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Determination of ammonium in methanol for industrial use - Ion chromatography

工业用甲醇中铵离子的测定 离子色谱法

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Determination of ammonium in methanol for industrial use - Ion chromatography

Warning – This document is not intended to explain all safety issues related to its use; the user is responsible for taking appropriate safety and health measures, which shall also comply with the relevant national regulations.

1 Scope

This document specifies the method for the determination of free ammonia and ammonium ions in methanol for industrial use by ion chromatography.

This document applies to the determination of free ammonia and ammonium ions in methanol for industrial use. When the injection volume is 50 μL, the lower limit of determination is 0.01 mg/L.

2 Normative references

The following documents are normatively referenced in this document and are indispensable for its application. For dated references, only the version corresponding to that date is applicable to this document; for undated references, the latest version (including all amendments) is applicable to this document.

GB/T 3723, Sampling of chemical products for industrial use - Safety in sampling

GB/T 6680, General rules for sampling liquid chemical products

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

GB/T 33087, Ultra pure water for instrumental analysis specification and test methods

GB/T 34672, Chemical reagent - General rules for the ion chromatography

3 Terms and definitions

No terms and definitions need to be defined in this document.

4 Method summary

When the samples enter the ion chromatography column with the eluent for exchange separation, use a conductivity detector to measure the ammonium ion content. Use the retention time to determine quality, and the working curve method to determine quantity.

5 Reagents and materials

Unless otherwise stated, use only guaranteed reagents in the analysis.

5.1 Water: in accordance with GB/T 33087.

5.2 Methanol: chromatographic pure, ammonium content less than 0.01 mg/L.

Note: Preparation method of ammonium-free methanol reagent – take 2 L of methanol; add 15 mL of 1.0 mol/L sulfuric acid solution; distill; discard 100 mL of distillate before and after; take the middle distillate as ammonium-free methanol reagent.

5.3 Nitrogen: volume fraction not less than 99.999%.

5.4 Ammonium ion standard substance: 1 000 mg/L, certified reference material.

5.5 Ammonium ion standard working solution: 50.00 mg/L.

Pipette 5.00 mL of ammonium ion standard substance (see 5.4) to a 100 mL volumetric flask; use water (see 5.1) to dilute to a constant volume.

5.6 Methanol solution: volume fraction 5%.

5.7 Methane sulfonic acid eluent stock solution: 1.0 mol/L.

5.8 Methane sulfonic acid eluent working solution: 2.0 mmol/L.

Pipette 4.00 mL of methane sulfonic acid eluent stock solution (see 5.7) into a 2 000 mL volumetric flask; use water (see 5.1) to dilute to a constant volume. Prepare before use.

Note: The eluent is prepared according to the working conditions of different chromatographic columns. The preparation method of the eluent given above is for reference only.

8.1.1 Set the instrument parameters with reference to Table A.1 – Reference ion chromatogram for determination of ammonium ions in methanol for industrial use by isocratic elution – in Appendix A.

8.1.2 Respectively pipette 0 mL, 0.10 mL, 0.20 mL, 0.40 mL, 1.00 mL, 2.00 mL, 4.00 mL of ammonium ion standard working solution (see 5.5) into 100 mL volumetric flasks; use methanol solution (see 5.6) to dilute and fix volume in turn, to obtain the ammonium ion series standard samples; measure through the sample introduction system. Refer to Figure B.1 in Appendix B for the reference ion chromatogram.

Note: When the sample contains calcium and magnesium ions, set the instrument parameters with reference to Table A.1 – Reference ion chromatographic conditions for determination of ammonium ions in methanol for industrial use – in Appendix A. For the reference ion chromatogram, see Figure B.2 in Appendix B.

8.1.3 Take the ammonium ion mass concentration as the abscissa and the corresponding peak area after deducting the blank as the ordinate, to draw a standard working curve, as shown in Formula (1).

$$A_i = a \times \rho_i^2 + b \times \rho_i + c \qquad \cdots\cdots (1)$$

Where:

A_i – peak area of ammonium ion in the standard solution i, in micro Siemens minute (μS•min);

ρ_i – mass concentration of ammonium ions in the standard solution i, in milligrams per liter (mg/L);

a – quadratic coefficient of the regression equation;

b – linear coefficient of the regression equation;

c – constant term of the regression equation.

8.2 Sample determination

8.2.1 Sample pretreatment

8.2.1.1 According to the column efficiency of the ion chromatography and the pressure resistance of the system, use water (see 5.1) to dilute the methanol sample to a methanol concentration of not more than 10% by volume; shake well.

8.2.1.2 Use a syringe (see 6.9) to pipette a certain amount of methanol sample (see 8.2.1.1); filter it through a syringe micromembrane filter (see 6.8); then, pass it through an organic matter pretreatment column (see 6.4) before testing.

8.2.2 Sample determination

Under the same chromatographic working conditions as drawing the standard working curve (see 8.1.1), inject the methanol sample solution (see 8.2.1.2) into the sample introduction system for determination. Measure twice in parallel.

9 Result calculation

9.1 Calculation of ammonium ion mass concentration in the sample

Calculate the mass concentration ρ_s of ammonium ions in the sample according to Formula (2), and the parameters a, b and c by Formula (1):

$$\rho_s = f\left(\frac{-b+\sqrt{b^2-4\times a\times(c-A_s)}}{2a}\right) \quad \cdots\cdots(2)$$

Where:

ρ_s – mass concentration of ammonium ions in methanol, in milligrams per liter (mg/L);

A_s – peak area of ammonium ion in methanol, in micro Siemens minute (μS•min);

f – sample dilution factor.

9.2 Result expression

For any sample, use the arithmetic mean of the results of two parallel determinations to represent the analysis result. The rounding off of the value shall be in accordance with GB/T 8170; keep the result to two decimal places.

10 Precision

10.1 Repeatability limit

In the same laboratory, the same operator uses the same equipment to test the same sample according to the same method, and the difference between the two test results does not exceed the repeatability limit (r), as shown in Table 1.

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