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# Textiles — Quantification of carbon fibre constituent element — Elemental analyser method

*Textiles — Quantification des éléments constitutifs des fibres de carbone — Méthode de l'analyseur élémentaire*





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This document was prepared by Technical Committee ISO/TC 38, *Textiles*.

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# Introduction

Carbon fibre has drawn much attraction in various industries due to its high stiffness, specific strength and anti-corrosion. These outstanding properties of carbon fibre enable the expansion of its application from textile usage to mechanical parts used in automobile and aircraft industry, if carbon fibre is used as a reinforced component in polymer matrix.

In order to accelerate the trend of productization using carbon fibre, there is a prerequisite that the carbon content in the fibre should be evaluated quantitatively. In addition, it is difficult to issue a test report because even an accredited test organization cannot provide a clear method of quantification.

X-ray photoelectron spectroscopy is one of the measurement method suitable for analysis of chemical components with quality and quantity. However, its detecting area is too small to cover the entire fibre.

This document aims to quantifz carbon content in textiles and textile products including PAN-based carbon fibre using elemental analyser (EA) and gas chromatography (GC), successively. Furthermore, this method can also analyse the contents of H and N, simultaneously.

# Textiles — Quantification of carbon fibre constituent element — Elemental analyser method

## 1 Scope

This document specifies a quantitative measurement of chemical constituent element on carbon fibre and its textile by an elemental analyser.

## 2 Normative references

There are no normative references in this document.

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/



**3.1**
**polyacrylonitrile**
**PAN**
synthetic, semicrystalline organic polymer resin for carbon fibre production



## 4 Principle

The carbon fibre constituent elements are determined with the quantification method by using an elemental analyser (EA). All types of textiles and textile product or samples, including PAN-based carbon fibre, are oxidised in a carbon fibre by dynamic flash combination method in a high purity oxygen environment, separated on gas chromatography column, and analysed using a thermal conductive detector (TCD). When the tin boat with sample is dropped in to the reactor, the oxygen environment triggers a strong exothermic reaction. Temperature rises approximately to 1 200 °C, causing the sample to combust. The combustion products are conveyed across the reactor, where oxidation is completed. Nitrogen oxides and sulfur trioxide are reduced to elemental nitrogen and sulfur dioxide and oxygen excess is retained. The gas mixture containing $N_2$, $CO_2$, $H_2O$ and $SO_2$ flows into the chromatographic column, where separation takes place. Eluted gases are sent to the TCD where electrical signals processed by the EA software provide percentages of nitrogen, carbon, hydrogen, and sulfur contained in the sample.

## 5 Reagents and materials

Unless otherwise specified, chemicals of analytical grade shall be used.

**5.1** **Toluene**, CAS No. 108-88-3[1)]

1) Chemical Abstracts Service (CAS) Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

**SAFETY PRECAUTIONS — The safety precautions for the harmful effects of this reagent shall be considered and shall be taken during use.**

**5.2 Helium,** with minimum purity of 99,999 % used as carrier gas.

**5.3 Oxygen,** with minimum purity of 99,999 %, used as oxidation gas.

**5.4 Standard and calibration standard materials,** is shown in the Table 1**.**

Standard materials shall be compounds not contained in the test sample and completely separated from other components in chromatogram analysis. The materials shall be inert to sample composition, and stable within a test temperature range and their purity shall be obviously known.

**Table 1 — List of standard and calibration standard material**

| Material | Compound | Purpose |
|---|---|---|
| Aspartic acid | $C_4H_7NO_4$ | Standard reference material |
| 2.5-Bis(5-tert-butyl-benzoxaz-ol-2-yl) thiophene (BBOT) | $C_{26}H_{26}N_2O_2S$ | Standard reference material |
| Sulfanilamide | $C_6H_8N_2SO_2$ | Calibration standard |
| L-Cystine | $C_6H_{12}N_2O_4S_2$ | Calibration standard |



## 6 Apparatus

**6.1 Vial**, with a capacity of approximately 25 ml.

**6.2 Desiccator**, containing desiccant (silica gel, calcium chloride anhydride, calcium sulfate anhydride) to dry solvent and cool down to test specimens.



**6.3 Volumetric graduated pipette**, with capacity of approximately 5 ml and 10 ml.

**6.4 Thermostatic ultrasonic bath**, capable by operating by a frequency of 40 kHz.

**6.5 Vacuum oven**, capable to dry test specimen at least at 80 °C.

**6.6 Analytical balance**, with a resolution of at least 0,01 mg for weighing the standard materials.

**6.7 Elemental analyser equipment**

**6.7.1 Oxidation reactor, GC column and adsorption trap**

The equipment shall be installed and used according to the manual provided by their manufacturer. All the parts coming in contact with a test specimen shall be made of materials which are resistant to the sample and do not generate any chemical change.

**6.7.2 TCD detector,** with gas supply for the detector, injected sample volume, separation ratio and sensitivity adjustment shall be optimized so that the signal (peak area) utilized in calculation is proportional to the material amount.

**6.7.3 Oven**, capable to dry at (105 ± 5) °C.

**6.7.4** **Tin boat**, with a disc-type holder in which a pretreated sample of carbon fibre is located for the EA measurement.

Any unavoidable deposit of dust shall be regularly removed.

# 7 Preparation of test specimen

## 7.1 General

**7.1.1** If the test specimen are yarns, unravel the fibres and cut them to about 50 mm ± 5 mm in length.

**7.1.2** If the test specimen is all kinds of fabrics, cut the specimen pieces to 50 mm × (50 mm ± 5 mm).

In case of woven fabric, unravel warp and weft yarns in order to get couple(s) of representative yarns from two different places of each direction.

Fabrics or yarns may contain of different components, so take this into account when collecting them.

## 7.2 Desizing

Cut test specimen about 50 mm in length and put test specimen in a vial (6.1). Add 20 ml of toluene (5.1). If the test specimen is not sufficiently immersed in the solvent, add more solvent. Close the vial tightly and extract the test specimen at (60 ± 5) °C for (60 ± 5) min in an ultrasonic bath. Cool down to room temperature to less than 27 °C.



After desizing, test specimens shall be dried at 80 °C for 5 h in a vacuum oven (6.5) and kept in the desiccator (6.2).

Do not touch the specimen with bare hands during drying and mass measurements.



## 7.3 Drying

The vial (6.1) and tin boat (6.7.4) dry at 105 °C to 110 °C for 4 h to 16 h in oven (6.7.3) to a constant mass.

After Drying, cool them in the desiccator (6.2).

Do not touch the specimen with bare hands during drying and mass measurements.

## 7.4 Cutting

Unravel the test specimen and cut them to about 10 mm. Cut this specimen to 1 mm or less and take 1 mg to 2 mg of specimen.

# 8 Test procedure

## 8.1 Preparation of dosing test specimen

**8.1.1** Break clumpy test specimen into pieces and drying completely.

**8.1.2** Weight the tin boat using analytical balances (6.6) and zero it.

**8.1.3** The dosing test specimen is placed in a tin boat and weight using analytical balances (6.6).

Do not touch the dosing test specimen with bare hands during drying and mass measurements.

### 8.2 Procedure

**8.2.1** Prepare the instrument for running dosing test specimens.

**8.2.2** The weighed dosing test specimen with tin boat (8.1.3) is located on a tin boat to be inserted into an elemental analyser.

The above measurement should be repeated at least five times from five different doses obtained from the test specimen.

The experimental parameters such as the amount of a sample, flow rates of carrier gases, etc. are described in Annex A in details.

**8.2.3** Summarize, edit and analyse results.

## 9 Calculations and display results

Electrical signal data converted by the software in the TCD give information on the amount (%) of carbon, hydrogen and nitrogen in the sample.

The quantification of the element that has been analysed is determined by comparing the values obtained from the analysis of the sample with the analysis of the use of a reference factor according to Formula (1):

$$K = (A_S - A_B) / ((T \, x \, W) / 100) \quad (1)$$



where

$K$ is the average K-Factor;



$A_S$ is the peak area or integral of standard material;

$A_B$ is the peak area or integral of blank;

$T$ is the theoretical percentage of standard material, in %;

$W$ is the mass of standard material, in g.

The calculation of the percentage of element (%) is given by Formula (2):

$$C_a = ((A_U - A_B) / K) / W \times 100 \quad (2)$$

where

$C_a$ is the calculated percentage of element, %;

$K$ is the average K-Factor;

$A_U$ is the peak area or integral of the unknown;

$A_B$ is the peak area or integral of the blank;

$W$ is the mass of the unknown, in g.

Test results shall be calculated up to two decimal places and end at one decimal place.