IMPURITIES

• **Residue on Ignition ⟨281⟩:** NMT 0.1%

Delete the following:

•• **Heavy Metals,** *Method I* ⟨231⟩

Sample solution: Dissolve 1 g in 23 mL of water, and add 2 mL of 1 N acetic acid.

Acceptance criteria: NMT 20 ppm • (Official 1-Jan-2018)

• **Limit of Hydroxylamine**

Buffer: 1.36 g/L of monobasic potassium phosphate in water, adjusted with 1 M potassium hydroxide to a pH of 7.4

Solution A: 1 mg/mL of pyridoxal 5-phosphate monohydrate in *Buffer*, prepared in a low-actinic flask fresh before use

Standard stock solution: 2.0 mg/mL of hydroxylamine hydrochloride in water

Standard solutions: Transfer 5.0, 10.0, and 15.0 mL of the *Standard stock solution* to separate 100-mL volumetric flasks, and dilute with water to volume.

Sample solution: Transfer 1500 mg of Acetohydroxamic Acid, previously dried, to a 100-mL beaker, and dissolve in a sufficient amount of water to cover the electrode of a calibrated pH meter (about 60 mL). While stirring, adjust with 0.05 M potassium hydroxide to a pH of 7.4. Transfer the contents of the beaker, with the aid of small portions of water, to a 100-mL volumetric flask, and dilute with water to volume.

Blank: Water

Analysis

Samples: *Standard solutions*, *Sample solution*, and *Blank*

Transfer 2.0 mL of each *Standard solution*, the *Sample solution*, and *Blank* into separate 100-mL volumetric flasks. To each flask add 4.0 mL of *Solution A*. After 8 min, accurately timed, dilute the contents of each flask with *Buffer* to volume.

Immediately determine the fluorescence intensities of the solutions from the *Standard solutions* and the *Sample solution* in a fluorometer at an excitation wavelength of 350 nm and an emission wavelength of 450 nm, setting the instrument to zero with the *Blank*. Determine the best-fit straight line from the fluorescence intensities of the three *Standard solutions* versus the hydroxylamine hydrochloride concentrations, in µg/mL. From the best-fit straight line, determine the concentration, in µg/mL, of hydroxylamine hydrochloride in the *Sample solution*.

Calculate the percentage of hydroxylamine in the portion of Acetohydroxamic Acid taken:

$$\text{Result} = (C_U/C) \times (M_{r1}/M_{r2}) \times 100$$

C_U = concentration of hydroxylamine hydrochloride in the *Sample solution* (mg/mL)
C = concentration of Acetohydroxamic Acid in the *Sample solution* (mg/mL)
M_{r1} = molecular weight of hydroxylamine, 33.03
M_{r2} = molecular weight of hydroxylamine hydrochloride, 69.50

Acceptance criteria: NMT 0.5%

SPECIFIC TESTS

• **Loss on Drying ⟨731⟩**

Analysis: Dry a sample over phosphorus pentoxide for 16 h.

Acceptance criteria: NMT 1.0%

• **Completeness of Solution ⟨641⟩:** A 1.0-g portion dissolves in 10 mL of water to yield a clear solution.

• **Color of Solution**

Sample solution: 200 mg/mL in water

Blank: Water

Instrumental conditions

(See *Ultraviolet-Visible Spectroscopy* ⟨857⟩.)

Mode: UV-Vis

Analytical wavelength: Between 400 and 750 nm

Cell: 1 cm

Acceptance criteria: The absorbance is NMT 0.050.

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers, and store in a cool, dry place.

• **USP Reference Standards ⟨11⟩**

USP Acetohydroxamic Acid RS

Acetohydroxamic Acid Tablets

DEFINITION

Acetohydroxamic Acid Tablets contain NLT 90.0% and NMT 110.0% of the labeled amount of acetohydroxamic acid ($C_2H_5NO_2$).

IDENTIFICATION

• **A.** Tablets produce a purple color when mixed with an acidic solution of ferric chloride.

ASSAY

• **Procedure**

Ferric chloride solution: 20 mg/mL of ferric chloride in 0.1 N hydrochloric acid

Standard solution: 500 µg/mL of USP Acetohydroxamic Acid RS in 0.1 N hydrochloric acid

Sample solution: Weigh, and finely powder NLT 20 Tablets. Transfer an accurately weighed portion of the powder, equivalent to about 500 mg of acetohydroxamic acid, to a 1000-mL volumetric flask, add about 500 mL of 0.1 N hydrochloric acid, and shake for 1 min. Dilute with 0.1 N hydrochloric acid to volume, and mix. Filter, discarding the first 40 mL of the filtrate. Use the clear filtrate.

Blank: 0.1 N hydrochloric acid

Analysis

Samples: *Standard solutions*, *Sample solution*, and *Blank*

Transfer 10.0 mL each of the *Standard solution*, *Sample solution*, and *Blank* to separate 100-mL volumetric flasks. To each flask add 50 mL of 0.1 N hydrochloric acid and 10.0 mL of *Ferric chloride solution*, and dilute with 0.1 N hydrochloric acid to volume. Without delay, concomitantly determine the absorbances of the solutions at the wavelength of maximum absorbance at about 502 nm, using the *Blank* to set the instrument.

Calculate the percentage of labeled amount of acetohydroxamic acid ($C_2H_5NO_2$) in the portion of Tablets taken:

$$\text{Result} = (A_U/A_S) \times (C_S/C_U) \times 100$$

A_U = absorbance of the *Sample solution*
A_S = absorbance of the *Standard solution*
C_S = concentration of USP Acetohydroxamic Acid RS in the *Standard solution* (µg/mL)
C_U = nominal concentration of acetohydroxamic acid in the *Sample solution* (µg/mL)

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

• **Dissolution,** *Procedure for a Pooled Sample* ⟨711⟩

Medium: 0.01 N hydrochloric acid; 900 mL

Apparatus 1: 100 rpm

Time: 30 min

Analysis: Calculate the percentage of the labeled amount of acetohydroxamic acid ($C_2H_5NO_2$) dissolved, using the procedure in the *Assay*, using a filtered portion of the solution under test as *Sample solution* in comparison with a *Standard solution* having a known