



DPC: 25/30474488 DC

BSI Group Headquarters

389 Chiswick High Road London W4 4AL

Tel: +44 (0)20 8996 9000

Fax: +44 (0)20 8996 7400

www.bsigroup.com

Date: 17 February 2025

Origin: International

Latest date for receipt of comments: 12 April 2025

Project No. 2023/01089

Responsible committee: CII/60 Surface chemical analysis

Interested committees:

Title: Draft BS ISO 16666 Surface chemical analysis - Total reflection X-ray fluorescence - Principles and general requirements.

Please notify admin.start@bsigroup.com if you are aware of any keywords that might assist in clarifying or identifying the standard or of the content of this standard:

- i) has any issues related to 3rd party IPR, patent or copyright
- ii) affects other national standard(s)
- iii) requires additional national guidance or information

WARNING: THIS IS A DRAFT AND MUST NOT BE REGARDED OR USED AS A BRITISH STANDARD. THIS DRAFT IS NOT CURRENT BEYOND 12 April 2025

This draft is issued to allow comments from interested parties; all comments will be given consideration prior to publication. No acknowledgement will normally be sent. **See overleaf for information on the submission of comments.**

No copying is allowed, in any form, without prior written permission from BSI except as permitted under the Copyright, Designs and Patent Act 1988 or for circulation within a nominating organization for briefing purposes. Electronic circulation is limited to dissemination by e-mail within such an organization by committee members.

Further copies of this draft may be purchased from BSI Shop <http://shop.bsigroup.com> or from BSI Customer Services, Tel: +44(0) 20 8996 9001 or email cservices@bsigroup.com. British, International and foreign standards are also available from BSI Customer Services.

Information on the co-operating organizations represented on the committees referenced above may be obtained from <https://standardsdevelopment.bsigroup.com/>.

E-mail: admin.start@bsigroup.com

Introduction

This draft standard is based on national and international discussions. Your comments on this draft are invited and will assist in the preparation of the consequent standard.

For international standards, comments will be reviewed by the relevant UK national committee before sending the consensus UK vote and comments to the international committee, which will then decide appropriate action. If the international standard is approved, it is usual for the text to be published as a British Standard.

For national standards, comments will be reviewed by the relevant UK national committee and the resulting standards published as a British Standard.

UK Vote

Please indicate whether you consider the UK should submit a negative (with supporting technical reasons) or positive vote on this draft. Please indicate if you are aware of any reason why this draft standard should not be published as a British Standard.

Submission of Comments

- **Annotated drafts are not acceptable and will be rejected.**
- All comments should be submitted online at <https://standardsdevelopment.bsigroup.com/>. You will need to register in order to comment.

Template for comments and secretariat observations

Date: xx/xx/20xx	Document: ISO/DIS xxxx
------------------	------------------------

1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex (e.g. 3.1)	Paragraph/ Figure/Table/Note	Type of comment	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	



DRAFT International Standard

ISO/DIS 16666

Surface chemical analysis — Total reflection X-ray fluorescence — Principles and general requirements

ICS: 71.040.40

ISO/TC 201/SC 10

Secretariat: JISC

Voting begins on:
2025-02-17

Voting terminates on:
2025-05-12

This document is circulated as received from the committee secretariat.

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENTS AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

Reference number
ISO/DIS 16666:2025(en)

Full document details: 25/30474488 DC

© ISO 2025  **Documents**
Document Sharing Hub



COPYRIGHT PROTECTED DOCUMENT

© ISO 2025

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms, definitions and abbreviated terms	1
3.1 Abbreviated terms	2
4 Physical background of TXRF	2
4.1 General	2
4.2 Definition of angle and X-ray path in TXRF geometry	4
4.3 Beam projection	5
4.4 X-ray standing waves (XSW)	5
4.5 Fluorescence intensity	7
5 Instrumental requirements	8
5.1 Description of various possible TXRF measurement setups	8
5.2 Beam conditioning	9
5.2.1 Requirement	9
5.2.2 Beam guidance	9
5.2.3 Beam modulation (tuning)	9
5.2.4 Measurement angles in TXRF	10
5.3 Reflector (sample carrier)	10
5.3.1 General requirements	10
5.3.2 Reflectors for chemical analysis	10
5.4 Reflector alignment	10
5.5 Detector	11
5.6 Sample station	11
5.7 Control unit	11
6 Calibration and quality control	11
6.1 Angle inspection	11
6.2 Energy calibration	12
6.3 Calibration of the element sensitivities	12
6.4 Inspection of the sample excitation	13
6.5 Inspection of the element detection	14
7 Spectra analysis and Reporting	14
7.1 Spectra Processing	14
7.2 Reporting Spectra	14
8 Samples	15
9 Qualitative and quantitative analysis	15
9.1 Qualitative analysis	15
9.2 Quantitative analysis	15
9.2.1 Methods of quantitative analysis	15
9.3 Preparation of a measurement for quantitative analysis	17
9.4 Procedure of a quantitative analysis by the element-specific sensitivity method (internal standard addition)	17
9.5 Verification of quantitative TXRF measurements	18
9.6 Traceability in TXRF	18
Annex A (informative) Measurement angles in TXRF, background correction, peak correction, spectrum deconvolution	20
Annex B (informative) Round-robin test for verification of the precision and accuracy of TXRF spectroscopy	23
Bibliography	24

Foreword

This document has been prepared by Sub-Committee SC10 “X-ray Reflectometry (XRR) and X-ray Fluorescence (XRF) Analysis” of the ISO TC201 “Surface chemical analysis”.

For current information on this document, please go to the ISO website (www.iso.org) and search for the document number in question.

Introduction

Through general technical developments total reflection X-ray fluorescence analysis (TXRF) could be developed into a robust independent physical method of instrumental analytical chemistry. TXRF is a distinct multi-element micro-method and a method of surface analysis. It is used for the simultaneous qualitative detection of the elements and quantification of the element masses. It should be noted that only small quantities of material are used for the analysis to benefit from all the advantages inherent to the procedure and thus to achieve correct analysis values. TXRF is used where also other atomic spectroscopic methods, such as atomic absorption spectrometry (AAS) or inductively coupled plasma-based techniques (ICP-AES and ICP-MS) are established in the field of element analysis. In addition, TXRF is used in surface analysis and complements techniques such as Auger electron spectroscopy (AES), Rutherford backscattering spectrometry (RBS) or secondary ion mass spectrometry (SIMS). The rapid and in part non-destructive measurement process of a TXRF configuration allows screening or qualitative analysis of unknown flat, film-like and particulate samples and is therefore a valuable addition to other analytical techniques.

The main difference between TXRF and the conventional X-ray fluorescence spectrometry (XRF) is the efficient way to excite the material to be examined to fluoresce which is achieved by a special geometrical arrangement in the spectrometer. The spectral background is drastically reduced, the fluorescence intensity increased resulting in a significant enhancement of the signal to noise ratio. Special sample treatment and sample presentation are required for this purpose. If these boundary conditions are met, TXRF achieves a high degree of accuracy and repeatability of the analyses, although a simplified calibration and quantification is used. The detection limits currently achievable with TXRF are approximately 10^{-12} g or 10^{10} atoms/cm² and are thus orders of magnitude lower than for XRF. The relative element concentrations are, depending on the matrix or the main components of the sample and for the elements to be examined, between 0,1 % and up to 10^{-9} for the low concentrations.

The present standard describes the physical principles and instrumentation required for TXRF analysis assuming that the conditions for total reflection are met, without referring to the samples' specificity. As a demonstration of the good reproducibility of the TXRF instruments, data from a round-robin test performed using centrally prepared and optimized samples are presented (see [Annex B](#)).

Other standards focussing on specific case studies and procedures are available for silicon wafers (ISO 14706 and 17331), environmental and biological samples (ISO TS 18507), and water (ISO 20289).

Surface chemical analysis — Total reflection X-ray fluorescence — Principles and general requirements

1 Scope

The document provides physical principles, description of instrumental requirements of TXRF spectrometers and general guidelines for calibration, method development and verification of TXRF measurements and quality control.

The document describes measurements at TXRF conditions, which are met in case of a fixed glancing angle below the critical angle of total reflection and considerably enhanced excitation radiation intensity. Although certain definitions of grazing incidence geometry are shown for clarification, this document excludes measurement setups working under such conditions.

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.

DIN 51003:2022, *Total reflection X-ray fluorescence – Principles and definitions*

ISO 18115-1, *Surface chemical analysis — Vocabulary — Part 1: General terms and terms used in spectroscopy*

ISO 20289:2018, *Surface chemical analysis — Total reflection X-ray fluorescence analysis of water*

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

ISO 14706:2014, *Surface chemical analysis — Determination of surface elemental contamination on silicon wafers by total-reflection X-ray fluorescence (TXRF) spectroscopy*

ISO 17331:2004, *Surface chemical analysis — Chemical methods for the collection of elements from the surface of silicon-wafer working reference materials and their determination by total-reflection X-ray fluorescence (TXRF) spectroscopy*

3 Terms, definitions and abbreviated terms

For the purposes of this document, the terms and definitions given in ISO 18115-1.

ISO maintains 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 Abbreviated terms

AAS	Atomic Absorption Spectroscopy
FWHM	Full Width at Half Maximum
GIXRF	Grating Incidence X-ray Fluorescence
ICP-MS	Inductively Coupled Plasma-Mass Spectroscopy
ICP-AES	Inductively Coupled Plasma –Atomic Emission Spectroscopy
SDD	Silicon Drift Detector
XRF	X-ray fluorescence
XRR	X-ray reflectometry
XSW	X-ray Standing Wave
TXRF	Total Reflection X-ray Fluorescence

4 Physical background of TXRF

4.1 General

TXRF is based on the excitation (and detection) of element-specific X-ray fluorescence radiation at a very small glancing angle (smaller than the critical angle of the total reflection of the sample carrier, see [Figure 3](#) and [4.2](#)) of the incident radiation^[1].

Total reflection has two beneficial effects on the measurement:

- 1) Since the primary beam penetrates only a few nanometres into the sample carrier, the proportion of radiation generated and detected by scattering and fluorescence excitation of the carrier material is significantly lower than, e.g. in classic XRF, which enables a lower background signal and thus better detection limits ([Figure 1](#))