

ICS 73.060.10

CCS D31



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

GB/T 6730.72-2016

**Iron ores - Determination of arsenic, chromium, cadmium, lead
and mercury content - Inductively coupled plasma mass
spectrometry**

铁矿石 砷、铬、镉、铅和汞含量的测定 电感耦合等离子体质谱法 (ICP-MS)

Issued on: February 24, 2016

Implemented on: November 1, 2016

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine;
Standardization Administration of the PRC**

Table of Contents

1 Scope
2 Normative references
3 Principles
4 Reagents
4.1 Hydrochloric acid, $\rho =$
4.2 Hydrofluoric acid, $\rho =$
4.3 Nitric acid, $\rho =$
5 Instruments
6 Sampling and specimen preparation
7 Analytical procedures
7.1 Sample amount Weigh
7.3 Blank test and verification test
8 Result calculation...
9 Test report

1 Scope

Part 72 of China's national series on the analysis of iron ores, determining arsenic, chromium, cadmium, lead and mercury by inductively coupled plasma mass spectrometry. These five elements are grouped for a reason that has nothing to do with metallurgy: they are the heavy metals that environmental and customs regulation cares about, and a cargo of ore has to be characterised for them whatever its iron content. ICP-MS is the right tool because it determines all five in one aspiration at the concentrations that matter, which the individual colorimetric methods elsewhere in this series cannot do without five separate preparations. Mercury is the difficult member of the set - volatile, prone to memory effects in the instrument's spray chamber, and lost during dissolution unless the digestion is closed - which is why its inclusion changes the sample preparation for all of them. This part applies to natural iron ore, iron concentrate, sinter and pellets, with the measuring range for each element given in the document.

This Part of GB/T 6730 specifies the method for the determination of arsenic, chromium, cadmium, lead, mercury in iron ore, by inductively coupled plasma mass spectrometry (ICP-MS). This Part applies to natural iron ore, iron concentrate, sinter and pellets. The measurement range is as shown in Table 1.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

GB/T 602 Chemical reagent - Preparations of standard solutions for impurity

GB/T 6379.2 Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

GB/T 6682 Water for analytical laboratory use - Specification and test methods

GB/T 6730.1 Iron ores - Preparation of predried test samples for chemical analysis

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

GB/T 12806 Laboratory glassware - One-mark volumetric flasks

GB/T 12807 Laboratory glassware - Graduated pipettes

3 Principles

The sample is digested by high-temperature sealed microwave acid. The treated solution is diluted with dilute nitric acid to constant volume. The ^{45}Sc , ^{72}Ge , ^{115}In , ^{209}Bi are used as internal standards, to directly carry out ICP-MS measurement.

4 Reagents

Unless otherwise specified, the reagents for analysis shall be of superior purity or purified to the superior purity level; the water shall comply with the grade 2 water specified in GB/T 6682.

4.3 Nitric acid, $\rho =$

4.4 Nitric acid, 2 + 98.

4.5 Standard stock solutions of chromium, arsenic, cadmium, mercury, lead: All are 1000 $\mu\text{g/mL}$ (nitric acid medium, medium volume concentration is about 5%), OR prepared using certified reference materials within the validity period.

4.6 The ^{45}Sc , ^{72}Ge , ^{115}In , ^{209}Bi standard solution: 1.0 $\mu\text{g/mL}$ (nitric acid medium, medium volume concentration is about 5%).

4.7 High-purity argon gas, which has a purity not less than 99.999%.

5 Instruments

5.1 General equipment and appliances Unless otherwise specified, routine laboratory equipment such as volumetric flasks and pipetting devices shall comply with Level A, which is specified in GB/T 12806 and GB/T 12807.

5.2 Inductively coupled plasma mass spectrometer For the instrument parameters and usage conditions, refer to the requirements in Appendix A. It is equipped with a hydrofluoric acid-resistant sampling system.

5.3 High temperature and pressure microwave digestion furnace Equipped with polytetrafluoroethylene (PTFE) high temperature resistant pressure sealed digestion tank (volume not more than 60 mL), its working conditions refer to the requirements in Appendix A. OR it may use other digestion equipment with the same function.

6 Sampling and specimen preparation

6.1 Laboratory specimens Specimens shall be taken and prepared in accordance with GB/T 10322.1. Generally, the specimen particle size shall be less than 100 μm . If the content of combined water or easy oxides in the specimen is high, the particle size shall be less than 160 μm .

6.2 Preparation of pre-dried specimens Thoroughly mix the laboratory specimens. Take specimens using the reduction method. Dry the specimen at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, in accordance with GB/T 6730.1.

7.1 Sample amount Weigh

0.1 g $\pm$

0.05 g of pre-dried specimen (see 6.2), accurate to 0.0001 g.

7.2 Number of measurements Referring to the procedures in Appendix B, measure the same pre-dried specimen at least twice independently and take the average value.

Note: "Independent" means that the results of the second and subsequent measurements are not affected by the results of the previous measurements. In this analytical method, this condition means repeated measurements by the same operator at different times or by different operators, including the use of appropriate recalibration.

7.3 Blank test and verification test

7.3.1 Blank test: Perform a blank test along with sample analysis.

7.3.2 Verification test: Analyze the same type of standard sample, along with the sample

Note 2: Such standard samples shall not be used, unless it is confirmed that there is no

deviation from the standard value. The acceptability of the analytical results for each specimen depends on the acceptability of the analytical results of the corresponding CRM or RM. If the CRM or RM results fail the accuracy test, the relevant specimen results are unacceptable and the specimen shall be re-dissolved and measured.

9 Test report

The test report shall include the following information:

- a) Name and address of the testing laboratory;
- b) Test report release date;
- c) The number of this Part;
- d) Necessary detailed description of the specimen itself;
- e) Analytical results;
- f) The number corresponding to the analytical result;