⟨345⟩ ASSAY FOR CITRIC ACID/CITRATE AND PHOSPHATE

INTRODUCTION

The following ion chromatographic general procedure is provided for the determination of citric acid/citrate and phosphate in compendial articles, when specified in the individual monographs. See *Ion Chromatography* ⟨1065⟩ for discussion of the theory and principles of measurements using ion chromatography.

ASSAY

• **PROCEDURE**

Mobile phase: 20 mM sodium hydroxide or potassium hydroxide from an appropriate volume of carbonate-free sodium hydroxide or potassium hydroxide solution of known concentration and water (resistivity NLT 18 megohm-cm). Alternatively, *Mobile phase* can be generated electrolytically using an automatic eluent generator. Protect the *Mobile phase* from atmospheric carbon dioxide.

Standard solution 1 (for the assay of citric acid/citrate only): 20 µg/mL of citrate ($C_6H_5O_7$) in freshly prepared 1 mM sodium hydroxide from USP Citric Acid RS

Standard solution 2 (for the concomitant assay of citrate and phosphate): 20 µg/mL of citrate ($C_6H_5O_7$) and 12 µg/mL of phosphate (PO_4) in freshly prepared 1 mM sodium hydroxide from USP Citric Acid RS and monobasic sodium phosphate

Sample solution (for the assay of citric acid/citrate): Nominally 20 µg/mL of citrate in freshly prepared 1 mM sodium hydroxide, unless otherwise stated in the monograph

Sample solution (for the assay of phosphate): Nominally 12 µg/mL of phosphate in freshly prepared 1 mM sodium hydroxide, unless otherwise stated in the monograph

Chromatographic system

(See *Chromatography* ⟨621⟩, *System Suitability*.)

Mode: LC

Detector: Conductivity with suppression

Columns

Analytical: 4-mm × 25-cm; 13-µm packing L61

Guard: 4-mm × 5-cm; 13-µm packing L61

Temperatures

Column: 30°

Detector: 35°

Suppressor: 4-mm membrane anionic autosuppressor or a suitable chemical suppression system

Flow rate: 2 mL/min

Injection volume: 10 µL

System suitability

Samples: *Standard solution 1* and/or *Standard solution 2*, as appropriate

[NOTE—The relative retention times for phosphate and citrate are 0.57 and 1.0, respectively.]

Suitability requirements

Tailing factor: NMT 2.0 for the citrate and/or phosphate peaks, as appropriate

Relative standard deviation: NMT 1.5% for six replicate injections for the citrate and/or phosphate peaks, as appropriate

Analysis

Samples: *Standard solution 1* and/or *Standard solution 2*, and *Sample solution*

Unless otherwise stated in the monograph, calculate the concentration of citrate or phosphate in the portion of the *Sample solution* taken:

$$\text{Result} = (r_U / r_S) \times C_S$$

r_U = peak response of the citrate or phosphate peak from the *Sample solution*

r_S = peak response of the citrate or phosphate peak from *Standard solution 1* or *Standard solution 2*

C_S = concentration of citrate or phosphate from *Standard solution 1* or *Standard solution 2* (µg/mL)

USP Reference Standards ⟨11⟩

USP Citric Acid RS