



BSI Standards Publication

**Iron ores — Wavelength  
dispersive X-ray fluorescence  
spectrometers — Determination  
of precision**

**National foreword**

This Published Document is the UK implementation of ISO/TR 18231:2016.

The UK participation in its preparation was entrusted to Technical Committee ISE/58, Iron ores.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2016.

Published by BSI Standards Limited 2016

ISBN 978 0 580 80818 0

ICS 73.060.10

**Compliance with a British Standard cannot confer immunity from legal obligations.**

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 31 May 2016.

**Amendments/corrigenda issued since publication**

Date	Text affected
------	---------------

---

TECHNICAL  
REPORT

**ISO/TR**  
**18231**

First edition  
2016-05-01

---

---

**Iron ores — Wavelength dispersive  
X-ray fluorescence spectrometers —  
Determination of precision**

*Minerais de fer — Spectromètres à fluorescence à rayons X à longueur  
d'onde dispersive — Détermination de la précision*



Reference number  
ISO/TR 18231:2016(E)



**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2016. Published in Switzerland

All rights reserved. Unless otherwise specified, 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  
Ch. de Blandonnet 8 • CP 401  
CH-1214 Vernier, Geneva, Switzerland  
Tel. +41 22 749 01 11  
Fax +41 22 749 09 47  
copyright@iso.org  
www.iso.org

# Contents

Page

<b>Foreword</b> .....	<b>iv</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Frequency of testing</b> .....	<b>1</b>
<b>3 Counter tests</b> .....	<b>2</b>
3.1 Counter resolution.....	2
3.1.1 General.....	2
3.1.2 Procedure.....	4
3.1.3 Assessment of results.....	6
3.2 Conductivity of the gas flow proportional counter window.....	6
3.2.1 General.....	6
3.2.2 Procedure.....	7
3.2.3 Assessment of results.....	7
3.3 Pulse shift corrector.....	7
3.3.1 General.....	7
3.3.2 Procedure.....	8
<b>4 Spectrometer tests</b> .....	<b>8</b>
4.1 General.....	8
4.2 Precision.....	9
4.2.1 General.....	9
4.2.2 Calculation of counting statistical error.....	10
4.3 Test specimen.....	11
4.3.1 General.....	11
4.3.2 Sequential spectrometers.....	11
4.3.3 Simultaneous spectrometers.....	11
4.4 Instrumental conditions.....	11
4.4.1 General.....	11
4.4.2 Sequential spectrometers.....	12
4.4.3 Simultaneous spectrometers.....	12
4.5 Stability test.....	12
4.6 Specimen rotation test.....	13
4.7 Carousel reproducibility test.....	13
4.8 Mounting and labelling reproducibility test.....	13
4.9 Comparison of sample holders.....	13
4.10 Comparison of carousel positions.....	14
4.11 Angular reproducibility.....	14
4.12 Collimator reproducibility (for sequential spectrometers fitted with an interchangeable collimator).....	14
4.13 Detector changing reproducibility (for sequential spectrometers fitted with more than one detector).....	14
4.14 Crystal changing reproducibility.....	14
4.15 Other tests.....	15
4.16 Note on glass bead curvature.....	15
<b>Determination of the dead time and the maximum usable count rate of the equipment</b> .....	<b>15</b>
5.1 General.....	15
5.2 Methods of determination of dead time.....	16
5.2.1 General.....	16
5.2.2 Recommended method for determining dead time.....	17
<b>Annex A (informative) Calculation of the coefficient of variation of duplicates</b> .....	<b>24</b>
<b>Bibliography</b> .....	<b>26</b>

## 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](http://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](http://www.iso.org/patents)).

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

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 442, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

## Introduction

If an X-ray fluorescence spectrometer is to be used for precise analyses, it needs to be functioning correctly to specification, that is, the errors associated with the various functions of the instrument have to be very small. It is important therefore that the spectrometer be tested to ensure that it is indeed functioning to deliver the required precision. The objective of this Technical Report is to set out tests that can be used to ascertain the extent of the errors and to suggest procedures for their rectification. These tests are not used to ascertain whether the instrument is operating optimally but to determine whether the instrument is capable of giving a preselected precision.

Currently in preview, click buy full version.

# Iron ores — Wavelength dispersive X-ray fluorescence spectrometers — Determination of precision

## 1 Scope

This Technical Report describes methods of test that can be applied to wavelength dispersive X-ray fluorescence (WD-XRF) spectrometers to ensure that the spectrometers are functioning in a manner that allows precise analyses to be made.

The tests outlined are designed to measure the errors associated with the operation of certain parts of the spectrometer. They are not designed to check every part of the spectrometer but only those parts that may be the common sources of error.

It is assumed that the performance of the instrument has been optimized according to the manufacturer's instructions. For all tests, the two-theta angle should be carefully set for the line being measured. The pulse height window should be set according to the manufacturer's instructions and should have a broad setting which may also include the escape peak for gas proportional counters. The instrument and detector gas environment should be as specified by the manufacturer, as should the power supply to the instrument.

NOTE Where no distinction has been made, it is assumed that a test is applicable to both sequential and simultaneous spectrometers.

## 2 Frequency of testing

Testing is not required to be carried out with each batch of analyses. The frequency of testing varies depending on the test involved. [Table 1](#) lists the suggested frequency with which each test should be carried out. Where specific problems are encountered, more frequent testing may be required and remediation work performed.