

KES

Analysis for resin of felt

KES T – M081

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Contents

1. Scope of Application
2. Equipment and Reagent
3. Qualitative Analysis
4. Quantitative Analysis

Additional Rules



1. Scope of application

This standard shall specify the method of qualitative analysis on resin included in felt using an infrared spectrometer

2. Equipment and reagent

- (1) Infrared spectrometer
- (2) Extraction equipment
- (3) Rotary-type vaporizer
- (4) Extraction solvent

Repeat the operation of extruding, refining, and confirming until it is able to confirm

benzene, methyl alcohol, and hydrochloric acid solution of $\frac{N}{10}$

Also, enrich hydrochloric acid of $\frac{N}{10}$, extracted solution, after neutralizing

- (5) Silicagel for column chromatography
- (6) Hexane
- (7) Chloroform

3. Quantitative analysis

- (1) Extraction

Take test sample of reagent 5 g, put it in 200 ml flask with reflux cooling system, add extracted solution of 150 ml to this, heat it to the boiling temperature of extracted solution, and filter it with glass filter after extracting it for an hour.

Put it in 200 ml mass flask and enrich the solution to be 2 ~ 3 ml using evaporator

- (2) Refining

After filling glass pipe with dimension of 10 ~ 15 mm inner diameter and 200 ~ 300 mm length with glass wool, put mixture of silicagel for column chromatography and hexane to be about 150mm height in it

Cover a filter paper on the silicagel again, put this in extracted fluid and add hexane, chloroform, and methanol of 100 mℓ orderly.

At this time, in case that it is not possible to confirm resin by the spectrum absorbing infrared and measured at each solution because of mixture, it would be required to operate this work and divide the mixture according to take a little from same solution for refinement

(3) Confirming

Measure the spectrum of extracted solution after refining by the method of peeling and KBr and compare it with standard spectrum chart pre-made (Refer to figure)

Table 1 Combination of resin and extracted solution

Re-sin Extrac- ted solution	Mela- mine serie- s	Urea series					Epoxy series	Acry- lic ester series	Metaa- -cryle- ster series	Polya- mide series	Sili- con series	Poly- Styre- ne series	Polye- Thyle- ne series	Poly- -ace late- vinyl se- ries	Poly- ure-t hane se- ries	Fluo- rine se- ries
		Me-l hyl role serie- s	Urea- mela- mine series	ciem dsal se- ries	Wool on se- ries	Ethyle- ne series										
Benze ne	×	×	×	×	×	×	○	○	○	○	○	○	○	○	○	○
Methan ol	△	×	×	○	○	○	○	○	×	△	×	×	×	○	△	×
N/10 hydroxi de acid	○	○	○	○	○	○	×	×	×	×	×	×	×	×	×	×

- : Qualitative extraction is possible
 - △ : Part of qualitative extraction is possible according to a fiber
 - ×
- ×

4. Quantitative analysis

4.1 Take a test sample of about 10 g, remove edge fiber appropriately and let it not to lose weight on testing

4.2 Equipment and reagent

(1) Drier

It must maintain the temperature of 105 ~ 110 °C

(2) Soap

A kind of KS M 2704 (Powder washing soap)

(3) Phthalic anhydride sodium

4.2 Operation

(1) Weigh it after drying reagent at drier in temperature of 105 ~ 110 °C to be contained quantity

(2) Wash the test sample in water of 25 ± 2 °C for 10 minutes and weigh it after drying like clause 4.3 (1)

(3) Treat the test sample in mixture (1 : 50) of 0.25 % soap fluid and 0.25 % anhydride sodium for 5 minutes remaining 90 °C, wash it in water of 90 °C and weigh it after drying

4.4 Calculation

$$\text{Water solution resin (\%)} = \frac{W_0 - W_1}{W_3} \times 100$$

$$\text{Soap solution resin (\%)} = \frac{W_1 - W_3}{W_3} \times 100$$

$$\text{Sticking resin (\%)} = \frac{W_2 - W_3}{W_3} \times 100$$

$$\text{Total resin (\%)} = \frac{W_0 - W_3}{W_3} \times 100$$

Here, W_0 : Weight of dries test sample

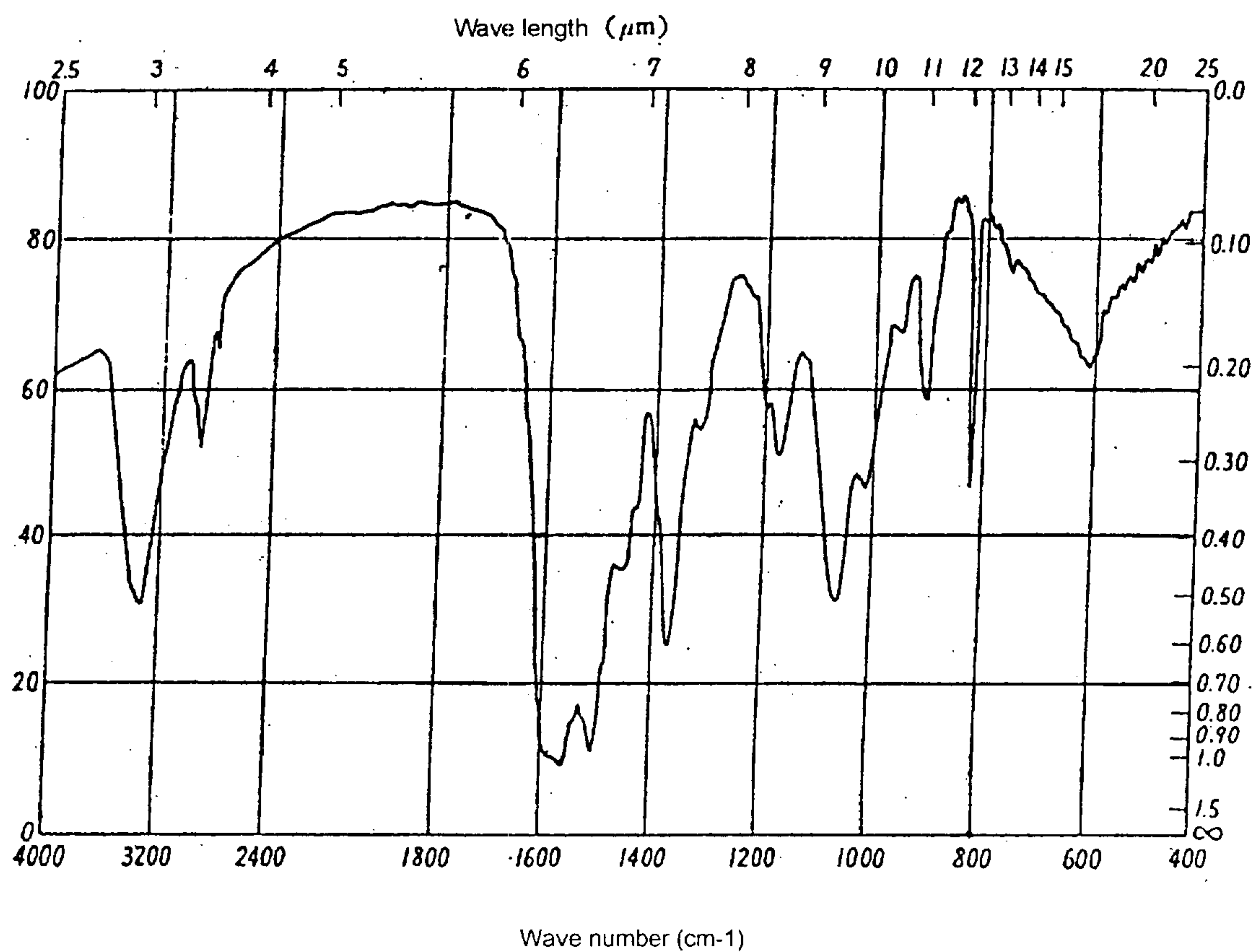
W_1 : Weight of test sample after extracting water solution

W_2 : Weight of test sample after extracting soap solution

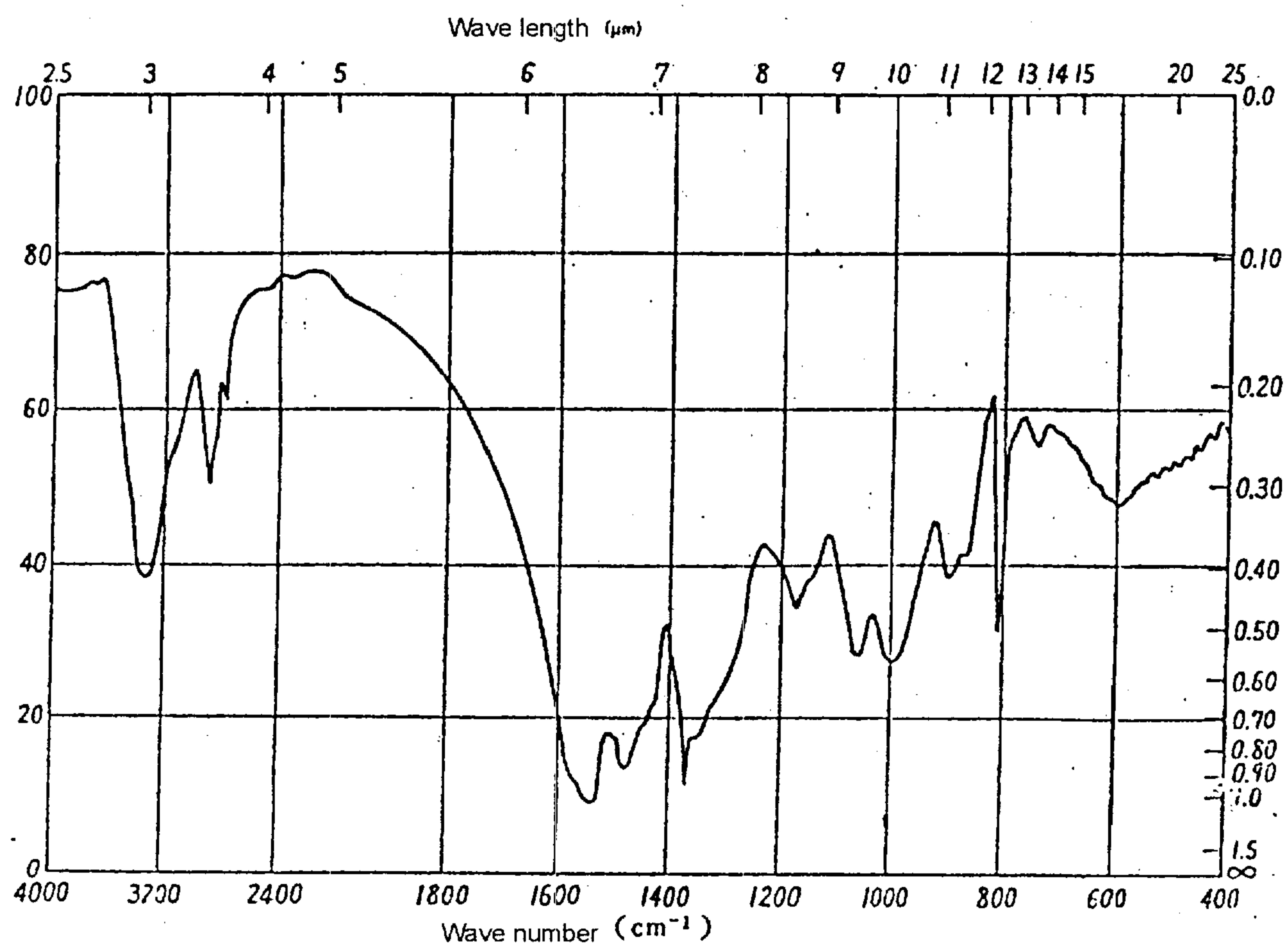
W_3 : Weight of test sample after extracting hydrochloric acid treating

Reference figure Infrared absorbing spectrum for processing each fiber

Reference figure 1 – 1 Melamine series thermosetting (K)



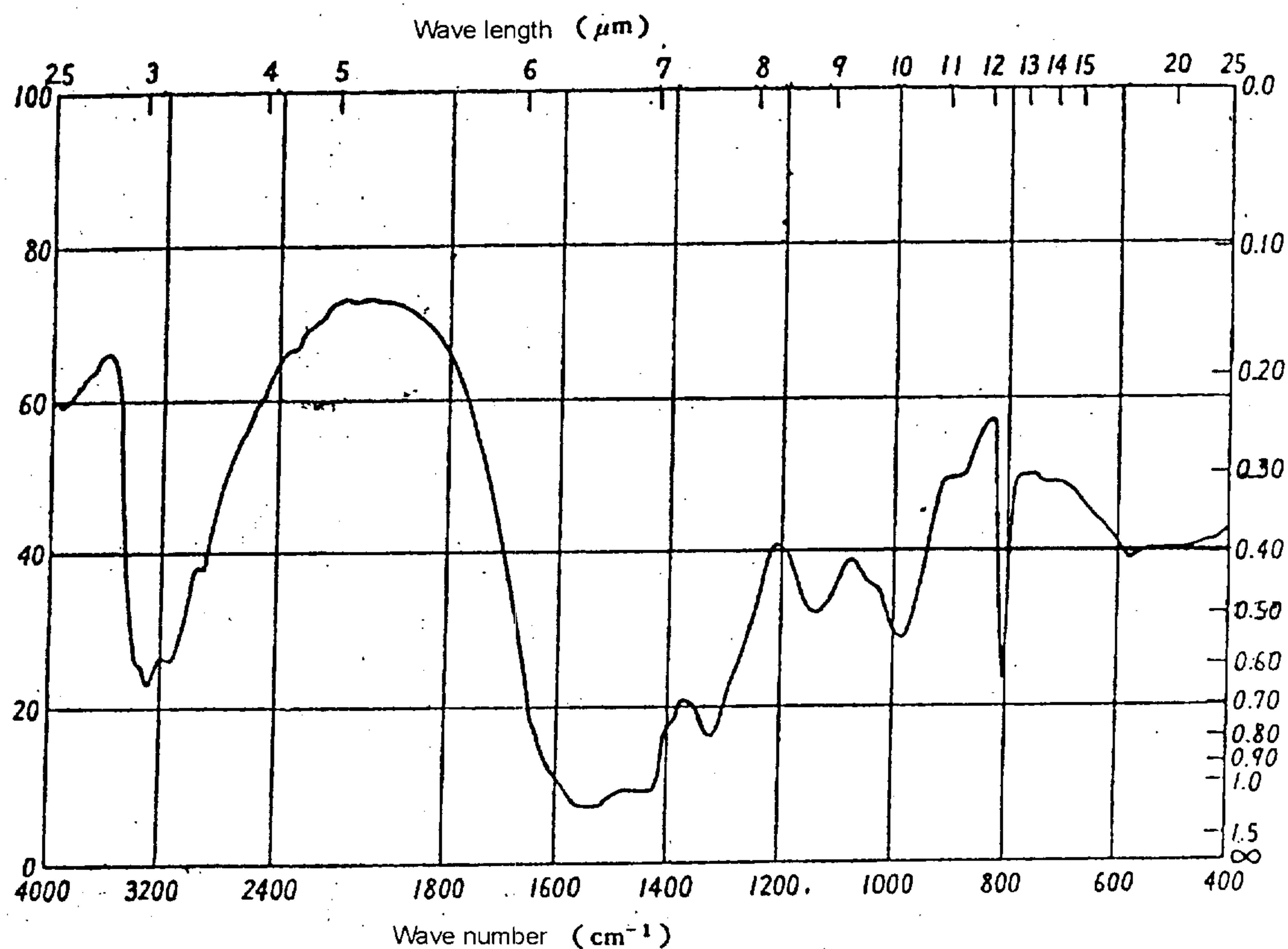
Reference figure 1 – 2 Melamine series catalyst hardening (K)



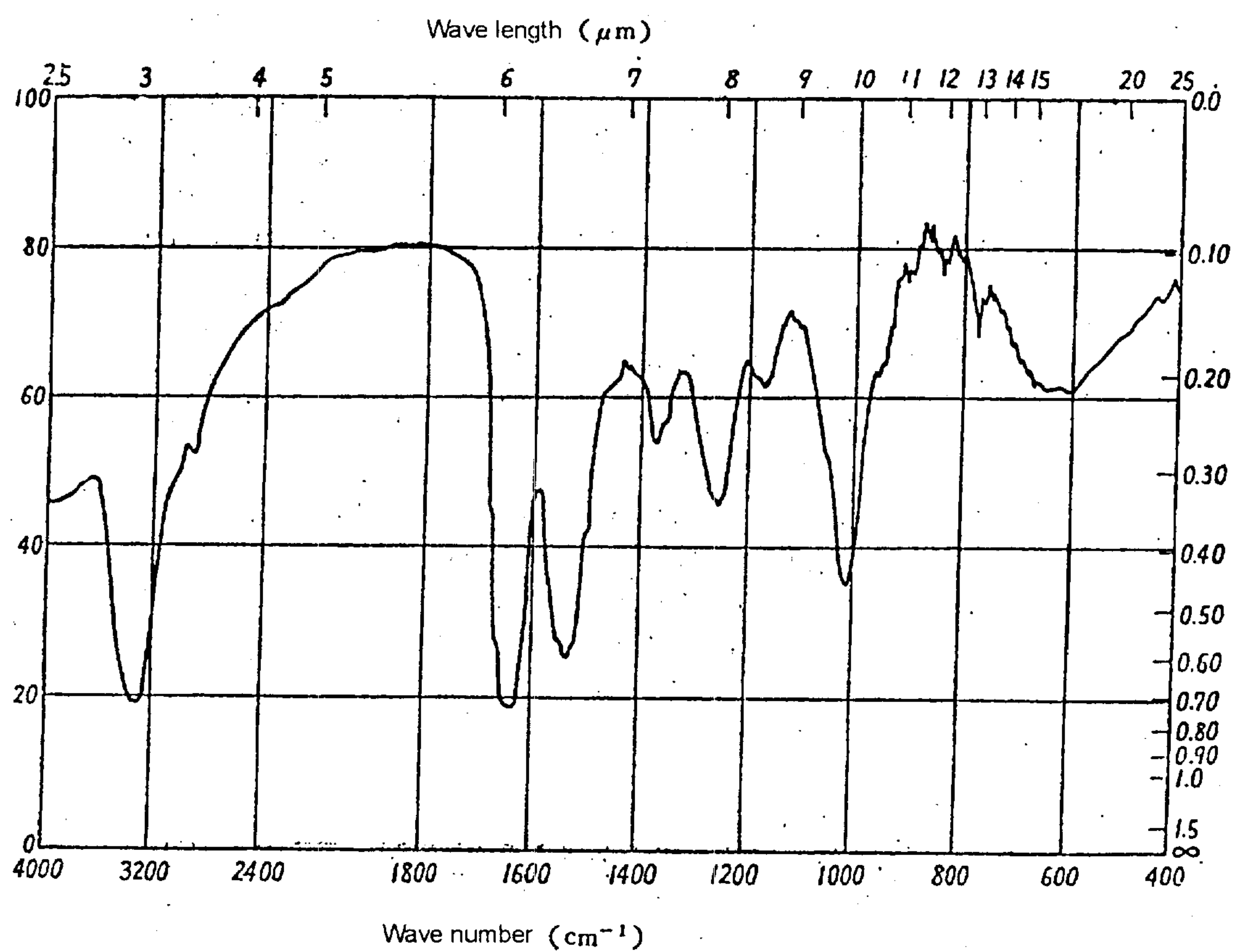
Reference figure 1 – 3 Melamine series thermosetting N/10 hydrochloric acid

extraction

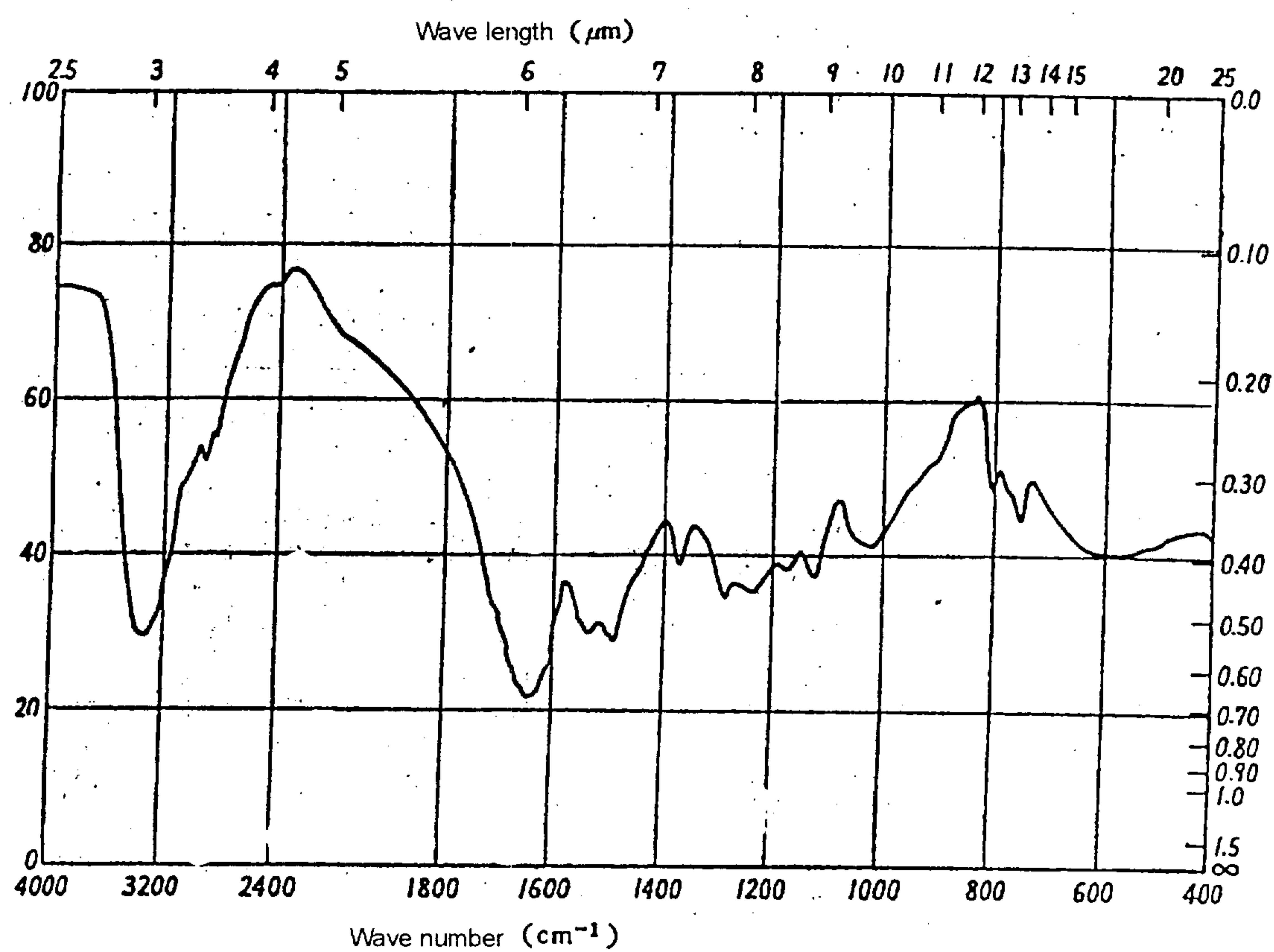
Sodium hydroxide neutralizing (K)



Reference figure 2 – 1 Urea series (High condensation type)

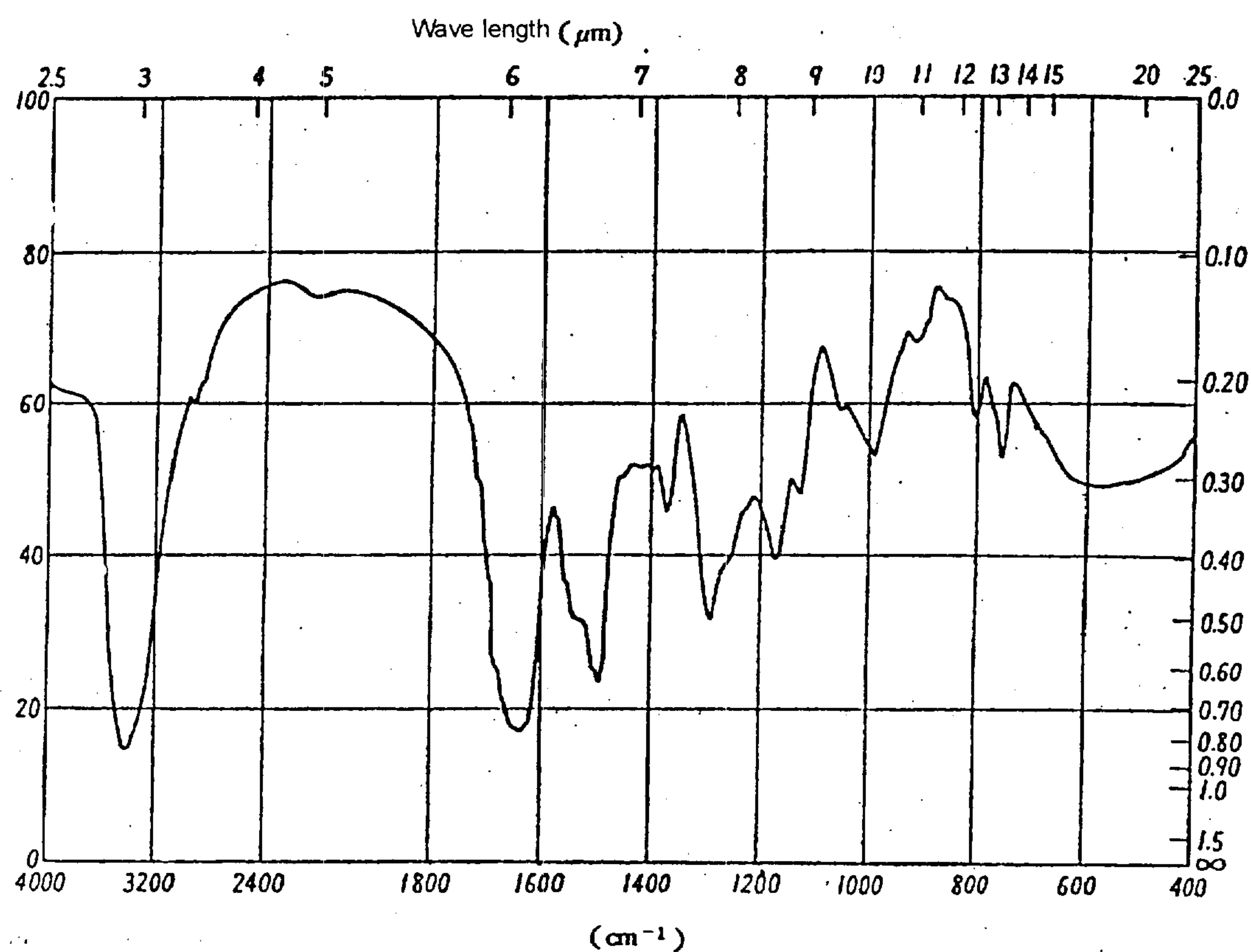


Reference figure 2 – 2 Urea type(Methyl role type) catalyst hardening (K)



Reference figure 2 – 3 Urea type(Methyl role type) catalyst hardening (K) N/10

Hydrochloric acid extraction sodium hydroxide neutralizing (K)



Reference figure 2 – 4 Urea series (glyocsal type) thermosetting (K)

