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**Test methods for resin finished
textiles**

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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 Textile Evaluation Technology Council (JTETC)/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 L 1041**:2000 is replaced with this Standard.

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Attention is drawn to the possibility that some parts of this Standard may conflict with patent rights, applications for a patent after opening to the public or utility model rights. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying any of such patent rights, applications for a patent after opening to the public or utility model rights.

Test methods for resin finished textiles

Introduction

This Japanese Industrial Standard was established in 1960 to specify the test methods for resin finished textiles. In the revision of this time, this Standard has been revised in a part of technical contents, and conformed to the revisions issued by the Ministerial Ordinance of Ministry of Health Labour and Welfare No. 104 (2004) and the Ministerial Ordinance of Ministry of Health Labour and Welfare No. 46 (2009), and also accommodated to the first editions of **ISO 14184-1** and **ISO 14184-2** published in 1998. In addition, the test method to discriminate whether the free formaldehyde is from resin or not newly prepared as Annex JE.

The **JIS** methods of the resin qualitative test (clause **6**), resin quantitative test (clause **7**) and free formaldehyde test (clause **8**) in this Standard are not given in the corresponding International Standards. A list of modifications with the explanations is given in Annex JF. Also Annex JA to Annex JE are the matters not given in the corresponding International Standards.

1 Scope

This Standard specifies the analysis and determination methods for resin finished textiles (including lace), and the determination method of free formaldehyde. However, when there are test methods separately specified in Japanese Industrial Standards for the colour fastness test and the physical test, the tests shall be carried out according to their test methods. The test when the fabric of resin finished textiles is damaged by chlorine is shown in Annex JD.

WARNING : This Standard calls for the use of substances¹⁾ and/or procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage. It has been assumed in the drafting of this Standard that the execution of its provisions is entrusted to appropriately qualified and experienced people.

Note ¹⁾ Information on substances which may damage the health are described in the Material Safety Data Sheet (MSDS) in detail.

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

ISO 14184-1:1998 *Textiles—Determination of formaldehyde—Part 1: Free and hydrolized formaldehyde (water extraction method)*

ISO 14184-2:1998 *Textiles—Determination of formaldehyde—Part 2: Released formaldehyde (vapour absorption method)* (overall evaluation: MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standards and **JIS** are

IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. For standards with the year indication, only the editions of the indicated year shall be applied and the revisions (including amendments) made thereafter shall not be applied. For those without the indication of the year, the most recent edition (including amendments) shall be applied.

- JIS K 0064 *Test methods for melting point and melting range of chemical products*
- JIS K 0117 *General rules for infrared spectrophotometric analysis*
- JIS K 3302 *Laundry bar soaps*
- JIS K 8001 *General rule for test methods of reagents*
- JIS K 8027 *Acetylacetone (Reagent)*
- JIS K 8101 *Ethanol (99.5) (Reagent)*
- JIS K 8180 *Hydrochloric acid (Reagent)*
- JIS K 8322 *Chloroform (Reagent)*
- JIS K 8355 *Acetic acid (Reagent)*
- JIS K 8359 *Ammonium acetate (Reagent)*
- JIS K 8625 *Sodium carbonate (Reagent)*
- JIS K 8848 *Hexane (Reagent)*
- JIS K 8858 *Benzene (Reagent)*
- JIS K 8872 *Formaldehyde solution (Reagent)*
- JIS K 8891 *Methanol (Reagent)*
- JIS L 0105 *General principles of physical testing methods for textiles*
 - NOTE : Corresponding International Standard: ISO 139:2005 *Textiles—Standard atmospheres for conditioning and testing* (MOD)
- JIS L 0208 *Glossary of terms used in textile industry—Testing*
- JIS P 3801 *Filter paper (for chemical analysis)*
- JIS R 3503 *Glass apparatus for chemical analysis*
- JIS Z 8401 *Guide to the rounding of numbers*
- ISO 3696:1987 *Water for analytical laboratory use—Specification and test methods*
- ISO 4793:1980 *Laboratory sintered (fritted) filters—Porosity grading, classification and designation*

3 Terms and definitions

For the purposes of this Standard, the terms and definitions given in **JIS L 0105** and **JIS L 0208** apply.

4 Types of test

The types of test are as follows.

- a) Resin qualitative test
- b) Resin quantitative test
- c) Free formaldehyde test
 - 1) **JIS method**
 - 1.1) **Method A (2.5 g method)** Mainly applicable to tests for textile products for infants within 24 months after the birth
 - 1.2) **Method B (1 g method)** Mainly applicable to tests for other textile products than those for Method A
 - 2) **ISO method**
 - 2.1) Water extraction method: Applicable to tests for all textile products (see Annex A)
 - 2.2) Gas absorption method: Applicable to tests for all textile products (see Annex B)
- d) Phloroglucinol method: Applicable to tests for all textile products (see Annex JB)
- e) Plottable jar method: Applicable to tests for all textile products (see Annex JC)
- f) Chlorine damage test method: Applicable to tests related to deterioration of fabric due to chlorine damage (see Annex JD)
- g) Hydrochloric acid extraction discrimination method: Applicable to the determination whether the detected free formaldehyde is from resin or by migration (see Annex JE)

5 Sampling and preparation of sample and test specimen

The sampling and preparation of sample and test specimen shall be according to clause 6 of **JIS L 0105**. However, the standard condition is not required to be applied to the resin qualitative test and the resin quantitative test because they are not influenced by temperature and humidity.

When using **ISO** method, Annex A or Annex B shall be referred to.

6 Resin qualitative test

6.1 Applicable resins

This test is applicable to resins of melamine series, urea series, epoxy series, acrylate series, methacrylate series, polyamide series, silicon series, polystyrene series, polyethylene series, polyvinyl acetate series, polyurethane series and fluorine series.

6.2 Test method

The test method is as follows.

a) **Extraction**

- 1) **Operation** Take about 5 g of test specimen from the sample, put this test specimen in a round bottom flask of 200 ml in capacity with a reflux condenser attached, and add 150 ml of extraction solvent to it. Heat it up to the boiling point of extraction solvent, carry out the extraction for 1 h and filter it with the glass filter using filter of pore size symbol 2 specified in **JIS R 3503** while it is warm. Furthermore, put it in an eggplant type flask of 200 ml in capacity and concentrate it down to 2 ml to 3 ml by using a rotary evaporator.
- 2) **Extraction solvent** The extraction solvent shall be benzene specified in **JIS K 8858**, methanol specified in **JIS K 8891** and 0.1 mol/L hydrochloric acid. Repeat the operation of extraction, refining and confirmation in this order until the resin is confirmed.

For the extracted solution by 0.1 mol/L hydrochloric acid, neutralize it with sodium hydroxide solution, then carry out the concentration operation.

- b) **Refining** After glass wool is stuffed in a column tube of 10 mm to 15 mm in inside diameter and 200 mm to 300 mm in length, pack the silica gel of 177 μm to 350 μm for column chromatography mixed turbidly in hexane specified in **JIS K 8848** up to about 150 mm in height. Furthermore, spread a filter paper of Class 6 specified in **JIS P 3801** on the upper end of silica gel. Put the extracted solution into it and drop about 100 ml each of hexane, chloroform specified in **JIS K 8322** and methanol specified in **JIS K 8891** in this order. Receive the outflow solution in an eggplant type flask of 100 ml in capacity for every solvent, and evaporate the solvent by using a rotary evaporator.

NOTE : When the resin cannot be confirmed by the infrared absorption spectrum measured for every solvent because of the mixed matters, the operations from the extraction are carried out again and the mixed matters should be separated by taking an aliquot of the same solvent finely in refining.

- c) **Confirmation** Measure the infrared absorption spectrum of the purified extractive according to the method specified in **JIS K 0117**, compare it with the infrared absorption spectrum diagram of the reference resin (see figure 1) prepared beforehand, and identify it.

Moreover, for the extractive which is not dissolved in the developing solvent and left on the filter paper, recover and dry it sufficiently, then measure the infrared absorption spectrum according to the method specified in **JIS K 0117**. An example of combination of resin with extraction solvent is shown in table 1.

Table 1 Example of combination between resin and extraction solvent (informative)

Extraction solvent	Resin															
	Melamine series	Urea series														
		Methylol series	Urea-melamine series	Glyoxal series	Uron series	Ethylene-urea series										
Benzene	×	×	×	×	×	×	○	○	○	○	○	○	○	○	○	○
Methanol	△	×	×	○	○	○	○	△	×	△	△	×	×	○	△	×
0.1 mol/L hydrochloric acid	○	○	○	○	○	○	×	×	×	×	×	×	×	×	×	×
○: capable of qualitative test by extraction																
△: capable of qualitative test by extraction depending on type of fibre																
×: not capable of qualitative test by extraction																

7 Resin quantitative test

7.1 Applicable resins

This test is applicable to the condensation resin finished textiles such as melamine series and urea series.

7.2 Test method

The test method is as follows.

- a) **Sampling** Two pieces of test specimen of about 1 g shall be taken from the sample.
- b) **Operation**
 - 1) Measure the absolute dry mass (m_0) of test specimen.
 - 2) Wash it with water at $25\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 10 min carefully so as not to unravel the cut edge, then dry it and measure the absolute dry mass (m_1).
 - 3) Treat this test specimen in aqueous solution (bath ratio 1:100) at $90\text{ }^{\circ}\text{C}$ containing 0.25 % soap of Class 1 specified in **JIS K 3302** and 0.25 % sodium carbonate specified in **JIS K 8625** for 5 min, wash it with hot water at $90\text{ }^{\circ}\text{C}$ sufficiently several times, then dry it and measure the absolute dry mass (m_2).
 - 4) Treat this cloth in 0.25 % hydrochloric acid (bath ratio 1:100) at $90\text{ }^{\circ}\text{C}$ for 30 min, which is prepared by dissolving 7 g of hydrochloric acid specified in **JIS K 8180** in water to make 1 L.
 - 5) Next, wash it with hot water at $90\text{ }^{\circ}\text{C}$ several times, wash it sufficiently, then dry it and measure the absolute dry mass (m_3).

- c) **Calculation** The amount of resin shall be calculated according to equation (1) to equation (4), and the mean value shall be rounded to one decimal place according to Rule B of **JIS Z 8401**.

$$M_1 = \frac{m_0 - m_1}{m_3} \times 100 \dots\dots\dots (1)$$

$$M_2 = \frac{m_1 - m_2}{m_3} \times 100 \dots\dots\dots (2)$$

$$M_3 = \frac{m_2 - m_3}{m_3} \times 100 \dots\dots\dots (3)$$

$$M = M_1 + M_2 + M_3 \dots\dots\dots (4)$$

where, ***M*** : total amount of resin (%)
 ***M*₁** : amount of resin washed out with water (%)
 ***M*₂** : amount of resin washed out with soap (%)
 ***M*₃** : fixed amount of resin (%)
 ***m*₀** : absolute dry mass of untreated test specimen (g)
 ***m*₁** : absolute dry mass after wash with water for 10 min (g)
 ***m*₂** : absolute dry mass after soap treatment (g)
 ***m*₃** : absolute dry mass after hydrochloric acid treatment (g)

8 Free formaldehyde test

8.1 JIS method

The **JIS** method shall be according to Method A (2.5 g method) or Method B (1 g method).

8.1.1 Preparation and storage of sample (common to Method A and Method B)

The preliminary drying and conditioning related to the preparation of sample shall not be carried out because it may cause the change in the elution amount of formaldehyde in the sample. The sample shall be put in a polyethylene bag, etc. sealed hermetically and stored until the test is carried out.

8.1.2 Preparation of reagents and standard solution

The preparation of reagents and standard solution shall be as follows.

- a) **Water** That of purified water of the Japanese Pharmacopeia or at least equal to it in quality
- b) **Acetylacetone reagent solution** For the preparation of acetylacetone reagent solution, dissolve 150 g of ammonium acetate specified in **JIS K 8359** in about 800 ml of water, add 3 ml of acetic acid specified in **JIS K 8355** and 2 ml of acetylacetone specified in **JIS K 8027** to it, shake to mix them well, then make it 1 000 ml with water. This reagent solution shall be prepared at every service.

- c) **Buffer solution** For the preparation of buffer solution, dissolve 150 g of ammonium acetate specified in **JIS K 8359** in about 800 ml of water, add 3 ml of acetic acid specified in **JIS K 8355** to it, shake to mix it well, then make it 1 000 ml with water.
- d) **Preparation of formaldehyde reference solution** The preparation of formaldehyde reference solution shall be as follows.

- 1) **Standardization of formaldehyde solution** Weigh 1 g of formaldehyde solution specified in **JIS K 8872** correctly and make it 100 ml with water. Take 10 ml aliquot of this solution, add 50 ml of 0.05 mol/L iodine solution, further add 20 ml of 1 mol/L potassium hydroxide solution, then leave it as it is at room temperature for 15 min. Add 15 ml of dilute sulfuric acid (reagent of the Japanese Pharmacopeia) to it, and titrate the excess iodine with 0.1 mol/L sodium thiosulfate solution by using starch reagent (the Japanese Pharmacopeia) as an indicator. In addition, carry out the blank test by using 10 ml of water.

The preparation and standardization of 0.05 mol/L iodine solution, 1 mol/L potassium hydroxide solution, 0.1 mol/L sodium thiosulfate solution shall be according to **JIS K 8001** or the reference solution for quantitative analysis of the Japanese Pharmacopeia shall be used.

The content rate of formaldehyde shall be obtained according to equation (5).

$$C_p = 1.5013 \times \frac{(V_1 - V_2) \times f}{1000} \times \frac{100}{10} \times \frac{1}{m} \times 100$$

$$= 1.5013 \times (V_1 - V_2) \times f \times \frac{1}{m} \dots\dots\dots (5)$$

where, C_p : content rate of formaldehyde in formaldehyde solution (%)

V_1 : titrated amount of 0.1 mol/L sodium thiosulfate solution used in blank test (ml)

V_2 : titrated amount of 0.1 mol/L sodium thiosulfate solution used in this test (ml)

f : titre of 0.1 mol/L sodium thiosulfate solution

m : amount of formaldehyde solution taken (g)

1.501 3 : mass of formaldehyde corresponding to 1 ml of 0.05 mol/L iodine solution (mg)

- 2) **Formaldehyde reference solution A** Weigh $400/C_p$ g of the formaldehyde solution standardized in **8.1.2 d) 1)** correctly and make it 100 ml accurately with water. Take 10 ml aliquot of this solution, repeat the operation to dilute to 10 times the amount with water five times, and make it the formaldehyde reference solution A.

NOTE : In 1 ml of this formaldehyde solution, 0.4 µg of formaldehyde is contained.

- 3) **Formaldehyde reference solution B** Weigh $400/C_p$ g of the formaldehyde solution standardized in **8.1.2 d) 1)** correctly and make it 100 ml accurately by adding water. Take 10 ml aliquot of this solution, repeat the operation to dilute to 10 times the amount with water four times, and make it the formaldehyde reference solution B.

NOTE : In 1 ml of this formaldehyde solution, $4.0\ \mu\text{g}$ of formaldehyde is contained.

- e) **Dimedone ethanol solution** For the preparation of 5,5-dimethyl-1,3-cyclohexane dione (dimedone) ethanol solution, add 1 g of 5,5-dimethyl-1,3-cyclohexane dione (dimedone) specified in Annex JA in ethanol (99.5) specified in **JIS K 8101**, and dissolve it to be 100 ml. This reagent shall be prepared at every service.

8.1.3 Method A (2.5 g method)

Method A (2.5 g method) shall be as follows.

- a) **Sampling and preparation of sample** Cut the sample finely, take its 2.50 g, put it in a flask with grounding stopper of 200 ml in capacity, add 100 ml of water measured accurately and stopper it hermetically, and impregnate water into the sample by shaking to mix.
- b) **Extraction** Extract this in water bath²⁾ at $40\ ^\circ\text{C} \pm 2\ ^\circ\text{C}$ for 1 h as shaking to mix sometimes, filter it with the glass filter using filter of pore size symbol 2 specified in **JIS R 3503** while it is warm, and take it as the extracted solution (E_A).

Note ²⁾ There may be a case where a stopper is detached after heating is started, therefore the stopper should be loosened one time to release the expanded air.

c) Colour development and measurement

1) Preparation of solutions for measurement

- 1.1) **Sample solution** (S) Take 5.0 ml of extracted solution (E_A) in a test tube with grounding stopper, add 5.0 ml of acetylacetone reagent solution prepared according to **8.1.2 b)**, stopper it hermetically and shake to mix it beforehand.
- 1.2) **Test control solution** (B) Take 5.0 ml of water in a test tube with grounding stopper, add 5.0 ml of acetylacetone reagent solution prepared according to **8.1.2 b)**, stopper it hermetically and shake to mix it beforehand.
- 1.3) **Correction solution** (S_0) Take 5.0 ml of extracted solution (E_A) in a test tube with grounding stopper, add 5.0 ml of buffer solution prepared according to **8.1.2 c)**, stopper it hermetically and shake to mix it beforehand.
- 1.4) **Correction control solution** (B_0) Take 5.0 ml of water in a test tube with grounding stopper, add 5.0 ml of buffer solution prepared according to **8.1.2 c)**, stopper it hermetically and shake to mix it beforehand.
- 1.5) **Standard test solution** (F_A) Take 5.0 ml of the formaldehyde reference solution A prepared according to **8.1.2 d) 2)** in a test tube with grounding stopper, add 5.0 ml of acetylacetone reagent solution prepared in **8.1.2 b)**, stopper it hermetically and shake to mix it beforehand.

- 2) **Colour development** Heat these solutions in water bath ²⁾ at $40\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 30 min, take them out from the water bath, then furthermore leave them in room as they are for 30 min.
- 3) **Measurement** Use an absorption cell (10 mm in layer length) in a spectrophotometer, and measure the absorbance at the maximum absorption wavelength between 412 nm and 415 nm as follows.
- 3.1) Measure the standard test solution (F_A) by taking the test control solution (B) as the control, and take it as the absorbance A_A .
- 3.2) Measure the sample solution (S) by taking the test control solution (B) as the control, and take it as the absorbance A .
- 3.3) Measure the correction solution (S_0) by taking the correction control solution (B_0) as the control, and take it as the absorbance A_0 .
- d) **Calculation of absorbance difference and elution amount** The free formaldehyde in the sample shall be calculated as the absorbance difference and the elution amount. The absorbance difference shall be rounded to two decimal places according to equation (6), and the elution amount shall be rounded to an integer according to equation (7) respectively by Rule B of **JIS Z 8401**.

$$A_F = A - A_0 \dots\dots\dots (6)$$

$$C = K_A \times \frac{A - A_0}{A_A} \times 100 \times \frac{1}{m} \dots\dots\dots (7)$$

where, A_F : absorbance difference corresponding to formaldehyde eluted from sample
 C : eluted amount of formaldehyde for 1 g of sample ($\mu\text{g/g}$)
 A : absorbance when extracted solution is operated with acetylacetone reagent solution
 A_0 : absorbance when extracted solution is operated with buffer solution
 A_A : absorbance when formaldehyde reference solution A is operated with acetylacetone reagent solution
 K_A : concentration of formaldehyde reference solution A ($\mu\text{g/ml}$)
 m : mass of sample taken (g)

- e) **Basis for confirmation of formaldehyde** When the value of A_F exceeds 0.05, the confirmation test shall be carried out according to f) 1) or f) 2), and whether the absorption at the wavelength where absorbance A is measured is caused by formaldehyde or not shall be confirmed.

f) **Confirmation test**

1) **Dimedone method**

1.1) **Preparation of solutions for confirmation**

- 1.1.1) **Confirmation solution** Take 5.0 ml of extracted solution (E_A) in a test tube with grounding stopper, add 1.0 ml of 5,5-dimethyl-1,3-cyclohexane dione (dimedone) ethanol solution, stopper it hermetically and shake to mix it beforehand.
- 1.1.2) **Confirmation control solution** Take 5.0 ml of water in a test tube with grounding stopper, add 1.0 ml of 5,5-dimethyl-1,3-cyclohexane dione (dimedone) ethanol solution, stopper it hermetically and shake to mix it beforehand.
- 1.2) **Operation** Heat the confirmation solution and the confirmation control solution in water bath ²⁾ at $40\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 10 min. Then, add 5.0 ml of acetylacetone reagent solution, stopper hermetically and shake to mix them, respectively.
- 1.3) **Colour development** Next, heat them in water bath ²⁾ at $40\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 30 min, take them out from the water bath, then further leave them as they are in room for 30 min.
- 1.4) **Measurement** Use an absorption cell (10 mm in layer length) in a spectrophotometer and measure the absorption spectrum including the wavelength measuring the absorbance A as follows.
- 1.4.1) Measure the absorption spectrum of sample solution (S) by taking the test control solution (B) as the control.
- 1.4.2) Measure the absorption spectrum of confirmation solution of 1.1.1) by taking the confirmation control solution of 1.1.2) as the control.
- 1.5) **Confirmation** Compare the both absorption spectra obtained in 1.4.1) and 1.4.2). When it is found that the peak observed at 412 nm to 415 nm in wavelength disappears or reduces at the absorption spectrum obtained in 1.4.2) from the absorption spectrum in 1.4.1), it can be confirmed that the absorbance A is caused by the absorption of formaldehyde.

2) High performance liquid chromatography

2.1) Preparation of solution for confirmation

- 2.1.1) **Sample solution (S)** Take 5.0 ml of extracted solution (E_A) in a test tube with grounding stopper, add 5.0 ml of acetylacetone reagent solution prepared according to 8.1.2 b), stopper it hermetically and shake to mix it beforehand.
- 2.1.2) **Standard test solution (F_A)** Take 5.0 ml of the formaldehyde reference solution A prepared according to 8.1.2 d) 2), add 5.0 ml of acetylacetone reagent solution prepared according to 8.1.2 b), stopper it hermetically and shake to mix it beforehand.
- 2.2) **Colour development** Heat the sample solution (S) and the standard test solution (F_A) in a water bath ²⁾ at $40\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 30 min, take it out from the water bath, then furthermore leave it as it is in room for 30 min.
- 2.3) **Operation** Take 10 μl of the sample solution (S) and the standard test solution (F_A) after colouring respectively, and carry out the test under the following condition.

- 2.3.1) **Column tube** That of stainless tube of 4.6 mm in inside diameter and 150 mm in length is used.
- 2.3.2) **Column packing material** Octadecylsilyl silica gel of 5 μm in particle size is used.
- 2.3.3) **Column temperature** 35 °C
- 2.3.4) **Detector** Ultraviolet-visible detector
- 2.3.5) **Detection wavelength** 412 nm to 415 nm
- 2.3.6) **Mobile phase** Acetonitrile:purified water (15:85 to 20:80)
- 2.3.7) **Flow rate** 1.0 ml/min
- 2.4) **Confirmation** When the peak having retention time coinciding to the retention time of the peak of formaldehyde-acetylacetone reaction product in the standard test solution (F_A) is present on the chromatogram of sample solution (S), it can be confirmed that the absorbance A is caused by the absorption of formaldehyde.

8.1.4 Method B (1 g method)

Method B (1 g method) shall be as follows.

- a) **Sampling and preparation of sample** Cut the sample finely, take its about 1 g, and weigh its mass correctly to two decimal places. Put it in a flask with grounding stopper of 200 ml in capacity, add 100 ml of water measured accurately and stopper it hermetically, and impregnate water into the sample by shaking to mix.
- b) **Extraction** Extract this in water bath ²⁾ at 40 °C $\pm$ 2 °C for 1 h shaking to mix sometimes, filter it with the glass filter using filter of pore size symbol 2 specified in **JIS R 3503** while it is warm, and take it as the extracted solution (E_B).
- c) **Colour development and measurement**
 - 1) **Preparation of solutions for measurement**
 - 1.1) **Sample solution (S)** Take 5.0 ml of extracted solution (E_B) in a test tube with grounding stopper, add 5.0 ml of acetylacetone reagent solution prepared according to **8.1.2 b)**, stopper it hermetically and shake to mix it beforehand.
 - 1.2) **Test control solution (B)** Take 5.0 ml of water in a test tube with grounding stopper, add 5.0 ml of acetylacetone reagent solution prepared according to **8.1.2 b)**, stopper it hermetically and shake to mix it beforehand.
 - 1.3) **Correction solution (S_0)** Take 5.0 ml of extracted solution (E_B) in a test tube with grounding stopper, add 5.0 ml of buffer solution prepared according to **8.1.2 c)**, stopper it hermetically and shake to mix it beforehand.
 - 1.4) **Correction control solution (B_0)** Take 5.0 ml of water in a test tube with grounding stopper, add 5.0 ml of buffer solution prepared according to **8.1.2 c)**, stopper it hermetically and shake to mix it beforehand.

- 1.5) **Standard test solution (F_B)** Take 5.0 ml of the formaldehyde reference solution B prepared according to **8.1.2 d) 3)** in a test tube with grounding stopper, add 5.0 ml of acetylacetone reagent solution prepared in **8.1.2 b)**, stopper it hermetically and shake to mix it beforehand.
- 2) **Colour development** Heat these solutions in water bath ²⁾ at 40 °C ± 2 °C for 30 min, take them out from the water bath, then furthermore leave them as they are in room for 30 min.
- 3) **Measurement** Use an absorption cell (10 mm in layer length) in a spectrophotometer, and measure the absorbance at the peak absorption wavelength between 412 nm and 415 nm as follows.
- 3.1) Measure the standard test solution (F_B) by taking the test control solution (B) as the control, and take it as the absorbance A_B.
- 3.2) Measure the sample solution (S) by taking the test control solution (B) as the control, and take it as the absorbance A.
- 3.3) Measure the correction solution (S₀) by taking the correction control solution (B₀) as the control, and take it as the absorbance A₀.
- d) **Calculation of elution amount** The free formaldehyde in the sample shall be calculated according to equation (8) and rounded to an integer by Rule B of **JIS Z 8401**.

$$C = K_B \times \frac{A - A_0}{A_B} \times 100 \times \frac{1}{m} \dots\dots\dots (8)$$

where, C : eluted amount of formaldehyde for 1 g of sample (µg/g)

K_B : concentration of formaldehyde reference solution B (µg/ml)

A : absorbance when extracted solution is operated with acetylacetone reagent solution

A₀ : absorbance when extracted solution is operated with buffer solution

A_B : absorbance when formaldehyde reference solution B is operated with acetylacetone reagent solution

m : mass of sample taken (g)

- e) **Basis for confirmation of formaldehyde** When the value of C exceeds 40 µg for 1 g of sample, the confirmation test shall be carried out according to f).

NOTE : 40 µg for 1 g of sample corresponds to 0.05 in absorbance.

- f) **Confirmation test**

- 1) **Dimedone method** It is confirmed that the absorbance at the wavelength where the absorbance A is measured is caused by the absorption of formaldehyde by carrying out the same operation as the dimedone method in **8.1.3 f) 1)** by using 5.0 ml of extracted solution (E_B).

- 2) **High performance liquid chromatography** It is confirmed that the absorbance at the wavelength where the absorbance A is measured is caused by the absorption of formaldehyde by carrying out the same operation as the high performance liquid chromatography in **8.1.3 f) 2)** by using 5.0 ml of extracted solution (E_B) and 5.0 ml of the formaldehyde reference solution B prepared in **8.1.2 d) 3)**.

8.2 ISO method

The free formaldehyde test according to **ISO** method shall be either of the following.

8.2.1 Water extraction method

The water extraction method shall be according to Annex A.

8.2.2 Vapour absorption method

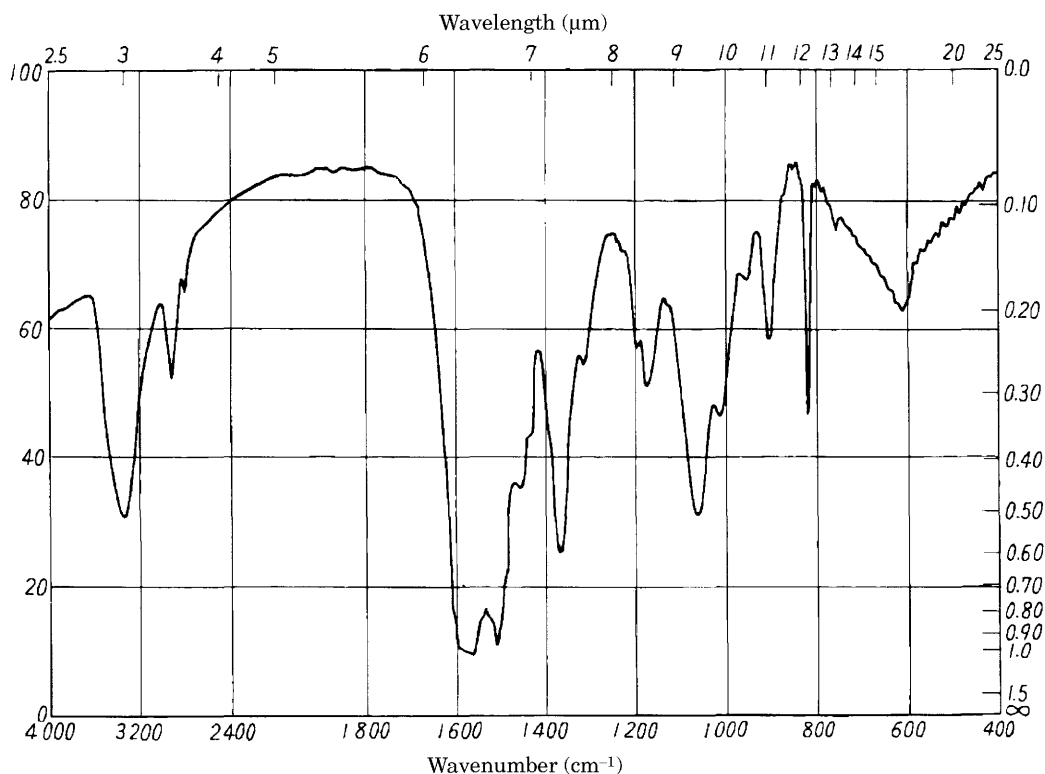
The vapour absorption method shall be according to Annex B.

9 Test report

The following items shall be described in the test report.

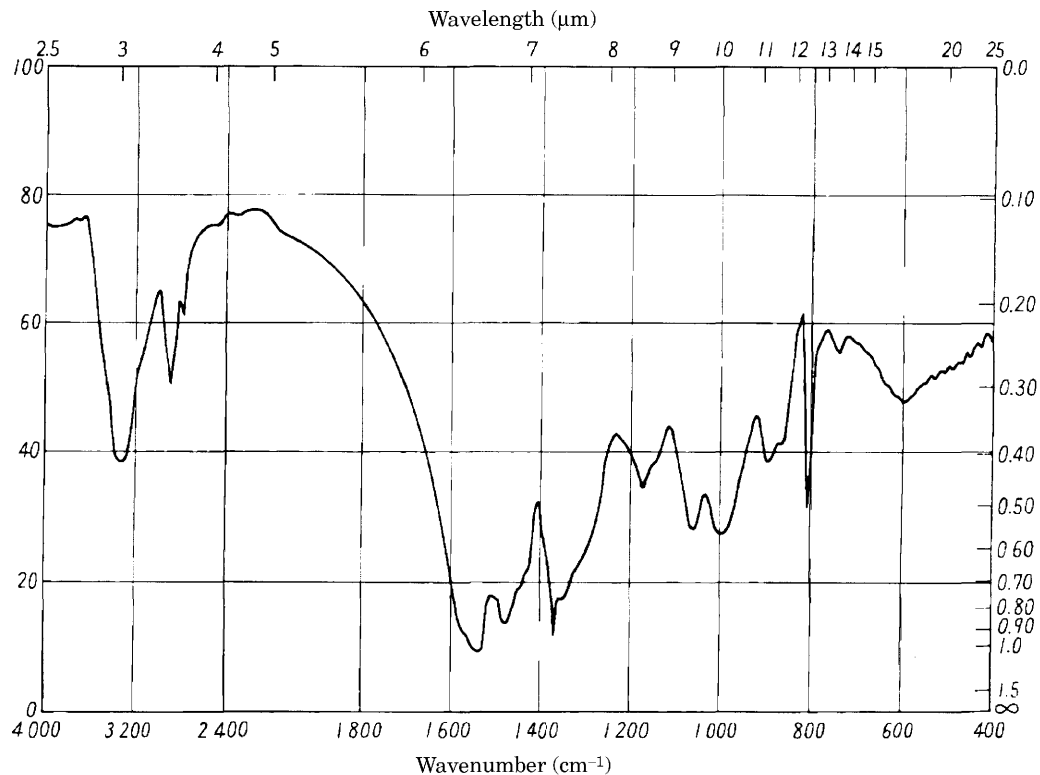
Refer to **A.9** or **B.9** for the case according to **ISO** method.

- a) Test date
- b) Number of this Standard
- c) Type of test
- d) Test method
- e) Test conditions
- f) Test results



NOTE : (K) in resin name means that the spectrum diagram is according to the potassium bromide pellet method.

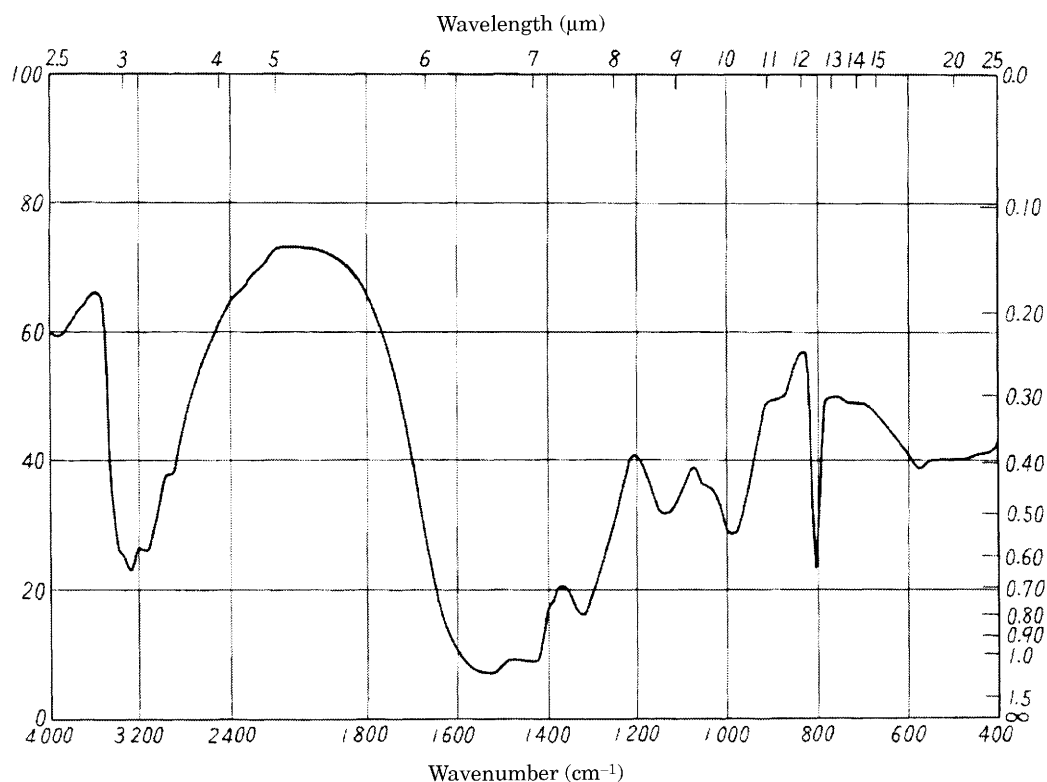
(1) Melamine series thermal hardening (K)



NOTE : (K) in resin name means that the spectrum diagram is according to the potassium bromide pellet method.

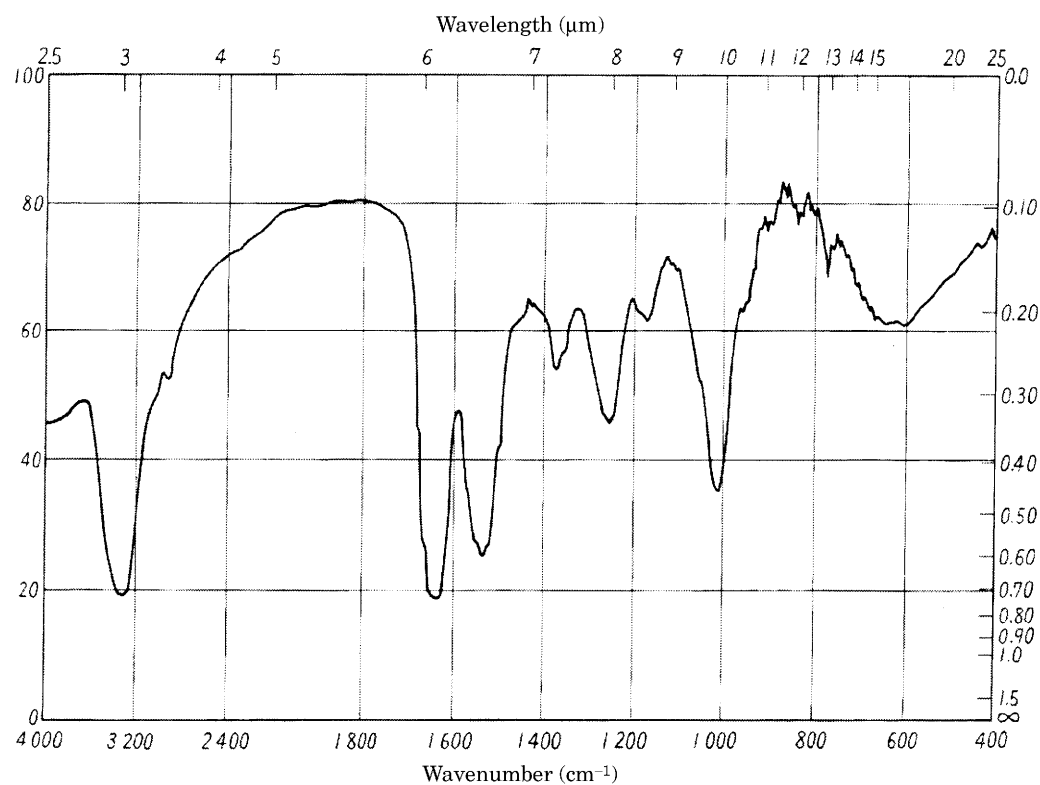
(2) Melamine series catalytic hardening (K)

Figure 1 Infrared absorption spectrum diagram of reference resin



NOTE : (K) in resin name means that the spectrum diagram is according to the potassium bromide pellet method.

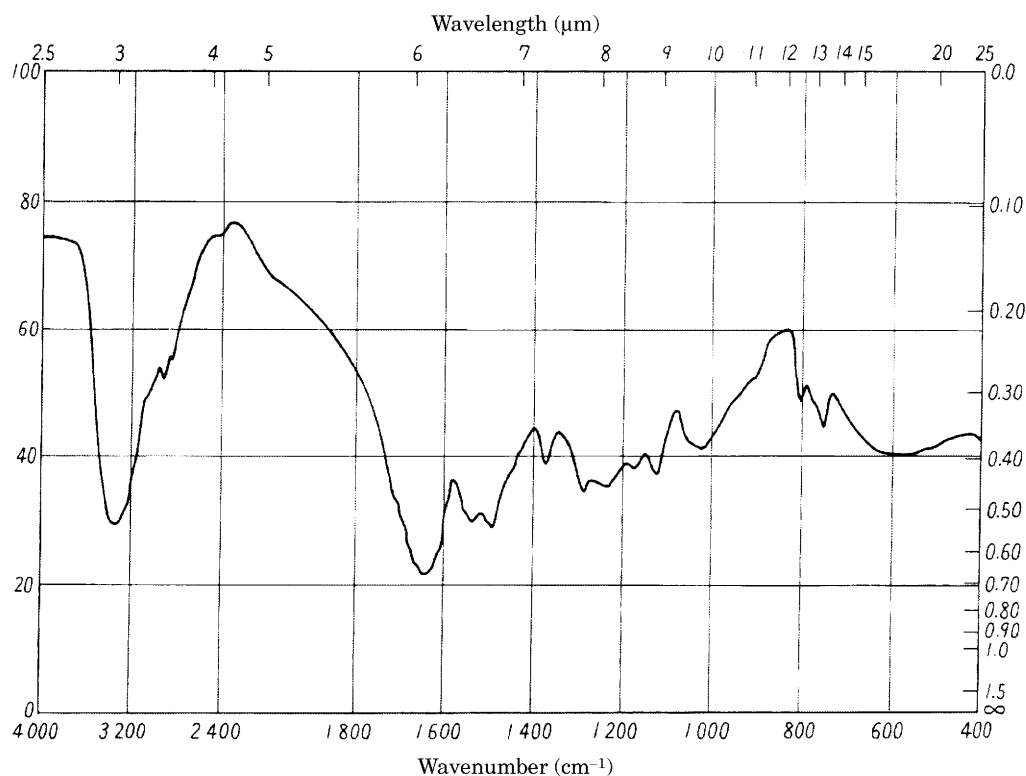
(3) Melamine series thermal hardening (0.1 mol/L hydrochloric acid extraction/sodium hydroxide neutralization) (K)



NOTE : (K) in resin name means that the spectrum diagram is according to the potassium bromide pellet method.

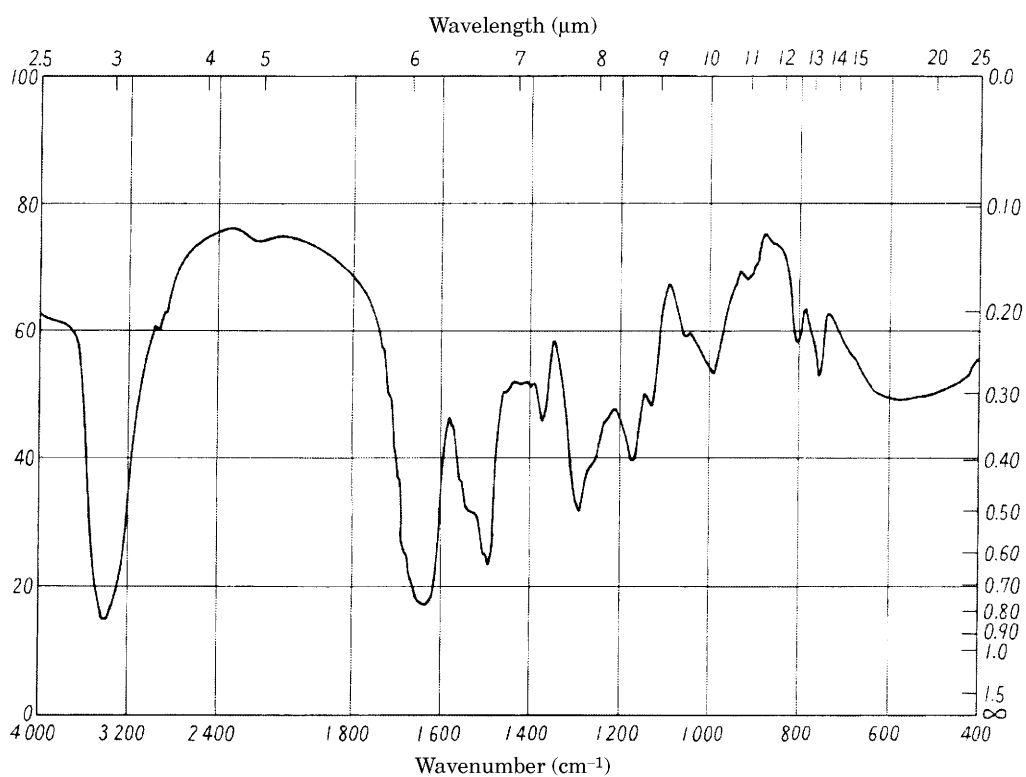
(4) Urea series (high condensation type) catalytic hardening (K)

Figure 1 (continued)



NOTE : (K) in resin name means that the spectrum diagram is according to the potassium bromide pellet method.

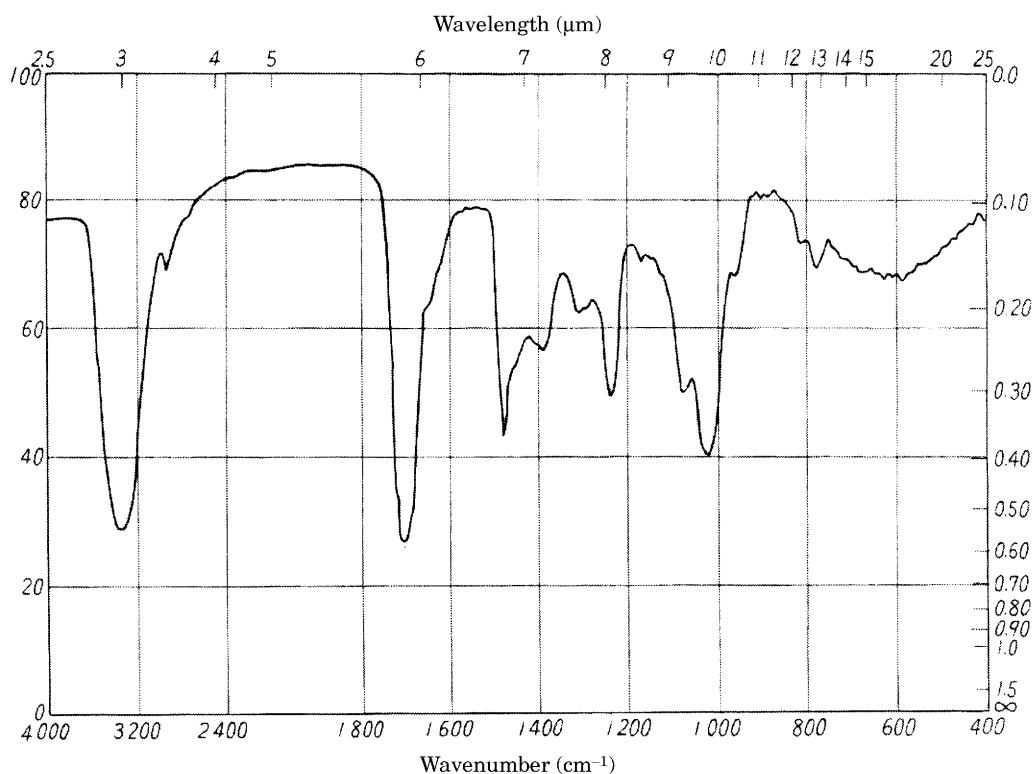
(5) Urea series (methylo type) catalytic hardening (K)



NOTE : (K) in resin name means that the spectrum diagram is according to the potassium bromide pellet method.

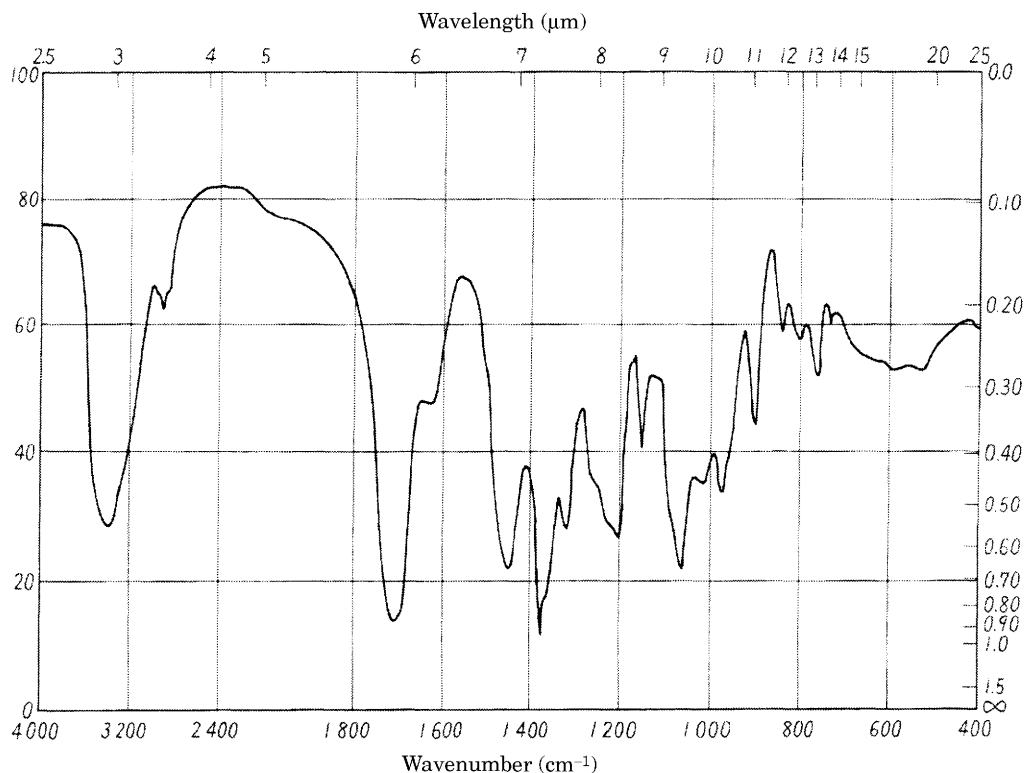
(6) Urea series (methylo type) catalytic hardening (0.1 mol/L hydrochloric acid extraction/sodium hydroxide neutralization) (K)

Figure 1 (continued)



NOTE : (K) in resin name means that the spectrum diagram is according to the potassium bromide pellet method.

(7) Urea series (glyoxal type) thermal hardening (K)



NOTE : (K) in resin name means that the spectrum diagram is according to the potassium bromide pellet method.

(8) Urea series (glyoxal type) catalytic hardening (K)

Figure 1 (continued)