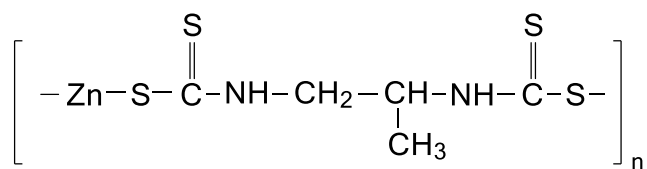


PROPINEB

177



$n > 1$

ISO common name	propineb
m.p.	Decomposed
v.p.	Negligible
Solubility	Insoluble in water and most organic solvents
Description	White powder
Stability	Decomposed under acid conditions
Formulation	Wettable powders and dustable powders

PROPINEB TECHNICAL

177/TC/M/-

1. **Sampling.** Take at least 200g. Fill the bottle completely and store them at a temperature below 20°C

2. Identity tests

2.1 HPLC. Use the reversed phase HPLC method below. The relative retention time of the propineb peak in the sample solution should not differ by more than 1.5% from that of the calibration solution (Fig. 1 and 2).

2.2 Infrared. Prepare potassium bromide discs from the sample and from propineb standard using 0.7 mg material and 400 mg of potassium bromide. Scan the discs from 4000 to 400 cm^{-1} . The spectrum produced from the sample should not differ significantly from that of the standard (Fig. 3).

2.3 Zinc. MT 154

3. propineb.

OUTLINE OF METHOD

The content of Propineb is determined by reversed phase HPLC using UV detector and external standardization.

REAGENTS

Propineb standard of known content. Store refrigerated.

Water HPLC Grade

Methanol HPLC grade

Disodium hydrogen phosphate

Sodium hydroxide

Tetrabutyl ammonium hydrogen sulfate

Sodium Sulfite

EDTA

CALIBRATION SOLUTION

a) Solution A. Weigh 3.72g EDTA, 1.42g disodium hydrogen phosphate, and 3.39g tetrabutylammonium hydrogen sulfate into a beaker, then add 1000 mL water. Place the beaker in an ultrasonic bath until the sample has been dissolved completely (about 5 min), mixed thoroughly, adjust the pH of the solution to 9.5 with saturated

sodium hydroxide solution.

- b) Solution B. Weigh 7.44 g EDTA, 1.42 g disodium hydrogen phosphate and 1 g sodium sulfite into a beaker, then add 1000 mL water. Place the beaker in an ultrasonic bath until the sample has been dissolved completely (about 5 min), mixed thoroughly, adjust the pH of the solution to 11.0 with saturated sodium hydroxide solution.
- c) Calibration solution. Weigh in duplicate Propineb standard containing 40 mg (accurate to 0.2mg) pure Propineb accurately into a 100 mL volumetric flask, add solution B (85 mL) and place the flasks in an ultrasonic bath until the sample has been dissolved completely (about 10 min, keep the bath temperature not higher than 20°C). Allow the solutions to back to room temperature, then make up to volume with solution B and mix thoroughly. Transfer 5 mL into 50 mL volumetric flask accurately, make up to volume with solution B and mix thoroughly. Prior to analysis, filter the solutions with 0.22µm filters .

APPARATUS

High performance liquid chromatograph equipped with a detector suitable for 280nm, a column oven can control at 20 °C and injection system capable of injecting 5µL. (Inject volume depends on the existing equipment.)

Liquid chromatographic column, Agilent Extent C18 (4.6×150mm, 5µm) or equivalent with the same selectivity.

Ultrasonic bath

Filter; 0.22µm

Analytical balance

pH meter

PROCEDURE

(a) Chromatographic conditions (typical)

Column: Agilent Extent C18 (4.6×150mm, 5µm) or equivalent

Mobile phase: Solution A- Methanol =65:35 (V/V)

Column Temperature: 20°C

Flow Rate: 1.0ml/min

Detector Wavelength: 280nm

Injection Volume: 5µL

Retention time: approximately 6.5min

Run time: 13min

Environment temperature: not higher than 22 °C

(b) Equilibration of the system.

Inject 5µL of the calibration solution C1 and repeat the injections until retention times and peak areas deviate by less than ± 0.5% from the mean for 3 successive injections.

(C) Sample preparation.

Prepare sample solutions in duplicate. Weigh the test item containing around 40mg

(accurate to 0.2mg) pure propineb accurately into a 100 mL volumetric flask, add solution B (85 mL) and place the flasks in an ultrasonic bath until the sample has been dissolved completely (about 5 min and keep the bath temperature not higher than 20 °C). Allow the solutions to back to Environment temperature, then make up to volume with solution B and mix thoroughly. Transfer 5 mL into 50 mL volumetric flask accurately, make up to volume with solution B and mix thoroughly. Prior to analysis, filter the solutions with 0.22µm filters

(d) Determination.

Inject 5µL of calibration solution C2 for two successive injections. The mean response factor for this solution should deviate by no more than 1% from that of C1 (see section (b), otherwise prepare the calibration solutions again. Inject in duplicate 5µL of each sample bracketing them by single injection of calibration solution (C1 or C2) using the following sequence: C1, S1, S1, C2, S2, S2, C1, etc .

(e) Calculation.

Calculate the mean value of each pair of response factors bracketing the two injections of a sample and use this value for calculating propineb content of the bracketed sample injections.

$$f_i = \frac{s \times P}{H_s} \dots\dots\dots$$

$$\text{Propineb content(X1)} = \frac{f \times H_w}{w} \text{ g/kg}$$

where:

- f_i = individual response factor
- f = mean response factor
- H_s = peak area of propineb in the calibration solution
- H_w = peak area of propineb in the sample solution
- s = mass of propineb reference standard in the calibration solution (mg)
- w = mass of sample taken (mg)
- P = purity of propineb reference standard (g/kg)

Repeatability r = 6 to 10 g/kg at 900g/kg active ingredient content

Reproducibility R = 11 to 19 g/kg at 900g/kg active ingredient content

PROPINEBWETTABLE POWDERS

177/WP/M/

1. Sampling. Take at least 200 g.

2. Identity tests

2.1 **HPLC.** As for propineb technical 177/TC/M/2.1

2.2 **Infrared.** As for propineb technical 177/TC/M/2.2

3. propineb. As for propineb technical 177/TC/M/3

4. Suspensibility

Reagents and apparatus as for 177/TC/M/3 and MT 184.1

a) Preparation of suspension and determination of sedimentation. MT 184.1

b) Determination of Propineb in the bottom 25ml of suspension. After removal of the top 225ml suspension transfer the remaining 25ml to a volumetric flask (100ml) and dilute to volume with Solution B, place the flasks in an ultrasonic bath until the sample has been dissolved completely (about 5 min and keep the bath temperature not higher than 20°C), then do the measurement and calculation in accordance with the analysis of Propineb technical method.

c) Calculation

$$\text{Suspensibility} = \frac{111(c - Q)}{c} \%$$

where:

c = mass of propineb in the sample taken for the preparation of the suspension (g)

Q = mass of propineb in the bottom 25 ml of suspension (g)

Repeatability r = 6 to 10 g/kg at 700g/kg active ingredient content

Reproducibility R = 11 to 19 g/kg at 700g/kg active ingredient content

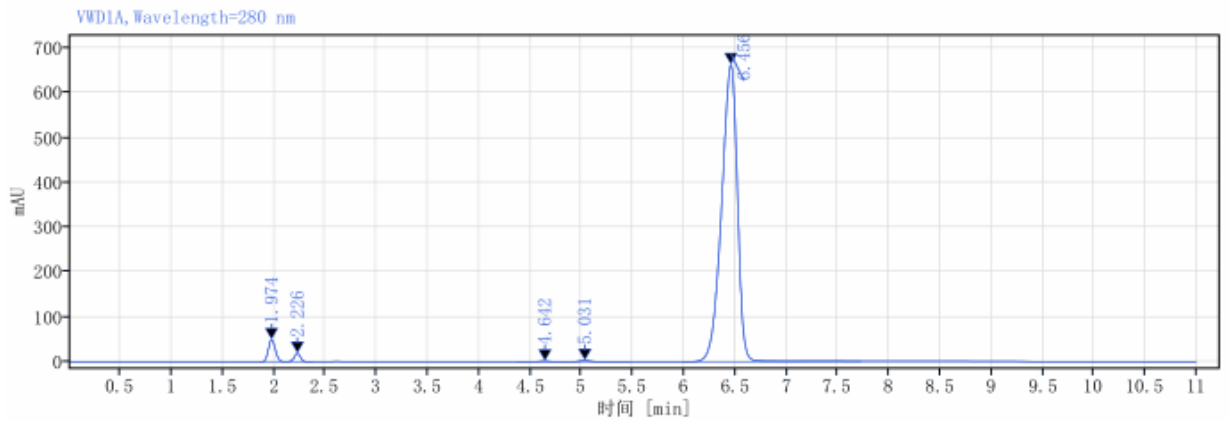


Fig. 1 Typical HPLC-Chromatogram of Propineb standard

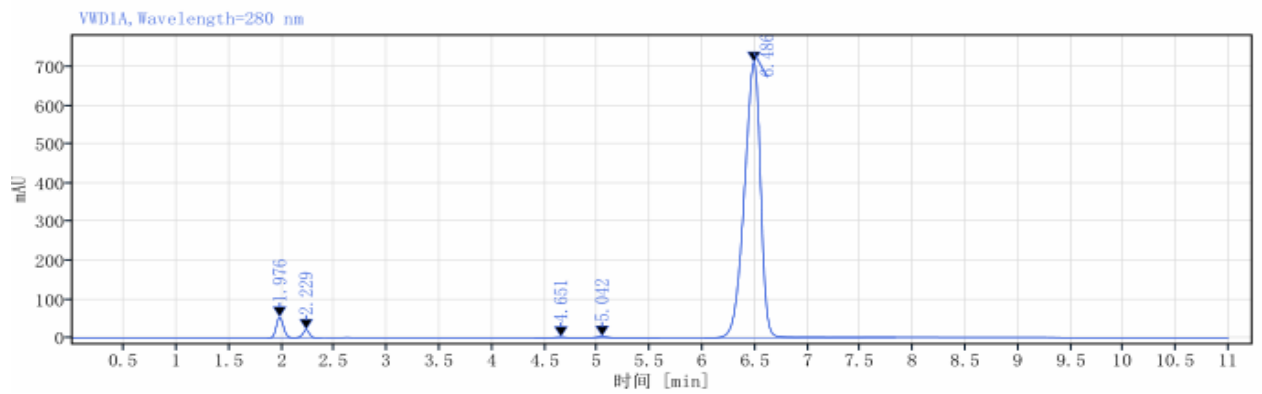


Fig. 2 Typical HPLC-Chromatogram of Propineb TC sample

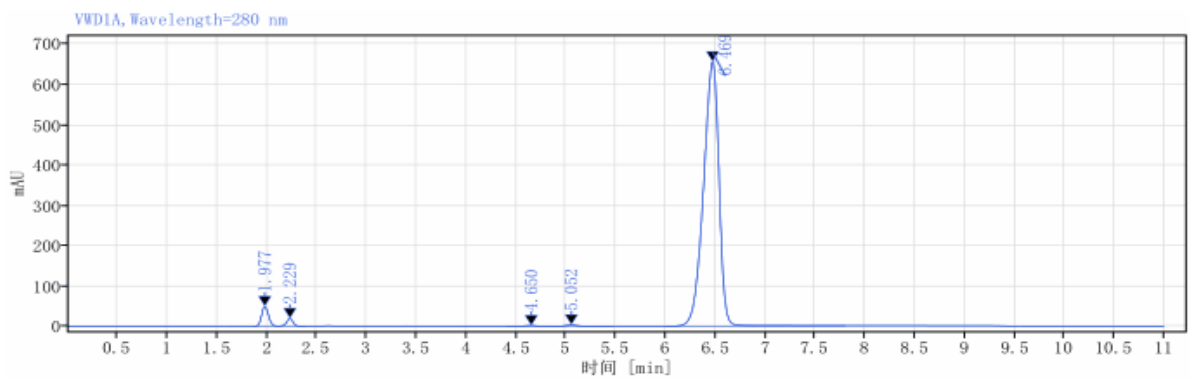


Fig. 3 Typical HPLC-Chromatogram of Propineb WP sample

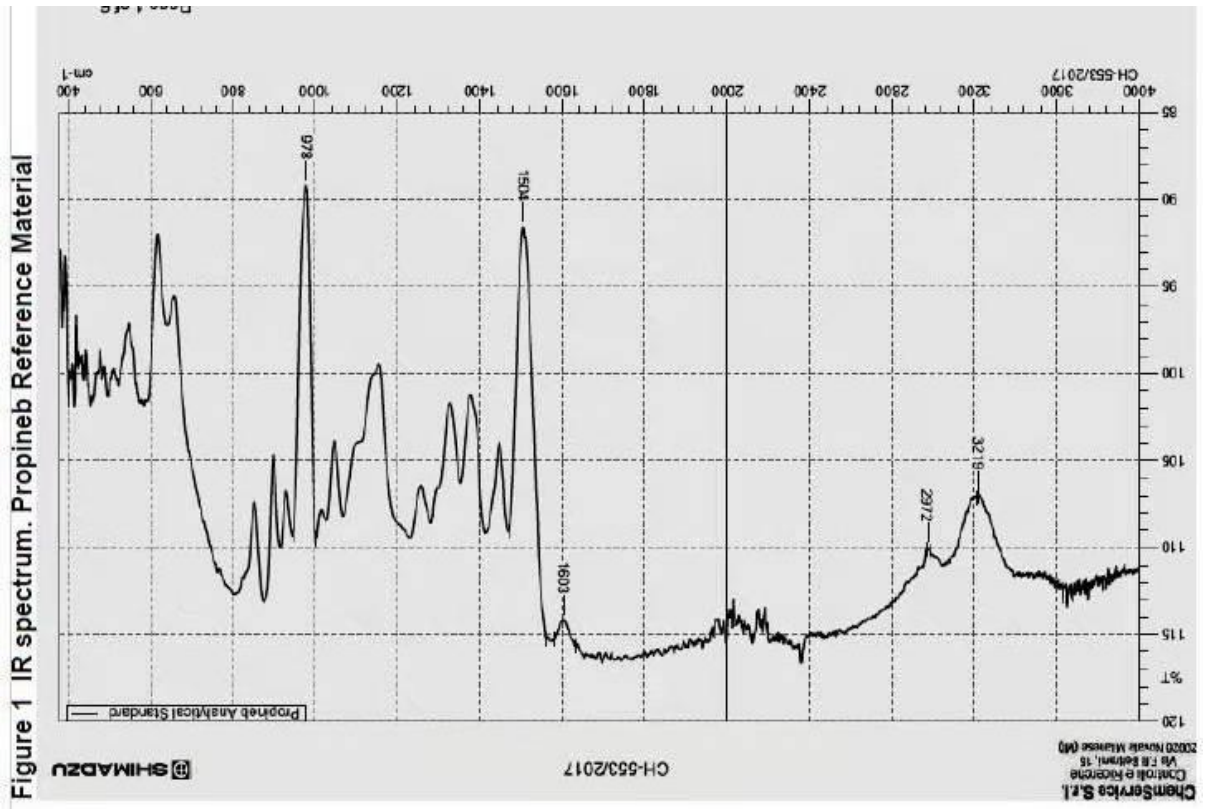


Fig. 4 Infrared spectra of Propineb