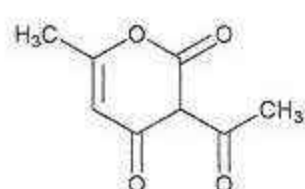


Dehydroacetic Acid



$C_8H_8O_4$ 168.15
 Keto form: 2H-Pyran-2,4(3H)-dione, 3-acetyl-6-methyl-;
 3-Acetyl-6-methyl-2H-pyran-2,4(3H)-dione [520-45-6].
 Enol form: 2H-Pyran-2-one, 3-acetyl-4-hydroxy-6-methyl-;
 3-Acetyl-4-hydroxy-6-methyl-2H-pyran-2-one [771-03-9].

DEFINITION

Dehydroacetic Acid contains NLT 98.0% and NMT 100.5% of dehydroacetic acid ($C_8H_8O_4$), calculated on the dried basis.

IDENTIFICATION

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

ASSAY

• PROCEDURE

Sample: 500 mg

Blank: 75 mL of neutralized alcohol

Titrimetric system

(See *Titrimetry* (541).)

Mode: Direct titration

Titrant: 0.1 N sodium hydroxide VS

Endpoint detection: Visual

Analysis: Transfer the *Sample* into a 250-mL conical flask, dissolve it in 75 mL of neutralized alcohol, and add phenolphthalein TS. Titrate with *Titrant* to a pink endpoint that persists for NLT 30 s. Perform a blank determination.

Calculate the percentage of dehydroacetic acid ($C_8H_8O_4$) in the *Sample* taken:

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

V_S = *Titrant* volume consumed by the *Sample* (mL)

V_B = *Titrant* volume consumed by the *Blank* (mL)

N = actual normality of the *Titrant* (mEq/mL)

F = equivalency factor, 168.2 mg/mEq

W = *Sample* weight (mg)

Acceptance criteria: 98.0%–100.5% on the dried basis

IMPURITIES

- **RESIDUE ON IGNITION** (281): NMT 0.1%

Delete the following:

- **HEAVY METALS, Method II** (231): NMT 10 $\mu\text{g/g}$ (Official 1-Jan-2018)

SPECIFIC TESTS

- **MELTING RANGE OR TEMPERATURE, Class I** (741):

109°–111°

- **LOSS ON DRYING** (731)

Analysis: Dry a sample at 80° for 4 h.

Acceptance criteria: NMT 1.0%

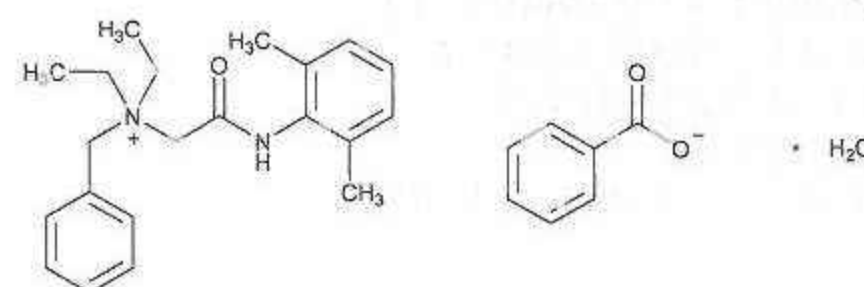
ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in well-closed containers. No storage requirements specified.

• USP REFERENCE STANDARDS (11)

USP Dehydroacetic Acid RS

Denatonium Benzoate



$C_{28}H_{34}N_2O_3 \cdot H_2O$ 464.60

$C_{28}H_{34}N_2O_3$ 446.59

Benzenemethanaminium, N-[2-[(2,6-dimethylphenyl)amino]-2-oxoethyl]-N,N-diethyl-, benzoate, monohydrate; Benzyl-diethyl-[(2,6-xylyl)carbamoyl]methyl ammonium benzoate monohydrate [86398-53-0].

Anhydrous [3734-33-6].

DEFINITION

Denatonium Benzoate, dried at 105° for 2 h, contains one molecule of water of hydration or is anhydrous. When dried at 105° for 2 h, it contains NLT 99.5% and NMT 101.0% of denatonium benzoate ($C_{28}H_{34}N_2O_3$).

IDENTIFICATION

- **A. INFRARED ABSORPTION** (197K)
- **B. ULTRAVIOLET ABSORPTION** (197U)
 - Analytical wavelength:** 263 nm
 - Sample solution:** 100 $\mu\text{g/mL}$
 - Medium:** Water
 - Acceptance criteria:** Absorptivities, calculated on the dried basis, do not differ by more than 3.0%.
- **C.**
 - Sample:** 150 mg
 - Analysis:** Dissolve the *Sample* in 10 mL of water, and add 15 mL of trinitrophenol TS.
 - Acceptance criteria:** A yellow precipitate is formed.
- **D.**
 - Sample:** 100 mg
 - Analysis:** Dissolve the *Sample* in 10 mL of water, and add 20 mL of 2 N sulfuric acid and 15 mL of ammonium reineckate TS. Mix, filter through a sintered-glass crucible using gentle suction, and wash thoroughly with water. Remove as much water as possible with suction, and then dry in an oven at 105° for 1 h.
 - Acceptance criteria:** The denatonium reineckate so obtained melts at about 170° (see *Melting Range or Temperature* (741)).

ASSAY

• PROCEDURE

Sample: 900 mg, previously dried

Blank: 50 mL of glacial acetic acid

Titrimetric system

(See *Titrimetry* (541).)

Mode: Direct titration

Titrant: 0.1 N perchloric acid VS

Endpoint detection: Visual

Analysis: Dissolve the *Sample* in 50 mL of glacial acetic acid, and add 1 drop of crystal violet TS. Titrate with *Titrant* to a green endpoint. Perform a blank determination, and make any necessary correction.

Calculate the percentage of denatonium benzoate ($C_{28}H_{34}N_2O_3$) in the portion of sample taken:

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

V_S = *Titrant* volume consumed by the *Sample* (mL)

V_B = *Titrant* volume consumed by the *Blank* (mL)