

Method of Test for Phosphoric Acid in Beverage

1. Scope

This method is applicable to the determination of phosphoric acid in cola beverage, and tea beverages.

2. Method

After dilution, phosphoric acid is determined by high performance ion chromatograph (HPIC).

2.1. Equipment

2.1.1. High performance ion chromatograph.

2.1.1.1. Detector: conductivity detector.

2.1.1.2. Column: IonPac® AS11-HC, 9 µm, 4 mm i.d. × 25 cm, or an equivalent product.

2.1.1.3. Guard column: IonPac® AG11-HC, 9 µm, 4 mm i.d. × 5 cm, or an equivalent product.

2.1.1.4. Anion Self-Regenerating Suppressor: AERS 500, 4 mm, or an equivalent product.

2.1.2. Ultrasonicator.

2.2. Chemicals

50% Sodium hydroxide solution, ion chromatograph grade;

Deionized water, resistivity $\geq 18 \text{ M}\Omega \cdot \text{cm}$ at 25°C;

85% Ortho phosphoric acid, reference standard.

2.3. Apparatus

2.3.1. Volumetric flask: 10 mL and 100 mL.

2.3.2. Membrane filter: 0.45 µm, Nylon.

2.4. Eluent

2.4.1. Solution A: deionized water.

2.4.2. Solution B:

Dilute 10.5 mL of 50% sodium hydroxide solution with 2000 mL of deionized water to 2000 mL. Filter with a membrane filter.

2.5. Standard solution preparation

Accurately weigh equivalent to 0.1 g of phosphoric acid reference standard and dilute with deionized water to 100 mL as the standard stock solution. Store in a refrigerator. When to use, dilute appropriate volume of the standard stock solution with deionized water to 1 - 15 µg/mL as the standard solutions.

2.6. Sample solution preparation

Sample containing carbon dioxide should remove carbon dioxide before sample solution preparation. Transfer about 5 g of the sample accurately weighed into a 100 mL-volumetric flask and dilute with deionized water to volume. Filter with a membrane filter. Take the filtrate as the sample solution.

2.7. Identification and quantification

Accurately inject 25 µL of the sample solution and the standard solutions into HPIC separately and operate according to the following conditions. Identify phosphoric acid based on the retention time. Calculate the amount of phosphoric acid in the sample by the following formula:

$$\text{The amount of phosphoric acid in the sample (g/kg)} = \frac{C \times V}{M \times 1000}$$

Where,

C: the concentration of phosphoric acid in the sample solution calculated by the standard curve (µg/mL)

V: the final make-up volume of the sample (mL)

M: the weight of sample (g)

HPIC operating conditions ^(note)

Detector: conductivity detector.

Column: IonPac[®] AS11-HC, 9 µm, 4 mm i.d. × 25 cm.

Guard column: IonPac[®] AG11-HC, 9 µm, 4 mm i.d. × 5 cm.

Anion Self-Regenerating Suppressor: AERS 500, 4 mm.

Column temperature: 30°C.

Injection volume: 25 µL.

Eluent : a gradient program of solvent A and solvent B is as follows.

Time(min)	A (%)	B (%)
0 → 13	65 → 65	35 → 35
13 → 14	65 → 20	35 → 80
14 → 19	20 → 20	80 → 80
19 → 20	20 → 65	80 → 35
20 → 25	65 → 65	35 → 35

Flow rate: 1.0 mL/min.

Note: All the parameters can be adjusted depending on the instruments used if the above conditions are not applicable.

Remark

1. Limit of quantitation (LOQ) for phosphoric acid is 0.02 g/kg.

2. Further validation should be performed when interference compounds appear in samples.
3. Phosphoric acid naturally exists in some foods; thus, it is not possible to determine whether it meets the regulations with only the test results. To conduct a comprehensive judgment, source of raw materials, processing procedures, inspection results and other relevant information should be considered.

References

1. Ruey-Juh Lin, Bin-Chiou Chang, Shin-Shou Chou. 1991. Ionic Chromatographic Studies on Phosphoric Acid in Beverage Produced in Taiwan. Drug and Food Inspection Agency Annual Report on Investigation and Research. 9: 483-490.
2. De Borba, B and Rohrer, J. 2018. Rapid determination of phosphate and citrate in carbonated soft drinks using a reagent-free ion chromatography system. Thermo Fisher Scientific Application Note169.
[<https://tools.thermofisher.com/content/sfs/brochures/AN-169-ICPhosphate-Citrate-Soft-Drinks-AN71409-EN.pdf>].