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BSI Standards Publication

Aerospace series — Non-metallic materials — Textiles — Test method — Determination of water soluble chloride and sulfate of aqueous extracts

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National foreword

This British Standard is the UK implementation of EN 4503:2013.

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

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Série aérospatiale - Matériaux non-métalliques - Textiles - Méthode d'essai - Détermination des chlorures et des sulfates des résidus aqueux

Luft- und Raumfahrt - Nichtmetallische Werkstoffe - Textilien - Prüfverfahren - Bestimmung von wasserlöslichem Chlorid und Sulfat in wässerigen Lösungen

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EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Page

Foreword

This document (EN 4503:2013) has been prepared by the Aerospace and Defence Industries Association of Europe - Standardization (ASD-STAN).

After enquiries and votes carried out in accordance with the rules of this Association, this Standard has received the approval of the National Associations and the Official Services of the member countries of ASD, prior to its presentation to CEN.

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of an identical text or by endorsement, at the latest by January 2014, and conflicting national standards shall be withdrawn at the latest by January 2014.

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Introduction

This standard is part of the series of EN non-metallic materials standards for aerospace applications. The general organisation of this series is described in EN 4385. This standard is a level 3 document as defined in EN 4385.

1 Scope

This European Standard specifies the determination of water soluble chloride and sulphate of aqueous extracts of textile materials.

This method has been written in response to an aerospace requirement for a method of extraction using hot water, this method should be used in conjunction with EN 4426.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 4385, *Aerospace series — Non-metallic materials – General organisation of standardization — Links between types of standards*

EN 4426, *Aerospace series — Non-metallic materials — Textiles — Test method — Determination of conductivity and pH of aqueous extracts* 1)

EN 20139, *Textiles — Standard atmospheres for conditioning and testing (ISO 139)*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

ISO 383, *Laboratory glassware — Interchangeable conical ground joints*

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

3 Terms and definitions

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

3.1
chloride
free water soluble Cl^- ion which could combine with free H^+ ions to produce acidic conditions which would be detrimental to the ageing of fabrics

3.2
sulfates
free water soluble SO4C ion which could combine with free H+ ions to produce acidic conditions which would be detrimental to the ageing of fabrics

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1) Published as ASD-STAN Prestandard at the date of publication of this standard

4 Health, safety and environment

This standard does not necessarily include all health and safety requirements associated with its use.

Persons using this standard shall be familiar with normal laboratory/test house practices.

It is the responsibility of the user to establish satisfactory health and safety practices and to ensure conformity with any European, National or local laws/regulations.

5 Principle/Technique

An aqueous extract is prepared using distilled water. The soluble chloride and sulphate are then measured by gravimetric method. A volumetric method may also be used to determine the amount of soluble chloride.

6 Resources

6.1 Apparatus/facilities

6.1.1 Round bottomed flasks of chemically resistant glass with a volume of 250 ml and a ground glass neck of size 24/29 in accordance with ISO 383

6.1.2 A glass stopper incorporating a stopcock with P.T.F.E. core liner to prevent sticking of the glass core in the neck of the stopcock

NOTE Grease shall not be used for this purpose.

6.1.3 Water-cooled condensers

6.1.4 Laboratory balance, accurate to 0,000 2 g

6.1.5 Sinter glass crucibles, porosity 4 in accordance with ISO 4793

6.1.6 Titration vessel and reference half-cell, with suitable pH meter as used with glass electrodes, set to read in mV

NOTE The half-cell is filled with silver oxalate suspension.

6.1.7 Sintered porcelain or silica gooch filter crucibles, porosity 4 in accordance with ISO 4793

6.1.8 Filter paper with the following nominal characteristics:

- mass of 100 g/m^2
- retention 2,5 μm
- initial filtration speed – slow
- thickness 0,2 mm
- ash content 0,007 %

NOTE Whatman 42 has been found suitable.

6.2 Materials/reagents

The following reagents are required and shall be of recognized analytical quality.

6.2.1 Potassium chloride solution, of concentration 0,001 mol/1, which has a conductivity of 12,780 mS/m at 20 °C

6.2.2 Distilled or deionized water, in accordance with EN ISO 3696. It should have a maximum conductivity of 1 mS/m. For pH determinations remove carbon dioxide from the water by boiling for 5 min, then cool in the absence of air before use.

6.2.3 Silver oxalate suspension, made by dissolving 14 g of sodium oxalate and 10 g of potassium nitrate in 1 litre of water (6.2.2) adding with constant stirring, 100 ml of silver nitrate (7.2.7). The stock suspension shall be kept in a dark glass bottle.

6.2.4 Nitric acid solution, concentrated

6.2.5 Nitric acid solution, approximately 0,5 % v/v

6.2.6 Nitric acid solution, approximately 10 % v/v

6.2.7 Silver nitrate solution, 0,1 mol/l

6.2.8 Silver nitrate solution, 0,01 mol/l

6.2.9 Sodium chloride solution, approximately 100 g/l

6.2.10 Hydrochloric acid solution, concentrated

6.2.11 Barium chloride solution, 20 g/l

6.3 Qualification of personnel

No specific technical requirements.

7 Test samples/test pieces

Samples shall be taken representative of the bulk and of sufficient size to provide all the test specimens required. All samples shall be kept identifiable to the bulk textiles which they represent. Cut the sample under test into pieces of such size that all parts readily wet out.

Care shall be taken to avoid contamination of samples and handling shall be kept to an absolute minimum.

NOTE Nominal 10 mm squares have been found suitable.

The samples are conditioned and tested in a standard atmosphere of (65 ± 2) % r.h. and (20 ± 2) °C in accordance with EN 20139 – Standard Temperate Atmosphere For Testing.

8 Test procedure

8.1 Preparation

Cut the sample into nominal 10 mm squares and weigh (5 ± 0,05) g of conditioned sample into flask (6.1.1).

To the flask containing the sample under test add (100 ± 0,1) ml of distilled water (6.2.2).

NOTE For sample weight of less than 5 g, the liquor ratio should be maintained at 1: 20, i.e. 1 g of sample to 20 ml of water.

Connect the flask containing the sample and water to the water cooled condenser (6.1.3). Quickly bring contents to the boil and continue to boil liquor gently for 60 minutes. After this period disconnect and remove flask from the condenser whilst liquor is still boiling close immediately with the glass stopcock (6.1.2).

Do not filter or make up volume but cool rapidly to (20 ± 2) °C to ensure partial vacuum is created to ensure the extract is not contaminated.

8.2 Determination of water soluble chloride

8.2.1 Gravimetric method. Prepare an extract as described in 8.1 If the textile contains chlorinated compounds, e.g. PCPL, carry out a preliminary extraction with a suitable solvent before preparing the extract as described in 8.1. Take a suitable measured portion, add 5 ml of concentrated nitric acid (6.2.4) per 100 ml of extract boil for 5 min and leave overnight. Filter through a filter paper pulp pad, wash with distilled water, and add a slight excess of silver nitrate solution (6.2.7) to the combined filtrate and washings. Heat the solution, protected from direct light, in a water bath until the precipitate is coagulated and the supernatant liquor is clear. Verify completeness of precipitation by adding a drop of silver nitrate solution (6.2.7) to the supernatant liquor. Allow to cool in the dark, and then filter through a tarred crucible (6.1.5). Wash the crucible with dilute nitric acid (6.2.5) until the washings give no opalescence when tested with sodium chloride solution (6.2.9).

Then dry the crucible initially at about 100 °C and finally to constant mass [2] at 130 °C to 150 °C. Carry out a blank determination and subtract the mass thus obtained from the mass obtained in the test. Calculate the water-soluble chlorides as described in .9.2.1.1.

8.2.2 Volumetric method. Before each test, verify that all parts of the apparatus are clean. Clean the silver wire electrodes with very fine abrasive or use a suitable chemical method. Discard and replace the silver oxalate suspension (6.2.3) at the first sign of darkening; to delay darkening, shield the half-cell from light when not in use. Shake the suspension bottle thoroughly before replenishing the half-cell.

Take particular care that electrolytes do not come into contact with the junctions between the silver wire electrodes and their leads to the pH meter. Flush the electrolyte junction after each determination by easing the stopper at the bottom of the half-cell to allow fresh oxalate suspension (6.2.3) to flow into the junction.

Prepare an extract as described in 8.1.

Take a suitable measured portion (a suitable test portion should contain a minimum of 0,2 mg of chloride ion), add 1 ml of nitric acid (6.2.6) per 100 ml of extract, boil for 5 min, cool rapidly to room temperature and transfer to the titration vessel. Start the stirrer, connect the electrodes to the pH meter, set to read in mV, and titrate with silver nitrate solution (6.2.8) until a reading of 0 mV is first indicated. Carry out a blank determination and calculate the water soluble chlorides as described in 9.2.1.2.

8.3 Determination of water-soluble sulfates by gravimetric analysis

Prepare an extract as described in 8.1. Take a measured portion, filter through a suitable filter paper (6.1.8) and wash with distilled water. Add concentrated hydrochloric acid (6.2.10) drop by drop until the solution is just acid to litmus, then add 1 ml of acid per 100 ml solution. Boil for 5 min and leave to cool overnight. Filter off any precipitate on a filter-paper pulp pad, wash with distilled water and heat the combined filtrate and washings to boiling. While still boiling add, drop by drop, 10 ml of hot barium chloride solution (6.2.11).

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2) That is until the residue suffers no more than 0,0005 g loss of mass on drying for a further 30 min.

Continue boiling for 30 min, then leave to cool overnight. Transfer the precipitate quantitatively to a crucible (6.1.5), and wash with cold water (6.2.2) until the washings are free from chloride. Heat the crucible and its contents, gently at first, and finally to constant mass* at 800 °C to 900 °C.

Carry out a blank determination and calculate the water-soluble sulphates as described in 9.2.2.

9 Expression of results

9.1 Individual results

State which method of extraction and what ratio of water to textile have been used,

NOTE For the calculation of the amount of chloride, and sulphate, it should be noted that 100 ml of extract are equivalent to 5,0 g of conditioned specimen, except for wool in any textile form and for felts and loose fibre masses of any composition, where 100 ml of extract are equivalent to 2,0 g of conditioned specimen.

In the equations given overleaf, masses are expressed in grams and volumes in millilitres.

9.2 Calculation

9.2.1 Water-soluble chlorides

The mass of chloride ion (P_c), as a percentage by mass of the conditioned mass of the specimen, is given by the equations in 9.2.1.1 and 9.2.1.2.

9.2.1.1 Gravimetric

a) For yarns or fabrics other than wool:

$$P_c = \frac{495 \times m}{V}$$

b) For felts, loose fibres and wool in any textile form:

$$P_c = \frac{1\,237 \times m}{V}$$

where:

m is the mass of residue, corrected for blank (g);

V is the volume of extract taken (ml).

c) The percentage of water soluble chlorides expressed as sodium chloride is:

$P_c \times 1{,}65$

9.2.1.2 Volumetric

a) For yarns or fabrics other than wool:

$$P_c = \frac{0{,}71 \times v}{V}$$

b) For felts, loose fibres and wool in any textile form:

$$P_c = \frac{1{,}77 \times v}{V}$$

where:

v is the volume of $AgNO_3$ used (6.8) corrected for blank (ml);

V is the volume of extract taken (ml).

c) The percentage of water soluble chlorides, expressed as sodium chloride is:

$P_c \times 1{,}65$

9.2.2 Water soluble sulfates

The mass of sulfate ion (P_s), as a percentage by mass of the conditioned mass of the specimen, is given by the following equations.

a) Yarns or fabrics other than wool:

$$P_s = \frac{823 \times m}{V}$$

b) Felts, loose fibres and wool in any textile form:

$$P_s = \frac{2\,058 \times m}{V}$$

where:

m is the mass of precipitate connected for blank (g);

V is the volume of extract taken (ml).

c) The percentage of water-soluble sulphates, expressed as anhydrous sodium sulphate is:

$P_s \times 1{,}48$

10 Measurement uncertainties

Not applicable.

11 Designation

Not applicable.

12 Test report

The report shall include the following information:

a) Reference to this European Standard, i.e. EN 4503;

b) Identification of sample tested (i.e. lot number, batch number);

c) The content of water-soluble chlorides as a percentage of the conditioned mass of the specimen, or expressed as sodium chloride;

d) The content of water-soluble sulphates as a percentage of the conditioned mass of the specimen, or expressed as anhydrous sodium sulphate;

e) Traceability to the individual who performed the test;

f) Date of test;

g) Details of any deviation from the test method.

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