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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 Protection 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 1570:2004** is replaced with this Standard.

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Wood preservatives

Introduction

This Japanese Industrial Standard was established in 1995, and has been revised twice before this revision. The last revision was made in 2004 and the revision at this time involves reviewing of copper/azole compound series and creosote oil, addition of azole/quarternary ammonium/neonicotinoid compound series, and reflection of latest status of production and applications of the product.

No corresponding International Standard has been established at this point.

1 Scope

This Standard specifies the wood preservatives for giving wood materials antiseptic and termite proofing performance which are used by pressure injection treatment.

2 Normative references

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

- JIS A 9002 *Preservative treatments of wood products by pressure processes*
- JIS B 7525 *Density hydrometers*
- JIS H 2121 *Electrolytic cathode copper*
- JIS K 0050 *General rules for chemical analysis*
- JIS K 0061 *Test methods for density and relative density of chemical products*
- JIS K 0102 *Testing methods for industrial wastewater*
- JIS K 0114 *General rules for gas chromatographic analysis*
- JIS K 0115 *General rules for molecular absorptiometric analysis*
- JIS K 0116 *General rules for atomic emission spectrometry*
- JIS K 0121 *General rules for atomic absorption spectrometry*
- JIS K 0124 *General rules for high performance liquid chromatography*
- JIS K 1503 *Acetone*
- JIS K 1571 *Wood preservatives—Performance requirements and their test methods for determining effectiveness*
- JIS K 2249 *Crude petroleum and petroleum products—Determination of density and petroleum measurement tables based on a reference temperature (15 °C)*
- JIS K 2435-2 *Benzene·Toluene·Xylene—Part 2: Toluene*
- JIS K 2435-3 *Benzene·Toluene·Xylene—Part 3: Xylene*
- JIS K 3362 *Test method of household synthetic detergent*

- JIS K 8012 *Zinc (Reagent)*
JIS K 8032 *Acetonitrile (Reagent)*
JIS K 8034 *Acetone (Reagent)*
JIS K 8101 *Ethanol (99.5) (Reagent)*
JIS K 8102 *Ethanol (95) (Reagent)*
JIS K 8117 *Dichloromethane for pesticide residue and polychlorinated biphenyl analysis (Reagent)*
JIS K 8125 *Calcium chloride (for U-tube) (Reagent)*
JIS K 8230 *Hydrogen peroxide (Reagent)*
JIS K 8271 *Xylene (Reagent)*
JIS K 8322 *Chloroform (Reagent)*
JIS K 8361 *Ethyl acetate (Reagent)*
JIS K 8464 *Cyclohexane (Reagent)*
JIS K 8541 *Nitric acid (Reagent)*
JIS K 8574 *Potassium hydroxide (Reagent)*
JIS K 8593 *Petroleum ether (Reagent)*
JIS K 8613 *Ammonium carbonate (Reagent)*
JIS K 8625 *Sodium carbonate (Reagent)*
JIS K 8660 *Copper (Reagent)*
JIS K 8680 *Toluene (Reagent)*
JIS K 8799 *Phenolphthalein (Reagent)*
JIS K 8839 *2-Propanol (Reagent)*
JIS K 8844 *Bromophenol blue (Reagent)*
JIS K 8891 *Methanol (Reagent)*
JIS K 8897 *Methylene blue (Reagent)*
JIS K 8951 *Sulfuric acid (Reagent)*
JIS K 8953 *Zinc sulfate heptahydrate (Reagent)*
JIS K 8983 *Copper (II) sulfate pentahydrate (Reagent)*
JIS K 8987 *Sodium sulfate (Reagent)*
JIS K 9005 *Phosphoric acid (Reagent)*
JIS K 9009 *Sodium dihydrogenphosphate dihydrate (Reagent)*
JIS K 9702 *Dimethyl sulfoxide (Reagent)*
JIS R 3503 *Glass apparatus for chemical analysis*
JIS R 3505 *Volumetric glassware*
JIS Z 8401 *Guide to the rounding of numbers*
JIS Z 8802 *Methods for determination of pH of aqueous solutions*

3 Principle (pressure injection treatment)

A pressure treatment device containing wood materials is filled with wood preservative (a diluted solution of wood preservative for some types of chemicals) and wood preservative is injected into the wood materials by means of pressure pump under higher pressure than the inside of the wood materials for impregnation into the inside of the wood materials, for the purpose of giving the wood materials the antiseptic and termite proofing performances. The treatment method is specified in **JIS A 9002**.

4 Terms and definitions

For the purposes of this Standard, the terms and definitions given in **JIS K 1571** and the following apply.

4.1 antiseptic performance

performance of wood preservatives to prevent the deterioration due to wood rot bacteria (wood rotting fungus)

4.2 termite proofing performance

performance of wood preservatives to prevent the vermin damage done by Formosan subterranean termite and *Reticulitermes speratus*

4.3 iron corrosion performance

corrosion performance against iron, of wood treated with a wood preservative

4.4 injection treatment

treatment to penetrate wood preservative into wood by using the mechanical pressure difference produced such as by depressurizing or pressurizing

4.5 water soluble wood preservatives

wood preservatives to be used by dissolving in water

4.6 emulsified wood preservatives

wood preservatives to be used by emulsifying in water

4.7 oil soluble wood preservatives

wood preservatives to be used by dissolving in organic solvent

4.8 oil based wood preservatives

wood preservatives in oily state to be used by stock solution as it is

4.9 designated concentration

the concentration of chemicals solution required for ensuring the quantity of absorption corresponding to the product standard of each pressure injection treated product given in **8 a)** of **JIS A 9002**

It shall be designated by the manufacturers.

5 Classification

Wood preservatives are classified as shown in table 1.

Table 1 Classification of wood preservatives

Division	Class		Symbol of class
Water soluble wood preservatives	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		CUAZ
	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		AZNA
	Metallic salts of naphthenic acid series		
Oil soluble wood preservatives		No. 1	NCU-E
		No. 2	NZN-E
		No. 3	VZN-E
Oil based wood preservatives		No. 1	NCU-O
		No. 2	NZN-O
	Azole/neonicotinoid compound series		AZN
Oil based wood preservatives	Creosote oil		A

6 Quality

The quality of wood preservatives of respective types, when tested in accordance with clause 8, shall conform to a relevant table among tables 2 to 12 according to the class.

Table 2 Quality of wood preservatives of quaternary ammonium compound series

Item		Quality		Sub-clause of relevant test
		No. 1 (AAC-1)	No. 2 (AAC-2)	
Content of active substances (mass fraction %)	Didecyl dimethyl ammonium chloride (hereafter referred to as "DDAC".)	30 to 55	—	8.3.1 and 8.4.1.1 or 8.4.1.2
	N,N-Didecyl-N-methyl-polyoxyethyl-ammonium propionate (hereafter referred to as "DMPAP".)	—	30 to 55	8.3.1 and 8.4.1.1 or 8.4.1.2
State of product		Liquid state		8.6
Water insoluble content (mass fraction %)		1 max.		8.7
pH		5.0 to 8.0		8.8

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

Item		Quality		Sub-clause of relevant test
		No. 1 (ACQ-1)	No. 2 (ACQ-2)	
Compound ratio of active substances (mass fraction %)	Copper compound (as CuO)	53 to 59	62 to 71	8.3.2 and 8.4.2.1 or 8.4.2.2
	N-Alkyl benzyl dimethyl ammonium chloride (hereafter referred to as “BKC”.)	41 to 47	—	8.3.2 and 8.4.3.1
	DDAC	—	29 to 38	8.3.2 and 8.4.1.1
Total content of active substances ^{a)} (mass fraction %)		16 min.	—	8.5.1 b)
		—	13 min.	8.5.2 b)
State of product		Liquid state		8.6
Water insoluble content (mass fraction %)		1 max.		8.7
pH		9.5 to 11.0		8.8
Note ^{a)} The total content of active substances means the total amount of copper compound and BKC or DDAC.				

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

Item		Quality	Sub-clause of relevant test
		CUAZ	
Compound ratio of active substances (mass fraction %)	Copper compound (as CuO)	98.6 to 99.0	8.3.3 and 8.4.2.1 or 8.4.2.2
	Cyproconazole ^{a)} [α -(4-chlorophenyl)- α -(1-cyclopropylethyl)-1H-1,2,4-triazole-1-ethanol]	1.0 to 1.4	8.3.3 and 8.4.4.1 or 8.4.4.2
Total content of active substances ^{b)} (mass fraction %)		11 min.	8.5.3 b)
State of product		Liquid state	8.6
Water insoluble content (mass fraction %)		1 max.	8.7
pH		8.7 to 11.5	8.8
Notes ^{a)} For the compound containing cyproconazole, there is the following patent: Patent registration: No. 3216946 "Microbicidal composition" Date of registration: August 3, 2001			
^{b)} The total content of active substances means the total amount of copper compound and cyproconazole.			

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

Item		Quality	Sub-clause of relevant test
		BAAC	
Compound ratio of active substances (mass fraction %)	Boron compound (as H_3BO_3)	23 to 27	8.3.4 and 8.4.5.1
	DDAC	71 to 79	8.3.4 and 8.4.1.1
Total content of active substances ^{a)} (mass fraction %)		25 min.	8.5.4 b)
State of product		Liquid state	8.6
Water insoluble content (mass fraction %)		1 max.	8.7
pH		5.0 to 9.0	8.8
Note ^{a)} The total content of active substances means the total amount of boron compound and DDAC.			

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

Item		Quality	Sub-clause of relevant test
		SAAC	
Compound ratio of active substances (mass fraction %)	DMPAP	99.0 to 99.9	8.3.5 and 8.4.1.1 or 8.4.1.2
	Silafluofen [4-Ethoxy phenyl (3-(4-fluoro-3-phenoxy-phenyl) propyl) dimethyl silane]	0.4 to 0.6	8.3.5 and 8.4.6.1
Total content of active substances ^{a)} (mass fraction %)		35 min.	8.5.5 b)
State of product		Liquid state	8.6
Water insoluble content (mass fraction %)		1 max.	8.7
pH		5.0 to 8.0	8.8
Note ^{a)} The total content of active substances means the total amount of DMPAP and [4-Ethoxy phenyl (3-(4-fluoro-3-phenoxy-phenyl) propyl) dimethyl silane].			

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

Item		Quality	Sub-clause of relevant test
		AZAAC	
Compound ratio of active substances (mass fraction %)	DMPAP	96.7 to 97.3	8.3.6 and 8.4.1.1 or 8.4.1.2
	Cyproconazole ^{a)} [α -(4-chlorophenyl)- α -(1-cyclopropylethyl)-1H-1,2,4-triazole-1-ethanol]	0.5 to 0.7	8.3.6 and 8.4.4.1 or 8.4.4.2
	Ethofenprox [2-(4-ethoxy phenyl)-2-methyl propyl-3-plienoxybenzyl ether]	2.2 to 2.6	8.3.6 and 8.4.7.1
Total content of active substances ^{b)} (mass fraction %)		40 min.	8.5.6 b)
State of product		Liquid state	8.6
Water insoluble content (mass fraction %)		1 max.	8.7
pH		5.0 to 8.0	8.8
Notes ^{a)} For the compound containing cyproconazole, there is the following patent: Patent registration: No. 3216946 "Microbicidal composition" Date of registration: August 3, 2001 ^{b)} The total content of active substances means the total amount of DMPAP, cyproconazole and ethofenprox.			

Table 8 Quality of wood preservatives of azole/quaternary ammonium/neonicotinoid compound series ^{a)}

Item		Quality	Sub-clause of relevant test
		AZNA	
Compound ratio of active substances (mass fraction %)	DDAC	95.0 to 97.5	8.3.7 and 8.4.1.1 or 8.4.1.2
	Tebuconazole [(α -(2-(4-chlorophenyl) ethyl) - α -(1,1-dimethyl ethyl)-1H-1,2,4-triazole-1-ethanol]	2.0 to 4.0	8.3.7 and 8.4.8.1
	Imidacloprid [1-[(6-chloro-3-pyridinyl) methyl] -4.5-dihydro-N-nitro-1H-imidazol-2-amine]	0.5 to 1.0	8.3.7 and 8.4.9.1
Total content of active substances ^{b)} (mass fraction %)		23 min.	8.5.7 b)
State of product		Liquid state	8.6
Water insoluble content (mass fraction %)		1 max.	8.7
pH		4.0 to 7.0	8.8
Notes ^{a)} For this wood preservative, there is the following patent: Patent registration: No. 4135979 "Water-based, solvent- and emulsifier-free microbicidal active compound combination" Date of registration: June 13, 2008 ^{b)} The total content of active substances means the total amount of DDAC, tebuconazole and imidacloprid.			

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

Item		Quality			Sub-clause of relevant test
		No. 1 (NCU-E)	No. 2 (NZN-E)	No. 3 (VZN-E)	
Content of active substances (mass fraction %)	Copper naphthenate (as Cu)	6.0 to 6.6	—	—	8.3.8 and 8.4.10.1
	Zinc naphthenate (as Zn)	—	8.0 to 8.8	—	8.3.8 and 8.4.11.1
	Tertiary zinc carboxylate (as Zn)	—	—	5.0 to 5.5	8.3.8 and 8.4.11.1
	Permethrin [3-phenoxy benzyl=2-(2,2-dichloro vinyl)-3,3-dimethyl-1-cyclopropane carboxylate]	—	—	0.50 to 0.55	8.3.8 and 8.4.12.1
Acid value of fatty acid ^{a)}		180 to 300		318 to 328	8.9
State of product		Liquid state			8.6
pH		6.0 to 7.0			8.8
Note ^{a)} The fatty acid means the carboxylic acid of terpene series.					

Table 10 Quality of wood preservatives of metallic salts of naphthenic acid series

Item		Quality		Sub-clause of relevant test
		No. 1 (NCU-O)	No. 2 (NZN-O)	
Content of active substances (mass fraction %)	Copper naphthenate (as Cu)	5.0 to 11.0	—	8.3.9 and 8.4.13.1
	Zinc naphthenate (as Zn)	—	5.0 to 11.0	8.3.9 and 8.4.14.1
Acid value of fatty acid		180 to 300		8.9
State of product		Liquid state		8.6
pH		—		—

Table 11 Quality of wood preservatives of azole/neonicotinoid compound series

Item		Quality	Sub-clause of relevant test
		AZN	
Compound ratio of active substances (mass fraction %)	Cyproconazole ^{a)} [α -(4-chlorophenyl)- α -(1-cyclopropylethyl)-1H-1,2,4-triazole-1-ethanol]	62 to 72	8.3.10 and 8.4.4.2
	Imidacloprid [1-[(6-chloro-3-pyridinyl) methyl] -4.5-dihydro-N-nitro-1H-imidazol-2-amine]	28 to 38	8.3.10 and 8.4.9.1
Total content of active substances ^{b)} (mass fraction %)		3.6 min.	8.5.8 b)
State of product		Liquid state	8.6
pH		—	—
Notes ^{a)} For the compound containing cyproconazole, there is the following patent: Patent registration: No. 3216946 “Microbicidal composition” Date of registration: August 3, 2001 ^{b)} The total content of active substances means the total amount of cyproconazole and imidacloprid.			

Table 12 Quality of creosote oil wood preservatives

Item		Quality	Sub-clause of relevant test
		A	
Density (g/cm ³ , 40 °C)		1.01 min.	8.4.15.1.1 or 8.4.15.1.2
Moisture content (volume fraction %)		3 max.	8.4.15.2
Engler degree ^{a)} (40/20 °C)		2.0 max.	8.4.15.3
Distillation test (dehydrated sample) (volume fraction %)	Percent recovered up to 235 °C	10 max.	8.4.15.4
	Percent recovered between 235 °C and 255 °C	60 min.	8.4.15.4
	Percent recovered between 255 °C and 270 °C	20 max.	8.4.15.4
	Percent recovered between 270 °C and 315 °C	5 min.	8.4.15.4
	Percent recovered up to 315 °C	85 min.	8.4.15.4
	Density of fraction between 235 °C and 315 °C (g/cm ³ , 40 °C)	1.02 min.	8.4.15.5.1 or 8.4.15.5.2
Toluene insoluble content (dehydrated sample) (mass fraction %)		0.5 max.	8.4.15.6
Fluidity test		No noticeable solid matter shall be produced.	8.4.15.7
Content of dibenzo [a, h] anthracene (mg/kg)		10 max.	8.4.15.8
Content of benzo [a] anthracene (mg/kg)		10 max.	8.4.15.9
Content of benzo [a] pyrene (mg/kg)		10 max.	8.4.15.10
Total content of active substances (mass fraction %)		Not specified	—
Note ^{a)} Engler degree means the relative viscosity to water.			

7 Performance

The antiseptic performance, termite proofing performance and iron corrosion performance of wood preservatives shall conform to the performance requirements specified in clause 4 of **JIS K 1571** when wood preservatives are tested at the respectively specified concentration according to **5.2.1.1**, **5.2.2.1** or **5.2.2.2**, **5.2.3**, **5.3.1.1**, **5.3.2.1** and **5.4.1** of **JIS K 1571**.

8 Test methods of quality

8.1 General matters

The general matters shall be as follows:

- a) The matters common to methods of chemical analysis shall be in accordance with **JIS K 0050**.
- b) The method of rounding off of numerical values shall be in accordance with **JIS Z 8401**.
- c) Atomic absorption spectrometry shall be in accordance with **JIS K 0121**.
- d) Molecular absorptiometric analysis shall be in accordance with **JIS K 0115**.
- e) ICP atomic emission spectrometry shall be in accordance with **JIS K 0116**.
- f) High performance liquid chromatography shall be in accordance with **JIS K 0124**.
- g) Gas chromatographic analysis shall be in accordance with **JIS K 0114**.
- h) The glass instruments to be used for test shall be in accordance with **JIS R 3503**.
- i) The glass volumenometer to be used for test shall be in accordance with **JIS R 3505**.

8.2 Sampling method

The sample representing the lot shall be sampled from the stock of products or the sampling nozzle, using an instrument suitable for each sample.

8.3 Preparation of test solution

8.3.1 Quaternary ammonium compound series (AAC-1, AAC-2)

Weigh out about 10 g of the sample taken in **8.2** to the nearest 0.1 mg, put it in a 1 000 ml volumetric flask, make up to the mark with distilled water and take it as the test solution.

8.3.2 Copper/quaternary ammonium compound series (ACQ-1, ACQ-2)

Weigh out about 0.8 g of the sample taken in **8.2** to the nearest 0.1 mg, put it in a 250 ml volumetric flask, make up to the mark with distilled water and take it as the test solution.

8.3.3 Copper/azole compound series (CUAZ)

Weigh out about 17 g of the sample taken in **8.2** to the nearest 0.1 mg, put it in a 500 ml volumetric flask, make up to the mark with distilled water and take it as the test solution.

8.3.4 Boron/quaternary ammonium compound series (BAAC)

Weigh out about 13.3 g of the sample taken in **8.2** to the nearest 0.1 mg, put it in a 1 000 ml volumetric flask, make up to the mark with distilled water and take it as the test solution.

8.3.5 Quaternary ammonium/non-ester pyrethroid compound series (SAAC)

Weigh out about 10 g of the sample taken in **8.2** to the nearest 0.1 mg, put it in a 1 000 ml volumetric flask, make up to the mark with distilled water and take it as the test solution.

8.3.6 Azole/quaternary ammonium/non-ester pyrethroid compound series (AZAAC)

Weigh out about 10 g of the sample taken in **8.2** to the nearest 0.1 mg, put it in a 1 000 ml volumetric flask, make up to the mark with distilled water and take it as the test solution.

8.3.7 Azole/quaternary ammonium/neonicotinoid compound series (AZNA)

Weigh out about 4 g of the sample taken in **8.2** to the nearest 0.1 mg, put it in a 100 ml volumetric flask, make up to the mark with distilled water and take it as the test solution.

8.3.8 Metallic salts of fatty acid series (NCU-E, NZN-E, VZN-E)

Weigh out about 1 g of the sample taken in **8.2** to the nearest 0.1 mg, put it in a 100 ml volumetric flask, make up to the mark with distilled water and take it as the test solution. In the case of permethrin of VZN-E, the sample taken in **8.2** shall be used as it is.

8.3.9 Metallic salts of naphthenic acid series (NCU-O, NZN-O)

Weigh out about 0.5 g of the sample taken in **8.2** 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 to cool, transfer it to a 500 ml volumetric flask by filtering it through a filter paper, wash sufficiently the tall beaker and the watch glass with distilled water, and add the washings to the volumetric flask. Make up to the mark with distilled water and take this solution as the test solution.

8.3.10 Azole/neonicotinoid compound series (AZN)

Weigh out about 1.5 g of the sample taken in **8.2** to the nearest 0.1 mg in a 50 ml eggplant-shaped 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 by washing the concentrated solution and the inside wall of eggplant type flask with a small amount of ethanol specified in **JIS K 8101**, make up to the mark with ethanol specified in **JIS K 8101**, and take this solution as the test solution.

8.3.11 Creosote oil (A)

Weigh out about 200 ml of the sample taken in **8.2** to the nearest 0.1 mg, and heat it to about 50 °C. Add about 60 g of the calcium chloride specified in **JIS K 8125** or about 100 g of salt having been dried at about 120 °C for 1 h or longer as a desiccant, mix them together sufficiently, then keep it at 50 °C for about 10 min. Allow it to cool, then filter it and take it as a dewatering test sample.

8.4 Analytical method for individual ingredients

8.4.1 Didecyl dimethyl ammonium chloride (DDAC) and N,N-didecyl-N-methyl-polyoxyethyl ammonium propionate (DMPAP)

8.4.1.1 Titration method

- a) **Summary** DDAC and DMPAP, being a cation, is titrated with anion solution while being agitated in water-chloroform system by using bromphenol blue (hereafter referred to as “BPB”) as an indicator.
- b) **Reagents** The reagents shall be as follows:
 - 1) **Anion solution (0.002 mol/L)**, prepared by weighing out 0.684 5 g of sodium tetraphenyl borate and dissolving in distilled water to make 1 000 ml.
 - 2) **Buffer solution (pH 10)**, prepared by dissolving 100 g of sodium carbonate specified in **JIS K 8625** and 100 g of sodium sulfate specified in **JIS K 8987** in distilled water to make 1 000 ml.
 - 3) **BPB solution (1 g/L)**, prepared by dissolving 0.1 g of BPB specified in **JIS K 8844** in distilled water to make 100 ml.
 - 4) **Chloroform**, specified in **JIS K 8322**.
- c) **Operation** Weigh out 2 ml of the test solution by using a volumetric pipette, and pour it into 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 after stoppering. Add anion solution (0.002 mol/L) little by little repeatedly by using a microburette while agitating after each addition, and confirm the change of the colour of the solution. Take the point where the blue colour of the chloroform layer vanishes and the water layer colour turns faint purple as the end point of titration.
- d) **Calculation** Calculate the amount of DDAC and DMPAP of each chemical obtained in c) and round the result to two significant figures.
 - 1) **AAC-1** Calculate the mass and content according to formula (1) and formula (2):

$$A_1 = \frac{t \times f \times 0.724 \times 1\,000}{1\,000 \times 2} \dots\dots\dots (1)$$

where, A_1 : mass of DDAC in the sample weighed out in **8.3.1** (g)

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

f : factor of 0.002 mol/L anion solution

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

$$A = \frac{A_1}{S} \times 100 \dots\dots\dots (2)$$

where, A : content of DDAC in the sample weighed out in **8.3.1** (mass fraction %)

S : mass of sample weighed out in **8.3.1** (g)

- 2) **AAC-2** Calculate the mass and content according to formula (3) and formula (4):

$$A_1 = \frac{t \times f \times 0.908 \times 1\,000}{1\,000 \times 2} \dots\dots\dots (3)$$

where, A_1 : mass of DMPAP in the sample weighed out in **8.3.1** (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)

$$A = \frac{A_1}{S} \times 100 \dots\dots\dots (4)$$

where, A : content of DMPAP in the sample weighed out in **8.3.1** (mass fraction %)

S : mass of sample weighed out in **8.3.1** (g)

- 3) **ACQ-2** Calculate the mass according to formula (5):

$$D = \frac{t \times f \times 0.724 \times 250}{1\,000 \times 2} \dots\dots\dots (5)$$

where, D : mass of DDAC in the sample weighed out in **8.3.2** (g)

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

f : factor of 0.002 mol/L anion solution

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

- 4) **BAAC** Calculate the mass according to formula (6):

$$D = \frac{t \times f \times 0.724 \times 1\,000}{1\,000 \times 2} \dots\dots\dots (6)$$

where, D : mass of DDAC in the sample weighed out in **8.3.4** (g)

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

f : factor of 0.002 mol/L anion solution

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

5) **SAAC** Calculate the mass according to formula (7):

$$D = \frac{t \times f \times 0.908 \times 1\,000}{1\,000 \times 2} \dots\dots\dots (7)$$

where, D : mass of DMPAP in the sample weighed out in **8.3.5** (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)

6) **AZAAC** Calculate the mass according to formula (8):

$$A_1 = \frac{t \times f \times 0.908 \times 1\,000}{1\,000} \dots\dots\dots (8)$$

where, A_1 : mass of DMPAP in the sample weighed out in **8.3.6** (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) **AZNA** Calculate the mass according to formula (9):

$$D_1 = \frac{t \times f \times 0.724 \times 100}{1\,000} \dots\dots\dots (9)$$

where, D_1 : mass of DDAC in the sample weighed out in **8.3.7** (g)

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

f : factor of 0.002 mol/L anion solution

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

8.4.1.2 High performance liquid chromatography

a) **Summary** The sample is electrically neutralized by an ion pair indicator and determination is performed by the high performance liquid chromatography with UV absorptiometric detection.

- b) **Reagents** The reagents shall be as follows:
- 1) **DDAC and DMPAP**, refined for determination and of previously known purity.
 - 2) **Sodium 2-naphthalene sulfonate solution**, prepared by weighing out 575 mg of sodium 2-naphthalene sulfonate, putting it in a 100 ml volumetric flask and making up to the mark with distilled water.
 - 3) **Phosphoric acid solution**, prepared by placing 17 ml of phosphoric acid specified in **JIS K 9005** in a 100 ml volumetric flask and making up to the mark with distilled water.
 - 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** to put it 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 make up to the mark with distilled water. Mix this solution and methanol at the ratio of 1:4 in volume percentage and take it as the mobile phase.
 - 6) **Standard solution for working curve**, prepared by weighing out approximately 50 mg of DDAC and DMPAP to the nearest 0.1 mg to put it in a 100 ml volumetric flask and making up to the mark with the mobile phase prepared in 5).
- c) **Apparatus** For the apparatus, a high performance liquid chromatograph shall be used.
- d) **Analytical conditions of high performance liquid chromatography** The analytical conditions of high performance liquid chromatography shall be in accordance with table 13.

Table 13 Analytical conditions of high performance liquid chromatography

Item	Condition
Column	ODS system column (I.D 4.6 mm × L 150 mm)
Mobile phase	In accordance with 8.4.1.2 b) 5) .
Mobile phase flow rate	1.0 ml/min
Column temperature	40 °C
Measurement wavelength	275 nm
Injection amount	10 µl

- e) **Operation** Weigh out 2 ml of the test solution by using a volumetric pipette, pour it into a 100 ml volumetric flask, and make up to the mark with mobile phase and take it as the analytical test solution. Inject 10 µl of the analytical test solution into high performance liquid chromatograph, and obtain the peak area of DDAC and DMPAP. Obtain the DDAC and DMPAP concentrations in the analytical test solution from the working curve.

- f) **Preparation of working curve** By diluting the standard solution for working curve with mobile phase, prepare solutions of which the DDAC and DMPAP concentrations are increased stepwise from 50 mg/L to 150 mg/L. Perform the same operation as above on these solutions and prepare at least three working curves of DDAC and DMPAP concentration against peak area. Working curves shall be prepared each time before use.
- g) **Calculation** Calculate the amount of DDAC and DMPAP obtained in e), for each chemical, and round the result to two significant figures.
- 1) **AAC-1 and AAC-2** Calculate the mass and content according to formula (10) and formula (11):

$$D_1 = D_0 \times \frac{100 \times 1\,000}{1\,000\,000 \times 2} \dots\dots\dots (10)$$

where, D_1 : mass of DDAC and DMPAP in the sample weighed out in **8.3.1** (g)

D_0 : concentration of DDAC and DMPAP in the analytical test solution obtained from working curve (mg/L)

$$D = \frac{D_1}{W} \times 100 \dots\dots\dots (11)$$

where, D : content of DDAC and DMPAP in the sample weighed out in **8.3.1** (mass fraction %)

W : mass of the sample weighed out in **8.3.1** (g)

- 2) **SAAC** Calculate the mass according to formula (12):

$$M = M_0 \times \frac{100 \times 1}{1\,000} \dots\dots\dots (12)$$

where, M : mass of DMPAP in the sample weighed out in **8.3.5** (g)

M_0 : concentration of DMPAP in the test solution obtained from working curve (mg/L)

- 3) **AZAAC** Calculate the mass according to formula (13):

$$A_1 = \frac{A \times 100 \times 1\,000}{1\,000\,000} \dots\dots\dots (13)$$

where, A_1 : mass of DMPAP in the sample weighed out in **8.3.6** (g)

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

- 4) **AZNA** Calculate the mass according to formula (14):

$$D_1 = \frac{d \times 100 \times 100}{1\,000\,000} \dots\dots\dots (14)$$

where, D_1 : mass of DDAC in the sample weighed out in **8.3.7** (g)

d : concentration of DDAC in the analytical test solution (mg/L)

8.4.2 Copper compound (as CuO)

8.4.2.1 Atomic absorption spectrometry

- a) **Summary** Copper is determined by the use of an atomic absorption spectrometer.
- 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 as follows: Dissolve copper (reagent) specified in **JIS K 8660**, electrolytic cathode copper specified in **JIS H 2121** or copper (II) sulfate pentahydrate specified in **JIS K 8983** in nitric acid of a suitable concentration specified in **JIS K 8541** to prepare a copper standard solution which is 0.05 mol/L to 0.15 mol/L in the final concentration of nitric acid (1 000 mg/L as Cu). Weigh out 10 ml of this solution by using a volumetric pipette to put it in a 100 ml flask and make up to the mark with distilled water.
- c) **Apparatus** For the apparatus, an atomic absorption spectrometer shall be used.
- d) **Operation** Weigh out 2 ml of ACQ test solution, and 1 ml of CUAZ test solution by using a volumetric pipette, pour it into a 1 000 ml volumetric flask, add 8 ml of sulfuric acid, then make up to the mark with distilled water and take it as the analytical test solution. Spray the analytical test solution into the flame according to clause 8 of **JIS K 0121**, read the absorbance at 324.8 nm in wavelength, obtain the concentrations of copper in the analytical test solution from the working curve.
- e) **Preparation of working curve** Weigh out aliquots of the copper standard solution for working curve (100 mg/L as Cu) in amounts increased stepwise from 0 ml to 5 ml by using a volumetric pipette to pour it in as many 100 ml volumetric flasks, add sulfuric acid so that they are in the same condition as the analytical test solution, and make up to the mark with distilled water. Perform the same operation as above on these solutions and prepare at least three working curves of copper quantity against absorbance. Working curves shall be prepared each time before use.
- f) **Formula** Calculate the amount of copper compound obtained in **d)** of each chemical and round the result to two significant figures.
 - 1) **ACQ** Calculate the mass according to formula (15):

$$C = \frac{c \times 1.252 \times 250 \times 1\,000}{1\,000\,000 \times 2} \dots\dots\dots (15)$$

where, C : mass of copper compound in the sample weighed out in **8.3.2** (as CuO) (g)

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