

Methods of testing cellulose acetate flake

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National foreword

This British Standard is a revision of BS 2880:1957 and BS 2880: Addendum No.1:1959 which are withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/82, Thermoplastic materials.

A list of organizations represented on this committee can be obtained on request to its secretary.

In this revision the test methods have been aligned with the latest versions of the relevant International Standards. In some case the precise ISO procedure has been invoked. In these cases it is intended that the ISO methods of test will be published as BS 2782 methods identical with the corresponding ISO standards. However, at the present time, although method numbers have already been allocated, some BS 2782 methods have not yet been published and, until they have, the corresponding ISO standards should be used.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard, having been prepared under the direction of the Plastics and Rubber Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 30 August 1991

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Amendments/corrigenda issued since publication

Date	Comments
28 February 2010	Supersession text amended

1 Scope

This standard describes methods of test for the following cellulose acetate flake properties:

- a) moisture content;
- b) ash content;
- c) acetic acid yield;
- d) bulk density;
- e) particle size distribution;
- f) free acidity;
- g) viscosity ratio of a 5 g/L solution at 25 °C;
- h) viscosity at a concentration of 60 g/L;
- i) viscosity at 25 % concentration at 25 °C;
- j) insoluble particles;
- k) original light absorption and change of light absorption after heating of plasticized cellulose acetate;
- l) the reduction in viscosity ratio on moulding.

NOTE The titles of the publications referred to in this standard are listed on the inside back cover.

2 Definition

For the purposes of this British Standard the following definition applies.

cellulose acetate flake

non-plasticized acetic acid esters of cellulose (excluding mixed acid esters) in flake form

3 Method 1. Determination of moisture content

3.1 Principle

A specified mass of material is heated at 105 °C for 3 h and the percentage loss of mass is used to determine the moisture content.

3.2 Sampling

Select a representative sample following the principles embodied in BS 5309 and BS 6001. Pack the sample in a moisture-proof container.

NOTE Good quality, clean, dry lever-top tins have been used satisfactorily as moisture-proof containers.

3.3 Procedure

Determine the moisture content of the sample selected in accordance with 3.2 by the method described in BS 2782-4:Method 431C.

NOTE BS 2782-4:Method 431C is identical with ISO 585.

4 Method 2. Determination of ash content

4.1 Principle

A given mass of material is heated at 575 °C to constant mass and the residual mass is taken as the ash content.

4.2 Sampling

Select a representative sample following the principles embodied in BS 5309 and BS 6001. Pack the sample in a moisture-proof container.

NOTE Good quality, clean, dry lever-top tins have been used satisfactorily as moisture-proof containers.

4.3 Procedure

Determine the ash content of the sample selected in accordance with 4.2 by the method described in BS 2782-4:Method 470C except that the temperature used shall be 575 °C.

NOTE BS 2782-4:Method 470C is identical with ISO 3451-3.

5 Method 3. Determination of acetic acid yield

5.1 Principle

Finely divided cellulose acetate stands in contact with a mixture of acetone and aqueous sodium hydroxide. The amount of alkali consumed in hydrolysing the cellulose acetate is then measured by titration.

NOTE The method described for the estimation of acetic acid yield has been found to be suitable for cellulose acetates of acetic acid yields ranging from about 40 % to a nominal 62.5 %. The term "acetic acid yield" is preferred for describing what has previously been known as "acetyl value".

5.2 Reagents

5.2.1 Sodium hydroxide solution, carbonate free, analytical reagent grade, nominal $c(\text{NaOH}) = 1 \text{ mol/L}$.

5.2.2 Dilute sodium hydroxide solution, carbonate free, analytical reagent grade, nominal $c(\text{NaOH}) = 0.5 \text{ mol/L}$.

5.2.3 Sulphuric acid, analytical reagent grade, nominal $c(\text{H}_2\text{SO}_4) = 0.5 \text{ mol/L}$.

NOTE In order to ensure that there will be a positive back titration value in the blank the strength of the sulphuric acid (5.2.3) should be greater than that of the sodium hydroxide (5.2.1).

5.2.4 Acetone, complying with BS 509-1.

5.2.5 Phenolphthalein, a solution comprising 5 g of phenolphthalein dissolved in a litre of ethanol.

5.3 Sampling

Select a representative sample following the principles embodied in BS 5309 and BS 6001. Pack the sample in a moisture-proof container.

NOTE Good quality, clean, dry lever-top tins have been used satisfactorily as moisture-proof containers.

5.4 Procedure

Grind at least 2 g of the sample selected in accordance with 5.3 so that all of it will pass through a sieve with a nominal aperture size of 212 μm complying with BS 410.

NOTE A beater-cross mill has been found suitable for grinding the material.

Place a test portion of the ground material equivalent to about 1.5 g of oven-dry material in a weighing bottle, dry for 3 h at a temperature of $110 \pm 2^\circ\text{C}$ and cool in a desiccator. Stopper the bottle and weigh. Transfer the contents of the bottle to a 300 mL stoppered borosilicate glass conical flask. Reweigh the stoppered bottle and determine the mass taken by difference.

Shake the test portion evenly over the base of the flask and, without lifting the flask from the bench, carefully run in 15 mL of water around the sides of the flask to ensure even distribution over the base; then add 65 mL of acetone (5.2.4). In order to prevent the formation of lumps, add the first 10 mL very slowly by pouring carefully around the sides of the flask while the flask is turned gently without its base leaving the bench. Allow the flask and contents to stand for 30 min and then shake for 3 h or allow to stand for between 16 h and 24 h.

Add 25 mL of sodium hydroxide solution (5.2.1) slowly with continual swirling. Shake for 3 h.

Wash down the stopper with water, adding approximately 50 mL of water to the contents of the flask. Add 25 mL of sulphuric acid (5.2.3) and about 0.5 mL of the phenolphthalein solution (5.2.5). Allow to stand, shaking if necessary, until any signs of pink coloration have disappeared from the insoluble matter. Titrate with dilute sodium hydroxide solution (5.2.2). Carry out a blank determination on the reagents used.

5.5 Calculation of result

Calculate the acetic acid yield (in %) using the following equation:

$$\text{Acetic acid yield} = \frac{3.002(A - B)}{W}$$

where

- W is the weight of the oven-dry test portion (in g);
- A is the volume of sodium hydroxide solution (5.2.2) required for the test portion (in mL);
- B is the volume of sodium hydroxide solution (5.2.2) required for the blank (in mL).

6 Method 4. Determination of bulk density

6.1 Principle

The material is allowed to flow under fixed conditions from a prescribed funnel into a measuring cylinder of predetermined mass. The density is then determined from the mass of material and its measured bulk volume.

6.2 Sampling

Select a representative sample following the principles embodied in BS 5309 and BS 6001. Pack the sample in a moisture-proof container.

NOTE Good quality, clean, dry lever-top tins have been used satisfactorily as moisture-proof containers.

6.3 Procedure

Carry out the determination of bulk density on the sample selected in accordance with 6.2 by the method described in BS 2782-6:Method 621A.

7 Method 5. Determination of the particle size distribution

7.1 Principle

A known amount of material is separated into differing amounts of material of specified minimum particle size by progressive sieving.