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Replacing GB/T 23978-2009

Determination of chloride in water-soluble dyes

水溶性染料产品中氯化物的测定

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Table of Contents

Determination of chloride in water-soluble dyes

1 Scope

This Standard specifies the determination method of chloride in water-soluble dyes.

This Standard applies to various water-soluble dyes.

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 601, Chemical reagent - Preparations of reference titration solutions

GB/T 603, Chemical reagent - Preparations of reagent solutions for use in test methods

GB/T 6682-2008, Water for analytical laboratory use - Specification and test methods (ISO 3696:1987, MOD)

GB/T 8170-2008, Rules of rounding off for numerical values & expression and judgement of limiting values

3 Determination methods

3.1 Ion chromatography

3.1.1 Determination principle

For the pre-treated water-soluble dye sample solution, use the ion chromatography, with retention time for qualitative determination and peak area external standard method for quantitative determination, to determine its chloride content (as NaCl).

3.1.2 General provisions

Unless otherwise specified, use only reagents that are identified as analytical reagents, and grade-3 water that is specified in GB/T 6682-2008. The

Burn the sodium chloride at 500 °C to a constant amount; then, weigh about 1.000 g (accurate to 0.000 1 g) in a 1 000 mL volumetric flask; add water to dissolve and dilute to a constant volume; prepare a sodium chloride solution whose concentration is 1 000 μg/mL. Then, prepare linear standard solutions of seven concentrations of 0.01 μg/mL, 0.05 μg/mL, 0.1 μg/mL, 0.5 μg/mL, 1.0 μg/mL, 5.0 μg/mL, and 10.0 μg/mL for use.

3.1.8 Determination

After the instrument is running stably, use a syringe to respectively draw the standard solution and sample solution to fill the quantitative loop for injection. After the chloride ion peak has flowed out (see Figure A.1 in Appendix A), use the chromatographic workstation to process the results. Determine the peak area of sodium chloride in the standard solution; draw a standard curve; then, calculate the chloride content in the sample according to the standard curve.

3.1.9 Result calculation

The standard curve equation of sodium chloride is calculated according to Formula (1):

$$A = k\rho - b \qquad \cdots\cdots(1)$$

The content of chloride (calculated as NaCl) in the sample is calculated, as mass fraction w_1 (%), according to Formula (2):

$$w_1 = \frac{\rho \times V_1 \times n}{m_1 \times 10^6} \times 100\% \qquad \cdots\cdots(2)$$

Where:

A -- peak area value of sodium chloride;

k -- constant of the sodium chloride standard curve equation, which is given by the chromatographic workstation;

ρ -- concentration of sodium chloride in the solution, in micrograms per milliliter (μg/mL);

b -- constant of the sodium chloride standard curve equation, which is given by the chromatographic workstation;

V_1 -- value of the final constant volume of the sample solution, in milliliters (mL);

n -- value of the dilution ratio of the sample;

m_1 -- value of the mass of the sample, in grams (g);

3.2.3.2 Nitric acid.

3.2.3.3 Nitric acid solution: the volume ratio of nitric acid to water = 1:1.

3.2.3.4 Nitrobenzene.

3.2.3.5 Silver nitrate standard titration solution: $c(AgNO_3) = 0.1$ mol/L.

3.2.3.6 Ferric ammonium sulfate test solution: Weigh 14 g of ferric ammonium sulfate; dissolve it in 100 mL of water; filter; add 10 mL of nitric acid; store in a brown bottle; place it in a refrigerator. The validity period is 6 months.

3.2.3.7 Ammonium thiocyanate standard titration solution: $c(NH_4SCN) = 0.1$ mol/L.

3.2.4 Preparation of sample solution

Weigh about 2 g (accurate to 0.000 1 g) of the sample; dissolve it in 150 mL of water; add 15 g of activated carbon; gently boil for 2 min ~ 3 min. Cool to room temperature; add 1 mL of nitric acid solution; place for 30 min (shake it occasionally). Use a Buchner funnel for suction filtration. If the filtrate is colored, add another 5 g of activated carbon; place for 1 h while shaking it from time to time; then, use a Buchner funnel for suction filtration (if there is still color, replace the activated carbon and repeat the operation until the filtrate is colorless). Use 10 mL of water to wash the activated carbon 3 times; combine and transfer the filtrate to a 250 mL volumetric flask; use water to fix volume; shake well; set aside.

3.2.5 Determination steps

Pipette 50.00 mL of the sample solution (3.2.4); place it in a 250 mL conical flask; add 2 mL of nitric acid solution (3.2.3.3), 10.00 mL of silver nitrate standard titration solution (3.2.3.5) and 5 mL of nitrobenzene; vigorously shake until the silver chloride precipitates and coagulates; add 1 mL of ferric ammonium sulfate test solution; use the ammonium thiocyanate standard titration solution to titrate the excess silver nitrate; the solution changes from light yellow to light red as the end point; keep it for 1 min. Do a blank test at the same time.

Note: If the chloride content is high, and the chloride ion in the sample cannot be completely precipitated with 10.00 mL of silver nitrate standard titration solution, dilute the sample solution.

3.2.6 Result calculation

The content of chloride (calculated as NaCl) in the sample is calculated, as mass fraction w_2 (%), according to Formula (3):

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