

**DEFINITION**

Acetyltriethyl Citrate contains NLT 99.0% of acetyltriethyl citrate (C<sub>14</sub>H<sub>22</sub>O<sub>8</sub>), 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 the *System suitability solution*, as obtained in the *Assay*.

**ASSAY**• **PROCEDURE**

**System suitability solution:** 30 mg/mL each of USP Acetyltriethyl Citrate RS and USP Triethyl Citrate RS in toluene

**Sample solution:** 30 mg/mL in toluene

**Chromatographic system**

(See *Chromatography* (621), *System Suitability*.)

**Mode:** GC, equipped with an on-column, temperature-programmable injector

**Detector:** Flame ionization

**Column:** 0.32-mm × 30-m, bonded with a 0.5-μm layer of phase G42

**Temperatures**

**Injector:** See Table 1.

**Detector:** 275°

**Column:** See Table 2.

Table 1

Start Temperature (°)	Ramp (°)	End Temperature (°)	Hold Time (min)
85	—	85	0.5
85	20	225	10

Table 2

Start Temperature (°)	Ramp (°)	End Temperature (°)	Hold Time (min)
80	—	80	0.5
80	20	220	10

**Flow rate:** 2.3 mL/min

**Carrier gas:** Helium

**Injection volume:** 1 μL

**System suitability**

**Sample:** *System suitability solution*

[NOTE—The relative retention times of 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

**Analysis**

**Sample:** *Sample solution*

Calculate the percentage of acetyltriethyl citrate (C<sub>14</sub>H<sub>22</sub>O<sub>8</sub>) in the portion of sample taken:

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

$r_U$  = peak area of acetyltriethyl citrate from the *Sample solution*

$r_T$  = sum of all the peaks excluding the solvent peak

**Acceptance criteria:** NLT 99.0% on the anhydrous basis

**IMPURITIES****Delete the following:**

- **HEAVY METALS, Method II (231):** NMT 10 μg/g (Official 1-Jan-2018)

**SPECIFIC TESTS**

- **SPECIFIC GRAVITY (841):** 1.135–1.139

- **REFRACTIVE INDEX (831):** 1.432–1.441

• **ACIDITY**

**Sample:** 32.0 g

**Analysis:** Dissolve the *Sample* in 30 mL of isopropyl alcohol, previously neutralized to bromothymol blue. Add bromothymol blue TS, and 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 0.3%

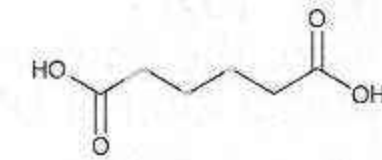
**ADDITIONAL REQUIREMENTS**

- **PACKAGING AND STORAGE:** Preserve in tight containers.

- **USP REFERENCE STANDARDS (11)**

USP Acetyltriethyl Citrate RS

USP Triethyl Citrate RS

**Activated Charcoal—see Activated Charcoal General Monographs****Adipic Acid**

C<sub>6</sub>H<sub>10</sub>O<sub>4</sub>

Hexanedioic acid;

1,4-Butanedicarboxylic acid [124-04-9].

146.14

**DEFINITION**

Adipic Acid contains NLT 99.0% and NMT 101.0% of C<sub>6</sub>H<sub>10</sub>O<sub>4</sub>, calculated on the dried basis.

**IDENTIFICATION**

- **A. INFRARED ABSORPTION** (197K)

**ASSAY**• **PROCEDURE**

**Sample:** 60 mg

**Titrimetric system**

(See *Titrimetry* (541).)

**Mode:** Direct titration

**Titrant:** 0.1 N sodium hydroxide VS

**Blank:** 50.0 mL of water

**Endpoint detection:** Colorimetric

**Analysis:** Dissolve the *Sample* in 50 mL of water. Add 0.2 mL of phenolphthalein TS, and titrate with 0.1 N sodium hydroxide VS to a permanent pale pink endpoint. Perform a blank determination. Calculate the percentage of adipic acid (C<sub>6</sub>H<sub>10</sub>O<sub>4</sub>) in the *Sample* taken:

$$\text{Result} = [(V - B) \times N \times F \times 100] / W$$

$V$  = titrant volume consumed by the *Sample* (mL)

$B$  = titrant volume consumed by the *Blank* (mL)

$N$  = titrant actual normality (mEq/mL)

$F$  = equivalency factor, 73.1 mg/mEq

$W$  = weight of the *Sample* (mg)