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

GB/T 12737-2008

Replacing GB/T 12737-1991

**Chemical products for industrial use -- General method for
determination of traces of sulphur compounds, as sulphate, by
reduction and titrimetry**

工业用化工产品中以硫酸根表示的痕量 硫化合物测定的通用方法 还原和滴定法
(English Translation)

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Foreword

This standard is equivalent to international standards ISO 6228. Determination of trace sulfur compounds 1980 (E) "industrial chemicals in sulfate represented Given the general method of reduction and titration. " this standard is the international standard ISO 6228. 1980 (E) on the same technical content, but contains the following editorial changes.

. "" Instead of using the decimal point --- "";

--- Use the "standard" instead of "this International Standard";

--- With "L" (L) in place of "l";

--- As clear operation, after 4.4 will be "the agent" plus "(also liquid)";

--- The reagent concentration percentage (m1/m1) to the mass fraction.

This standard replaces GB/T 12737-1991 "General method of trace chemical products Determination of Sulphate reduction titration." this standard compared with GB/T 12737-1991, the main changes are as follows.

--- Respect for the original ISO , also prepared using liquid hydrogen iodate by the mass fraction of more than 55% to about 57% concentrated hydrochloric acid change 38% (Excerpts 44, 1991, version 3.6.);

--- Remove the mercury standard titration solution of acetic acid formulas or mercury nitrate standard titration solution concentration (edition 44, 1991 Version 3.6.);

--- Removed Sulfate (SO₄ dollars) is calculated content (edition 44, 1991 Version 3.6.);

--- Adjust some interfering ions maximum permissible limit (Excerpts Appendix B, 1991 edition Appendix A);

--- Remove the pre-treatment method, formic acid, oxalic acid sample (Excerpts Appendix A, 1991 edition of Appendix B);

--- Remove the purification method hydroiodic (1991 edition Appendix C).

The Standard Appendix A, Appendix B and Appendix C are informative appendices.

The standard proposed by China Petroleum and Chemical Industry Association.

This standard by the National Standardization Technical Committee chemical inorganic chemicals Club (SAC/TC63/SC1) centralized.

Drafting of this standard. Shandong Entry-Exit Inspection and Quarantine Tianjin, Tianjin Chemical Engineering Research and Design Institute.

1 Scope

This standard specifies the general method of trace sulfur compounds of industrial chemical products to indicate sulfate reduction and measured --- titration.

Standards can be used in chemical products of the method of sample handling methods shall be defined so that the converted to sulfate sulfur compounds, if necessary General Procedure for any improvements that may be required.

2 Applications

This method is applicable to the determination of the solution, or in some cases directly measured in a sample sulfate (SO_2 -4) content in the range of $4.5\mu\text{g} \sim 450\mu\text{g}$. The test solution was measured and taken as the water content should not exceed 2mL. Elements in the Samples interference should not exceed Over the allowable limit, this limit according to the standard method of adopting the related chemical products made in regulations.

Sulfur compounds in the product should be in the form of sulphate, of other sulfur-containing compounds to the sample should be pretreated to obtain an appropriate Sulfate-containing test solution.

This method is expected precision of $\pm 5\%$.

NOTE: hydrochloric acid in place of the reducing solution, can be determined in the presence of sulfide (but non-polysulfide) other sulfur compounds.

Principle 3

If desired, the first change in a sample of sulfur compounds into sulfate, and then use the hydrogen iodide acid and phosphinic acid (hypophosphorous acid) in the presence of hydrochloric acid mixed the composition of sulfate ions reduced to hydrogen sulfide. A stream of nitrogen and hydrogen sulfide out of the absorption with acetone solution of sodium hydroxide. To 1,5 Phenyl-3-thioxo Kabbah hydrazone (dithizone) as an indicator, with mercuric acetate or nitrate mercury standard titration solution titration sulfur ions.

4 Reagents

Analysis using analytical grade reagents and only distilled water or water of appropriate purity.

4.1

acetone.

4.2

nitrogen, oxygen-free.

4.3

Sodium hydroxide. 40g/L solution.

4.4

reducing solution.

Equipped with a reflux condenser 1000mL spherical grinding mouth three-necked flask (Figure 1), in the case of nitrogen (4.2), and sequentially added.

--- 100mLrho (density) near 1.71g/mL, from about 57% by mass of hydroiodic acid solution;

--- 25mLrho (density) near 1.21g/mL, from about 50 wt.% Hypophosphorous acid (H_3PO_2) solution;

--- 100mLrho (density) near 1.19g/mL, about 38% of the mass fraction of hydrochloric acid solution.

Installed reflux condenser, nitrogen slowly (4.2) was blown into the mixture boiled under reflux for about 4h.

In keeping cool to room temperature under a nitrogen stream.

The reagent (reducing solution) is placed with a ground glass-stoppered dark glass bottles stored in the dark, glass bottles require advance with nitrogen (4.2) blowing original the air kept under a nitrogen atmosphere.

The solution is stable for a few weeks.

Note: This reagent (reducing solution) should be prepared in a fume hood to drain the release of hydrogen chloride.

GB/T 12737-2008/ISO 6228. 1980