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Wood preservatives

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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 Wood Preserving Association (JWPA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14. Consequently **JIS K 1570 : 1998** is replaced with this Standard. There is the possibility that the compliance with this Standard may infringe on the following patent right.

Title of invention: Antiseptic compositions

Date of registration of creation: October 27, 1992

This description does not give any influence on the validity or scope of the above-mentioned patent right.

The patentee of this patent right has assured to the Japanese Industrial Standards Committee that the patentee has an intention to consent to any person's execution of the said patent right under the non-discriminatory and reasonable condition.

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Wood preservatives

1 Scope This Japanese Industrial Standard specifies the wood preservatives to be used for the injection treatment to give the antiseptic property and termite proofing property to wood.

2 Normative references The standards listed in attached table 1 contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards (including amendments) shall be applied.

3 Definitions For the purpose of this Standard, the following definitions apply.

- a) **wood preservatives**, the chemicals for wood treatment having antiseptic performance and/or termite proofing property.
- b) **injection treatment**, the treatment to penetrate wood preservative into wood by using the mechanical pressure difference such as depressurizing, pressurizing.
- c) **oil based wood preservatives**, the wood preservatives in oily state to be used by stock solution as it is.
- d) **oil soluble wood preservatives**, the wood preservatives to be used by dissolving in organic solvent.
- e) **water soluble wood preservatives**, the wood preservatives to be used by dissolving in water.
- f) **emulsified wood preservatives**, the wood preservatives to be used by emulsifying in water.
- g) **designated concentration**, the concentration of chemicals solution required for ensuring the quantity of absorption corresponding to the product standard of the treated products respectively. That shall be designated by the manufacturers.

4 Classification The classification of wood preservatives shall be in accordance with table 1.

Table 1 Classification of wood preservatives

Division	Class		Symbol of class
Water soluble wood preservative	Quarternary ammonium compound series	No. 1	AAC-1
		No. 2	AAC-2
	Copper/quarternary ammonium compound series	No. 1	ACQ-1
		No. 2	ACQ-2
	Copper/azole compound series	No. 1	CUAZ-1
		No. 2	CUAZ-2
		No. 3	CUAZ-3
	Boron/quarternary ammonium compound series		BAAC
	Quaternary ammonium/non-ester pyrethroid compound series		SAAC
	Azole/quarternary ammonium/non-ester pyrethroid compound series		AZAAC
Emulsified wood preservatives	Metallic salts of fatty acid series	No. 1	NCU-E
		No. 2	NZN-E
		No. 3	VZN-E
Oil soluble wood preservatives	Metallic salts of naphthenic acid series	No. 1	NCU-O
		No. 2	NZN-O
	Azole/neonicotinoid compound series		AZN
Oil based wood preservatives	Creosote oil		A

5 Quality The quality shall conform to tables 2 to 11 respectively when tested according to clause 7 corresponding to the class of wood preservatives.

Table 2 Quality of quaternary ammonium compound series of wood preservatives

Item		Quality	
		No. 1 (AAC-1)	No. 2 (AAC-2)
Compound ratio of effective composition (mass percentage %)	Didecyl dimethyl ammonium chloride	30 to 55	—
	N,N-Didecyl-N-methyl-polyoxyethyl-ammonium propionate	—	30 to 55
State of product		Liquid state	
Water insoluble content (mass percentage %)		1 max.	
pH		5.0 to 8.0	

Table 3 Quality of copper/quaternary ammonium compound series of wood preservatives

Item		Quality	
		No. 1 (ACQ-1)	No. 2 (ACQ-2)
Compound ratio of effective composition (mass percentage %)	Copper compound (as CuO)	53 to 59	62 to 71
	N-Alkyl benzyl dimethyl ammonium chloride	41 to 47	—
	Didecyl dimethyl ammonium chloride	—	29 to 38
Total effective composition ⁽¹⁾ (mass percentage %)		At least marked content. 16 min.	At least marked content. 13 min.
State of product		Liquid state	
Water insoluble content (mass percentage %)		1 max.	
pH		9.5 to 11.0	

Note ⁽¹⁾ The total effective composition means the total amount of copper compound and N-alkyl benzyl dimethyl ammonium chloride or didecyl dimethyl ammonium chloride.

Table 4 Quality of copper/azole compound series of wood preservatives

Item		Quality		
		No. 1 (CUAZ-1)	No. 2 (CUAZ-2)	No. 3 (CUAZ-3) ⁽³⁾
Compound ratio of effective composition (mass percentage %)	Copper compound (as CuO)	53 to 57	98.6 to 99.0	
	Boron compound (as H ₃ BO ₃)	40 to 46	—	
	Tebuconazole [α-(2-(4-chlorophenyl) ethyl)-α-(1,1-dimethyl ethyl)-1H-1,2,4-triazole-1-ethanol]	1.6 to 2.4	—	
	Cyproconazole [α-(4-chlorophenyl)-α-(1-cyclopropyl ethyl)-1H-1,2,4-triazole-1-ethanol]	—	1.0 to 1.4	
Total effective composition ⁽²⁾ (mass percentage %)		At least marked content. 20 min.	At least marked content. 11 min.	
State of product		Liquid state		
Water insoluble content (mass percentage %)		1 max.		
pH		9.0 to 10.0	8.7 to 9.3	8.7 to 11.5

Notes ⁽²⁾ The total effective composition means the total amount of copper compound, tebuconazole or cyproconazole and boron compound.

⁽³⁾ CUAZ-3 includes lignin sulfonate as auxiliaries in addition to this.

Table 5 Quality of boron/quaternary ammonium compound series of wood preservatives⁽⁴⁾

Item		Quality
Compound ratio of effective composition (mass percentage %)	Boron compound (as H ₃ BO ₃)	23 to 27
	Didecyl dimethyl ammonium chloride	71 to 79
Total effective composition ⁽⁵⁾ (mass percentage %)		At least marked content. 25 min.
State of product		Liquid state
Water insoluble content (mass percentage %)		1 max.
pH		5.0 to 9.0

Notes ⁽⁴⁾ Regarding the wood preservatives of 5.0 to 8.0 in pH among them, there is the following patent.

Patent registration: No. 1707718, "Antiseptic compositions"

Date of registration: October 27, 1992

⁽⁵⁾ The total effective composition means the total amount of boron compound and didecyl dimethyl ammonium chloride.

Table 6 Quality of quaternary ammonium/non-ester pyrethroid compound series of wood preservatives

Item		Quality
Compound ratio of effective composition (mass percentage %)	N,N-Didecyl-N-methyl-polyoxyethyl-ammonium propionate	99.0 to 99.9
	Silafluofen [4-Ethoxy phenyl [3-(4-fluoro-3-phenoxy-phenyl) propyl] dimethyl silane]	0.4 to 0.6
Total effective composition ⁽⁶⁾ (mass percentage %)		Any larger one of either at least marked content or 35
State of product		Liquid state
Water insoluble content (mass percentage %)		1 max.
pH		5.0 to 8.0

Note ⁽⁶⁾ The total effective composition means the total amount of N,N-didecyl-N-methyl-polyoxyethyl-ammonium propionate and 4-ethoxyphenyl [3-(4-fluoro-3-phenoxyphenyl) propyl] dimethyl silane.

Table 7 Quality of azole/quaternary ammonium/non-ester pyrethroid compound series of wood preservatives

Item		Quality
Compound ratio of effective composition (mass percentage %)	N,N-Didecyl-N-methyl-polyoxyethyl-ammonium propionate	96.7 to 97.3
	Cyproconazole [α -(4-chlorophenyl)- α -(1-cyclopropyl ethyl)-1H-1,2,4-triazole-1-ethanol]	0.5 to 0.7
	Ethofenprox [2-(4-ethoxy phenyl)-2-methyl propyl-3-phenoxy benzyl ether]	2.2 to 2.6
Total effective composition ⁽⁷⁾ (mass percentage %)		Any larger one of either at least marked content or 40
State of product		Liquid state
Water insoluble content (mass percentage %)		1 max.
pH		5.0 to 8.0

Note ⁽⁷⁾ The total effective composition means the total amount of N,N-didecyl-N-methyl-polyoxyethyl-ammonium propionate, cyproconazole and ethofenprox.

Table 8 Quality of metallic salts of fatty acid series of wood preservatives

Item		Quality		
		No. 1 (NCU-E)	No. 2 (NZN-E)	No. 3 (VZN-E)
Compound ratio of effective composition (mass percentage %)	Copper naphthenate (as Cu)	6.0 to 6.6	—	—
	Zinc naphthenate (as Zn)	—	8.0 to 8.8	—
	Tertiary zinc carboxylate (as Zn)	—	—	5.0 to 5.5
	Permethrin [3-phenoxy benzyl=2-(2,2-dichloro vinyl)-3,3-dimethyl-1-cyclopropane carboxylate]	—	—	0.50 to 0.55
Acid value of fatty acid ⁽⁸⁾		180 to 300		318 to 328
State of product		Liquid state		
pH		6.0 to 7.0		

Note ⁽⁸⁾ The fatty acid means the carboxylic acid of terpene series.

Table 9 Quality of metallic salts of naphthenic acid series of wood preservatives

Item		Class and quality	
		No. 1 (NCU-O)	No. 2 (NZN-O)
Compound ratio of effective composition (mass percentage %)	Copper naphthenate (as Cu)	5.0 to 11.0	—
	Zinc naphthenate (as Zn)	—	5.0 to 11.0
Acid value of naphthenic acid		180 to 300	
State of product		Liquid state	

Table 10 Quality of azole/neonicotinoid compound series of wood preservatives

Item		Class and quality
Compound ratio of effective composition (mass percentage %)	Cyproconazole [α -(4-chlorophenyl)- α -(1-cyclopropyl ethyl)-1H-1,2,4-triazole-1-ethanol]	62 to 72
	Imidacloprid [1-[(6-chloro-3-pyridinyl) methyl]-4,5-dihydro-N-nitro-1H-imidazol-2-amine]	28 to 38
Total effective composition ⁽⁹⁾ (mass percentage %)		Any larger one of either at least marked content or 3.6
State of product		Liquid state

Note ⁽⁹⁾ The total effective composition means the total amount of cyproconazole and imidacloprid.

Table 11 Quality of creosote oil of wood preservatives⁽¹⁰⁾

Item		Quality
Density (g/cm ³ , 40 °C)		1.03 min.
Moisture content (volume percentage %)		3 max.
Engler degree (40/20 °C)		2.0 max.
Distillation test (dehydrated sample) (volume percentage %)	Percent recovered up to 235 °C	25 max.
	Percent recovered between 235 °C and 315 °C	40 min.
	Percent recovered up to 315 °C	50 min.
Density of fraction between 235 °C and 315 °C (g/cm ³ , 40 °C)		1.02 min.
Toluene insoluble content (dehydrated sample) (mass percentage %)		0.5 max.
Fluidity test		Noticeable solid matter shall not be produced.

Note ⁽¹⁰⁾ The creosote oil of wood preservatives and the products treated by that shall not be used for the use other than industrial use such as rail road sleeper use. Provided that this rule shall be excluded for 10 ppm or under content of benzo (a) pyrene, benz (a) anthracene and dibenz (a,h) anthracene respectively.

6 Performance The performance of wood preservatives shall conform to the requirements of performance specified in clause 6 of **JIS K 1571** when tested at the respectively specified concentration according to 4.2.1.1, 4.2.2, 4.2.3, 4.3.1.1, 4.3.2.1 and 4.4.1 of **JIS K 1571**.

7 Test methods

7.1 General matter Unless specified especially in this Standard, the general matter shall be as follows:

- The common matter in methods of chemical analysis shall be in accordance with **JIS K 0050**.
- The rounding-off method for numerical values shall be in accordance with **JIS Z 8401**.
- Atomic absorption spectrochemical analysis shall be in accordance with **JIS K 0121**.
- Molecular absorption spectrophotometry shall be in accordance with **JIS K 0115**.
- ICP atomic emission spectrometry shall be in accordance with **JIS K 0116**.
- High performance liquid chromatographic analysis shall be in accordance with **JIS K 0124**.
- Gas chromatographic analysis shall be in accordance with **JIS K 0114**.
- The glass instruments to be used for test shall be in accordance with **JIS R 3503**.
- The glass volumenometer to be used for test shall be in accordance with **JIS R 3505**.

7.2 Quaternary ammonium compound series of wood preservatives

7.2.1 Sampling method The sampling method shall be in accordance with **JIS K 2420**. Provided that the sample shall be taken from an optional place under well-mixed condition.

7.2.2 Preparation of sample solution Weigh out the sample to the nearest 0.1 mg so as to be approximately 10 g as didecyl dimethyl ammonium chloride (hereafter referred to as "DDAC") or N,N-didecyl-N-methyl-polyoxyethyl-ammonium propionate (hereafter referred to as "DMPAP"), and put it in a 1 000 ml volumetric flask, add water up to the marked line, and take it as the sample solution.

7.2.3 DDAC

7.2.3.1 Titration method

- a) **Summary** DDAC, being a cation, is titrated by using anion solution as agitating in water-chloroform system with BPB as an indicator.
- b) **Reagents** The reagents shall be as follows:
 - 1) **Anion solution (0.002 mol/L)**, prepared so that 0.684 5 g of sodium tetraphenyl borate is weighed out and dissolved in water to make 1 000 ml.
 - 2) **Buffer solution (pH 10)**, prepared so that 100 g of sodium carbonate specified in **JIS K 8625** and 100 g of sodium sulfate specified in **JIS K 8987** are dissolved in water to make 1 000 ml.
 - 3) **BPB solution (1 g/L)**, prepared so that 0.1 g of bromophenol blue specified in **JIS K 8844** is dissolved in water to make 100 ml.
 - 4) **Chloroform**, specified in **JIS K 8322**.
- c) **Operation** Weigh out 1 ml of sample solution with using a volumetric pipette, pour in a 200 ml Erlenmeyer flask, add 50 ml of buffer solution, 50 ml of chloroform and five drops of BPB solution (1 g/L), and agitate it as stoppering. Add anion solution (0.002 mol/L) repeatedly little by little by using a microburette, agitate well every time of adding, and confirm the change of colour. Take the point, where the blue colour of chloroform layer varnishes and the water layer colours to pale purple, as the end point of titration, and calculate the quantity of DDAC according to formula (1) and formula (2) to round off to two significant figures.

$$A_1 = \frac{t \times 0.724 \times 100}{1\,000} \dots\dots\dots (1)$$

where, A_1 : mass of DDAC in the sample weighed out in **7.2.2** (g)
 t : quantity of anion solution (0.002 mol/L) required for titration (ml)
0.724: mass of DDAC equivalent to 1 ml of anion solution (0.002 mol/L) (mg)

$$A = \frac{A_1}{S} \dots\dots\dots (2)$$

where, A : DDAC content in the sample weighed out in **7.2.2**
 (mass percentage %)

 S : mass of the sample weighed out in **7.2.2** (g)

7.2.3.2 High performance liquid chromatography

- a) **Summary** The sample is electrically neutralized by an ion pair indicator, and determined according to the high performance liquid chromatography with UV absorptiometric detection.
- b) **Reagents** The reagents shall be as follows:
 - 1) **DDAC**, refined for determination and known of the purity previously.
 - 2) **Sodium 2-naphthalene sulfonate solution**, prepared so that 575 mg of sodium 2-naphthalene sulfonate is weighed out, poured in a 100 ml volumetric flask and water is added up to the masked line.
 - 3) **Phosphoric acid solution**, prepared so that 17 ml of phosphoric acid specified in **JIS K 9005** is put in a 100 ml volumetric acid and water is add up to the marked line.
 - 4) **Methanol**, for high performance liquid chromatography.
 - 5) **Mobile phase**, prepared as follows: Weigh out 780 mg of sodium dihydrogenphosphate dihydrate specified in **JIS K 9009**, pour in a 500 ml volumetric flask, add 10 ml of the solution prepared in 2) and 2 ml of the solution prepared in 3) respectively and add water up to the marked line. Mix this solution and methanol at the rate of 1 to 4 in volume percentage and take it as the mobile pleas.
 - 6) **Standard solution for working curve**, prepared so that approximately 20 mg of DDAC is weighed out to the nearest 0.1 mg, poured in a 100 ml volumetric flask, and the mobile phase prepared in 5) is added up to the marked line.
- c) **Apparatus** For the apparatus, a high performance liquid chromatograph shall be used.
- d) **Analytical condition of high performance liquid chromatography** The analytical condition of high performance liquid chromatography shall be in accordance with table 12.

Table 12 Analytical condition of high performance liquid chromatography

Item	Condition
Column	ODS system of column I.D. 4.6 mm × L. 150 mm
Mobile phase	In accordance with 7.2.3.2 b) 5).
Mobile phase flow rate	1.0 ml/min
Column temperature	40 °C
Measuring wavelength	275 nm
Range	0.016 AUFS
Injection amount	10 µl

- e) **Operation** Weigh out 1 ml of the sample solution by using a volumetric pipette, pour it in a 100 ml volumetric flask, pour mobile phase up to the marked line, and take it as the analytical sample solution. Inject 10 µl of the analytical sample solution into the high performance liquid chromatograph, and obtain the peak area of DDAC. Obtain the DDAC concentration in the analytical sample solution from working curve, and calculate the content in the sample according to formula (3) and formula (4).

$$D_1 = D_0 \times \frac{100}{1\,000} \dots\dots\dots (3)$$

where, D_1 : mass of DDAC in the sample weighed out in **7.2.2** (g)
 D_0 : concentration of DDAC in the analytical sample solution obtained from working curve (mg/L)

$$D = \frac{D_1}{W} \times 100 \dots\dots\dots (4)$$

where, D : DDAC content in the sample weighed out in **7.2.2** (mass percentage %)
 W : mass of the sample weighed out in **7.2.2** (g)

- f) **Preparation of working curve** Dilute the standard solution for working curve by mobile phase and prepare stepwise so as to be approximately 50 mg/L to 150 mg/L in the concentration of DDAC. Operate this solution similarly and prepare the working curve of DDAC concentration to peak area. The preparation of working curve shall be carried out each time when used.

7.2.4 DMPAP

7.2.4.1 Titration method

- a) **Summary** DMPAP, being a cation, is titrated by using anion solution as agitating in water-chloroform system with BPB as an indicator.
- b) **Reagents** The reagents shall be as follows:

- 1) **Anion solution (0.002 mol/L)**, prepared so that 0.684 5 g of sodium tetraphenyl borate is weighed out and dissolved in water to make 1 000 ml.
 - 2) **Buffer solution (pH 10)**, prepared so that 100 g of sodium carbonate specified in **JIS K 8625** and 100 g of sodium sulfate specified in **JIS K 8987** are dissolved in water to make 1 000 ml.
 - 3) **BPB solution (1 g/L)**, prepared so that 0.1 g of bromophenol blue specified in **JIS K 8844** is dissolved in water to make 100 ml.
 - 4) **Chloroform**, specified in **JIS K 8322**.
- c) **Operation** Weigh out 1 ml of the sample solution by using a volumetric pipette, pour it in a 200 ml Erlenmeyer flask, add 50 ml of buffer solution, 50 ml of chloroform and 5 drops of BPB solution (1 g/L) and agitate as stoppering. Add anion solution (0.002 mol/L) little by little repeatedly by using a microburette, agitate every time of adding, and confirm the change of colour. Take the point, where the blue colour of chloroform layer varnishes and the water layer colour to paint purple, as the end point of titration, and calculate the quantity of DMPAP according to formula (5) and formula (6) to round-off to two significant figures.

$$A_1 = \frac{t \times 0.908 \times 100}{1\ 000} \dots\dots\dots (5)$$

where, A_1 : mass of DMPAP in the sample weighed out in **7.2.2** (g)
 t : quantity of anion solution (0.002 mol/L) required for titration (ml)
 0.908 : mass of DMPAP equivalent to 1 ml of 0.002 mol/L anion solution (mg)

$$A = \frac{A_1}{S} \times 100 \dots\dots\dots (6)$$

where, A : DMPAP content (mass percentage %)
 S : mass of the sample weighed out in **7.2.2** (g)

7.2.4.2 High performance liquid chromatography

- a) **Summary** The sample is electrically neutralized by an ion pair indicator and DMPAP is determined according to the high performance liquid chromatography with UV absorptiometric detection.
- b) **Reagents** The reagents shall be as follows:
 - 1) **DMPAP**, refined for determination and known of the purity previously.
 - 2) **Sodium 2-naphthalene sulfonate solution**, prepared so that 575 mg of sodium 2-naphthalene sulfonate is weighed out, put in a 100 ml volumetric flask and water is added up to the marked line.
 - 3) **Phosphoric acid solution**, prepared so that 17 ml of phosphoric acid specified in **JIS K 9005** in a 100 ml volumetric flask and water is added up to the marked line.
 - 4) **Methanol**, for high performance liquid chromatography.

- 5) **Mobile phase**, prepared as follows: Weigh out 780 mg of sodium dihydrogen-phosphate dihydrate specified in **JIS K 9009**, put in a 500 ml volumetric flask, further add 10 ml of the solution prepared in 2) and 2 ml of the solution prepared in 3) respectively, and add water up to the marked line. Mix this solution and methanol at the rate of 1 to 4 in volume percentage and take it as the mobile phase.
- 6) **Standard solution for working curve**, prepared so that approximately 20 mg of DMPAP is weighed out to the nearest 0.1 mg, put in a 100 ml volumetric flask and the mobile phase prepared in 5) is added up to the marked line.
- c) **Apparatus** For the apparatus, a high performance liquid chromatograph shall be used.
- d) **Analytical condition of high performance liquid chromatography** The analytical condition of high performance liquid chromatography shall be in accordance with table 13.

Table 13 Analytical condition of high performance liquid chromatography

Item	Condition
Column	ODS system of column I.D. 4.6 mm × L. 150 mm
Mobile phase	In accordance with 7.2.4.2 b) 5) .
Mobile phase flow rate	1.0 ml/min
Column temperature	40 °C
Measuring wavelength	275 nm
Range	0.016AUFS
Injection amount	10 µl

- e) **Operation** Weigh out 1 ml of the sample solution by using a volumetric pipette, pour it in a 100 ml volumetric flask, pour mobile phase up to the marked line and take it as the analytical sample solution. Inject 10 µl of the analytical sample solution into high performance liquid chromatograph, and obtain the peak area of DMPAP. Obtain the DMPAP concentration in the analytical sample solution from working curve, and calculate the content in the sample according to formula (7) and formula (8).

$$M_1 = M_0 \times \frac{100}{1\,000} \dots\dots\dots (7)$$

where, M_1 : mass of DMPAP in the sample weighed out in **7.2.2** (g)
 M_0 : concentration of DMPAP in the analytical sample solution obtained from working curve (mg/L)

$$M = \frac{M_1}{W} \times 100 \dots\dots\dots (8)$$

where, M : concentration of DMPAP in the sample weighed out in **7.2.2** (mass percentage %)

W : mass of the sample weighed out in **7.2.2** (g)

- f) **Preparation of working curve** Dilute the standard solution for working curve by mobile phase and prepare stepwise so as to be approximately 50 mg/L to 150 mg/L of the concentration of DMPAP. Operate this solution similarly and prepare the working curve of DMPAP concentration to peak area. The preparation of working curve shall be carried out each time when used.

7.2.5 State of product The state of product shall be visually checked.

7.2.6 Water insoluble content The water insoluble content shall be as follows.

7.2.6.1 Operation Agitate the analytical sample solution sufficiently, weigh out 100 ml of the analytical sample solution by using a 100 ml measuring cylinder and filter by using a mass-known glass filter, 1 G4. Wash the residue with water sufficiently, dry it in a dryer at 105 °C to 110 °C until it reaches a constant weight, leave it cool in a desiccator as it is, and weigh the mass of residue.

7.2.6.2 Calculation The water insoluble content shall be calculated according to formula (9).

$$Q = \frac{R \times 10}{S \times A / 100} \times 100 \dots\dots\dots (9)$$

where, Q : water insoluble content (mass percentage %)

R : mass of residue (g)

A : concentration of DDAC or DMPAP (mass percentage %)

S : mass of the sample taken in **7.2.2** (g)

7.2.7 pH The pH shall be measured on the analytical sample solution prepared in **7.2.2** according to **JIS Z 8802**.

7.3 Copper/quaternary ammonium compound series of wood preservatives

7.3.1 Sampling method The sampling of representative sample shall be in accordance with **JIS K 2420**. Provided that the sample shall be taken from an optional place under well-mixed condition.

7.3.2 Preparation of sample solution Weigh out the sample to the nearest 0.1 mg so as to be approximately 0.8 g as effective composition, put it in a 250 ml volumetric flask, add water up to the marked line, and take it as the sample solution.

7.3.3 Copper compound

7.3.3.1 Atomic absorption spectrochemical analysis

- a) **Summary** The sample is dissolved in water, then copper is determined according to atomic absorption spectrochemical analysis.

- b) **Reagents** The reagents shall be as follows:
- 1) **Sulfuric acid**, specified in **JIS K 8951**.
 - 2) **Copper standard solution for working curve (100 mg/L as Cu)**, prepared so that 10 ml of copper standard solution (1 000 mg/L as Cu) specified in **JIS K 0010** is weighed out by using a volumetric pipette, poured in 100 ml flask and water is added up to the marked line.
- c) **Apparatus** For the apparatus, an atomic absorption spectrochemical analyzer shall be used.
- d) **Operation** Weigh out 2 ml of the sample solution by using a volumetric pipette, pour it in a 1 000 ml volumetric flask, add 8 ml sulfuric acid, then add water up to the marked line, and take it as the analytical sample solution. Spray the analytical sample solution into flame according to clause 6 of **JIS K 0121**, read the absorbance at 324.8 nm in wavelength, obtain the concentrations of copper in the analytical sample solution from the working curve and calculate the quantity of copper compound in the analytical sample solution according to formula (10).

$$C = \frac{c \times 1.252 \times 250 \times 1\,000}{10^6 \times 2} \dots\dots\dots (10)$$

where, C : mass of copper compound (as CuO) in the sample solution weighed out in **7.3.2** (g)

 c : concentration of copper (as Cu) in the analytical sample solution (mg/L)

 1.252 : factor to convert Cu into CuO

- e) **Preparation of working curve** Weigh out 0 ml to 5 ml of the copper standard solution for working curve (100 mg/L as Cu) stepwise by using a volumetric pipette, pour it in a 100 ml volumetric flask, add sulfuric acid so as to become the same condition as the analytical sample solution, and then add water up to the marked line. Operate this solution similarly and prepare the working curve of the quantity of copper to absorbance. The preparation of working curve shall be carried out each time when used.

7.3.3.2 ICP atomic emission spectrometry

- a) **Summary** The sample is dissolved in water, and then copper is determined according to ICP atomic emission spectrometry.
- b) **Reagents** The reagents shall be as follows:
- 1) **Sulfuric acid**, specified in **JIS K 8951**.
 - 2) **Copper standard solution for working curve (100 mg/L as Cu)**, being in accordance with **7.3.3.1 b) 2)**.
- c) **Apparatus** For the apparatus, an ICP atomic emission spectrometer shall be used.
- d) **Operation** Weigh out 2 ml of sample solution by using a volumetric pipette, pour it in a 1 000 ml volumetric flask, add 8 ml of sulfuric acid, then add water up to the marked line, and take it as the analytical sample solution. Measure the emission intensity of copper in the analytical sample solution by using ICP

atomic emission spectrometer, obtain the concentration of copper in the analytical sample solution from the working curve, and calculate the mass of copper compound in the sample solution according to formula (11).

$$C = \frac{c \times 1.252 \times 250 \times 1\,000}{10^6 \times 2} \dots\dots\dots (11)$$

where, C : mass of copper compound (as CuO) in the sample solution weighed out in **7.3.2** (g)

c : concentration of copper in the analytical sample solution (mgCu/L)

1.252 : factor to convert Cu into CuO

7.3.4 N-Alkylbenzyl dimethyl ammonium chloride

7.3.4.1 Summary N-Alkylbenzyl dimethyl ammonium chloride (hereafter referred to as “BKC”) is titrated by using sodium lauryl sulfate solution as agitating in water-chloroform system with using methylene blue as an indicator.

7.3.4.2 Reagents The reagents shall be as follows:

- a) **Sodium lauryl sulfate solution (0.005 mol/L)**, prepared so that approximately 1.5 g of sodium lauryl sulfate, of which the purity is previously measured according to **JIS K 3362**, is weighed out to the nearest 0.1 mg, and dissolved in water to make 1 000 ml.
- b) **Methylene blue solution (0.03 g/L)**, prepared as follows: Add 12 g of sulfuric acid specified in **JIS K 8951** in 500 ml of water gradually as agitating. Add 0.03 g of methylene blue (dihydrate) specified in **JIS K 8897** and 50 g of sodium sulfate specified in **JIS K 8987** into this solution, dissolve them by agitating and add water to make 1 000 ml.
- c) **Chloroform**, specified in **JIS K 8322**.

7.3.4.3 Operation Weigh out 2 ml of the sample solution by using a volumetric pipette, pour it in a 100 ml Erlenmeyer flask with a ground stopper, add 20 ml of chloroform, 20 ml of water and 2 ml of methylene blue solution (0.03 g/L) and titrate by sodium lauryl sulfate solution (0.005 mol/L) with using of a 5 ml micro burette as agitating. Add sodium lauryl sulfate solution (0.005 mol/L) little by little repeatedly, agitate sufficiently each time of adding, and confirm the change in colour of the solution. Take the time, when the blue colour of the upper layer (water layer) is the same grade as the blue colour of the lower layer (chloroform layer) or the lower layer is slightly more blue than the upper, as the end point. In addition, operate similarly without adding the sample solution, take this as blank test, calculate the mass of BKC in the sample solution according to formula (12).

$$B = \frac{(t_1 - t_2) \times f \times 354 \times 0.005 \times 250}{1\,000 \times 2} \dots\dots\dots (12)$$

where, B : mass of BKC in the sample weighed out in **7.3.2** (g)

t_1 : quantity of sodium lauryl sulfate solution (0.005 mol/L) required for titration (ml)

- t_2 : quantity of sodium lauryl sulfate solution (0.005 mol/L)
required for blank test (ml)
 f : factor of sodium lauryl sulfate solution (0.005 mol/L)
354 : average molecular weight of BKC

7.3.5 DDAC

7.3.5.1 Summary DDAC, being a cation, is titrated by using anion solution as agitating in water-chloroform system with BPB as an indicator.

7.3.5.2 Reagents The reagents shall be in accordance with 7.2.3.1 b).

7.3.5.3 Operation Weigh out 2 ml of the sample solution by using a volumetric pipette, determine DDAC according to 7.2.3.1, and calculate the mass of DDAC in the sample solution according to formula (13).

$$D = \frac{(t_1 - t_2) \times 362 \times 0.002 \times 250}{1\,000 \times 2} \dots\dots\dots (13)$$

- where, D : mass of DDAC in the sample weighed out 7.3.2 (g)
 t_1 : quantity of anion solution (0.002 mol/L) required for
titration (ml)
 t_2 : quantity of anion solution (0.002 mol/L) required for
blank test (ml)
362 : average molecular weight of DDAC

7.3.6 Calculation for compound ratio of effective composition

7.3.6.1 ACQ-1 The compound ratio of effective composition in ACQ-1 shall be calculated from the results of 7.3.3 and 7.3.4 according to formula (14) and formula (15), and rounded off to two significant figures.

$$C_0 = \frac{C}{C + B} \times 100 \dots\dots\dots (14)$$

$$B_0 = \frac{B}{C + B} \times 100 \dots\dots\dots (15)$$

- where, C_0 : compound ratio of copper compound in the sample (as
CuO) (mass percentage %)
 B_0 : compound ratio of BKC in the sample (as BKC) (mass
percentage %)
 C : see formula (10) or formula (11)
 B : see formula (12)

7.3.6.2 ACQ-2 The compound ratio of effective composition in ACQ-2 shall be calculated from the results of 7.3.3 and 7.3.5 according to formula (16) and formula (17), and rounded off to two significant figures.

$$C_0 = \frac{C}{C + D} \times 100 \quad \text{..... (16)}$$

$$D_0 = \frac{D}{C + D} \times 100 \quad \text{..... (17)}$$

where, C_0 : compound ratio of copper compound in the sample (as CuO) (mass percentage %)
 D_0 : compound ratio of DDAC in the sample (as DDAC) (mass percentage %)
 C : see formula (10) or formula (11)
 D : see formula (13)

7.3.7 Method of obtaining for effective composition content

7.3.7.1 ACQ-1 The effective composition content in ACQ-1 shall be calculated from the results of 7.3.3 and 7.3.4 according to formula (18) and rounded off to two significant figures.

$$T = \frac{C + B}{S} \quad \text{..... (18)}$$

where, T : effective composition content (mass percentage %)
 S : mass of the sample weighed out in 7.3.2 (g)
 C : see formula (10) or formula (11)
 B : see formula (12)

7.3.7.2 ACQ-2 The effective composition content in ACQ-2 shall be calculated from the results of 7.3.3 and 7.3.5 according to formula (19) and rounded off to two significant figures.

$$T = \frac{C + D}{S} \times 100 \quad \text{..... (19)}$$

where, T : effective composition content (mass percentage %)
 S : mass of the sample weighed out in 7.3.2 (g)
 C : see formula (10) or formula (11)
 D : see formula (13)

7.3.8 State of product The state of product shall be visually checked.

7.3.9 Water insoluble content The water insoluble content shall be as follows.

7.3.9.1 Operation Agitate the sample solution sufficiently, weigh out 100 ml of the sample solution by using a 100 ml measuring cylinder, and filter it with using a mass-known glass filter, 1 G4. Wash the residue sufficiently with water, dry it in a dryer at 105 °C to 110 °C until it reaches a constant weight, leave it cool in a desiccator as it is, and weigh the mass of the residue.

7.3.9.2 Calculation Calculate the water insoluble content according to formula (20) for ACQ-1 or formula (21) for ACQ-2.

$$Q = \frac{R \times 250}{(C + B) \times 100} \times 100 \quad \text{..... (20)}$$

$$Q = \frac{R \times 250}{(C + D) \times 100} \times 100 \quad \text{..... (21)}$$

where, Q : water insoluble content (mass percentage %)
 R : mass of residue (g)
 C : see formula (10) or formula (11)
 B : see formula (12)
 D : see formula (13)

7.3.10 pH The pH shall be measured on the sample solution prepared in 7.3.2 according to JIS Z 8802.

7.4 Copper/azole compound series of wood preservatives

7.4.1 Sampling method The sampling of representative sample shall be in accordance with JIS K 2420. Provided that the sample shall be taken from an optional place under well-mixed condition.

7.4.2 Preparation of sample solution Agitate the representative sample sufficiently, weigh out from it to the nearest 0.1 mg so as to be approximately 17 g as effective composition, dissolve it in a small amount of water, transfer it in a 500 ml volumetric flask, add water up to the marked line, and take it as the sample solution.

7.4.3 Copper compound The mass of copper compound shall be in accordance with 7.3.3. Provided that it shall be calculated by using formula (22) for calculation formula.

$$C = \frac{c}{1\,000} \times 1.252 \times \frac{1\,000}{2} \times \frac{5}{1\,000} \quad \text{..... (22)}$$

where, C : mass of copper compound in the same weighed out in 7.4.2 (as CuO) (g)
 c : concentration of copper in the analytical sample solution (as Cu) (mg/L)
1.252 : factor to convert Cu into CuO

7.4.4 Boron compound

7.4.4.1 Molecular absorption spectrophotometry

a) **Summary** The absorbance of the complex, which is produced by the reaction of boric acid with carminic acid in conc. sulfuric acid, is measured and boric acid is determined.

b) **Reagents** The reagents shall be as follows:

- 1) **Sulfuric acid**, specified in **JIS K 8951**.
- 2) **Hydrogen peroxide**, specified in **JIS K 8230**.
- 3) **Carminic acid solution**, prepared so that 0.125 g of carminic acid is weighed out, put in a 500 ml volumetric flask, dissolved in 300 ml of sulfuric acid, and further sulfuric acid is added up to the marked line.
- 4) **Iron (II) sulfate solution**, prepared so that 5 g of iron (II) sulfide specified in **JIS K 8948** is weighed out in a 100 ml volumetric flask, dissolved by adding 3 ml of sulfuric acid specified in **JIS K 8951** and water and further water is added up to the marked line.
- 5) **Boric acid standard stock solution**, prepared so that boric acid specified in **JIS K 8863** is dried in a desiccator containing sulfuric acid for 5 h, then 1.250 g of it is weighed out in a 500 ml volumetric flask and dissolved in 250 ml of water, further water is added up to the marked line.

c) **Operation** Weigh out 5 ml of the sample solution by using a volumetric pipette, pour it in a 500 ml rounded-bottom flask, with Kjeldahl trap bulb and interchangeable ground joint, of quartz glass or non-boric acid glass, and add 5 ml of hydrogen peroxide and 2 ml of sulfuric acid. After the contents have become transparent and been concentrated until white fume has generated, leave it cool as it is, transfer the decomposed solution in the rounded-bottom flask into a 500 ml volumetric flask, add water up to the marked line, and take this solution as the diluted sample solution.

Weigh out 2 ml of the diluted sample solution by using a volumetric pipette, pour it in a 25 ml volumetric flask, add 3 drops of hydrochloric acid and 3 drops of iron (II) sulfate solution to mix and further add 10 ml of sulfuric acid and mix them. After cooling down to room temperature, add 10 ml of carminic acid solution accurately and mix them gently. After adding sulfuric acid up to the marked line, leave it for 45 min as it is and take it as the analytical sample solution. Transfer a portion of analytical sample solution to an absorption cell, measure the absorbance at 600 nm in wavelength and obtain the concentration of boric acid in the analytical sample solution from the working curve prepared previously.

d) **Calculation** The mass of boron compound in the sample shall be calculated according to formula (23).

$$B = \frac{b}{1\,000} \times \frac{25}{2} \times \frac{100}{5} \times \frac{500}{1\,000} \dots\dots\dots (23)$$

where, B : mass of boron compound in the sample weighed out in **7.4.2** (as H_3BO_3) (g)

b : concentration of boric acid in the analytical sample solution (as H_3BO_3) (mg/L)

7.4.4.2 ICP atomic emission spectrometry

a) **Summary** The sample solution is sprayed in inductively coupled plasma and boron is determined by measuring the emission caused by boron at 249.773 nm in wavelength.

- b) **Reagents** Boron standard solution (1 mg/ml as B), being in accordance with 47.3 a) 2) of JIS K 0102.
- c) **Apparatus** For the apparatus, an ICP atomic emission spectrometer shall be used.
- d) **Operation** Weigh out 1 ml of the sample solution by using a volumetric pipette, pour it in a 1 000 ml volumetric flask, add water up to the marked line, and take this solution as the analytical sample solution. The concentration of boron in the analytical sample solution shall be obtained according to 47.3 of JIS K 0102.
- e) **Preparation of working curve** The preparation of working curve shall be in accordance with 47.3 c) 3) of JIS K 0102.
- f) **Calculation** The mass of boron compound in the sample shall be calculated according to formula (24).

$$B = \frac{b}{1\,000} \times 5.718 \times 1\,000 \times \frac{500}{1\,000} \dots\dots\dots (24)$$

where, *B* : mass of boron compound in the sample weighed out in 7.4.2 (as H₃BO₃) (g)
 b : concentration of boron in the analytical sample solution (as B) (mg/L)
 5.718 : factor to convert B into H₃BO₃

7.4.5 Tebuconazole

7.4.5.1 Summary The tebuconazole in the sample solution is determined according to high performance liquid chromatography.

7.4.5.2 Reagents The reagents shall be as follows:

- a) **Tebuconazole solution**, prepared so that approximately 0.125 g of tebuconazole (the purity is known and not less than 90 %) is weighed out to the nearest 0.1 mg, put in a 100 ml volumetric flask, dissolved in 50 ml of methanol specified in JIS K 8891 and methanol is added up to the marked line.
- b) **Solvent for working curve**, prepared so that the mixed solution of 80 ml of methanol specified in JIS K 8891 and 20 ml of water is furthermore mixed with 1.5 ml of acetic acid specified in JIS K 8355.
- c) **Methanol**, specified in JIS K 8891.
- d) **Acetic acid**, specified in JIS K 8355.
- e) **Mobile phase**, prepared so that 80 ml of methanol specified in JIS K 8891 and 20 ml of water are mixed.

7.4.5.3 Apparatus For the apparatus, a high performance liquid chromatograph shall be used.

7.4.5.4 Analytical condition of high performance liquid chromatography The analytical condition of high performance liquid chromatography shall be in accordance with table 14.

Table 14 Analytical condition of high performance liquid chromatography

Item	Condition
Column	ODS system of column (I.D. 4.6 mm, L. 150 mm)
Mobile phase	In accordance with 7.4.5.2 e).
Mobile phase flow rate	0.8 ml/min
Column temperature	40 °C
Measuring wavelength	220 nm
Injection amount	10 µl

7.4.5.5 Operation Weigh out 10 ml of the sample solution by using a volumetric pipette, pour it in a 50 ml volumetric flask, adjust pH to approximately 4.0 by adding 0.8 ml of acetic acid, add methanol up to the marked line, further filter it by using a filter of 0.45 µm in pore diameter, and take this solution as analytical sample solution. Inject 10 µl of the analytical sample solution into a high performance liquid chromatograph, obtain the peak area of tebuconazole, and obtain the concentration of tebuconazole from the working curve.

7.4.5.6 Preparation of working curve Prepare the tebuconazole solution by using the solvent for working curve step wise so as to be 10 µg/ml to 50 µg/ml, and filter it by using a filter of 0.45 µm in pore diameter. Inject 10 µl of the filtrate in a high performance liquid chromatograph, and prepare the working curve by operating so similarly as **7.4.5.4**.

7.4.5.7 Calculation The mass of tebuconazole in the sample shall be calculated according to formula (25).

$$T = \frac{t}{1\,000} \times \frac{50}{10} \times \frac{500}{1\,000} \dots\dots\dots (25)$$

where, T : mass of tebuconazole in the sample weighed out in **7.4.2** (g)

t : concentration of tebuconazole in the analytical sample solution (mg/L)

7.4.6 Cyproconazole

7.4.6.1 Gas chromatography

- a) **Summary** The cyproconazole in the analytical sample solution is determined according to gas chromatography.
- b) **Reagents** The reagents shall be as follows:
 - 1) **Ethanol**, specified in **JIS K 8101**.
 - 2) **Toluene**, specified in **JIS K 8680**.
 - 3) **Ethyl acetate**, specified in **JIS K 8361**.

- 4) **Acetone**, specified in **JIS K 8034**.
 - 5) **Cyclohexane**, specified in **JIS K 8464**.
 - 6) **Ammonium carbonate solution**, prepared so that 80 g of ammonium carbonate specified in **JIS K 8613** is put in a 1 000 ml volumetric flask, dissolved by adding water and further water is added up to the marked line.
 - 7) **Ethyl acetate/cyclohexane solution**, prepared so that ethyl acetate specified in **JIS K 8361** and cyclohexane specified in **JIS K 8464** are mixed at the rate of 2 to 3 (volume percentage).
 - 8) **Porous diatomaceous earth column**, being a column of approximately 2.5 cm in inside diameter filled with granular diatomaceous earth for column chromatograph of approximately 20 ml in retention, or that having the performance equivalent to this.
 - 9) **Silica gel minicolumn**, being a column of approximately 1.0 cm in inside diameter and approximately 2.5 cm in length filled with approximately 700 mg of silica gel for column chromatograph, or that having the performance equivalent to this.
 - 10) **Cyproconazole solutio**, prepared so that approximately 50 mg of the reference material of cyproconazole (the purity is known and not less than 95 %) is weighed out to the nearest 0.1 mg, put in a 50 ml volumetric flask, dissolved by adding ethanol specified in **JIS K 8101**, and further ethanol is added up to the marked line.
 - 11) **Solvent for working curve**, prepared so that 60 ml of acetonitrile specified in **JIS K 8032** and 40 ml of water are mixed.
- c) **Apparatus** For the apparatus, a gas chromatographic analyzer shall be used.
- d) **Analytical condition of gas chromatograph** The analytical condition of gas chromatograph shall be in accordance with table 15.

Table 15 Analytical condition of gas chromatograph

Item	Condition
Stationary phase liquid (film thickness)	[5 % phenyl] cross-linking type methyl siloxane (0.25 µm)
Tube for column (inside diameter × length)	Molten silica/capillary tube (0.32 mm × 30 cm)
Column temperature	60 °C, 1 min → (20 °C/min) → 240 °C, 10 min → (20 °C/min) → 260 °C
Injection temperature	250 °C
Injection amount	2 µl
Combustion gas	Hydrogen 30 ml/min, air 370 ml/min
Make up gas	He 30 ml/min
Carrier gas	He Split Vent 93 ml/min, Purge Vent 1 ml/min
Detector	Flame ionization detector (FID)

e) **Operation** The operation shall be as follows:

- 1) Weigh out 10 ml of the sample solution by using a volumetric pipette, pour it in a 50 ml volumetric flask, add ammonium carbonate solution up to the marked line, and take it as the diluted sample solution.
- 2) Weigh out 20 ml of the diluted sample solution by using a volumetric pipette, add into a porous diatomaceous earth column and hold it for 10 min.
- 3) Attach an injector to the porous diatomaceous earth column, elute by 120 ml of toluene, receive the eluate in a 200 ml rounded-bottom flask, and distil this solution by using a rotary evaporator (hot water bath temperature, 40 °C) under reduced pressure.
- 4) Dissolve the residue by using 10 ml of toluene as dividing in 3 times.
- 5) Wash the silicagel minicolumn attached with an injector of 10 ml in capacity by using 10 ml of ethyl acetate, and further wash by 10 ml of toluene. Pass the whole solution mentioned above in 4) into the silicagel minicolumn finished of washing, and discard the out flow solution.
- 6) Pass 5 ml of ethyl acetate/cyclohexane solution, and discard the out flow solution.
- 7) Then, elute by using 10 ml of ethyl acetate, receive the eluate in a 100 ml rounded-bottom flask, and distil this solution by using a rotary evaporator (hot water bath temperature, 40 °C) under reduced pressure.
- 8) Dissolve the residue by using acetone to be a constant volume (10 ml), and take this solution as the analytical sample solution.
- 9) Inject 2 µl of the analytical sample solution into the gas chromatographic analyzer, obtain the peak area of cyproconazole, and obtain the concentration of cyproconazole from the working curve.
- 10) **Preparation of working curve** Prepare the cyproconazole solution by using the solvent for working curve stepwise so as to be 10 µg/ml to 50 µg/ml, inject 2 µl of it in the gas chromatographic analyzer, operate as similarly as in 7.4.6.1 d), and prepare the working curve.

f) **Calculation** The mass of cyproconazole in the sample shall be calculated according to formula (26).

$$Y = \frac{y}{1\,000} \times \frac{10}{20} \times \frac{50}{10} \times \frac{500}{1\,000} \dots\dots\dots (26)$$

where, Y: mass of cyproconazole in the sample weighed out in 7.4.2 (g)

y: concentration of cyproconazole in the analytical sample solution (mg/L)

7.4.6.2 High performance liquid chromatography

a) **Summary** The cyproconazole in the sample solution is determined according to the high performance liquid chromatography.

- b) **Reagents** The reagents shall be as follows:
- 1) **Cyproconazole solution**, prepared so that approximately 50 mg of the reference material of cyproconazole (the purity is known and not less than 95 %) is weighed out to the nearest 0.1 mg, put in a 50 ml volumetric flask, dissolved by adding ethanol specified in **JIS K 8101**, and further ethanol is added up to the marked line.
 - 2) **Phosphoric acid/ethanol solution**, prepared so that 10 g of phosphoric acid specified in **JIS K 9005**, which is put in a 100 ml volumetric flask and added with water to the marked line, and ethanol specified in **JIS K 8101** are mixed at the rate of 1 to 9 in volume.
 - 3) **Mobile phase**, prepared by mixing 60 ml of acetonitrile specified in **JIS K 8032** and 40 ml of water.
- c) **Apparatus** For the apparatus, a high performance liquid chromatograph shall be used.
- d) **Analytical condition of high performance liquid chromatography** The analytical condition of high performance liquid chromatography shall be in accordance with table 16.

Table 16 Analytical condition of high performance liquid chromatography

Item	Condition
Column	ODS system column (I.D. 4.6 mm, L. 150 mm)
Mobile phase	In accordance with 7.4.6.2 b) 3) .
Mobile phase flow rate	1.0 ml/min
Column temperature	40 °C
Measuring wavelength	221 nm
Injection amount	10 µl

- e) **Operation** Weigh out 10 ml of the sample solution by using a volumetric pipette, pour it in a 50 ml volumetric flask, add phosphoric acid/ethanol solution up to the marked line, further filter by using a filter of 0.45 µm in pore diameter, and take this solution as the analytical sample solution. Inject 10 µl of the analytical sample solution into the high performance liquid chromatograph, obtain the peak area of cyproconazole, and obtain the concentration of cyproconazole from the working curve.
- f) **Preparation of working curve** Prepare the cyproconazole solution by using ethanol specified in **JIS K 8101** stepwise so as to be 10 µg/ml to 50 µg/ml, and filter it by using a filter of 0.45 µm in pore diameter. Inject 10 µl of the filtrate in the high performance liquid chromatograph and prepare the working curve by operating as similarly as in **7.4.6.2 d)**.
- g) **Calculation** The mass of cyproconazole in the sample shall be calculated according to formula (27).

$$Y = \frac{y}{1\,000} \times \frac{50}{10} \times \frac{500}{1\,000} \dots\dots\dots (27)$$

where, Y : mass of cyproconazole in the sample weighed out in
 7.4.2 (g)
 y : concentration of cyproconazole in the analytical sample
 solution (mg/L)

7.4.7 Calculation of compound ratio of effective composition

7.4.7.1 CUAZ-1 The compound ratio of effective composition in CUAZ-1 shall be calculated from the results of **7.4.3**, **7.4.4** and **7.4.5** according to formula (28), formula (29) and formula (30) and rounded off to two significant figures.

$$C_0 = \frac{C}{C+B+T} \times 100 \dots\dots\dots (28)$$

$$B_0 = \frac{B}{C+B+T} \times 100 \dots\dots\dots (29)$$

$$T_0 = \frac{T}{C+B+T} \times 100 \dots\dots\dots (30)$$

where, C_0 : compound ratio of copper compound in the sample (as
 CuO)
 B_0 : compound ratio of boron compound in the sample (as
 H₃BO₃)
 T_0 : compound ratio of tebuconazole in the sample
 C : copper compound content in the sample solution (as
 CuO) (g)
 B : boron compound content in the sample solution (as
 H₃BO₃) (g)
 T : tebuconazole content in the sample solution (g)

7.4.7.2 CUAZ-2 The compound ratio of effective composition in CUAZ-2 shall be calculated from the results of **7.4.3** and **7.4.6** according to formula (31) and formula (32), and rounded off to two significant figures.

$$C_0 = \frac{C}{C+Y} \times 100 \dots\dots\dots (31)$$

$$Y_0 = \frac{Y}{C+Y} \times 100 \dots\dots\dots (32)$$

where, C_0 : compound ratio of copper compound in the sample (as
 CuO)
 Y_0 : compound ratio of cyproconazole in the sample
 C : copper compound content in the sample solution (as
 CuO) (g)
 Y : cyproconazole content in the sample solution (g)

7.4.7.3 CUAZ-3 The compound ratio of effective composition in CUAZ-3 shall be calculated from the results of 7.4.3 and 7.4.6 according to formula (33) and formula (34), and rounded off to two significant figures.

$$C_0 = \frac{C}{C+Y} \times 100 \quad \text{..... (33)}$$

$$Y_0 = \frac{Y}{C+Y} \times 100 \quad \text{..... (34)}$$

where, C_0 : compound ratio of copper compound in the sample (as CuO)

Y_0 : compound ratio of cyproconazole in the sample

C : copper compound content in the sample solution (as CuO) (g)

Y : cyproconazole content in the sample solution (g)

7.4.8 Obtaining method for effective composition content

7.4.8.1 CUAZ-1 The effective composition content in CUAZ-1 shall be calculated from the results of 7.4.3, 7.4.4 and 7.4.5 according to formula (35), and rounded off to two significant figures.

$$A = \frac{C+B+T}{S} \times 100 \quad \text{..... (35)}$$

where, A : effective composition content (mass percentage %)

S : mass of the sample weighed out in 7.4.2 (g)

C : C in formula (22)

B : B in formulae (23) and (24)

T : T in formula (25)

7.4.8.2 CUAZ-2 The effective composition content in CUAZ-2 shall be calculated from the results of 7.4.3 and 7.4.6 according to formula (36), and rounded off to two significant figures.

$$A = \frac{C+Y}{S} \times 100 \quad \text{..... (36)}$$

where, A : effective composition content (mass percentage %)

S : mass of the sample weighed out in 7.4.2 (g)

C : C in formula (22)

Y : Y in formulae (26) and (27)

7.4.8.3 CUAZ-3 The effective composition content in (CUAZ-3) shall be calculated from the results of 7.4.3 and 7.4.6 according to formula (37), and rounded off to two significant figures.

$$A = \frac{C+Y}{S} \times 100 \quad \text{..... (37)}$$

where, A : effective composition content (mass percentage %)
 S : mass of the sample weighed out in 7.4.2 (g)
 C : C in formula (22)
 Y : Y in formulae (26) and (27)

7.4.9 State of product The state of product shall be visually checked.

7.4.10 Water insoluble content

7.4.10.1 Operation Agitate the sample solution sufficiently, weigh out 100 ml of the sample solution by using a 100 ml measuring cylinder, and filter it by using the mass-known glass filter, 1 G4. Wash well the residue by water, dry it in a dryer at 105 °C to 110 °C until it reaches a constant weight, leave it cool in a desiccator as it is, and weigh the mass of the residue.

$$Q = \frac{R}{S \times A/100} \times \frac{250}{100} \times 100 \dots\dots\dots (38)$$

where, Q : water insoluble content (mass percentage %)
 R : mass of the residue (g)
 A : effective composition content of the sample (mass percentage %)
 S : mass of the sample weighed out in 7.4.2 (g)

7.4.11 pH The pH shall be measured on the sample solution prepared according to 7.4.2 according to JIS Z 8802.

7.5 Boron/quaternary ammonium compound series of wood preservatives

7.5.1 Sampling method The sampling of the representative sample shall be in accordance with JIS K 2420. Provided that the sample shall be taken from an optional place under well-mixed condition.

7.5.2 Preparation of sample solution Weigh out the sample to the nearest 0.1 mg so as to be approximately 13.3 g as effective composition, put it in a 1 000 ml volumetric flask, add water up to the marked line, and take this solution as the sample solution.

7.5.3 Boron compound

7.5.3.1 ICP atomic emission spectrometry

- a) **Summary** The sample solution is sprayed in inductively coupled plasma, and boron is determined by measuring the emission caused by boron at 249.773 nm in wavelength.
- b) **Reagents** The reagents shall be as follows.

Boron reference solution (1 mg/ml as B), being in accordance with 47.3 a) 2) of JIS K 0102.

- c) **Apparatus** For the apparatus, an ICP atomic emission spectrometer shall be used.
- d) **Operation** Weigh out 4 ml of the sample solution by using a volumetric pipette, pour it in a 1 000 ml volumetric flask, add water up to the marked line, and take this solution as the analytical sample solution. The boron content of the analytical sample solution shall be obtained according to ICP atomic emission spectrometry specified in **47.3** of **JIS K 0102**.
- e) **Preparation of working curve** The preparation of working curve shall be in accordance with **47.3 c) 3)** of **JIS K 0102**.
- f) **Calculation** The mass of boron in the sample solution shall be calculated according to formula (39).

$$B = \frac{b \times 5.718 / 1\,000}{1\,000 \times 4} \dots\dots\dots (39)$$

where, B : mass of boron compound in the sample weighed out in **7.5.2** (as H_3BO_3) (g)
 b : concentration of boron in the analytical sample solution (mg/L)
 5.718 : factor to convert B into H_3BO_3

7.5.4 DDAC The DDAC content shall be in accordance with **7.2.3.1**. Provided that the calculation shall be carried out by using formula (40).

$$D = \frac{(t_1 - t_2) \times 362 \times 0.002 \times 1\,000}{1\,000 \times 2} \dots\dots\dots (40)$$

where, D : mass of DDAC in the sample weighed out in **7.5.2** (g)
 t_1 : quantity of anion solution (0.002 mol/L) required for titration (ml)
 t_2 : quantity of anion solution (0.002 mol/L) required for blank test (ml)
 362 : molecular weight of DDAC

7.5.5 Calculation for compound ratio of effective composition The compound ratio of effective composition shall be calculated from the results of **7.5.3** and **7.5.4** according to formula (41) and formula (42), and rounded off to two significant figures.

$$B_0 = \frac{B}{B + D} \times 100 \dots\dots\dots (41)$$

$$D_0 = \frac{D}{B + D} \times 100 \dots\dots\dots (42)$$

where, B_0 : compound ratio of boron compound in the sample
 D_0 : compound ratio of DDAC in the sample
 B : mass of boron compound in the sample (as H_3BO_3) (g)
 D : mass of DDAC in the sample (g)

7.5.6 Obtaining method for effective composition content The effective composition content shall be calculated from the results of 7.5.3 and 7.5.4 according to formula (43). The calculated value shall be rounded off to two significant figures.

$$A = \frac{B + D}{S} \times 100 \quad \text{..... (43)}$$

where, A : effective composition content in the sample (mass percentage %)

S : mass of the sample weighed out in 7.5.2 (g)

B : mass of boron compound in the sample (as H_3BO_3) (g)

D : mass of DDAC in the sample (g)

7.5.7 State of product The state of product shall be visually checked.

7.5.8 Water insoluble content The water insoluble content shall be as follows.

7.5.8.1 Operation Agitate the sample solution sufficiently, weigh out 100 ml of the sample solution by using a 100 ml measuring cylinder, and filter it by using a mass-known glass filter, 1 G4. Wash the residue by water sufficiently, dry it in a dryer at 105 °C to 110 °C until it reaches a constant weight, leave it cool in a desiccator as it is, and weigh the mass of the residue.

7.5.8.2 Calculation The water insoluble content shall be calculated according to formula (44).

$$Q = \frac{R}{S \times A / 1\,000} \times 100 \quad \text{..... (44)}$$

where, Q : water insoluble content (mass percentage %)

R : mass of the residue (g)

A : effective composition content (mass percentage %)

S : mass of the sample (g)

7.5.9 pH The pH shall be measured on the sample solution prepared in 7.6.2 according to JIS Z 8802.

7.6 Quaternary ammonium/non-ester pyrethroid compound series of wood preservatives

7.6.1 Sampling method The sampling of the representative sample shall be in accordance with JIS K 2420. Provided that the sample shall be taken from an optional place under well-mixed condition.

7.6.2 Preparation of sample solution Weigh out the sample to the nearest 0.1 mg so as to be approximately 10 g as effective composition, put it in a 1 000 ml volumetric flask, add water up to the marked line, and take this solution as the sample solution.

7.6.3 DMPAP The DMPAP content shall be in accordance with **7.2.4.1** or **7.2.4.2**. The calculation shall be carried out by using formula (45) or formula (46).

7.6.3.1 Titration method

$$D = \frac{(t_1 - t_2) \times 454 \times 0.002 \times 1\,000}{1\,000 \times 2} \dots\dots\dots (45)$$

where, D : mass of DMPAP in the sample weighed out in **7.6.2** (g)
 t_1 : quantity of anion solution (0.002 mol/L) required for titration (ml)
 t_2 : quantity of anion solution (0.002 mol/L) required for blank test (ml)
454 : average molecular weight of DMPAP

7.6.3.2 High performance liquid chromatography

$$M = M_0 \times \frac{100 \times 1}{1\,000} \dots\dots\dots (46)$$

where, M : mass of DMPAP in the sample weighed out in **7.6.2** (g)
 M_0 : concentration of DMPAP in the sample solution obtained from working curve (mg/L)

7.6.4 Silafluofen

7.6.4.1 Summary The silafluofen is determined according to high performance liquid chromatography.

7.6.4.2 Reagents The reagents shall be as follows:

- a) **Acetonitrile**, specified in **JIS K 8032** (for high performance liquid chromatography).
- b) **Silafluofen reference solution (100 mg/L)**, prepared so that 10 mg of silafluofen reference material for analysis (at least 97 % in purity) is put in a 100 ml volumetric flask and acetonitrile is added up to the marked line.
- c) **Methanol**, specified in **JIS K 8891** (for high performance liquid chromatography).
- d) **Calibration standard solution**, prepared so that 5 ml to 15 ml of silafluofen reference solution (100 mg/L) is weighed out in 20 ml volumetric flask stepwise and accurately, and acetonitrile is added up to the marked line. This solution is analyzed according to the high performance liquid chromatography and the working curve is prepared from the relation of silafluofen concentration to peak area.
- e) **Mobile phase**, prepared so that 65 ml of acetonitrile specified in **JIS K 8032**, 15 ml of methanol specified in **JIS K 8891** and 20 ml of water are mixed.

7.6.4.3 Apparatus For the apparatus, a high performance liquid chromatograph shall be used.

7.6.4.4 Analytical condition of high performance liquid chromatography The analytical condition of high performance liquid chromatography shall be in accordance with table 17.

Table 17 Analytical condition of high performance liquid chromatography

Item	Condition
Column	ODS system column I.D. 4.6 mm × L. 150 mm
Mobile phase	In accordances with 7.6.4.2 e).
Mobile phase flow rate	2.0 ml/min
Column temperature	40 °C
Measuring wavelength	230 nm
Injection amount	10 µl

7.6.4.5 Operation Inject 10 µl of the sample solution in the high performance liquid chromatograph and obtain the peak area of silafluofen at 230 nm in wavelength. Obtain the concentration of silafluofen in the sample solution from the working curve, and calculate the mass of silafluofen in the sample according to formula (47).

$$S = \frac{R}{1\,000} \dots\dots\dots (47)$$

where, **S** : mass of silafluofen in the sample weighed out **7.6.2** (g)
 R : concentration of silafluofen in the sample solution (mg/L)

7.6.5 Calculation for compound ratio of effective composition The compound ratio of effective composition shall be calculated from the results of **7.6.3** and **7.6.4** according to formula (48) and formula (49). The calculated value shall be rounded off to two significant figures.

$$S_0 = \frac{S}{S + D} \times 100 \dots\dots\dots (48)$$

$$D_0 = \frac{D}{S + D} \times 100 \dots\dots\dots (49)$$

where, **S**₀ : compound ratio of silafluofen in the sample
 D₀ : compound ratio of DMPAP in the sample
 S : mass of silafluofen in the sample (g)
 D : mass of DMPAP in the sample (g)

7.6.6 Obtaining method for effective composition content The effective composition content shall be calculated from the results of **7.6.3** and **7.6.4** according to formula (50). The calculated values shall be rounded off to two significant figures.

$$A = \frac{S+D}{T} \times 100 \dots\dots\dots (50)$$

where, A : effective composition content in the sample (mass percentage %)
 T : mass of the sample weighed out in **7.6.2** (g)
 S : mass of silafluofen in the sample (g)
 D : mass of DMPAP in the sample (g)

7.6.7 State of product The state of product shall be visually checked.

7.6.8 Water insoluble content The water insoluble content shall be as follows.

7.6.8.1 Operation Agitate the sample solution sufficiently, weigh out 100 ml of the sample solution by using a 100 ml measuring cylinder, and filter it by using a mass known glass filter, 1 G4. Wash the residue by water sufficiently, dry it in a dryer at 105 °C to 110 °C until it reaches a constant weight, leave it cool in a desiccator as it is, and weigh the mass of the residue.

7.6.8.2 Calculation The water insoluble content shall be calculated according to formula (51).

$$Q = \frac{R \times 10}{S \times A/100} \times 100 \dots\dots\dots (51)$$

where, Q : water insoluble content (mass percentage %)
 R : mass of the residue (g)
 A : effective composition content (mass percentage %)
 S : mass of the sample (g)

7.6.9 pH The pH shall be measured on the sample solution, prepared according to **7.6.2**, according to **JIS Z 8802**.

7.7 Azole/quaternary ammonium/non-ester pyrethroid compound series of wood preservatives

7.7.1 Sampling method The sampling of the representative sample shall be in accordance with **JIS K 2420**. Provided that the sample shall be taken from an optional place under well-mixed condition.

7.7.2 Preparation of sample solution Weigh out the sample to the nearest 0.1 mg so as to be approximately 10 g as effective composition, put in a 1 000 ml volumetric flask, add water up to the marked line, and take this solution as the sample solution.

7.7.3 Cyproconazole The cyproconazole content shall be in accordance with **7.4.6.2**, and calculated by using formula (52).

$$C_{y1} = \frac{C_y \times 25 \times 1\,000}{1\,000 \times 1\,000 \times 10} \dots\dots\dots (52)$$

where, C_{y1} : mass of cyproconazole in the sample weighed out in
7.7.2 (g)

C_y : concentration of cyproconazole in the analytical sample
solution (mg/L)

7.7.4 Ethofenprox

7.7.4.1 Summary The ethofenprox in the sample solution is determined according to high performance liquid chromatography.

7.7.4.2 Reagents The reagents shall be as follows:

- a) **Ethofenprox solution**, prepared so that approximately 0.1 g of ethofenprox (the purity is known and at least 90 %) is weighed out to the nearest 0.1 mg, put in a 100 ml volumetric flask, dissolved in 50 ml of methanol specified in **JIS K 8891**, and methanol is added up to the marked line.
- b) **Mobile phase** The acetonitrile for high performance liquid chromatography shall be used.

7.7.4.3 Apparatus For the apparatus, a high performance liquid chromatograph shall be used.

7.7.4.4 Analytical condition of high performance liquid chromatography The analytical condition of high performance liquid chromatography shall be in accordance with table 18.

Table 18 Analytical condition of high performance liquid chromatography

Item	Condition
Column	ODS system column I.D. 4.6 mm × L. 150 mm
Mobile phase	In accordance with 7.7.4.2 b).
Mobile phase flow rate	1.0 ml/min
Column temperature	30 °C
Measuring wavelength	254 nm
Injection amount	10 µl

7.7.4.5 Operation Take 5 ml of the sample solution by using a volumetric pipette, pour it in a 25 ml volumetric flask, add methanol specified in **JIS K 8891** up to the marked line, and take this solution as the analytical sample solution. Filter this solution by using a filter of 0.45 µm in pore diameter, inject 10 µl of the filtrate in the high performance liquid chromatograph, obtain the peak area of ethofenprox, and obtain the quantity of ethofenprox from the working curve.

7.7.4.6 Calculation The mass of ethofenprox in the sample solution shall be obtained according to formula (53).

$$E_1 = \frac{E \times 25 \times 1\,000}{1\,000 \times 1\,000 \times 5} \dots\dots\dots (53)$$

where, E_1 : mass of ethofenprox in the sample weighed out in **7.7.2** (g)

E : concentration of ethofenprox in the analytical sample solution (mg/L)

7.7.4.7 Preparation of working curve Prepare the ethofenprox solution stepwise by using methanol so as to be 10 µg/ml to 100 µg/ml, and filter this solution by a filter of 0.45 µm in pore diameter. Inject 10 µl of the filtrate into the high performance liquid chromatograph, and prepare the working curve by operating as similarly as in **7.7.4.5**.

7.7.5 DMPAP content

7.7.5.1 Titration method The titration method shall be in accordance with **7.2.4.1**). The mass of DMPAP shall be calculated by using formula (54).

$$A_1 = t \times f \times 0.908 \times \frac{1\,000}{1\,000} \dots\dots\dots (54)$$

where, A_1 : mass of DMPAP in the sample weighed out in **7.7.2** (g)

t : quantity of 0.002 mol/L anion solution required for titration (ml)

f : factor of 0.002 mol/L anion solution

0.908 : mass of DMPAP equivalent to 1 ml of 0.002 mol/L anion solution (mg)

7.7.5.2 High performance liquid chromatography The high performance liquid chromatography shall be in accordance with **7.2.4.2**. The mass of DMPAP shall be calculated by using formula (55).

$$A_1 = \frac{A \times 100 \times 1\,000}{1\,000 \times 1\,000} \dots\dots\dots (55)$$

where, A_1 : mass of DMPAP in the sample weighed out in **7.7.2** (g)

A : concentration of DMPAP in the analytical sample solution from the working curve (mg/L)

7.7.6 Calculation for compound ratio of effective composition The compound ratio of effective composition shall be calculated from the results of **7.7.3**, **7.7.4** and **7.7.5** according to formula (56), formula (57) and formula (58), and rounded off to two significant figures.

$$A_0 = \frac{A_1}{A_1 + C_{y1} + E_1} \times 100 \dots\dots\dots (56)$$

$$Cy_0 = \frac{Cy_1}{A_1 + Cy_1 + E_1} \times 100 \quad \dots\dots\dots (57)$$

$$E_0 = \frac{E_1}{A_1 + Cy_1 + E_1} \times 100 \quad \dots\dots\dots (58)$$

where, A_0 : compound ratio of DMPAP in the sample
 Cy_0 : compound ratio of cyproconazole in the sample
 E_0 : compound ratio of ethofenprox in the sample
 A_1 : DMPAP content in the sample solution (g)
 Cy_1 : cyproconazole content in the sample solution (g)
 E_1 : ethofenprox content in the sample solution (g)

7.7.7 Obtaining method for effective composition content The effective composition content shall be calculated from the results of **7.7.3**, **7.7.4** and **7.7.5** according to formula (59) and rounded off to two significant figures.

$$T = \frac{A_1 + Cy_1 + E_1}{S} \times 100 \quad \dots\dots\dots (59)$$

where, T : effective composition content (mass percentage %)
 S : mass of the sample taken in **7.7.2** (g)

7.7.8 State of product The state of product shall be visually checked.

7.7.9 Water insoluble content The water insoluble content shall be as follows.

7.7.9.1 Operation Agitate the sample solution sufficiently, weigh out 100 ml of the sample solution by using a 100 ml measuring cylinder, and filter it by a mass-known glass filter, 1 G4. Wash the residue by water sufficiently, dry it in a dryer at 105 °C to 110 °C until it reaches a constant weight, leave it cool in a desiccator as it is, and weigh the mass of the residue.

7.7.9.2 Calculation The water insoluble content shall be calculated according to formula (60).

$$Q = \frac{R}{S \times T / 1\,000} \times 100 \quad \dots\dots\dots (60)$$

where, Q : water insoluble content (mass percentage %)
 R : mass of the residue (g)
 T : effective composition content (mass percentage %)
 S : mass of the sample weighed out in **7.7.2** (g)

7.7.10 pH The pH shall be measured on the sample solution prepared in **7.7.2** according to **JIS Z 8802**.

7.8 Metallic salts of fatty acid series of wood preservatives

7.8.1 Sampling method The sampling of the representative sample shall be in accordance with **JIS K 2420**. Provided that the sample shall be taken from an optional place under well-mixed condition.

7.8.2 Preparation of sample solution Weigh out approximately 1 g of the sample to the nearest 0.1 mg, put it in a 100 ml volumetric flask, add water, agitate it sufficiently, then add water up to the marked line, and take this solution as the sample solution.

7.8.3 Copper naphthenate

7.8.3.1 Summary The organic matter in the sample is heated to decompose by using acid, and then copper is determined according to atomic absorption spectrochemical analysis.

7.8.3.2 Reagents The reagents shall be as follows:

- a) **Sulfuric acid**, specified in **JIS K 8951**.
- b) **Copper standard solution for working curve (0.1 mg/ml as Cu)**, prepared so that 10 ml of copper standard solution (1 000 mg/L as Cu) specified in **JIS K 0010** is weighed out by using a volumetric pipette, poured in a 100 ml volumetric flask and water is added to the marked line.
- c) **Hydrogen peroxide**, specified in **JIS K 8230**.

7.8.3.3 Apparatus For the apparatus, an atomic absorption spectrochemical analyzer shall be used.

7.8.3.4 Operation Weigh out 10 ml of the sample solution by using a volumetric pipette, pour it in a 50 ml volumetric flask, add water to the marked line and agitate until it becomes uniform. Weigh out 1 ml from it by using a volumetric pipette, pour it in a 500 ml rounded-bottom flask with trap bulb, add 1 ml of hydrogen peroxide and 4 ml of sulfuric acid, and heat to dissolve it gradually on a sand bath. After leaving it cool as it is, transfer it in a 100 ml volumetric flask, wash the inside of the rounded-bottom flask with trap bulb by water sufficiently, and add the washings to the volumetric flask. Add water up to the marked line, and take this solution as the analytical sample solution. Spray the analytical sample solution into flame according to clause 6 of **JIS K 0121**, and read the indicated value at 324.8 nm in wavelength. Obtain the concentration of copper in the analytical sample solution from the working curve and calculate the copper content according to formula (61) and formula (62).

$$C_0 = c \times 50 \quad \text{..... (61)}$$

$$C = \frac{C_0}{S} \times 100 \quad \text{..... (62)}$$

where, C_0 : mass of copper naphthenate (as Cu) in the sample weighed out in **7.8.2** (g)

c : concentration of copper (as Cu) in the analytical sample solution (mg/ml)

C : copper naphthenate content (as Cu) (mass percentage %)

S : mass of the sample (g)

7.8.3.5 Preparation of working curve Weigh out 0 ml to 4 ml of the copper standard solution for working curve (0.1 mg/ml as Cu) by using a volumetric pipette stepwise, pour it in a 100 ml volumetric flask, add sulfuric acid (1+4) so as to become the same condition as the analytical sample solution, and then add water up to the marked line. Prepare the working curve of copper amount to indicated value by operating similarly on this solution. The preparation of working curve shall be carried out each time when used.

7.8.4 Zinc naphthenate

7.8.4.1 Summary The organic matter in the sample is heated to decompose by acid, and then zinc is determined according to atomic absorption spectrochemical analysis.

7.8.4.2 Reagents The reagents shall be as follows:

- Sulfuric acid**, specified in **JIS K 8951**.
- Zinc standard solution for working curve (0.05 mg/ml as Zn)**, prepared so that 5 ml of zinc standard solution (1 000 mg/L as Zn) specified in **JIS K 0011** is weighed out by using a volumetric pipette, poured in a 100 ml volumetric flask, and water is added up to the marked line.
- Hydrogen peroxide**, specified in **JIS K 8230**.

7.8.4.3 Apparatus For the apparatus, an atomic absorption spectrochemical analyzer shall be used.

7.8.4.4 Operation Weigh out 10 ml of the sample solution by using a volumetric pipette, pour it in a 50 ml volumetric flask, add water to the marked line, and agitate it until it becomes uniform. Take 1 ml out of it by using a volumetric pipette, pour it in a 500 ml rounded-bottom flask with trap bulb, add approximately 1 ml of hydrogen peroxide and 4 ml of sulfuric acid, and heat to dissolve it gradually on sand bath. After leave it cool as it is, transfer it to a 100 ml volumetric flask, wash the inside of rounded-bottom flask with trap bulb and put it in the volumetric flask together. Add water up to the marked line, and take this solution as the analytical sample solution. Spray the analytical sample solution into flame according to clause 6 of **JIS K 0121**, and read the indicated value at 213.8 nm in wavelength. Obtain the concentration of zinc in the analytical sample solution from the working curve and calculate the zinc content according to formula (63) and formula (64).

$$Z_0 = z \times 50 \dots\dots\dots (63)$$

$$Z = \frac{Z_0}{S} \times 100 \dots\dots\dots (64)$$

where, Z_0 : mass of zinc naphthenate (as Zn) in the sample weighed out in 7.8.2 (g)

z : concentration of zinc (as Zn) in the analytical sample solution (mg/ml)

Z : zinc naphthenate content (as Zn) (mass percentage %)

S : mass of the sample (g)

7.8.4.5 Preparation of working curve Weigh out 0 ml to 4 ml of the zinc standard solution for working curve (0.05 mg/ml as Zn) stepwise by using a volumetric pipette, pour it in a 100 ml volumetric flask, add sulfuric acid so as to become the same condition as the analytical sample solution, and then add water up to the marked line. Prepare the working curve of zinc amount to indicated value by operating this solution similarly. The preparation of working curve shall be carried out each time when used.

7.8.5 Zinc tertiary carboxylate content

7.8.5.1 Summary The organic matter in the sample is heated to decomposed by acid, and then zinc is determined according to atomic absorption spectrochemical analysis.

7.8.5.2 Reagents The reagents shall be as follows:

- a) **Sulfuric acid**, specified in **JIS K 8951**.
- b) **Zinc standard solution for working curve (0.05 mg/ml as Zn)**, prepared so that 5 ml of the zinc standard solution (1 000 mg/L as Zn) specified in **JIS K 0011** is weighed out by using a volumetric pipette, poured in a 100 ml volumetric flask and water is added up to the marked line.
- c) **Hydrogen peroxide**, specified in **JIS K 8230**.

7.8.5.3 Apparatus For the apparatus, an atomic absorption spectrochemical analyzer shall be used.

7.8.5.4 Operation Weigh out 10 ml of the sample solution by using a volumetric pipette, pour it in a 50 ml volumetric flask, add water up to the marked line, and agitate it until it becomes uniform. Weigh out 1 ml out of it by using a volumetric pipette, pour it in a 500 ml rounded-bottom flask with trap bulb, add approximately 1 ml of hydrogen peroxide and 4 ml of sulfuric acid, and heat to decompose it gradually on sand bath. After leaving it cool as it is, transfer it to a 100 ml volumetric flask, wash the inside of rounded-bottom flask with trap bulb by water sufficiently, and add the washings to the volumetric flask. Add water to the marked line, and take this solution as the analytical sample solution. Spray the analytical sample solution into flame according to clause 6 of **JIS K 0121**, and read the indicated value at 213.8 nm in wavelength. Obtain the concentration of zinc in the analytical sample solution from the working curve, and calculate the zinc content according to formula (65) and formula (66).

$$V_0 = z \times 50 \dots\dots\dots (65)$$

$$V = \frac{V_0}{S} \times 100 \dots\dots\dots (66)$$

where, V_0 : mass of zinc tertiary carboxylate (as Zn) in the sample weighed out in **7.8.2** (g)
 z : concentration of zinc (as Zn) in the analytical sample solution (mg/ml)
 V : zinc tertiary carboxylate content (as Zn) (mass percentage %)
 S : mass of the sample (g)

7.8.5.5 Preparation of working curve Weigh out 0 ml to 4 ml of the zinc standard solution for working curve (0.05 mg/ml as Zn) by using a volumetric pipette stepwise, pour it in a 100 ml of volumetric flask, add sulfuric acid so as to become the same condition as the analytical sample solution, and then add water up to the marked line. Prepare the working curve of zinc amount to indicated value by operating this solution similarly. The preparation of working curve shall be carried out each time when used.

7.8.6 Permethrin

7.8.6.1 Summary The sample is diluted by acetone and determined according to gas chromatograph internal standard method.

7.8.6.2 Reagents The reagents shall be as follows:

- a) **Permethrin reference material**, refined for determination and the purity is previously known as at least 90 %.
- b) **Acetone**, specified in **JIS K 1503**.
- c) **Di-n-octyl phthalate**, being on the market as reagent.
- d) **Permethrin solution**, prepared as follows: Weigh out approximately 0.25 g of permethrin to the nearest 0.1 mg, put it in a 100 ml volumetric flask, add acetone to the marked line, take this solution as the permethrin standard solution. Weigh out approximately 0.26 g of di-n-octyl phthalate to the nearest 0.1 mg, put it in a 100 ml volumetric flask, add acetone up to the marked line, and take this solution as the internal standard solution. Weigh out 2 ml of permethrin standard solution and 2 ml of internal standard solution by using a volumetric pipette, pour them in a 50 ml volumetric flask and add acetone up to the marked line.

7.8.6.3 Apparatus For the apparatus, a gas chromatographic analyzer shall be used.

7.8.6.4 Conditions of gas chromatograph The condition of gas chromatograph shall be in accordance with table 19.

Table 19 Condition of gas chromatograph

Item	Condition
Stationary liquid (load)	DEGS (Diethyleneglycol succinate) (2 %)
Stationary carrier	Informative [chromosorb W (HP) (149 mesh to 177 mesh)]
Column pipe (I.D. and Length, mm × m)	(glass made, 3 × 1)
Column temperature (°C)	215
Injection temperature (°C)	250
Hydrogen gas pressure (kPa)	88.3
Air pressure (kPa)	49.0
Nitrogen gas flow rate (ml/min)	50
Detector	Flame ionization detector (FID)

7.8.6.5 Operation Weigh out approximately 1 g of the sample to the nearest 0.1 mg, put it in a 50 ml volumetric flask, add 2 ml of internal standard solution, add acetone up to the marked line, take this solution as the analytical sample solution, and inject 2 µl of this solution into the column. Allow the obtained chromatogram to flow out in order of internal standard material and permethrin, and separate permethrin into two peaks. Sum up these two peak areas and obtain the peak area of permethrin in the analytical sample solution. Obtain the ratio to the peak area of permethrin in the permethrin solution obtained by the similar operation, obtain the amount of permethrin in the analytical sample solution from the permethrin content in the permethrin solution, and calculate the permethrin content according to formula (67) and formula (68).

$$P_0 = \frac{St}{50} \times \frac{p}{pn} \dots\dots\dots (67)$$

$$P = \frac{P_0}{S} \times 100 \dots\dots\dots (68)$$

where, P_0 : mass of permethrin in the sample weighed out in **7.8.2**
(g)

P : permethrin content (mass percentage %)

St : mass of standard permethrin (g)

p : chromatogram peak area ratio of permethrin in the analytical sample solution to internal standard material

pn : chromatogram peak area ratio of permethrin in the standard sample solution to internal standard material

S : mass of the sample (g)

7.8.7 Acid value of fatty acid

7.8.7.1 Summary Fatty acid is separated from the sample and the acid value is obtained by titration using potassium hydroxide solution.

7.8.7.2 Reagents The reagents shall be as follows:

- a) **2-Propanol**, specified in **JIS K 8839**.
- b) **Petroleum ether**, specified in **JIS K 8593**.
- c) **Sulfuric acid (1+9)**, prepared by using sulfuric acid specified in **JIS K 8951**.
- d) **Potassium hydroxide—ethanol solution (0.5 mol/L)**, prepared so that 30 g of potassium hydroxide specified in **JIS K 8574** is diluted by the smallest amount of water and diluted by ethyl alcohol (95) specified in **JIS K 8102** to make 1 L, and filtered after 2 days or 3 days elapse.
- e) **Solvent**, prepared as follows: The mixed solution of ethyl ether—ethyl alcohol at the volume ratio of 1 to 1 or 2 to 1, or the mixed solution of benzene—ethyl alcohol at the volume ratio of 1 to 1 or 2 to 1 is used. These solutions are neutralized by the above-mentioned 0.5 mol/L potassium hydroxide solution using phenolphthalein as an indicator just before used.
- f) **Phenolphthalein solution (10 g/L)**, prepared by using phenolphthalein specified in **JIS K 8799** and ethanol (95) specified in **JIS K 8102**.

7.8.7.3 Operation Take 5 g of the sample in a 400 ml beaker, dissolve in 50 ml of 2-propanol, add 40 ml of sulfuric acid (1+9) and agitate them. Transfer this solution to a 250 ml separating funnel as washing by 20 ml of petroleum ether, further add 30 ml of petroleum ether. Agitate the separating funnel sufficiently, after placed quietly, transfer the lower layer to another 250 ml separating funnel, add 10 ml of sulfuric acid (1+9) and 30 ml of petroleum ether, agitate them sufficiently, placed quietly, discard the lower layer part, and put the upper layer part together, as washing by 20 ml of petroleum ether, to the upper layer part in the previous separating funnel. Agitate this extracted solution sufficiently again, discard the separated matter in the lower layer, transfer this solution to a 500 ml Erlenmeyer flask as filtering by using a filter paper, and evaporate petroleum ether on water bath. Transfer it on a mass-known evaporating dish as washing by 10 ml of petroleum ether, evaporate petroleum ether on water bath, dry it in a dryer at 105 °C until it reaches a constant weight, and weigh the mass. In this case, never place it in the dryer for not less than 2 h.

Add 100 ml of neutral solvent and several drops of phenolphthalein solution to the contents, and agitate sufficiently until the sample has been dissolved completely. Titrate this solution by using potassium hydroxide solution, and take the point, where the pale pink colour of indicator has continued for 30 s, as the end point. The acid value of fatty acid shall be calculated according to formula (43).

$$A = \frac{56.11 \times 0.5 \times D \times f}{C_1 - C_2} \dots\dots\dots (69)$$

- where, *A* : acid value of fatty acid
 D : required amount of potassium hydroxide solution (0.5 mol/L) (ml)
 f : factor of potassium hydroxide solution (0.5 mol/L)
 *C*₁ : total mass of residue and evaporating dish (g)
 *C*₂ : mass of evaporating dish (g)

7.8.8 pH The sample is adjusted so as to be the specified concentration, and the pH shall be determined according to **JIS Z 8802**.

7.8.9 State of product The state of product shall be visually checked.

7.9 Metallic salts of naphthenic acid series of wood preservatives

7.9.1 Sampling method The sampling of the representative sample shall be in accordance with **JIS K 2420**. Provided that the sample shall be taken from an optional place under well-mixed condition.

7.9.2 Preparation of sample solution Weigh out approximately 0.5 g of the sample to the nearest 0.1 mg in a 300 ml tall beaker, add approximately 10 ml of hydrogen peroxide specified in **JIS K 8230** and 4 ml of sulfuric acid specified in **JIS K 8951**, place a watch glass on it and heat to decompose it gradually on sand bath. After leaving it cool as it is, transfer it to a 500 ml volumetric flask as filtering with a sheet of filter paper, wash the tall beaker and the watch glass by water sufficiently, add the washings to the volumetric flask, add water up to the marked line and take this solution as the sample solution.

7.9.3 Copper naphthenate

7.9.3.1 Summary The copper in the sample solution is determined according to atomic absorption spectrochemical analysis.

7.9.3.2 Reagents The reagents shall be as follows:

- a) **Sulfuric acid**, specified in **JIS K 8951**.
- b) **Sulfuric acid (1+4)**, prepared by using sulfuric acid specified in **JIS K 8951**.
- c) **Sulfuric acid (1+124)**, prepared by using sulfuric acid specified in **JIS K 8951**.
- d) **Copper standard solution for working curve (100 mg/L as Cu)**, prepared so that 10 ml of copper standard solution (1 000 mg/L as Cu) specified in **JIS K 0010** is weighed out by using a volumetric pipette, poured in a 100 ml volumetric flask, and 4 ml of sulfuric acid (1+4) is added, then water is added to the marked line.
- e) **Hydrogen peroxide**, specified in **JIS K 8230**.

7.9.3.3 Apparatus For the apparatus, an atomic absorption spectrochemical analyzer shall be used.

7.9.3.4 Operation Weigh out 4 ml of the sample solution by using a volumetric pipette, pour it in a 100 ml volumetric flask, add sulfuric acid (1+124) up to the marked line, and take this solution as the analytical sample solution. Spray the analytical sample solution into flame according to clause 6 of **JIS K 0121**, read the absorbance at 324.8 nm in wavelength, and obtain the concentration of copper in the analytical sample solution from the working curve. The copper content in the sample shall be calculated according to formula (70).

7.9.3.5 Calculation The amount of copper in the sample shall be calculated according to formula (70).

$$C_0 = c \times 0.0125 \dots\dots\dots (70)$$

where, C_0 : amount of copper in the sample (as Cu) weighed out in **7.9.2** (g)

c : concentration of copper in the analytical sample solution (as Cu) (mg/L)

7.9.3.6 Obtaining method for effective composition content The effective composition content shall be calculated from the results of **7.9.3.5** according to formula (71). The calculated value shall be rounded off to two significant figures.

$$C = \frac{C_0}{S} \times 100 \dots\dots\dots (71)$$

where, C : copper content in the sample (as Cu) (mass percentage %)

C_0 : amount of copper in the sample (as Cu) weighed out in **7.9.2** (g)

S : mass of the sample (g)

7.9.3.7 Preparation of working curve Weigh out 0 ml to 5 ml of the copper standard solution for working curve (100 mg/L as Cu) stepwise by using a volumetric pipette, pour them in a 100 ml volumetric flask and add sulfuric acid (1+124) to the marked line so as to become the same condition as the analytical sample solution. Operate this solution as similarly as the sample solution, and prepare the working curve of copper amount to absorbance. The preparation of working curve shall be carried out each time when used.

7.9.4 Zinc naphthenate

7.9.4.1 Summary The zinc in the sample solution is determined according to atomic absorption spectrochemical analysis.

7.9.4.2 Reagents The reagents shall be in accordance with **7.9.3.2**. Provided that the zinc standard solution for working curve shall be as follows.

Zinc standard solution for working curve (100 mg/L as Zn), prepared so that 10 ml of the zinc standard solution (1 000 mg/L as Zn) specified in **JIS K 0011** is weighed out by using a volumetric pipette, poured in a 100 ml volumetric flask, and 4 ml of sulfuric acid (1+4) is added, then water is added up to the marked line.

7.9.4.3 Apparatus For the apparatus, an atomic absorption spectrochemical analyzer shall be used.

7.9.4.4 Operation Operate the sample solution prepared in **7.9.2** as similarly as in **7.9.3.4**, and take this solution as the analytical sample solution. Spray the analytical sample solution into flame according to clause **6** of **JIS K 0121**, read the absorbance at 213.9 nm in wavelength, calculate the zinc content in the analytical sample solution from the working curve and obtain the concentration.

7.9.4.5 Calculation The amount of zinc in the sample shall be calculated according to formula (72).

$$Z_0 = z \times 0.0125 \dots\dots\dots (72)$$

where, Z_0 : amount of zinc in the sample (as Zn) weighed out in
7.9.2 (g)
 z : concentration of zinc in the analytical sample solution
(as Zn) (mg/L)

7.9.4.6 Obtaining method for effective composition content The effective composition content shall be calculated from the result of 7.9.4.5 according to formula (73). The calculated value shall be rounded off to two significant figures.

$$Z = \frac{Z_0}{S} \times 100 \dots\dots\dots (73)$$

where, Z : zinc content in the sample (as Zn) (mass percentage %)
 Z_0 : amount of zinc in the sample (as Zn) weighed out in
7.9.2 (g)
 S : mass of the sample (g)

7.9.4.7 Preparation of working curve Weigh out 0 ml to 5 ml of the zinc standard solution for working curve (100 mg/L as Zn) stepwise by using a volumetric pipette, pour it in a 100 ml volumetric flask and add sulfuric acid (1+124) up to the marked line so as to become the same condition as the analytical sample solution. Operate this solution as similarly as the analytical sample solution and prepare the working curve of zinc amount to absorbance. The preparation of working curve shall be carried out each time when used.

7.9.4.8 Acid value of fatty acid The acid value of fatty acid shall be in accordance with 7.8.7.

7.9.4.9 State of product The state of product shall be visually checked.

7.10 Azole/neonicotinoid compound series of wood preservatives

7.10.1 Sampling method The sampling of the representative sample shall be in accordance with JIS K 2420. Provided that the sample shall be taken from an optional place under well-mixed condition.

7.10.2 Preparation of sample solution Weigh out approximately 1.5 g of the sample to the nearest 0.1 mg in a 50 ml eggplant type flask, add approximately 2 ml of dimethyl sulfoxide specified in JIS K 9702, and concentrate this solution in hot water at approximately 45 °C by using a rotary evaporator until the liquid amount becomes approximately 2 ml. Transfer into a 25 ml volumetric flask as washing the concentrated solution and the inside wall of eggplant type flask by using a small amount of ethanol specified in JIS K 8101, add ethanol specified in JIS K 8101 up to the marked line, and take this solution as the sample solution.

7.10.3 Cyproconazole The cyproconazole content shall be in accordance with 7.4.6.2.

7.10.3.1 Summary The cyproconazole in the sample is determined according to the external standard method of high performance liquid chromatography.

7.10.3.2 Reagents The reagents shall be as follows:

- a) **Standard stock solution for working curve**, prepared so that approximately 270 mg of cyproconazole (the purity is known and not less than 95 %) is weighed out to the nearest 0.1 mg, poured in a 25 ml volumetric flask and ethanol specified in **JIS K 8101** is added to the marked line.
- b) **Mobile phase**, prepared by mixing 60 ml of acetonitrile specified in **JIS K 8032** and 40 ml of water.

7.10.3.3 Apparatus For the apparatus, a high performance liquid chromatograph shall be used.

7.10.3.4 Analytical condition of high performance liquid chromatography
The analytical condition of high performance liquid chromatography shall be in accordance with table 20.

Table 20 Analytical condition of high performance liquid chromatography

Item	Condition
Column	ODS system column I.D. 4.6 mm × L. 150 mm
Mobile phase	In accordance with 7.10.3.2 b).
Mobile phase flow rate	1.0 ml/min
Column temperature	40 °C
Measuring wavelength	221 nm
Injection amount	10 µl

7.10.3.5 Operation Filter the sample solution by using a filter of 0.45 µm in pore diameter, inject 10 µl of the filtrate into the high performance liquid chromatograph, obtain the peak area of cyproconazole, and obtain the amount of cyproconazole from the working curve.

7.10.3.6 Preparation of working curve Weigh out 1 ml to 5 ml of the stock solution for working curve stepwise by using a volumetric pipette, pour in a 25 ml volumetric flask, and add ethanol specified in **JIS K 8101** up to the marked line. Filter this solution by using a filter of 0.45 µm pore diameter, inject 10 µl of the filtrate into the high performance liquid chromatograph, and prepare the working curve of the concentration of cyproconazole to the peak area. The preparation of working curve shall be carried out each time when used.

7.10.3.7 Calculation The mass of cyproconazole in the sample shall be calculated according to formula (74).

$$C_y = \frac{c \times 25}{10^6} \dots\dots\dots (74)$$

where, C_y : cyproconazole content in the sample (g)
 c : concentration of cyproconazole in the sample solution (mg/L)

7.10.4 Imidacloprid

7.10.4.1 Summary The imidacloprid in the sample is determined according to the external standard method of high performance liquid chromatography.

7.10.4.2 Reagents The reagents shall be as follows:

- a) **Standard stock solution for working curve**, prepared so that approximately 130 mg of imidacloprid (the purity is known and not less than 98 %) is weighed out to the nearest 0.1 mg, poured in a 25 ml volumetric flask, and ethanol specified in **JIS K 8101** is added up to the marked line.
- b) **Mobile phase**, prepared by mixing 60 ml of acetonitrile specified in **JIS K 8032** and 40 ml of water.

7.10.4.3 Apparatus For the apparatus, a high performance liquid chromatograph shall be used.

7.10.4.4 Analytical condition of high performance liquid chromatography
The analytical condition of high performance liquid chromatography shall be in accordance with table 21.

Table 21 Analytical condition of high performance liquid chromatography

Item	Condition
Column	ODS system column I.D. 4.6 mm × L. 150 mm
Mobile phase	In accordance with 7.10.4.2 b).
Mobile phase flow rate	1.0 ml/min
Column temperature	40 °C
Measuring wavelength	271 nm
Injection amount	10 µl

7.10.4.5 Operation Filter the sample solution by using a filter of 0.45 µm in pore diameter, inject 10 µl of the filtrate into the high performance liquid chromatograph, obtain the peak area of imidacloprid and obtain the amount of imidacloprid from the working curve.

7.10.4.6 Preparation of working curve Weigh out 1 ml to 5 ml of the stock solution for working curve stepwise by using a volumetric pipette, pour it in a 25 ml volumetric flask, and add ethanol specified in **JIS K 8101** up to the marked line. Filter this solution by using a filter of 0.45 µm in pore diameter, inject 10 µl of the filtrate into the high performance liquid chromatograph, and prepare the working curve of the concentration of imidacloprid to the peak area. The preparation of working curve shall be carried out each time when used.

7.10.4.7 Calculation The amount of imidacloprid in the sample shall be calculated according to formula (75).

$$I = \frac{i \times 25}{10^6} \dots\dots\dots (75)$$

where, I : imidacloprid content in the sample (g)
 i : concentration of imidacloprid in the sample solution (mg/L)

7.10.5 Calculation for compound ratio of effective composition The compound ratio of effective composition shall be calculated from the results of **7.10.3** and **7.10.4** according to formula (76) and formula (77). The calculated value shall be rounded off to two significant figures.

$$Cy_0 = \frac{Cy}{Cy + I} \times 100 \dots\dots\dots (76)$$

$$I_0 = \frac{I}{Cy + I} \times 100 \dots\dots\dots (77)$$

where, Cy_0 : compound ratio of cyproconazole in the sample
 I_0 : compound ratio of imidacloprid in the sample
 Cy : cyproconazole content in the sample (g)
 I : imidacloprid content in the sample (g)

7.10.6 Obtaining method for effective composition content The effective composition content shall be calculated from the results of **7.10.3**, **7.10.4** and **7.10.5** according to formula (78). The calculated value shall be rounded off to two significant figures.

$$A = \frac{(Cy + I)}{S} \times 100 \dots\dots\dots (78)$$

where, A : effective composition content in the sample (mass percentage %)
 S : mass of the sample weighed out in **7.10.2** (g)
 Cy : Cy in formula (74)
 I : I in formula (75)

7.10.7 State of product The state of product shall be visually checked.

7.11 Creosote oil of wood preservatives

7.11.1 Sampling and preparation of sample The sampling of the representative sample shall be in accordance with **JIS K 2420**. Provided that the representative sample shall be taken from an optional place under well-mixed condition. One litre of the representative sample, well-mixed, shall be taken as the sample.

7.11.2 Test methods The test methods shall be as follows:

- a) **Density** Take the sample heated at approximately 40 °C in a dried and clean glass cylinder of approximately 40 mm in inside diameter and approximately 350 mm in length, put it in a water tank adjusted at approximately 40 °C, float No. 7 or No. 6 density hydrometer of large size, 14 pieces set specified in **JIS B 7525** gently, and leave it as it is for 10 min.

In such a case, the difference in temperature between the water tank and the sample shall not be 5 °C or over. After placed gently, read the graduation of hydrometer. Remove the hydrometer, immediately put in a thermometer and agitate, and measure the temperature of sample. The temperature of the sample, at this time, shall be 35 °C to 45 °C.

The density shall be calculated from the graduation of hydrometer and temperature according to formula (79).

$$d = d_1 + 0.000\ 70(t - 40) - 0.000\ 6 \dots\dots\dots (79)$$

where, d : density at 40 °C (g/cm³)

d_1 : graduation of hydrometer read out

t : measuring temperature

0.000 6 : temperature correction value

- b) **Moisture content**

- 1) **Apparatus** The apparatus shall be as follow:

- 1.1) **Receiver for determination of moisture content**, as shown in figure 1, and being 5 ml in capacity, graduated at interval of 0.05 ml and marked with figures indicating the graduation at interval of 0.5 ml.

Moreover, a spiral wire of length so as to reach to the liquid surface shall be attached to the lowest part of the side pipe of receiver for determination of moisture content so as to avoid bumping.

- 1.2) **Rounded-bottom flask**, being 500 ml in capacity.

- 2) **Reagents** The reagents shall be as follows:

- 2.1) **Toluene**, prepared by dehydrating pure toluene specified in **JIS K 2435** or toluene specified in **JIS K 8680**, respectively.

- 2.2) **Xylene**, prepared by dehydrating 5 degrees of xylene specified in **JIS K 2435** or xylene specified in **JIS K 8271**, respectively.

- 3) **Operation** Take approximately 100 ml of the sample in a 500 ml dried, clean rounded bottom flask, add 100 ml of toluene or xylene to this solution, and mix sufficiently by agitating.

Moreover, the test shall be carried out so that water is previously poured up to the graduation of 0 ml in the receiver for determination moisture content.

Unit: mm

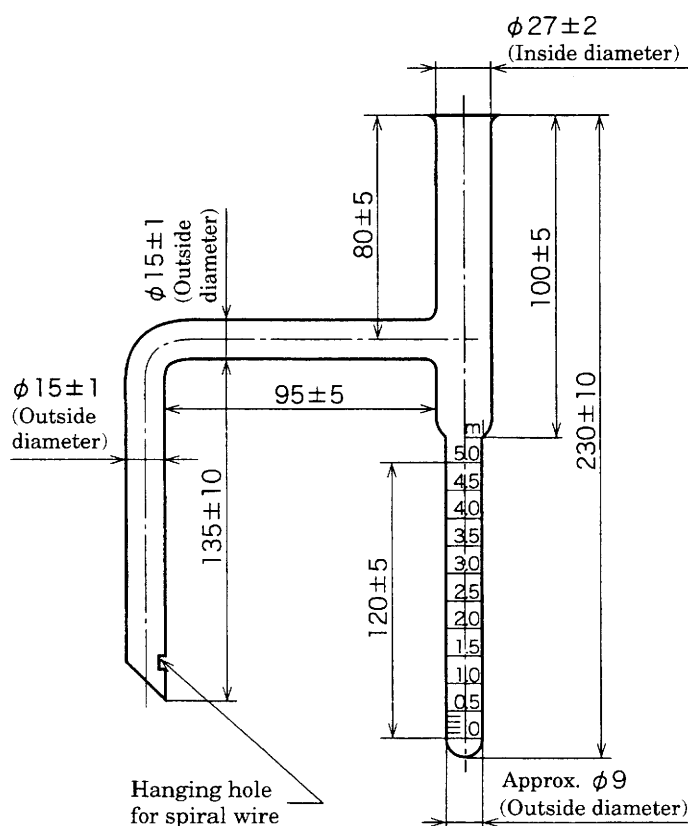


Figure 1 Receiver for determination of moisture content

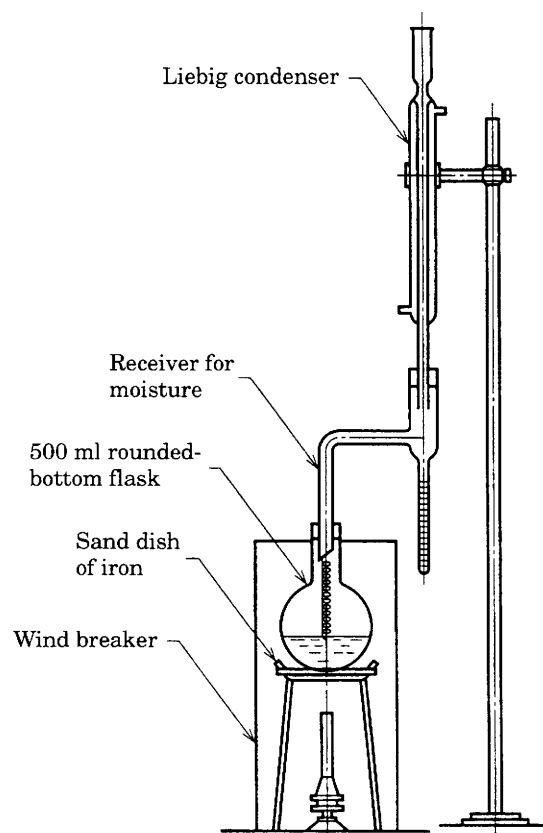


Figure 2 An example of receiver for determination of moisture content

c) **Engler degrees**

- 1) **Apparatus** The apparatus shall be as follows:
 - 1.1) **Engler viscometer**, as shown in table 22 and figure 3.

Table 22 Sample container of brass for Engler viscometer

Unit: mm

Item	Dimension
Inside diameter	106.0 ± 1.0
Height from the lower end of outlet to marking pointer	52.0 ± 0.5
Height from the lower end of cylinder part to marking pointer	25.0 ± 1.0
Length of outlet	20.0 ± 0.1
Inside diameter at the upper end of outlet	2.90 ± 0.02
Inside diameter at the lower end of outlet	2.80 ± 0.02
Length of projected part of outlet	3.00 ± 0.03

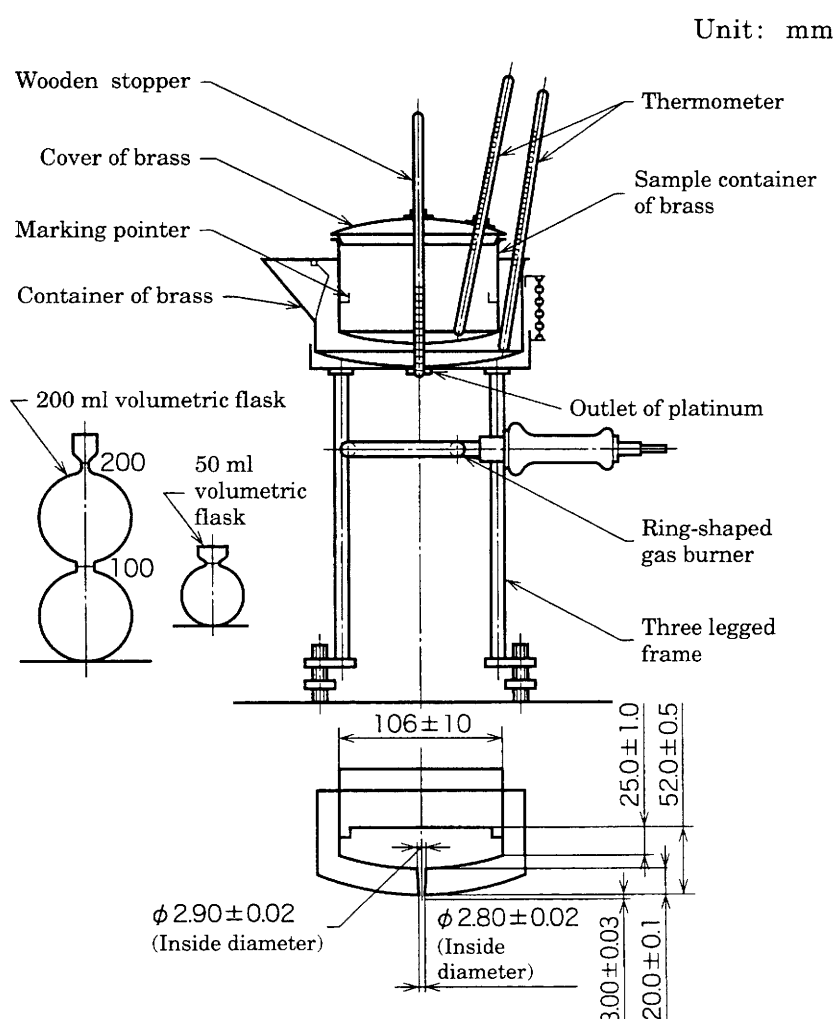


Figure 3 An example of Engler viscometer

- 2) **Operation** Wash the inside of sample container of brass and the outlet by toluene or xylene and water successively, clog the outlet with a wooden stopper, and fill up to the top end of marking pointer in the container by water at 20 °C. In such a time, the outer cylinder shall be filled with the water at the same temperature. Then, loose the wooden stopper slightly,

stop under the condition where the water is going to drop from the end of outlet, further add a small amount of water to fit the water surface in the container to the marking pointer accurately.

Then, place a 200 ml volumetric flask just below the outlet, pull out the wooden stopper, let water flow down so as not to touch on the wall of volumetric flask, and measure the number of seconds required for 200 ml of out flow with a stop watch. Take the mean value of three measured results as the flow-out time of water.

Then, after drying the outlet and container sufficiently, make the temperature of sample $40\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$, put it in the container up to the marking pointer, pour water at $41\text{ }^{\circ}\text{C}$ to $42\text{ }^{\circ}\text{C}$ in the outer cylinder, then make the temperature of sample $40\text{ }^{\circ}\text{C}$ accurately. Measure once the flow-out time (second) in a 200 ml volumetric flask, and calculate Engler degree ($40/20\text{ }^{\circ}\text{C}$) according to formula (80).

$$E = \frac{T_1}{T_2} \dots\dots\dots (80)$$

where, E : Engler degree ($40/20\text{ }^{\circ}\text{C}$)

T_1 : flow-out time of the sample (s)

T_2 : flow-out time of water (s)

d) **Distillation test**

1) **Apparatus** The apparatus shall be as follows (see figure 4).

1.1) **Distillation flask**, being 250 ml in capacity.

1.2) **Thermometer**, being a cylindrical mercury thermometer, carved with graduation at interval of $1\text{ }^{\circ}\text{C}$ in the range between $100\text{ }^{\circ}\text{C}$ and $360\text{ }^{\circ}\text{C}$, and marked with the number indicating the graduation at interval of $10\text{ }^{\circ}\text{C}$, and the dimensions shall be as shown in table 23.

Table 23 Thermometer

Unit: mm

Item	Dimension
Outside diameter of stem	5 to 7
Length of mercury bulb	10 to 20
Distance from the lower end of mercury bulb to $360\text{ }^{\circ}\text{C}$ of graduation	280 to 350

Unit: mm

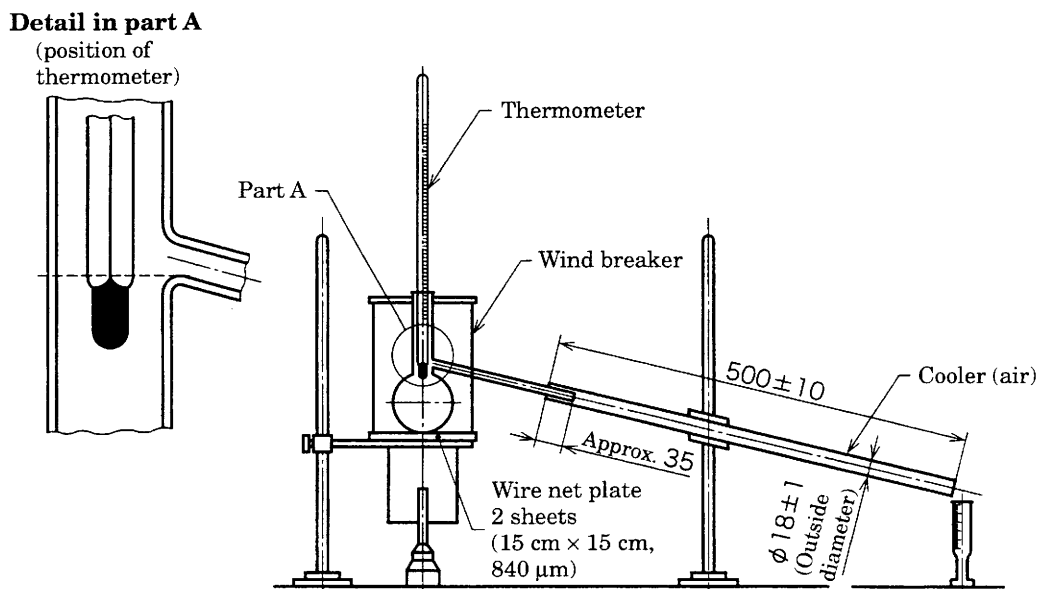


Figure 4 An example of distillation apparatus

- 1.3) **Cooler**
- 1.4) **Receiver (A)**, as shown in figure 5, being a measuring cylinder having approximately 260 mm in whole length, 100 ml in capacity, carved with graduation at the interval of 0.2 ml in the range between 0 ml and 7 ml, at the interval of 1 ml in the range between 7 ml to 10 ml and at the interval of 10 ml in the range between 10 ml and 100 ml.
- 1.5) **Receiver (B)**, being a measuring cylinder having approximately 28 mm in inside diameter, approximately 220 mm in whole length and 100 ml in capacity, and carved with graduation at the interval of 1 ml.
- 1.6) **Wind breaker and cover**, of slate sheet of approximately 5 mm in thickness, as shown in figure 6.

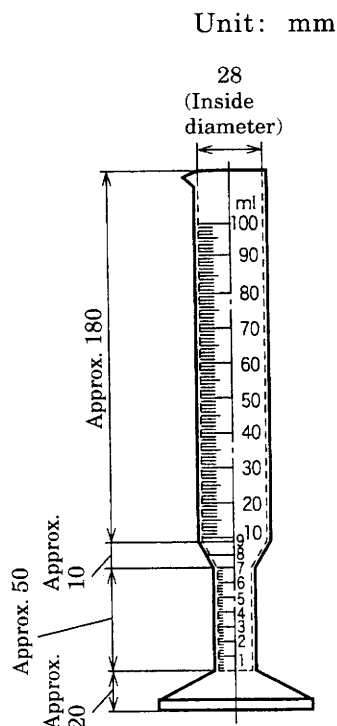


Figure 5 Receiver (A)

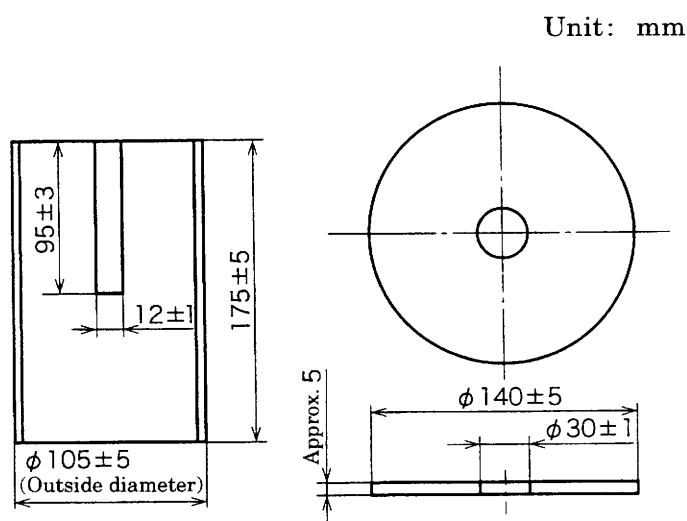


Figure 6 Wind breaker and cover

- 2) **Reagent** The reagent shall be as follows:
 - 2.1) **Calcium chloride**, specified in **JIS K 8125**.
- 3) **Preparation of dehydrated sample** Heat approximately 200 ml of the sample at approximately 50 °C, add approximately 60 g of calcium chloride specified in **JIS K 8125** as a desiccant, agitate sufficiently, then keep it at approximately 50 °C for approximately 10 min. After leaving it cool as it is, filter it and take it as the dehydrated sample.

- 3.1) **Operation** Take 100 ml⁽¹¹⁾ of the dehydrated sample in a dried, clean distillation flask and fix a thermometer with cork stopper. Fix a thermometer so that the upper end of mercury bulb is positioned at the lower joint of the branch pipe of distillation flask, then place on an appropriate heating apparatus. Connect the branch pipe so as to insert in the cooler by approximately 35 mm, place the receiver (A) at the lower end of the cooler, and assemble the whole apparatus as shown in figure 4.

After distillation begins, heat⁽¹²⁾ so as to distil from the cooler into the receiver at the rate of 60 drops per minute. When something is crystallized in the cooler during distillation, heat appropriately to dissolve and let it flow down.

When reaching to 235 °C in such a way, replace by the receiver (B). When reaching to 315 °C, immediately stop heating, let the distillate left in the cooler flow down in the receiver, and weigh the distillate of the receiver (A) and receiver (B).

When some crystals are precipitated on the liquid surface of distillate in the receiver, heat the receiver to dissolve crystals, and weigh the amount of distillate.

Notes ⁽¹¹⁾ When the volume percentage of moisture content in the sample is 0.5 % or lower, it may be used for the test as it is.

⁽¹²⁾ When the adjustment of distillation from cooler is difficult because of the precipitation of crystal, the number of drops falling from the branch pipe of the flask may be read.

4) Density of fraction in the range between 235 °C to 315 °C

- 4.1) **Apparatus** The apparatus shall be as follows.

Pycnometer, as shown in figure 7.

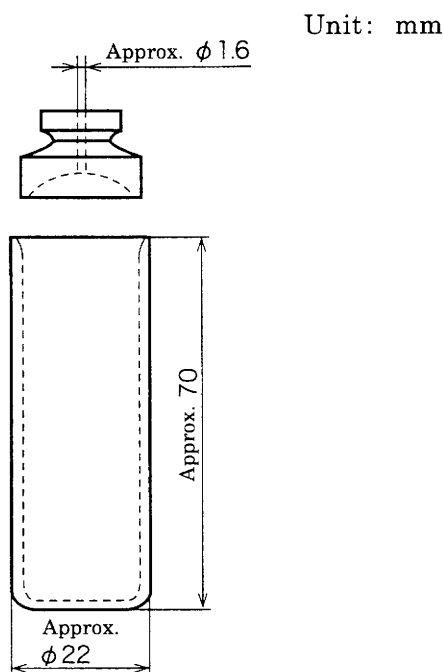


Figure 7 Pycnometer

- 4.2) **Operation** Weigh the mass of a dried, clean pycnometer with stopper. Then, fill this pycnometer with water at 40 °C until the water over flows, place it in a water vessel maintained at 40 °C previously for 30 min or over, and take out, remove by absorbing the water in a capillary of stopper rapidly with using of a piece of paper or others, and let the position of liquid coincide with the marked line of capillary. After wiping off the moisture adhering to the outside by using a piece of dry cloth, weigh the mass.

Then, remove the water in this pycnometer, dry it, then pour the sample at 40 °C in it to carry out the same operation, finally wipe off the moisture, oil and others adhering to the outside of pycnometer completely, and then weigh the mass. The density of the sample at 40 °C shall be calculated according to formula (81).

$$d = [(M_3 - M_1)/(M_2 - M_1)] \times 0.992 \ 2 \dots\dots\dots (81)$$

where, d : density at 40 °C (g/cm³)

M_1 : mass of pycnometer (g)

M_2 : mass of (pycnometer + water) (g)

M_3 : mass of (pycnometer + sample) (g)

0.992 2 : factor to convert the density at 40 °C to the density at 4 °C

e) **Toluene insoluble content**

- 1) **Apparatus** The apparatus shall be as follows:

1.1) **Rounded bottom flask**, being 125 ml in capacity.

1.2) **Glass filter**, being a crucible type glass filter, 1 G4, specified in **JIS R 3503**.

- 2) **Reagents** The reagents shall be as follows:

2.1) **Toluene**, being pure toluene specified in **JIS K 2435** or toluene specified in **JIS K 8680**.

2.2) **Acetone**, specified in **JIS K 1503** or **JIS K 8034**.

- 3) **Operation** Weigh out approximately 10 g of the sample to the nearest 1 mg in a 125 ml rounded bottom flask, add approximately 50 ml of toluene heated at 50 °C to 60 °C previously, agitate them sufficiently, then fix a reflux condenser and heat it until the solution boils.

After heating, immediately suction and filter by using a glass filter. Then, wash the residue and the glass filter by using heated toluene until the washings become colourless, further wash the residue four times with each approximately 5 ml of acetone, then dry the filter at approximately 110 °C for 60 min, leave it cool in a desiccator as it is, then weigh the mass and calculate the toluene insoluble content according to formula (82).

$$U = [(M_1 - M_2) \times 100/S] \times [100/(100 - M_3)] \dots\dots\dots (82)$$

where, U : toluene insoluble content (mass percentage %)

M_1 : mass of (filter + residue) (g)

M_2 : mass of filter (g)

M_3 : moisture content of the sample obtained according to
7.11.2 b) (volume percentage %)

S : mass of the sample

f) **Fluidity test**

1) **Apparatus** The apparatus shall be as follows:

1.1) **Erlenmeyer flasks**, being 100 ml and 300 ml in capacity.

1.2) **Filter**, being a funnel type glass filter, 3 G3, specified in **JIS R 3503**.

1.3) **Thermometers**

1.3.1) **Cylindrical mercury thermometer**, in which the graduation range is 0 °C to 100 °C, carved at intervals of 1 °C in graduation.

1.3.2) **Double wall mercury thermometer with immersion line**, of which the graduation surface is opal plate, and the dimensions and specifications are in accordance with table 24.

Table 24 Double wall mercury thermometer with immersion line

Item	Dimensions
Temperature range	15 °C to 45 °C
Liquid	Mercury
Gas filled on the liquid	Nitrogen
Minimum graduation	0.1 °C
Whole length	330 mm to 350 mm
Outside diameter of stem	6 mm to 7 mm
Outside diameter of large mercury bulb	5 mm to 6 mm
Length of large mercury bulb	10 mm to 15 mm
Distance from the lower end of large mercury bulb to the lowest graduation line	100 mm to 105 mm
Distance from the upper end of thermometer to the highest graduation line	20 mm to 30 mm
Distance from the lower end of large mercury bulb to immersion line	100 mm
Tolerance of temperature	0.1 °C

1.4) **Thermostatic water tank**, capable of regulating at 32.0 °C ± 0.5 °C.

1.5) **Dryer**, being a thermostatic dryer capable of regulating at approximately 80 °C.

1.6) **Water bath**, capable of receiving a 300 ml Erlenmeyer flask in size.

1.7) **Suction filtering apparatus**, capable of receiving a 100 ml Erlenmeyer flask in size appropriately.

2) **Reagent** The reagent shall be as follows.

2.1) **Desiccant**, being calcium chloride specified in **JIS K 8125**.

3) **Operation**

3.1) **Preparation of test sample** Take approximately 100 ml of the sample, which is made uniform by agitating sufficiently, in a 300 ml Erlenmeyer flask, and fix a cylindrical mercury thermometer to the neck part of the flask by using a cork stopper. Immerse the flask in water bath, and heat it to 60 °C to 80 °C as shaking. After adding approximately 30 g of desiccant and shaking sufficiently, keep the sample at 60 °C to 80 °C for 10 min.

Then, take out the filter placed in a dryer regulated previously at approximately 80 °C for 5 min, immediately assemble the filtering apparatus, and filter the sample by decantation method. When approximately 50 ml of the filtrate is obtained, stop to filter and take this filtrate as the test sample.

3.2) **Operation** Take approximately 50 ml of the test sample in a 100 ml Erlenmeyer flask, and fix the double wall mercury thermometer to the neck part of the flask by using a cork stopper. In such a case, make the large mercury bulb of the thermometer so as to be immersed in the test sample. Immerse the flask in the water bath, heat the test sample to 35 °C to 40 °C, and confirm there is not any solid matter in the test sample, then put the flask in the thermostatic water tank regulated at $32\text{ °C} \pm 0.5\text{ °C}$ and make the solution surface of the test sample so as to be below the water surface in the thermostatic water tank. From the time when the temperature of the test sample has reached to $32.0\text{ °C} \pm 0.5\text{ °C}$, keep it at the same temperature for 2 h, then take out the flask, and immediately examine whether any solid matter is produced on the solution surface of the sample or not.

8 Marking The following information shall be marked.

- a) Title and number of this Standard
- b) Symbols of classification
- c) Effective composition content
- d) Name of manufacturer or its abbreviation
- e) Month and year of manufacture or its abbreviation
- f) Serial number or lot number

Remarks : The item **c)** shall not apply to creosote oil.

Attached table 1 Normative references

JIS B 7525	<i>Density hydrometers</i>
JIS K 0010	<i>Reference materials—Standard solution—Copper</i>
JIS K 0011	<i>Zinc standard solution</i>
JIS K 0050	<i>General rules for chemical analysis</i>
JIS K 0102	<i>Testing methods for industrial wastewater</i>
JIS K 0114	<i>General rules for gas chromatographic analysis</i>
JIS K 0115	<i>General rules for molecular absorptiometric analysis</i>
JIS K 0116	<i>General rules for atomic emission spectrometry</i>
JIS K 0121	<i>General rules for atomic absorption spectrochemical analysis</i>
JIS K 0124	<i>General rules for high performance liquid chromatography</i>
JIS K 1503	<i>Acetone</i>
JIS K 1571	<i>Qualitative standards and testing methods of wood preservatives</i>
JIS K 2420	<i>Method of sampling for aromatic hydrocarbons and tar products</i>
JIS K 2435	<i>Benzene • Toluene • Xylene</i>
JIS K 3362	<i>Testing methods for synthetic detergent</i>
JIS K 8032	<i>Acetonitrile</i>
JIS K 8034	<i>Acetone</i>
JIS K 8101	<i>Ethanol (99.5)</i>
JIS K 8102	<i>Ethanol (95)</i>
JIS K 8125	<i>Calcium chloride (for U-tube)</i>
JIS K 8230	<i>Hydrogen peroxide</i>
JIS K 8271	<i>Xylene</i>
JIS K 8322	<i>Chloroform</i>
JIS K 8355	<i>Acetic acid</i>
JIS K 8361	<i>Ethyl acetate</i>
JIS K 8464	<i>Cyclohexane</i>
JIS K 8574	<i>Potassium hydroxide</i>
JIS K 8593	<i>Petroleum ether</i>
JIS K 8613	<i>Ammonium carbonate</i>
JIS K 8625	<i>Sodium carbonate</i>
JIS K 8680	<i>Toluene</i>
JIS K 8799	<i>Phenolphthalein</i>
JIS K 8839	<i>2-Propanol</i>
JIS K 8844	<i>Bromophenol blue</i>

Attached table 1 (concluded)

JIS K 8863	<i>Boric acid</i>
JIS K 8891	<i>Methanol</i>
JIS K 8897	<i>Methylene blue</i>
JIS K 8948	<i>Iron (II) sulfide</i>
JIS K 8951	<i>Sulfuric acid</i>
JIS K 8987	<i>Sodium sulfate</i>
JIS K 9005	<i>Phosphoric acid</i>
JIS K 9009	<i>Sodium dihydrogenphosphate dihydrate</i>
JIS K 9702	<i>Dimethyl sulfoxide</i>
JIS R 3503	<i>Glass apparatus for chemical analysis</i>
JIS R 3505	<i>Volumetric glassware</i>
JIS Z 8401	<i>Guide to the rounding of numbers</i>
JIS Z 8802	<i>Methods for determination of pH of aqueous solutions</i>

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