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**Testing methods for quantitative analysis
of fibre mixtures of textiles — Part 2:
Testing methods for quantitative analysis
of fibre mixtures**

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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 1030-2:2006** 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.

JIS L 1030 consists of the following two parts under the general title “*Testing methods for quantitative analysis of fibre mixtures of textiles*”:

Part 1: Testing methods for fibre identification

Part 2: Testing methods for quantitative analysis of fibre mixtures

Testing methods for quantitative analysis of fibre mixtures of textiles — Part 2 : Testing methods for quantitative analysis of fibre mixtures

Introduction

This Japanese Industrial Standard has been prepared based on the first editions of ISO 1833-1, ISO 1833-2, ISO 1833-3, ISO 1833-4, ISO 1833-5, ISO 1833-7, ISO 1833-8, ISO 1833-9, ISO 1833-10, ISO 1833-11, ISO 1833-12, ISO 1833-13, ISO 1833-14, ISO 1833-15, ISO 1833-16, ISO 1833-17, ISO 1833-18, ISO 1833-19 and ISO 1833-21 published in 2006 and the first editions of ISO 1833-6 and ISO 17751 published in 2007, the first edition of ISO 1833-20 published in 2009 and the first edition of ISO 1833-24 published in 2010 with some modifications of the technical contents.

The portions with continuous sidelines or dotted underlines are the matters in which the contents of the corresponding International Standard have been modified. A list of modifications with explanations is given in Annex JA.

1 Scope

This Standard specifies three kinds of test methods, namely the release method, the dissolution method and the microscopy as the test methods for obtaining the mixture ratio of fibre mixed in textile goods. Before these test methods are applied, it is required that all fibres mixed in textile goods are identified. The fibre identification is specified in JIS L 1030-1.

Generally, for those textile products of fibre mixtures to which the release method is applicable, the release method shall be used, and for those to which it is not applicable, the dissolution method shall be used. For the textile products of fibre mixtures to which neither of the above methods is applicable, the microscopy shall be used.

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 people appropriately qualified and/or experienced in handling chemicals.

Note ¹⁾ Information on substances which may damage the health is described in the Safety Data Sheet (SDS) in detail.

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

ISO 1833-1 : 2006 *Textiles — Quantitative chemical analysis — Part 1 :*

General principles of testing

- ISO 1833-2 : 2006 *Textiles — Quantitative chemical analysis — Part 2 : Ternary fibre mixtures*
- ISO 1833-3 : 2006 *Textiles — Quantitative chemical analysis — Part 3 : Mixtures of acetate and certain other fibres (method using acetone)*
- ISO 1833-4 : 2006 *Textiles — Quantitative chemical analysis — Part 4 : Mixtures of certain protein and certain other fibres (method using hypochlorite)*
- ISO 1833-5 : 2006 *Textiles — Quantitative chemical analysis — Part 5 : Mixtures of viscose, cupro or modal and cotton fibres (method using sodium zincate)*
- ISO 1833-6 : 2007 *Textiles — Quantitative chemical analysis — Part 6 : Mixtures of viscose or certain types of cupro or modal or lyocell and cotton fibres (method using formic acid and zinc chloride)*
- ISO 1833-7 : 2006 *Textiles — Quantitative chemical analysis — Part 7 : Mixtures of polyamide and certain other fibres (method using formic acid)*
- ISO 1833-8 : 2006 *Textiles — Quantitative chemical analysis — Part 8 : Mixtures of acetate and triacetate fibres (method using acetone)*
- ISO 1833-9 : 2006 *Textiles — Quantitative chemical analysis — Part 9 : Mixtures of acetate and triacetate fibres (method using benzyl alcohol)*
- ISO 1833-10 : 2006 *Textiles — Quantitative chemical analysis — Part 10 : Mixtures of triacetate or polylactide and certain other fibres (method using dichloromethane)*
- ISO 1833-11 : 2006 *Textiles — Quantitative chemical analysis — Part 11 : Mixtures of cellulose and polyester fibres (method using sulfuric acid)*
- ISO 1833-12 : 2006 *Textiles — Quantitative chemical analysis — Part 12 : Mixtures of acrylic, certain modacrylics, certain chlorofibres, certain elastanes and certain other fibres (method using dimethylformamide)*
- ISO 1833-13 : 2006 *Textiles — Quantitative chemical analysis — Part 13 : Mixtures of certain chlorofibres and certain other fibres (method using carbon disulfide/acetone)*
- ISO 1833-14 : 2006 *Textiles — Quantitative chemical analysis — Part 14 : Mixtures of acetate and certain chlorofibres*

(method using acetic acid)

- ISO 1833-15 : 2006 *Textiles — Quantitative chemical analysis — Part 15 : Mixtures of jute and certain animal fibres (method by determining nitrogen content)*
- ISO 1833-16 : 2006 *Textiles — Quantitative chemical analysis — Part 16 : Mixtures of polypropylene fibres and certain other fibres (method using xylene)*
- ISO 1833-17 : 2006 *Textiles — Quantitative chemical analysis — Part 17 : Mixtures of chlorofibres (homopolymers of vinyl chloride) and certain other fibres (method using sulfuric acid)*
- ISO 1833-18 : 2006 *Textiles — Quantitative chemical analysis — Part 18 : Mixtures of silk and wool or hair (method using sulfuric acid)*
- ISO 1833-19 : 2006 *Textiles — Quantitative chemical analysis — Part 19 : Mixtures of cellulose fibres and asbestos (method by heating)*
- ISO 1833-20 : 2009 *Textiles — Quantitative chemical analysis — Part 20 : Mixtures of elastane and certain other fibres (method using dimethylacetamide)*
- ISO 1833-21 : 2006 *Textiles — Quantitative chemical analysis — Part 21 : Mixtures of chlorofibres, certain modacrylics, certain elastanes, acetates, triacetates and certain other fibres (method using cyclohexanone)*
- ISO 1833-24 : 2010 *Textiles — Quantitative chemical analysis — Part 24 : Mixtures of polyester and certain other fibres (method using phenol and tetrachloroethane)*
- ISO 17751 : 2007 *Textiles — Quantitative analysis of animal fibres by microscopy — Cashmere, wool, speciality fibres and their blends (Overall evaluation : MOD)*

The symbols which denote the degree of correspondence in the contents between the relevant International Standard 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. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS K 0050 *General rules for chemical analysis*

JIS K 8085 *Ammonia solution (Reagent)*

JIS K 8102 *Ethanol (95) (Reagent)*

JIS K 8111 *Zinc chloride (Reagent)*

JIS K 8161 *Dichloromethane (Reagent)*

JIS K 8180 *Hydrochloric acid (Reagent)*

JIS K 8264 *Formic acid (Reagent)*

JIS K 8305 *meta-Cresol (Reagent)*

JIS K 8322 *Chloroform (Reagent)*

JIS K 8361 *Ethyl acetate (Reagent)*

JIS K 8500 *N,N-Dimethylformamide (Reagent)*

JIS K 8576 *Sodium hydroxide (Reagent)*

JIS K 8680 *Toluene (Reagent)*

JIS K 8863 *Boric acid (Reagent)*

JIS K 8891 *Methanol (Reagent)*

JIS K 8951 *Sulfuric acid (Reagent)*

JIS K 8962 *Potassium sulfate (Reagent)*

JIS K 9001 *Potassium thiocyanate (Reagent)*

JIS K 9003 *Liquid paraffin (Reagent)*

JIS L 0105 *General principles of physical testing methods for textiles*

NOTE : Corresponding International Standard : ISO 139 *Textiles — Standard atmospheres for conditioning and testing* (MOD)

JIS L 1013 *Testing methods for man-made filament yarns*

JIS L 1030-1 *Testing methods for quantitative analysis of fibre mixtures — Part 1 : Testing methods for fibre identification*

JIS R 3503 *Glass apparatus for chemical analysis*

JIS Z 8401 *Guide to the rounding of numbers*

3 Terms and definitions

For the purpose of this Standard, the following terms and definitions apply.

3.1 mixture ratio

in textile goods composed of more than one kind of fibre, the ratio of mass of each kind of component fibre to the total mass of component fibres, expressed by mass percentage

3.2 bone dry mass

mass of specimen obtained when it has attained a constant mass after being left to

stand in a ventilated oven at a temperature of $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$

The constant mass here refers to the state of specimen in which, in mass measurements made at a minimum of 15 min intervals, the mass measured before and after 15 min interval no longer differs more than 0.1 %. However, for fibre of 0.0 % in official regain, the mass of sample under standard condition is regarded as bone dry mass.

3.3 mixture ratio based on bone dry mass

mixture ratio obtained from the bone dry mass

3.4 mixture ratio based on corrected mass

the mixture ratio obtained from the mass when the moisture corresponding to official regain as shown in table 1 is contained in the mixed fibre respectively

Table 1 Official regain of fibre

Kind of fibre		Name of fibre	Official regain (%)
Cotton		Cotton	8.5
Wool or animal hair		Wool or animal hair	15.0 ^{a)}
Silk		Silk	12.0 ^{b)}
Linen		Flax and ramie	12.0
Viscose fibre	Those with average degree of polymerization 450 or greater	Polynosic	11.0
	Others	Rayon	11.0
		Modal	11.0
Cuprammonium fibre		Cupro	11.0
Purified cellulose fibre		Lyocell	11.0
Acetate fibre	Those of which at least 92 % of hydroxyl is acetylated	Triacetate	3.5
	Those manufactured by dissolution method	Acetate	6.5
Promix fibre		Promix	5.0
Nylon fibre		Nylon	4.5
Aramid fibre		Aramid	7.0
Vinylon fibre		Vinylon	5.0
Polyvinylidene chloride-based synthetic fibre		Vinylidene	0.0
Polyvinylchloride-based synthetic fibre		Polyvinylchloride	0.0
Polyester-based synthetic fibre		Polyester	0.4
Polyacrylnitrile-based synthetic fibre	Those of which the mass ratio of acrylonitrile is 85 % or greater	Acrylic	2.0
	Others	Modacrylic	2.0
Polyethylene-based synthetic fibre		Polyethylene	0.0
Polypropylene-based synthetic fibre		Polypropylene	0.0
Polyurethane-based synthetic fibre		Polyurethane	1.0
Polychlal fibre		Polychlal	3.0
Polylactide fibre		Polylactide	0.5
Polyalkylene paraoxybenzoate-based synthetic fibre		Benzoate	0.4
Glass fibre		Glass fibre	0.0
Carbon fibre		Carbon fibre	0.0
Metallic fibre		Metallic fibre	0.0
Feather		Feather	13.0

Table 1 (concluded)

Kind of fibre		Name of fibre	Official regain (%)
Other fibres	Natural fibre	Natural fibre	12.0
		Jute	13.75
	Artificial fibre	Cellulose fibre	11.0
		Others ^{c)}	0.0
		Others ^{c)}	0.0
Notes ^{a)} Generally applied value. ^{b)} Applies to degummed silk. ^{c)} For fibres with official regain other than 0.0 %, an appropriate moisture regain can be applied based on consideration of conventional practices by inspection institutes and relevant industry. In this case, the applied moisture regain shall be stated in the test report.			

4 Classification of tests

The classification of tests shall be as follows.

- a) **Release method** This method shall be generally used for all fibre mixture textile products to which it is applicable.
- b) **Dissolution method** This method shall be used for fibre mixture textile products to which the release method is not applicable.

The parenthesized numerals are the subclauses in this Standard in which the respective test methods are given.

- 1) 60 % Sulfuric acid method (6.2.1)
- 2) 70 % Sulfuric acid method (6.2.2)
- 3) Warm 75 % sulfuric acid method (6.2.3)
- 4) 75 % Sulfuric acid method (6.2.4)
- 5) 80 % Sulfuric acid method (6.2.5)
- 6) Concentrated sulfuric acid method I (6.2.6)
- 7) Concentrated sulfuric acid method II (6.2.7)
- 8) 20 % Hydrochloric acid method (6.2.8)
- 9) 35 % Hydrochloric acid method (6.2.9)
- 10) Mixed acid method (6.2.10)
- 11) 2.5 % Sodium hydroxide method (6.2.11)
- 12) 5 % Sodium hydroxide method (6.2.12)
- 13) 30 % Sodium hydroxide method (6.2.13)
- 14) Cuprammonium method (6.2.14)
- 15) Glacial acetic acid method (6.2.15)

- 16) Hot glacial acetic acid method (6.2.16)
- 17) 70 % Acetone method (6.2.17)
- 18) 80 % Acetone method (6.2.18)
- 19) 100 % Acetone method (6.2.19)
- 20) Warm 100 % acetone method (6.2.20)
- 21) Sodium hypochlorite method (6.2.21)
- 22) Sodium zincate method (6.2.22)
- 23) Formic acid/zinc chloride mixed solution method (6.2.23)
- 24) 75 % Formic acid method (6.2.24)
- 25) 80 % Formic acid method (6.2.25)
- 26) Warm dimethylformamide method (6.2.26)
- 27) Hot dimethylformamide method I (6.2.27)
- 28) Hot dimethylformamide method II (6.2.28)
- 29) Dichloromethane method (6.2.29)
- 30) 65 % Potassium thiocyanate method (6.2.30)
- 31) Benzyl alcohol method (6.2.31)
- 32) Tetrahydrofuran method (6.2.32)
- 33) Phenol/tetrachloroethane mixed solution method (6.2.33)
- 34) *meta*-Cresol method (6.2.34)
- 35) Carbon disulfide/acetone azeotropic mixed solution method (6.2.35)
- 36) Benzene/cyclohexanone mixed solution method (6.2.36)
- 37) Boiling xylene method (6.2.37)
- 38) Chloroform method (6.2.38)
- 39) Dimethylacetamide method (6.2.39)
- 40) Cyclohexane method (6.2.40)
- c) **Microscopy** This method is used for fibres to which neither the release method nor the dissolution method is applicable.
- d) **Method by determining nitrogen content (Annex A)**
- e) **Heating method (Annex B)**
- f) **Ternary fibre mixtures (individual dissolution method) (Annex C)**

5 Release method

5.1 Application

This test method is applied to the calculation of mixture ratio of the textile goods which are woven, knitted with or constituted of the yarn of different kinds of fibre and which can be unravelled to be separated into different kinds of fibre.

5.2 Summary

The sample is taken from the textile goods which are woven, knitted with or constituted of yarns of different kinds of fibre so as to contain all yarns constituting one unit of design, and then is unravelled to be separated into yarns of different kinds of fibre. The masses of respective yarns are obtained and expressed by percentage.

5.3 Test site

The test shall be carried out in the room under the conditions of usual temperature and humidity.

5.4 Apparatus and instrument

The following apparatus and instrument shall be used.

- a) **Desiccator**, specified in JIS R 3503, containing self-indicating silica gel.
- b) **Ventilated oven**, for drying specimens at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.
- c) **Chemical balance**, capable of weighing to the nearest 0.000 2 g.
- d) **Soxhlet extraction apparatus**, of sufficient size to give a volume equal to 20 times the mass, in grams, of the sample, or any other apparatus giving equal results.
- e) **Erlenmeyer flask**, specified in JIS R 3503, having a capacity of 200 ml or over, and a glass ground-in stopper.
- f) **Weighing bottle**, sufficiently large to contain the crucible type filter, or an equivalent instrument.

5.5 Reagent

Petroleum ether, distilling between $30\text{ }^{\circ}\text{C}$ to $60\text{ }^{\circ}\text{C}$.

5.6 Sampling

The sample, of 1 g or over, containing all yarns constituting one unit of design is taken from the textile goods which are woven, knitted with or constituted of yarns of different kinds of fibre. Other conditions shall be in accordance with clause 6 of **JIS L 0105**.

5.7 Pre-treatment of sample

5.7.1 Soxhlet extraction method

Put the air-dried sample and petroleum ether (5.5) in a Soxhlet extraction apparatus [5.4 d)], and extract for 1 h by circulating at least 6 times per hour. Stand the sample so as to allow evaporation of petroleum ether, immerse it in water at ordinary tem-

perature for 1 h, then immerse it further in warm water of $65\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ at least for 1 h. The bath ratio at immersion shall be 100 : 1, and the solution shall be stirred once in a while. Wring the sample to remove excess water, and then allow it to dry naturally.

NOTE : For the unbleached vegetable fibres (for example jute, coco fibre), all natural non-fibrous matters cannot be removed by the usual pre-treatment using water and petroleum ether.

5.7.2 Other extraction methods

When non-fibrous matter is not extracted by petroleum ether or water, remove it according to the following method so as not to change all fibrous weave.

- a) In the case of other fibre excluding silk, wool, animal hair, acetate, promix, polyvinylchloride and vinylidene, the following method shall be used.
 - 1) Put the sample in 100 times its amount of 0.5 % sodium carbonate (anhydrous) solution, and treat in a boiling water bath for 15 min.
 - 2) Carry out the washing twice in hot water at $60\text{ }^{\circ}\text{C}$ to $70\text{ }^{\circ}\text{C}$ for 10 min, and then carry out washing in water and drying.
- b) For mixtures with wool, animal hair or polyester, the following method shall be used.
 - 1) Put the sample in 100 times its amount of approximately 0.3 % non-ionic surface active agent aqueous solution, and treat it for 15 min at $60\text{ }^{\circ}\text{C}$ to $70\text{ }^{\circ}\text{C}$ while stirring.
 - 2) Wash sufficiently with water and dry.
- c) For mixtures with polyurethane, the following method shall be used.
 - 1) Shake the sample vigorously together with 50 times its amount of petroleum ether in an Erlenmeyer flask with ground-in stopper [5.4 e)] for 10 min, then, repeat the procedure by using the same amount of fresh petroleum ether.
 - 2) After drying to remove the petroleum ether, throw the sample in hot water and further treat for 5 min. Wash with water and dry, and remove the natural non-fibrous matters in fibres contained in the sample and the substance added during manufacturing process.
- d) For mixtures of vinylidene, polyvinylchloride or polypropylene fibre that are not treated with sizing or resin processing, the following method shall be used.
 - 1) Put the sample in 100 times its amount of approximately 1 % nonionic surface active agent aqueous solution, and immerse it for 15 min at $23\text{ }^{\circ}\text{C}$ to $25\text{ }^{\circ}\text{C}$ while stirring occasionally.
 - 2) Pull out the sample, wash it sufficiently with water, and dry to remove the natural non-fibrous matters in fibre contained in the sample and the substance added during manufacturing process.
- e) For mixtures of the fibre in c) treated with sizing or resin processing, the following method shall be used.

- 1) In addition to the treatment in c) and d), put the sample in 100 times its amount of 0.25 % hydrochloric acid solution, and treat in a boiling water bath for 15 min.
- 2) Wash this successively with hot water, 0.2 % ammonia solution and water, and dry. In the case of vinylon other than formalized vinylon, carry out the operation described in b) in place of the 0.25 % hydrochloric acid treatment, and in this case, no pre-treatment is performed for formalized vinylon.

Apart from the pre-treatment in 5.7.1 and 5.7.2, discolouration, which would not interfere with the operation in 5.8, or any suitable pre-treatment using other solvent may be performed.

5.8 Operation

5.8.1 Separation of yarn

Take a sample containing all yarns constituting one unit of design, 1 g or over in amount, from the pre-treated sample. Unravel the sample and separate it into yarns of different kinds of fibre, to supply it as specimen.

5.8.2 Drying

Put specimens in separate weighing bottles [5.4 f)], and dry them in an ventilated oven [5.4 b)] at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ until they reach constant mass.

5.8.3 Cooling

Transfer the weighing bottles into a desiccator [5.4 a)] and leave them to cool to ordinary temperature.

5.8.4 Weighing

Take out the cooled weighing bottles from the desiccators and weigh them by a chemical balance [5.4 c)] within 2 min. Take out the specimen from the weighing bottle after weighing, immediately weigh the weighing bottle again and obtain the bone dry mass of specimen from the difference of weighed values. In this procedure, always weigh to the nearest 0.000 2 g.

5.9 Calculation

5.9.1 Mixture ratio based on bone dry mass

The mixture ratio shall be obtained according to the following formulae (1) to (5).

a) In the case of binary fibre mixtures

$$x_A = \frac{W_A}{W_A + W_B} \times 100 \dots\dots\dots (1)$$

$$x_B = 100 - x_A \dots\dots\dots (2)$$

where, x_A : mixture ratio based on bone dry mass of fibre A (%)
 x_B : mixture ratio based on bone dry mass of fibre B (%)
 W_A : bone dry mass of fibre A (g)
 W_B : bone dry mass of fibre B (g)

b) **In the case of ternary fibre mixtures**

$$x_A = \frac{W_A}{W_A + W_B + W_C} \times 100 \quad \dots\dots\dots (3)$$

$$x_B = \frac{W_B}{W_A + W_B + W_C} \times 100 \quad \dots\dots\dots (4)$$

$$x_C = 100 - (x_A + x_B) \quad \dots\dots\dots (5)$$

where, x_A : mixture ratio based on bone dry mass of fibre A (%)
 x_B : mixture ratio based on bone dry mass of fibre B (%)
 x_C : mixture ratio based on bone dry mass of fibre C (%)
 W_A : bone dry mass of fibre A (g)
 W_B : bone dry mass of fibre B (g)
 W_C : bone dry mass of fibre C (g)

5.9.2 **Mixture ratio based on corrected mass**

The mixture ratio based on corrected mass shall be obtained according to the following formulae (6) to (12).

a) **In the case of binary fibre mixtures**

$$X_A = \frac{W_A \times \left(1 + \frac{R_A}{100}\right)}{W_A \times \left(1 + \frac{R_A}{100}\right) + W_B \times \left(1 + \frac{R_B}{100}\right)} \times 100 \quad \dots\dots\dots (6)$$

$$X_B = 100 - X_A \quad \dots\dots\dots (7)$$

where, X_A : mixture ratio based on corrected mass of fibre A (%)
 X_B : mixture ratio based on corrected mass of fibre B (%)
 W_A : bone dry mass of fibre A (g)
 W_B : bone dry mass of fibre B (g)
 R_A : official regain of fibre A as shown in table 1 (%)
 R_B : official regain of fibre B as shown in table 1 (%)

The mixture ratio based on corrected mass, with the dried mass added with the fibrous matter and non-fibrous matter removed by pre-treatment, shall be obtained according to the following formula:

$$X_A = \frac{W_A \times \left(1 + \frac{R_A + N_A}{100}\right)}{W_A \times \left(1 + \frac{R_A + N_A}{100}\right) + W_B \times \left(1 + \frac{R_B + N_B}{100}\right)} \times 100 \quad \dots\dots\dots (8)$$

$$X_B = 100 - X_A \quad \dots\dots\dots (9)$$

where, X_A : mixture ratio based on corrected mass of fibre A (%)
 X_B : mixture ratio based on corrected mass of fibre B (%)
 W_A : bone dry mass of fibre A (g)

- W_B : bone dry mass of fibre B (g)
 R_A : official regain of fibre A as shown in table 1 (%)
 R_B : official regain of fibre B as shown in table 1 (%)
 N_A : decrease rate of fibre A produced by pre-treatment and/or percentage of non-fibrous matter to be added (%)
 N_B : decrease rate of fibre B produced by pre-treatment and/or percentage of non-fibrous matter to be added (%)

b) In the case of ternary fibre mixtures

$$X_A = \frac{W_A \times \left(1 + \frac{R_A}{100}\right)}{W_A \times \left(1 + \frac{R_A}{100}\right) + W_B \times \left(1 + \frac{R_B}{100}\right) + W_C \times \left(1 + \frac{R_C}{100}\right)} \times 100 \dots\dots\dots(10)$$

$$X_B = \frac{W_B \times \left(1 + \frac{R_B}{100}\right)}{W_A \times \left(1 + \frac{R_A}{100}\right) + W_B \times \left(1 + \frac{R_B}{100}\right) + W_C \times \left(1 + \frac{R_C}{100}\right)} \times 100 \dots\dots\dots(11)$$

$$X_C = 100 - (X_A + X_B) \dots\dots\dots(12)$$

- where, X_A : mixture ratio based on corrected mass of fibre A (%)
 X_B : mixture ratio based on corrected mass of fibre B (%)
 X_C : mixture ratio based on corrected mass of fibre C (%)
 W_A : bone dry mass of fibre A (g)
 W_B : bone dry mass of fibre B (g)
 W_C : bone dry mass of fibre C (g)
 R_A : official regain of fibre A as shown in table 1 (%)
 R_B : official regain of fibre B as shown in table 1 (%)
 R_C : official regain of fibre C as shown in table 1 (%)

The mixture ratio based on corrected mass, with the dried mass added with the fibrous matter and non-fibrous matter removed by pre-treatment, shall be obtained according to formulae (8) and (9) in a).

5.10 Confirmation

The measurements shall be carried out twice and the mean value shall be obtained. When the difference of not less than 1.5 is found in the value of mixture ratio based on bone dry mass according to the calculation method in 5.9, measurement shall be further carried out at least twice and the mean value of these measured values shall be obtained. In this case, the results of the first two measurements shall be discarded. The mean value shall be calculated to one decimal place in accordance with Rule B specified in JIS Z 8401.

6 Dissolution method

6.1 Common matters

Matters common to the test methods for determining the mixture ratio of mixtures of two or more kinds of fibre shall be as follows.

6.1.1 Application

This Standard is applicable to the test methods for determining the mixture ratio of mixtures containing two or more kinds of fibre.

The test methods specified are applicable to fibres in any textile form. Where certain textile forms are excepted, these are listed in the "Application" of the appropriate sub-clause.

6.1.2 Summary

After the identification of the components of a mixture, one component is removed, usually by selective solution, the insoluble residue is weighed, and the proportions of soluble components are calculated from the loss in mass. Generally, the fibre in the larger proportion or fibre with the least damage on residual fibre is removed first.

6.1.3 Test condition

Because bone dry mass is determined, it is unnecessary to condition the specimen. The analysis is carried out under ordinary room conditions.

6.1.4 Apparatus and instrument

The apparatus and instrument shall be as follows.

- a) **Crucible type glass filter**, with a sintered disk filter of 30 ml to 40 ml in capacity and with pore size of 90 μm to 150 μm . This filter should be provided with either a watch glass or glass ground-in stopper. In place of this crucible type glass filter, other equivalent apparatus or instrument giving identical results (hereafter referred to as "filter") may be used.
- b) **Weighing bottle**, of sufficient size capable of containing a crucible type filter or that equivalent to this.
- c) **Desiccator**, specified in JIS R 3503, containing self-indicating silica gel.
- d) **Ventilated oven**, for drying specimens at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.
- e) **Chemical balance**, with a resolution of 0.000 2 g.
- f) **Soxhlet extraction apparatus**, of sufficient size to give a volume equal to 20 times the mass, in grammes, of the sample, or any other apparatus giving equal results.
- g) **Additional apparatus**, to be specified in individual relevant clauses in this Standard.

6.1.5 Reagents

Reagents shall be as follows.

- a) **Petroleum ether**, redistilled, distilling between $30\text{ }^{\circ}\text{C}$ to $60\text{ }^{\circ}\text{C}$.
- b) **Water**, either purified water specified by Japanese Pharmacopoeia or water of quality equal to at least A2 specified in JIS K 0050.

c) **Additional reagent**, to be specified in individual relevant clauses in this Standard.

6.1.6 Sampling

Take a laboratory test sample that is representative of the laboratory bulk sample and sufficient to provide all the specimens, each of at least 1 g, that are required. Fabrics may contain yarns of different composition and account should be taken of this fact in the sampling of the fabric.

6.1.7 Pre-treatment of sample

The pre-treatment of sample shall be carried out by the method specified in 5.7. Additional pre-treatment is applied where the sample contains finishes which are insoluble in both water and petroleum ether [6.1.5 a)]. In the case of samples treated with special dye or processing, to which the operation in 6.2 would not be effective, discolouration or other treatment shall be additionally performed by a suitable method.

See Annex D for example of methods of removal of non-fibrous matters applicable where non-fibrous matters are known.

6.1.8 Procedure

The procedure shall be as follows.

- a) **In the case of binary fibre mixtures** Cut the sample, pre-treated according to 6.1.7, into suitable lengths, mix sufficiently, take approximately 1 g therefrom and obtain the bone dry mass. Then, select the operation method suitable for the composition of sample from the operation methods given in 6.2. Generally, the fibre in the larger proportion or fibre with the least damage in residual fibre is removed first. Then, carry out the treatment specified in the selected operation method to remove the soluble fibre and obtain the bone dry mass of the residue. Furthermore, obtain the composition of sample from the calculation in 6.1.10. The test methods applicable to respective combinations of fibres are shown in table CB.2.
- b) **In the case of ternary fibre mixtures** In the case of ternary fibre mixtures, the successive dissolution method in which a combination of two types of dissolution methods selected from among the operation methods in 6.2 is performed shall be applied. At this time, usually select the two operation methods by which the two fibres of larger composition are dissolved respectively or two operation methods with least damage on residual fibre. Namely, use one piece of cut specimen, remove the first dissolved part of fibre at the first stage test and obtain the total bone dry mass of residual two kinds of fibre. Then, remove the second dissolved part of fibre at the second stage test and obtain the bone dry mass of the third residual fibre. Furthermore, obtain the composition of sample from the calculation in 6.1.10. The test method corresponding to the combination of component fibres shall be shown in table CB.3. As regards mixtures of four kinds or more, carry out the test by applying this correspondingly.

The individual dissolution method in which the bone dry mass of each fibre is determined by respective dissolution methods using two or more kinds of specimens in the case of ternary fibre mixture is specified in Annex C.

6.1.9 Operation

The operation shall be as follows.

a) Common matters

- 1) **Drying** Put each of the specimens in separate weighing bottles [6.1.4 b)] and carry out all drying operations in a ventilated oven [6.1.4 d)] at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ until constant mass is achieved.
- 2) **Drying and specimen** Dry the specimen in a weighing bottle with its stopper beside it. After drying, close the weighing bottle before taking out the specimen from the oven, and then transfer the weighing bottle rapidly into the desiccators [6.1.4 c)]. The same operation shall be carried out when a similar apparatus to crucible type filter [6.1.4 a)] is used.
- 3) **Drying of crucible type filter and residue** Dry the filter with its stopper or cover beside it in the oven. After drying, close the filter and transfer it quickly to a desiccator.
- 4) **Cooling** Conduct all cooling operations until complete cooling to room temperature is attained, with the desiccators placed beside the balance [6.1.4 e)].
- 5) **Weighing** After cooling, complete the weighing of the weighing bottle or filter, to an accuracy of 0.000 2 g, within 2 min of its removal from the dessicator. The filter shall have been weighed prior to this procedure.

Do not handle the filters, specimens or residues with bare hand during the drying, cooling and weighing operations.

- b) **Test operation** Take from the pre-treated laboratory test sample a test specimen weighing about 1 g. Normally, cut yarn or dissected cloth into suitable lengths. Dry the specimen in a weighing bottle, cool it in a dessicator and weigh it. Transfer the specimen to the glass vessel specified in the appropriate clause of this Standard, reweigh the weighing bottle immediately, and obtain the dry mass of the specimen by the difference.

Carry out completely the applicable test operation specified in this Standard, examine the residue microscopically, or otherwise, as appropriate, to check that the treatment has in fact completely removed the soluble fibre.

6.1.10 Calculation

The mass of the insoluble fibre shall be expressed as a percentage of the total mass of fibre in the mixture. The bone dry mass based on a) shall be obtained by calculation of the result based on the mass (corrected mass) when the moisture corresponding to the official regain in table 1 is included in bone dry mass as shown in b) or the mass in which the moisture corresponding to the official regain in table 1 and the mass corresponding to the fibrous matter removed by the pre-treatment, and non-fibrous matter are included in bone dry mass, as shown in c).

Obtain the percentage of soluble fibre by the difference. When the correction to the soluble fibre is required, calculate according to a) 1.1). The calculation method used

shall be expressed, and in the cases of the method in **b)** and the method in **c)**, the value of mass corresponding to the percentage included shall be also expressed.

a) Method based on bone dry mass (mixture ratio based on bone dry mass)

1) In the case of binary fibre mixtures

- 1.1) In the case of carrying out the correction to dissolved fibre, formulae (13) and (14) shall be used :

$$x_A = \frac{(W - W') \times f_G}{W} \times 100 \quad \dots\dots\dots(13)$$

$$x_B = 100 - x_A \quad \dots\dots\dots(14)$$

where, x_A : mixture ratio based on bone dry mass of soluble fibre A (%)
 x_B : mixture ratio based on bone dry mass of residual fibre B (%)
 W : bone dry mass of specimen (g)
 W' : bone dry mass before correction of residual fibre B (g)
 f_G : correction factor to soluble fibre A
The value of f_G is indicated in table 3.

- 1.2) In the case of carrying out the correction to residue, formulae (15) and (16) shall be used :

$$x_A = 100 - x_B \quad \dots\dots\dots(15)$$

$$x_B = \frac{W' \times f}{W} \times 100 \quad \dots\dots\dots(16)$$

where, x_A : mixture ratio based on bone dry mass of soluble fibre A (%)
 x_B : mixture ratio based on bone dry mass of residual fibre B (%)
 W : bone dry mass of specimen (g)
 W' : W' bone dry mass before correction of residual fibre B (g)
 f : correction factor to mass change due to the reagent of residual fibre B
The value of f is indicated in table 3.

2) In the case of ternary fibre mixtures (successive dissolution method)

- 2.1) In the case of carrying out the correction to residue, formulae (17) to (19) shall be used :

$$x_A = 100 - (x_C + x_B) \quad \dots\dots\dots(17)$$

$$x_B = \frac{(W_{BC} - W' \times f_1 \times f_2) \times f_3}{W} \times 100 \quad \dots\dots\dots(18)$$

$$x_C = \frac{W' \times f_1 \times f_2}{W} \times 100 \quad \dots\dots\dots(19)$$

- where,
- x_A : mixture ratio based on bone dry mass of fibre A dissolved at the first time (%)
 - x_B : mixture ratio based on bone dry mass of fibre B dissolved at the second time (%)
 - x_C : mixture ratio based on bone dry mass of residual fibre C (%)
 - W : bone dry mass of specimen (g)
 - W' : bone dry mass before correction of residue C (g)
 - W_{BC} : sum of bone dry mass of residual fibre B and residual fibre C after dissolving fibre A (g)
 - f_1 : correction factor to mass change due to the reagent of residual fibre C in the case of dissolving fibre A
 - f_2 : correction factor to mass change due to the reagent of residual fibre C in the case of dissolving fibre B
 - f_3 : correction factor to mass change due to the reagent of residual fibre B in the case of dissolving fibre A
- The values of f_1 , f_2 and f_3 are indicated in table 3.

See table CB.3 for examples of this successive dissolution method applied for ternary fibre mixtures.

- b) **Method based on corrected mass (the mass when the moisture corresponding to the official regain in table 1 is included in bone dry mass) (mixture ratio based on corrected mass)**
- 1) **In the case of binary fibre mixtures** For the mixture ratio based on corrected mass (the mass when the moisture corresponding to the official regain in table 1 is included in bone dry mass), formulae (20) and (21) shall be used :

$$X_A = 100 - X_B \dots\dots\dots (20)$$

$$X_B = \frac{x_B \times \left(1 + \frac{a_2}{100}\right)}{x_A \times \left(1 + \frac{a_1}{100}\right) + x_B \times \left(1 + \frac{a_2}{100}\right)} \times 100 \dots\dots\dots (21)$$

- where,
- X_A : percentage of soluble fibre A containing moisture corresponding to official regain (mixture ratio based on corrected mass) (%)
 - X_B : percentage of residual fibre B containing moisture corresponding to official regain (mixture ratio based on corrected mass) (%)
 - x_A : mixture ratio based on bone dry mass of soluble fibre A (%)
 - x_B : mixture ratio based on bone dry mass of residual fibre B (%)
 - a_1 : official regain to be added to soluble fibre A (%)
 - a_2 : official regain to be added to residual fibre B (%)

The respective values of a_1 and a_2 are indicated in table 1.

- 2) **In the case of ternary fibre mixtures (applicable to both successive dissolution method and individual dissolution method)** For the mixture ratio based on corrected mass (the mass when the moisture corresponding to the official regain in table 1 is included in bone dry mass), formulae (22) and (24) shall be used :

$$X_A = \frac{x_A \times \left(1 + \frac{a_1}{100}\right)}{x_A \times \left(1 + \frac{a_1}{100}\right) + x_B \times \left(1 + \frac{a_2}{100}\right) + x_C \times \left(1 + \frac{a_3}{100}\right)} \times 100 \quad \dots\dots\dots(22)$$

$$X_B = \frac{x_B \times \left(1 + \frac{a_2}{100}\right)}{x_A \times \left(1 + \frac{a_1}{100}\right) + x_B \times \left(1 + \frac{a_2}{100}\right) + x_C \times \left(1 + \frac{a_3}{100}\right)} \times 100 \quad \dots\dots\dots(23)$$

$$X_C = \frac{x_C \times \left(1 + \frac{a_3}{100}\right)}{x_A \times \left(1 + \frac{a_1}{100}\right) + x_B \times \left(1 + \frac{a_2}{100}\right) + x_C \times \left(1 + \frac{a_3}{100}\right)} \times 100 \quad \dots\dots\dots(24)$$

where, X_A : percentage of fibre A containing moisture corresponding to official regain (mixture ratio based on corrected mass) (%)
 X_B : percentage of fibre B containing moisture corresponding to official regain (mixture ratio based on corrected mass) (%)
 X_C : percentage of fibre C containing moisture corresponding to official regain (mixture ratio based on corrected mass) (%)
 x_A : mixture ratio based on bone dry mass of fibre A (%)
 x_B : mixture ratio based on bone dry mass of fibre B (%)
 x_C : mixture ratio based on bone dry mass of fibre C (%)
 a_1 : official regain to be added to fibre A (%)
 a_2 : official regain to be added to fibre B (%)
 a_3 : official regain to be added to fibre C (%)
The respective values of a_1 , a_2 and a_3 are indicated in table 1.

- c) **Method based on the bone dry mass including the percentage of moisture and the following clause (mixture ratio based on corrected mass before treatment)**

- 1) **In the case of binary fibre mixtures** For the mixture ratio based on the bone dry mass including the percentage of water content and the following 1.1) and 1.2) (mixture ratio based on corrected mass before treatment), formulae (25) and (26) shall be used :

- 1.1) fibrous matter removed by pre-treatment
1.2) non-fibrous matter

$$X_A = 100 - X_B \dots\dots\dots (25)$$

$$X_B = \frac{x_B \times \left[1 + \frac{(a_2 + b_2)}{100} \right]}{x_A \times \left[1 + \frac{(a_1 + b_1)}{100} \right] + x_B \times \left[1 + \frac{(a_2 + b_2)}{100} \right]} \times 100 \dots\dots\dots (26)$$

- where, X_A : percentage of soluble fibre A in mixtures including the percentage of moisture and non-fibrous matter (mixture ratio based on corrected mass before treatment) (%)
- X_B : percentage of residual fibre B in mixtures including the percentage of moisture and non-fibrous matter (mixture ratio based on corrected mass before treatment) (%)
- x_A : mixture ratio based on bone dry mass of soluble fibre A (%)
- x_B : mixture ratio based on bone dry mass of residual fibre B (%)
- a_1 : official regain to be added to soluble fibre A (%)
- a_2 : official regain to be added to residual fibre B (%)
- b_1 : decrease rate of soluble fibre A produced by pre-treatment and/or percentage of non-fibrous matter to be added to soluble fibre A (%)
- b_2 : decrease rate of residual fibre B produced by pre-treatment and/or percentage of non-fibrous matter to be added to residual fibre B (%)

The respective values of a_1 and a_2 are shown in table 1.

2) In the case of ternary fibre mixtures (applicable to both successive dissolution method and individual dissolution method) For the mixture ratio based on the bone dry mass including the percentage of water content and the following 2.1) and 2.2) (mixture ratio based on corrected mass before treatment), formulae (27) to (29) shall be used:

2.1) fibrous matter removed by pre-treatment

2.2) non-fibrous matter

$$X_A = \frac{x_A \times \left[1 + \frac{(a_1 + b_1)}{100} \right]}{x_A \times \left[1 + \frac{(a_1 + b_1)}{100} \right] + x_B \times \left[1 + \frac{(a_2 + b_2)}{100} \right] + x_C \times \left[1 + \frac{(a_3 + b_3)}{100} \right]} \times 100 \dots\dots\dots (27)$$

$$X_B = \frac{x_B \times \left[1 + \frac{(a_2 + b_2)}{100} \right]}{x_A \times \left[1 + \frac{(a_1 + b_1)}{100} \right] + x_B \times \left[1 + \frac{(a_2 + b_2)}{100} \right] + x_C \times \left[1 + \frac{(a_3 + b_3)}{100} \right]} \times 100 \dots\dots\dots (28)$$

$$X_C = \frac{x_C \times \left[1 + \frac{(a_3 + b_3)}{100} \right]}{x_A \times \left[1 + \frac{(a_1 + b_1)}{100} \right] + x_B \times \left[1 + \frac{(a_2 + b_2)}{100} \right] + x_C \times \left[1 + \frac{(a_3 + b_3)}{100} \right]} \times 100 \dots\dots\dots(29)$$

where, X_A : percentage of fibre A in mixtures including the percentage of moisture and non-fibrous matter (mixture ratio based on corrected mass before treatment) (%)

X_B : percentage of fibre B in mixtures including the percentage of moisture and non-fibrous matter (mixture ratio based on corrected mass before treatment) (%)

X_C : percentage of fibre C in mixtures including the percentage of moisture and non-fibrous matter (mixture ratio based on corrected mass before treatment) (%)

x_A : mixture ratio based on bone dry mass of fibre A (%)

x_B : mixture ratio based on bone dry mass of fibre B (%)

x_C : mixture ratio based on bone dry mass of fibre C (%)

a_1 : official regain to be added to fibre A (%)

a_2 : official regain to be added to fibre B (%)

a_3 : official regain to be added to fibre C (%)

b_1 : decrease rate of fibre A produced by pre-treatment and/or percentage of non-fibrous matter to be added to fibre A (%)

b_2 : decrease rate of fibre B produced by pre-treatment and/or percentage of non-fibrous matter to be added to fibre B (%)

b_3 : decrease rate of fibre C produced by pre-treatment and/or percentage of non-fibrous matter to be added to fibre C

The respective values of a_1 , a_2 and a_3 are shown in table 1.

Where a special pre-treatment has been used in c), the values of b_1 , b_2 and b_3 should be determined by submitting each of the pure fibre constituents to pre-treatment applied in the analysis. Pure fibres are those free from all non-fibrous material except that which they normally contain (either naturally or because of the manufacturing process) in the state (unbleached, bleached) in which they are found in the material to be analysed.

6.1.11 Confirmation and precision

The confirmation and precision shall be as follows.

- a) **Confirmation of test results** The measurement shall be carried out twice and the mean of the measured values shall be obtained. It shall be confirmed that the difference exceeding 1.5 % is not found in the value of mixture ratio based on bone dry mass according to the calculation method of 6.1.10 a). When difference exceeding 1.5 % is found, two or more measurements shall be further carried out and the mean of the measured values shall be obtained. At this time, the first two test results shall be discarded. The mean value shall be calculated to one decimal place

according to Rule B of **JIS Z 8401**.

- b) **Precision of test methods** The precision indicated in each test method relates to the reproducibility. This refers to the reliability, i.e. the closeness of agreement between experimental values obtained by operators in different laboratories or at different times, using the same method on specimens of an identical, consistent mixture.

The reproducibility is expressed by confidence limits of the results for a confidence level of 95 %, i.e. the difference between two results in a series of analyses made in different laboratories would be exceeded only in five cases out of 100, when the standard method is applied to an identical consistent mixture.

6.2 Operation method

6.2.1 60 % sulfuric acid method

6.2.1.1 Application

This method is applied to the binary fibre mixtures of rayon or cupro fibre with cotton, flax (linen) or ramie fibre after removal of non-fibrous matter.

6.2.1.2 Summary

The rayon or cupro is dissolved out from the mixture of a known bone dry mass with 60 % sulfuric acid. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of rayon or cupro is obtained by difference.

6.2.1.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Erlenmeyer flask**, specified in **JIS R 3503**, 200 ml or over in capacity, provided with a glass ground-in stopper.
- b) **Mechanical shaker**, capable of shaking at 200 reciprocations per minute with 40 mm to 70 mm amplitude.
- c) **Glass beaker**, specified in **JIS R 3503** and 100 ml or over in capacity.

6.2.1.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **60 % Sulfuric acid** Pour 343 ml of concentrated sulfuric acid of approximately 1.84 g/ml density specified in **JIS K 8951** into approximately 368 ml of water gradually while cooling and stirring, until the density of 1.498 g/ml (20 °C) or 1.494 g/ml (25 °C) is obtained.

NOTE : Adding water to concentrated sulfuric acid is very dangerous; mixing should always be performed by adding concentrated sulfuric acid to water.

- b) **Diluted ammonia solution** Dilute approximately 28 % ammonia solution specified in **JIS K 8085** with water to make approximately 1 % diluted ammonia solution.

6.2.1.5 Test operation

After completing the operation specified in 6.1.9 b), proceed to the following operation.

To the specimen contained in an Erlenmeyer flask [6.2.1.3 a)], add 100 ml of 60 % sulfuric acid [6.2.1.4 a)] at 23 °C to 25 °C per gram of specimen, stopper the flask and shake vigorously by the mechanical shaker [6.2.1.3 b)] for 10 min. Leave it to stand for 5 min and then shake it for 5 min to dissolve rayon or cupro. After filtering this through the weighed filter by suction, wash the residue on the funnel first with 60 % sulfuric acid of the same amount and the same temperature as before and then with water. Then, transfer this into another glass beaker [6.2.1.3 c)] and neutralize it with approximately 50 ml per gram of specimen of diluted ammonia solution (approximately 1 %) [6.2.1.4 b)]. After filtering this again through the filter by suction, wash the residue on the funnel with water. Dry, cool and weigh the filter and the residue.

NOTE : Adding water to sulfuric acid is very dangerous; when washing with water is performed after filtering by suction, make sure that the suction bottle is replaced with an empty one or the content is discarded prior to washing.

6.2.1.6 Calculation

Calculate the results as specified in 6.1.10. The values of the correction factor f shall be as given in table 3.

6.2.1.7 Confirmation

Confirmation shall be in accordance with 6.1.11 a).

6.2.2 70 % Sulfuric acid method

6.2.2.1 Application

This method is applied to the binary fibre mixtures of cotton, flax (linen), ramie, rayon, cupro or triacetate fibre with wool, animal hair, polyvinylchloride, polyester, modacrylic, polypropylene, benzoate, aramid or polylactide fibre, the binary fibre mixtures of vinylon fibre with polyester, polypropylene, benzoate, aramid or polylactide fibre and the binary fibre mixtures of acetate fibre with polyvinyl chloride, modacrylic or polylactide fibre after removal of non-fibrous matter.

6.2.2.2 Summary

Cotton, flax (linen), ramie, rayon, cupro, acetate, triacetate or vinylon is dissolved out from the mixture of a known bone dry mass with 70 % sulfuric acid. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of bone dry mass of the mixture. The percentage of cotton, flax (linen), ramie, rayon, cupro, acetate, triacetate or vinylon is obtained by difference.

6.2.2.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Erlenmeyer flask**, specified in JIS R 3503, 200 ml or over in capacity and provided

with a glass ground-in stopper.

- b) **Mechanical shaker**, capable of shaking at reciprocation of 200 cycles per minute with 40 mm to 70 mm amplitude.
- c) **Glass beaker**, specified in **JIS R 3503** and 100 ml or over in capacity.

6.2.2.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **70 % Sulfuric acid** Add gradually 400 ml of concentrated sulfuric acid of approximately 1.84 g/ml density specified in **JIS K 8951**, while cooling and stirring, to approximately 267 ml of water until the density of 1.610 g/ml (20 °C) or 1.606 g/ml (25 °C) is obtained.

NOTE : Adding water to concentrated sulfuric acid is very dangerous; mixing should always be performed by adding concentrated sulfuric acid to water.

- b) **Diluted ammonia solution** Dilute approximately 28 % ammonia solution specified in **JIS K 8085** with water to make approximately 1 % diluted ammonia solution.

6.2.2.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

To the specimen contained in an Erlenmeyer flask [**6.2.2.3 a)**], add 100 ml of 70 % sulfuric acid [**6.2.2.4 a)**] at 23 °C to 25 °C per gram of specimen, stopper the flask and shake it by mechanical shaker [**6.2.2.3 b)**] at least for 10 min to dissolve cotton, flax (linen), ramie, rayon, cupro, acetate, triacetate or vinylon. After filtering this through the weighed filter by means of suction, wash the residue on funnel first with 70 % sulfuric acid of the same amount and the same temperature as before and then with water. Then, transfer this to another glass beaker [**6.2.2.3 c)**], and neutralize with approximately 50 ml per gram of specimen of diluted ammonia solution (approximately 1 %) [**6.2.2.4 b)**]. Filter this again through the filter by suction, and wash the residue on the funnel with water. Dry, cool and weigh the filter and residue.

NOTE : Adding water to sulfuric acid is very dangerous; when washing with water is performed after filtering by suction, make sure that the suction bottle is replaced with an empty one or the content is discarded prior to washing.

6.2.2.6 Calculation

Calculate the results as specified in **6.1.10**. The values of the correction factor f shall be as given in table 3.

6.2.2.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.3 Warm 75 % sulfuric acid method

6.2.3.1 Application

This method is applied to the binary fibre mixtures of natural or regenerated cellulose fibre with polyester fibre after removal of non-fibrous matter.

6.2.3.2 Summary

Cellulose fibre is dissolved out from the mixture of a known bone dry mass with 75 % sulfuric acid. The residue is collected, washed, dried and weighed. The mass is corrected and expressed as a percentage of the bone dry mass of the mixture. The percentage of cellulose fibre is obtained by difference.

6.2.3.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Erlenmeyer flask**, specified in **JIS R 3503**, 200 ml or over in capacity and provided with a glass ground-in stopper.
- b) **Heating apparatus**, capable of maintaining the temperature of flask at $50^{\circ}\text{C} \pm 5^{\circ}\text{C}$ (e.g. water bath with a thermostat).

6.2.3.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **75 % Sulfuric acid** Prepare by adding carefully, while cooling, 700 ml of concentrated sulfuric acid of approximately 1.84 g/ml density specified in **JIS K 8951** to 350 ml of water. After cooling this solution to room temperature, dilute with water to make 1 L. The concentration shall be in the range of 73 % to 77 % sulfuric acid.

NOTE : Adding water to concentrated sulfuric acid is very dangerous; mixing should always be performed by adding concentrated sulfuric acid to water.

- b) **Diluted ammonia solution** Dilute approximately 28 % ammonia solution specified in **JIS K 8085** with water to make approximately 1 % ammonium diluted solution.

6.2.3.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

To the specimen contained in an Erlenmeyer flask [**6.2.3.3 a)**], add 200 ml of 75 % sulfuric acid [**6.2.3.4 a)**] per gram of specimen. Stopper the flask and shake carefully for moisturizing the specimen. Maintain the Erlenmeyer flask at the temperature of $50^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 1 h, while, at approximate intervals of 10 min, shake the Erlenmeyer flask and the contents gently. Filter the contents in the Erlenmeyer flask through the weighed filter by suction. Rinse the Erlenmeyer flask with a small amount of 75 % sulfuric acid, and transfer all residue to the filter. After filtering the filter by suction, fill the filter with fresh 75 % sulfuric acid and wash the residue. Do not filter before the solution has completely drained through the filter or 1 min has elapsed. Wash the residue with cold water several times continuously, wash it twice with diluted ammonia solution [**6.2.3.4 b)**], and then wash it further with water to complete rinsing. Wash the filter by filtering by suction at every process. Do not apply suction until each washing solution has drained through the filter. After final filtering by suction, dry,

cool and weigh the filter and the residue.

NOTE : Adding water to sulfuric acid is very dangerous; when washing with water is performed after filtering by suction, make sure that the suction bottle is replaced with an empty one or the content is discarded prior to washing.

6.2.3.6 Calculation

Calculate the results as specified in 6.1.10. The value of the correction factor f shall be as given in table 3.

6.2.3.7 Confirmation

Confirmation shall be in accordance with 6.1.11 a).

6.2.3.8 Precision

For homogeneous mixtures of textile materials, the confidence limits of the results obtained by this method are not greater than ± 1 % for the confidence level of 95 %

6.2.4 75 % Sulfuric acid method (room temperature)

6.2.4.1 Application

This method is applied to the binary fibre mixtures of silk, and wool or animal hair after removal of non-fibrous matter.

6.2.4.2 Summary

Silk is dissolved out from the mixture of a known bone dry mass with 75 % sulfuric acid ²⁾. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of silk is obtained by difference.

Note ²⁾ The wild silk such as tussah silk is not completely dissolved with 75 % sulfuric acid.

6.2.4.3 Additional apparatus

Erlenmeyer flask, specified in JIS R 3503, 200 ml or over in capacity and provided with a glass ground-in stopper.

6.2.4.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **75 % Sulfuric acid** Prepare by adding carefully, while cooling, 700 ml of concentrated sulfuric acid of approximately 1.84 g/ml density specified in **JIS K 8951** to 350 ml of water. After cooling this solution to room temperature, dilute with water to make 1 L. The concentration in the range of 73 % to 77 % sulfuric acid is permissible.

NOTE : Adding water to concentrated sulfuric acid is very dangerous; mixing should always be performed by adding concentrated sulfuric acid to water.

- b) **Diluted sulfuric acid** Add gradually 100 ml of concentrated sulfuric acid of ap-

proximately 1.84 g/ml density specified in **JIS K 8951** to 1 900 ml of water.

- c) **Diluted ammonia solution** Dilute approximately 28 % concentrated ammonia solution specified in **JIS K 8085** with water to make approximately 1 % ammonia diluted solution.

6.2.4.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

To the specimen contained in an Erlenmeyer flask (**6.2.4.3**), add 100 ml of 75 % sulfuric acid [**6.2.4.4 a)**] per gram of specimen, stopper the flask, shake vigorously (preferably by means of a mechanical shaker) and leave it to stand in vertical position at room temperature for 30 min. Shake it again and leave it to stand for another 30 min. Shake it for the final time, and filter the contents in the Erlenmeyer flask by pouring it through a weighed filter. Rinse the residual fibre in the Erlenmeyer flask with a small amount of 75 % sulfuric acid. Filter it by suction, and wash the residue on the filter with 50 ml of diluted sulfuric acid solution [**6.2.4.4 b)**], 50 ml of water [**6.1.5 b)**] and 50 ml of diluted ammonia solution [**6.2.4.4 c)**] continuously. At each process of the operation, leave the residue to stand for about 10 min before suction so as to allow the residue to be fully immersed in the solution. Finally, carry out the rinsing, and immerse the residue in water for 30 min. After filtering the filter by suction, dry, cool and weigh the filter and the residue.

NOTE : Adding water to sulfuric acid is very dangerous; when washing with water is performed after filtering by suction, make sure that the suction bottle is replaced with an empty one or the content is discarded prior to washing.

6.2.4.6 Calculation

Calculate the results as specified in **6.1.10**. The value of the correction factor f shall be as given in table 3.

6.2.4.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.4.8 Precision

For homogeneous mixtures of textile materials, the confidence limits of the results obtained by this method shall be not greater than ± 1 % for the confidence level of 95 %.

6.2.5 80 % Sulfuric acid method

6.2.5.1 Application

This method is applied to the binary fibre mixtures of polyurethane fibre with polyester or polylactide fibre after removal of the non-fibrous matter.

6.2.5.2 Summary

The polyurethane is dissolved out from the mixture of a known bone dry mass with 80 % sulfuric acid. The residue is collected, washed, dried and weighed. The mass is

corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of polyurethane is obtained by difference.

NOTE : The solubility of polyurethane is different depending on the type.

6.2.5.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Erlenmeyer flask**, specified in **JIS R 3503**, 200 ml or over in capacity and provided with a glass ground-in stopper.
- b) **Mechanical shaker**, capable of shaking at 200 reciprocations per minute with 40 mm to 70 mm amplitude.
- c) **Glass beaker**, specified in **JIS R 3503**, 100 ml or over in capacity.

6.2.5.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **80 % Sulfuric acid** Pour gradually, while cooling and stirring, 460 ml of concentrated sulfuric acid of approximately 1.84 g/ml density specified in **JIS K 8951** in approximately 185 ml of water, until the density of 1.727 g/ml (20 °C) or 1.722 g/ml (25 °C) is obtained.

NOTE : Adding water to concentrated sulfuric acid is very dangerous; mixing should always be performed by adding concentrated sulfuric acid to water.

- b) **Diluted ammonia solution** Dilute approximately 28 % concentrated ammonia solution specified in **JIS K 8085** with water to make approximately 1 % ammonia diluted solution.

6.2.5.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

To the specimen contained in an Erlenmeyer flask [**6.2.5.3 a)**], add 100 ml of 80 % sulfuric acid [**6.2.5.4 a)**] at 23 °C to 25 °C per gram of specimen, stopper the flask and shake it by the mechanical shaker [**6.2.5.3 b)**] for at least 10 min and dissolve polyurethane. After filtering this through the weighed filter by suction, wash the residue on funnel first with 80 % sulfuric acid of the same amount and the same temperature as before and then with water. Then, transfer this to another glass beaker [**6.2.5.3 c)**] and neutralize with approximately 50 ml per gram of specimen of diluted ammonia solution (approximately 1 %) [**6.2.5.4 b)**]. After filtering this through the filter by suction again, wash the residue on funnel with water. Dry, cool and weigh the filter and the residue.

NOTE : Adding water to sulfuric acid is very dangerous; when washing with water is performed after filtering by suction, make sure that the suction bottle is replaced with an empty one or the content is discarded prior to washing.

6.2.5.6 Calculation

Calculate the results as specified in **6.1.10**. The value of the correction factor f shall be as given in table 3.

6.2.5.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.6 Concentrated sulfuric acid method I

6.2.6.1 Application

This method is applied to the binary fibre mixtures of the chlorofiber of chlorination treated goods or untreated goods based on single polymerization of vinyl chloride with cotton, rayon, cupro, acetate, triacetate, nylon, polyester, certain acrylic fibres or certain modacrylic fibres after removal of non-fibrous matter.

NOTE : Certain modacrylic fibres means that which is dissolved when immersed in concentrated sulfuric acid of 1.84 g/ml density.

This method is applied in place of the methods described in **6.2.27** and **6.2.35** when it is clarified that chlorofibre is not completely dissolved in hot dimethyl formamide solution or the azeotropic mixture solution of carbon disulfide acetone in the preliminary test.

6.2.6.2 Summary

The constituent fibres other than chlorofibre are dissolved out from the mixture of a known bone dry mass with concentrated sulfuric acid of approximately 1.84 g/ml density. The residue composed of chlorofibre is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of the constituent fibre other than chlorofibre is obtained by difference.

6.2.6.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Erlenmeyer flask**, specified in JIS R 3503, is not less than 200 ml in capacity and is provided with a glass ground-in stopper.
- b) **Glass rod**, having a flat tip.

6.2.6.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **Concentrated sulfuric acid** Use the concentrated sulfuric acid of approximately 1.84 g/ml density specified in JIS K 8951 as it is without dilution.
- b) **50 % Diluted sulfuric acid**
- c) **Diluted ammonia solution** Dilute approximately 28 % concentrated ammonia solution specified in JIS K 8085 with water to make approximately 1 % ammonia diluted solution.

6.2.6.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

To the specimen contained in the Erlenmeyer flask [**6.2.6.3 a)**], add 100 ml of the concentrated sulfuric acid [**6.2.6.4 a)**] per gram of specimen. Allow the contents of the flask to remain at room temperature for 10 min, and during that time, stir the test specimen occasionally by means of the glass rod [**6.2.6.3 b)**]. If a woven or knitted fabric is being treated, wedge it between the wall of flask and the glass rod and exert a light pressure in order to separate the material dissolved by the concentrated sulfuric acid.

Decant the liquid in the Erlenmeyer flask through the weighed filter. Add a fresh portion of concentrated sulfuric acid to the flask and repeat the same operation. Transfer the contents of the flask to the filter, and transfer the fibrous residue there with the aid of the glass rod. If necessary, add a little concentrated sulfuric acid to the flask in order to remove any fibres adhering to the wall. Drain the filter using suction. After emptying the flask, wash the residue in the filter successively with the 50 % sulfuric acid solution [**6.2.6.4 b)**], water, the diluted ammonia solution [**6.2.6.4 c)**], draining the filter using suction after each addition until the water drained from the filter is neutral when tested by a litmus paper. Do not apply suction during the washing operation, but only after the liquid has drained through the filter. Finally, dry the filter and residue, then cool and weigh them.

NOTE : Adding water to concentrated sulfuric acid is very dangerous; when washing with water is performed after filtering by suction, make sure that the suction bottle is replaced with an empty one or the content is discarded prior to washing.

6.2.6.6 Calculation

Calculate the results as specified in **6.1.10**. The value of the correction factor f shall be as given in table 3.

6.2.6.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.6.8 Precision

For homogeneous mixtures of textile materials, the confidence limits of the results obtained by this method are not greater than ± 1 % for the confidence level of 95 %.

6.2.7 Concentrated sulfuric acid method II

6.2.7.1 Application

This method is applied to the binary fibre mixture of polyester fibre with polypropylene fibre after removal of non-fibrous matter.

6.2.7.2 Summary

The polyester fibre is dissolved out from the mixture of a known bone dry mass with concentrated sulfuric acid. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of polyester fibre is obtained by difference.

6.2.7.3 Additional apparatus

Glass beaker, specified in **JIS R 3503** and is not less than 100 ml in capacity.

6.2.7.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **Concentrated sulfuric acid** Use the concentrated sulfuric acid of approximately 1.84 g/ml in density specified in **JIS K 8951** as it is without dilution.
- b) **Diluted ammonia solution** Dilute approximately 28 % concentrated ammonia solution specified in **JIS K 8085** with water to make approximately 1 % ammonia diluted solution.
- c) **70 % Sulfuric acid**, specified in **6.2.2.4 a)**.

6.2.7.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

Take the specimen in a glass beaker (**6.2.7.3**), add to this 100 ml of concentrated sulfuric acid [**6.2.7.4 a)**] at 23 °C to 25 °C per gram of specimen, and stir it occasionally for 10 min to dissolve polyester fibre. After filtering this through the weighed crucible type filter by suction, wash the residue on funnel first with 70 % concentrated sulfuric acid [**6.2.7.4 c)**] of the same amount and the same temperature as before and then with water. Then, transfer this to another glass beaker, neutralize it by adding about 50 ml of approximately 1 % diluted ammonia solution [**6.2.7.4 b)**] per gram of specimen, and after filtering this through the filter by suction again, wash the residue on funnel with water. Dry, cool and weigh the filter and the residue.

NOTE : Adding water to concentrated sulfuric acid is very dangerous; when washing with water is performed after filtering by suction, make sure that the suction bottle is replaced with an empty one or the content is discarded prior to washing.

6.2.7.6 Calculation

Calculate the results as specified in **6.1.10**. The value of the correction factor f shall be as given in table 3.

6.2.7.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.8 20 % Hydrochloric acid method

6.2.8.1 Application

This method is applied to the binary fibre mixtures of nylon or formalized vinylon fibre with cotton, wool, animal hair, flax (linen), ramie, rayon, cupro, promix, polyvinyl chloride, polyester, acrylic, modacrylic, polypropylene, polyurethane, benzoate, aramid or polylactide fibre after removal of non-fibrous matter. This method is applicable to formalized vinylon only when it has been confirmed that vinylon can be completely dissolved.

6.2.8.2 Summary

The nylon or formalized vinylon fibre is dissolved out from the mixture of a known bone dry mass with 20 % hydrochloric acid. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of nylon or formalized vinylon fibre is obtained by difference.

6.2.8.3 Additional apparatus

Glass beaker, specified in **JIS R 3503** and is not less than 100 ml in capacity.

6.2.8.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **20 % Hydrochloric acid** Dilute hydrochloric acid specified in **JIS K 8180** with approximately the same amount of water so that the density of 1.102 g/ml (20 °C) can be obtained.
- b) **Diluted ammonia solution** Dilute approximately 28 % concentrated ammonia solution specified in **JIS K 8085** with water to make approximately 1 % ammonia diluted solution.

6.2.8.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

Take the specimen in a glass beaker (**6.2.8.3**), add to this 100 ml of 20 % hydrochloric acid [**6.2.8.4 a)**] at room temperature per gram of specimen, and stir it occasionally for 15 min to dissolve vinylon or nylon fibre. After filtering this through the weighed filter by suction, wash the residue on funnel first with 20 % hydrochloric acid of the same amount and the same temperature as before and then with water. Then, transfer this to another glass beaker, neutralize it by adding about 50 ml of approximately 1 % diluted ammonia solution [**6.2.8.4 b)**] per gram of specimen, and after filtering this through the filter again by suction, wash the residue on funnel with water. Dry, cool and weigh the filter and the residue.

6.2.8.6 Calculation

Calculate the results as specified in **6.1.10**. The value of the correction factor f shall be as given in table 3.

6.2.8.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.9 35 % Hydrochloric acid method

6.2.9.1 Application

This method is applied to the binary fibre mixtures of silk with wool, animal hair, aramid or polylactide fibre after removal of non-fibrous matter.

6.2.9.2 Summary

The silk is dissolved out from the mixture of a known bone dry mass with 35 % hydrochloric acid. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of silk is obtained by difference.

6.2.9.3 Additional apparatus

Glass beaker, specified in **JIS R 3503** and not less than 100 ml in capacity.

6.2.9.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **35 % Hydrochloric acid** Dilute hydrochloric acid with water to make the density 1.177 g/ml (20 °C).
- b) **Diluted ammonia solution** Dilute approximately 28 % concentrated ammonia solution specified in **JIS K 8085** with water to make approximately 1 % ammonia diluted solution.

6.2.9.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

Take the specimen in a glass beaker (**6.2.9.3**), add to this 100 ml of 35 % hydrochloric acid [**6.2.9.4 a)**] at room temperature per gram of specimen, and stir it occasionally for 15 min to dissolve silk. After filtering this through the weighed filter by suction, wash the residue on funnel first with 35 % hydrochloric acid of the same amount and the same temperature as before and then with water. Then, transfer this to another glass beaker, neutralize it by adding about 50 ml of approximately 1 % diluted ammonia solution [**6.2.9.4 b)**] per gram of specimen, and after filtering this through the filter again by suction, wash the residue on funnel with water. Dry, cool and weigh the filter and the residue.

6.2.9.6 Calculation

Calculate the results as specified in **6.1.10**. The value of the correction factor f shall be as given in table 3.

6.2.9.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.10 Mixed acid method

6.2.10.1 Application

This method is applied to the binary fibre mixtures of mercerized cotton with polynosic fibre after removal of non-fibrous matter.

6.2.10.2 Summary

The polynosic fibre is dissolved out from the mixture of a known bone dry mass with mixed acid. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The

percentage of polynosic fibre is obtained by difference.

6.2.10.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Erlenmeyer flask**, specified in **JIS R 3503**, not less than 200 ml in capacity and is provided with a glass ground-in stopper.
- b) **Mechanical shaker**, capable of shaking at 200 reciprocations per minute with 40 mm to 70 mm amplitude.
- c) **Glass beaker**, specified in **JIS R 3503** and not less than 100 ml in capacity.

6.2.10.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **Mixed acid solution** Measure out accurately 800 ml of 35 % hydrochloric acid specified in **JIS K 8180** at approximately 15 °C in a beaker and add gradually 200 ml of 70 % sulfuric acid at the same temperature while stirring.
- b) **Diluted ammonia solution** Dilute approximately 28 % concentrated ammonia solution specified in **JIS K 8085** with water to make approximately 1 % ammonia diluted solution.

6.2.10.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation. At this time, unravel the specimen into yarns and cut to an approximate length of 1 cm. If yarns are cut to a smaller length than this, the damage rate of cotton may increase. To the specimen contained in an Erlenmeyer flask with a glass ground-in stopper [**6.2.10.3 a)**], add 100 ml of the mixed acid solution [**6.2.10.4 a)**] at approximately 23 °C to 25 °C per gram of specimen, and shake it by the mechanical shaker [**6.2.10.3 b)**] for 10 min to dissolve polynosic fibre. After filtering this through the weighed filter by suction, wash the residue on funnel first with the mixed acid solution of the same amount and the same temperature as before and then with water. Then, transfer this to another glass beaker [**6.2.10.3 c)**], neutralize it by adding about 50 ml of approximately 1 % diluted ammonia solution [**6.2.10.4 b)**] per gram of specimen, and after filtering this through the filter by suction again, wash the residue on funnel with water. Dry, cool and weigh the filter and the residue.

6.2.10.6 Calculation

Calculate the results as specified in **6.1.10**. The value of the correction factor f shall be as given in table 3.

6.2.10.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.11 2.5 % Sodium hydroxide method

6.2.11.1 Application

This method is applied to the binary fibre mixtures of wool or animal hair with poly-

ester or acrylic fibre after removal of non-fibrous matter.

6.2.11.2 Summary

Wool or animal hair is dissolved out from the mixture of a known bone dry mass with 2.5 % sodium hydroxide. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of wool or animal hair is obtained by difference.

6.2.11.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Heating apparatus**, capable of maintaining the temperature of the glass beaker in the range between 95 °C and 100 °C (e.g. a water bath with a thermostat).
- b) **Glass beaker**, specified in **JIS R 3503** and is not less than 100 ml in capacity.

6.2.11.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **2.5 % Sodium hydroxide solution** Weigh out 25.7 g of sodium hydroxide specified in **JIS K 8576**, and dissolve it in water to make approximately 1 L so that the density is 1.026 g/ml (20 °C).
- b) **Diluted hydrochloric acid** Dilute hydrochloric acid (approximately 35 %) specified in **JIS K 8180** with water and make approximately 2 % diluted hydrochloric acid.

6.2.11.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

Take the specimen in a glass beaker [**6.2.11.3 b)**], add to this 50 ml of 2.5 % sodium hydroxide solution [**6.2.11.4 a)**] per gram of specimen, and stir it occasionally for 20 min in a boiling water to dissolve wool or animal hair. After filtering this through the weighed filter by suction, wash the residue on funnel with warm water. Then, transfer this to another glass beaker, treat it with about 25 ml of approximately 2 % diluted hydrochloric acid [**6.2.11.4 b)**] per gram of specimen, and after filtering this through the filter by suction again, wash the residue on funnel with water. Dry, cool and weigh the filter and residue.

6.2.11.6 Calculation

Calculate the results as specified in **6.1.10**. The value of the correction factor f shall be as given in table 3.

6.2.11.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.12 5 % Sodium hydroxide method

6.2.12.1 Application

This method is applied to the binary fibre mixtures of wool, animal hair or silk with

cotton, rayon, cupro, nylon, vinylon, polypropylene, polychlal or aramid fibre after removal of non-fibrous matter.

6.2.12.2 Summary

Wool, animal hair or silk is dissolved out from the mixture of a known bone dry mass with 5 % sodium hydroxide. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of wool, animal hair or silk is obtained by difference.

6.2.12.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Heating apparatus**, capable of maintaining the temperature of the glass beaker in the range between 95 °C and 100 °C (e.g. water bath with a thermostat).
- b) **Glass beaker**, specified in **JIS R 3503** and not less than 100 ml in capacity.

6.2.12.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **5 % Sodium hydroxide solution** Weigh out 52.8 g of sodium hydroxide specified in **JIS K 8576**, dissolve it in water to make approximately 1 L so that the density is 1.054 g/ml (20 °C).
- b) **Diluted hydrochloric acid** Dilute approximately 35 % hydrochloric acid specified in **JIS K 8180** with water to make approximately 2 % diluted hydrochloric acid solution.

6.2.12.5 Test operation

After completing the operation described in **6.1.9 b)**, proceed to the following operation.

Take the specimen in a glass beaker [**6.2.12.3 b)**], add to this 50 ml of 5 % sodium hydroxide solution [**6.2.12.4 a)**] per gram of specimen, stir it occasionally for 15 min in a boiling water bath to dissolve wool, animal hair or silk. After filtering this through the weighed crucible type filter by suction, wash the residue on funnel with warm water. Then, transfer this to another glass beaker, treat it with about 25 ml of approximately 2 % diluted hydrochloric acid [**6.2.12.4 b)**] per gram of specimen, and after filtering this through the filter by suction again, wash the residue on funnel with water. Dry, cool and weigh the filter and residue.

6.2.12.6 Calculation

Calculate the results as specified in **6.1.10**. The value of the correction factor f shall be as given in table 3.

6.2.12.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.13 30 % Sodium hydroxide method

6.2.13.1 Application

This method is applied to the binary fibre mixtures of polyester fibre with benzoate fibre after removal of non-fibrous matter.

6.2.13.2 Summary

The polyester fibre is dissolved out from the mixture of a known bone dry mass with 30 % sodium hydroxide. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of polyester fibre is obtained by difference.

6.2.13.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Heating apparatus**, capable of maintaining the temperature of the glass beaker in the range between 95 °C and 100 °C (e.g. water bath with a thermostat).
- b) **Glass beaker**, specified in **JIS R 3503** and is not less than 100 ml in capacity.

6.2.13.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **30 % Sodium hydroxide solution** Weigh out 400 g of sodium hydroxide specified in **JIS K 8576**, dissolve it in water to make up to approximately 1 L and so that the density is 1.328 g/ml (20 °C).
- b) **Diluted hydrochloric acid solution** Dilute approximately 35 % hydrochloric acid specified in **JIS K 8180** with water to make approximately 2 % diluted hydrochloric acid solution.

6.2.13.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation. Take the sample in a glass beaker [**6.2.13.3 b)**], add to this 200 ml of 30 % sodium hydroxide solution [**6.2.13.4 a)**] per gram of specimen, and stir it occasionally for 30 min in a boiling water bath to hydrolyze polyester fibre. Then add 600 ml of boiling water per gram of specimen, stir it and dissolve. After filtering this through the weighed filter by suction, wash the residue on funnel with hot water. Transfer this to another glass beaker, treat it with about 25 ml of approximately 2 % diluted hydrochloric acid [**6.2.13.4 b)**] per gram of specimen, and after filtering this through the filter by suction again, wash the residue on funnel with water. Dry, cool and weigh the filter and residue.

6.2.13.6 Calculation

Calculate the results as specified in **6.1.10**. The correction factor value of f shall be 1.07.

6.2.13.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.14 Cuprammonium method

6.2.14.1 Application

This method is applied to the binary fibre mixtures of cotton, flax (linen), ramie, rayon or cupro fibre with promix, vinylon, polyvinyl chloride, polyester, acrylic, modacrylic, polypropylene, polychlal, benzoate or aramid fibre, and the binary fibre mixtures of silk with promix fibre after removal of non-fibrous matter.

6.2.14.2 Summary

The cotton, silk, flax (linen), ramie, rayon or cupro fibre is dissolved out from the mixture of a known bone dry mass with cuprammonium solution. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of cotton, silk, flax (linen), ramie, rayon or cupro fibre is obtained by difference.

6.2.14.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Erlenmeyer flask**, specified in **JIS R 3503**, not less than 200 ml in capacity and provided with a glass ground-in stopper.
- b) **Mechanical shaker**, capable of shaking at 200 reciprocations per minute with 40 mm to 70 mm amplitude.
- c) **Glass beaker**, specified in **JIS R 3503** and not less than 100 ml in capacity.
- d) **Glass filter**, of pore size symbol 1 specified in **JIS R 3503**.

6.2.14.4 Additional reagents

The additional reagents to be used shall be as follows.

a) Preparation of cuprammonium solution

- 1) Put pure copper piece, approximately 28 % cooled ammonia solution specified in **JIS K 8085** and 10 g of saccharose per litre of the ammonia solution in the preparation device for cupro ammonium solution as shown in figure 1, feed air while using light suction to prepare the solution in several hours, and preserve it in a cool and dark place. Leave it to stand under this condition for at least 10 h and take the supernatant.

This solution contains $13 \text{ g/L} \pm 2 \text{ g/L}$ of Cu and not less than 200 g/L of NH_3 .

- 2) Alternatively, put 25 g of copper hydroxide in a 1 L narrow-mouthed glass bottle, add 1 L of 25 % ammonia solution, seal hermetically with a rubber stopper, shake it gently to dissolve and leave it to stand in a cool place for a while. Take the supernatant by using a glass filter [6.2.14.3 d)] without suction.

This solution contains $7.5 \text{ g/L} \pm 0.5 \text{ g/L}$ of Cu and not less than 200 g/L of NH_3 .

3) Determination of copper and ammonia in cuprammonium solution

- 3.1) **Determination of copper (method to quantify as copper oxide)** Take 10 ml of test solution in an evaporation dish, evaporate ammonia on a boiling water bath and after removing moisture on a sand pan or in an oven, incandesce it for 3 h to

5 h and quantify copper as copper oxide. At this time, 0.001 g of copper oxide is equivalent to 0.000 8 g of copper.

- 3.2) **Determination of ammonia** Add approximately 50 ml of water to 1 ml of test solution, and titrate it with 0.5 mol/L sulfuric acid with 0.02 % solution of methyl red as an indicator. At this time, 1 ml of 0.5 mol/ L sulfuric acid is equivalent to 0.017 g of ammonia.

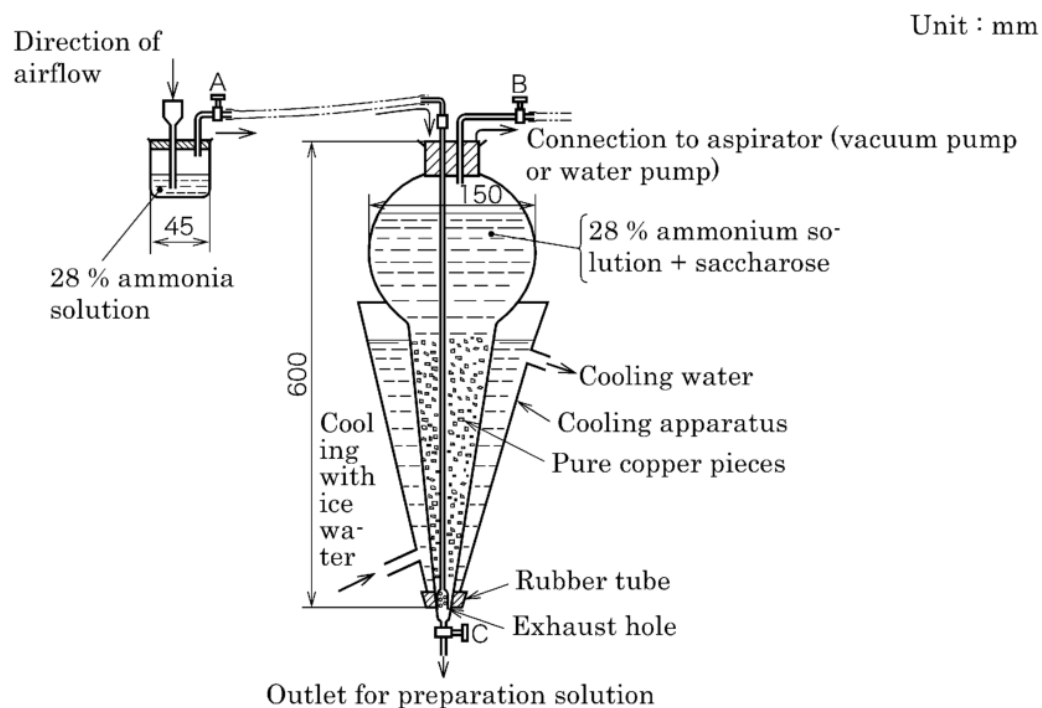


Figure 1 Preparation device for cuprammonium solution

- b) **Diluted ammonia solution** Dilute approximately 28 % concentrated ammonia solution specified in **JIS K 8085** with water to make approximately 1 % ammonia diluted solution.
- c) **Diluted hydrochloric acid solution** Dilute approximately 35 % hydrochloric acid specified in **JIS K 8180** with water to make approximately 2 % diluted hydrochloric acid solution.

6.2.14.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

If the specimen is difficult to dissolve with cuprammonium solution [**6.2.14.4 a)**], in advance treat it with 50 ml of 5 % sodium hydroxide solution per gram of specimen in a boiling water bath for 15 min, then wash it with water and dehydrate. This treatment cannot be applied to the mixtures of silk.

To the specimen contained in an Erlenmeyer flask [**6.2.14.3 a)**], add 100 ml of cuprammonium solution approximately at 18 °C to 22 °C per gram of specimen, shake it

vigorously by the mechanical shaker [6.2.14.3 b)] for 30 min to dissolve cotton, flax (linen), ramie, cupro, rayon or silk. After filtering this through the weighed filter by suction, wash the residue on the funnel with the cuprammonium solution of the same amount, the same temperature and the same quality as before, then with 50 ml of approximately 1 % diluted ammonia solution [6.2.14.4 b)] per gram of specimen, and then with sufficient amount of water successively. Then, transfer this to another glass beaker [6.2.14.3 c)], treat it with about 25 ml of approximately 2 % diluted hydrochloric acid [6.2.14.4 c)] per gram of specimen, and after filtering this through the filter by suction again, wash the residue on the funnel with water. Dry, cool and weigh the filter and residue.

6.2.14.6 Calculation

Calculate the results as specified in 6.1.10. The value of correction factor f shall be as specified in table 3.

6.2.14.7 Confirmation

Confirmation shall be in accordance with 6.1.11 a).

6.2.15 Glacial acetic acid method

6.2.15.1 Application

This method is applied to the binary fibre mixtures of acetate fibre with certain chlorofibre or chlorinated chlorofibre after removal of non-fibrous matter.

6.2.15.2 Summary

The acetate fibre is dissolved out from the mixture of a known dry mass with glacial acetic acid. The residue is collected, washed, dried and weighed. The mass is expressed as a percentage of the bone dry mass of the mixture. The percentage of acetate fibre is obtained by difference.

6.2.15.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Erlenmeyer flask**, specified in JIS R 3503, not less than 200 ml in capacity and provided with a glass ground-in stopper.
- b) **Mechanical shaker**, capable of shaking at 200 reciprocations per minute with 40 mm to 70 mm amplitude.

6.2.15.4 Additional reagent

Glacial acetic acid, distilling between 117 °C to 119 °C.

NOTE : The toxic effects of this reagent shall be borne in mind and full precautions shall be taken during use.

6.2.15.5 Test operation

After completing the operation specified in 6.1.9 b), proceed to the following operation.

To the specimen contained in an Erlenmeyer flask [6.2.15.3 a)], add 100 ml of glacial acetic acid (6.2.15.4) per gram of specimen, stopper the flask and shake it by mechanical shaker [6.2.15.3 b)] for 20 min. Pour the supernatant in the Erlenmeyer flask into the weighed filter. Repeat this operation twice by using a fresh 100 ml of glacial acetic acid each time so that it is performed 3 times in total. Then, transfer the residue to the filter and perform suction. Rinse the residue and filter with 100 ml of glacial acetic acid, and rinse further with water 3 times. After each rinsing and before performing suction, allow the solution to drop through the filter for 2 min. Dry, cool and weigh the filter and residue.

6.2.15.6 Calculation

Calculate the results as specified in 6.1.10. The value of correction factor f shall be 1.00.

6.2.15.7 Confirmation

Confirmation shall be in accordance with 6.1.11 a).

6.2.15.8 Precision

For homogeneous mixtures of textile materials, the confidence limits of the results obtained by this method are not greater than ± 1 % for the confidence level of 95 %.

6.2.16 Hot glacial acetic acid method

6.2.16.1 Application

This method is applied to the binary fibre mixtures of nylon fibre with formalized vinylon or aramid fibre after removal of non-fibrous matter.

6.2.16.2 Summary

The nylon fibre is dissolved out from the mixture of a known bone dry mass with hot glacial acetic acid. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of nylon fibre is obtained by difference.

6.2.16.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Heating apparatus**, capable of maintaining the temperature of the glass beaker in the range between 95 °C and 100 °C (e.g. a water bath with a thermostat).
- b) **Glass beaker**, specified in JIS R 3503 and not less than 100 ml in capacity.

6.2.16.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **Glacial acetic acid**, distilling between 117 °C to 119 °C.

NOTE : The toxic effects of this reagent shall be borne in mind and full precautions shall be taken during use.

- b) **Diluted ammonia solution** Dilute approximately 28 % concentrated ammonia so-

lution specified in **JIS K 8085** with water to make approximately 1 % ammonia diluted solution.

6.2.16.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

Take the specimen in a glass beaker [**6.2.16.3 b)**], add to this 100 ml of glacial acetic acid [**6.2.16.4 a)**] per gram of specimen, and put this in a heating apparatus [**6.2.16.3 a)**] for 30 min while stirring occasionally to dissolve nylon fibre. After filtering this through the weighed filter by suction, wash the residue on the funnel first with glacial acetic acid of the same amount and the same temperature as before and then with warm water. Then, transfer this to another glass beaker, neutralize it by adding water and 50 ml of approximately 1 % diluted ammonia solution [**6.2.16.4 b)**] per gram of specimen, and after filtering this through the filter by suction again, wash the residue on the funnel with water. Dry, cool and weigh the filter and residue.

6.2.16.6 Calculation

Calculate the results as specified in **6.1.10**. The value of correction factor f shall be as given in table 3.

6.2.16.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.17 70 % Acetone method

6.2.17.1 Application

This method is applied to the binary fibre mixtures of acetate fibre with triacetate fibre after removal of non-fibrous matter.

6.2.17.2 Summary

The acetate fibre is dissolved out from the mixture of a known bone dry mass with 70 % acetone aqueous solution. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of acetate fibre is obtained by difference.

6.2.17.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Erlenmeyer flask**, specified in **JIS R 3503**, not less than 200 ml in capacity and provided with a glass ground-in stopper.
- b) **Mechanical shaker**, capable of shaking at 200 reciprocations per minute with 40 mm to 70 mm amplitude.

6.2.17.4 Additional reagent

70 % Acetone aqueous solution Dilute 700 ml of acetone with water to make 1 L.

6.2.17.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

To the specimen contained in an Erlenmeyer flask [**6.2.17.3 a)**], add 80 ml of 70 % acetone aqueous solution (**6.2.17.4**) per gram of specimen and shake the Erlenmeyer flask by the mechanical shaker [**6.2.17.3 b)**] for 1 h. Then, filter the solution in the Erlenmeyer flask through the weighed filter. Add 60 ml of 70 % acetone aqueous solution to the residue in the Erlenmeyer flask, shake it by hands and filter the solution in the Erlenmeyer flask through the filter. Repeat this operation twice and transfer the residue to the filter by the final operation. Fill the filter with 70 % acetone aqueous solution again, and leave it to stand until the solution has drained the filter. After filtering it by suction finally, dry, cool and weigh the filter and the residue.

6.2.17.6 Calculation

Calculate the results as specified in **6.1.10**. The value of correction factor f shall be as specified in table 3.

6.2.17.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.17.8 Precision

For homogeneous mixtures of textile materials, the confidence limits of the results obtained by this method are not greater than ± 1 % for the confidence level of 95 %.

6.2.18 80 % Acetone method

6.2.18.1 Application

This method is applied to the binary fibre mixtures of acetate fibre with modacrylic or polychlral fibre.

6.2.18.2 Summary

The acetate fibre is dissolved out from the mixture of a known bone dry mass with 80 % acetone aqueous solution. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of acetate fibre is obtained by difference.

6.2.18.3 Additional apparatus

Glass beaker, specified in **JIS R 3503** and not less than 100 ml in capacity.

6.2.18.4 Additional reagent

80 % Acetone aqueous solution Dilute 800 ml of acetone with 1 L of water.

6.2.18.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

Take the specimen in a glass beaker (**6.2.18.3**), add to this 100 ml of 80 % acetone aqueous solution (**6.2.18.4**) at 23 °C to 25 °C per gram of specimen, stir it occasionally

for 30 min to dissolve the acetate fibre. After filtering this through the weighed filter by suction, wash the residue on the funnel first with 80 % acetone aqueous solution of the same amount and the same temperature as before and then with water. Dry, cool and weigh the filter and residue.

6.2.18.6 Calculation

Calculate the results as specified in 6.1.10. The value of correction factor f shall be as given in table 3.

6.2.18.7 Confirmation

Confirmation shall be in accordance with 6.1.11 a).

6.2.19 100 % Acetone method

6.2.19.1 Application

This method is applied to the binary fibre mixtures of acetate fibre with cotton (scoured or bleached), wool, animal hair, silk, flax (linen), ramie, rayon, cupro, promix, regenerated protein fibre, nylon, vinylon, polyester, acrylic, polypropylene, benzoate, aramid and glass fibre after removal of non-fibrous matter. It is not applicable to modacrylic fibres or acetate fibres that have been deacetylated on the surface.

6.2.19.2 Summary

The acetate fibre is dissolved out from the mixture of a known bone dry mass with 100 % acetone. The residue is collected, washed, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of acetate fibre is obtained by difference.

6.2.19.3 Additional apparatus

Erlenmeyer flask, specified in JIS R 3503, not less than 200 ml in capacity and provided with a glass ground-in stopper.

6.2.19.4 Additional reagent

100 % Acetone, distilling between 55 °C to 57 °C.

6.2.19.5 Test operation

After completing the operation specified in 6.1.9 b), proceed to the following operation.

To the specimen contained in an Erlenmeyer flask (6.2.19.3), add 100 ml of 100 % acetone (6.2.19.4) per gram of specimen, shake the Erlenmeyer flask and leave it to stand for 30 min at room temperature. Then, pour the solution in the Erlenmeyer flask into the weighed filter. Carry out the extraction twice more (so that it is carried out three times in total), each time taking 15 min so that the whole treatment time with 100 % acetone becomes 1 h. Wash the residue in the filter with 100 % acetone and filter it by suction. Fill the filter with 100 % acetone and leave it to stand for 30 min at room temperature until the solution has drained the filter. After filtering it by suction finally, dry, cool and weigh the filter and residue.

6.2.19.6 Calculation

Calculate the results as specified in 6.1.10. The value of correction factor f shall be as specified in table 3.

6.2.19.7 Confirmation

Confirmation shall be in accordance with 6.1.11 a).

6.2.19.8 Precision

For homogeneous mixtures of textile materials, the confidence limits of the results obtained by this method are not greater than ± 1 % for the confidence level of 95 %.

6.2.20 Warm 100 % acetone method

6.2.20.1 Application

This method is applied to the binary fibre mixtures of acrylic fibre with modacrylic fibre after removal of non-fibrous matter.

6.2.20.2 Summary

The modacrylic fibre is dissolved out from the mixture of a known bone dry mass with warm 100 % acetone. The residue is collected, washed, dried and weighed. The mass is corrected if necessary, and expressed as a percentage of the bone dry mass of mixture. The percentage of modacrylic fibre is obtained by difference.

6.2.20.3 Additional apparatus

The additional apparatus to be used shall be as follows.

- a) **Heating apparatus**, capable of maintaining the temperature of a glass beaker at 45 °C to 50 °C (e.g. a water bath with a thermostat).
- b) **Glass beaker**, specified in JIS R 3503 and not less than 100 ml in capacity.

6.2.20.4 Additional reagent

100 % acetone, distilling between 55 °C to 57 °C.

6.2.20.5 Test operation

After completing the operation specified in 6.1.9 b), proceed to the following operation.

Take the specimen in a glass beaker [6.2.20.3 b)], add to this 100 ml of 100 % acetone (6.2.20.4) per gram of specimen, stir it occasionally at a temperature of 45 °C to 50 °C for 30 min to dissolve modacrylic fibre. After filtering this through the weighed filter by suction, wash the residue on the funnel with 100 % acetone of the same amount and the same temperature as before. Dry, cool and weigh the filter and the residue.

6.2.20.6 Calculation

Calculate the results as specified in 6.1.10. The value of correction factor f shall be as given in table 3.

6.2.20.7 Confirmation

Confirmation shall be in accordance with **6.1.11 a)**.

6.2.21 Sodium hypochlorite method

6.2.21.1 Application

This method is applied to the binary fibre mixtures of one of the protein fibres and one of the non-protein fibres as shown in the following, after removal of non-fibrous matters.

- Protein fibre : wool, chemically treated wool or other animal-hair fibres, and silk (raw silk, scoured silk, raw tussah and bleached tussah)
- Non-protein fibre : cotton, flax (linen), ramie, rayon, cupro, nylon, polyvinyl chloride, chlorofibre, polyester, acrylic, modacrylic, polypropylene, benzoate, polyurethane and glass fibre

NOTE : In the case of containing several kinds of protein fibre, in respect of this method, the whole amount can be obtained, however, the individual determination is impossible.

6.2.21.2 Summary

The protein fibre is dissolved out from the mixture of a known bone dry mass with alkaline sodium hypochlorite. The residue is collected, washed with water, dried and weighed. The mass is corrected, if necessary, and expressed as a percentage of the bone dry mass of the mixture. The percentage of protein fibre is obtained by difference.

6.2.21.3 Additional apparatus

Glass beaker, specified in **JIS R 3503** and not less than 100 ml in capacity.

6.2.21.4 Additional reagents

The additional reagents to be used shall be as follows.

- a) **Sodium hypochlorite solution** Prepare a sodium hypochlorite solution of 1 mol concentration by adding sufficient amount of sodium hydroxide specified in **JIS K 8576** so as to obtain concentration of 5 g per litre. This solution may be standardized by iodine determination method, however the concentration of this solution shall be within the range between 0.9 mol and 1.1 mol.
- b) **Diluted acetic acid** Dilute 5 ml of glacial acetic acid with water to make 1 L.

6.2.21.5 Test operation

After completing the operation specified in **6.1.9 b)**, proceed to the following operation.

To the specimen contained in a glass beaker (**6.2.21.3**), add 100 ml of sodium hypochlorite solution [**6.2.21.4 a)**] per gram of specimen, stir it vigorously so as to moisten the specimen, then leave it to stand for 30 min while stirring vigorously once in a while. After filtering the contents in the beaker through the weighed filter, rinse the beaker with a small amount of sodium hypochlorite solution and transfer all the residue to the filter. Filter the filter by suction, treat the residue with 50 ml of diluted acetic acid so-