SRI LANKA STANDARD 25:1981
UDC 677.014.23/.24:543.05

METHOD FOR
THE REMOVAL OF NON-FIBROUS MATTER PRIOR TO QUANTITATIVE ANALYSIS OF FIBRE MIXTURES
(SECOND REVISION)

BUREAU OF CEYLON STANDARDS

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SLS 25 : 1981

[redacted]

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SRI LANKA STANDARD

METHOD FOR THE REMOVAL OF NON-FIBROUS MATTER PRIOR TO QUANTITATIVE ANALYSIS OF FIBRE MIXTURES

(SECOND REVISION)

FOREWORD

This Sri Lanka Standard Specification was authorized for adoption and publication by the Council of the Bureau of Ceylon Standards on 1981-11-26, after the draft, finalized by the Drafting Committee on Test Methods for Textiles, had been approved by the Textiles Divisional Committee.

This revision of CS 25 is technically identical with the ISO/TR 5090. In the first revision, a method for the removal of added matter was described but in the present standard, the term *added matter* has been replaced by *non-fibrous matter*. Dyes are not considered as non-fibrous matter and hence the procedure for the removal of dyes has been eliminated in this standard. The other change is that this revised method is extended to include procedures for the removal of more types of non-fibrous matter.

It is common practice for various additions to be made to fibres, yarns and fabrics for the purposes of assisting processing and manufacture or modifying the properties of the finished material. These usually result in an appreciable increase in mass and often affect the solubility of the fibres. It must also be borne in mind that fibres generally contain a small proportion of naturally occuring non-fibrous substances. The removal of these non-fibrous substances is therefore necessary before procedures for quantitative chemical analysis can be conducted successfully.

The removal of certain types of non-fibrous matter, particularly when more than one substance is present, may demand the exercise of considerable chemical resource, and each material to be treated for removal of its non-fibrous matter should be regarded as an individual problem. The procedures suggested in this standard may not be complete, and it should not be assumed that those described in the Appendix will have no effect on the physical and chemical properties of the textile materials concerned. Furthermore, these procedures are only applicable where the non-fibrous substances are known or can be identified with certainty.

In this standard, dyes are not considered as non-fibrous matter but as an integral part of the textile, and are not therefore mentioned. Some prints are made with resin bonded pigments which cannot be regarded as part of the fibre substance. They involve a greater addition of mass to the fibre than dyes and it would be desirable to remove them, but it is rarely if ever possible to do so. Similarly, certain finishes cannot be removed.

It may be assumed that Soxhlet extraction under the conditions described in the Appendix will ensure adequate removal of oils, fats and waxes. With other non-fibrous substances, it is necessary, wherever possible, to check that removal is complete.

CAUTION - Since certain hazards are associated with reagents and solvents employed in the methods given below, these methods should be used only by persons acquainted with the hazards and the precautions to be taken.

All values given in this standard are in SI units.

In the preparation of this standard, valuable assistance derived from related publications of the International Organization for Standardization is gratefully acknowledged.

1 SCOPE AND FIELD OF APPLICATION

This standard describes procedures for the removal of certain commonly found types of non-fibrous substances from fibres. Fibres to which the procedures are applicable and those to which they are not applicable are listed in the table, in relation to the non-fibrous substances to be removed. Identification of the non-fibrous matter and of the fibres present is not covered by this standard.
In certain cases, the elimination of all the added matter is impracticable. The quantity remaining should not effect the quantitative analysis; on the other hand it is essential to minimize the chemical degradation of the fibres.

2 DEFINITION

For the purpose of this standard, the following definition shall apply:

2.1 non-fibrous matter : Processing aids such as lubricants and sizes (but excluding jute-batching oils), and naturally occurring non-fibrous substances.

3 PRINCIPLE

Where possible, removal of non-fibrous matter by a suitable solvent.

NOTE - In many cases the removal of certain finishes involves their chemical modifications. In addition, chemical degradation of the fibre substances cannot always be avoided.

4 APPARATUS

The apparatus required is part of the normal equipment of a chemical laboratory.

TABLE 1 - Use of procedures for removal of non-fibrous matter
(see Appendix A)

Sl. no.	Non-fibrous matter to be removed	Fibres in the presence of which the procedure is applicable	Method of test		Fibres in the presence of which the procedure is not applicable
			Ref. to	Reagents	
i	Oils, fats and waxes	Most fibres	A.1	Light petroleum, Soxhlet	Elastane
ii	Soaking oils	Nett silk	A.2	Toluene/ methanol, Soxhlet	-
iii	Starch	Cotton (see Note 1) Linen (see Note 2) Viscose Spun silk Jute (see Note 3) and most other fibres	A.3	Amylase then boiling water	-
iv	Locust-bean gum and starch	Cotton (see Note 1) Viscose Spun silk	A.4	Boiling water then A.3	-
v	Tamarind seed size	Cotton (see Note 1)	A.5	Boiling water twice	-
vi	Acrylic (size or finish)	Most fibres (see Note 4)	A.6	2 g/l soap, 2 g/l NaOH, 70 °C to 75 °C, rinse in water	Protein, deacetylated acetate, acetate, triacetate acrylic, modacrylic
vii	Gelatin and polyvinyl alcohol	Most fibres	A.7	1 g/l non-ionic detergent, 1 g/l anionic detergent, 1 g/l Na_2CO_3	Protein, deacetylated acetate, acetate, triacetate
viii	Starch and polyvinyl alcohol	Cotton polyester	A.8	A.3 followed by A.7	Protein, deacetylated acetate, acetate, triacetate
ix	Polyvinyl acetate	Most fibres	A.9	Acetone, Soxhlet	Deacetylated acetate, acetate, triacetate, chlorofibre
x	Linseed oil sizes	Viscose crepe yarns	A.10	A.1 followed by A.7	Protein, deacetylated acetate, acetate, triacetate
xi	Amino-formaldehyde resins	Cotton Cupro Viscose Modal Deacetylated acetate Acetate Triacetate Polyester Polyamide (nylon)	A.11	Orthophosphoric acid/urea, 80 °C 10 minutes rinse in water, then $NaHCO_3$	Asbestos

(to be contd.)

NOTES

1 Grey cotton loses mass when treated by these methods. The loss amounts to approximately 3 per cent of the final oven-dry mass.

2 Linen loses mass when treated by this method. The loss depends on the types of yarn from which the fabric is produced.
Losses are approximately as follows:
bleached yarns 2 per cent, boiled yarns 3 per cent and grey yarns 4 per cent.

3 Jute loses mass by approximately 0.5 per cent when treated by this method.

4 Polyamide 6.6 (nylon 6.6) may undergo a loss in mass of fibre substance of up to 1 per cent when treated by this method. The loss in mass of polyamide 6 (nylon) may vary between 1 per cent and 3 per cent.

TABLE (contd.)

Sl. no.	Non-fibrous matter to be removed	Fibres in the presence of which the procedure is applicable	Method of test		Fibres in the presence of which the procedure is not applicable
			Ref. to	Reagents	
xii	Bitumen, creosote and tar	Most fibres	A.12	Dichloromethane (methylene-chloride), Soxhlet	Deacetylated acetate, acetate, triacetate, modacrylic, chlorofibre
xiii	Cellulose ethers	Most fibres	A.13.1	Soak in cold water	-
		Cotton	A.13.2	175 g/l NaOH solution at 10°C, neutralized in 0.1 N acetic acid	Viscose, deacetylated acetate, triacetate, modacrylic, acrylic
xiv	Cellulose nitrate	Most fibres	A.14	Soaking in acetone, one hour	Deacetylated acetate, acetate, triacetate
xv	Polyvinyl chloride	Most fibres	A.15	Soaking in tetrahydrofuran (do not recover by distillation)	Deacetylated acetate acetate, triacetate, chlorofibre
xvi	Oleates	Most fibres	A.16	0.2 N HCl, extraction in dichloromethane, Soxhlet	Deacetylated acetate, acetate, triacetate modacrylic, chlorofibre, polyamide (nylon), asbestos
xvii	Oxides of chromium, iron and copper	Cupro Viscose Modal Deacetylated acetate Acetate Triacetate	A.17	10 g/l hydrated oxalic acid at 80°C, neutralized with NH_4OH	-
xviii	Pentachlorophenyl laurate	Most fibres	A.18	Toluene, Soxhlet	Polyethylene, polypropylene
xix	Polyethylenes	Most fibres	A.19	Boiling toluene under reflux	Polypropylene
xx	Polurethanes	Polyamide (nylon) Cupro Viscose Modal Deacetylated acetate Acetate Triacetate	A.20	Dimethyl sulphoxide or dichloromethane, if possible 50 g/l NaOH, ethanol at 50°C	Deacetylated acetate, acetate, triacetate, polyester, acrylic, modacrylic
xxi	Natural rubber and styrenebutadiene, neoprene, nitrite	Cupro Viscose Modal Deacetylated acetate Acetate Triacetate Glass	A.21	Swell in benzene, scrape, heat in molten p-dichlorobenzene after 45 minutes add one part tertbutyl hydroperoxide per four parts of p-dichlorobenzene, cool to 60°C, add benzene	All synthetic fibres
xxii	Silicones	Most fibres	A.22	Hydrofluoric acid 50 ml to 60 ml per litre, 65°C	Polyamide (nylon) glass
xxiii	Tin weighting	Silk	A.23	0.5 N hydrofluoric acid	-
xxiv	Wax-based waterproof finishes	Cotton Protein Polyester Polyamide (nylon)	A.24	Dichloromethane, Soxhlet if metallic complex: 1 g/l formic acid and 5 g/l soap	Deacetylated acetate, acetate, triacetate, modacrylic, chlorofibre

APPENDIX A

PROCEDURES FOR REMOVAL OF NON-FIBROUS MATTER

A.1 OILS, FATS AND WAXES (EXCEPT SOAKING OILS)

Extract the specimen in a Soxhlet apparatus with light petroleum (distilling between 40 °C and 60 °C) for at least one hour at a minimum rate of six cycles per hour.

A.2 SOAKING OILS

Extract the specimen in a Soxhlet apparatus with a mixture of one volume of toluene with three volumes of methanol as the solvent, for at least two hours at a minimum rate of six cycles per hour.

NOTE - There is an accepted method for the removal of soaking oils form silk that includes benzene but because of the toxic properties of benzene the above method is suggested.

A.3 STARCH

Immerse the specimen in a freshly prepared solution containing 0.1 per cent (m/m) of a non-ionic wetting agent together with an appropriate amylase preparation, using a liquor/specimen ratio of 100:1. The concentration of the amylase preparation and the pH, temperature and time of treatment should be those recommended by the manufacturer. Transfer the specimen to boiling water and boil it for 15 minutes. Test for complete removal of starch using a dilute aqueous solution of iodine in potassium iodide. When all the starch is removed, rinse the specimen thoroughly in water, squeeze or mangle it, and dry it.

A.4 LOCUST-BEAN GUM AND STARCH

Boil the specimen in water for 5 minutes, using a liquor/specimen ratio of 100:1. Repeat the procedure with a fresh portion of water. Follow this by the procedure described in A.3.

A.5 TAMARIND SEED SIZE

Boil the specimen in water for 5 minutes, using a liquor/specimen ratio of 100:1. Repeat this procedure with a fresh portion of water.

NOTE - Size prepared from coarsely ground undercorticated tamarind seed powder may not be completely removed by this procedure.

A.6 ACRYLIC SIZE

Immerse and agitate the specimen for 30 minutes in at least 100 times its own mass of a solution containing 2 g/l of soap or other suitable detergent and 2 g/l of sodium hydroxide at 70 °C to 75 °C. Give 3 minutes to 5 minutes rinses in distilled water at 85 °C, squeeze, mangle or centrifuge, and dry the specimen.

A.7 GELATIN AND POLYVINYL ALCOHOL

Treat the specimen in a solution (using a minimum liquor/specimen ratio of 100:1) containing 1 g/l of non-ionic surfactant, 1 g/l of anionic surfactant and 1 g/l of anhydrous sodium carbonate, for 90 minutes at 50 °C followed by 90 minutes in the same bath at 70 °C to 75 °C. Wash the specimen and dry it.

A.8 STARCH AND POLYVINYL ALCOHOL

Conduct the procedure described in **A.3**, followed by the procedure described in **A.7**, with intermediate drying.

A.9 POLYVINYL ACETATE

Extract the specimen in a Soxhlet apparatus with acetone for at least 3 hours at a minimum rate of six cycles per hour.

A.10 LINSEED OIL SIZES

Conduct the procedure described in **A.1**, followed by the procedure described in **A.7**.

A.11 AMINO-FORMALDEHYDE RESINS

Extract the specimen with a solution of 25 g/l of 50 per cent orthophosphoric acid and 50 g/l of urea at 80 °C for 10 minutes, using liquor/specimen ratio of 100:1. Wash the specimen in water, drain, wash it in a 0.1 per cent sodium bicarbonate solution, and finally wash it thoroughly in water.

NOTE - This method causes some damage to cupro, viscose, modal, deacetylated acetate, acetate and triacetate.

A.12 BITUMEN, CREOSOTE AND TAR

Extract the specimen with dichloromethane (methylene chloride) in a Soxhlet apparatus. The duration of treatment depends on the amount of non-fibrous matter present, and it may be necessary to renew the solvent.

NOTE - Extraction of jute with dechloromethane will also remove the batching oil, which may be present to the extent of 5 per cent or more.

A.13 CELLULOSE ETHERS

A.13.1 Methyl cellulose soluble in cold water

Soak the specimen in cold water for 2 hours. Rinse the specimen repeatedly in cold water, with vigorous squeezing.

A.13.2 Cellulose ethers insoluble in water but soluble in alkali

Immerse the specimen for 30 minutes in a solution containing approximately 175 g/l of sodium hydroxide cooled to a temperature of 5 °C to 10 °C. Then work the specimen thoroughly in a fresh portion of reagent, rinse it well in water, neutralize it with approximately 0.1 N acetic acid, rinse it again in water, and dry it.

A.14 CELLULOSE NITRATE

Immerse the specimen in acetone at room temperature for one hour, using a liquor/specimen ratio of 100:1. Drain, wash the specimen in three portions of fresh acetone, and allow the entrained solvent to evaporate.

A.15 POLYVINYL CHLORIDE

Immerse the specimen in tetrahydrofuran at room temperature for one hour, using a liquor/specimen ratio of 100:1. If necessary, scrape off the softened polyvinyl chloride. Drain, wash the specimen in three portions of fresh tetrahydrofuran, drain and allow the entrained to evaporate.

CAUTION - Because of the risk of explosion, tetrahydrofuran should not be recovered by distillation.

A.16 OLEATES

Immerse the specimen in approximately 0.2 N hydrochloric acid at ambient temperature until it is thoroughly wetted. Wash the specimen well and dry it. Extract the specimen in a Soxhlet apparatus with dichloromethane (methylene chloride) for one hour at a minimum rate of six cycles per hour.

A.17 OXIDES OF CHROMIUM, IRON AND COPPER

NOTE - This method is not applicable if dyes containing chromium have been applied to the material under test.

Immerse the specimen in a solution containing 14 g/l of hydrated oxalic acid at 80 °C for 15 minutes, using a liquor/specimen ratio of 100:1. Wash it thoroughly (any copper present will remain as the colourless oxalate, remove this with 1 per cent acetic acid at 40 °C for 15 minutes, and wash the specimen). Neutralize the specimen with ammonia and wash it thoroughly in water. Squeeze, mangle or centrifuge, and dry it.

A.18 PENTACHLOROPHENYL LAURATE (PCPL)

Extract the specimen in a Soxhlet apparatus with toluene for 4 hours at a minimum rate of six cycles per hour.

A.19 POLYETHYLENES

Extract the specimen in boiling toluene under relfux.

NOTE - The material must be completely immersed in the boiling solvent.

A.20 POLYURETHANES

No completely satisfactory method is available but the following have been found suitable.

Some polyurethanes can be removed by dissolution in dimethyl sulphoxide or dichloromethane (methylene chloride), and subsequent repeated washing of the specimen with fresh quantities of solvent. When the fibre composition of the specimen permits, some polyurethanes can be removed by hydrolysis in a boiling aqueous solution containing 50 g/l of sodium hydroxide. Alternatively an aqueous solution containing 50 g/l of sodium hydroxide and 100 g/l of ethanol may be used at a temperature above 50 °C.

NOTE - Dimethyl sulphoxide has toxic properties.

A.21 NATURAL RUBBERS, AND STYRENE-BUTADIENE, NEOPRENE, NITRILE AND MOST OTHER SYNTHETIC RUBBERS

No completely satisfactory method is available but the following has been found useful.

Soak the specimen in a hot volatile solvent which swells it considerably (for example benzene), and when it is fully swollen remove as much of the rubber as possible by scraping. It may be possible in some cases, where the textile fibres are exposed, to wet only the textile face, and strip the rubber and textile layers apart almost at once. Continue by heating the residual specimen, with constant stirring, in 50 or more times its mass of molten p-dichlorobenzene; use a flat-bottomed flask with an attached wide-bore condenser (to allow adequate access or air), and preferably a magnetic stirrer and hot-plate.

After 45 minutes, add 1 part 70 per cent tert-butyl hydroperoxide per 4 parts-p-dichorobenzene present. Boil until decomposition of the rubber is complete (2 hours is an average time). Cool the flask to about 60 °C and add an equal volume of benzene. Filter and wash the textile component repeatedly in warm benzene.

Nitrile rubber (for example acrylonitride-butadiene rubber) may require the addition of the same volume of nitrobenzene as of tert-butyl hydroperoxide to speed up the dissolution process.

NOTES

1 Natural rubber should dissolve after being boiled in p-dichlorobenzene alone for several hours in the presence of air. Dissolution may also be effected by heating in diphenyl ether at 150 °C to 160 °C for two hours and then washing the specimen in benzene.

2 The above treatments are strongly oxidative in character and the properties of the textile material may be affected appreciately.

A.22 SILICONES

Scour the specimen in a solution containing 50 ml/l to 60 ml/l of 40 per cent hydrofluoric acid in a polyethylene vessel at 65 °C for 45 minutes. Thoroughly wash the specimen, neutralize it, and scour it in a solution containing 2 g/I of soap at 60 °C for one hour.

A.23 TIN WEIGHTING

Immerse the specimen in 0.5 N hydrofluoric acid in a polyethelene vessel at 55 °C for 20 minutes, stirring occasionally. Rinse in warm water. Immerse the specimen in a 2 per cent solution of sodium carbonate at 55 °C for 20 minutes. Wash the specimen in warm water, squeeze, mangle or centrifuge, and dry it.

A.24 WAX-BASED WATERPROOF FINISHES

Extract the specimen in Soxhlet apparatus with dichloromethane (methylene chloride) for at least 3 hours at a minimum of six cycles per hour. Then, to remove any metallic complexes, scour the specimen in a solution containing 10 g/l of formic acid and 5 g/l of acid stable surfactant at 80 °C for 15 minutes. Wash the specimen thoroughly in water until it is free from acid.

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