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Textiles — Determination of index ingredient from coloured textile —

Part 3: **Myrobalan**

Textiles — Détermination d'indicateurs d'ingrédients de textiles colorés —
Partie 3: Mirobolant

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Foreword

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

A list of all parts in the ISO 22195 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

There is no doubt that dyeing plays the most important role in expressing the colour of clothes. Until the invention of synthetic dyes capable of expressing diverse colours, materials obtained from nature to dye fabric have been used. Typically, colourants were obtained from plants or various materials were extracted from minerals or insects. When dyeing fabrics using materials derived from these natural substances, it becomes necessary to identify which substances the colourant was derived from. In other words, there has been a demand to confirm whether a fabric is dyed using a natural substance.

A test method is developed to identify which type of natural substances has been used.

Textiles — Determination of index ingredient from coloured textile —

Part 3: Myrobalan

1 Scope

This document specifies a test method for the determination of the index ingredient of chemicals in coloured fabric with myrobalan.

2 Normative references

The following document is referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms and definitions

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

ISO and IEC maintain terminological 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
myrobalan
common name for fruits of *Terminalia Chebula* and *T. Bellirica* which are a genus of large deciduous plants in the *Terminalia* family

Note 1 to entry: The genus and its best-known species are commonly known as myrobalan. They grow throughout South and Southeast Asia including in India, Sri Lanka, Bhutan, Nepal, Thailand, and China. The plant's fruits are rich in tannin such as chebulagic acid, ellagitannins and phenolic compounds. They give yellow, khaki and grey colour to the dyed textile.

3.2
coloured
expressing of colours to textiles by dyeing or printing

3.3
natural colourant
colourant obtained from plants, wood, rocks, soil, insects or any other thing existing on earth without any chemical reaction adopted before colouring of textiles

4 Principle

Natural colourants usually contain several chemical constituents. Depending on the type of natural colourant, each contains a distinctive chemical. This characteristic chemical remains in the fabric dyed

with natural colourant. Therefore, analysis of natural coloured fabrics by chromatography can detect characteristic chemicals depending on the kind of natural colourant.

NOTE If the index component chebulagic acid is detected through this test method, it cannot be said that it is necessarily stained with chebulic myrobalan alone. However, based on this principle, applying this test method to unknown coloured fabrics or textiles is useful to provide a minimum amount of information that can be used to confirm whether the fabric is coloured using chebulic myrobalan.

5 Reagent

Unless otherwise specified, use only reagents of recognized HPLC grade.

5.1 Water, glass double distilled water or grade 2 water in accordance with ISO 3696.

5.2 Methanol (CAS 67-56-1).

5.3 Acetonitrile (CAS 75-05-8).

5.4 Formic acid (CAS 64-18-6), volume fraction of 30 %.

5.5 Chebulagic acid (CAS 23094-71-5), reference standard with percentage purity indication e.g. 85,0 % or more.

It is recommended to observe the Safety Data Sheet for Chemicals (FISPQ) of each reagent.

6 Apparatus

6.1 Analytical balance, resolution at 0,001 g.

6.2 Ultrasonic water bath, to be set up at (30 ± 2) °C.

6.3 Borosilicate glass container, 50 ml.

6.4 Rotary evaporator.

6.5 Membrane filter, with 0,2 µm pore size.

6.6 High performance liquid chromatograph with mass spectroscopy (HPLC-MS).

7 Procedure

7.1 Standard preparation

Stock solution of chebulagic acid is prepared in methanol containing 1 000 mg/l.

7.2 Preparation of test specimen

Cut the test specimen into pieces of approximately 5 mm × 5 mm and approximately 1 g. Weigh it to the nearest 0,01 g, and then place it into the glass container (6.3).

Pipette 10 ml of methanol (5.2) each into the other glass container (6.3) and pour it to cut test specimen containing glass container (6.3). Place the glass container containing the test specimen into an

ultrasonic bath (6.2) at (30 ± 2) °C for (20 ± 1) min. Afterwards, let the extract cool down to room temperature. Dilute if necessary.

Filter about 1 ml of the extracted solution into a HPLC vial using disposable syringe equipped with a membrane filter (6.5).

7.3 Analysis

The detection and identification of chebulagic acid is conducted using HPL-MS (6.6). The recommended chromatographic conditions are given in Annex A.

7.4 Determination and calculation

7.4.1 Determination of chebulagic acid

Comparison between analyses of standard and test specimen through 7.3 can show the result of existence of chebulagic acid in test specimen.

Detection of chebulagic acid may vary due to conditions of coloured test specimen. In this case, the amount of test specimen and extraction solution can be modified, and concentration of extracted solution can be adopted. The modified test specimen preparation conditions should be described in test result.

7.4.2 Calibration curve

Calibration curves with standards of chebulagic acid at 1 mg/l, 10 mg/l, 20 mg/l, 50 mg/l, and 100 mg/l are prepared with at least 5 calibration points.

NOTE Concentration ranges for the calibration standards are subject to change upon the need of each laboratory and equipment used.

For quantification, the calibration curve shall have a correlation coefficient greater than 0,995(R^2 greater than 0,990).

7.4.3 Calculation of chebulagic acid

Calculate the concentration of each chebulagic acid in the test specimen in mg/kg by Formula (1)

$$C_s = \frac{C_1}{W} \times f \times V \quad (1)$$

where

C_S is the concentration of each chebulagic acid in the test specimen, in mg/kg;

C_1 is the concentration of each chebulagic acid in the test specimen solution, in mg/l;

W is the mass of the test specimen, in g;

f is the dilution factor;

V is the final extraction volume, in ml;

8 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 22195-3:2023;

b) identification of the test specimen;

c) the concentration of chebulagic acid, in mg/kg

d) conditions of chromatographic analysis;

e) any deviation from the specified procedure in this document;

f) date of the test.