Myristic Acid

$C_{14}H_{28}O_2$ 228.37
Tetradecanoic acid [544-63-8].

DEFINITION
Myristic Acid is obtained from coconut oil and other fats. It contains NLT 97.0% of myristic acid ($C_{14}H_{28}O_2$).

ASSAY
• **FATS AND FIXED OILS,** *Fatty Acid Composition* ⟨**401**⟩
System suitability solution: Prepare as directed in the chapter, except that only stearic acid and palmitic acid are used.
Sample solution: Prepare as directed in the chapter for the *Test solution*.
Standard solution: Prepare as directed for the *Sample solution* using 100 mg of USP Myristic Acid RS instead of the substance to be examined.
Chromatographic system: Prepare as directed in the chapter.
Injection size: 1 µL
System suitability
(See *Chromatography* ⟨621⟩*, System Suitability*.)
Sample: *System suitability solution*
Suitability requirements
Resolution: NLT 1.5 between methyl stearate and methyl palmitate
Analysis
Samples: *Standard solution* and *Sample solution*
Record the chromatograms, and identify the methyl myristate peak in the chromatogram from the *Sample solution* by comparing the retention times of the peaks in that chromatogram with those in the chromatogram from the *Standard solution*. Measure the responses for all of the peaks in the chromatogram from the *Sample solution*, excluding the solvent peak.
Calculate the percentage of myristic acid ($C_{14}H_{28}O_2$) in the portion of Myristic Acid taken:

$$\text{Result} = (A/B) \times 100$$

A = peak response for methyl myristate from the *Sample solution*
B = sum of all peak responses in the *Sample solution*, except the solvent peak
Acceptance criteria: NLT 97.0%

IMPURITIES
• **RESIDUE ON IGNITION** ⟨**281**⟩**:** NMT 0.1%
• **LIMIT OF LEAD**
[NOTE—Select reagents having as low a lead content as practicable, and store all solutions in high-density polyethylene containers. Rinse all plastic and glassware thoroughly with warm 8 N nitric acid followed by deionized water.]
Standard stock solution: Dissolve 160 mg of lead nitrate in 100 mL of water containing 1 mL of nitric acid. Dilute with water to 1000 mL.
Standard solutions: [NOTE—Prepare these solutions on the day of use.] Transfer 10.0 mL of *Standard stock solution* to a 100-mL volumetric flask, and dilute with water to volume. Each mL of this solution contains the equivalent of about 10 µg of lead. Dilute accurately measured volumes of the diluted *Standard stock solution* with water to obtain solutions having known concentrations of 1, 2, and 5 µg of lead/mL.
Sample solution: Transfer 5 g of Myristic Acid to an evaporating dish. Add 5 mL of a 25% sulfuric acid solution, and distribute the sulfuric acid uniformly through the sample. Within a hood, place the dish on a steam bath to evaporate most of the water. Place the dish on a burner, and slowly pre-ash the sample by expelling most of the sulfuric acid. Place the dish in a muffle furnace that has been set at 525°, and ash the sample until the residue appears free from carbon. Prepare a blank by ashing 5 mL of a 25% sulfuric acid solution. Cool, and cautiously wash down the inside of each evaporation dish with water. Treat both the sample and the blank as follows. Add 5 mL of 1 N hydrochloric acid. Place each dish on a steam bath, and evaporate to dryness. To each dish add 1.0 mL of 3 N hydrochloric acid and about 5 mL of water, and heat briefly on a steam bath to dissolve any residue. Transfer each solution quantitatively to a 10-mL volumetric flask, and dilute with water to volume.
Instrumental conditions
(See *Spectrophotometry and Light-Scattering* ⟨851⟩.)
Mode: Atomic absorption spectrophotometry
Analytical wavelength: 283.3 nm at the lead emission line
Slit-width: 0.7 nm
Lamp: Lead electrodeless discharge
Flame: Air–acetylene with a suitable burner head
Blank: Water. [NOTE—Perform a blank determination following the manufacturer's operating instructions.]
Analysis
Samples: *Standard solutions*, *Sample solution*, and *Blank*
Determine the corrected absorbance values by subtracting the absorbance of the *Blank* from the absorbance of each of the *Standard solutions* and from the absorbance of the *Sample solution*. Prepare a standard curve by plotting the corrected absorbance values of the *Standard solutions* versus their corresponding concentration, in µg/mL. From the calibration curve, determine the lead concentration in the *Sample solution*.
Calculate the lead content, in ppm, in the portion of Myristic Acid taken:

$$\text{Result} = (C/W_S) \times V$$

C = measured concentration of lead in the *Sample solution* from the standard curve (µg/mL)
W_S = weight of Myristic Acid taken (g)
V = final volume of the *Sample solution*, 10 mL
Acceptance criteria: NMT 2 ppm

SPECIFIC TESTS
• **CONGEALING TEMPERATURE** ⟨**651**⟩**:** 48°–55.5°
• **FATS AND FIXED OILS,** *Acid Value* ⟨**401**⟩**:** 242–249
• **FATS AND FIXED OILS,** *Iodine Value* ⟨**401**⟩**:** NMT 1.0
• **FATS AND FIXED OILS,** *Saponification Value* ⟨**401**⟩**:** 242–251
• **FATS AND FIXED OILS,** *Unsaponifiable Matter* ⟨**401**⟩**:** NMT 1%
• **WATER DETERMINATION,** *Method I* ⟨**921**⟩**:** NMT 0.2%

ADDITIONAL REQUIREMENTS
• **PACKAGING AND STORAGE:** Preserve in well-closed containers. No storage requirements specified.
• **USP REFERENCE STANDARDS** ⟨**11**⟩
USP Myristic Acid RS

Myristyl Alcohol

DEFINITION
Myristyl Alcohol contains NLT 90.0% of myristyl alcohol ($C_{14}H_{30}O$), the remainder consisting chiefly of related alcohols.

IDENTIFICATION
• **A.** The retention time of the major peak of the *Sample solution* corresponds to that of the *System suitability solution,* as obtained in the *Assay*.

ASSAY
• **PROCEDURE**
System suitability solution: 9 mg/mL of USP Myristyl Alcohol RS and 1 mg/mL of USP Cetyl Alcohol RS in alcohol
Sample solution: 10 mg/mL of Myristyl Alcohol in dehydrated alcohol
Chromatographic system
(See *Chromatography* ⟨621⟩*, System Suitability*.)

Mode: GC
Detector: Flame ionization
Column: 3-mm × 2-m, packed with 10% liquid phase G2 on support S1A
Temperature
Column: 205°
Detector: 250°
Injector port: 275°
Carrier gas: Helium
Injection size: 2 µL
System suitability
Sample: *System suitability solution*
Suitability requirements
Resolution: NLT 4.0 between cetyl alcohol and myristyl alcohol
Relative standard deviation: NMT 1.5%
Analysis
Sample: *Sample solution*
Calculate the percentage of myristyl alcohol ($C_{14}H_{30}O$) in the portion of Myristyl Alcohol taken:

$$\text{Result} = (r_U/r_T) \times 100$$

r_U = peak area of myristyl alcohol from the *Sample solution*
r_T = sum of the peak areas except the solvent peak from the *Sample solution*
Acceptance criteria: NLT 90.0%

SPECIFIC TESTS
- **MELTING RANGE OR TEMPERATUE,** *Class II* ⟨**741**⟩**:** 36° to 42°
- **FATS AND FIXED OILS,** *Acid Value* ⟨**401**⟩**:** NMT 2
- **FATS AND FIXED OILS,** *Iodine Value* ⟨**401**⟩**:** NMT 1
- **FATS AND FIXED OILS,** *Hydroxyl Value* ⟨**401**⟩

Sample: Place 2 g in a dry, glass-stoppered, 250-mL flask, and add 2 mL of pyridine, followed by 10 mL of toluene. To the mixture add 10.0 mL of a solution of acetyl chloride, prepared by mixing 10 mL of acetyl chloride with 90 mL of toluene. Insert the stopper in the flask, and immerse in a water bath heated at 60° to 65° for 20 min. Add 25 mL of water, again insert the stopper in the flask, and shake vigorously for several minutes to decompose the excess acetyl chloride.
Titrimetric system
(See *Titrimetry* ⟨541⟩.)
Mode: Residual titration
Titrant: Acetyl chloride
Back titrant: 1 N sodium hydroxide VS
Blank: Proceed as directed for the *Sample*, omitting Myristyl Alcohol.
Endpoint detection: Colorimetric
Analysis: Add 0.5 mL of phenolphthalein TS to the *Sample* and *Blank*. Titrate each to a permanent pink endpoint with 1 N sodium hydroxide VS, shaking the flask vigorously toward the end of the titration to maintain the contents in an emulsified condition.
Calculate the hydroxyl value:

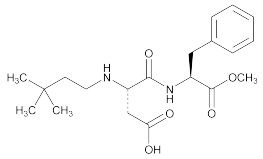
$$\text{Result} = [(V_U - V_B) \times F]/W$$

V_U = volume of 1 N sodium hydroxide consumed by the *Sample* (mL)
V_B = volume of 1 N sodium hydroxide consumed by the *Blank* (mL)
F = equivalent weight of potassium hydroxide, 56.1 mg/mEq
W = weight of the *Sample* (g)

Acceptance criteria: 250–267

ADDITIONAL REQUIREMENTS
- **PACKAGING AND STORAGE:** Preserve in well-closed containers.
- **USP REFERENCE STANDARDS** ⟨**11**⟩
USP Cetyl Alcohol RS
USP Myristyl Alcohol RS

Neotame



$C_{20}H_{30}N_2O_5$ 378.46
L-Phenylalanine, *N*-[*N*-(3,3-dimethylbutyl)-L-α-aspartyl]-1-methyl ester;
N-[*N*-(3,3-Dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester [165450-17-9].

DEFINITION
Neotame contains NLT 97.0% and NMT 102.0% of neotame ($C_{20}H_{30}N_2O_5$), calculated on the anhydrous basis.

IDENTIFICATION
- **A. INFRARED ABSORPTION** ⟨**197K**⟩

ASSAY
- **PROCEDURE**

Mobile phase: Dissolve 3.0 g of sodium 1-heptanesulfonate in 740 mL of water in a suitable 1000-mL vessel, and add 3.8 mL of triethylamine. Adjust the resulting solution with phosphoric acid to a pH of 3.5, and dilute with water to 750 mL. Add 250 mL of acetonitrile, and adjust with phosphoric acid to an apparent pH of 3.7.
Standard solution: 1.0 mg/mL of USP Neotame RS in *Mobile phase*
Sample solution: 1.0 mg/mL of Neotame in *Mobile phase.* [NOTE—This solution is stable for up to 32 h when stored at a temperature of 0°–10°.]
Chromatographic system
(See *Chromatography* ⟨621⟩*, System Suitability*.)
Mode: LC
Detector: UV 210 nm
Column: 4.6-mm × 10-cm; packing L1
Column temperature: 45°
Flow rate: 1.5 mL/min
Injection size: 25 µL
System suitability
Sample: *Standard solution*
Suitability requirements
Tailing factor: NMT 2.0
Relative standard deviation: NMT 2.0%
Analysis
Samples: *Standard solution* and *Sample solution*
Calculate the percentage of neotame ($C_{20}H_{30}N_2O_5$) in the portion of Neotame taken:

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

r_U = peak response from the *Sample solution*
r_S = peak response from the *Standard solution*
C_S = concentration of USP Neotame RS in the *Standard solution* (mg/mL)
C_U = concentration of Neotame in the *Sample solution* (mg/mL)