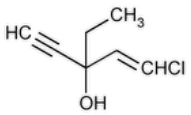
Status: Currently Official on 14-Feb-2025
Official Date: Official as of 01-May-2020
Document Type: USP Monographs
DocId: GUID-8F608AED-4C24-4AEE-86F9-C5AFE63AAEB1_2_en-US
DOI: https://doi.org/10.31003/USPNF_M31290_02_01
DOI Ref: 7yt5c

Ethchlorvynol



C_7H_9ClO 144.60

1-Penten-4-yn-3-ol, 1-chloro-3-ethyl-.

1-Chloro-3-ethyl-1-penten-4-yn-3-ol CAS RN®: 113-18-8; UNII: 6EIM3851UZ.

» Ethchlorvynol contains not less than 98.0 percent and not more than 100.0 percent of *E*-ethchlorvynol (C_7H_9ClO), calculated on the anhydrous basis.

Packaging and storage—Preserve in tight, light-resistant glass or polyethylene containers, using polyethylene-lined closures.

USP Reference standards ⟨11⟩—

USP Ethchlorvynol RS

Identification—

Change to read:

A: ▲*Spectroscopic Identification Tests* ⟨197⟩*, Infrared Spectroscopy: 197F*▲ (CN 1-May-2020) ·

B: Dissolve about 1 g in 20 mL of methanol. To 1 mL of the solution, add about 4 drops of 6 N ammonium hydroxide, mix, then add silver nitrate TS, a few drops at a time: a yellowish white precipitate is formed, and it at first redissolves, but becomes insoluble when an excess of silver nitrate TS has been added.

C: To 10 mL of the solution prepared in *Identification* test *B* add 5 mL of freshly prepared *m*-phenylenediamine hydrochloride-oxalic acid solution (prepared by dissolving 1 g of *m*-phenylenediamine hydrochloride and 1 g of oxalic acid in 35 mL of water and filtering, if necessary): a reddish orange color is produced in about 3 minutes.

Refractive index ⟨831⟩**:** between 1.476 and 1.480.

Acidity—Dissolve 5.0 mL of Ethchlorvynol in 50 mL of a mixture of equal volumes of water and methanol that has been neutralized to the phenolphthalein endpoint with 0.1 N sodium hydroxide. Add 1 mL of phenolphthalein TS, and titrate with 0.10 N sodium hydroxide to a pink endpoint: not more than 1.7 mL of 0.10 N sodium hydroxide is required for neutralization.

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

Chromatographic purity—Calculate the percentage of each peak, other than the *E*-ethchlorvynol peak, observed in the chromatogram of the Ethchlorvynol obtained as directed in the *Assay* taken by the same formula:

$$100r_i/r_t$$

in which r_i is the response of each secondary peak and r_t is the sum of all of the peaks observed in the chromatogram: not more than 0.2% of toluene, not more than 1.5% of *Z*-ethchlorvynol, and not more than 0.3% of any other impurity is found, and the total of all observed impurities is not more than 2.0%.

Assay—

Resolution solution—Add 2.5 µL of toluene to 0.5 mL of USP Ethchlorvynol RS, and mix.

Chromatographic system (see *Chromatography* ⟨621⟩)—The gas chromatograph is equipped with a thermal conductivity detector and a 1.8-m × 4-mm glass column (pretreated with 10% dimethyldichlorosilane in toluene) packed with 10% phase G16 on 60- to 80-mesh support S1AB. The column is maintained at about 160°, and the injector and the detector are maintained at about 200°. The carrier gas is dry helium, flowing at a rate of about 30 mL per minute. Chromatograph the *Resolution solution*, and record the peak responses as directed for *Procedure:* the relative retention times for toluene, β-chlorovinylethyl ketone (if present), *Z*-ethchlorvynol, and *E*-ethchlorvynol are about 0.1, 0.2, 0.8, and 1.0, respectively; the resolution, *R,* between the *Z*- and *E*-ethchlorvynol peaks is not less than 1.0; and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure— [Note—Use peak areas where peak responses are indicated.]

Inject about 3 µL of Ethchlorvynol into the chromatograph, record the chromatogram, and measure the responses for all of the peaks.

Calculate the percentage of *E*-ethchlorvynol (C_7H_9ClO) in the specimen of Ethchlorvynol taken by the formula:

$$100r_e/r_t$$

in which r_e is the response of the *E*-ethchlorvynol peak obtained in the chromatogram of the Ethchlorvynol and r_t is the sum of the responses of all of the peaks observed in the chromatogram.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
ETHCHLORVYNOL	Documentary Standards Support	SM42020 Small Molecules 4

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. Information currently unavailable

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