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GB/T 6730.55-2019

Replacing GB/T 6730.55-2019

Iron ores - Determination of tin content - Flame atomic absorption spectrometric method

铁矿石　锡含量的测定　火焰原子吸收光谱法

(ISO 11534:2006, Iron ores – determination of tin - Flame atomic absorption spectrometric method, MOD)

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Foreword

GB/T 6730, Iron ores, comprises dozens of parts.

This Part is the 55th part of GB/T 6730.

This Part was drafted in accordance with the rules given in GB/T 1.1-2009.

This Part replaces GB/T 6730.55-2004, *Iron ores – Determination of tin content – Flame atomic absorption spectrometric method*. The significant technical changes with respect to GB/T 6730.55-2004, in addition to some editorial changes, are as follows:

-- it adds in apparatus high temperature furnace suitable for heating to 1 000°C ~ 1 020°C (see 5.4);

-- it changes the requirements for sample amount, accurate to 0.000 2 g (see 7.2; 7.2 of edition 2004);

-- it changes the precision regression equation (see 8.2.1; 8.2.1 of edition 2004);

-- it changes the trueness check equation (see 8.2.4; 8.2.4 of edition 2004).

This Part was redrafted by modifying and adopting ISO 11534:2006, *Iron ores – Determination of tin – Flame atomic absorption spectrometric method*.

The technical differences between this Part and ISO 11534:2006, and the reasons, are as follows:

-- with respect to normative references, some adjustments are made in this Part resulting in some technical differences, in order to adapt to the technical conditions in China. The adjustments are intensively reflected in “Normative references” of Clause 2. This Part:

- adds the references including GB/T 6379.1, GB/T 6379.2, GB/T 7728, GB/T 8170, GB/T 12807, GB/T 12808 (see Clause 5, 8.2.1, 8.2.5);
- deletes ISO 648;
- uses GB/T 6682 modifying and adopting the international standard to replace ISO 3696 (see Clause 4);
- uses GB/T 6730.1 modifying and adopting the international standard to replace ISO 7764 (see 6.2);
- uses GB/T 10322.1 equivalently adopting the international standard to replace ISO 3082 (see 6.1);
- uses GB/T 12806 non-equivalently adopting the international standard to replace 1042 (see Clause 5);

-- it adjusts the sequence and numbers of the clauses in the “Reagents”

Iron ores – Determination of tin content – Flame atomic absorption spectrometric method

Warning – The operators using this Part shall have practical experiences in the work in a normal laboratory. This Part does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this Part to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This Part of GB/T 6730 specifies the flame atomic absorption spectrometric method for the determination of the mass fraction in iron ores.

This Part applies to mass fractions of tin between 0.001% and 0.015% in natural ores, iron ore concentrates and agglomerates, including sinter products.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition dated applies to this document. For undated references, the latest edition of the referenced documents (including all amendments) applies to this document.

GB/T 6739.1, *Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions* (GB/T 6379.1-2004, ISO 5725-1:1994, IDT)

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 6379.1-2004, ISO 5725-1:1994, IDT)

GB/T 6682, *Water for analytical laboratory use – Specification and test methods* (GB/T 6682-2008, ISO 3696:1987, MOD)

GB/T 6730.1, *Iron ores – Preparation of predried test samples for chemical analysis* (GB/T 6730.1-2016, ISO 7764:2006, MOD)

4.5 Hydrofluoric acid, ρ about 1.13 g/mL.

4.6 Hydrochloric acid, 1+1.

4.7 Hydrochloric acid, 2+3.

4.8 Sulfuric acid, 1+1.

4.9 Ascorbic acid solution, 200 g/l, freshly prepared just prior to use.

4.10 Potassium iodide/ascorbic acid solution: dissolve 90 g of potassium iodide in water, add 30 g of ascorbic acid and 30 mL of hydrochloric acid (4.3), and dilute with water to 200 mL. This solution shall be freshly prepared just prior to use.

4.11 Tri-n-octyl phosphine oxide (TOPO)/4-methyl-2 pentanone (MIBK) solution: dissolve 1 g of TOPO in 100 mL of MIBK.

4.12 Tin standard solution, 200 μg/mL.

Dissolve 0.1 000 g of tin metal (purity > 99.5%) in a platinum crucible (with cover) with 5 mL of hydrochloric acid (4.3). After cooling, transfer the solution to a 500 mL volumetric flask, add 200 mL of hydrochloric acid (4.6), and dilute to volume with hydrochloric acid (4.6), and mix. A certified tin standard solution, which is commercially available, can also be used.

4.13 Tin standard solution, 20 μg/mL.

Transfer 10 mL of tin standard solution (4.12) to a 100 mL volumetric flask, dilute to volume with hydrochloric acid (4.6) and mix.

5 Apparatus

Unless specified otherwise, ordinary laboratory apparatus, including one-mark pipettes, graduated pipettes and one-mark volumetric flasks complying with the specifications of GB/T 12806, GB/T 12807 and GB/T 12808 respectively, and the following.

5.1 Platinum crucible, of capacity 25 ml to 30 ml.

5.2 Platinum rod.

5.3 Separating funnel, 200 mL.

5.4 High temperature furnace, suitable for heating at 1 000°C to 1 020°C.

5.5 Atomic absorption spectrometer, equipped with a dinitrogen oxide/acetylene burner.

Carry out the analysis at least in duplicate in accordance with Annex A, independently, on one predried test sample.

NOTE: The expression “independently” means that the second and any subsequent result is not affected by the previous result(s). For the particular analytical method, this condition implies that the repetition of the procedure is carried out either by the same operator at a different time or by a different operator including, in either case, appropriate recalibration.

7.2 Test portion

Taking several increments, weigh, to the nearest 0.000 2 g, approximately 1.00 g of the predried test sample obtained in accordance with 6.2. The test portion should be taken and weighed quickly to avoid re-absorption of moisture.

7.3 Blank test and check test

7.3.1 Blank test

When the analysis is carried out, carry out the blank test; all reagents are from the same reagent bottle.

7.3.2 Check test

When the analysis is carried out, carry out the check test on the same type of reference samples.

7.4 Determination

7.4.1 Decomposition of the test portion

Transfer the test portion (7.2) to a crucible (5.1), moisten with a few drops of water, add 2 ml of sulfuric acid (4.8) and 6 ml of hydrofluoric acid (4.5), mix well using a platinum rod (5.2) and wash the rod with water. Using a low-temperature electric furnace, heat the crucible gently at first, then to white fumes, and continue heating until no fumes are observed. Place the crucible in a high temperature furnace (5.4) at 1 000°C to 1 020°C for 30 min. Remove the platinum crucible and cool. Add 4.8 g the mixed solvent (4.1) of sodium carbonate and sodium tetraborate, put on the platinum cover, place int the high temperature furnace for fusion for 30 min at 1 000°C ~ 1 020°C; remove to cool. Place the platinum crucible and cover in a 200 ml beaker. Add 50 ml of hydrochloric acid (4.7), cover the beaker with a watch glass and heat in a water bath at about 90°C until dissolution of the melt is complete. Wash out the crucible and cover and cool. In order to avoid the volatilization of tin, they shall be placed in a water bath at about 90°C.

7.4.2 Treatment of the test solution

Transfer the solution (7.4.1) to a 200 ml separating funnel having a mark indicating 100

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