

HG

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

HG/T 3811-2006

Bromides for industrial use - Test methods

工业溴化物试验方法

(English Translation)

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1 Scope

This standard specifies the test method of bromide for industrial use.

This standard is applicable to bromide for industrial use. This standard specifies the determination technology of product. When preparing the test solution, the standards related to the analyzed product shall be referenced to conduct the necessary revision to this standard, so as to make it more appropriate for the determination of product.

2 Normative references

The articles contained in the following documents have become part of this standard when they are quoted herein. For the dated documents so quoted, all subsequent modifications (including all corrections) or revisions made thereafter do not apply to this standard.

However, the parties who reach an agreement according to this standard are encouraged to study whether the latest versions of these documents may be used. For the undated documents so quoted, the latest versions (including all modification sheets) apply to this document.

GB/T 3049-1986 Chemical products for industrial use - General method for determination of iron content - 1,10-Phenanthroline spectrophotometric method [ISO 6685:1982(E) IDT]

GB/T 6682-1992 Water for analytical laboratory use - Specification and test methods (eqv ISO 3696:1987)

GB/T 9724-1988 Chemical reagent - General rules for the determination of pH

HG/T 3696.1 Inorganic chemical products - Preparations of standard volumetric solutions for chemical analysis

HG/T 3696.2 Inorganic chemical products - Preparations of standard solutions for chemical analysis

HG/T 3696.3 Inorganic chemical products - Preparations of reagent solutions for chemical analysis

3.1 Safety warning

Some reagents used in this test are poisonous or corrosive. Special attention must be paid during operation! If splashed to skin, wash with plenty of water. If the condition is severe, take the injured to hospital for professional medical treatment.

3.2 General stipulations

Unless otherwise specified, all the reagents used in this standard refer to analytic reagents; water is Grade-3 water as stipulated in GB/T 6682. Unless otherwise specified, all the standard titration solutions, standard impurity solutions, preparations and products used in the test shall be in accordance with HG/T 3696.1, HG/T 3696.2 or HG/T 3696.3 for preparation.

3.3.1.1 Method summary

Under the nitric acid condition, add the starch indicator; use 216-type silver electrode as the indicating electrode, 217-type double salt bridge saturated calomel electrode as the reference electrode; use the standard silver nitrate solution for titration.

3.3.1.2 Reagent

3.3.1.2.1

Nitric acid solution. 1+4.

3.3.1.2.2

Standard silver nitrate titration solution. $c(\text{AgNO}_3)$, about 0.1 mol/L.

3.3.1.2.3

Starch solution. 10g/L (prepared before use).

3.3.1.3 Instruments

3.3.1.3.1 216-type silver electrode.

3.3.1.3.2 217-type double salt bridge saturated calomel electrode.

3.3.1.4 Analytical procedure

Take 0.4g of sample, accurate to 0.01g, into a 250ml beaker. Add 100ml of water for dissolution; add 10ml of nitric acid and 10ml of starch indicator solution. Use 216-type silver electrode as the indicating electrode; use 217-type double salt bridge saturated calomel electrode as the reference electrode. The standard silver nitrate titration solution is used for

titration. At the early stage of titration, add a certain amount of standard silver nitrate titration solution. When approaching the end-point, gradually add 0.1ml per time (increased if necessary). Record the total volume and the corresponding potential E after each addition of silver nitrate. Calculate the difference $\Delta 2E$ between the continuously increased potentials, $\Delta 1E$ and $\Delta 1E$. The maximum value of $\Delta 1E$ is the titration end-point; after the end-point, continue to record an additional potential value E .

The data record format and the calculation of the volume of the consumed standard titration solution (V) when reaching the titration end-point are shown in annex A.

3.3.1.5 Result calculation

Bromide content is counted by the mass fraction w_1 , its value is expressed by %, calculated in accordance with formula (1). m - the mass of specimen, unit. g; M - the molar mass of bromide, unit. g/mol; K - the coefficient to convert chloride to bromide; ω_2 - the mass fraction of chloride, unit. %.

3.4 Determination of chloride content

3.4.1

Warning. When determining chloride, sample pre-treatment requires bromine evaporation. This product is toxic, it must be carried out in the well-ventilated fume cupboard.

3.4.2.1 Method summary

Use nitric acid to oxidize bromide ion to bromine; and heat it for evaporation. Under the nitric acid condition, add the starch indicator; use 216-type silver electrode as the indicating electrode; use 217-type double salt bridge saturated calomel electrode as the reference electrode. Use standard silver nitrate titration solution for titration.

3.4.2.2 Reagent

3.4.2.2.1

Nitric acid solution. 2 + 3.

3.4.2.2.2

Starch solution. 10g/L (prepared before use).

3.4.2.2.3

Standard silver nitrate titration solution. $c(\text{AgNO}_3)$, about 0.05 mol/L or 0.02 mol/L.

3.4.2.3 Instruments

3.4.2.3.1 216-type silver electrode (silver electrode vulcanized);

3.4.2.3.2 217-type double salt bridge saturated calomel electrode.

3.4.2.4 Analytical procedure

Take about 4g of sample, accurate to 0.01g, into in a 250ml beaker. Add 50ml of nitric acid solution; put it in the water bath (80°C) in the fume cupboard. Heat the beaker until the yellow color fades away. Use a small amount of water to wash the beaker wall; continue to heat the beaker for 15min; wait for cooling down; then, add water to 50ml in the beaker. Add 10ml of starch indicator solution; use 216-type silver electrode as the indicating electrode and 217-type double salt bridge saturated calomel electrode as the reference electrode; use standard silver nitrate titration solution for titration. At the early stage of titration, add a certain amount of standard silver nitrate titration solution. When approaching the end-point, gradually add 0.1ml per time (increased if necessary). Record the total volume after each addition of silver nitrate and the corresponding potential E.

Calculate the difference $\Delta 2E$ between the continuously increased potentials, $\Delta 1E$ and $\Delta 1E$.

The maximum value of $\Delta 1E$ is the titration end-point; record an additional potential value Use water to wash the wall of flask; heat it in water bath for 15min. After it is cooled, transfer it to a 250ml volumetric flask. Dilute it by adding water to the scale; shake it. Use the transfer pipette to transfer 10ml of solution into a 50ml colorimetric tube; dilute it by adding water to 25ml. Add 1ml of nitric acid and 1ml of silver nitrate solution; dilute it by adding water to 50ml; mix it evenly. The turbidity the solution generates shall not exceed that of the standard chloride turbidimetric solution.

Preparation of standard chloride turbidimetric solution. use the transfer pipette to transfer the standard chloride solution, that is prepared in accordance with the chloride content as stipulated in the product standard, into a 125ml conical flask. Handle the same procedure as the test solution.

3.5.1 Method summary

Under acid condition, add barium chloride and sulfate radical to generate sedimentation. Use the visual turbidimetry method for determination.

3.5.2 Reagent

3.5.2.1

Anhydrous ethanol.