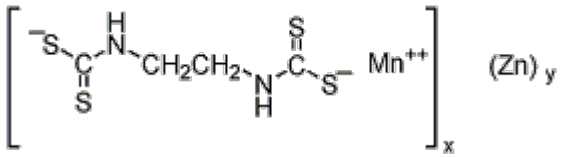
MANCOZEB

34



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ISO common name	Mancozeb
Chemical compositon	Co-ordination complex of zinc and maneb containing 20% manganese and 2.5% zinc
m.p.	Decomposes
v.p.	Negligible
Solubility	Insoluble in water and most organic solvents
Description	Yellow powder
Stability	Decomposed under acid conditions
Formulation	Wettable powders, water dispersible granules, suspension concentrates and dustable powders

MANCOZEB TECHNICAL
34/TC/M/-

1. **Sampling.** Take at least 200 g. Fill the bottles completely and store them (no longer than 4 weeks) at temperature below 20℃

2. Identity tests

2.1 HPLC

The identification test can be carried out simultaneously with the determination of mancozeb The relative retention time of the mancozeb peak in the sample solution should not differ by more than 1.5% from that of the calibration solution.
Chromatogram of Mancozeb standard see Fig 1
Chromatogram of Mancozeb sample see Fig 2

2.2 Infrared

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

3. Mancozeb.

OUTLINE OF METHOD
Mancozebwas determined by reversed phase high performance liquid chromatography using UV detector and external standardization.

REAGENTS

Water: de-ionized HPLC Grade
Methanol: HPLC grade
Disodium hydrogen phosphate: 98%
Sodium hydroxide solution:10%
Tetrabutyl ammonium hydrogen sulfate: 98%
Sodium Sulfite : 98.5%
EDTA: 99%
Mancozeb reference standard of known purity, store refrigerated
Mobile phase： buffer solution A
(a) Calibration solution.
Preparation of buffer solution A: Weigh 3.72g EDTA, 1.42g disodium hydrogen phosphate, and 3.39g tetrabutyl ammonium hydrogen sulfate in 1000mL flask to get an aqueous solution .Fill to the mark with water. Ensure concentration of EDTA, dipotassium hydrogen phosphate, and tetrabutyl ammonium hydrogen sulfate are 0.01mol/L separately. Adjust pH value to 9.5-10.0 with sodium hydroxide solution.
Preparation of solution B: Add 1g sodium sulfite into 1000ml buffer solution A.

Solution A and B cannot be used after 24 hours.
Preparation of standard solution: Weigh in duplicate accurately approximately100mg (accurate to 0.0002g) of mancozeb standard in 100mL flask, Add solution B about 30 ml and place the flask in an ultrasonic bath for 5 mins. Allow to cool the solution to ambient temperature, and fill to the mark with solution B. Mix thoroughly. Pipette accurately 1mL solution into 25mL volumetric flask. Dilute to the mark with solution B and Mix thoroughly.

APPARATUS

High-performance liquid chromatography equipped with a detector suitable for operation at 282nm, a constant-temperature column compartment and an injector capable of delivering 20 ul
Column stainless steel, 250mm × 4.6mm (i.d) columns, C18 (pH 9.5-10.0) packed with octadecyl silane filler，or equivalent
Ultrasonic Bath
Electronic integrator

PROCEDURE
(a) Liquid chromatographic conditions (typical)
Column: C18 pH 9.5-10.0 (250 mm x 4.6 mm) packed with octadecyl silane filler
Eluent: Methanol / buffer solution A (33:77 % v/v)
Column Temperature:29℃
Flow Rate: 0.9 ml/min^{-1}
Detector Wavelength：282nm
Injection Volumn: 20μL
Rention time: approximately 14min

(b) System equilibration
While the instrument is stabilized, several calibration standard injections before analysis until two adjacent injections mancozeb area relative changes less than 1.5% and absolute value of retention time less than 0.5min.

(c) Preparation of sample solution: Weigh (accurate to 0.0002g) accurately sufficient sample to contain about 100mg pure mancozeb into 100mL flask. Add solution B about 30 ml and place the flask in an ultrasonic bath for 5 mins. Allow to cool the solution to ambient temperature, and fill to the mark with solution B. Mix thoroughly. Pipette accurately 1mL solution into 25mL volumetric flask. Dilute to the mark with solution B and Mix thoroughly.

(d) Determination: Inject 20 μl portions of calibration solution before analysis until two adjacent injections mancozeb area relative changes less than 1.5%.
The analysis sequence each day should be as follows:
calibration solution, sample solution, sample solution, calibration standard

（e）Calculation

Calculate the percentage by content (ω 1) of Mancozeb as formula (1)

$$f_i = \frac{s \times P}{H_s} \quad \text{……（1）}$$

$$\text{mancozeb content} = \frac{f \times H_w}{w} \quad \text{g/kg}$$

where:

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

Repeatability r = 9 to 19 g/kg at 850kg/kg active ingredient content
Reproducibility R =15 to 24 g/kg at 850kg/kg active ingredient content

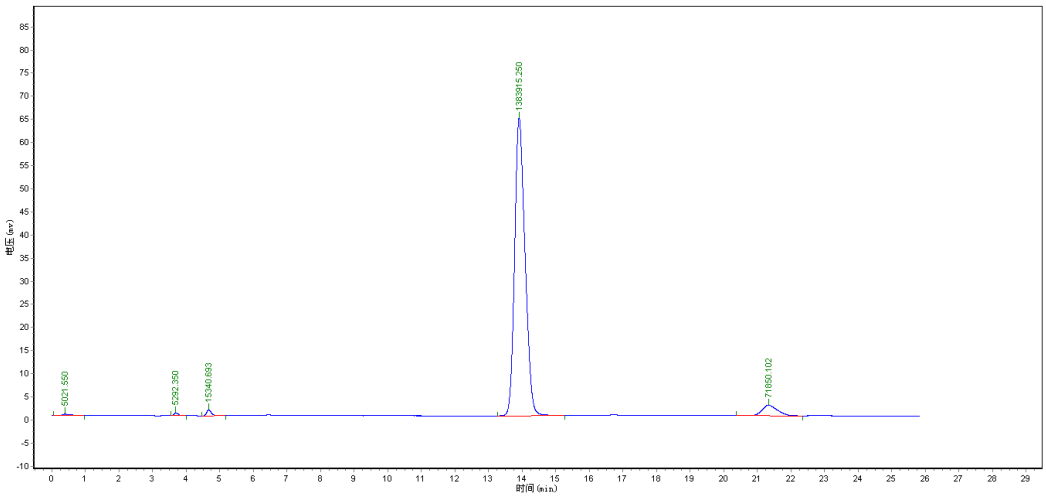

Fig 1 Chromatogram of Mancozeb standard

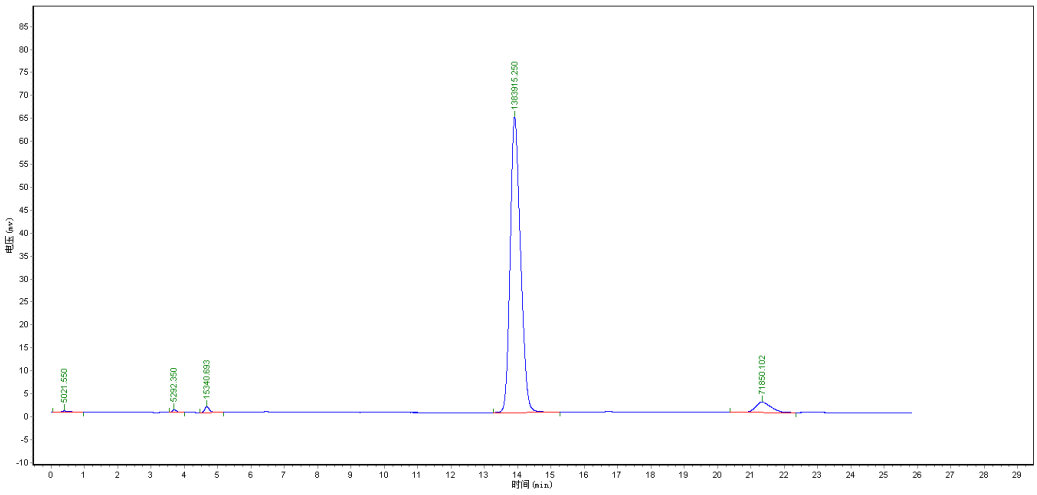

Fig 2 Chromatogram of Mancozeb sample

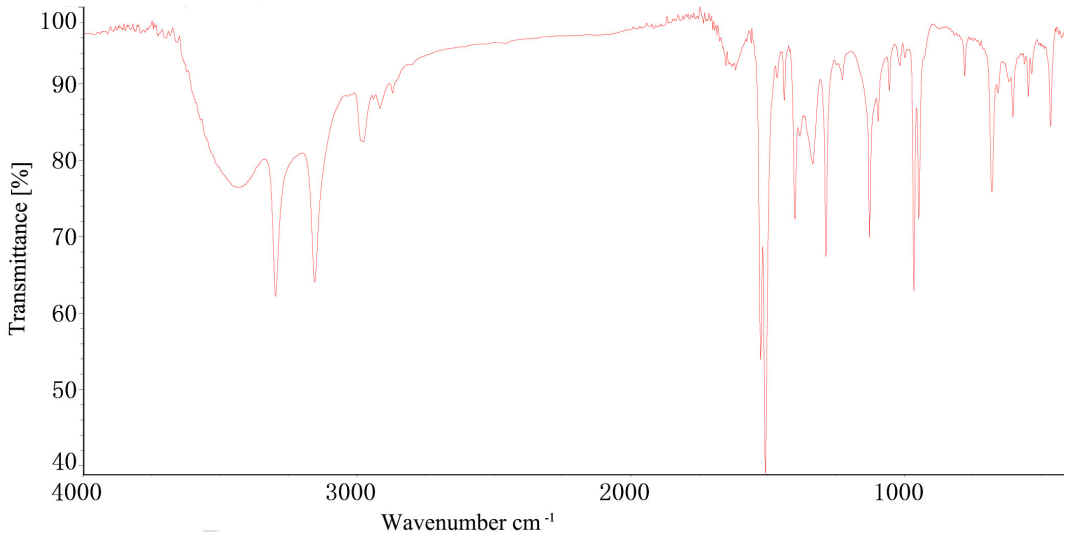

Fig 3 Infrared spectra of Mancozeb

MANCOZEB WETTABLE POWDERS

34/WP/M/-

1. **Sampling.** Take at least 500 g. Fill the bottles completely and store them (no longer than 4 weeks) at temperature below 20℃

2. **Identity tests.** As for mancozeb technical 34/TC/M/2

3. **Mancozeb.** As for mancozeb technical 34/TC/M/3

   Repeatability r= 9 to 19 g/kg at 830kg/kg active ingredient content
   Reproducibility R =15to24 g/kg at 830kg/kg active ingredient content

4. **Suspensibility**
   REAGENTS AND APPARATUS As for 34/TC/M and MT 184

(a) *Preparation of suspension ad determination of sedimentation.* MT 184.
(b) *Determination of Mancozeb in the bottom 25ml of suspension.* After removal of the top 225ml suspension transfer the 25ml remaining quantitatively to a volumetric flask (100ml) and dilute to volume with Solution B [as for 34/TC/M/3(a)], sonication until the solution is transparent, then do the measurement and calculation in accordance with the analysis of Mancozeb technical method.

(c)Calculation

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

where:

c = mass of mancozeb in the sample taken for the preparation of the suspension (g)
Q = mass of mancozeb in the bottom 25 ml of suspension (g)