .
- c) Place the flat weighing bottle in the drying oven, and heat. At this time, remove the stopper and place it near the bottle, and heat them at the same time. Select the heating temperature, for the solid sample, from a temperature equal to or lower than the melting point or the decomposition point, for the liquid sample, from the temperature equal to or lower than the boiling point or the decomposition point, and from the range between 50 °C to 105 °C. In the case where the specified temperature is 60 °C and over, the heating time shall be 2 h, in the case where the specified temperature is under 60 °C, it shall be 3 h.

The heating conditions for the typical organic accelerators and vulcanizing agents are shown in Table 3, and those for the antidegradant are shown in Table 4.

- d) After heating, transfer the flat weighing bottle and stopper to the desiccator and allow them to cool to ordinary temperature. Weigh the weighing bottle and stopper to the nearest 0.1 mg. Record the mass as m_3 .
- e) Repeat procedures **a)** to **d)** to give a second result.

Table 3 Heating temperature and heating time (typical organic accelerators and vulcanizing agents)

Type [abbreviation ^{a)}]	Heating temperature °C	Heating time h	Type [abbreviation ^{a)}]	Heating temperature °C	Heating time h
BA	100 ± 2	2	CMBT ^{a)}	55 ± 2	3
DPG			CBS		
DOTG			TBBS		
MBT			DCBS		
MBTS			TETD		
ZMBT			DPTH		
ZDMC			DPTT		
ZDEC			ZDBC		
CuDMC			TeDEC		
ZEPC			DETU		
FeDMC ^{a)}			DBTU		
ETU	80 ± 2	2	TMU ^{a)}	50 ± 2	3
MBSS			DIBS		
TMTM					
TMTD					
BQD					
DBQD					
Note ^{a)} The abbreviations used in this Standard are referred to those specified in ISO 6472 . However, for the following abbreviations not specified in ISO 6472 , the compound names are shown. — CMBT: Cyclohexylamine salt of 2-mercaptobenzothiazole — FeDMC: Ferric dimethyldithiocarbamate — TMU: Trimethylthiourea					

Table 4 Heating temperature and heating time (typical antidegradants)

Type [abbreviation ^{a)}]	Heating temperature °C	Heating time h	Type [abbreviation ^{a)}]	Heating temperature °C	Heating time h
ADPA	100 ± 2	2	ETMQ	75 ± 2	2
SPH			AANA		
MBI			PAN		
ZMBI			ODPA	70 ± 2	2
<i>o</i> -MBq24			SDPA		
<i>p</i> -BBp14			DCD ^{a)}		
<i>p</i> -TBq14			TMQ	50 ± 2	3
DBHQ			NDBC		
DAHQ			<i>o</i> -MBp14		
Note ^{a)} The abbreviations used in this Standard are referred to those specified in ISO 6472 . However, for the following abbreviation not specified in ISO 6472 , the compound name is shown. — DCD: 4,4'-Dicumyldiphenylamine					

7.2.3.3 Calculation

Calculate the loss on heating from each mass measured using the following formula, and obtain the average value of two results measured.

$$w_v = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where, w_v : loss on heating (%)

m_1 : mass of the flat weighing bottle and stopper (g)

m_2 : mass of the flat weighing bottle, stopper and sample before heating (g)

m_3 : mass of the flat weighing bottle, stopper and sample after heating (g)

7.2.4 Expression of test result

Round the result in accordance with **JIS Z 8401** and express it to two decimal places.

7.2.5 Precision

For the precision, the values obtained in the survey using 10 different accelerators and nine different antidegradants are shown in **C.1**.

7.2.6 Test report

The test report shall contain at least the following information.

- Record of sample
- Name and number of this Standard

- c) Test method (method A or B)
- d) Test conditions (heating temperature and time)
- e) Laboratory temperature and humidity
- f) Mass of the sample
- g) Test results
- h) Any operation not included in this Standard as well as any unusual features noted during the determination
- i) Date of the test

7.3 Sieve residue

7.3.1 General

The powder sample is passed through a sieve, with water in a defined manner. The residue left on the sieve is then dried and weighed. The ratio of the residue is obtained as sieve residue. Generally, this method is suitable for powders of particle size up to 150 µm. However, when the sample is soluble in, or swollen by, water, the test shall be carried out using other solvents which the sample is insoluble in and easily volatile at a temperature under the melting point of the sample.

7.3.2 Water and instruments

The water and instruments shall be as follows.

7.3.2.1 Water, as specified in 7.1 of JIS K 0050.

7.3.2.2 Sieve, as specified in JIS Z 8801-1 or that agreed between the interested parties.

7.3.2.3 Flat brush, with soft bristles, not damages the shape of samples.

7.3.2.4 Drying oven

7.3.2.5 Desiccator, as specified in 4.3.

7.3.2.6 Balance, capable of weighing to the nearest 0.1 mg.

7.3.2.7 Beaker, of capacity 100 ml to 250 ml.

7.3.2.8 Evaporating dish, about 120 mm in diameter.

7.3.3 Procedure of test method

The procedure shall be as follows.

- a) Dry the sieve in the oven maintained at a temperature between 105 °C and 110 °C. Transfer it in the desiccator, allow it to cool to ordinary temperature and weigh its mass to the nearest 1 mg.
- b) Repeat the procedure of a) until the loss in mass on heating for 15 min is 1 mg or less.

- c) Weigh out a sample of between about 5.0 g and 10 g to the nearest 1 mg, and place it in the 100 ml to 250 ml beaker. Record the mass as m_0 . Moisten it with 25 ml of water. Mix it thoroughly with a glass stirring rod to make sure that whole sample is moistened. If the sample is not moistened well, about 1 % of surfactant to water may be added.
- d) Add about 50 ml of water and mix thoroughly. Decant the particles floating on the liquid onto the sieve.
- e) Pour about 50 ml of water on the remainder of the sample. Decant it onto the sieve in the same manner as above. Transfer the whole sample on the sieve by repeating this procedure.
- f) Wash the sample through the sieve by pouring successive small amounts of water on to the sieve and shaking the sieve. Then put the sieve in an about 120 mm evaporating dish in diameter. Pour water into the dish until it reaches a level 15 mm above the screen and sweep the surface of the screen with the brush at a rate of one stroke per second.
- g) After every 20 strokes, lift the sieve from the dish to drain water through the screen and, in addition, renew the water in the dish after every 40 strokes.
- h) Repeat this procedure until no more of the sample passes through the sieve. Wash the solid matter adhering to the brush onto the sieve with water. Wash the sieve thoroughly with water.
- i) For the sample specified in Tables 3 and 4, dry the sieve in the oven under the heating condition specified in Tables 3 and 4 for 30 min. For other sample, dry the sieve in the oven maintained at the temperature not to fuse the sample for 30 min. Allow it to cool in the desiccator, then weigh it. Repeat this procedure until it reaches constant mass and calculate the mass of the residue by subtracting the mass of the sieve initially recorded from the final mass of the sieve plus dry residue. Record the mass as m_1 .

7.3.4 Calculation

Calculate the sieve residue using the following formula. If the number of test is larger than $n=2$ (see clause 6A), obtain the average value.

$$w_s = \frac{m_1}{m_0} \times 100$$

where, w_s : sieve residue (%)

m_0 : mass of the sample (g)

m_1 : mass of the residue (g)

7.3.5 Expression of test result

Round the result in accordance with JIS Z 8401 and express it to one decimal place.

7.3.6 Precision

For the precision, the values obtained in the survey using eight different accelerators are shown in **C.2**.

7.3.7 Test report

The test report shall contain at least the following information.

- a) Record of sample
- b) Name and number of this Standard
- c) Size of opening of the sieve used
- d) Mass of the sample
- e) Test condition (drying temperature)
- f) Laboratory temperature and humidity
- g) Test results
- h) Any operation not included in this Standard as well as any unusual features noted during the determination
- i) Date of the test

7.4 pH of water extract

7.4.1 General

For pH of water extract, extract the rubber compounding ingredient with water, and measure the pH of the water using a pH-meter. There are two methods for preparation of test solution as follows.

- a) **Method A**, extract the sample with water by boiling.
- b) **Method B**, extract the sample with water at ordinary temperature.

7.4.2 Water, apparatus and instruments

The water, apparatus and instruments shall be as follows.

7.4.2.1 Water, carbon-dioxide-free water prepared by Annex E of **JIS K 0050**.

7.4.2.2 pH-meter, adjusted according to Annexes A and B and any one of those types in Table A.3 or A.4 depending on the purpose of the test.

7.4.2.3 Thermometer, of PMF-30 specified in **JIS B 7410**.

7.4.2.4 Hard glass conical flask, of capacity 200 ml.

7.4.2.5 Balance, capable of weighing to the nearest 1 mg.

7.4.2.6 Funnel, made of hard glass

7.4.2.7 Filter paper, as specified in **JIS P 3801**.

7.4.2.8 Shaker, shaker, magnetic stirrer or other apparatus.

7.4.3 Preparation of test solution

7.4.3.1 Method A

The preparation method of test solution according to Method A shall be as follows.

- a) Weigh about 5.0 g of sample into the hard glass conical flask. Add 100 ml of water and boil for 5 min.
- b) Compensate for the loss of water resulting from boiling by adding water. Boil the solution gently. Then allow it to cool it to ordinary temperature with the flask stoppered.
- c) Take the solution or slurry, filtered after cooling, as the test solution.

If it settles out quickly, the supernatant solution may be used.

7.4.3.2 Method B

The preparation method of test solution according to Method B shall be as follows.

- a) Weigh about 5.0 g of sample into the hard glass conical flask. Add 100 ml of water, plug and shake it for 5 min.
- b) Take the solution or slurry, filtered after mixing, as the test solution.

If it settles out quickly, the supernatant solution may be used.

7.4.4 Procedure

The procedure shall be in accordance with the glass electrode method specified in **JIS Z 8802**.

7.4.5 Expression of test results

The pH shall be expressed as shown in the following example. If the number of test is larger than $n=2$ (see clause 6A), obtain the average value.

Example : pH 3.8 (20 °C) Type III glass electrode pH-meter

7.4.6 Test report

The test report shall contain at least the following information.

- a) Record of the sample
- b) Name and number of this Standard
- c) Type of pH-meter used and the name of the manufacturer
- d) Mass of the sample
- e) Method of preparation of the test solution (method A or B)
- f) pH standard solutions used to check the accuracy of the pH-meter (see Annex A)
- g) Temperature of test
- h) Laboratory temperature and humidity
- i) Test results
- j) Any operation not included in this Standard as well as any unusual features noted during the determination
- k) Date of the test

7.5 Melting point

7.5.1 General

For determination of the melting point of rubber compounding ingredients, use one of three methods in Table 5.

Table 5 Principle and characteristic of test method for melting point

Test method	Clause	Principle	Characteristic
Visual method	7.5.2	Heat the sample in a container indirectly by a heating medium and obtain the melting point visually.	It is capable of measuring by general apparatus. It is also capable of measuring the dark-coloured sample. The difference of measured value between the persons in charge of measurement appears easily. [There are the transmittance method (see Annex JB) and the reflectance method (see Annex JC) as the automatic method.]
Dropping point method	7.5.3	Heat the sample attached to the thermometer and obtain the melting point visually.	It is used for measuring the waxy sample at 80 °C or under in melting point.
DSC method	7.5.4	Obtain the melting point by using a differential scanning calorimeter (DSC).	It is an automatic measurement and labour saving. The difference of measured value between the persons in charge of measurement does not appear. Other information than melting point can be obtained at the same time. The price of apparatus is comparatively high.

7.5.2 Visual method

7.5.2.1 Instruments and apparatus

The instruments and apparatus shall be as follows.

7.5.2.1.1 Apparatus, an example of construction and dimensions of melting point measurement apparatus is shown in Figure 2.

7.5.2.1.2 Sample holder, made of hard glass. Shape and dimensions are as shown in Figure 2.

7.5.2.1.3 Heating medium, water or silicone oil, etc. The silicone oil shall not degrade at the measurement temperature and shall have a dynamic viscosity at 25 °C between 50 mm²/s and 100 mm²/s.

7.5.2.1.4 Plug, made of tetrafluoroethylene, rubber or cork.

7.5.2.1.5 Thermometer, of No. 1 to No. 6 with immersion line (stick shaped) specified in Japanese Pharmacopeia (12th revision). In the case where the anticipated melting point is 70 °C or lower, SOP-57 to 59 specified in **JIS B 7410** may be used.