~~DRAFT INTERNATIONAL STANDARD~~

**ISO/FDIS 5533**

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Secretariat:- SAC

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

*Textiles~~— quantification de la teneur en fibre~~ — Quantification des éléments constitutifs des fibres de carbone — Méthode ~~d'analyse~~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 ~~been~~ drawn much attraction in various industries due to its high stiffness, specific strength and anti-corrosion. These outstanding properties of carbon fibre enable ~~us to expand~~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 ~~which is~~ suitable for ~~anaysis~~analysis of chemical components with quality and quantity. However, its detecting area is too small to cover the entire ~~fiber~~fibre.

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

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# 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 terminologicalterminology databases for use in standardization at the following addresses:

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

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

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

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## 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 each element 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 be combustedcombust. 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 chemicals shall be used.

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

**SAFETY PRECAUTIONS — The safety precautions for the harmful effects of this reagent shall be ~~borne in mind,~~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.~~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-benzoxazol-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 |

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## 6 Apparatus

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

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**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.

[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.



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