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Methods of test for bromide for industrial use

工业溴化物试验方法

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Foreword

Please note that some contents in this standard may involve patents. The issuer of this standard does not undertake the responsibility to identify these patents.

Annex A of this standard is informative.

This standard was proposed by China Petroleum and Chemical Industry Federation.

This standard shall be under the jurisdiction of National Standardization Technical Committee of Chemical (SAC/TC63/SC1).

The main drafting organizations of this standard: Qingdao Entry-exit Inspection and Quarantine Bureau, Jiangsu World Chemical Co., Ltd., Shandong Weifang Longwei Industrial Co., Ltd., Tianjin Chemical Research & Design Institute.

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Methods of test for bromide for industrial use

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 Test methods

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 Determination of bromide content

3.3.1 Potentiometric titration (arbitration)

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: $c(AgNO_3)$, 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 Δ_2E between the continuously increased potentials, Δ_1E and Δ_1E. The maximum value of Δ_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 Potentiometric titration (arbitration method)

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: $c(AgNO_3)$, 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 Δ_2E between the continuously increased potentials, Δ_1E and Δ_1E. The maximum value of Δ_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 Determination of sulfate content

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.

3.5.2.2 Hydrochloric acid solution: 1 + 3.

3.5.2.3 Stannous chloride solution: 400g/L.

3.5.2.4 Barium chloride dihydrate solution: 100g/L.

3.5.2.5 Standard sulfate solution: 0.10mg of sulfate (SO_4) per 1ml of solution.

Take 10.00ml of standard sulfate solution prepared in accordance with HG/T 3696.2 into 100ml volumetric flask. Dilute it by adding water to the scale and shake the flask.

3.5.3 Analytical procedure

Take (1.00±0.01)g of specimen into a 50ml colorimetric tube; add 1ml of hydrochloric acid solution. Then add stannous chloride solution until the color of the solution fades away. Add 10ml of water; shake the tube. Add 5ml of anhydrous ethanol; shake the tube continuously; then add 3ml of barium chloride solution. Dilute it by adding water to the scale; shake it. Wait for 10min; the turbidity that it displays shall not be greater than that of standard turbidimetric solution.

Preparation of standard turbidimetric solution: use the transfer pipette to transfer the standard sulfate solution, that is prepared in accordance with the sulfate content as stipulated in the product standard at the specified amount, into a 50ml colorimetric tube. Add water to 10ml. Handle the same procedure for the test solution.

3.6 Determination of bromate content

Method summary: bromate oxidizes potassium iodide to iodine, under the acid condition.

Preparation of standard colorimetric solution: use the transfer pipette to transfer the standard iodide solution, that is prepared in accordance with the iodide content as stipulated in the product standard, into a 25ml colorimetric tube. Add water to 18ml. Handle the same procedure for the test solution.

3.8 Determination of iron content

3.8.1 Method summary

The same as article 2 in GB/T 3049-1986.

3.8.2 Reagent

The same as article 3 in GB/T 3049-1986.

3.8.3 Instrumentation

The same as article 4 in GB/T 3049-1986.

3.8.4 Analytical procedure

3.8.4.1 Drawing of working curve

The absorption cell at the thickness of 3cm and the corresponding standard iron solution shall be selected in accordance with article 5.3 in GB/T 3049-1986, so as to draw the working curve.

3.8.4.2 Preparation of test solution

Take 2g of specimen, accurate to 0.01g. Put it in the evaporating dish; add 2ml of sulfuric acid solution (1+1). Heat the evaporating dish slowly until sulfuric acid evaporates completely. Cool down; transfer it to a 100ml volumetric flask; dilute it by adding water to 60ml.

3.8.4.3 Preparation of blank test solution

Except for not adding specimen, other operational procedure is the same as article 3.8.4.2.

3.8.4.4 Determination

Take the test solution and blank test solution; the following operation shall start from and follow “use ammonia solution (3.2) or hydrochloric acid solution (3.1) to adjust pH value to 2” in article 5.4 of GB/T 3049-1986; use 3cm absorption cell for determination.

3.8.5 Result calculation

Iron content is counted by the mass fraction w_3 of iron (Fe), its value is expressed by %, calculated in accordance with formula (5):

$$w_3=\frac{(m_1-m_0)\times 10^{-6}}{m}\times 100 \quad \cdots\cdots (5)$$

Where,

m_1 — the mass of iron found out from the working curve based on the absorbance of test solution, unit: μg;

m_0 — the mass of iron found out from the working curve based on the absorbance of blank test solution, unit: μg;

m — the mass of specimen, unit: g

3.9 Determination of pH value

3.9.1 Apparatus

Acidimeter: accuracy is 0.02 pH in unit.

3.9.2 Analytical procedure

Take (5.0±0.1) g of specimen; use the water free of carbon dioxide; dilute it by adding water to 100ml; conduct the determination in accordance with the relevant stipulations given in article 6 of GB/T 9724-1988.

3.10 Determination of calcium, magnesium or other substance undissolved in ammonia solution

3.10.1 Method summary

In the specimen, oxalic acid reacts with the added ions such as calcium and magnesium, to generate sedimentation. Burn the sedimentation in the high temperature furnace; calculate the mass of substance that is not soluble in ammonia solution, based on the quantity of sedimentation.

3.10.2 Reagent

3.10.2.1 Ammonium oxalate solution: 40g/L.

3.10.2.2 Diammonium hydrogen phosphate solution: 100g/L.

3.10.2.3 Ammonia solution: 1 + 9.

3.10.2.4 Ammonia solution: 1+39.

3.10.3 Apparatuses

3.10.3.1 Porcelain crucible.

3.10.3.2 High temperature furnace: temperature can be controlled at (600±20)°C.

3.10.4 Analytical procedure

Take 10g of specimen, accurate to 1mg. Dissolve it in 75ml of water; add 5ml of ammonium oxalate solution and 2ml of diammonium hydrogen phosphate solution; then, Where:

m_1 — the mass of specimen after being dried, unit: g;

m — the mass of specimen, unit: g.

3.12 Determination of heavy metal content

3.12.1 Method summary

Under acid condition, sulfide reacts with heavy metal ion to produce colored sulfide. Compare it with the standard colorimetric solution.

3.12.2 Reagent

3.12.2.1 Acetic acid solution: 1+2.

3.12.2.2 Saturated hydrogen sulfide water (prepared before use).

3.12.2.3 Lead standard solution: 0.01 mg of lead (Pb) per 1ml of solution.

Take 1.00ml of lead standard solution, that is prepared in accordance with HG/T 3696.2, into a 100ml volumetric flask; dilute the lead standard solution with water; shake the flask gently.

3.12.3 Analytical procedure

Take (6.00±0.01)g of specimen into a 50ml colorimetric tube. Add 20ml of water for dissolution. Add 0.2ml of acetic acid solution. Add 10ml of hydrogen sulfide water; dilute it by adding water to 50ml; stand for 10min. The color of the solution shall not be darker than that of the standard solution.

Preparation of standard turbidimetric solution: According to the lead content as stipulated in the product standard, use the transfer pipette to transfer a specified amount of lead standard solution; place it into a 50ml colorimetric tube. Add 1.0g of specimen and 20ml of water. Handle it in the same-way same-time as the test solution.

------ The End ------