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METHOD FOR
DETERMINATION OF THE FLUIDITY OF COTTON, RAYONS AND CELLULOSE ACETATE IN CUPRAMMONIUM HYDROXIDE SOLUTION
(FIRST REVISION)

SRI LANKA STANDARDS INSTITUTION

Gr.8

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FOREWORD

This standard was approved by the sectoral committee on Textiles Clothing & Leather and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2000-07-27.

This standard was first published in 1974. This revision incorporate some changes in techniques and apparatus, and also provides a procedure for mixtures of cellulosic fibers.

It is found that for many chemical treatments that weaken cellulose the cuprammonium fluidity increase linearly with the percentage fall in strength over a wide range. For practical purposes, therefore, the cuprammonium fluidity enables a distinction to be made between mechanical and chemical damages and affords a very convenient measure of the extent of chemical attack that the cellulose has undergone.

When mixtures of cellulosic fibers are to be tested somewhat different concentrations apply, depending on the nature of the blend and on the source of the non cotton components. It would be more appropriate to estimate the fluidity of the cotton component only. Since the majority of mixtures are prepared before any treatment is applied which is likely to affect the fluidity it can be assumed that any increase in the fluidity value for the cotton component reflects an increase in the fluidity value for the other component, although it should be noted that these changes may not be proportional.

Because of these considerations three basic methods for calculating test specimen size and test results are given, the first dealing with pure fibre, the second and the third dealing with mixtures of cotton with cellulosic man-made fibres.

In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with CS 102.

In the preparation of this standard the valuable assistance derived from the following publication is gratefully acknowledged.

BS 2610 : 1978 Method of test for the determination of cuprammonium fluidity of cotton and certain cellulosic man-made fibres.

1 SCOPE

This Sri Lanka Standard specifies a method for the determination of the cuprammonium fluidity of cotton, viscose, cupro, modal, deacetylated acetate, acetate or triacetate, and blends of cotton with cellulosic man-made fibres, in cuprammonium hydroxide solution.

2 REFERENCES

SLS 16 Standard atmospheres for conditioning and testing textiles
SLS 25 Removal of non-fibrous matter prior to quantitative analysis of fibre mixtures
CS 102 Presentation of numerical values
CS 124 Test sieves

3 DEFINITIONS

For the purpose of this standard the following definitions shall apply:

3.1 kinematic fluidity (of a solution) : The reciprocal of the kinematic viscosity of the solution.

3.2 cuprammonium fluidity (of a specimen of cotton) : The kinematic fluidity of a solution in the specified cuprammonium solvent containing 0.5g of cellulose per 100 ml of solution divided by the density of the solvent. A value of 0.94g/cm^3 for the density is sufficiently accurate.

3.3 cuprammonium fluidity (of a specimen of rayon) : Fluidity of a solution containing 2.0g of cellulose per 100 ml of standard cuprammonium solvent at 20 °C.

3.4 cuprammonium fluidity (of a specimen of cellulose acetate or triacetate) : Fluidity of solution containing acetate or triacetate equivalent to 2.0g of regenerated cellulose per 100 ml of standard cuprammonium solvent at 20 °C.

4 PRINCIPLE

A weighed specimen of the material is dissolved in cuprammonium hydroxide solvent of specified composition in a defined capillary viscometer. The viscometer and its contents are brought to a temperature of 20 °C. The solution at 20 °C is allowed to flow through the capillary of the viscometer, and the time is determined for the meniscus to fall from one fixed mark to the next. The cuprammonium fluidity is calculated by means of a formula.

5 REAGENT

Cuprammonium hydroxide solvent containing 15 ± 0.1g of copper, 200 ± 5g of ammonia, and less than 0.5g nitrous acid per litre (cuprammonium solvent). The solution shall be free from any deposit. Solvent shall be prepared and tested before use as given in Appendix **A**.

6 APPARATUS

6.1 Viscometer and fittings, The viscometer shall be of the form illustrated in Figure 1(**a**) consisting of a glass tube A sealed to a capillary tube E, both being made of precision - bore tubing with the dimensions set out below.

Glass tube A internal diameter of 10 ± 0.01 mm, and wall thickness ranging from 1 mm to 1.5 mm.

The capillary E with an internal diameter of 0.88 ± 0.01 mm and external diameter of 6 ± 2 mm and 25 ± 0.5 mm long.

The instrument is fitted at its upper end with a plastic stopper incorporating a narrow plastic tube provided with a small plastic plug, and at its lower end with rubber tubing and screw clip. The body of the viscometer is provided with three timing rings B,C,D etched around its circumference at vertical heights of 242 ± 0.5 mm, 122 ± 0.5 mm and 62 ± 0.5 mm, respectively.

6.2 Glass jackets, for the support of the viscometer in the thermostat during the time required to bring the temperature to 20 °C while the time of outflow is being measured. Suitable designs are given in **Figure 1 (b)** for the first operation and **Figure 1 (c)** for the second. Other designs may be equally convenient, provided that the viscometer is held vertically while the solution flows out.

6.3 Brass supports, for the jackets, with plumb-line or other means of verifying that the jacket is vertical while the time of the flow is being measured. One suitable design is illustrated in Figure **1 (d).**

6.4 Steel rod, 1.6 mm diameter, approximately 300 mm long.

6.5 Blackened glass container, as illustrated in Figure **2** for cuprammonium solvent with an unblackened strip approximately 5 mm wide down the container.

6.6 Short graduated burette, preferably graduated in intervals of 0.7 ml.

6.7 Rotational device, for rotating the filled viscometers end-over-end at a speed of not more than 4 rpm.

6.8 Glass thermostat, with stirrer, capable of being maintained at 20 ± 0.2 °C. A glass accumulator jar approximately 250 mm x 220 mm x 350 mm is suitable. The vessel is conveniently illuminated from behind by means of a cylindrical electric light bulb and reflector.

6.9 Split-finger second timer or stop watch graduated in seconds and fifths of a second.

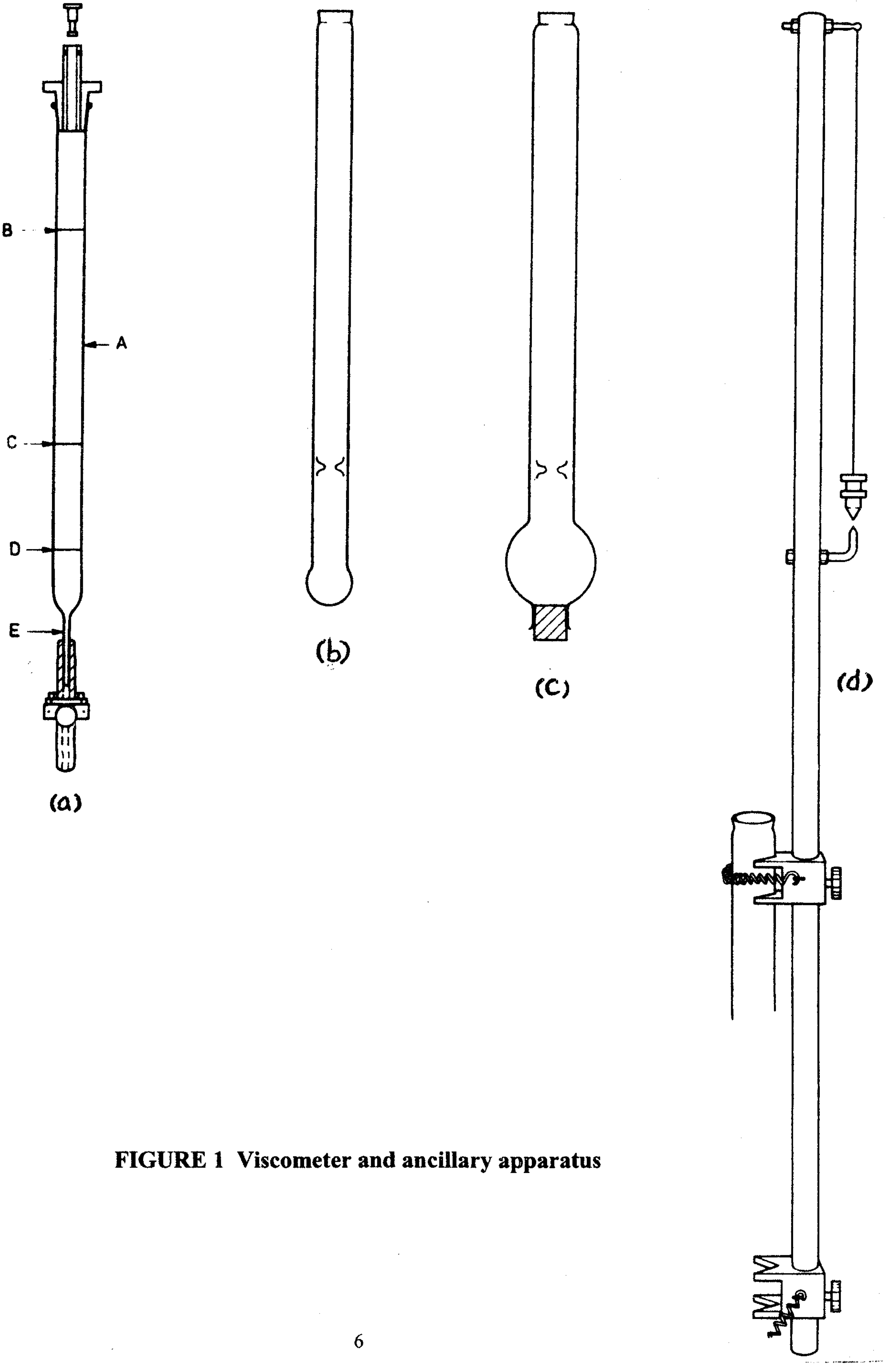

FIGURE 1 Viscometer and ancillary apparatus

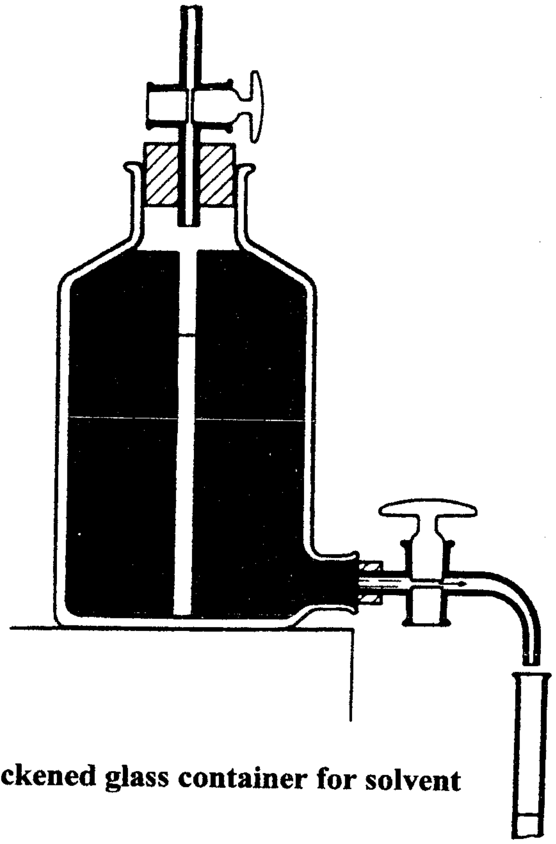

FIGURE 2 - Blackened glass container for solvent

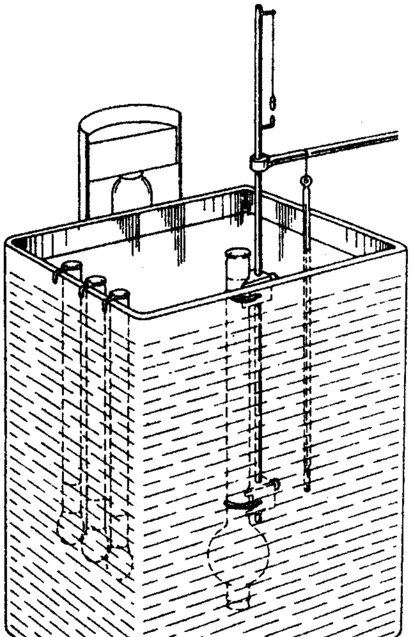

FIGURE 3 - Observation tank with apparatus

7 TEST PROCEDURE

7.1 Preparation of test specimens

Remove all non cellulosic impurities as specified in **SLS 25**. If a blend is to be subjected to test determine the composition using an appropriate method before determining fluidity using a fresh specimen. Cut yarns or loose fibres into lengths of 1 mm to 2 mm and in case of cloth first divide into narrow strips and then diagonally into short fragments to breakdown both warp and weft.

NOTE :

certain methods do not degrade the cellulose, while others do, so that correct fluidity values cannot be obtained when these methods are used to remove added matter.

7.2 Mass of specimen

7.2.1 *Pure cotton*

Weigh to the nearest milligram, sufficient of the material depending on the volume of the viscometer used, to give a 0.500 ± 0.001 per cent solution of dry cotton cellulose assuming 6 per cent moisture content.

7.2.2 *Cellulosic man-made fibre*

7.2.2.1 *Pure cellulosic man-made fibre*

Weigh, to the nearest milligram sufficient of the material, depending on the volume of the viscometer used, to give a 2.000 ± 0.001 per cent solution of cellulosic man-made fibre assuming 10 per cent moisture content.

7.2.2.2 *Acetate or triacetate*

The mass of acetate or triacetate required to give a 2 per cent solution of cellulose is given by the following formula.

$$\text{Required mass of acetate or triacetate (in mg) (M)} = 2V_o \frac{1}{100-m} \times \frac{142.9}{142.9-A}$$

where,

V_o is the volume of the viscometer (in ml) as determined in Appendix F,
m is the percentage of moisture (in g of water per 100 g air-dry cellulose acetate); and
A is the percentage acetic acid yield of acetate or triacetate

Mass of air dry mixture (M) = $\frac{C_m V_o}{100}$

where,

C_m is the concentration, in g/100 ml, obtained from Table 1 or as calculated from the formula; and
V_o is the volume, in ml, of the viscometer as determined in Appendix **B**.

Method 1 - Use the appropriate concentration (given in table 1) depending on the amount of cotton in the mixture.

TABLE 1 - Concentration in solvent cotton mixtures

Percentage Cotton in mixture on dry basis	Concentration (C_m) grams of air-dry mixture per 100 ml solvent
80	0.762
75	0.807
66.7	0.875
60	0.948
50	1.088
40	1.267
33.3	1.424
25	1.649

Method 2 - This method gives a concentration which equals that which would have been obtained, if the components had been separate, the whole of the cotton dissolved at a concentration of 0.5 per cent, the whole of the cellulosic man made fibre being dissolved at a concentration of 2 per cent and the two solutions then mixed. Calculate the concentration using the following formula :

Concentration, in grams per 100 ml, of air dry mixture in the solvent(C_m) = $\frac{1}{1.5\,R_c + 0.5}$

where,

R_c is mass, in grams, of cotton per gram of air - dry mixture.

7.3 Calibration of the viscometer

7.3.1 *Calibrating liquids*

7.3.1.1 *Aqueous glycerol solution,* containing approximately 65 per cent (m/m) of commercially pure glycerol. The kinematic fluidity of this solution shall be 8 s /cm^2 at 20 ± 0.2°C.

7.3.1.2 *Phenylethanal,* (solution) commercially pure, further purified by distillation. The kinematic fluidity of this substance shall be 7.14 s/cm^2 at 20 ± 0.2 °C.

7.3.2 *Calculation of kinetic energy correction constant (K)*

Allow the calibration liquid (7.3.1) of known kinematic fluidity to flow through the viscometer (sufficiently slowly to avoid the need for a kinetic energy correction) at 20 ± 0.2 °C. Measure the time taken to flow between B and D timing marks. Calculate the kinetic energy constant (K) as follows:

a) When the calibration liquid used is glycerol

kinetic energy correction constant (K) = 1.131 VT

b) When the calibration liquid used is phennylethanol

kinetic energy correction constant (K) = 0.117 VT

where,

V is the volume, in ml, between the timing marks B and D (See Note); and
T is the time taken, in seconds, to flow between B and D.

NOTE :

This measurement is required only approximately. Add water to the viscometer from a burette and measure the volume required to raise the liquid level from D to B.

7.3.3 *Calculation of viscometer constant (C)*

Calculate the viscometer constant (C) as follows assuming the density of the cuprammonium solvent and of dilute solutions of cellulose in cuprammonium to be 0.94 g/cm^3.

$$\text{Viscometer constant} = \frac{Fd}{0.94} \times \{T - (K/T)\}$$

where,

F is the kinematic fluidity, in s/cm^3, for the particular calibrating liquid (see 7.3.1);
T is the time taken, in seconds, to flow between Band D; and
K is the kinetic energy correction constant, calculated as in 7.3.2

7.3.4 *Calculation of cuprammonium fluidity of cellulose solutions*

When $T \geq 200$ seconds

Cuprammonium fluidity of cellulose solution $(F) = \frac{C}{T}$

When $T < 200$ seconds

Cuprammonium fluidity of cellulose solution $(F) = \frac{C}{(t - (k/t))}$

where,

C is the viscometer constant calculated as in 7.3.3;
T is the time taken, in seconds, to flow between B and D; and
K is the kinetic energy correction constant.

7.4 Determination of flow time

7.4.1 Transfer the required amount of fibre determined as in **7.2** to the viscometer **(6.1),** as described in Appendix **C.** Close the lower capillary (E) of the viscometer. Fill the viscometer to 2/3 of its capacity with cuprammonium solvent. Open the lower clip momentarily to ensure that the solvent completely fills the capillary.

7.4.2 Add 0.7 ml mercury from the burette **(6.6)** to assist mixing and dissolution of the specimen. Clamp the viscometer in a vertical position. Introduce the weighed specimen into the viscometer and rapidly mix with the solvent by means of the steel rod **(6.4).** Ensure thorough mixing, to avoid air bubbles and to allow free movement of the mercury.

7.4.3 Fill the viscometer (approximately 2 mm from the top) with cuprammonium solvent. Insert the stopper so that excess liquid, overflows displacing all air, and then insert the plug. Wrap the viscometer in black cloth or exclude light in some other suitable way. Attach to the rotating device and rotate the viscometer overnight.

NOTE

Agitation for this period suffices for most of the viscous solutions but in some cases complete dissolution may only be achieved by pre-treatment. Pre treatment of fibers which are difficult to dissolve is given in Appendix ***D****.*

7.4.4 Remove the viscometer from the rotating device. Withdraw the lower clip and rubber tube. Cautiously loosen the stopper to enable the mercury to run out through the lower capillary, assisting it if necessary, by gently tapping the viscometer. Replace the stopper firmly. Support the viscometer in a jacket (6.2) in the thermostat. And allow to stand until the required temperature is attended (10 minutes to 15 minutes).

7.4.5 Mount the viscometer in a jacket in an accurately vertical position in the thermostat. Remove the stopper and allow the solution to flow freely through the capillary E. Measure by means of a stop watch the time in seconds, for the meniscus to fall from **B** to **C**, and **B** to **D**. The intermediate reading (Time of flow from B to C) serving to detect any considerable irregularity indicates partial choking of the capillary (See Appendix **E**).

NOTE

Observation of the meniscus is facilitated by placing the lamp behind the tank in line with the viscometer (See Figure 3).

8 CALCULATION AND EXPRESSION OF RESULTS

8.1 Pure fibre (cotton or cellulosic man-made fibre)

Cuprammonium fluidity, F shall be calculated using the observed time t (in seconds) as follows (same as 7.2.4)

8.1.1 When $t \geq 200$ s $F = C^1 / t$

8.1.2 When $t < 200$ s $F = C^1 / (t - [K/t])$

where,

C^1 is the Viscometer constant;
t is the Time of flow in seconds; and
K is the kinetic energy correction constant.

The final value shall be expressed to the nearest 0.1 unit of cuprammonium fluidity.

8.2 Mixture of cotton with cellulosic man-made fibre

Fluidity (F_c) of the cotton component shall be calculated using the cuprammonium fluidity (F_m) of the mixture, as in **8.1**. Fluidity of the cotton component F_c, shall then be calculated using one of the formula depending on the method used (See **7.2.3**).

Method 1 $F_c = 1.4 F_m$

Method 2 $F_c = 1.15 F_m - 2.2$

The result is expressed to the nearest unit of cuprammonium fluidity.

APPENDIX A
PREPARATION AND TESTING OF CUPRAMMONIUM HYDROXIDE SOLUTION

Prepare the solution by blowing air through a mixture of metallic copper and concentrated aqueous ammonia containing 1 g of cane sugar per litre and cooled in ice. The ammonia solution should contain about 220 g of ammonia per litre and may be made by diluting ammonia (relative density 0.880) with water in the proportion 73:27 (V/V). Pass the air stream through an ammonia solution of similar concentration before it enters the reaction vessel. Any convenient arrangement of apparatus may be used for the preparation. The copper should be in the form of a powder fine enough to pass through a 63 µm sieve, conforming to of **CS 124,** and there should be 60g of copper per litre of solution. The metal should be well agitated and the air stream broken into fine bubbles.

The powdered copper should not contain more than traces of iron, chlorides or sulphate, nor more than 0.2 per cent of zinc. It may be prepared by precipitation from a copper salt with iron or zinc, or by electrolysis. The preparation should be carried to such a stage that the standard copper concentration is appreciably exceeded. The solution should be analysed for copper and ammonia as described in **A.1**, **A.2** and then it is a matter of calculation to decide the concentration of aqueous ammonia, with which the preparation must be diluted, and the extent of such dilution, in order to obtain a solution of the standard concentration.

A.1 DETERMINATION OF COPPER

Boil 25 ml of the solvent in a 250 ml conical flask until the ammonia is removed. Acidify the solution with dilute sulfuric acid. Boil to remove nitrous acid, the last traces of which are removed by adding a small amount of bromine, and again boiling.

Make the solution slightly alkaline with ammonia and add 7 ml, of glacial acetic acid followed by 3g to 5g of potassium iodide. Solvent shall be kept in a dark place after addition of pottasum iodide. Titrate the liberated iodine with 0.1 mol/l sodium thiosulphate solution, using soluble starch as indicator. If T_c is the sodium thiosulphate titre in ml the copper content of the cuprammonium in g/l is 0.508 T_c .

A.2 DETERMINATION OF AMMONIA

Run 2 ml of the solvent from a pipette into 25 ml of 1 mol/l sulphuric acid and titrate the excess acid with 1 mol/l sodium hydroxide solution using methyl red as indicator. The titration is complete when the red colour just disappears. If T_a is the alkali titre, in ml, the 'apparent' ammonia content is given by the formula

$$\text{'Apparent' ammonia content (A) g/l} = \frac{17.03 \times (50 - T_a)}{2}$$

This must be corrected by subtracting from it the ammonia equivalent of the copper, which is 0.536 c g/l, where c is the copper concentration in g/l.

A.3 DETERMINATION OF NITRITE

Run 10 ml of the solvent into a mixture of 25 ml of 0.04 mol/l ammonium ceric sulphate and 100 ml of 1 mol/l sulphuric acid. After 10 min titrate the excess of ceric sulphate with 0.04 mol/l ammonium ferrous sulphate, using 0.5 ml ferroin as indicator. The content of nitrous acid in g/l is equal to 0.094 x titre difference in ml.

APPENDIX B
MEASUREMENT OF THE TOTAL VOLUME (V_O) ENCLOSED BY THE VISCOMETER

Weigh the empty viscometer. Fill with water as it would be with cuprammonoium solvent and weigh again. Substract 0.7 ml of which is the volume of the mercury stirrer from the volume determined.

APPENDIX C
WEIGHING AND INTRODUCTION OF SPECIMENS

6.1 Cotton

Weigh the specimen in a tube 23 mm high by 18 mm in diameter (approximately) and transfer it to the viscometer via a short-stemmed glass funnel of 35 mm diameter with the aid of a small camelhair brush.

6.2 Cellulose man made fibres

Weigh the specimen in a tube 90 mm long by 7 mm in diameter (approximately), with a stopper in one end and eject it directly from this into the viscometer by means of a glass rod.

APPENDIX D
PRE-TREATMENT OF FIBRES WHICH ARE DIFFICULT TO DISSOLVE

In certain cases, namely native (unscoured) cotton, very mildly scoured cotton, mineral dyed or chrome proofed cotton, cotton dyed with sulfur black and certain reactive dyes, the fibre will not dissolve completely in cuprammonium solution and a pre-treatment is necessary. For grey cotton and cotton dyed with sulfuric blacks, boiling for 1 h under reflux in one per cent sodium hydroxide solution is normally sufficient to ensure complete solubility with little or no reduction in the relative molecular mass which would result in an increased fluidity value. For cotton dyed with reactive dyes, boiling for 2 h in 4 per cent sodium carbonate solution is sufficient for some reactive dyes, e.g. those based on dichlorotriazine.

With mineral dyed or chrome proofed fabrics, or fabrics that have been finished with aminoformaldehyde resins, where treatments with acid are required to confer solubility in cuprammonium, the results are of very limited value because of the hydrolytic action of the acid on the cellulose. An attempt is made to allow for this by subtracting the fluidity of a similarly treated control sample from that of the sample under examination but this is only partially satisfactory because the resin or mineral dyeing may exert some protective action on the cellulose.

Mineral dyed or proofed cotton fabrics may be solubilized by two or three 10 minute treatments with 1per cent oxalic acid solution at 70 °C. Where reactive dyed fabrics have been resin finished, a satisfactory result cannot be obtained even with a control. Resin-finished fabrics that have been cured under conditions leading to the formation of a significant number of methylene cross-links cannot be solubilized.

APPENDIX E
POSITION AND USE OF THE MIDDLE TIMING MARK

The middle ring C is positioned so that for normal viscous flow the time t_1 for the meniscus to sink from B to C is equal to the time t_2 for it to sink from C to D. When making measurements, the time of flow to C, as well as to D, is noted and the ratio t_1 / t_2 is recorded. It should approximate to unity when the material under test is fairly highly degraded. The ratios to be expected for cotton materials of different fluidity are shown in Table 1; any gross difference between these values and the observed value for t_1 / t_2 indicates that the capillary has become partially choked by undissolved material during the flow of the liquid. In practice this happens only rarely and then generally with cotton specimens of low fluidity that dissolve relatively slowly in cuprammonium; it is seldom, if ever, encountered with cellulosic man-made fibres.

TABLE 1 - Ratios of cuprammonium fluidity to t_1 / t_2

F	1.5	2.0	2.5	3.0	3.5	4.0
t_1 / t_2	0.67	0.73	0.78	0.80	0.83	0.84
F	4.5	5.0	7.0	10.0	15.0	21.3
t_1 / t_2	0.86	0.87	0.92	0.94	0.98	1.00

* F is measured in units of cuprammonium fluidity.

If the capillary has become partially choked, rinse the viscometer with water. Immerse in chromic acid mixture for several hours, rinse with water and dry. Immerse the viscometer fittings in dilute acetic acid solution, wash with water and dry in a warm place. The determination can then be repeated.

NOTE :

Chromic acid is a corrosive oxidizing agent and should be handled with care. It is recommended that only experienced laboratory staff should be employed.

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