

SN

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

SN/T 2697-2010

**Determination of sulfur, phosphorus, arsenic and chloride in
coal for import and export - X-ray fluorescence spectrometric
method**

进出口煤炭中硫、磷、砷和氯的测定 X射线荧光光谱法

(English Translation)

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1 Scope

This Part of SN/T 3323 specifies potassium dichromate titration, the method to determine the ferrous oxide content in mill scale.

This Part is applicable to the determination of ferrous oxide content in mill scale. The determination range is 10%~60%.

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

GB/T 6730.6-1986, Methods for chemical analysis of iron ores - The ferric chloride- sodium acetate volumetric method for the determination of metallic iron content

GB/T 6730.7-1986, Methods for chemical analysis of iron ores - The sulfosalicylic acid photometric method for the determination of metallic iron content

3 Method summary

In the absence of air, use hydrochloric acid and sodium fluoride to decompose the specimen. Use sodium diphenylamine sulfonate as indicator. Determine ferrous and

metallic iron content by potassium dichromate volumetric method. After subtracting the amount of metallic iron, the amount of ferrous iron is obtained.

4 Reagents

Unless otherwise stated, all reagents are analytically pure and above. Water is grade two water specified in GB/T 6682.

4.1

Sodium fluoride.

4.2

Sodium bicarbonate.

4.3

Hydrochloric acid ($\rho=1.19\text{g/mL}$).

4.4

Saturated sodium bicarbonate solution.

4.5 Mixed sulfuric acid: Put 200mL of sulfuric acid ($\rho=1.84\text{g/mL}$) under stirring, then

pour into 500mL of water slowly. Then, add 300mL of phosphoric acid ($\rho=1.70\text{g/mL}$).

Mix well.

4.6 Sodium diphenylamine sulfonate solution (2g/L): Weigh 0.2g of sodium

diphenylamine sulfonate and dissolve in 100mL of water.

4.7 Potassium dichromate standard titration solution (0.0050mol/L): Weigh 1.4709g of

potassium dichromate (reference reagent) that has been pre-dried at 150°C for 1h, and dissolve in water. Transfer into a 1000mL volumetric flask. Use water to dilute to the scale. Mix well.

4.8 Ferrous ammonium sulfate solution (about 0.03mol/L): Weigh 11.82g of ferrous

ammonium sulfate $[(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$ and dissolve in sulfuric acid (5+95). Transfer into a 1000mL volumetric flask. Use sulfuric acid (5+95) to dilute to the scale. Mix well.

5 Instruments and equipment

5.1

Balance

The sensitivity is 0.1mg.

5.2

Geiger funnel

250mL.

6 Specimen

The specimen shall pass through the sieve mesh of 0.100mm.

7.1 Test material

Weigh about 0.15g of specimen (Chapter 6), accurate to 0.0001g.

Weigh two portions of test material for parallel determination. Take the average value as the result.

7.2 Blank test

Conduct the blank test, along with test material.

7.3 Determination

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