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Iron ores — Determination of chlorine content — X-ray fluorescence spectrometric method

Minerais de fer — Dosage du chlore — Méthode par spectrométrie de fluorescence de rayons X

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Foreword

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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 summarizes the results of inter-laboratory testing for the determination of chlorine in iron ores by X-ray fluorescence. The method was developed by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*. As no other methods for determination of chlorine exist in ISO/TC 102, the method was designed to complement method ISO 9516-1. A method for water soluble chloride does exist (ISO 9517) but the range of application of ISO 9517 is significantly less than the XRF method described in this Technical Report. The method described in this Technical Report represents the first attempt of the committee to determine total chloride.

Evaluation of the data from the inter-laboratory test indicated that the method could not be considered for publication as an International Standard as the precision of the method was less good than the precision of the method for water soluble chloride described in ISO 9517. In addition, a test on the trueness of the method was not possible as no potential test samples that were certified for Cl were available. Although the test samples used were characterized using neutron activation, the XRF values were biased with respect to the neutron activation values and the neutron activation method itself was not considered to be a standard method.

Although the method was not considered suitable for publication as an International Standard, it was agreed that the method was otherwise suitable for use in the industry and the committee agreed that it should be published as a Technical Report.

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Iron ores — Determination of chlorine content — X-ray fluorescence spectrometric method

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

1 Scope

This Technical Report sets out a wavelength dispersive X-ray fluorescence procedure for the determination of chlorine in iron ores.

The method is applicable to a concentration range of 0,027 % to 1,15 % of chlorine in iron ores regardless of mineralogical type.

It is not intended that this method be used for the purpose of trade in iron ores due to the precision of the method.

2 Principle

The glass discs for X-ray fluorescence measurement are prepared by incorporating the test portion of the iron ore sample, via fusion, into a borate glass disc using a casting procedure. By using a fused glass disc, particle size effects are eliminated.

The method is applicable to data from simultaneous and sequential X-ray fluorescence spectrometers.

Calibration is carried out using pure chemicals, with chlorine added as a stock solution of sodium chloride. Because the oxygen of the flux is the dominant element in the glass disc, and because oxygen is a heavy absorber to Cl Kα, matrix effects are small and calibration is based on a linear relationship between concentration and measured fluorescent intensity. Background measurements are made to determine net line intensities.

3 Reagents and materials

3.1 During analysis, use only reagents of recognized high purity.

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

3.2 Iron (III) oxide (Fe_2O_3), nominally 99,999 % Fe_2O_3.

The iron (III) oxide should contain less than 3 µg/g of chlorine. It should initially be heated at 1 000 °C in a platinum crucible for a minimum of 12 h to reduce contaminant concentrations and cooled in a desiccator. Subsequently, if required, it should be re-ignited at 1 000 °C for 1 h and cooled in a desiccator.

3.3 Sodium chloride (NaCl), 13,2 g/l solution.

Analytical grade sodium chloride should be dried at 105 °C for 1 h and cooled in a desiccator. Weigh 13,2 g dried sodium chloride into a 1 000 ml one-mark volumetric flask and dilute to volume.

3.4 Desiccant, should be a freshly-regenerated self-indicating silica gel.

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