Trichloromonofluoromethane

CCl_3F 137.37
Methane, trichlorofluoro-.
Trichlorofluoromethane [75-69-4].

» Trichloromonofluoromethane contains not less than 99.6 percent and not more than 100.0 percent of CCl_3F, calculated on the anhydrous basis.

Packaging and storage—Preserve in tight cylinders, and avoid exposure to excessive heat.

Identification—The IR absorption spectrum of it, determined in a 10-cm cell with sodium chloride windows, at atmospheric pressure, exhibits maxima, among others, at the following wavelengths, in µm: 4.67 (*m*), 5.95 (*m*), 7.28 (*s*), 8.06 (*m*), 9.2 (*vs*), 10.7 (*vs*), 11.8 (*vs*), and 13.4 (*m*). The stronger maxima are best obtained at pressures less than 10 mm of mercury.

Boiling temperature: approximately 24°, determined as directed under *Approximate Boiling Temperature* (see *Aerosols, Nasal Sprays, Metered-Dose Inhalers, and Dry Powder Inhalers* ⟨601⟩).

Water: not more than 0.001%, determined as directed under *Water Content* (see *Aerosols, Nasal Sprays, Metered-Dose Inhalers, and Dry Powder Inhalers* ⟨601⟩ and *Method Ic* under *Water Determination* ⟨921⟩).

High-boiling residues: not more than 0.01%, determined as directed for *High-Boiling Residues*, *Method I*, under *Aerosols, Nasal Sprays, Metered-Dose Inhalers, and Dry Powder Inhalers* ⟨601⟩.

Chromatographic purity—In the chromatogram recorded for the *Assay*, identify the peaks due to dichlorodifluoromethane and dichlorotetrafluoroethane from relative retention times observed on chromatographing the *Resolution preparation:* the sum of these two peak areas is not greater than 0.2% of the total of all peak areas; and the sum of the areas of all peaks other than that for trichloromonofluoromethane is not greater than 0.4% of the total of all peak areas.

Inorganic chlorides—Place 5 mL of anhydrous methanol in a test tube, add 3 drops of a saturated solution of silver nitrate in anhydrous methanol, shake, and add 7 g of Trichloromonofluoromethane: no opalescence or turbidity is produced.

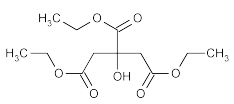
Assay—

Resolution preparation and *Chromatographic system*—Proceed as directed in the *Assay* under *Dichlorodifluoromethane.*

Assay preparation—Introduce the liquid phase of Trichloromonofluoromethane into an evacuated headspace vial.

Procedure—Inject the gas phase headspace *Assay preparation* into the chromatograph, record the chromatogram, and measure the peak areas. Calculate the percentage (a/a) of CCl_3F in the portion of Trichloromonofluoromethane taken.

Triethyl Citrate



$C_{12}H_{20}O_7$ 276.29

DEFINITION
Triethyl Citrate contains NLT 99.0% and NMT 100.5% of $C_{12}H_{20}O_7$, calculated on the anhydrous basis.

IDENTIFICATION
- **A. Infrared Absorption** ⟨197F⟩
- **B.** The retention time of the major peak of the *Sample solution* corresponds to that of a similar preparation of USP Triethyl Citrate RS, as obtained in the *Assay*.

ASSAY
- **Procedure**

System suitability solution: 30 mg/mL each of USP Triethyl Citrate RS and USP Acetyltriethyl Citrate RS in toluene
Sample solution: 30 mg/mL of Triethyl Citrate in toluene
Chromatographic system
(See *Chromatography* ⟨621⟩, *System Suitability*.)
Mode: GC
Detector: Flame ionization
Column: 0.32-mm × 30-m; 0.5-µm layer of phase G42
Temperature
Injector: 225°
Detector: 275°
Column: See the temperature program table below.

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
80	—	80	0.5
80	20	220	20

Flow rate: 2.3 mL/min
Carrier gas: Helium
Injection type: Split, 30:1
Injection size: 1 µL
System suitability
Sample: *System suitability solution*
[NOTE—The relative retention times for triethyl citrate and acetyltriethyl citrate are 0.9 and 1.0, respectively.]
Suitability requirements
Resolution: NLT 1.5 between triethyl citrate and acetyltriethyl citrate
Relative standard deviation: NMT 2.0% (determined from both the triethyl citrate and acetyltriethyl citrate peaks, based on area percentage calculation)
Analysis
Sample: *Sample solution*
[NOTE—Measure all of the peak areas, excluding the solvent peak.]
Calculate the percentage of $C_{12}H_{20}O_7$ in the portion of Triethyl Citrate taken:

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

r_U = peak area for triethyl citrate
r_T = sum of the area responses of all the peaks
Acceptance criteria: 99.0%–100.5% on the anhydrous basis

IMPURITIES
Inorganic Impurities
- **Heavy Metals,** *Method II* ⟨**231**⟩**:** NMT 10 ppm

SPECIFIC TESTS
- **Specific Gravity** ⟨**841**⟩**:** 1.135–1.139
- **Refractive Index** ⟨**831**⟩**:** 1.439–1.441
- **Acidity**

Neutralized isopropyl alcohol: To a suitable quantity of isopropyl alcohol add 2–3 drops of bromothymol blue TS and just sufficient 0.10 N sodium hydroxide dropwise to produce a faint blue color. [NOTE—Prepare *Neutralized isopropyl alcohol* just prior to use.]
Sample solution: 32.0 g of Triethyl Citrate in 30 mL of *Neutralized isopropyl alcohol*
Analysis: Add bromothymol blue TS. Titrate with 0.10 N sodium hydroxide to a faint blue endpoint.
Acceptance criteria: NMT 1.0 mL of 0.10 N sodium hydroxide is required.

• **WATER DETERMINATION,** *Method I* ⟨**921**⟩**:** NMT 0.25%

ADDITIONAL REQUIREMENTS
- **PACKAGING AND STORAGE:** Preserve in tight containers.
- **USP REFERENCE STANDARDS** ⟨**11**⟩
  USP Acetyltriethyl Citrate RS
  USP Triethyl Citrate RS

Medium-Chain Triglycerides

Glycerides, mixed decanoyl and octanoyl.
Caprylic and capric triglycerides.

» Medium-Chain Triglycerides consist of a mixture of triglycerides of saturated fatty acids, mainly of caprylic acid ($C_8H_{16}O_2$) and capric acid ($C_{10}H_{20}O_2$). The fatty acids are derived from the oil extracted from the hard, dried fraction of the endosperm of *Cocos nucifera* L. or from the dried endosperm of *Elaeis guineensis* Jacq. They contain not less than 95 percent of saturated fatty acids with 8 and 10 carbon atoms.

Packaging and storage—Preserve in tight containers, protected from light. Store at temperatures not exceeding 25°.

Labeling—Where it is intended for use in parenteral nutrition, it is so labeled.

Appearance—The substance is clear and not more intensely colored than a solution prepared immediately before use by mixing 2.4 mL of ferric chloride CS and 0.6 mL of cobaltous chloride CS with *Diluent,* prepared as directed below, to make 10.0 mL, and diluting 5.0 mL of the solution so obtained with *Diluent* to make 10.0 mL. Make the comparison by viewing the substance and the solution downward in matched color-comparison tubes against a white surface (see *Color and Achromicity* ⟨631⟩).

Diluent—Transfer 27.5 mL of hydrochloric acid to a 1000-mL volumetric flask, and dilute with water to volume.

Identification—

A: It meets the requirements of the test for *Saponification value.*

B: It meets the requirements of the test for *Fatty acid composition.*

Specific gravity ⟨841⟩**:** between 0.93 and 0.96, at 20°.

Acid value ⟨401⟩**:** not more than 0.2.

Hydroxyl value ⟨401⟩**:** not more than 10.

Iodine value ⟨401⟩**:** not more than 1.0.

Peroxide value ⟨401⟩**:** not more than 1.0.

Saponification value ⟨401⟩**:** between 310 and 360, determined on 1.0 g.

Unsaponifiable matter ⟨401⟩**:** not more than 0.5%, determined on 5.0 g.

Fatty acid composition ⟨401⟩**—**The fatty acid fraction of Medium-Chain Triglycerides exhibits the following composition, as determined in the section *Fatty Acid Composition.* Disregard any peak with an area less than 0.05% of the total area:

Carbon-Chain Length	Number of Double Bonds	Percentage (%)
6	0	≤2.0
8	0	50.0–80.0
10	0	20.0–50.0
12	0	≤3.0
14	0	≤1.0

Viscosity ⟨911⟩**:** between 25 and 33 centipoises determined at 20 ± 0.1° with a capillary viscosimeter.

Refractive index ⟨831⟩**:** between 1.440 and 1.452, at 20°.

Alkaline impurities—Dissolve 2.0 g of Medium-Chain Triglycerides in a mixture of 1.5 mL of alcohol and 3.0 mL of ethyl ether. Add 0.05 mL of bromophenol blue TS, and titrate with 0.01 N hydrochloric acid to a yellow endpoint: not more than 0.15 mL of 0.01 N hydrochloric acid is required.

Water, *Method I* ⟨921⟩**:** not more than 0.2%.

Total ash ⟨561⟩**:** not more than 0.1%, determined on 2.0 g.

Heavy metals, *Method II* ⟨231⟩**—**[NOTE—Use this test for Medium-Chain Triglycerides intended for use other than in parenteral nutrition.]

Test solution—Transfer 2.0 g of Medium-Chain Triglycerides to a quartz crucible, add 0.5 g of magnesium oxide, and mix. Ignite the crucible to dull redness until a homogeneous white or grayish-white mass is obtained. Ignite at 800° for 1 hour, cool, and dissolve the residue by adding two 5-mL portions of diluted hydrochloric acid. Add 0.1 mL of phenolphthalein TS and then ammonium hydroxide until a pink color is obtained. Cool, add glacial acetic acid until the solution is decolorized, then add 0.5 mL in excess, and dilute with water to 20.0 mL.

Standard solution—To 0.5 g of magnesium oxide add 2.0 mL of *Standard Lead Solution,* and evaporate to dryness at 105° for 1 hour. Using the same conditions as prescribed for the *Test solution,* ignite, dissolve in diluted hydrochloric acid, add ammonia and then acetic acid, and dilute with water to 20.0 mL.

Procedure— To 12 mL of the *Test solution,* add 2.0 mL of *pH 3.5 Acetate Buffer,* mix, add to 1.2 mL of thioacetamide-glycerin base TS, and mix immediately. To 10 mL of the *Standard solution,* add 2.0 mL of the *Test solution,* add 2.0 mL of *pH 3.5 Acetate Buffer,* mix, add to 1.2 mL of thioacetamide-glycerin base TS, and mix immediately. Prepare a blank, using a mixture of 10 mL of water and 2.0 mL of the *Test solution.* Compared to the blank, the *Standard solution* shows a light brown color. Dilute both the *Test solution* and the *Standard solution* with water to 50 mL, allow to stand for 2 minutes, and view downward over a white surface: any brown color from the *Test solution* is not darker than that of the solution from the *Standard solution* (not more than 10 µg per g).

Limit of chromium—[NOTE—Use this test for Medium-Chain Triglycerides intended for use in parenteral nutrition.]

Test stock solution—Transfer about 50 g of Medium-Chain Triglycerides to a 100-mL volumetric flask, dissolve in and dilute with diisobutyl ketone to volume.

Test solution—Transfer 4.0 mL of *Test stock solution* to a 10-mL volumetric flask, and dilute with diisobutyl ketone to volume.

Chromium standard solution—Transfer about 0.283 g of potassium dichromate, previously dried at 105° for 4 hours and accurately weighed, to a 1000-mL volumetric flask, and dilute with water to volume. Immediately before use, dilute this solution with water to 1000 times its volume. This solution contains the equivalent of 0.1 µg of chromium per mL.

Standard solutions—Into each of three 10-mL volumetric flasks, transfer 4.0 mL of *Test stock solution,* add 0.5, 1.0, and 2.0 mL, respectively, of *Chromium standard solution,* and dilute with diisobutyl ketone to volume. These solutions contain 0.005 µg, 0.01 µg, and 0.02 µg of chromium per mL, respectively.

Procedure—Concomitantly determine the absorbances of the *Standard solutions* and the *Test solution* at least three times each, at the wavelength of maximum absorbance at 357.8 nm, with a suitable atomic absorption spectrophotometer (see *Spectrophotometry and Light-Scattering* ⟨851⟩) equipped with a graphite furnace and a chromium hollow-cathode lamp, using argon as the carrier gas. Record the average of the steady readings for each of the *Standard solutions* and the *Test solution.* Plot the absorbances of the *Standard solutions* and the *Test solution* versus the concentration of added chromium. Draw the straight line best fitting the points, and extrapolate the line until it meets the concentration axis. The distance between this point and the intersection of the axes represents the concentration of