

Australian Standard[®]

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

This Australian Standard was prepared by Committee MN/2, Iron Ores. It was approved on behalf of the Council of Standards Australia on 19 December 1995 and published on 4 April 1996.

The following interests are represented on Committee MN/2:

Australasian Institute of Mining and Metallurgy
Australian Mining Industry Council
Bureau of Steel Manufacturers of Australia
CSIRO - Institute of Minerals, Energy and Conservation
Chamber of Mines of W.A.
Department of Primary Industries and Energy
Pilbara Iron Ore Producers Association
The Royal Australian Chemical Institute

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RECONFIRMATION

OF

AS 2563—1996

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

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NOTES

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PREFACE

This Standard was prepared by the Standards Australia Committee on Iron Ores, as a revision of AS 2563—1982, *Wavelength dispersive X-ray fluorescence spectrometers—Methods of test for determination of precision*, which was prepared by the Committee on Aluminium Ores.

If an X-ray fluorescence spectrometer is to be used for precise analyses it needs to be functioning properly, i.e. 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 correctly. The objective of this Standard 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 used not to ascertain whether the instrument is operating optimally, but whether the instrument is capable of giving a preselected precision.

The Standard now contains a discussion of the problem that scintillation counters may deteriorate and that spectrometers may show instability after changes to kV and mA settings. In addition, the Standard has been modified to cover the higher counting rates that modern spectrometers can handle, including automatic pulse-height correction.

The term ‘informative’ has been used in this Standard to define the application of the Appendix to which it applies. An informative appendix is for information and guidance only.

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STANDARDS AUSTRALIA

Australian Standard

**Wavelength dispersive X-ray fluorescence spectrometers—
Determination of precision**
**SECTION 1 SCOPE AND FREQUENCY OF
TESTING**

1.1 SCOPE This Standard sets out methods of test that can be applied to wavelength dispersive X-ray fluorescence spectrometers to ensure that the spectrometers are functioning in a manner that allows precise analyses to be carried out.

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 are known to be common sources of unacceptable error.

It is assumed that the performance of the instrument has been optimized according to the manufacturer's instructions.

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

1.2 FREQUENCY OF TESTING Testing is not required to be carried out with each batch of analyses. The frequency of testing should vary depending on the test involved. Table 1.1 lists the suggested frequency with which each test should be carried out. Where specific problems are encountered, more frequent testing may be required.

**TABLE 1.1
SUGGESTED FREQUENCY OF PRECISION TESTS**

Frequency	Test
Half-yearly	Conductivity of gas-flow proportional counter window Resolution of gas-flow proportional counter Angular reproducibility Collimator reproducibility Detector changing reproducibility Crystal changing reproducibility General stability Resolution of scintillation and sealed gas counters
Yearly	Carousel reproducibility Comparison of carousel positions Comparison of sample holders

The frequencies with which the tests listed in Table 1.1 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 shall be made when the spectrometer is re-commissioned.

SECTION 2 COUNTER TESTS

2.1 COUNTER RESOLUTION

2.1.1 General Impurities in the flow gas may cause gas-flow proportional counters to become gradually contaminated, resulting in both a shift and a broadening of the energy distribution (pulse height) curve. Similarly, sealed gas counters and scintillation counters may, for various reasons, exhibit the same gradual deterioration. This can, ultimately, adversely affect the measurements. Impurities in detected gas can be minimized by the use of gas filters.

The resolution (R) of a counter is related to its energy distribution curve, and is given by the peak width at half height (W) expressed as a percentage of the maximum of the pulse amplitude distribution (V), using the following equation:

$$R(\%) = \frac{W}{V} \times 100 \quad \dots (1)$$

The theoretical resolution is given by—

$$R = Fn^{1/2} \quad \dots (2)$$

where

F = the Fano factor

n = 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)

Using the theoretical relationship, it is possible to re-express the resolution in terms of a constant and the energy of the incident radiation. For a gas counter, the expression becomes—

$$R(\%) = \frac{38.3}{E^{1/2}} \quad \dots (3)$$

where

E = the energy of the incident radiation, in kiloelectron-volts

Hence, for Cu $K\alpha$ ($E = 8.04$ keV), the theoretical resolution of a gas counter is 13.5%.

For a scintillation counter—

$$R(\%) = \frac{120}{E^{1/2}} \quad \dots (4)$$

and for Cu $K\alpha$ the resolution should be approximately 40%.

In practice, however, the actual resolution achieved (R_{ac}) is found to be of the form—

$$R_{ac} = kR \quad \dots (5)$$

where

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