TECHNICAL REPORT

ISO/TR 18231

First edition 2016-05-01

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

erais nde disp. Minerais de fer — Spectromètres à fluorescence à rayons X à longueur





© ISO 2016, Published in Switzerland

aroduced or utilized c

Te internet or an !

or ISO's memb 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
Fore	eword		iv
Intr	oductio	n	v
1	Scop	е	1
2	<.O.	uency of testing	
3	_	ter tests	
3	3.1	Counter resolution	
	3.1	3.1.1 General	
		3.1.2 Procedure	
		3.1.3 Assessment of results	
	3.2	Conductivity of the gas flow proportional counter window	
	0.2	3.2.1 General	
		3.2.2 Procedure	
		3.2.3 Assessment of results	
	3.3	Pulse shift corrector	
		3.3.1 General	
		3.3.2 Procedure	
4	_	t rometer tests General	8
	4.1		
	4.2	Precision	
		4.2.1 General 4.2.2 Calculation of counting statistical error	
	4.3	4.2.2 Calculation of counting statistical error Test specimen	10 11
	4.3		
		4.3.1 General 4.3.2 Sequential spectrometers	
	4.4	4.3.3 Simultaneous spectrometers Instrumental conditions	
	4.4	4.4.1 General	
		4.4.2 Sequential spectrometers	
		4.4.3 Simultaneous spectrometers	
	4.5	Stability test	
	4.6	Specimen rotation test	
	4.7	Carousel reproducibility test	13
	4.8	Mounting and loading 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	1
	1.12	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	
	4.16	Note on glass bead curvature	
5	Doto		
	5.1	rmination of the dead time and the maximum usable count rate of the equipmen General	
	5.1	Methods of determination of dead time	
	٥.۷	5.2.1 General	
		5.2.2 Recommended method for determining dead time	
		<u> </u>	
Ann	ex A (inf	formative) Calculation of the coefficient of variation of duplicates	24
Bibl	iograph	y	26

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).

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 102, 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 See tests aether the 1. 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.

This document is a previous general ded by tills

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.