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**Test methods of coating powders for
electrical insulation**

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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 Japan Electrical Insulating and Advanced Performance Materials Industrial Association (JEIA)/ Japanese Industrial 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 C 2161** : 1997 has been replaced with this Standard.

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Test methods of coating powders for electrical insulation

Introduction

This Japanese Industrial Standard has been prepared based on the first edition of **IEC 60455-2-2** published in 1984. Some alterations and additions are made, however, involving the addition of test methods that are considered necessary, but not given originally in the said International Standard.

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

1 Scope

This Standard specifies test methods of thermosetting coating powders for electrical insulation to be used for insulation of electrical and electronic components.

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

IEC 60455-2-2:1984 *Specification for solventless polymerisable resinous compounds used for electrical insulation — Part 2: Methods of test — Test methods for coating powders for electrical purposes* (MOD)

The 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**.

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 standards (including amendments) indicated below shall be applied.

JIS B 7411 *Solid-stem general purpose liquid-in-glass thermometers*

JIS B 7502 *Micrometer callipers*

JIS B 7507 *Vernier, dial and digital callipers*

JIS C 1102-2 *Direct acting indicating analogue electrical measuring instruments and their accessories Part 2: Special requirements for ammeters and voltmeters*

JIS C 1602 *Thermocouples*

JIS C 1604 *Resistance thermometer sensors*

JIS C 2320 *Electrical insulating oils*

JIS G 3141 *Cold-reduced carbon steel sheet and strip*

- JIS K 5600-1-4 *Testing methods for paints — Part 1: General rule — Section 4: Standard panels for testing*
- JIS K 5600-5-2 *Testing methods for paints — Part 5: Mechanical properties of film — Section 2: Cupping test*
- JIS K 5600-5-5 *Testing methods for paints — Part 5: Mechanical property of film — Section 5: Scratch hardness (Stylus method)*
- JIS K 6850 *Adhesives — Determination of tensile lap-shear strength of rigid-to-rigid bonded assemblies*
- JIS K 7100 *Plastics — Standard atmospheres for conditioning and testing*
- JIS R 6252 *Abrasive papers*
- JIS S 6006 *Pencil, coloured pencils and leads for them*
- JIS S 6050 *Plastics erasers*
- JIS Z 8401 *Guide to the rounding of numbers*
- JIS Z 8704 *Temperature measurement — Electrical methods*
- JIS Z 8801-1 *Test sieves — Part 1: Test sieves of metal wire cloth*
- ISO 683-1 *Heat-treatable steels, alloy steels and free-cutting steels — Part 1: Direct-hardening unalloyed and low-alloyed wrought steel in form of different black products*
- ISO 11357-2 *Plastics — Differential scanning calorimetry (DSC) — Part 2: Determination of glass transition temperature*
- ISO 11359-2 *Plastics — Thermomechanical analysis (TMA) — Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature*
- IEC 60216-1 *Electrical insulating materials — Properties of thermal endurance — Part 1: Ageing procedures and evaluation of test results*

3 Terms and definitions

For the purpose of this Standard, the following terms and definitions apply.

3.1 loss on heat

the reduction in mass of a sample due to volatile matter when the sample is heated

It is expressed as the percentage of the mass loss to the mass before heating.

3.2 cut-through resistance

the temperature of a specimen at the instant when a metal wire under specified load breaks the coating of the specimen and comes in contact with the edge of the steel square bar of the specimen

3.3 edge coverage

the ratio of edge thickness to face thickness of the coating when the film is formed on a steel square bar

4 Classification of tests

The classification of tests shall be as given in table 1.

Table 1 Classification of tests

Classification		Applicable clause for test method	Classification		Applicable clause for test method
Characteristics test of powder	Bulk density	7.1	Characteristics test of cured materials	Specific permittivity and dissipation factor	8.10
	Pourability	7.2		Volume resistivity	8.11
	Particle size distribution	7.3		Resistance to tracking	8.12
	Softening temperature	7.4		Resistance to chemicals	8.13
	Gel time	7.5		Resistance to vapour of solvents	8.14
	Loss on heat	7.6		Thermal endurance	8.15
Characteristics test of cured materials	Appearance of coating	8.2		Edge coverage	8.16
	Thickness	8.3		Thermal shock resistance	8.17
	Porosity of coating	8.4		Barcol hardness	8.18
	Scratch hardness	8.5		Pencil hardness	8.19
	Cut-through resistance	8.6		Glass transition temperature	8.20
	Impact strength	8.7		Coefficient of thermal expansion	8.21
	Cupping resistance	8.8		Water absorption rate of boiling	8.22
	Electric strength	8.9		Tensile shear strength	8.23
NOTE : In the characteristics test of powder, items 7.1 to 7.5 are for evaluating capability of coating work by the powder, and in the characteristics test of cured materials, items 8.3 to 8.15 and 8.17 to 8.23 are for evaluating the characteristics of coating, and items 8.2 and 8.16 are for evaluating adhesion of the coating.					

5 Test conditions

The test conditions shall be in accordance with Grade 3 [temperature $(23 \pm 5) ^\circ\text{C}$, humidity $(50^{+20}_{-10}) \%$] stated in clause 5 of **JIS K 7100**.

6 Rounding off of numerical values

The measured values or average values obtained in each test shall be rounded off in accordance with **JIS Z 8401** to the number of digits shown in table 2.

Table 2 Rounding

Classification	Units	Round to:	Classification	Units	Round to:
Bulk density	g/cm ³	Second decimal place	Specific permittivity	—	First decimal place
Pourability	s	Integer	Dissipation factor	—	Third decimal place
Particle size distribution	%	First decimal place	Volume resistivity	Ω·m	Two significant figures
Softening temperature	°C	Integer	Resistance to tracking	—	Integer
Gel time	s	Integer	Resistance to chemicals	%	First decimal place
Loss on heat	%	Second decimal place	Edge coverage	%	Integer
Thickness	mm	Second decimal place	Barcol hardness	—	Integer
Scratch hardness	g	Integer	Glass transition temperature	°C	Integer
Cut-through resistance	°C	Integer	Coefficient of thermal expansion	1/°C	Two significant figures
Impact strength	cm	Integer	Water absorption rate of boiling	%	Second decimal place
Cupping resistance	mm	First decimal place	Tensile shear strength	MPa	Two significant figures
Electric strength	kV/mm	Integer			

7 Characteristics tests of powders

7.1 Bulk density

7.1.1 Apparatus

The measurement test of bulk density shall use the following apparatus:

- a) **Bulk density measuring apparatus** The bulk density measuring apparatus shall be that of brass or stainless steel with the shape and dimensions shown in figure 1. The funnel shall be such that its lower opening can be covered and the receiver (measuring cylinder) shall have a volume of $(100 \pm 0.5) \text{ cm}^3$, an internal diameter of $(40 \pm 0.5) \text{ mm}$ and smoothly finished internal surface.

Unit : mm

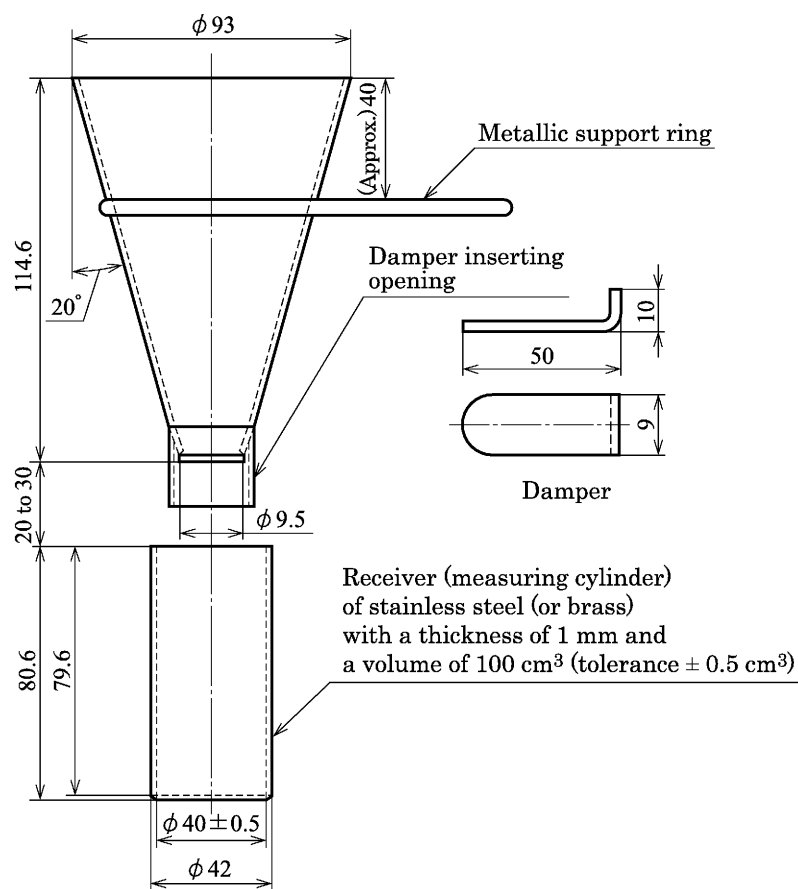


Figure 1 Bulk density measuring apparatus

b) **Balance** The balance shall have a reciprocal sensitivity of 0.1 g or better.

7.1.2 Test sample

About 120 ml of powder shall be mixed well and used as the test sample.

7.1.3 Procedure

Insert the damper into the lower opening of the funnel and put about 120 ml of powder into the funnel. Draw out the damper quickly and let the powder fall into the receiver placed 20 mm to 30 mm below the lower opening of the funnel. If the powder adheres to the funnel wall, tap lightly the funnel wall or poke the powder with a thin rod to let it fall.

Scrape off the powder protruding above the receiver with a flat plate and then weigh the mass of the receiver containing the sample correctly to the nearest 0.1 g.

Carry out the measurement three times and obtain the average value.

7.1.4 Calculation

Calculate the bulk density by the following equation:

$$D = \frac{W_1 - W_2}{V}$$

where, D : bulk density (g/cm³)
 W_1 : mass of receiver containing sample (g)
 W_2 : mass of receiver (g)
 V : volume of receiver (cm³)

7.1.5 Report

Report the following matter:

- a) Bulk density (g/cm³)

7.2 Pourability

7.2.1 Apparatus

The pourability test shall use the following apparatus:

- a) **Pourability test apparatus** An apparatus consisting of a funnel, funnel support, supporting rod and supporting base as illustrated in figure 2 shall be used. The funnel shall be as shown in figure 3.

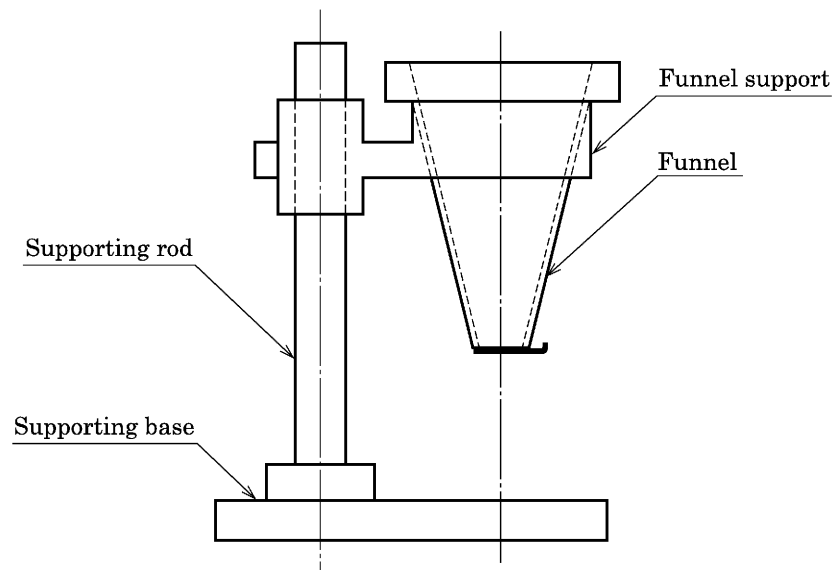


Figure 2 Pourability test apparatus

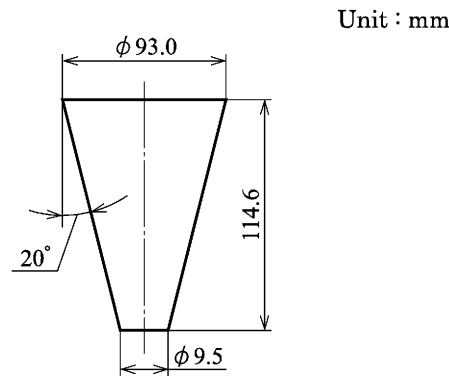


Figure 3 Funnel for pourability test

b) **Stop-watch or timer**

c) **Balance** The balance shall have a reciprocal sensitivity of 0.1 g or better.

7.2.2 Procedure

Work the powder sample of about 300 g (amount enough for three tests described below) fine until there is no lump or coagulation, weigh (100 ± 0.1) g of sample accurately and quietly transfer it into the funnel whose bottom orifice is closed. The orifice part at this point shall be filled with the powder. Start the stopwatch or the timer at the instant when the orifice is opened, and stop it at the instant when the last powder leaves the orifice, to read the time. Carry out the measurements three times and obtain the average value.

If either of the following occurs, terminate the measurement and discard it as being outside the application of the fluidity test:

- a) flow-out of powder from the orifice stops twice in three measurements; or
- b) flow-out of powder from the orifice stops once or more in the further conducted three measurements following the initial three measurements in which the flow-out has stopped once.

7.2.3 Report

Report the following matters:

- a) Flow-out time (s)
- b) If the measurement is terminated, state "Outside the application of fluidity test".

7.3 Particle size distribution

7.3.1 Dry sieving method

7.3.1.1 Apparatus

The dry sieving method shall use the following apparatus:

- a) **Sieve vibrator** The sieve vibrator shall be of mechanical vibration system, and can accommodate 6 sieves of 150 mm or 200 mm diameter and 45 mm depth. The vibration shall be 4.00 Hz to 4.83 Hz and the hammering shall be 2.28 Hz to 2.75 Hz.

- b) **Sieves** The sieves shall be sieves of metal wire cloth specified in **JIS Z 8801-1**, of required sieve size which has been decided upon by agreement between the parties concerned with acceptance.
- c) **Balance** The balance shall have a reciprocal sensitivity of 0.01 g or better.

7.3.1.2 Sample

Take 20 g to 50 g of the powder per one test, weigh it accurately with a balance, and supply it as the sample.

7.3.1.3 Procedure

Pile up the sieves from the ones with smaller sieve size, attach a receiving pan to the bottom, put the sample in the uppermost sieve and place the cover. Carry out the sieving by means of the sieving vibrator for $30 \text{ min} \pm 30 \text{ s}$. Weigh the mass of the so sieved sample left on each sieve accurately to 0.01 g.

7.3.1.4 Calculation

Obtain the mass ratio in the percentage of the sample of each particle size to the total mass before sieving to the first decimal place.

7.3.1.5 Report

Report the following matter:

- a) Residue on sieve of each sieve size (%)

7.3.2 Vacuum sieving method

7.3.2.1 Apparatus

The vacuum sieving method shall use the following apparatus:

- a) **Vacuum sieving apparatus** The vacuum sieving apparatus shall be capable of maintaining a vacuum of $125 \text{ mm} \pm 12.5 \text{ mm}$ ($16\,665 \text{ Pa} \pm 1\,667 \text{ Pa}$) of water as measured by a manometer. The revolving nozzle shall revolve at a speed of $(25 \pm 2) \text{ rev/min}$.

NOTE : Increasingly higher vacuums up to 700 mm (93 325 Pa) of water are maintained for smaller sized sieves.

- b) **Balance** The balance shall have a reciprocal sensitivity of 0.000 1 g or better.
- c) **Sieves** The sieves shall be sieves of metal wire cloth specified in **JIS Z 8801-1**, of required sieve size which has been decided upon by agreement between the parties concerned with acceptance.
- d) **Transparent cover**

7.3.2.2 Sample

The sample shall be 10.0 g for sieve size $32 \mu\text{m}$ or larger, and 1.0 g for sieve size smaller than $32 \mu\text{m}$.

7.3.2.3 Procedure

If samples are not to be transferred for weighing, weigh the mass of each of the sieves, covers and samples to 0.1 g. If samples are to be transferred from the sieves, weigh the sample to 0.000 1 g, and put the sample into the sieve.

Put the transparent cover on the sieve and place the sieve in the vacuum sieving apparatus.

The vacuum shall be 150 mm of water for sieve sizes larger than 74 μm , 250 mm of water for sieve sizes 74 μm to 20 μm , and 700 mm of water for sieve sizes less than 20 μm .

Operate the sieving apparatus for $10 \text{ min} \pm 15 \text{ s}$.

Remove the sieve and cover from the sieving apparatus.

When the sample is not transferred from the sieve, weigh to 0.1 g the sample retained on the sieve or adhering to the cover together with the mass of the sieve, and obtain the residual of sample by subtracting the tare mass from the total mass.

If the sample is transferred from the sieve, transfer the sample to the balance by carefully brushing both sides of the sieve and cover, then weigh the mass of sample to 0.000 1 g.

Repeat the procedure for each sieve used.

7.3.2.4 Calculation

Obtain the percentage of the mass of powder retained on each sieve to the total powder before sieving to the first decimal place.

7.3.2.5 Report

Report the following matter:

- a) Residue for each sieve (%)

7.4 Softening temperature

7.4.1 Apparatus

The measurement of softening temperature shall use the following apparatus:

- a) **Softening temperature measuring apparatus** The softening temperature measuring apparatus shall be the Kofler hot bench shown in figure 4. It shall consist of a hot plate having temperature gradient from one side to the other and a scale plate which shows the softening temperature of the sample. The temperature gradient shall be capable of being controlled from the room temperature to 250 °C to 300 °C, and the scale plate which indicates the temperature shall be so constructed that it can be adjusted by means of the standard material with a known melting point.

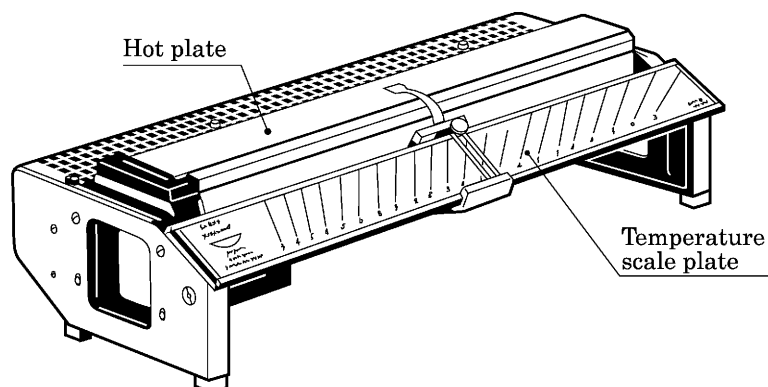


Figure 4 Softening temperature measuring apparatus

7.4.2 Procedure

Set the softening temperature measuring apparatus in a draught-free area and pre-heat it until the temperature of the hot plate attains equilibrium. Adjust the temperature indication by using a standard material having a melting point close to that of the sample.

Put a small amount of the powder on the plate by spreading it in a zone so that the anticipated melting point becomes the centre of temperature band of 20 °C. About 1 min after, brush off the powder towards the low temperature of the temperature gradient with a soft brush, take the point where the trace of burned powder is observed as the softening point, and read the temperature indication of this point.

Carry out the measurement three times with the same sample and obtain the average value.

7.4.3 Report

Report the following matter:

- a) Softening temperature (°C)

7.5 Gel time

7.5.1 Method A

7.5.1.1 Apparatus

Method A of gel time measurement shall use the following apparatus:

- a) **Hot plate** The hot plate shall have a smooth surface and be capable of maintaining the temperature to within ± 1 °C.
- b) **Stop-watch or timer**
- c) **Stirrer** The stirrer shall be a wood spatula approximately 2 mm $\times$ 10 mm $\times$ 150 mm or non-heat conducting stirrer with similar dimensions.
- d) **Surface pyrometer**
- e) **1 ml measuring spoon**

7.5.1.2 Procedure

Place the hot plate in a draught-free area or three-sided, approximately 300 mm high draught shield, and heat it to the specified temperature. The temperature of the hot plate shall be measured with the surface pyrometer.

Measure 1 ml of the sample with a 1 ml measuring spoon, and place it on the surface of the hot plate. Immediately spread the sample with the spatula into a 50 mm diameter circle. When the sample becomes “wetted”, start the stop-watch or timer. Keep rubbing the molten sample in a circular motion at a rate of (60 ± 5) revolutions per minute until the sample ceases to flow and forms an immovable gel, stop the stop-watch or timer and record the time.

Carry out the measurement three times with the same sample and take the average value.

NOTE : The hot plate may be previously coated with thin release agent, to facilitate the removal of gelated sample from the hot plate.

7.5.1.3 Report

Report the following matters:

- Gel time (s)
- Measurement temperature ($^{\circ}\text{C}$)

7.5.2 Method B

7.5.2.1 Apparatus

Method B of gel time measurement shall use the following apparatus:

- Hot plate** The hot plate shall have the structure and dimensions shown in figure 5 and be capable of maintaining the temperature to within $\pm 1^{\circ}\text{C}$.

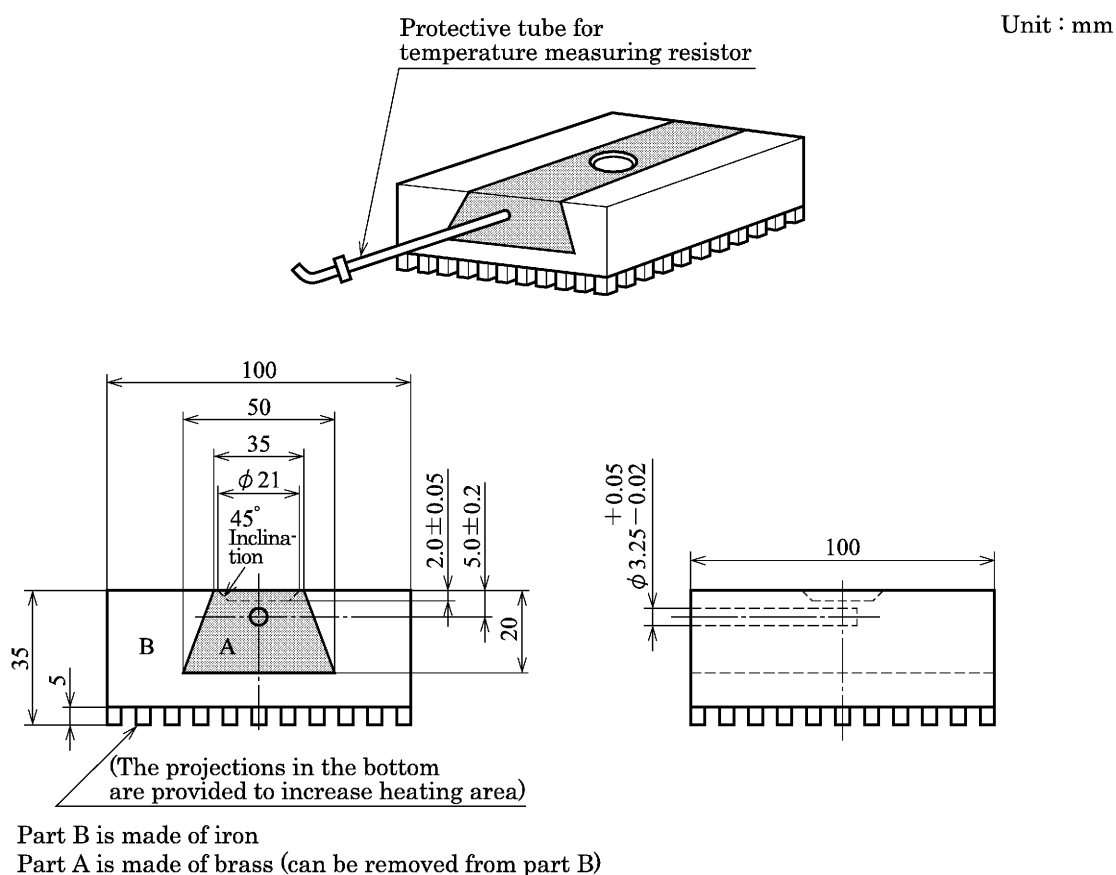


Figure 5 Hot plate

- Stop-watch or timer**
- Stirring pin** The stirring pin shall have the dimensions as shown in figure 6.

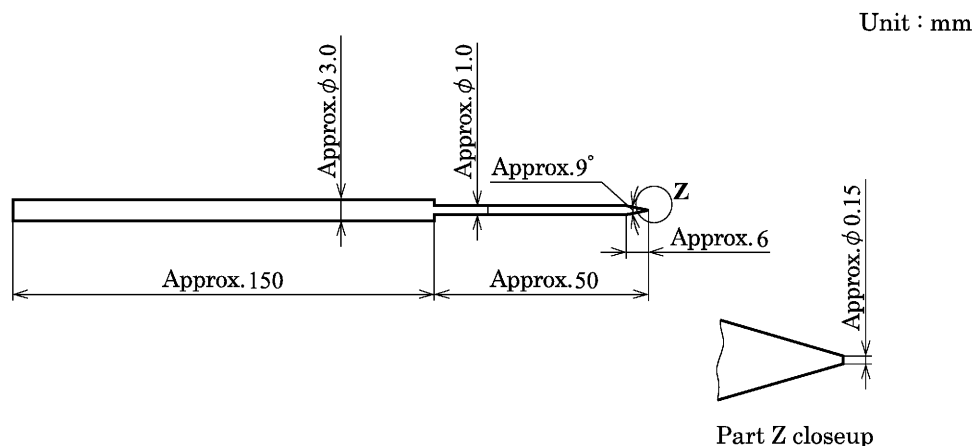


Figure 6 Stirring pin

- d) **Thermometer** The thermometer shall consist of a combination of the Class A resistance thermometer sensor specified in table 2 in clause 4 of **JIS C 1604** and a stainless steel protective tube with 3.2 mm outer diameter, and shall indicate or record the temperature by connection of the resistance thermometer sensor to the measuring instrument of at least Grade C specified in 10.7 of **JIS Z 8704** or any equivalent recording thermometer.

7.5.2.2 Procedure

Place the hot plate in a draught-free area or three-sided, approximately 300 mm high draught shield.

Weigh about 0.1 g of sample and put it on the recess of the hot plate, and melt it by spreading with the stirring pin into about 20 mm diameter circle quickly. Holding the stirring pin at an angle of about 30° to the plane, stir the sample with the pin tip by moving it in circles at a rate of (60 ± 5) revolutions per minute. When stirring of the entire sample becomes difficult or the sample ceases to adhere to the pin tip, i.e., the sample has formed an immovable gel, take this point as the end point, and take the time from placing the sample on the hot plate to the end point as the gel time. The sample should stay on the hot plate as far as possible during measurement.

Carry out the measurement three times with the same sample, and take the average value.

7.5.2.3 Report

Report the following matters:

- Gel time (s)
- Measurement temperature (°C)

7.6 Loss on heat

7.6.1 Apparatus

The test of loss on heat shall use the following apparatus:

- Container** The container shall be of about 60 mm diameter, having a shape like Petri dish, and made of glass, tinplate or aluminum.

- b) **Thermostatic chamber** The thermostatic chamber shall be of hot air circulating system equipped with an exhaust port, which is capable of maintaining the temperature to within ± 2 °C.
- c) **Balance** The balance shall have a reciprocal sensitivity of 1 mg or better.

7.6.2 Procedure

Dry the container in a dryer maintained at the temperature of (105 ± 2) °C, cool it in a desiccator and measure the mass of the container. Spread (2 ± 0.1) g of sample on the bottom of the container and weigh it correctly to 1 mg and heat it at (105 ± 2) °C for 1 h, or at (230 ± 3) °C for 5 min. After completion of the heating, take out the container from the thermostatic chamber, cool it in a desiccator, weigh the mass and obtain the mass of residue in the container.

Carry out the measurement with three samples and obtain the average value.

7.6.3 Calculation

Calculate the loss on heat by the following equation:

$$A = \frac{m_1 - m_2}{m_1} \times 100$$

where, A : loss on heat (%)
 m_1 : mass of sample before heating (g)
 m_2 : mass of sample after heating (g)

7.6.4 Report

Report the following matters:

- a) Loss on heat (%)
b) Heating temperature for test (°C)

8 Characteristics tests of cured materials

8.1 Preparation of specimens

8.1.1 Classification of specimens

The specimens prepared according to one of the following shall be used:

- a) Apply the powder paint on a metal plate coated with ethylene tetrafluoride resin, silicone oil, etc., and after allowing to cure, pull off the cured material from the plate and then cut it into the specified size.
- b) Apply the powder paint on a steel plate or a steel square bar of the specified shape and dimensions by the specified method and then allow to cure.
- c) Mould the specimen by hot-pressing the powder paint in a metallic mould.

NOTE : Take sufficient care not to leave bubbles in the cured material during forming.

8.1.2 Formation of coating

After defatting a metal plate sufficiently using such solvents as alcohol or acetone and drying well, form a coating on the plate by either of the following methods:

- a) fluidized bed coating method [see “Powder coating technology handbook (third edition)”]
- b) electrostatic coating method [see “Powder coating technology handbook (third edition)”]

8.2 Appearance of coating

The appearance shall be examined by normal eyesight for colour tone, flatness, inclusion of foreign matters, etc., preferably under diffused daylight.

8.3 Thickness

The thickness of coating shall be measured by means of an outside micrometer caliper specified in JIS B 7502 or a microscope; or of a magnetic instrument for magnetic base materials.

8.4 Porosity of coating

8.4.1 Apparatus

The test of porosity shall use the following apparatus:

- a) **High-voltage generator** A high-voltage generator to be used shall be as follows:
 - 1) The high-voltage generator shall generate a d.c. test voltage which is adjustable between + 5 % and -10 %.
 - 2) The internal resistance shall be chosen so that the steady state short-circuit current has an average value of (3 ± 0.3) mA.
 - 3) The high voltage testing head shall be equipped with a current limiting resistor to limit the current during flashover at high voltage to 10 mA to 50 mA.
 - 4) One pole of the high-voltage generator shall be connected to the protective conductor or to the body of the high-voltage generator. The other pole of the generator shall lead to the high-voltage testing head through a screened high-voltage cable. The high-voltage generator shall be provided with a device which optically and/or acoustically clearly indicates each spark at the test electrode.
- b) **High-voltage testing head** The testing head shall be provided with an electrically conductive sheath which is connected to the protective conductor of the apparatus.
- c) **Electrodes** The electrodes shall consist of either flexible metal brushes or conductive rubber.

8.4.2 Specimens

The specimens shall be prepared by coating a steel sheet as specified in clause 3 of JIS G 3141 of approximate dimensions of 150 mm × 200 mm × 1 mm by the method specified in 8.1.2. Five specimens shall be prepared.

8.4.3 Procedure

Adjust the test voltage to 15 kV/mm, and move the electrode at a maximum speed of 4 cm/s across the surface of the specimen. During this time, carefully observe the change of test voltage. Pores and flaws are indicated by visible sparks on the test electrode and simultaneously by an optical and/or acoustic signal.