INTERNATIONAL STANDARD 1159

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Plastics — Vinyl chloride-vinyl acetate copolymers — Determination of vinyl acetate

Plastiques — Copolymères chlorure de vinyle-acétate de vinyle — Détermination de la teneur en acétate de vinyle

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FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 1159 was developed by Technical Committee ISO/TC 61, *Plastics*.

It was submitted directly to the ISO Council, in accordance with clause 6.13.1 of the Directives for the technical work of ISO. It cancels and replaces ISO Recommendation R 1159-1970, which had been approved by the member bodies of the following countries :

Australia	Germany	Romania
Austria	Greece	Sweden
Belgium	Hungary	Switzerland
Brazil	India	Turkey
Canada	Ireland	United Kingdom
Chile	Italy	U.S.A.
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Finland	New Zealand	
France	Poland	

No member body had expressed disapproval of the document.

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Plastics — Vinyl chloride-vinyl acetate copolymers — Determination of vinyl acetate

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining the percentage of vinyl acetate in vinyl chloride-vinyl acetate copolymers.

2 PRINCIPLE

Dissolution of a test portion in purified tetrahydrofuran, and hydrolysis of the acetate groups by ethanolic potassium hydroxide solution.

Back-titration of excess potassium hydroxide with sulphuric acid, using thymol blue as indicator.

Argentometric titration of hydrogen chloride liberated during hydrolysis.

3 REAGENTS

3.1 Potassium hydroxide, ethanolic solution approximately 0,5 N.

Dissolve 33 g of solid potassium hydroxide (KOH) in 500 ml of ethanol. Make up to 1 000 ml, allow to stand overnight and decant the clear portion of the solution.

3.2 Potassium hydroxide, ethanolic solution approximately 0,2 N.

Prepare the solution in the same way as in 3.1, but dissolving only 13,5 g of solid potassium hydroxide in the ethanol.

3.3 Sulphuric acid, 0,1 N standard volumetric solution.

3.4 Sulphuric acid, 0,05 N standard volumetric solution.

3.5 Silver nitrate, 0,1 N standard volumetric solution.

3.6 Silver nitrate, 0,05 N standard volumetric solution.

3.7 Pure tetrahydrofuran, further purified as described in 7.2.

3.8 Ethanol-water mixture (1 : 1 by volume), neutralized to thymol blue, prepared from freshly boiled distilled water.

3.9 Thymol blue indicator solution.

Dissolve 0,1 g of thymol blue in 100 ml of ethanol.

3.10 Potassium hydroxide, solid pellets.

3.11 Potassium chromate, 50 g/l solution.

4 APPARATUS

4.1 Burette, 25 ml, for acidimetry.

4.2 Burette, 10 ml, for argentometry.

4.3 Electromagnetic stirrer.

4.4 Thermostatically controlled water bath, to maintain a temperature of 30 ± 0,5 °C (see 7.1).

4.5 Analytical balance, to weigh to an accuracy of ± 0,000 1 g.

4.6 Volumetric flask, 100 ml capacity, with ground glass stopper.

4.7 Pipettes, of 1, 5, 20 and 30 ml capacity.

5 PROCEDURE

5.1 The time necessary for a quantitative saponification of vinyl acetate groups depends on the vinyl acetate content, the concentration of the ethanolic potassium hydroxide solution used, the temperature and the mass of the test portion. Consequently, choose the appropriate conditions as indicated in the table below, taking into account the assumed vinyl acetate content of the copolymer and the temperature at which saponification will take place.

5.2 Accurately weigh out the test portion of dry copolymer indicated in the table into the flask (4.6) (see 7.3) and add 20 ml of tetrahydrofuran (3.7) by means of a pipette. The dissolution of the copolymer is facilitated by using the magnetic stirrer (4.3). After complete dissolution, immerse the flask in the thermostatted bath at 30 °C and leave it for 10 min; add 5 ml of ethanolic potassium hydroxide solution of the concentration indicated in the table and thoroughly mix the contents of the flask by a gentle swirling motion. In case partial precipitation of the copolymer occurs at this stage, it must be redissolved by stirring. Allow hydrolysis to take place for the period of time prescribed in the table.

5.3 After hydrolysis, add 30 ml of ethanol-water mixture (3.8) dropwise, while stirring. This addition causes the copolymer to precipitate with a fine grain. Add 1 ml of thymol blue solution (3.9). While stirring, titrate the excess potassium hydroxide with sulphuric acid solution of the concentration indicated in the table until the deep green colour changes to orange. Under the same test conditions, carry out a blank test in the absence of copolymer, titrating from blue to yellow.

5.4 After completed acidimetric titration, add 1 ml of sulphuric acid solution (3.3), and titrate the mixture potentiometrically with silver nitrate solution of the concentration indicated in the table, stirring continuously. The volume of silver nitrate solution used is equivalent to the hydrogen chloride split off during hydrolysis of the copolymer (see 7.4).

6 EXPRESSION OF RESULTS

6.1 When using 0,1 N sulphuric acid and silver nitrate solutions, the vinyl acetate content, as a percentage by mass, is given by the formula :

$$\frac{0{,}860\,9\,(V_1 - V_2 - V_3)}{m}$$

6.2 When using 0,05 N sulphuric acid and silver nitrate solutions, the vinyl acetate content, as a percentage by mass, is given by the formula :

$$\frac{0{,}430\,4\,(V_1 - V_2 - V_3)}{m}$$

where

V_1 is the volume, in millilitres, of sulphuric acid used in the blank test;

V_2 is the volume, in millilitres, of sulphuric acid used in the determination;

V_3 is the volume, in millilitres, of silver nitrate solution used;

m is the mass, in grams, of the test portion.

6.3 Carry out two determinations. If they differ by more than 0,4 % of the vinyl acetate content, the test shall be repeated. Report the average of two acceptable determinations.

7 NOTES ON PROCEDURE

7.1 If no thermostatically controlled bath is available, it is permissible to proceed at room temperature. The appropriate conditions are given in the table.

7.2 Purification of tetrahydrofuran : Tetrahydrofuran often contains matter reacting with potassium hydroxide. Using solvent containing such impurities leads to results higher than theoretical.

TABLE — **Conditions for hydrolysis of vinyl chloride-vinyl acetate copolymers**

Assumed content of vinyl acetate	Mass of test portion	Normality of ethanolic KOH solution	Normality of H_2SO_4 and $AgNO_3$ solutions	Time of hydrolysis, h		
				using a bath (4.4) at 30 ± 0,5 °C	at room temperature :	
% (*m*/*m*)	g				20 to 25 °C	25 to 30 °C
0 to 5	0,4 to 0,5	0,2	0,05	2	3,5	2,5
5 to 10	0,18 to 0,2	0,2	0,05	2	3,5	2,5
10 to 30	0,18 to 0,2	0,5	0,1	1,5	2,5	2
30 to 60	0,18 to 0,2	0,5	0,1	2	3,5	2,5
60 and above	0,13 to 0,15	0,5	0,1	3	6	4