SLOVENSKI STANDARD
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Splošne metode preskušanja pigmentov in polnil - 19. del: Določevanje nitratov, topnih v vodi (metoda s salicilno kislino) (ISO/FDIS 787-19:2020)

General methods of test for pigments - Part 19: Determination of water-soluble nitrates (Salicylic acid method) (ISO/FDIS 787-19:2020)

Allgemeine Prüfverfahren für Pigmente und Füllstoffe - Teil 19: Bestimmung der wasserlöslichen Nitrate (Salicylsäure-Verfahren) (ISO/FDIS 787-19:2020)

Méthodes générales d'essais des pigments - Partie 19: Détermination des nitrates solubles dans l'eau (Méthode à l'acide salicylique) (ISO/FDIS 787-19:2020)

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Part 19: Determination of water-soluble nitrates (Salicylic acid method)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 256, *Pigments, dyestuffs and extenders*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 298, *Pigments and extenders*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 787-19:1974), which has been technically revised.

The main changes compared to the previous edition are as follows:

— the normative references have been updated;

— the document has been editorially revised.

A list of all parts in the ISO 787 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.

General methods of test for pigments —

Part 19: Determination of water-soluble nitrates (Salicylic acid method)

1 Scope

This document specifies a general method of test for determining the water-soluble nitrates in a sample of pigments by a spectrophotometric method using salicylic acid.

ISO 787-13 specifies a method for determining the water-soluble nitrates in a sample of pigments using Nessler`s method.

2 Normative references

The following documents are 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 648, *Laboratory glassware — Single-volume pipettes*

ISO 835, *Laboratory glassware — Graduated pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

No terms and definitions are listed in this document.

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 http://www.electropedia.org/

4 Principle

The nitrate present in the extract of the pigment sample is used to nitrate salicylic acid in sulphuric acid medium. The nitro-compound formed is of an intensive yellow colour in alkaline solution and the colour is measured spectrometrically at a wavelength of 410 nm.

5 Reagents

All reagents used shall be of recognized analytical reagent quality. Distilled water, or water of equivalent purity, shall be used.

5.1 Sulphuric acid, ρ = 1,84 g/ml.

5.2 Sulphuric acid, 5 N.

5.3 Ethanol, a volume fraction of 95 %.

5.4 Sodium salicylate, 5 g/l.

5.5 Sodium hydroxide, 300 g/l solution.

5.6 Sodium hydroxide, 4 N solution.

5.7 Potassium nitrate, dried at 120 °C and cooled in an desiccator.

6 Apparatus

6.1 Spectrophotometer, suitable for measurements at a wavelength of 410 nm.

6.2 10 mm cells for use with spectrophotometer.

6.3 ph meter.

6.4 One-mark volumetric flask, of a capacity 50 ml, 100 ml, 250 ml and 500 ml, according to ISO 1042.

6.5 Pipettes, capacity 10 ml, according to ISO 648 and ISO 835.

7 Sampling

Take a representative sample of the product to be tested according to ISO 15528.

8 Preparation and calibration graph

8.1 Standard solution I

Weigh 163 ± 0,1 mg of the potassium nitrate (5.7), dissolve it in water in the 100 ml one-mark volumetric flask, make up to the mark and mix well.

8.2 Standard solution II

Pipette 10 ml of the standard solution I in to a 500 ml one-mark volumetric flask, make up to the mark and mix well.

8.3 Construction of graph

Pipette 2 ml, 4 ml, 6 ml, 8 ml and 10 ml of the standard solution II (corresponding to 0,04 mg, 0,08 mg, 0,12 mg, 0,16 mg and 0,2 mg of NO_3 respectively) into separate 100 ml beakers.

To each beaker, add 1 ml of sodium salicylate solution (5.4), evaporate to dryness on a water-bath and allow cooling in a desiccator. Moisten each dried residue with 1 ml of the sulphuric acid (5.1) and allow standing in the desiccator for 10 min. Afterwards, wash the contents into separate 50 ml one-mark volumetric flasks with water, add 10 ml of the sodium hydroxide solution (5.5) to each and cool to room temperature.

Make up to the mark with water and mix well. Determine and record the absorbance of each of the solutions at 410 nm in 10 mm cells against a solution prepared in the same way as the previous solutions but omitting the nitrate solution.

Construct a graph of absorbance against the mass of NO_3 in milligrams.

9 Procedure

9.1 Pipette into a 250 ml one-mark volumetric flask 50 ml of the clear aqueous extract obtained, as appropriate for the pigment under test, by the hot extraction method (see ISO 787-3), or the cold extraction method (see ISO 787-8). Make up to the mark with water and mix.

If the aqueous extract contains chromate proceed as follows:

Place 50 ml of the clear aqueous extract into a 250 ml glass beaker and add 5 ml of sulphuric acid (5.2) and 2 ml of ethanol (5.3). Heat the solution until the chromate present is reduced as indicated by the blue-green colour of the solution and the absence of aldehyde odour; take care to avoid losses by splashing. Cool and add sodium hydroxide solution (5.6) until just alkaline. Cool again and adjust the pH to 8 ± 0,5 measured by the pH meter (6.3). Filter through filter paper and wash with hot water, collecting the filtrate and washings in a 250 ml one-mark volumetric flask. Cool, make up the mark and mix.

9.2 Pipette 10 ml of this solution to a 100 ml glass beaker.

If the nitrate content is found to be greater than 0,1 %, carry out a second determination, using 5 ml of solution.

9.3 Add to the beaker 1 ml of sodium salicylate solution (5.4) and proceed as specified in Clause 8, including the determination of the absorbance at 410 nm.

9.4 From the known absorbance of the test solution, determine from the calibration graph the corresponding mass of nitrate in milligrams.

10 Expression of results

Calculate the water–soluble nitrate content expressed as NO_3, as a percentage by mass, with Formula (1):

$$\frac{25a}{2m} \tag{1}$$

where

a is the mass, in milligrams, of NO_3 corresponding to the absorbance of the test solution;

m is the mass, in grams, of the pigment from which the clear aqueous extract was obtained.

NOTE If 5 ml of the extract was taken because the nitrate content was greater than 0,1 %, the formula becomes $\frac{25a}{m}$.

11 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested (type, identification, etc.);

b) a reference to this document, i.e. ISO 787-19:2020;

c) the result of the test, and weather the hot or the cold extraction method was used;

d) any deviation, agreed or otherwise, from the test procedure specified;

e) the date of the test.