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Determination of Inorganic Sulfites and Bisulfites in Cosmetics

化妆品中限用物质无机亚硫酸盐类和亚硫酸氢盐类的测定

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Determination of Inorganic Sulfites and Bisulfites in Cosmetics

1 Scope

This Standard specifies the reagents and materials, instruments and apparatuses, analysis steps, result calculation, recovery and precision, allowable difference and the like of titration and ion chromatography of the determination of sulfites and bisulfites in cosmetics.

Note: The user selects titration or ion chromatography according to the configuration of the instrument. If the results are inconsistent, the ion chromatography method will prevail.

This Standard applies to the determination of sulfites and bisulfites in cosmetics.

For the contents of sulfites and bisulfites, the quantitation-limit of titration in this Standard is 50 mg/kg; the detection-limit of ion chromatography is 2 mg/kg, and the quantitation-limit is 10 mg/kg.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the dated version applies to this document. For undated references, the latest edition (including all amendments) applies to this document.

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

3 Titration

3.1 Principle

Under the heating condition, the free sulfur dioxide in the sample reacts excessively with hydrogen peroxide to form sulfuric acid; then, use alkali standard solution to titrate the resulting sulfuric acid. Thus, the content of free sulfur dioxide in the sample can be obtained.

3.2 Reagents and materials

Unless otherwise specified, the used reagents are analytical reagents; the water is that in accordance with 3.2.1.

3.4.3 Titration

Take down the pear-shaped flask; use a small amount of water to wash the upper end and let it flow into the pear-shaped flask; use sodium hydroxide standard solution (3.2.8) to titrate the solution in the pear-shaped flask to an olive color; record the volume of the consumed sodium hydroxide standard solution; at the same time, do a blank test.

3.4.4 Blank test

Except that the sample is not weighed, it is carried out according to the above steps of distillation and titration.

3.4.5 Parallel test

The contents of sulfites and bisulfites in the sample shall be determined from the average of two independent parallel test results.

3.5 Result calculation

The contents of inorganic sulfites and bisulfites in the sample (calculated as sulfur dioxide) is calculated according to Formula (1):

$$X=\frac{c\times(V-V_0)\times 32}{m}\times 1\,000 \quad \cdots\cdots\cdots\cdots (1)$$

Where:

X -- the content of inorganic sulfites and bisulfites in the sample (calculated as sulfur dioxide), in milligrams per kilogram (mg/kg);

c -- the mass concentration of the sodium hydroxide standard solution, in moles per liter (mol/L);

V -- the volume of sodium hydroxide standard solution that is consumed to determine the sample, in milliliters (mL);

V_0 -- the volume of sodium hydroxide standard solution that is consumed in the blank test, in milliliters (mL);

32 -- the mass of sulfur dioxide that is equivalent to 1.00 mL of sodium hydroxide standard solution [c(NaOH) = 1.00 mol/L], in milligrams.

m -- sampling amount, in grams (g);

The calculation result shall keep two significant figures.

3.6 Recovery and precision

4.2.8 Sodium sulfite grade-2 standard intermediate solution 5.00 mg/L (content based on sulfur dioxide): accurately transfer 2.50 mL of sodium sulfite grade-1 standard intermediate solution (4.2.7) to a 10 mL volumetric flask; use water to dilute to the mark; shake well. Store it at 0°C ~ 4°C out of the sun. Formulate when needed.

4.2.9 Sodium sulfite standard working solution: accurately transfer 0.20 mL, 0.50 mL, 1.00 mL, 2.00 mL, 3.00 mL and 4.00 mL of sodium sulfite grade-2 standard intermediate solution to a 10 mL volumetric flask respectively; use water to dilute to the mark; shake well. The concentrations are 0.10 mg/L, 0.25 mg/L, 0.50 mg/L, 1.00 mg/L, 1.50 mg/L and 2.00 mg/L respectively (content based on sulfur dioxide); prepare when needed.

4.3 Instruments and apparatuses

4.3.1 Ion chromatograph: equipped with a conductivity detector.

4.3.2 Analytical balance: sensitivity of 1 mg.

4.3.3 Analytical balance: sensitivity of 0.01 g.

4.3.4 Volumetric flasks: 10 mL and 100 mL.

4.3.5 Colorimetric tube with a stopper: 10 mL.

4.3.6 RP C_{18} ion chromatography solid-phase extraction column (1.0 mL), or a pre-treatment column with equivalent performance to remove organic matters. Use 5 mL of methanol (4.2.3) and 10 mL of water to activate before use; place for 30 min before use, or carry out activation with reference to the extraction column technical file.

4.3.7 Centrifuge: (speed ≥ 8 000 r/min).

4.3.8 Filter: pore size of 0.22 μm.

4.3.9 Vortex oscillator.

4.3.10 Pipette or pipettor: 200 μL, 1 mL, and 5 mL.

4.4 Analysis steps

4.4.1 Preparation of sample solution

Weigh 2 g (accurate to 0.01 g) of sample; place it in a 10 mL colorimetric tube with a stopper; use acetonitrile (4.2.2) to dilute to the mark of 10 mL; vortex for 2 min; centrifuge at 8 000 r/min for 5 min.

Accurately transfer 0.50 mL of the supernatant to another 10 mL colorimetric tube with a stopper; use water to dilute to the mark of 10 mL; mix well.

Determine the sample solution; obtain the concentration (mg/L) of sulfites and bisulfites in the measurement solution from the standard curve.

4.4.6 Blank test

Except that the sample is not weighed, it is carried out according to the above steps.

4.4.7 Parallel test

The contents of sulfites and bisulfites in the sample shall be determined from the average of two independent parallel test results.

4.5 Result calculation

The contents of sulfites and bisulfites in the sample (calculated as sulfur dioxide) is calculated according to Formula (2):

$$X = \frac{(c - c_0) \times V_1 \times V_3 \times 1\,000}{m \times V_2 \times 1\,000} \quad \cdots\cdots (2)$$

Where:

X -- the contents of sulfites and bisulfites in the sample (calculated as sulfur dioxide), in milligrams per kilogram (mg/kg);

c -- the measured value of sodium sulfite in the sample solution (calculated as sulfur dioxide), in milligrams per liter (mg/L);

c_0 -- the measured value of sodium sulfite in the sample blank solution (calculated as sulfur dioxide), in milligrams per liter (mg/L);

V_1 -- the fix-volume of the sample when it is extracted with acetonitrile; in milliliters (mL);

V_3 -- the fix-volume of the extract when it is diluted with water; in milliliters (mL);

m -- sample mass, in grams (g).

V_2 -- the fix-volume of the pipetted extract when it is diluted with water; in milliliters (mL);

1 000 -- unit conversion coefficient.

The calculation result shall keep two significant figures.

4.6 Recovery and precision

For sulfites and bisulfites (calculated as sulfur dioxide) in the range of 10.0

Appendix A

(Informative)

Calibration method of sodium sulfite standard solution

A.1 Principle

In a weakly acidic solution, add an excess of iodine oxide sulfite. Take the starch as the indicator; use the sodium thiosulfate solution to titrate the excess iodine.

A.2 Reagents and materials

Unless otherwise indicated, the reagents are all analytical reagent, and the water is distilled water or water of comparable purity.

A.2.1 Iodine standard solution [$c(1/2I_2) = 0.1$ mol/L].

A.2.2 Sodium thiosulfate standard titration solution [$c(Na_2S_2O_3) = 0.1$ mol/L].

A.2.3 Glacial acetic acid (CH_3COOH).

A.2.4 Starch indicator solution (10 g/L): weigh 1 g of soluble starch; use a little water to mix and make it a paste; slowly pour in 100 mL of boiling water while stirring; boil for 2 min; let cool for use; prepare when needed.

A.3 Instrument

A.3.1 Pipette.

A.3.2 Iodine flask.

A.3.3 Burette.

A.4 Analysis steps

Pipette 10 mL of sodium sulfite standard solution into a 250 mL iodine flask; add 100 mL of water; accurately add 20 mL of 0.1mol/L iodine standard solution, 5 mL of glacial acetic acid; shake well; place in the dark; after 2 minutes, quickly use the standard solution of sodium thiosulfate 0.1mol/L to titrate to pale yellow; add 0.50 mL of starch indicator; continue to titrate until it is colorless; do a blank test at the same time.

A.5 Result calculation

Calculate the concentration of sodium sulfite standard solution according to formula (A.1):

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