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Surface chemical analysis — Total reflection X-ray fluorescence analysis of water

Analyse chimique des surfaces — Analyse par fluorescence de rayons X en réflexion totale d'eau

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Foreword

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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 document 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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This document was prepared by Technical Committee ISO/TC 201, *Surface Chemical Analysis*, Subcommittee SC 10, *X-ray Reflectometry (XRR) and X-ray Fluorescence (XRF) Analysis*.

This second edition cancels and replaces the first edition (ISO 20289:2018) which has been technically revised.

The main changes are as follows:

- updated Clause 2 and bibliography;
- editorial changes and correction of verbal forms;
- revision of Annex A removing the use of relative uncertainty;

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

Total reflection X-ray fluorescence (TXRF) spectroscopy is a surface sensitive technique which can be used to obtain compositional information about different kinds of samples. ISO/TS 18507 provides guidelines for the characterization of biological and environmental samples with TXRF.

TXRF is suitable for quantitative elemental analysis of liquid samples deposited as thin films on clean and well-polished reflectors, by means of internal standard calibration[1][2].

This document provides guidance and requirements for the quantitative elemental analysis of water by means of TXRF instrumentation.

Surface chemical analysis — Total reflection X-ray fluorescence analysis of water

1 Scope

This document specifies a chemical method for technicians working with total reflection X-ray fluorescence (TXRF) instrumentation to perform measurements of water samples, according to good practices, with a defined degree of accuracy and precision. Target users are identified among laboratories performing routine analysis of large numbers of samples, which also conform with ISO/IEC 17025.

This document specifies a method to determine the content of elements dissolved in water (e.g. drinking water, surface water and ground water). This document is also applicable for determining elements in waste waters and eluates, taking into account the specific and additionally occurring interferences. This document does not specify sampling, dilution and pre-concentration methods.

Elements determined using the method specified in this document can depend on the X-ray source of the instrument. This document does not specify health, safety or commercial aspects.

The determinable concentrations depend on the matrix and the interferences encountered. In drinking water and relatively unpolluted waters, the limit of quantification lies between 0,001 mg/l and 0,01 mg/l for most of the elements. The range of concentrations typically lies between 0,001 mg/l and 10 mg/l, depending on the element and predefined requirements.

Annex A reports an example of uncertainty calculation. Annex B provides an example report on validation of the method for TXRF analysis of water performed with instrumentation that has Mo as the X-ray source and uses Ga as the internal calibration standard.

Quantification limits of most elements are affected by blank contamination and depend predominantly on the laboratory air-handling facilities available, on the purity of reagents and the cleanliness of labware.

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/TS 18507:2015, *Surface chemical analysis — Use of Total Reflection X-ray Fluorescence spectroscopy in biological and environmental analysis*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 18507 and the following 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
calibration standard
known standard solutions, prepared from the primary dilution solution or stock solutions, containing the elements of interest or the internal standard element

3.2
linear calibration range
concentration range over which the instrument response is linear

3.3
sensitivity
slope of the linear fitting of the analytical curves, functional relationship between fluorescence intensity and concentration

3.4
limit of detection
minimum element concentration that can be identified with 99 % confidence that the element concentration is different from the blank

Note 1 to entry: See 10.3.

3.5
sample
water solution to be analysed

3.6
specimen
solution containing internal standard prepared for TXRF analysis

3.7
replicate
sample carrier with deposited residue

3.8
internal standard content
known amount of an element used to normalize the variation in the fluorescence X-ray intensities of the other elements in a residue

3.9
quality control sample
reference material obtained from an outside source, with known element concentrations used to check accuracy and precision

Note 1 to entry: See Clause 12.

3.10
limit of quantification
smallest concentration of an element that can be reliably quantified

Note 1 to entry: See 11.2.

4 Symbols and abbreviated terms

C	concentration (or mass per volume density) in mg/l
LOD	limit of detection
LOQ	limit of quantification
RSD	relative standard deviation
S	sensitivity
TXRF	total reflection X-ray fluorescence

5 Safety

This test method uses X-ray radiation. Consequently, it is important to avoid exposing any part of the body to the X-rays produced by the apparatus. A risk assessment and materials safety data sheets (MSDS or SDS) should be readily available.

6 Interferences

The electrical conductivity of the water sample to be analysed should be lower than 2 mS/cm. The presence of particles and suspended solids can affect accuracy and precision of TXRF analysis due to self-absorption effects. Sea water or matrix rich wastewaters may be diluted or filtered to reduce possible interferences (see 9.2) and background contributions (see 10.3). All the reagents shall be analysed to check the possible presence of impurities. Interferences can be caused by contaminant elements due to uncleaned glassware (see 7.1), sample preparation environment (see 9.1) or reflectors (see 9.4). Pile-up or sum peaks can arise if some elements are present in high concentration. Peaks overlap should be considered.

7 Apparatus

7.1 General

For reagents and specimen preparation, use ordinary laboratory glassware or plasticware unless otherwise stated. The apparatus for preparation of the samples shall be calibrated and cleaned.

Check periodically the accuracy of micropipettes and volumetric flasks used in the volumetric method, by taking an exact volume of water with the micropipette and weighing it on a high-precision calibrated balance. The corresponding relative standard deviation (RSD) of all the micropipettes and glassware used shall be indicated and considered for the calculation of the overall method RSD.

7.2 Labware

The use of uncoloured material is preferable.

7.2.1 One-mark volumetric flasks, 10,00 ml ± 0,02 ml, made of glass, PE, PFA or PP.

7.2.2 Plastic micropipettes, from 1 µl to 10 µl and from 100 µl to 1 000 µl (minimum accuracy 2 %), made of PE, PFA or PP.

7.2.3 Plastic beakers, 100 ml and 1 000 ml, made of PE, PFA, PP or PTFE.

7.2.4 Plastic test tubes, 1,5 ml, 50 ml and 100 ml, made of PE, PFA, PP or PTFE.

7.2.5 Syringe with 0,2 µm pore size filters.

7.2.6 Carrier-stand made of PFA or PTFE.

7.3 Drying apparatus

7.3.1 Use one of the following.

a) Infrared lamp.

b) Glass ceramic heating plate with a power controller.

c) Vacuum vessel fitted with gas flow equipment.

7.3.2 Drying temperature should be lower than 60 °C to avoid the loss of volatile elements and compounds. Due to heat treatment, some elements can evaporate or diffuse into the X-ray reflector. Therefore, the recovery rate for elements has to be determined especially for fast diffusers, for example Cu or Ni, or for most volatile elements and/or compounds, such as As, Cl, Hg or SiF.

8 Reagents, standards and materials

Use only reagents of recognized analytical grade and only bi-distilled water or water of equivalent purity. The level of contamination of blanks shall be periodically checked and taken into account for quantification.

8.1 Ultra-pure water, for example water with conductivity less than 0,05 µS/cm, commercially available or obtained by purification systems.

8.2 Ultra-pure nitric acid, for example nitric acid (CAS 7697-37-2) solution with concentration from 65 % to 70 % wt.

8.3 Cleaning solution, for example liquid detergent, concentrated, alkaline and foaming, suitable for manual cleaning, soaking, ultrasonic baths and brushes systems (CAS 1310-73-2).

8.4 Silicone solution in isopropanol, for example silicone solution (CAS 67-63-0) in isopropanol if the level of contamination is acceptable.

8.5 Internal standard solution, for example 1 000 mg/l standard solution in 0,5 mol/l nitric acid (CAS 69365-72-6) specified for AAS or ICP.

8.6 Multi-element standard solution, for example 1 000 mg/l standard solution in nitric acid specified for AAS or ICP containing elements of interest.

8.7 Diluted internal standard solutions

The internal standard content in the specimen should be comparable to the amount of element to be quantified. Diluted internal standard solutions may be necessary to add a defined sample amount to prepare the specimen. Prepare fresh diluted internal standard solutions, either by volume or, preferably, by weight starting from internal standard solutions (8.5). Select the right dilution steps required to obtain the final defined concentration of internal standard element in the specimen. Take into account the accuracy of all the steps involved. Check the contamination level of the prepared solutions and take it into account for quantification.

EXAMPLE Diluted gallium internal standard solution 100 mg/l gallium solution

Prepare diluted gallium standard solution using either method a) or method b) below:

a) Transfer 1 000 µl of gallium standard solution (8.5) to a plastic or glass 10 ml one-mark volumetric flask (7.2.1) with a plastic micropipette (7.2.2), add under 9 ml of ultra-pure water (8.1), finally make up to the mark with ultra-pure water (8.1) and mix.

b) Weigh a 10 ml plastic beaker (7.2.3) to the nearest 0,001 g. Transfer 1 000 µl of gallium standard solution (8.5) to the beaker with a plastic micropipette (7.2.2), add under 9 ml of ultra-pure water (8.1), finally add ultra-pure water (8.1) to make the mass up to 10 g (plus the mass of the beaker) to the nearest 0,01 g and mix.

8.8 Validity of reference material

The validity of the reference material (for example the expiration date) should also be checked.

The use of calibrated balances is recommended. Precision should be indicated and considered for the overall method uncertainty estimation (for example 10,0 mg ± 0,5 mg).