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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).

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).

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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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