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ISO 11127-7

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Preparation of steel substrates before application of paints and related products — Test methods for non-metallic blast-cleaning abrasives —

Part 7:

Determination of water-soluble chlorides

Préparation des subjectiles d'acier avant application de peintures et de produits assimilés — Méthodes d'essai pour abrasifs non métalliques destinés à la préparation par projection —

Partie 7: Détermination des chlorures solubles dans l'eau

Reference number
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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).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 35, *Paints and varnishes*, Subcommittee SC 12, *Preparation of steel substrates before application of paints and related products,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 139, *Paints and varnishes,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 11127-7:2011), which has been technically revised.

The main changes are as follows:

- an Introduction has been added;
- the scope has been expanded to include spectrophotometric and ion chromatographic methods of analysis;
- Clauses 3, 4, 9 and 10 have been added;
- the list of apparatus in Clause 5 has been expanded;
- Clause 8 has been renamed;
- Annex A has been deleted.

A list of all parts in the ISO 11127 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

Amperometric titration against silver nitrate has been the established method of determining water-soluble chlorides in non-metallic abrasives since the first edition of this document. Subsequently, other methods of analysis offering faster and more sensitive determinations, namely ion chromatography and spectrophotometry, have gained popularity. This document specifies three methods of analysis for water-soluble chlorides.

The spectrophotometric determination of water-soluble chloride relies on the dissociation of mercuric thiocyanate by chloride ions. In the presence of ferric ion, the liberated thiocyanate ion forms the dark red coloured ferric thiocyanate complex in a concentration which is proportional to the original chloride ion concentration. The concentration of the ferric thiocyanate is determined by measuring the absorbance at 450 nm. This method can be used to determine chloride concentrations as low as 0,1 mg/l. Commercially available reagent kits offer determinations in the ranges 0,1 mg/l to 25 mg/l and 1 mg/l to 70 mg/l. Portable spectrophotometers allow the adoption of this method as field method of determination of water-soluble chloride. This method has also been adapted for flow injection analysis.

The determination of chloride by ion chromatograph separates the chloride from other water-soluble anions by liquid chromatography, applying an anion exchange resin as stationary phase, and aqueous solutions of carbonate, hydrogencarbonate, hydroxide as eluent. The detection is carried out using a conductivity detector (CD). This method permits the determination of both chloride and sulfate at the same time. The determination can be used for chloride concentrations down to 0,1 mg/l.

Preparation of steel substrates before application of paints and related products — Test methods for non-metallic blast-cleaning abrasives —

Part 7:
Determination of water-soluble chlorides

1 Scope

This document specifies three methods for the determination of water-soluble chlorides in non-metallic blast-cleaning abrasives, namely, amperometric titration, spectro-photometry and ion chromatography.

This document is part of the ISO 11127 series dealing with the sampling and testing of non-metallic abrasives for blast-cleaning.

The types of non-metallic abrasive and requirements on each are contained in the ISO 11126 series.

The ISO 11126 series and the ISO 11127 series have been drafted as a coherent set of International Standards on non-metallic blast-cleaning abrasives.

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

ISO 11127-1, *Preparation of steel substrates before application of paints and related products — Test methods for non-metallic blast-cleaning abrasives — Part 1: Sampling*

ISO 10304-1, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate*

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
cuvette
absorption cell

3.2
eluent
liquid phase used to achieve separation and transport of analytes

4 Reagent

Use only reagents of recognized analytical grade and only water of at least grade 3 purity in accordance with ISO 3696 (for ion chromatography water of grade 1 purity in accordance with ISO 3696 shall be used).

4.1 Sulfuric acid, concentrated, approximately 96 % (by mass), $\rho \approx 1{,}84$ g/ml.

4.2 Silver nitrate, standard volumetric solution, $c(AgNO_3) = 0{,}01$ mol/l.

4.3 Chloride reagent kit, as supplied by manufacturer comprising of 4.3.1 and 4.3.2.

4.3.1 Mercuric thiocyanate, in methyl alcohol.

4.3.2 Ferric nitrate, in perchloric acid.

4.4 Chloride standard solution, $\rho = 1\ 000$ mg/l.

4.5 Sodium hydrogen carbonate, $NaHCO_3$.

4.6 Sodium carbonate, Na_2CO_3.

4.7 Potassium hydroxide, KOH.

5 Apparatus

Common laboratory apparatus and glassware, together with the following:

5.1 Equipment for amperometric titration, any commercial equipment being suitable.

5.2 Micro-burette

5.3 Balance, capable of weighing to an accuracy of 0,1 g.

5.4 Grade A measuring cylinder, graduated 100:1 ml.

5.5 Micro-pipette, capable of measuring 0,1 ml to 1,0 ml.

5.6 Spectrophotometer, any commercial equipment being suitable.

5.7 Cuvette

5.8 Ion chromatograph system, generally comprising 5.8.1 to 5.8.7.

5.8.1 Eluent reservoir, and a degassing unit.

5.8.2 Metal-free HPLC pump

5.8.3 Sample injection system, incorporating a sample loop of appropriate volume (e.g. 0,02 ml).

5.8.4 Anion column, with specified separating performance.

5.8.5 Conductivity detector

5.8.6 Recording device

5.8.7 Pre-columns

5.9 **Membrane filter**, mesh size 0,45 µm.

6 Sampling

Take a representative sample of the product to be tested, in accordance with ISO 11127-1.

7 Procedure

7.1 Carry out the determination in duplicate.

7.2 Weigh a test portion of (100 ± 0,1) g of the sample (m_0) into a 250 ml flask and add (100 ± 0,5) ml of water. Shake for 5 min and allow to stand for 1 h. Then shake again for 5 min and allow to settle. If the solution does not completely clear, filter it by any suitable method.

8 Analysis by amperometric titration

8.1 Take 25 ml of the solution from 7.2. Add 0,1 ml of sulfuric acid (4.1) using micro-pipette (5.5) and dilute to approximately 75 ml with water.

The pipette shall not be used by mouth.

8.2 Titrate the solution with silver nitrate (4.2) from the micro-burette (5.2), noting the end point as the voltage at which the pointer of the galvanometer reverses direction.

8.3 Calculation

Calculate the water-soluble chloride content $w(\text{Cl}^-)$ of the abrasive, expressed as a percentage by mass, using Formula (1):

$$w\left(\text{Cl}^-\right)=\frac{V\times 0{,}000\,355\times 4}{m_0}\times 100 \quad (1)$$

where

m_0	is the mass, in grams, of the test portion;
V	is the volume, in millilitres, of silver nitrate solution (4.2) used;
4	is the sample fraction factor;
0,000 355	is the factor for the conversion of millilitres of silver nitrate solution, $c(\text{AgNO}_3)$ = 0,01 mol/l, to grams of Cl.

If the duplicate determinations differ by more than 10 % (relative to the higher result), repeat the procedure described in Clause 7.

Calculate the mean of two valid determinations and report the result to the nearest 0,000 1 %.

9 Analysis by spectrophotometry

9.1 Commercially available spectrophotometers may have pre-programmed methods using their reagents to determine chloride concentrations which allow direct reading of the chloride concentration. Users of these instruments should follow the manufacturer's instructions for calibration and determination of chloride. The instructions in 9.2 to 9.14 apply where no pre-programmed methods are available.

9.2 Turn on spectrophotometer and allow to warm up. Select read out in absorbance units. Adjust wavelength to 460 nm.

9.3 Prepare 250 mg/l of chloride solution by pipetting 25 ml of 1 000 mg/l standard solution of chloride (4.4) into an 100 ml volumetric flask and diluting to 100 ml with de-ionised water.

9.4 Pipette 1 ml, 2 ml, 3 ml, 4 ml and 5 ml of the 250 mg/l chloride solution (9.3) into five 50 ml volumetric flasks and fill each to the mark with de-ionised water to prepare calibration standards of 0,000 5 %, 0,001 %, 0,001 5 %, 0,002 0 % and 0,002 5 %, respectively.

9.5 Prepare a blank sample by adding 10 ml of de-ionised water to a 100 ml Erlenmeyer flask and add 2 ml of mercuric thiocyanate reagent (4.3.1). Swirl to mix. Add 1 ml of ferric nitrate (4.3.2), swirl and allow to stand for 3 min.

9.6 Pour the blank sample into a 10 mm cuvette (5.7) and place in a spectrophotometer cell compartment. Close the lid and zero the instrument as directed by the manufacturer.

9.7 Add 10 ml 0,000 5 % calibration standard to the Erlenmeyer flask and add 2 ml of mercuric thiocyanate reagent (4.3.1). Swirl to mix. Add 1 ml of ferric nitrate (4.3.2), swirl to mix and allow colour to develop for 3 min.

9.8 Pour the sample into a clean 10 mm cuvette, place it in the spectrophotometer cell compartment, close the lid and read the absorbance. Record absorbance.

9.9 Repeat 9.6 and 9.7 for each of the calibration standards in turn.

9.10 Plot absorbance against chloride concentration and draw a calibration curve.

9.11 Repeat 9.6 and 9.7 for the solution (7.2).

9.12 Read chloride content of the solution (7.2) from the calibration curve. Record result.

9.13 Repeat 9.10 and 9.11 for the duplicate sample.

9.14 If the duplicate determinations differ by more than 10 % (relative to the higher result), repeat the procedure described in Clause 7.

Calculate the mean of two valid determinations and report the result to the nearest 0,000 1 %.

10 Analysis by ion chromatography

10.1 Take the solution from 7.2 and filter it through a membrane filter with a mesh size of 0,45 µm.

Samples/abrasives with a high concentration of metals (e.g. Fe or Mn) should be treated before the analysis for higher precision. The metals should be separated from the analyte (e.g. by a cation exchange sample preparation cartridge or an ion exchange column).

10.2 The solution shall be analysed in accordance with ISO 10304-1.

10.3 Calculate the water-soluble chloride content $w(\text{Cl}^-)$ of the abrasive, expressed as a percentage by mass, using Formula (2):

$$w\left(\text{Cl}^-\right)=\frac{\beta\left(\text{Cl}^-\right)\times V}{1\,000\times m_0}\times 100 \tag{2}$$

where

$\beta(\text{Cl}^-)$	is the concentration of chloride in the solution in mg/l, which was analysed by ion chromatography;
V	is the volume, in litres, of the solution;
m_0	is the mass, in grams, of the test portion;
1 000	is the factor, in milligrams per grams, for the conversion of chloride concentration to percentage by mass of Cl.

10.4 If the duplicate determinations differ by more than 10 % (relative to the higher result), repeat the procedure described in Clause 7.

Calculate the mean of two valid determinations and report the result to the nearest 0,000 1 %.

11 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested, in accordance with the appropriate part of the ISO 11126 series);

b) a reference to this document (ISO 11127-7:2022);

c) analysis method;

d) the result of the test;

e) any deviation from the test method specified;

f) any unusual features observed

g) the date of the test;

h) the name of the person who carried out the test.