

JIS

JAPANESE INDUSTRIAL STANDARD

**Testing methods of
solventless liquid resins
for electrical insulation**

JIS C 2105—1992

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In the event of any doubt arising,
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Testing methods of solventless liquid
resins for electrical insulation

C 2105-1992

1. Scope

This Japanese Industrial Standard specifies the testing methods of solventless liquid resins for electrical insulation and the materials in cured form used for impregnation, molding, embedding, etc. for electric machinery and apparatus, and electrical and electronic components.

Remarks 1. The standards cited in this Standard are shown in Attached Table 1.

2. The International standard corresponding to this Standard is given below.

IEC 455-2 (1977) Specification for solventless polymerisable resinous compounds used for electrical insulation. Part 2: Methods of test

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

2. Classification of tests

Tests are classified as shown in Table 1.

Table 1. Classification of tests

Kind of test (liquid characteristics)	Applicable clause for test method	Kind of test (cured material characteristics)	Applicable clause for test method
Appearance	5.	Appearance of cured material	21.
Color number	6.	Hardness	22.
Specific gravity	7.	Specific gravity of cured material	23.
Viscosity	8.	Relative shrinkage	24.
Relative viscosity change	9.	Flexural strength and flexural modulus of elasticity	25.
Ash content	10.	Tensile strength, elongation and tensile modulus of elasticity	26.
Filler content	11.	Impact strength	27.
Volatile content	12.	Compressive strength	28.
Acid value	13.	Bond strength	29.
Hydroxyl value	14.	Coefficient of linear expansion	30.
Epoxy value	15.	Thermal conductivity	31.
Drying time of coated film	16.	Resistance to cracking	32.
Adhesion of coated film	17.	Temperature of deflection under load	33.
Gel-time	18.	Thermal endurance	34.
Curing	19.	Loss in mass on heating	35.
Influence on enameled wire	20.	Flame retardance	36.
		Water absorption	37.
		Resistance to oil	38.
		Resistance to chemicals	39.
		Dielectric breakdown strength	40.
		Volume resistivity	41.
		Dissipation factor and permittivity	42.
		Arc resistance	43.
		Tracking resistance	44.

3. Test conditions

3.1 Testing place Unless otherwise specified atmospheric conditions of testing place shall be the ordinary temperature $20 \pm 15^{\circ}\text{C}$ and the ordinary humidity (65 ± 20) % specified in JIS Z 8703.

3.2 Sampling procedure Products of each job number is considered as one lot, and the product is well stirred if necessary. Then the sample of about 0.5 kg shall be taken into each clean container by taking care not to include impurities and the container shall be closely plugged to avoid evaporation of the volatile content.

3.3 Disposition of sample The sample shall be stored in a cool and dark place as far as possible until initiation of the test. Each test on liquid sample and manufacture of test pieces shall be commenced within 72 h from the sampling.

The above procedures shall be commenced immediately for products to which curing agent has been added.

4. Rounding off of numerical values

The measured values or the average values obtained in each test shall be rounded off to the places or the significant figures shown in Table 2 according to JIS Z 8401.

Table 2. Rounding off of numerical values

Classification	Place or number of figures to which rounded off
(Liquid characteristic)	
Specific gravity	Two places of decimals
Viscosity	Two significant figures
Relative viscosity change	
Ash content	
Filler content	
Volatile content	Two significant figures
Acid value	
Hydroxyl value	Three significant figures
Epoxy value	
Adhesion of coated film	Two significant figures Three significant figures
Middle part	
Lower part	
Gel-time	One place of decimal
Curing	One place of decimal Integer
Minimum curing time	
Highest exothermic temperature	

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Table 2. (Continued)

Classification	Place or number of figures to which rounded off
(Cured material characteristics)	
Hardness	Integer
Specific gravity of cured material	Two places of decimals
Relative shrinkage	Two significant figures
Flexural strength Flexural modulus of elasticity Tensile strength Tensile modulus of elasticity	Integer
Elongation	Two significant figures
Impact strength Compressive strength Bond strength	Integer
Coefficient of linear expansion Thermal conductivity	Two significant figures
Resistance to cracking Temperature of deflection under load	Integer
Loss in mass on heating	Two significant figures
Flame retardance	Integer
Water absorption Dielectric breakdown strength Volume resistivity Dissipation factor Permittivity	Two significant figures
Arc resistance	Three significant figures
Tracking resistance	Two significant figures

5. Appearance test

5.1 Apparatus Suitable ones shall be selected from the test tubes specified in JIS R 3503 and used as the apparatus.

5.2 Procedure Put the sample into the test tube to a depth 30 mm or more, and examine for the presence of impurities by looking at the tube from the side under diffused daylight.

5.3 Report Enter the appearance in the report.

6. Color number test

The color number test shall be carried out, according to the degree of coloration, by Hasen's color number method for light color, and by Gardner's color number method for deep color.

(1) Hasen's color number method

(1.1) Apparatus The apparatus shall be as follows:

(a) Test tube The test tube shall be that specified in JIS R 3503.

(b) Hasen's standard color solution Dissolve 1.246 g of the potassium chloroplatinate of special grade of JIS K 8163 and 1.000 g of the cobalt chloride of special grade of JIS K 8129 into 100 ml of the hydrochloric acid of special grade of JIS K 8180, make the solution 1000 ml by adding distilled water or demineralized water to prepare the standard stock solution.

Make the Hasen's standard color solution by diluting this standard stock solution with distilled water or demineralized water at the rate shown in Table 3, put about 100 ml of this solution into the test tube of (a), tightly plug the tube and store it in a cool and dark place.

Table 3. Hasen's color standard solution

Unit: ml

Hasen's color number	Standard stock solution	Distilled water or demineralized water	Hasen's color number	Standard stock solution	Distilled water or demineralized water
0	0	100	100	20	80
10	2	98	120	24	76
20	4	96	140	28	72
30	6	94	160	32	68
40	8	92	180	36	64
50	10	90	200	40	60
60	12	88	250	50	50
70	14	86	300	60	40
80	16	84	400	80	20
90	18	82	500	100	0

- (1.2) Procedure Take the liquid sample of almost the same volume as the standard color solution contained in test tube, into a new test tube, and stand both test tubes vertically side by side, after the bubbles disappear. Place white paper or white screen behind the tubes, carry out colorimetry visually from the side under diffused daylight, select the standard color solution corresponding to the sample and take it as the color number of the sample.

(2) Gardner's color number method

- (2.1) Apparatus The apparatus shall be as follows:

- (a) Gardner's color-number tube The Gardner's color number tube means one set of 18 tubes made of colorless transparent glass tube to the shape and dimensions illustrated in Fig. 1 and numbered from 1 to 18. Each tube contains the Gardner's standard color solution of the respective number.
- (b) Gardner's standard color solution The Gardner's standard color solution is the solution of the composition shown in Table 4, and when the color of solution is measured in accordance with JIS Z 8722 on the solution of 10 mm layer, the measured chromaticity coordinates shall coincide with the values of Table 4.

Store the Gardner's standard color solution in a cool and dark place.

Fig. 1. Gardner's color number tube

Unit: mm

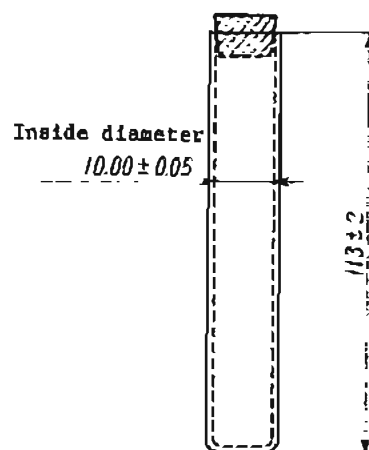


Table 4. Composition and chromaticity coordinates of Gardner's standard color solution

Gardner's color number	Potassium chloro-platinate solution ⁽¹⁾	Iron cobalt solution			Chromaticity coordinates	
	Number of grams of potassium chloro-platinate to be dissolved in 1000 ml of 0.1 mol/l hydrochloric acid	Ferrous chloride solution ⁽²⁾ Number of ml	Cobalt chloride solution ⁽³⁾ Number of ml	Hydrochloric acid (1+17) Number of ml	x	y
1	0.550	—	—	—	0.319 0	0.327 1
2	0.865	—	—	—	0.324 1	0.334 4
3	1.330	—	—	—	0.331 5	0.345 6
4	2.080	—	—	—	0.343 3	0.363 2
5	3.035	—	—	—	0.357 8	0.382 0
6	4.225	—	—	—	0.375 0	0.404 7
7	6.400	—	—	—	0.402 2	0.436 0
8	7.900	—	—	—	0.417 9	0.453 5
9	—	3.8	3.0	93.2	0.433 8	0.464 8
10	—	5.1	3.6	91.3	0.449 0	0.477 5
11	—	7.5	5.3	87.2	0.483 6	0.480 5
12	—	10.8	7.6	81.6	0.508 4	0.463 9
13	—	16.6	10.0	73.4	0.539 5	0.445 1
14	—	22.2	13.3	64.5	0.565 4	0.429 5
15	—	29.4	17.6	53.0	0.587 0	0.411 2
16	—	37.8	22.8	39.4	0.606 0	0.393 3
17	—	51.3	25.6	23.1	0.627 5	0.372 5
18	—	100.0	0.0	0.0	0.647 5	0.352 5

Note ⁽¹⁾ Potassium chloroplatinate solution Take 1000 ml of 0.1 mol/l hydrochloric acid, and dissolve potassium chloroplatinate of the amount shown in Table 4 into the acid to prepare this solution.

⁽²⁾ Ferrous chloride solution Dissolve about 5 portions of ferrous chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), in mass ratio, by adding about 1.2 portions of hydrochloric acid (1+17) and adjust the color by adding hydrochloric acid (1+17) so that the color becomes equal to the solution immediately after making by dissolving about 3.0 g of potassium dichromate into 100 ml of sulfuric acid (specific gravity 1.84), to prepare this solution.

⁽³⁾ Cobalt chloride solution Dissolve one portion of cobalt chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$), in mass ratio, by adding 3 portions of hydrochloric acid (1+17) to prepare this solution.

- (2.2) Procedure The procedure shall be as specified in (1.2).
However, colored glass of the same color may be used in lieu of the Gardner's standard color number tube.

(3) Report Enter the following items in the report:

- (a) Kind of test method
- (b) Color number

7. Specific gravity test

7.1 Equipment and apparatus The equipment and apparatus shall be as follows:

- (1) Hydrometer The hydrometer specified in JIS B 7525.
- (2) Measuring cylinder The measuring cylinder of 200 ml capacity specified in JIS R 3505.
- (3) Even balance The even balance of weighing capacity 500 g and sensitivity 0.5 g.

7.2 Procedure The procedure shall be as follows:

- (1) Hydrometer method Carry out the measurement at $25 \pm 0.5^\circ\text{C}$ by using the hydrometer of 7.1 (1).
- (2) Measuring cylinder method Put the sample of about 100 ml into a measuring cylinder whose mass is known taking care not to include bubbles, measure the mass with the even balance and calculate the specific gravity from the formula given below. Previously maintain the sample and measuring cylinder at a temperature $25 \pm 0.5^\circ\text{C}$.

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

where, d : specific gravity

m_1 : mass of measuring cylinder (g)

m_1 : mass of measuring cylinder containing sample (g)

m_2 : mass of water equal to volume of sample (g)

7.3 Report Enter the following items in the report:

- (1) Kind of test method (hydrometer method or measuring cylinder method)
- (2) Specific gravity

8. Viscosity test

The viscosity test shall be as specified in 8. (viscosity test) of JIS C 2103.

9. Relative viscosity change test

9.1 Equipment and apparatus The equipment and apparatus shall be as follows:

- (1) Viscometer The Brookfield viscometer shall be used.
- (2) Tall beaker The tall beaker of 300 ml capacity specified in JIS R 3503.

9.2 Procedure Add the specified amount of the appropriate curing agent specified for each product to the product under test and mix them uniformly to prepare the specimen. Take about 250 g of the specimen so prepared into the tall beaker, measure the viscosity at the prescribed temperature, slightly plug the beaker, allow it to stand at the prescribed temperature for the prescribed duration, and measure the viscosity of specimen again at the temperature of first measurement.

9.3 Calculation Carry out the calculation by means of following formula:

$$\eta_v = \frac{\eta - \eta_0}{\eta_0} \times 100$$

where, η_0 : relative viscosity change (%)

η : viscosity of sample after addition of curing agent
[mPa·s{cP} or Pa·s{P}]

η : viscosity of sample after allowed to stand [mPa·s{cP}
or Pa·s{P}]

9.4 Report Enter the following items in the report:

- (1) Test conditions
 - (a) Name and added amount of curing agent (g)
 - (b) Temperature (°C) and duration (h) of standing
 - (c) Temperature at viscosity measurement (°C), rotor number and number of revolutions of rotor (min⁻¹)
- (2) Relative viscosity change (%)

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10. Ash content test

10.1 Equipment and apparatus The equipment and apparatus shall be as follows:

- (1) Chemical balance A chemical balance with reciprocal sensitivity 1 mg.
- (2) Crucible A ceramic crucible for chemical analysis or a platinum crucible for chemical analysis.
- (3) Burning equipment The burning equipment shall be an electric furnace or a gas burner which can heat the specimen up to 900°C.
- (4) Desiccator The desiccator specified in JIS R 3503.

10.2 Procedure Put the sample of about 10 g in the crucible of an adequate size which has been previously made a constant weight by strong heating, and measure the mass correctly to the nearest 1 mg with a chemical balance. Place the lid on the crucible and place the crucible in the burning equipment previously heated at 200°C to 280°C. Progressively raise the temperature of burning equipment unit it reaches $900 \pm 25^\circ\text{C}$, and then maintain this temperature for 2 h. The lid of crucible may be removed during the heating, if necessary, but it shall be placed before completion of the heating. After a lapse of two hours, cool the crucible to the room temperature in a desiccator for about 30 min, and immediately measure the mass to the nearest 1 mg with the chemical balance.

10.3 Calculation Carry out the calculation by means of the following formula:

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

where, A_s : ash content (%)

m_0 : mass of empty crucible (g)

m_1 : mass of sample and crucible before heating (g)

m_2 : mass of sample and crucible after heating (g)

10.4 Report Enter the following items in the report:

- (1) Test conditions [heating temperature (°C) and duration of heating (h)]
- (2) Ash contents (%)

11. Filler content test

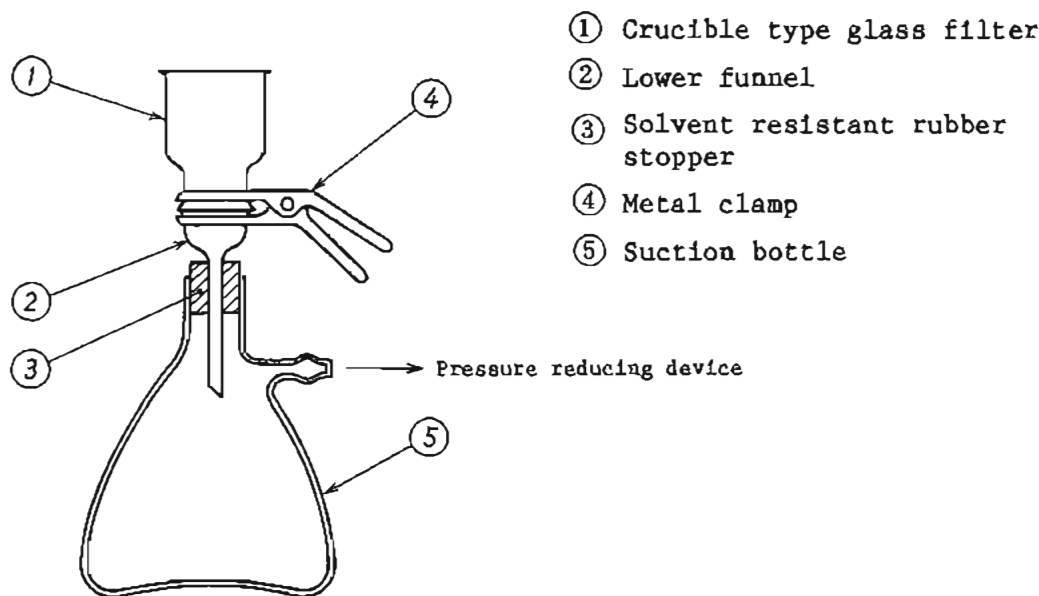
11.1 Igniting method The igniting method shall be as stated in 10.

11.2 Extraction method

11.2.1 Equipment and apparatus The equipment and apparatus shall be as follows:

- (1) Chemical balance A chemical balance with reciprocal sensitivity 1 mg.
- (2) Conical flask (Erlenmeyer flask) The Erlenmeyer flask specified in JIS R 3503.
- (3) Crucible type glass filter The crucible type sintered-glass filter specified in JIS R 3503.
- (4) Desiccator The desiccator specified in JIS R 3503.
- (5) Thermostatic chamber A thermostatic chamber of hot air circulating type which can maintain the prescribed temperature.
- (6) Equipment The equipment consists of a crucible type sintered-glass filter, a lower funnel and a suction bottle as illustrated in Fig. 2. Fix the crucible type filter and the lower funnel by gripping with a metal clamp.

Fig. 2. Filtering equipment (an example)



11.2.2 Solvent Use the acetone specified in JIS K 8034, methylene chloride or other suitable solvent.

11.2.3 Preparation of crucible type glass filter Dry the crucible type glass filter in a thermostatic chamber maintained at 105°C to 110°C for 30 min, take it out of the chamber, allow it to cool in a desiccator at the room temperature for about 30 min and weigh it. Repeat the drying, cooling and weighing until the crucible type glass filter becomes a constant weight, and then attach it to the filtering equipment. Unless otherwise specified the desiccant in the desiccator shall be silica gel.

11.2.4 Procedure The procedure shall be as follows:

- (1) Take about 3 g of sample in a conical flask and weigh them correctly to the nearest 1 mg. Completely dissolve the sample by adding the solvent of 50 ml gradually at the room temperature while shaking the conical flask is continuously.
- (2) Wet the crucible type glass filter attached to the filtering equipment with a small amount of the solvent, carefully pour the sample solution in the conical flask into the crucible type glass filter of the filtering equipment by tilting method and carry out the suction gradually by adjusting the suction pressure until the sample solution finished its dripping through the crucible type glass filter. Wash the conical flask with a small amount of the solvent and transfer the insoluble content to the crucible type glass filter while washing. Use a stirring stick of glass or the like to completely transfer the insoluble contents, if necessary.
- (3) Clean the crucible type glass filter using a small amount of solvent each time until the filtrate becomes colorless.
- (4) After removal of solvent by sufficient suction, detach the crucible type glass filter from the filtering equipment.
- (5) Dry the crucible type glass filter to which the insoluble content is adhering in the thermostatic chamber at a temperature appropriate for the solvent used.
- (6) Take out the crucible type glass filter from the thermostatic chamber, cool it in the desiccator for about 30 min and then weigh it. Repeat the drying, cooling and weighing until the crucible type glass filter becomes a constant weight.

11.2.5 Calculation Carry out the calculation by means of the following formula:

$$F_c = \frac{m_1}{m_s} \times 100$$

where, F_c : filler content (%)

m_s : mass of sample (g)

m_1 : increment in mass of crucible type glass filter (g)

11.3 Report Enter the following items in the report:

- (1) Test method (state whether the igniting method or extraction method is used)
- (2) Test conditions [heating conditions (temperature and duration), for igniting method; name, number and pore symbol of filter plate of crucible type glass filter, and kind of solvent used (e.g. crucible type filter IG3, acetone) for extraction method.]
- (3) Filler content (%)

12. Volatile content test

12.1 Equipment and apparatus The equipment and apparatus shall be as follows:

- (1) Thermostatic chamber A thermostatic chamber of hot air circulating type which can maintain the prescribed temperature.
- (2) Flat-bottom dish A flat-bottom dish made of glass or metal with a inside diameter of about 60 mm and a depth of about 20 mm.
- (3) Chemical balance A chemical balance with reciprocal sensitivity 1 mg.
- (4) Desiccator The desiccator specified in JIS R 3503.

12.2 Procedure Take about 1.5 g of resin from the sample and put it on the flat-bottom dish previously weighed, measure their mass correctly to the nearest 1 mg, place the specimen horizontally in a thermostatic chamber, and heat it at the prescribed temperature for the prescribed duration. Cool the specimen in the desiccator and then measure the mass to the nearest 1 mg.

12.3 Calculation Carry out the calculation by means of the following formula:

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

where, V_m : volatile content (%)

m_0 : mass of flat-bottom dish (g)

m_1 : mass of flat-bottom dish and sample before heating (g)

m_2 : mass of flat-bottom dish and sample after heating (g)

12.4 Report Enter the following items in the report:

- (1) Test conditions [heating temperature (°C) and duration of heating (h)]
- (2) Volatile content (%)

13. Acid-value test

13.1 Equipment and apparatus The equipment and apparatus shall be as follows:

- (1) Chemical balance A chemical balance with reciprocal sensitivity 1 mg.
- (2) Buret The buret of 25 ml capacity specified in JIS R 3505.
- (3) Conical flask The conical flask of 300 ml capacity specified in JIS R 3503.