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Guidelines for good XRF laboratory practice for the iron ore industry

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National foreword

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TECHNICAL REPORT

ISO/TR 18336

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Guidelines for good XRF laboratory practice for the iron ore industry

Lignes directrices de bonnes pratiques de laboratoire de spectrométrie de fluorescence de rayons X pour l'industrie du minerais de fer

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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).

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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*.

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Introduction

This Technical Report is intended for use in conjunction with other International Standards for the chemical analysis of iron ores. Although it was written for a high through-put iron ore laboratory, the procedures described can be modified to suit other industry or laboratory requirements. Some laboratories may find the recommended frequency of testing recommended by this Technical Report to be excessive for their situation or the precision required by them. In this case, the operator may use their informed discretion to adapt the recommendations of the guidelines to their situation.

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Guidelines for good XRF laboratory practice for the iron ore industry

1 Scope

This Technical Report specifies recommended quality control procedures for XRF laboratories operating within the iron ore industry.

2 Reagents

All reagents (including fusion fluxes and calibration reagents) should be purchased from reputable suppliers and should meet the minimum requirements for purity as listed in ISO 9516-1. All reagents should have a batch number and, where available, a certificate of analysis. Details of purchased reagents (supplier, amount purchased, quality, and batch number) should be recorded. These records should include what the reagents are used for. For batches of flux, the records should indicate which samples were analysed with a particular batch.

2.1 Fusion flux

As the levels of contamination may vary from batch to batch of flux, the purity of fusion fluxes should be checked prior to use. This can be achieved by fusing duplicate beads of pure silica and iron with the new flux, and analysing these along with beads prepared using a previously tested (certified) flux. Background concentrations should not exceed 10 ppm to 20 ppm (as compared to a certified batch of flux) for each of the following oxides Mn_3O_4, SnO_2, V_2O_5, Cr_2O_3, Co_3O_4, NiO, CuO, ZnO, As_2O_3, PbO, BaO, Na_2O and Cl and the sum of the positive differences should not exceed 40 ppm to 50 ppm.

The concentrations of the oxides should not exceed 0,01 % for each of the following oxides Fe_2O_3, SiO_2, CaO, Al_2O_3, TiO_2, MgO, K_2O and P_2O_5, (the absolute sum of the differences should not exceed 0,02 %), while backgrounds should not differ by more than 0,01 %. Sulfur (reported as SO_3) can frequently vary by 0,05 %. Where flux does not conform to specifications, a second duplicate set of beads (made with old and new flux) should be prepared by a different operator on the same day, or by the same operator on a different day. If the material fails to meet the minimum specifications, the supplier of non-conforming flux should be contacted and a replacement batch obtained and tested.

Where non-significant deviations are observed for major and trace elements between flux batches, these beads can be used to update calibration intercepts. In all cases, records of calibration prior and after amendment should be kept.

Prior to calibration amendment, the concentration levels of all previously analysed blank beads (prior to calibration amendment) should be plotted, and trends noted. If consecutive sets of duplicate beads show consistent positive concentration increases, previous beads should be refused and re-run, and the trends confirmed or negated.

Where laboratories elect to use additive fluxes (oxidizing, release agents or internal standard compounds), the homogeneity of the flux should be tested, assessed and compared against the quoted quality or against a flux batch that is known to be homogenous. Testing methods include direct measurement of added analytes, or indirect measurement of a quality parameter (ignition loss). An example of flux testing results can be found in Annex A.

As calibrations would have been amended, trends will be seen as negative values progressing towards a more positive result. If past expected trends cannot be replicated, the XRF instrument (calibration, monitor) should be inspected. If previously seen trends are repeated, flux suppliers should be contacted, and the problem discussed.

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2.2 Calibration reagents

Reagents used for XRF recalibration should be checked in a similar manner to that used to check flux (by preparing both the old reagent and the new reagent in the same type of flux). Here the level of contaminants should not exceed those reported on the reagent supplier's certificate of analysis. Please note that it is common for reagent suppliers to report reagent purity based on an "as difference" basis. Consequently, a 99,999 % reagent may have only been analysed for a single contaminant whose content is less than 1 ppm. However, this reagent may contain other contaminants which have not been analysed whose concentration may be significant.

Alternatively, if no in-house high purity reagent is available, small quantities of analysed reagents may be obtained from reputable laboratories. In addition, where reagents are suspected of contamination, they can be externally analysed.

3 Apparatus

All equipment used to prepare and measure fused glass beads should be checked on a regular basis in accordance with the schedule set out in Table 1. The frequencies defined in Table 1 are those required by a high through-put iron ore laboratory. As this Technical Report is a guideline rather than a prescriptive standard, laboratories with lesser demands can modify the figures accordingly.

3.1 Bead preparation equipment

All equipment used for the production of fused glass beads (such as balances, fusion ware, fusion furnaces and sample drying equipment) should be installed, maintained and operated in accordance to the manufacturer's recommendations. The external surfaces of all fusion furnaces should be inspected at the commencement of each shift for excessive dust and glass fragments or glass spills. If any spillage is detected it should be cleaned up before further use. On a weekly basis, or if spillage has occurred, fusion furnaces should be allowed to cool and the interior should be inspected for faults (broken or loose furnace linings that may contaminate samples) and cleaned (and repaired if necessary). Fusion furnace temperatures should be checked weekly for accuracy and uniformity of temperature using a calibrated reference thermocouple. As a general rule, an independent performance audit of bead making equipment (including any environmental factors) should be performed yearly.

Table 1 — Summary of frequency of tests and procedures

Frequency	Test
Each shift	Run Monitors[a]. Monitor data should be checked for drift (3.2.4). If problems/issues are suspected within the instrument, then increased monitor samples should be run throughout the day. At the commencement of every shift, all laboratory personnel involved in the production of XRF fusion beads should prepare at least one quality control sample (reference or certified reference material) in duplicate (5.1).
	At the commencement of every shift, all laboratory personnel involved in the production of XRF fusion beads should prepare at least one quality control sample (reference or certified reference material) in duplicate (5.1). These specimens should be measured and the results evaluated before any unknown specimens are validated.
	Monitor chilled water flow rate and temperature (3.2.2). Monitor compressed air pressure (3.2.2). Monitor detector gas flow rate and pressure (3.2.2). Monitor instrument error logs (3.2.2). Inspect fusion furnaces and clean if necessary (3.1).

[a] Monitor is used exclusively for drift correction. Where the power settings have not changed more than 1 kW from the nominal operating power and the spectrometer has been shown to have achieved stability over a desired time frame (3.2). Monitor updates can be performed for the corresponding time frame.

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Table 1 *(continued)*

Frequency	Test
Weekly	Check resolution and pulse shift for flow detectors (3.2.2). Perform disc making precision tests for new operators. Once competent, perform checks monthly (4.3). Clean interior and exterior of fusion furnaces (3.1). Check furnace temperature and uniformity of temperature (3.1).
Fortnightly	Back up spectrometer data files, calibrations and configurations (3.2.3). Measure flatness of moulds and casting dishes (4.4).
Monthly	Perform disc making precision tests for competent operators (4.3) or automated equipment. Clean monitors (3.2.4).
Three-monthly	Prepare synthetic calibration standards (SynCals) in quadruplicate and compare results to calibration data (5.3).
Half-yearly and prior to calibration or after major repairs	Perform precision tests in ISO/TR 18231. Perform long-term stability tests in ISO/TR 18231. Perform detector linearity tests in ISO/TR 18231.
Yearly	Check all bead preparation equipment (balances, fusion ware, fusion furnaces and environmental conditions) (3.1).

[a] Monitor is used exclusively for drift correction. Where the power settings have not changed more than 1 kW from the nominal operating power and the spectrometer has been shown to have achieved stability over a desired time frame (3.2). Monitor updates can be performed for the corresponding time frame.

3.2 XRF Spectrometer

3.2.1 General

All XRF instruments should be tested to ensure conformance to ISO/TR 18231 for instrument precision and detector linearity. Precision testing should be performed twice yearly, or whenever major repairs to an XRF (replacement of detector, X-ray tube, generator or measurement electronics) or to the chilled water circuit has been performed.

For all instrument tests, a relative precision of better than 0,03 % coefficient of variation is required for the analysis of Fe in iron ores, and so spectrometer precision and linearity tests should be carried out using accumulated count rates of 2×10^7 counts for each measurement. Where separate counting channels are used for the different elements (simultaneous instruments), or where the detector is changed, the dead time of each channel and each detector should be determined independently, using an appropriate wavelength for the detector/crystal combination.

Long term (24 h) XRF stability tests should be performed using various kV and mA settings, detector, collimator, and crystal combinations. All tests should be conducted biannually at a significance level of 0,03 % (2×10^7 counts), and immediately prior to performing an XRF calibration. Instruments should exhibit short-term stability in the order of 0,03 % over a 6 h to 12 h period. If an XRF fails to meet desired precisions, instrument manufacturers should be consulted and the cause of poor instrument stability rectified.

Additional spectrometer tests can be found in Annexes I, J and L.

3.2.2 Each shift/weekly spectrometer checks

As the operation and performance of an XRF spectrometer is highly dependent on the quality of external services such as chilled water (flow rate and temperature), compressed air (pressure) and detector gas (flow rate and pressure), the status of these supplies should be monitored on a daily basis and the

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results recorded in a check sheet. In addition, error logs should be checked daily and corrective action taken when faults are reported. As spectrometers possess internal services (such as chilled water flow, temperature and conductivity, internal spectrometer temperature and instrument vacuum levels), the status of these should be monitored. Note that as all modern XRF software possesses spectrometer status screens, these should be configured to be open in a minimised window so that the status of spectrometer services can be monitored.

As the performance and condition of spectrometer flow detectors are highly dependent on the quality of detector gas, the resolution and pulse shift should be checked on a weekly basis. Checks should be performed with regard to the manufacturer's recommended procedure. Minor changes in pulse shift (≥2, ≤5) should be corrected (note as spectrometers typically have different detector voltage settings for each crystal, all should be checked when tests and amendments are performed). Significant changes in detector resolution or pulse shift position should be investigated as these may be caused by window failure or gas contamination (leaks).

3.2.3 Hardware and software backup

A backup of spectrometer data files, calibrations and configurations should be performed every two weeks. This backup should be stored on a remote computer or shared data storage area. XRF software can be stored in a secure area within the laboratory.

It is also highly recommended that XRF software and spectrometer data (calibration files and configuration settings) be loaded on several computers (laptops preferred) which possess the necessary hardware (serial ports) required to connect and communicate with the spectrometer. These computers will serve as a hardware and software backup.

3.2.4 Instrument monitors

To compensate for drifts in X-ray tube output intensity, all X-ray measurements should be made relative to a monitor disc. 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. The requirement of the monitor disc is that it be stable over a long period. Also, the monitor should contain sufficient of each element to ensure that the intensity of each analytical line is much higher than the intensity of the background and can be measured with the required precision in a reasonable time. Suitable stable monitor discs made for the analysis of iron ore are available commercially.

The essential property of the monitor is its long-term stability. It should be flat so that it can be placed reproducibly in the sample holder of the XRF, and the analytical surface should be polished for easy cleaning. Monitors should be cleaned in accordance with manufacturer's specifications or every two months with ethanol or acetone. In-house prepared borate monitors are not recommended as they are unstable and deteriorate over time.

The accumulated counts for the various elements should be such that they are higher than those from iron ores, so that precision is not limited by the monitor. For the major elements, including Fe, the count rate may be less than observed from iron ores, but it should be high enough that, in a short counting time, the counting error is sufficiently small. Monitors should be run every 4 h to 6 h during, and prior to the commencement of routine analyses.

Monitor count times can be determined at the time of calibration using the formula below.

The monitor count time *TM* (in seconds) for each element is given by Formula (1):

$$TM = 2 \times \frac{RS \times T}{RM} \tag{1}$$

where

RS is the intensity, c/s, from the calibration standard SynCal, measured for 10 s (see ISO 9516-1);

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