Mode: LC
Detector: Fluorescence
Excitation wavelength: 384 nm
Emission wavelength: 406 nm
Columns
Guard: 4.6-mm × 7.5-cm; 5-µm packing L1
Analytical: 4.6-mm × 25-cm; 5-µm packing L1
Flow rate: 1 mL/min
Injection volume: 10 µL. [NOTE—If an autosampler is used, the sample loop should be flushed with acetonitrile between injections.]
Analysis
Samples: *Standard solutions*, *Sample solution*, and *Blank*
Inject the *Standard solutions* and construct a four-point calibration curve using the peak area versus concentration (ng/µL). Inject each *Blank* and *Sample solution*, and determine its benzo[*a*]pyrene concentration (ng/µL) from the standard calibration curve.
Calculate the benzo[*a*]pyrene content, in µg/kg, in the portion of Conjugated Linoleic Acids–Free Fatty Acids taken:

$$\text{Result} = (C - C_0) \times (V/M)$$

C = concentration of benzo[*a*]pyrene obtained from the calibration curve for the oil sample (ng/µL)
C_0 = concentration of benzo[*a*]pyrene obtained from the calibration curve for the blank (ng/µL)
V = volume of acetonitrile and tetrahydrofuran (1:1) added to the vial, 300 µL
M = mass of the oil sample (g)
Acceptance criteria: NMT 2 µg/kg

SPECIFIC TESTS
- **WATER DETERMINATION ⟨921⟩, *Method I, Method Ia*:** NMT 0.1%
- **FATS AND FIXED OILS ⟨401⟩, *Procedures, Acid Value*:** 195–204
- **FATS AND FIXED OILS, ⟨401⟩, *Procedures, Peroxide Value*:** NMT 5.0
- **FATS AND FIXED OILS, ⟨401⟩, *Procedures, Fatty Acid Composition***
Standard solution: Prepare as directed for the *Test Solution*, except use 100 mg of USP Conjugated Linoleic Acids–Free Fatty Acids RS.
Sample solution: Prepare as directed in the *Standard solution*, except replace USP Conjugated Linoleic Acids–Free Fatty Acids RS with Conjugated Linoleic Acids–Free Fatty Acids.
System suitability
Sample: *Standard solution*
[NOTE—The relative retention times for the fatty acid components are shown in *Table 1*.]
Suitability requirements
Resolution: NLT 1.5 between CLA isomer *c*9, *t*11 and CLA isomer *t*10, *c*12
Chromatogram similarity: The chromatogram of the *Standard solution* is similar to the reference chromatogram provided with the lot of USP Conjugated Linoleic Acids–Free Fatty Acids RS being used.
Acceptance criteria: Conjugated Linoleic Acids–Free Fatty Acids exhibit the composition profile of fatty acids in *Table 1*.

Table 1

Fatty Acid	Shorthand Notation	Relative Retention Time	Area Percentage
Palmitic acid	16:0	0.82	≤9
Stearic acid	18:0	0.92	≤5
Oleic acid	18:1	0.93	≤20

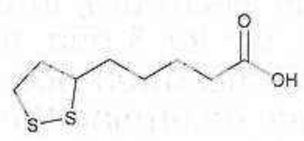
Table 1 *(Continued)*

Fatty Acid	Shorthand Notation	Relative Retention Time	Area Percentage
Linoleic acid	18:2	0.96	≤3
Conjugated linoleic acid	18:2	—	≥78
CLA isomer *c*9, *t*11	18:2	1.00	≥37.5
CLA isomer *t*10, *c*12	18:2	1.01	≥37.5
CLA isomers *trans trans*	18:2	1.03	≤2.0

ADDITIONAL REQUIREMENTS
- **PACKAGING AND STORAGE:** Preserve in tight, light-resistant containers. Store in a cool, dry place, and protect from light, heat, and air.
- **USP REFERENCE STANDARDS ⟨11⟩**
USP Conjugated Linoleic Acids–Free Fatty Acids RS

▲*USP41*

Alpha Lipoic Acid



$C_8H_{14}O_2S_2$ 206.33
Thioctic acid;
1,2-Dithiolane-3-pentanoic acid;
1,2-Dithiolane-3-valeric acid [1077-28-7].

DEFINITION
Alpha Lipoic Acid contains NLT 99.0% and NMT 101.0% of $C_8H_{14}O_2S_2$, calculated on the dried basis.

IDENTIFICATION
- **A.** The retention time of the peak for alpha lipoic acid of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.
- **B. INFRARED ABSORPTION ⟨197K⟩**

ASSAY
- **PROCEDURE**
Buffer solution: 0.68 g/L of monobasic potassium phosphate
Mobile phase: Methanol, *Buffer solution*, and acetonitrile (58:46:9). Adjust with phosphoric acid solution (8.3 in 100) to a pH of 3.0–3.1.
Standard solution: 1.0 mg/mL of USP Alpha Lipoic Acid RS in *Mobile phase*
Sample solution: 1.0 mg/mL of Alpha Lipoic Acid in *Mobile phase*
Chromatographic system
(See *Chromatography ⟨621⟩, System Suitability*.)
Mode: LC
Detector: UV 215 nm
Column: 4.6-mm × 250-mm; packing L1
Column temperature: 35°
Flow rate: 1.2 mL/min
Injection size: 20 µL
System suitability
Sample: *Standard solution*
Suitability requirements
Column efficiency: NLT 10,000 theoretical plates
Tailing factor: NMT 2.0 for the alpha lipoic acid peak

DS Monographs