TECHNICAL
SPECIFICATION

**ISO/TS
9516-4**

First edition
2021-11

Iron ores — Determination of various elements by X-ray fluorescence spectrometry —

Part 4:
Performance-based method using fusion preparation method

Reference number
ISO/TS 9516-4:2021(E)

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

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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2 *Chemical analysis*.

A list of all parts in the ISO 9516 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

X-ray fluorescence (XRF) spectrometry is a multi-elemental non-destructive analytical methodology used for quantitative and qualitative determinations. It is element specific covering an elemental range from boron (B) to uranium (U).

Once the sample has been dissolved into a borate glass it may be introduced to the spectrometer for analysis. The sample is then irradiated by intense radiation from an X-ray tube. Analysis of fused glass beads offers advantages over pressed powder techniques as it eliminates particle size effects, and it produces a homogeneous specimen for each element.

In some instances, the relationship between intensity (or intensity ratios) and concentration can be linear. For most analytes there is no direct straightforward relationship between intensity and concentration. With samples of differing compositions, the X-rays are absorbed differently in the different samples giving rise to what are generally referred to as matrix effects. These inter-element effects can be corrected using mathematical models derived from the known physics of X-rays.

Calibration can be based on binary standards (prepared from pure oxides or liquid solutions), reference materials, secondary standards, or combinations therewith.

Iron ores — Determination of various elements by X-ray fluorescence spectrometry —

Part 4:

Performance-based method using fusion preparation method

WARNING — This document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document specifies a performance-based method for the chemical analysis of natural and processed iron ores by fused bead wavelength and energy dispersive X-ray fluorescence (XRF).

It is applicable to all elements of interest when adequate calibrations have been established.

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.

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods*

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

ISO 7764, *Iron ores — Preparation of predried test samples for chemical analysis*

ISO Guide 31, *Reference materials — Contents of certificates, labels and accompanying documentation*

ISO Guide 35, *Reference materials — Guidance for characterization and assessment of homogeneity and stability*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain 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 http://www.electropedia.org/

3.1
primary standard
standard that is designated or widely acknowledged as having the highest metrological qualities and whose value is accepted without reference to other standards of the same quantity

Note 1 to entry: The concept of a primary standard is equally valid for base quantities and derived quantities.

Note 2 to entry: A primary standard is never used directly for measurement other than for comparison with other primary standards or reference standards.

3.2
reference material
RM

standard, generally having the highest metrological quality available at a given location or in a given organization, from which measurements made there are derived

3.3
certified reference material
CRM

material, e.g. iron ores, supplied by an organization conforming to the requirements for the competence of *reference material* (3.2) producers

Note 1 to entry: The requirements for the competence of reference material producers are given in ISO 17034.

Note 2 to entry: CRMs shall be supplied with a certificate of analysis giving information on the average value and standard deviation (with and between laboratory precision statistics) and measurement of uncertainty in accordance with ISO Guide 31.

3.4
accepted reference value
ARV

value that serves as an agreed upon reference for comparison, and which is derived as:

a) a theoretical or established value, based on scientific principles;

b) an assigned or certified value, based on experimental work of some national or international organization;

c) a consensus or certified value, based on collaborative experimental work under the auspices of a scientific or engineering group.

Note 1 to entry: When none of the above are available, the ARV is the expectation of the (measurable) quantity, i.e. the mean of a specified population of measurements.

3.5
referee method

<analytes of commercial significance> method that is independent of other methods (i.e. calibrated with primary standards or able to arrive at the final results using direct measurements or calculations from known physical/chemical laws) and is in the initial stages of the traceability chain

Note 1 to entry: Primary methods include gravimetry, titrimetry, coulometry and isotope dilution mass spectrometry.

4 Reagents and materials

4.1 Pure reagents

Reagents shall be of analytical quality and, wherever possible, be pure oxides or carbonates, except for the calibration of such elements as sulfur, chlorine, bromine, or phosphorus, which do not form stable oxides or carbonates, where some guarantee of stoichiometry is required.

Reagents shall be free of (or corrected for) the presence of moisture (and, in the case of oxides, carbon dioxide) when weighed out for fusion.

Reagents shall be used in a known stoichiometry in terms of content. In order to achieve this, they can be treated before use. Generally, the oxides of iron, silicon, manganese, aluminium, titanium and magnesium shall be heated to 1 000 °C. More information can be found in ISO 9516-1. The procedures specified in ISO 9516-1 ensure that the correct oxidation state is obtained.

All laboratory reagents used for referee methods shall be traceable.

Where reagents have been ignited, they should be covered during cooling in the desiccator and weighed as soon as possible.

4.2 Flux

High purity lithium borate or sodium borate fluxes should be used. Prior to using, the levels of contamination shall be checked. Good results have been obtained with a mixture that contains 6 parts of $Li_2B_4O_7$ and 11 parts of $LiBO_2$ by mass. This composition is a eutectic and can be used at relatively low temperatures.

NOTE 1 This mixture is commercially available under the designation 12:22.

NOTE 2 A typical sample to flux ratio is 1 unit of sample for 10 units of flux.

4.3 Releasing agent

To facilitate the casting of the sample-flux mixture and the releasing of the resultant bead after cooling, a releasing agent such as ammonium iodide (NH_4I) or lithium iodide (LiI) is recommended. Alternatively, lithium bromide (LiBr) can be used.

If a bromide-containing releasing agent is used, the line overlap of the Br L-lines with Al Kα shall be taken into account.

NOTE Fluxes with an integrated releasing agent are commercially available.

4.4 Oxidizing agents

Generally, no oxidizing agents are required if the iron is present as hematite (Fe_2O_3). However, if the samples to be analysed contain, for example, magnetite (Fe_3O_4) or if a gas burner is used without oxygen supplement, then the addition of an oxidizing agent to the flux can be required. In those cases, the use of lithium nitrate ($LiNO_3$) is recommended.

NOTE 1 Sodium nitrate can also be used as an oxidizing agent. This will render the analysis of sodium impossible.

NOTE 2 Typical mass of the oxidizing agent added is 0,5 units to 1 unit by mass for every unit of sample.

4.5 Certified reference materials

CRMs prepared in accordance with 5.2 may be used to establish calibration and to validate calibration.

4.6 Reference materials

RMs can be materials, e.g. iron ores, homogenized and prepared by a laboratory. The reference analysis of a RM shall be the average result from interlaboratory co-operative testing involving at least four laboratories able to meet the performance criteria.

5 Sampling and samples

5.1 Laboratory sample

For analysis, use a laboratory sample of less than 100 µm particle size which has been taken and prepared in accordance with ISO 3082. In the case of ores having significant contents of combined water or easily oxidizable compounds, use a particle size of less than 160 µm.

5.2 Preparation of test samples, CRMs and RMs

5.2.1 General

Depending on the ore type, proceed in accordance with either 5.2.2 or 5.2.3.

5.2.2 Ores having significant contents of combined water or easily oxidizable compounds

Prepare an air-equilibrated test sample in accordance with ISO 2596 with the following types of ore:

a) processed ores containing metallic iron;

b) natural or processed ores in which the non-oxidized sulfur content is higher than 0,2 % mass fraction;

c) natural or processed ores in which the content of combined water is higher than 2,5 % mass fraction.

NOTE Loss on ignition can be used as an estimate of combined water.

5.2.3 Ores outside the scope of 5.2.2

Prepare a pre-dried test sample by thoroughly mixing the laboratory sample and, taking multiple increments, extracting a test sample in such a manner that it is representative of the whole contents of the container. The pre-dried test portion shall be prepared in accordance with ISO 7764.

5.3 Test portion

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a manner that it is representative of the whole contents of the container. One disc from each test sample shall be prepared.

At least one CRM, of the same type (mineralogy and chemistry) as the ore used in the test discs, should be prepared and analysed.

6 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

6.1 Analytical balance, capable of weighing to four decimal places.

6.2 Crucibles and moulds. Articles made of platinum alloy with certified purity. The recommended alloys for making the pieces are platinum, platinum/gold, rhodium or platinum/rhodium.

The amount of sample and flux used should respect the volume of the crucible and mould available. It is important to take into account possible projections caused by the movement of the fusion machines during the fusion process.

The surface of the moulds should be perfectly flat throughout the life of the article. Regular polishing and checking the bottom alignment are recommended.

6.3 Fusion equipment.

Any gas heating, electric heating or induction heating equipment may be used. Equipment shall be capable of maintaining a temperature of at least 1 050 °C with capacity to vary temperature, time and agitation intensity.

The temperature shall be checked regularly with an optical pyrometer. It is important that during these measurements, the crucible contains a mass of flux similar to the mass that is used in the analysis.

In machines with more than one independent melting position, it is necessary to check their temperature homogeneity. Burners on gas machines and chambers in electrical machines shall maintain temperature stability during the melting process.

For gas burners, to minimize the loss of sulfur during the fusion, provisions shall be made to add oxygen to the flame. Alternatively, oxidizing aids can be added to the flux, see 4.4.

6.4 Electric furnace, capable of maintaining a temperature of at least 1 050 °C.

6.5 Gas burner.

When gas burners are used the temperature of the melt shall be in the range of 1 000 °C to 1 050 °C. To minimize the loss of sulfur during the fusion, provisions shall be made to add oxygen to the flame. Alternatively, oxidising aids can be added to the flux, see 4.4.

The temperature shall be checked regularly with an optical pyrometer. It is important that during these measurements, the crucible contains a mass of flux similar to the mass that is used in the analysis.

6.6 Fusion machine, with gas heating station(s) or induction furnaces, normally with agitation capacity and which allows the creation of fusion programmes with variation of time, agitation intensity and temperature. In machines with more than one independent melting position, it is necessary to check their temperature homogeneity. Burners, on gas machines and chamber in resistances on electrical machines, shall maintain temperature stability during the melting process.

6.7 XRF spectrometer.

Any wavelength dispersive XRF spectrometer equipped with a vacuum path, provided that the instrument has been checked. Performance checks can be carried out in accordance with ISO 9516-1, accumulating at least 4 × 10^7 counts for each measurement. At this number of counts, the counting statistical error will be limited to about 0,016 %. This can depend on the count rate. In case of doubt, check with the instrument manufacturer. See also 7.1.3.

Although this method is written for WDXRF equipment, its principles may also be applied to the use of EDXRF instrumentation.

Modern instruments generally have some form of dead time correction, and although the correction is not always perfect, it should be acceptable for this method, which is tolerant of small errors in dead time correction.

7 Measurements

7.1 General

7.1.1 Analytical line

For the analyte elements Fe, Si, Ca, Mn, Al, Ti, Mg, P, S and K, the $K\alpha_{1,2}$ (K-$L_{2,3}$) is recommended. For iron, the $K\alpha_{1,2}$ is the preferred line, provided the count rate is well within the dynamic range of the detector system used. If the count rate is too high, then the $K\beta_{1,3}$ (K-$M_{2,3}$) can be used. If analytes with an atomic number higher than 52 are analysed, the preferred line is the $L\alpha_1$ (L_3-M_5). For the determination of lead (if included), the $L\beta_1$ (L_2-M_4) should be used. Furthermore, if both As and Pb are analysed, then the interference of the Pb $L\alpha_1$ (L_3-M_5) on As $K\alpha_{1,2}$ (K-$L_{2,3}$) shall be taken into account or arsenic shall be determined using the As $K\beta_{1,3}$ (K-$M_{2,3}$) line. If barium is analysed, then either the $K\alpha_{1,2}$ (or K-$L_{2,3}$), the $L\alpha_1$ (L_3-M_5) or the $L\beta_1$ (L_2-M_4) can be used, depending upon which gives the best results.

7.1.2 Voltage and current

Voltages of 50 kV to 60 kV are recommended for the measurements of all the analytes with atomic number 22 or higher. For the other analytes, voltages between 25 kV and 40 kV can be used. It is highly recommended to adjust the current for each of these voltage settings in such a way that all the measurements are done at constant power of the generator.

For the best possible stability, it is advisable to work at the same constant voltage and constant current for all analytes. Tube voltages of around 40 kV to 50 kV are adequate.

7.1.3 Measuring times

The measuring time in combination with the count rate determine the counting statistical error. The measuring time should be chosen in such a way that the counting statistical error is lower than 0,5 % for Si, Ca, Ti and Al. This requirement can typically be met by accumulating at least 40 000 counts. For Fe, the counting statistical error should be at most 0,016 %. At low count rates, 4×10^7 counts need to be accumulated.

The counting statistical error for a detection system with a given dead time and for a given number of counts accumulated increases with increasing count rate. The effect becomes noticeable when the product of the dead time (expressed in seconds) and the count rate (expressed in counts per second) is 0,15 or higher. In that case, longer measuring times shall be used. At count rates below 300 000 counts per second, the effect can be neglected in most modern instrumentation. Consult the instrument manufacturer for more detail and guidance.

7.1.4 Background measurements

Measurements of the background are not required. However, if elements are not measured at trace levels, background measurements can improve the accuracy of determination at trace levels.

8 Calibration and validation

8.1 Principles

The calibration equations and inter-element corrections are established using calibration standards produced using fused beads made from pure reagents (or combinations thereof), CRMs, RMs or any combination of these. The validity of the calibration is confirmed by analysing one or more CRMs, representative of the range of analyses being undertaken, that are not used for calibration. The ongoing calibration monitoring for intensity, bead preparation and calibration drift is determined immediately after calibration and at intervals during the analytical process.

It is recommended to use several CRMs whose composition brackets the intended range of the method.

NOTE CRMs used for the validation cannot have been used for the calibration.

8.2 Preparation of fusion beads

For the preparation of fused beads, see ISO 9516-1.

8.3 Calibration and validation samples

Prepare a series of beads from pure reagents, CRMs, RMs or combinations of these as calibration standards. The series shall cover the range from the minimum to the maximum values for each element being analysed.

8.4 Validation of the calibration

8.4.1 Specimens

Prepare one or more CRMs, not used as a calibration standard and having a composition within the calibration range for each element being analysed.

When only one validation CRM is being used, select a sample in the middle of the concentration ranges. The concentration ranges of the method should then be very limited.

Where several validation CRMs are used, selected samples should cover high and low concentrations.

If no suitable CRM is available, choose an RM that satisfies the above criteria. It is recommended that each RM be validated by at least four independent laboratories.

8.4.2 Trueness validation

The trueness of the analytical method shall be checked by applying it to a CRM or a RM.

Calculate the analytical result, μ_c, for the CRM/RM, and compare it with the reference or certified value A_c. There are two possibilities:

a) $|\mu_c\text{-}A_c| \leq C$, in which case the difference between the reported result and the certified/reference value is statistically insignificant;

b) $|\mu_c\text{-}A_c| > C$, in which case the difference between the reported result and the certified/reference value is statistically significant.

where

μ_c is the final result for the CRM/RM;

A_c is the certified/reference value for the CRM/RM;

C is a value dependent on the type of CRM/RM used, see Formula (1).

$$C = 2\sqrt{\frac{s_\text{Lc}^2 + \frac{s_\text{Wc}^2}{n_\text{Wc}}}{N_\text{c}} + \sigma_\text{L}^2 + \frac{\sigma_\text{d}^2}{n}} \tag{1}$$

where

s_Lc is the between-laboratories standard deviation of the certifying laboratories;

s_Wc is the within-laboratory standard deviation of the certifying laboratories;

n_Wc is the average number of replicate determinations in the certifying laboratories;

N_c is the number of certifying laboratories;

n is the number of replicate determinations carried out on the CRM/RM;

σ_L is the independent duplicate standard deviation;

σ_d is the between-laboratories standard deviation.

The following procedure should be used when the information on the RM certificate is incomplete:

— if there are sufficient data to enable the between-laboratories standard deviation to be estimated, delete the expression $s_\text{Wc}^2 / n_\text{Wc}$ and regard s_Lc as the standard deviation of the laboratory means;

— if the certification has been made by only one laboratory or if the interlaboratory results are missing, using Formula (2):

$$C = 2\sqrt{2\sigma_{\mathrm{L}}^{2} + \frac{\sigma_{\mathrm{d}}^{2}}{n}} \qquad (2)$$

CRMs used for this purpose shall be prepared and certified in accordance with ISO Guide 35.

Analytical performance shall be assessed.

The analytical performance of a method shall be assessed with respect to precision and accuracy during the method development and validation phase.

8.5 Calibration maintenance

8.5.1 Monitor disc

To compensate for instrumental drift, all X-ray measurements shall be made relative to one or more monitor discs. Although different monitor discs could be used for each component, it is most convenient to use a single disc, containing all components to be measured.

NOTE The term “monitor” is used to indicate either a single disc or several discs that together make up the drift monitor samples.

Monitor discs are commercially available, but they can also be made from synthetic materials or a sample of ore. If an ore sample is used as the basis for the monitor, then the contents of some or all of the analytes can be boosted by spiking it with the relevant pure compounds. Fusion can be done in accordance with the procedure in 8.2 or ISO 9516-1. To ensure that overall high count-rates are attainable, the sample to flux ratio can be made higher than for the other samples.

Ensure that the monitors are stable over repeated and prolonged exposure to X-rays.

8.5.2 Duplicate monitor discs

It is highly recommended to procure or prepare at least two identical monitor discs (or sets of monitor discs if more than one disc is used) at the same time. Measure the different monitors to a high precision in a very short time span and store one of the discs in a secure place in a desiccator: this is the backup monitor. Use the other for the monitor measurements: this is the current monitor. Measure both monitors once a year or when the current monitor measurement shows a significant deviation from the previous measurement. Compare their intensities. If the intensities are not significantly different, the measurements on the current monitor are still representative for the instrumental drift.

If the intensities are significantly different, try cleaning the current monitor and measure it again. If the intensities between the current and backup monitor are still significantly different, compare the intensities of the backup monitor with the original intensities. If these are not significantly different, the cause of the intensity difference is in the current monitor; otherwise, the cause is with the instrument.

8.5.3 Monitor measurements

The period between successive monitor measurements should not exceed 8 h. It is anticipated that within that time interval, the intensity difference for the Fe measurement does not exceed 0,1 %. If experience indicates that within this time the intensity difference exceeds 0,1 %, then the maximum period between successive monitor measurements shall be shortened to ensure that the maximum difference does not exceed 0,1 %.

If the intensity for one or more of the monitor discs changes significantly between successive monitor measurements (more than 1 % relative for Fe, Si, Ca or Al), then the cause of the change should be determined and fixed. If a backup monitor (see 8.5.2) is available, the procedure suggested in 8.5.2 can be followed to help trace the cause of the aberrant intensities. If the change in intensity persists consistently, then the new changed monitor intensity can be accepted and updated by recalculating the new drift factors. Otherwise, it can be necessary to perform a re-calibration or calibrate again. However, quality control (QC) samples should be measured to confirm that the updated drift factors act sufficiently to pass the QC validation checks, or the instrument shall be recalibrated by measuring all the calibration standards.

In order to ensure that the measurements on the monitor disc are statistically relevant and that the application of monitor correction do not adversely affect the precision of the measurements, it is recommended that the counting statistical error on monitor measurements is significantly smaller than that of the other measurements. The counting statistical error should be, at most, half of that of the other measurements. This is most easily obtained by using longer measuring times.

8.5.4 Quality control measurements

8.5.4.1 Quality control material

It is essential that a sufficiently large and homogeneous sample quantity is available for QC material. The sample should be an iron ore, to ensure that problems with the sample preparation of routine ore samples are easily detected. A large, finely ground and well mixed quantity of a typical ore material from the site is well suited for this purpose. Ensure the sample preparation methods in Clause 5 are applied.

In order to monitor the quality and reproducibility of both the XRF system and the fused bead sample preparation setup, it is recommended to measure two types of QC samples on a periodic basis. These are discussed in the 8.5.4.2 and 8.5.4.3.

NOTE A quantity of around 5 kg is suitable to cover the useful lifetime of the equipment. A larger mass is required if the material is to be shared among different sample preparation machines.

8.5.4.2 Monitoring sample preparation

QC sample 1: For monitoring sample preparation reproducibility, prepare a fused bead specimen of the QC material prepared in 8.5.4.1 fresh every day or at a frequency practically suitable, and measure on the XRF. Monitor the results periodically.

It is recommended that the beads for this QC are prepared fresh and by using the same fusion equipment with the same recipe as the routine unknowns. The storage, handling, and measuring of these beads should also be identical to those of the routine specimens.

Every fusion machine used should be tested in this way separately. A fresh disk should be made for every machine that is intended to be used for sample preparation during the day.

It is advisable to run this test at the beginning of each shift.

8.5.4.3 Monitoring instrument reproducibility

QC sample 2: For monitoring XRF reproducibility, prepare a fused bead at the time of calibration setup, and measure the same bead on a daily basis or at a frequency practically suitable, e.g. once every two days. Monitor the results periodically. When the quality of such a fused bead degrades over a longer period (several days or months), discard and prepare a new fused bead sample of the same sample and continue the process. A small bias in results between the previous fused bead and new one is not relevant as the procedure is followed mainly to monitor the instrument reproducibility.

NOTE The material for this test can be a CRM or an RM, but it also possible to use a specimen made from the material for 8.5.4.2.

The two QC measurements should normally fall within two standard deviations of the average values of the statistical process control (SPC) plot. For QC sample 1 method, the standard deviation limits for the SPC monitoring should be calculated by preparing multiple fused beads of the same sample by multiple operators over a period of multiple days. A good data set to calculate the limits is around 30 to 50 independent measurements, collected in a reasonable amount of time (e.g. a few weeks maximum).

9 Reporting

9.1 General

Reporting can be done either as the oxide or the element. If conversion from oxide to element is performed, the oxide factors from Table 1 can be used.

Table 1 — Factors for conversion of oxide contents to element contents

Oxide	Element	Conversion factor
Fe_2O_3	Fe	0,699 4
SiO_2	Si	0,467 4
CaO	Ca	0,714 7
Mn_3O_4	Mn	0,720 3
Al_2O_3	Al	0,529 3
TiO_2	Ti	0,599 5
MgO	Mg	0,603 1
P_2O_5	P	0,436 4
SO_3	S	0,400 5
K_2O	K	0,830 2
SnO_2	Sn	0,787 7
V_2O_5	V	0,560 2
Cr_2O_3	Cr	0,684 2
Co_3O_4	Co	0,734 2
NiO	Ni	0,785 8
CuO	Cu	0,798 9
ZnO	Zn	0,803 4
As_2O_3	As	0,757 4
PbO	Pb	0,928 3
BaO	Ba	0,895 7

9.2 Calculation of results

This subclause only applies if measurements on duplicate fused beads are available.

If two or more beads are made from the same sample, the final result for Fe (or Fe_2O_3) is the arithmetic mean of the acceptable analytical values for the test sample. The result is calculated to four decimal places and rounded off to the second decimal place as follows:

- where the figure in the third decimal place is less than 5, it is discarded and the figure in the second decimal place is kept unchanged;
- where the figure in the third decimal place is 5 and there is a figure other than 0 in the fourth decimal place, or where the figure in the third decimal place is greater than 5, the figure in the second decimal place is increased by one;
- where the figure in the third decimal place is 5 and there is no figure other than 0 in the fourth decimal place, the 5 is discarded and the figure in the second decimal place is kept unchanged if it is 0, 2, 4, 6 or 8 and is increased by one if it is 1, 3, 5, 7 or 9.