

ICS 73.060.10

CCS D31



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 6730.87-2023

**Iron ores - Determination of total iron and other multi-element
content - Wavelength dispersive X-ray fluorescence
spectrometry (cobalt internal standard method)**

铁矿石 全铁及其他多元素含量的测定 波长色散X射线荧光光谱法（钴内标法）

Issued on: August 6, 2023

Implemented on: March 1, 2024

**Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC**

Table of Contents

1	Scope
2	Normative references
3	Terms and definitions
4	Principle
5	Reagents and materials
5.22	Ammonium bromide (NH ₄ Br) solution, 300 g/L. Dissolve
5.23	Ammonium iodide (NH ₄ I) solution, 300 g/L. Dissolve
6	Instruments and equipment
7	Sampling and sample preparation
8	Preparation of glass pieces
8.2	Preparation of glass pieces for calibration
8.3	Pre-oxidize and then melt lithium tetraborate flux base
8.4	Pre-oxidize and then melt in an internal standard flux crucible
8.4.1	Weigh
8.5	Pre-oxidize and then melt boric acid
8.5.1	Take
8.6.1	Weigh
9	Instrument preparation
10	Analysis steps...
11	Result calculation and representation...

Foreword

This document was drafted in accordance with the rules given in GB/T 1.1-2020 "Directives for standardization - Part

1. Rules for the structure and drafting of standardizing documents". This document is Part 87 of GB/T 6730 "Iron ores". See Annex A for the published parts of GB/T 6730. Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. The issuing authority shall not be held responsible for identifying any or all such patent rights. This document was proposed by China Iron and Steel Industry Association. This document shall be under the jurisdiction of National Technical Committee on Iron Ores and Direct Reduced Irons of Standardization Administration of China (SAC/TC 317). Main drafting organizations of this document. Wuhan University of Science and

Technology, Jiangsu Shagang Group Co., Ltd., Guangxi Liuzhou Iron and Steel Group Co., Ltd., Guangdong Zhongnan Iron and Steel Co., Ltd., Jiangsu Yonggang Group Co., Ltd., Hunan Valin Lianyuan Iron and Steel Co., Ltd., Zhongxin Iron and Steel Group Co., Ltd., Panxi Vanadium Titanium Inspection and Testing Institute, Metallurgical Industry Information Standards Institute. Main drafters of this document. Zhou Shuangqing, Zhu Qingyou, Xu Jianping, Zhang Jiming, Zhu Chunyao, Li Jiao, Huang Zhimin, Xing Wenqing, Yu Lei, Zhao Jie, Chen Haiyan, Wu Yuan, Xiao Xing, Zhuo Wenkeng, Zhang Zhongting, Li Zijing, Sun Hao, Chen Zibin, Zhu Rong, Li Shijing, Zhu Zhijin, Wu Chaochao.

1 Scope

Part 87 of China's national series on the analysis of iron ores, determining total iron and fifteen other elements by wavelength dispersive X-ray fluorescence with a cobalt internal standard. XRF is how ore is actually analysed at scale: a fused bead or pressed pellet is presented to the instrument and every element of interest is reported in minutes, against the hours that the wet chemical methods elsewhere in this series require for one element each. Its weakness is that the result depends on the sample's matrix and preparation, and an internal standard is the classical correction for that - cobalt is added at a known level, and each element's response is referred to cobalt's, so variations that affect the whole measurement cancel. This part covers total iron together with silicon, aluminium, calcium, magnesium, manganese, titanium, vanadium, phosphorus, copper, lead, chromium, zinc, arsenic, potassium and sulfur, and applies to iron ore, iron concentrate, sinter and pellets over the ranges given in the document.

This document specifies the use of wavelength dispersive X-ray fluorescence spectrometry (cobalt internal standard method) for the determination of all iron, silicon, aluminum, calcium, magnesium, manganese, titanium, vanadium, phosphorus, copper, lead, chromium, zinc, arsenic, potassium and sulfur content. This document is applicable to the determination of the following elements in iron ore, iron concentrate, sinter and pellets. The measurement range is shown in Table 1.

2 Normative references

The following referenced documents are indispensable for the application 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.

GB/T 6682, Water for analytical laboratory use -- Specification and test methods
Element Measurement range (mass fraction) % Measurement range (mass fraction) %

GB/T 6730.1, Iron ores -- Preparation of pre-dried test samples for chemical analysis

GB/T 8170, Rules of rounding off for numerical values and expression and judgement of limiting values

GB/T 10322.1, Iron ores - Sampling and sample preparation procedures

GB/T 15000.3, Directives for the work of reference materials -- Part

3.Reference materials -- Characterization and assessment of homogeneity and stability

GB/T 16597, Analytical methods of metallurgical products -- General rule for X-ray fluorescence spectrometric methods JJG 810, Verification Regulation for Wavelength Dispersive X-Ray Fluorescence Spectrometers YB/T 082, Specification for certified reference materials for metallurgical product analysis

3 Terms and definitions

There are no terms or definitions to be defined in this document.

4 Principle

The sample and the cobalt internal standard added accurately in quantitative amounts are melted into a borate glass piece with the flux. Measure the X-ray fluorescence intensity of the element to be measured.

5 Reagents and materials

Unless otherwise stated, only approved analytically pure reagents and grade 3 or above distilled water complying with GB/T 6682 or water with equivalent purity are used in the analysis.

5.1 Iron dioxide (Fe_2O_3), guaranteed reagent. Burn ferric oxide at 1000°C for at least 1 h and cool to room temperature in a desiccator.

5.8 Calcium carbonate (CaCO_3), guaranteed reagent. Bake calcium carbonate at 105°C for at least 1 hour and cool to room temperature in a desiccator.

5.9 Magnesium oxide (MgO), guaranteed reagent. Burn magnesium oxide at 1000°C for at least 1 h and cool to room temperature in a desiccator. Weigh immediately.

5.10 Aluminum trioxide (Al_2O_3), guaranteed reagent. Ignite aluminum oxide at 1000°C for at least 2 h and cool to room temperature in a desiccator.

5.11 Manganese tetroxide (Mn_3O_4), guaranteed reagent. Bake manganese tetroxide at 105°C for at least 2 h and cool to room temperature in a desiccator.

5.12 Ammonium dihydrogen phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$), guaranteed reagent. Bake ammonium dihydrogen phosphate at 105°C for at least 1 h and cool to room temperature in a desiccator.

5.13 Potassium carbonate (K_2CO_3), guaranteed reagent. Bake potassium carbonate at 105°C for at least 1 h and cool to room temperature in a desiccator.

5.22 Ammonium bromide (NH₄Br) solution, 300 g/L. Dissolve

15.0 g of ammonium bromide in 50 mL of water.

5.23 Ammonium iodide (NH₄I) solution, 300 g/L. Dissolve

15.0 g of ammonium iodide in 50 mL of water.

5.24 Standard samples/standard materials. They are used for establishing calibration curves and quality control. The selected standard samples/standard materials shall comply with the regulations of GB/T 15000.3 and YB/T 082. The content of each analyzed element shall cover the analysis range and have an appropriate gradient.

6 Instruments and equipment

6.1 Wavelength dispersive X-ray fluorescence spectrometer The X-ray fluorescence spectrometer shall meet the requirements of JJG 810 and GB/T 16597.

6.2 Analytical balance The accuracy is

0.1 mg.

6.4 Melting furnace Electrically heated, high frequency heated or gas heated melting furnace. The maximum working temperature is not less than 1100°C. Temperature control accuracy is ±10°C.

6.5 Crucible and mold (or crucible doubles as mold) The crucible is made of platinum-gold alloy (95% Pt.5% Au) and has a volume sufficient to accommodate the weighed flux and test material. The mold shall be thick enough to prevent deformation after heating.

7 Sampling and sample preparation

7.1 Laboratory specimens Samples shall be taken and prepared in accordance with the provisions of GB/T 10322.1. The particle size of the specimen shall be less than 100 μm

7.2 Preparation of pre-dried specimens Prepare pre-dried specimens according to the provisions of GB/T 6730.1.

8 Preparation of glass pieces

8.1 General When preparing glass pieces, the weighing amount of the test material is

0.4 g~

0.8 g, accurate to 0.0001 g.

8.2 Preparation of glass pieces for calibration