

Plastics piping systems — Determination of moisture content in thermoplastics by coulometry

The European Standard EN 12118 : 1997 has the status of a
British Standard

ICS 23.040.20

National foreword

This British Standard is the English language version of EN 12118 : 1997, published by the European Committee for Standardization (CEN).

This standard has the secondary identifier BS 2782 : Part 11 : Method 1103C, so that it is cross-referenced within the existing series of British Standards which cover related test methods for plastics materials and plastics piping components.

The UK participation in its preparation was entrusted to Technical Committee PRI/61, Plastics piping systems and components, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

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

The test method supports other standards that are in preparation by CEN for the specification of plastics piping and ducting systems and components.

It may be used for the revision or amendment of other national standards, but it should not be presumed to apply to any existing standard or specification which contains or makes reference to a different test method until that standard/specification has been amended or revised to make reference to this method.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled 'International Standards Correspondence Index', or by using the 'Find' facility of the BSI Standards Electronic Catalogue.

WARNING. This British Standard, which is identical with EN 12118 : 1997, does not necessarily detail all the precautions necessary to meet the requirements of the Health and Safety at Work etc. Act 1974. Attention should be paid to any appropriate safety precautions and the method should be operated only by trained personnel.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 4, an inside back cover and a back cover.

This British Standard, having been prepared under the direction of the Sector Board for Materials and Chemicals, was published under the authority of the Standards Board and comes into effect on 15 March 1998

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Amendments issued since publication

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Descriptors: Plastic tubes, plastics, chemical analysis, determination of content, water, coulometric methods

English version

Plastics piping systems — Determination of moisture content in thermoplastics by coulometry

Systèmes de canalisations en plastiques —
Détermination par coulométrie de la teneur en eau
dans les matières thermoplastiques

Kunststoff-Rohrleitungssysteme — Bestimmung des
Feuchtegehaltes in Thermoplasten mittels
Coulometrie

This European Standard was approved by CEN on 1997-05-28. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 155, Plastics piping systems and ducting systems, the secretariat of which is held by NNI.

The material-dependent parameters and/or performance requirements are incorporated in the system standard(s) concerned.

This standard is one of a series of standards on test methods which support system standards for plastics piping systems and ducting systems.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 1998, and conflicting national standards shall be withdrawn at the latest by February 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This standard specifies a method for the determination of moisture content in thermoplastics. This method is only applicable to thermoplastics for which the melting point is below 160 °C. The method is suitable for measuring the moisture content down to 0,005 %.

This method determines the total moisture content in the test piece and includes surface moisture and moisture contained within the test piece. Because the test piece is converted to a molten state, it is assumed that all moisture is expelled.

2 Principle

After calibration of a moisture analyser (see 4.1 and 6.2), a test piece is heated to (165 ± 5) °C for 10 min. The oven cell and the measuring cell are flushed with dry nitrogen. In the measuring cell, the moisture reacts chemically with phosphorus pentoxide, producing phosphoric acid.

By electrolysis, the phosphoric acid is again decomposed into water and phosphorus pentoxide.

The energy required for the electrolysis is proportional to the amount of water.

NOTE. It is assumed that the following test parameters are set by the standard making reference to this standard:

- a) the sampling procedure (see 5.1);
- b) the number of test pieces (see 5.2).

3 Materials

3.1 *Phosphorus pentoxide* (analytical grade).

3.2 *Dry nitrogen*, containing less than 5 ppm¹⁾ water content.

4 Apparatus

4.1 Equipment for reference purposes

4.1.1 *Moisture analyser*, with oven temperature capable of being controlled at (165 ± 5) °C and capable of measuring quantities of water by mass to an accuracy of within 0,1 mg, typically by use of a counter to measure energy consumption related to the mass of water involved.

4.1.2 *Nitrogen supply regulator*, capable of providing a flow rate of (70 ± 10) ml/min.

4.1.3 *Weighing cup*, of corrosion-resistant material, e.g. nickel.

4.1.4 *Weighing scale*, with accuracy of 0,01 mg.

4.1.5 *Capillary glass tube*, of 2 µl capacity, with connections to suit the moisture analyser.

4.2 Alternative equipment

Alternative equipment to that described in 4.1 may be used, provided that a correlation can be demonstrated with the results obtained using equipment conforming to 4.1.

5 Test pieces

5.1 Preparation

The test piece shall comprise a portion of 0,6 g to 0,7 g from a sample of granular material or cut from an end-product. The source and form of the sample shall be as specified in the referring standard.

5.2 Number

The number of test pieces shall be as specified in the referring standard.

6 Procedure

6.1 General

This procedure assumes the use of a counter as described in 4.1. For other forms of read-out (see 4.2), it is necessary to modify the procedure or calculations accordingly to suit readings obtained in place of Q_b and Q_c (see 6.2).

6.2 Calibration

6.2.1 Make a blank determination as follows.

- a) If applicable, set the counter to zero. Put the cup (4.1.3) in the oven and heat it at (165 ± 5) °C for 10 min.
- b) Switch off the heater and cool by dry nitrogen flow for 3 min. Then read the counter and record the count, Q_b .

6.2.2 Obtain a calibration value as follows.

- a) Introduce into the moisture analyser, in place of a test piece, the capillary glass tube (see 4.1.5) containing 2 µl of distilled water or an equivalent amount of sodium tartrate dihydrate.
- b) Heat the tube at (165 ± 5) °C for 10 min.
- c) Read the counter and record the count, Q_c .

6.2.3 Calculate the calibration factor, f_c , using the following equation:

$$f_c = 0,5(Q_c - Q_b)$$

If the value of f_c lies within the range 0,9 to 1,1, consider the test conditions to be satisfactory.

NOTE. The blank value (see 6.2.1) represents the quantity of moisture introduced into the apparatus by opening the oven, and the moisture contained in the nitrogen current.

6.3 Test

6.3.1 Weigh the test piece in a dry cup (4.1.3).

6.3.2 Record the actual mass, m_g , of the test piece, to the nearest 0,1 mg.

6.3.3 Introduce the cup into the oven (4.1.1).

6.3.4 Keep the cup in the oven for 10 min at (165 ± 5) °C, then switch off the heater and allow the cup to cool for the next 3 min. After this time, read the counter, and record the count, Q_s .

¹⁾ppm = parts per million.

6.3.5 Calculate the total moisture content, m_w , by using the following equation:

$$m_w = f_c \frac{(Q_s - Q_b)}{m_g}$$

where

- f_c is the calibration factor;
- Q_b is the blank count number, in micrograms (μg);
- Q_s is the test portion piece count number, in micrograms (μg);
- m_g is the actual mass of the test portion piece, in grams (g);
- m_w is the total moisture content, in (mass/mass) $\times 10^6$ (e.g. $\mu\text{g/g}$ or mg/kg).

7 Test report

The test report shall include the following information:

- a) reference to this standard and to the referring standard;
- b) full identification of the test piece;
- c) the calibration factor, f_c ;
- d) the total moisture content, m_w , in milligrams per kilogram (mg/kg) (see **6.3.5**) or in percent mass;
- e) any factors which may have affected the results, such as any incidents or any operating details not specified in this standard;
- f) the date of test.

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