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

GB/T 1506-2016

Replacing GB/T 1506-2002

**Manganese ores - Determination of manganese content -
Potentiometric method and ammonium iron(II) sulphate
titrimetric method**

锰矿石 锰含量的测定 电位滴定法和硫酸亚铁铵滴定法

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009. This standard replaces GB/T 1506-2002 "Manganese ores - Determination of manganese content - Potentiometric method and ammonium iron (II) sulphate titrimetric method". As compared with GB/T 1506-2002, the main technical changes are as follows: - ADD the content of normative references; - MODIFY the expression of requirements for the reagents and water used; - ADD the contents of the reagents used; - ADD the provisions on the number of determinations; - MODIFY the operational statement of the blank value determination; - MODIFY the added water volume for the preparation of N-phenyl anthranilic acid indicator solution (0.2 g/L); - MODIFY the operational statement of the dissolution of the sample by ammonium nitrate oxidation; - MODIFY the operational statement of the dissolution of the

sample by perchloric acid oxidation; - MODIFY the amount of nitric acid added when the sample is dissolved by perchloric acid oxidation; - ADD notes on the applicability of the two sample dissolution methods; - MODIFY the operational statement before titration with ammonium ferrous sulfate standard solution; - ADD the acceptance of the analytical value of the standard sample; - ADD the calculation of the final result; - ADD the requirements for the test report; - ADD Appendix A. This standard was proposed by the China Iron and Steel Association. This standard shall be under the jurisdiction of the National Standards Committee for Pig Iron and Ferroalloys (SAC/TC 318). Drafting organizations of this standard. Angang Mining Group, Changsha Mining and Metallurgy Research Institute Co., Ltd., Metallurgical Industry Information Standards Institute. The main drafters of this standard. Tian Hong, Chen Zhihua, Gao Jingjun, Zhang Bin, Yang Lin, Sun Deming, Wang Dan, Ma Caiyun, Lu Chunsheng. This standard replaces the standard previously issued as follows: - GB/T 1506-1979, GB/T 1506-2002. Manganese ores - Determination of manganese content - Potentiometric method and ammonium iron (II) sulphate titrimetric method Warning - The personnel using this standard shall have practical experience in formal laboratory work. This standard does not address all possible safety issues. It is the responsibility of the user to take appropriate safety and health measures and to ensure compliance with the conditions set by the relevant national regulations.

1 Scope

GB/T 1506-2016 is the Chinese national standard on manganese ores - determination of manganese content - potentiometric method and ammonium iron(ii) sulphate titrimetric method, in the field of mining and minerals. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 29 August 2016 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 July 2017. Classification: ICS 73.060.20, CCS D32. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This standard specifies the determination of manganese in manganese ore by potentiometric method and ammonium iron (II) sulphate titrimetric method. This standard is applicable to the determination of manganese content in manganese ore and manganese concentrates with vanadium content (mass fraction) not more than 0.05%, the determination range (mass fraction) is as follows: manganese content of potentiometric titration $\geq 15.00\%$; manganese content of ammonium iron (II) sulphate titrimetric method is $8.00\% \sim 60.00\%$.

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) are applicable to this standard.

GB/T 2011 Method of sampling and sample preparation of manganese ores in bulk

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

3.2.5 Nitric acid, rho =

3.2.6 Sulfuric acid, 1+1.

3.2.7 Hydrochloric acid, 1+4.

3.2.8 Sodium carbonate solution, 50 g/L.

3.2.9 Sodium pyrophosphate ($\text{Na}_2\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$) solution, 120 g/L. It is prepared 24 hours before use.

3.2.10 Filter type crucible, No.3.

3.5 Analytical procedures

3.5.1 Number of analyses PERFORM at least two reproducibility analyses on the same sample.

3.5.2 Sample quantity WEIGH

1.00 g of air-dried sample to the nearest 0.0001 g. WEIGH the specimen in accordance with

GB/T 14949.8 to determine the wet moisture content.

3.5.3 Blank test PERFORM blank test along with the sample.

3.5.4 Determination

3.5.4.1 Decomposition of samples PLACE the sample (see 3.5.2) in a 300 mL Teflon beaker, ADD a few drops of water to wet it, ADD 20 mL of hydrochloric acid (see 3.2.2) and 2 mL ~ 3 mL of nitric acid (see 3.2.5), HEAT the solution to drive off the nitrogen oxidation.

3.5.4.2 Residue treatment TRANSFER the filter paper containing the residue into a platinum crucible, DRY it, ASH it, BURN it at 600 °C ~ 700 °C, ADD 2 g of sodium carbonate (see 3.2.1), MELT it at 900 °C ~ 1000 °C. REMOVE the crucible and COOL it down.

3.5.4.3 Potentiometric titration PIPETTE 100 mL of the solution into a 500 mL beaker containing 250 mL of sodium pyrophosphate solution (see 3.2.9), CONTINUE stirring whilst adding [if there is a precipitate, USE less solution or INCREASE the sodium pyrophosphate solution (see 3.2.9), to ensure that the solution is clear]. 3.2.13) to titrate it, until the potentiometric titrator (see 3.3.2) has obvious potential sharp change or pointer deflection, as the end point.

3.6 Calculation of results Calculate the manganese content (mass fraction) in the sample in accordance with formula (3) or formula (4), expressed in %.

3.7 Allowable difference The difference in analysis results shall not be greater than the allowable difference listed in Table 1.

4 Method 2.ammonium iron (II) sulphate titrimetric method

4.1 Principle After the sample is dissolved by hydrochloric acid, nitric acid and phosphoric acid, in the phosphoric acid medium, ammonium phosphate or perchloric acid is added to oxidize manganese to trivalent, the N-benzo anthranilic acid is used as an indicator, use ammonium iron (II) sulphate standard titration solution to titrate it.

4.2 Reagents and materials During the analysis, only approved analytical reagents and waters of grade 3 and above purity in accordance with GB/T 6682 are used.

4.2.1 Ammonium nitrate, solid.

4.2.6 Sulfuric acid, 1 + 1.

4.2.7 Sulfuric acid, 5 + 95.

4.2.8 Sulfuric acid, 1 + 4.

4.2.9 Potassium dichromate standard solution [$c(1/6 K_2Cr_2O_7) = 0.04000 \text{ mol/L}$]. WEIGH 1.9615 g of reference potassium dichromate (pre-dried at 150 °C for 2 h and cooled to room temperature in a desiccator),

4.2.10.1 Preparation WEIGH

15.68 g of ammonium iron (II) sulphate, DISSOLVE it in 1000 mL of sulfuric acid (5 + 95) solution, MIX it uniformly.

4.2.10.2 Calibration PIPETTE

25.00 mL of potassium dichromate standard solution (see 4.2.9) into a 250 mL conical flask, ADD 40 mL of sulfuric acid (see 4.2.8) and 5 mL of phosphoric acid (see 4.2.2), USE the ammonium iron (II) sulphate standard titration solution (see 4.2.10) to titrate it until the orange color disappears, ADD 2 drops of the N-benzoanthranilic acid indicator solution (see 4.2.11), CONTINUE adding it until the solution becomes green, which is the end point.