Standard solutions: Transfer 1.0, 2.0, 3.0, 4.0, and 5.0 mL of *Standard stock solution B* to separate 100-mL volumetric flasks. Dilute the contents of each flask with 0.125 N hydrochloric acid to volume to obtain concentrations of 0.5, 1.0, 1.5, 2.0, and 2.5 µg/mL of zinc.

Sample solution: Finely powder NLT 20 tablets. Transfer an equivalent to 5 tablets to a porcelain crucible. Heat the crucible in a muffle furnace maintained at 550° for 6–12 h, and cool. Add 60 mL of hydrochloric acid, and boil gently on a hot plate or steam bath for 30 min, intermittently rinsing the inner surface of the crucible with 6 N hydrochloric acid. Cool, and quantitatively transfer the contents of the crucible to a 100-mL volumetric flask. Rinse the crucible with small portions of 6 N hydrochloric acid, and add the rinsings to the flask. Dilute with water to volume, and filter, discarding the first 5 mL of the filtrate. Dilute this solution quantitatively, with 0.125 N hydrochloric acid to obtain a nominal concentration of 2 µg/mL of zinc.

Instrumental conditions

(See *Spectrophotometry and Light-Scattering* ⟨851⟩.)

Mode: Atomic absorption spectrophotometry

Analytical wavelength: 213.8 nm

Lamp: Zinc hollow-cathode

Flame: Air–acetylene

Blank: 0.125 N hydrochloric acid

Analysis

Samples: *Standard solutions* and *Sample solution*

Determine the absorbances of the solutions against the *Blank.* Plot the absorbances of the *Standard solutions* versus the concentration, in µg/mL, of zinc, and draw the straight line best fitting the five plotted points. From the graph so obtained, determine the concentration, *C*, in µg/mL, of zinc in the *Sample solution*.

Calculate the percentage of the labeled amount of zinc (Zn) in the portion of Tablets taken:

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

C = determined concentration of zinc in the *Sample solution* (µg/mL)

C_U = nominal concentration of zinc in the *Sample solution* (µg/mL)

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

- **DISINTEGRATION AND DISSOLUTION ⟨2040⟩**

Medium: Water; 900 mL

Apparatus 2: 75 rpm

Time: 60 min

Analysis: Proceed as directed in *Method 1* or *Method 2* for *Strength*, making any necessary volumetric adjustments.

Sample solution: If *Method 1* is used, pipet 10.0 mL of the filtered pooled solution under test to a 50-mL volumetric flask, and dilute with 2% nitric acid solution to 50 mL. If *Method 2* is used, dilute the filtered pooled solution under test with 0.125 N hydrochloric acid to a concentration falling within the range of the *Standard solutions*.

Calculate the percentage of the labeled amount of zinc (Zn) dissolved:

$$\text{Result} = C \times (V_M/a) \times (D/L) \times 100$$

C = concentration of zinc in the *Sample solution* (mg/L)

V_M = volume of *Medium*, 900 mL

a = aliquot of solution under test (mL)

D = dilution factor to prepare the *Sample solution* from the aliquot taken

L = label claim (mg/Tablet)

Tolerances: NLT 75% of the labeled amount of zinc is dissolved.

- **WEIGHT VARIATION ⟨2091⟩:** Meet the requirements

SPECIFIC TESTS

- **MICROBIAL ENUMERATION TESTS ⟨2021⟩:** The total aerobic microbial count does not exceed 10^3 cfu/g, and the total combined yeast and mold count does not exceed 10^2 cfu/g.
- **ABSENCE OF SPECIFIED MICROORGANISMS ⟨2022⟩:** It meets the requirements of the test for absence of *Escherichia coli*.

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in well-closed containers.
- **LABELING:** The label states the quantity of zinc in terms of mg/Tablet.

Zinc and Vitamin C Lozenges

DEFINITION

Zinc and Vitamin C Lozenges contain NLT 90.0% and NMT 110.0% of the labeled amount of zinc (Zn) derived from substances generally recognized as safe and furnishing an ionizable form of zinc; and NLT 90.0% and NMT 120.0% of the labeled amount of vitamin C, as ascorbic acid ($C_6H_8O_6$), sodium ascorbate ($C_6H_7NaO_6$), or calcium ascorbate dihydrate ($C_{12}H_{14}CaO_{12} \cdot 2H_2O$). It contains no other vitamins or minerals for which nutritional value is claimed. It may contain other labeled added substances or additional ingredients in amounts that are unobjectionable.

IDENTIFICATION

- **A.**

Analysis: Proceed as directed in *Strength* for *Content of Zinc*.

Acceptance criteria: The *Sample solution* produces line emissions or absorptions at the characteristic wavelengths for zinc.

- **B.**

Analysis: Triturate a quantity of finely powdered Lozenges with sufficient alcohol to obtain a solution containing the equivalent of 20 mg/mL of ascorbic acid, sodium ascorbate, or calcium ascorbate dihydrate, and filter. Add 1 mL of 0.1 N hydrochloric acid to 4 mL of the filtrate from Lozenges containing sodium ascorbate or calcium ascorbate.

Acceptance criteria: A portion of the filtrate reduces alkaline cupric tartrate TS slowly at room temperature but more readily upon heating.

STRENGTH

- **CONTENT OF ZINC**

Procedure 1

[NOTE—A standard stock solution is commercially available at different zinc concentrations, which may be used for preparation of *Standard stock solution*. Necessary volumetric adjustment can be made in the *Standard solution*. Concentrations of the *Standard solution* and the *Sample solution* may be modified to fit the linear or working range of the instrument.]

Standard stock solution: Dissolve 625 mg of zinc oxide, weighed, and previously ignited to constant weight, in 10 mL of nitric acid, and add water to make 500.0 mL. This solution contains 1000 µg/mL of zinc.

Standard solution: To a 500-mL volumetric flask add 200 mL of water and 10 mL of nitric acid, and mix thoroughly. Pipet 10.0 mL of the *Standard stock solution* into the volumetric flask, and dilute with water to vol-

ume to obtain a solution having a known concentration of about 20 µg/mL of zinc.

Sample solution: Weigh and finely powder NLT 20 Lozenges. Transfer an accurately weighed portion of the powdered Lozenges, equivalent to about 0.1 g of zinc, to a 50-mL flask. Add 10 mL of nitric acid, and heat the solution on a hot plate to boil gently, during which process fuming evolves. Boil the solution for an additional 30 min with constant swirling, during which no fuming should be observed. Cool the solution to room temperature, quantitatively transfer all of the solution to a 500-mL volumetric flask, dilute with water to volume, and mix. Pipet 25.0 mL of this solution into a 250-mL volumetric flask, add 5 mL of nitric acid, dilute with water to volume, mix, and filter.

Inductively coupled plasma system

(See *Plasma Spectrochemistry* ⟨730⟩.)

Mode: Atomic emission spectroscopy

Analytical wavelength: 206.20 nm

[NOTE—The operating conditions may be developed and optimized based on the manufacturer's recommendation. A typical setting includes radio frequency (RF) power of about 1300 watts, argon torch flow of about 15 L/min, argon auxiliary flow of about 0.2 L/min, and a nebulizer flow rate of about 0.8 L/min.]

Blank: 2% nitric acid solution

Analysis

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

Calculate the percentage of the labeled amount of zinc (Zn) in the portion of Lozenges taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = response from the *Sample solution*
r_S = response from the *Standard solution*
C_S = concentration of zinc in the *Standard solution* (µg/mL)
C_U = nominal concentration of zinc in the *Sample solution* (µg/mL)

Acceptance criteria: 90.0%–110.0% of the labeled amount of zinc

Procedure 2

Standard stock solution A: Dissolve zinc oxide in 5 M hydrochloric acid with warming, if necessary, to obtain a solution with a concentration of 3.89 mg/mL. Dilute with water to obtain a solution with a concentration of 1000 µg/mL of zinc.

Standard stock solution B: 50 µg/mL of zinc from *Standard stock solution A* in 0.125 N hydrochloric acid

Standard solutions: Transfer 1.0, 2.0, 3.0, 4.0, and 5.0 mL of *Standard stock solution B* to separate 100-mL volumetric flasks. Dilute the contents of each flask with 0.125 N hydrochloric acid to volume to obtain solutions containing 0.5, 1.0, 1.5, 2.0, and 2.5 µg/mL of zinc.

Sample solution: Finely powder NLT 20 Lozenges. Transfer an equivalent to 5 Lozenges to a porcelain crucible. Heat the crucible in a muffle furnace maintained at 550° for 6–12 h, and cool. Add 60 mL of hydrochloric acid, and boil gently on a hot plate or steam bath for 30 min, intermittently rinsing the inner surface of the crucible with 6 N hydrochloric acid. Cool, and quantitatively transfer the contents of the crucible to a 100-mL volumetric flask. Rinse the crucible with small portions of 6 N hydrochloric acid, and add the rinsings to the flask. Dilute with water to volume, and filter, discarding the first 5 mL of the filtrate. Dilute this solution quantitatively with 0.125 N hydrochloric acid to obtain a nominal concentration of 2 µg/mL of zinc.

Instrumental conditions

(See *Spectrophotometry and Light-Scattering* ⟨851⟩.)

Mode: Atomic absorption spectrophotometry

Analytical wavelength: 213.8 nm

Lamp: Zinc hollow-cathode

Flame: Air–acetylene

Blank: 0.125 N hydrochloric acid

Analysis

Samples: *Standard solutions* and *Sample solution*

Determine the absorbances of the solutions against the *Blank*. Plot the absorbances of the *Standard solutions* versus the concentration, in µg/mL, of zinc, and draw the straight line best fitting the five plotted points. From the graph so obtained, determine the concentration, *C*, in µg/mL, of zinc in the *Sample solution*.

Calculate the percentage of the labeled amount of zinc (Zn) in the portion of Lozenges taken:

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

C = determined concentration of zinc in the *Sample solution* (µg/mL)
C_U = nominal concentration of zinc in the *Sample solution* (µg/mL)

Acceptance criteria: 90.0%–110.0% of the label claim

- **CONTENT OF VITAMIN C**

Sample solution: Transfer NLT 20 Lozenges to a 1000-mL volumetric flask containing 250 mL of metaphosphoric–acetic acids TS. Insert the stopper in the flask, and shake by mechanical means for 30 min or until the lozenges have disintegrated completely. Dilute with water to volume. Transfer a portion of the solution to a centrifuge tube, and centrifuge until a clear supernatant is obtained. Quantitatively dilute the clear supernatant with water, if necessary, to obtain a solution containing 0.5 mg/mL of ascorbic acid.

Blank: A mixture of 5.5 mL of metaphosphoric–acetic acids TS and 15 mL of water

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Direct titration

Titrant: Standard dichlorophenol–indophenol VS

Endpoint detection: Visual, a rose-pink color that persists for at least 5 s

Analysis: Transfer a volume of the *Sample solution*, equivalent to 2 mg of ascorbic acid, into a 50-mL conical flask. Add 5 mL of metaphosphoric–acetic acids TS, and titrate with *Titrant*. Correct for the volume of the *Titrant* consumed by the *Blank*.

Calculate the percentage of the labeled amount of ascorbic acid ($C_6H_8O_6$) in the portion of the Lozenges taken:

$$\text{Result} = \{[(V_S - V_B) \times F]/W\} \times 100$$

V_S = *Titrant* volume consumed by the *Sample solution* (mL)
V_B = *Titrant* volume consumed by the *Blank* (mL)
F = ascorbic acid equivalent of the *Titrant* (mg/mL)
W = nominal weight of ascorbic acid taken for *Analysis* (mg)

Acceptance criteria: 90.0%–120.0% of the labeled amount

SPECIFIC TESTS

- **MICROBIAL ENUMERATION TESTS ⟨2021⟩:** The total aerobic microbial count does not exceed 10^3 cfu/g, and the total combined yeasts and molds count does not exceed 10^2 cfu/g.
- **ABSENCE OF SPECIFIED MICROORGANISMS ⟨2022⟩:** It meets the requirements of the test for absence of *Escherichia coli*.

PERFORMANCE TESTS

- **DISINTEGRATION AND DISSOLUTION ⟨2040⟩**
  Medium: 0.1 N hydrochloric acid; 900 mL
  Apparatus 2: 75 rpm
  Time: 60 min
  Analysis: Determine the amount of zinc (Zn) and vitamin C dissolved, using the procedures in *Strength* for *Content of Zinc* and *Content of Vitamin C*, making any necessary volumetric adjustments. [NOTE—Proceed without delay in the vitamin C determination.]
  Calculate the percentage of the labeled amount of zinc (Zn) dissolved:

$$\text{Result} = C \times (V_M/a) \times (D/L) \times 100$$

  C = measured concentration of Zinc in the *Sample solution* (mg/mL)
  V_M = volume of *Medium*, 900 mL
  a = aliquot of solution under test taken (mL)
  D = dilution factor to prepare the *Sample solution* from the aliquot taken
  L = labeled amount of zinc (mg/Tablet)
  Calculate the percentage of the labeled amount of vitamin C, as ascorbic acid ($C_6H_8O_6$), dissolved:

$$\text{Result} = (V_S - V_B) \times F \times [(V_M/a)/L] \times 100$$

  V_S = *Titrant* volume consumed by the *Sample solution* (mL)
  V_B = *Titrant* volume consumed by the *Blank* (mL)
  F = concentration of the *Titrant* in terms of the equivalent of ascorbic acid (mg/mL)
  V_M = volume of *Medium*, 900 mL
  a = volume of the aliquot taken for *Analysis*
  L = labeled amount of ascorbic acid (mg/Tablet)
  Tolerances: NLT 75% of the labeled amount of zinc (Zn) and vitamin C is dissolved.
- **WEIGHT VARIATION ⟨2091⟩:** Meet the requirements

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in well-closed containers.
- **LABELING:** The label states the quantity of zinc and vitamin C as ascorbic acid in mg per Lozenge, and the salt form of zinc and the chemical form of vitamin C present in the Lozenges.

Zinc Gluconate—*see Zinc Gluconate General Monographs*