
**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*

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Foreword

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The committee responsible for this document is ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

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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.

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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.

Table 1 — Suggested frequency of precision tests

Frequency	Test
Monthly	Resolution of the gas-flow proportional counter Resolution of the scintillation and sealed gas counters Operation of the pulse height shift corrector ^a
Half yearly	Conductivity of gas-flow proportional counter window General stability Collimator reproducibility Detector changing reproducibility Crystal changing reproducibility Angular reproducibility
Yearly	Carousel reproducibility Comparison of carousel positions Comparison of sample holders Sample loading and unloading
^a The position of the pulse height peak should also be checked after changing a bottle of detector gas since a variation in the methane content of the gas will change the position of the peak.	

The frequencies with which the tests listed in [Table 1](#) are carried out are suggested on the basis that there have been no changes to the spectrometer. If mechanical or electronic maintenance of a major nature is carried out, the appropriate tests should be made before the spectrometer is taken back into routine service.

3 Counter tests

3.1 Counter resolution

3.1.1 General

3.1.1.1 Theoretical resolution

Impurities in the flow gas and contamination of the anode wire may cause gas flow proportional counters to gradually deteriorate, which will result in both a shift and a broadening of the energy distribution (pulse height) curve. Similarly, scintillation counters and sealed gas counters may, for various reasons, exhibit the same gradual deterioration. This can, ultimately, adversely affect the measurements. Impurities in detector gas can be minimized by the use of gas filters.

The resolution (*RES*) of a counter is related to its energy distribution curve, and is given by the measured peak width at half height (*W*) expressed as a percentage of the maximum of the pulse amplitude distribution (*V*), using Formula (1) where the values of *W* and *V* are in terms of arbitrary units (which vary between instrument manufacturers) obtained from the X-axis (see [Figure 1](#)):

$$RES = \frac{W}{V} \times 100 \quad (1)$$

The theoretical resolution (*RES_{th}*), using the full width at half height of a Gaussian distribution, can be calculated using the following formulae:

$$RES_{th} = 2,36 \sigma \quad (2)$$

$$\sigma = \sqrt{n} \quad (3)$$

Expressed as a percentage relative to n , Formula (3) becomes:

$$\sigma = \frac{100}{\sqrt{n}} (\text{in } \%) \quad (4)$$

where

n is the number of primary electrons per incident photon (gas counters) or number of photoelectrons collected by the first dynode of the photomultiplier tube (scintillation counters), calculated using Formula (5):

$$n = \frac{E_x}{V_i} \quad (5)$$

E_x is the energy of the incident radiation, in kilo electron volts (keV);

V_i is the effective ionization potential of Argon for a flow counter, in kilo electron volts (keV) = 0,026 4.

Substituting Formula (5) into Formula (4), and Formula (4) into Formula (2) gives:

$$RES_{th} = \frac{236 \times \sqrt{0,026\ 4}}{\sqrt{E_x}} = \frac{38,4}{\sqrt{E_x}} \quad (6)$$

Hence, for Cu K α ($E = 8,04$ keV), the theoretical resolution of an Ar gas counter is 13,5 %.

3.1.1.2 Scintillation counter

For a scintillation counter:

$$RES_{th} = \frac{128}{\sqrt{E_x}} \quad (7)$$

and for Cu K α , the resolution should be approximately 45 %.^[1]

3.1.1.3 Practical resolution

In practice, however, the measured resolution achieved (RES_m) is given in Formula (8):

$$RES_m = k R \quad (8)$$

where

k is a factor that varies with the design of the counter, phosphor efficiency (scintillation counters), diameter, cleanliness and composition of the anode wire (gas counters).

For a well-designed and clean gas-flow proportional counter, k should be less than 1,15. Hence, for such a counter, RES_m should be less than 15,6 % for Cu K α radiation. For the scintillation counter, this value should be less than 52 %.