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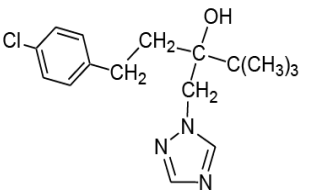
SEVEN CONTINENT

Tebuconazole



CIPAC Collaborative Trial
CIPAC 5345M, small scale study

CIPAC Collaborative Study of a
Gas Chromatographic Analysis of Tebuconazole Technical Material
and Formulated Products

By

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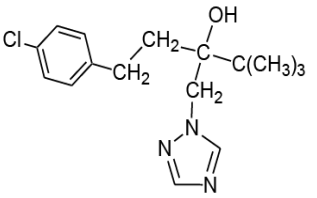
Presented at the CIPAC meeting in Braunschweig
June, 2023

Tebuconazole
494



ISO common name Tebuconazole

Chemical name (RS)-1-P-(4-chlorophenyl-4,4-dimethyl-3-(1*H*-1,2,4-triazole-ylmethyl)pentan-1-ol (IUPAC);
(±)α-[2-(4-chlorophenyl)ethyl]- α-(1,1-dimethylethyl)-1*H*-1,2,4-triazole-1-ethanol (CA)

CAS No	107534-96-3
Empirical formula	$C_{16}H_{22}ClN_3O$
RMM	307.8
Melting point	99.2±0.5°C (372±0.5K)
Vapor pressure	3.1×10^{-8} mPa (25ºC)
Density	1.25 (26°C)
Solubility	In water 32 g/L, (20ºC); In Methanol ＞250g/L, (25°C) In 1,2-dichloroethane ＞250g/L, (25°C)
Description	White powder.
Stability	Stable to elevated temperatures, and to photolysis and hydrolysis in pure water, under sterile conditions; hydrolysis DT50 >1 y (pH 4-9, 22 °C)

TEBUCONAZOLE TECHNICAL
494/TC/(M)/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 Infrared. prepare a film between potassium bromide plates and scan from 4000 to 400cm-1. The spectrum obtained from the sample should not differ significantly from that of the reference grade material. (Fig. 1)
2.2 GLC. Use the GLC method described below. The relative retention time of Tebuconazole in the sample solution should not deviate by more than 2% from that of the calibration solution.

3 Tebuconazole

OUTLINE OF THE METHOD.
The content of Tebuconazole (g/kg) is determined by capillary gas chromatography with split injection, using dicyclohexyl phthalate as internal standard.

3.1 Determination of Tebuconazole by gas chromatography

REAGENTS

Tebuconazole reference standard, of known content

Dicyclohexyl phthalate Internal standard. Must not contain impurities with the same retention time as Tebuconazole.

Acetone reagent grade≥99.5%.

Internal standard solution. Prepare a single stock of 6mg/ml internal standard solution, of sufficient volume for all samples to be analyzed. For example, to prepare 250ml stock solution, dissolve 1.5g (to the nearest 0.01 g) dicyclohexyl phthalate in acetone in a volumetric flask (250 ml) and make up to the mark with the same solvent.

Calibration solutions C1 and C2. Weigh in duplicate (to the nearest 0.01 mg) approximately 50mg (s in mg) of the Tebuconazole reference standard into

separate suitable vessels. Add by pipette internal standard solution (10 ml). Mix thoroughly. (calibration solutions C1, C2, chromatogram of C1 see Fig. 2).

APPARATUS

Gas chromatograph equipped with a split/splitless injection and a flame ionisation detector.

Capillary column, fused silica, 30 m x 0.32 (i.d.) mm, with a HP-5 bounded phase and a film thickness of 0.25 µm, or equivalent with the same selectivity.

Electronic integrator or data system

Ultrasonic bath

PROCEDURE

(a) Operating conditions (typical):

Injector type split injection

Split Ratio 20:1

Injection volume 0.2 µL

Injector temperature 280 °C

Detector type flame ionization

Detector temperature 300 °C

Oven temperature 240 °C hold 8min, ramp rate15°C/min, to 260°C, hold 4 min

Flowrates carrier gas helium: 2mL/min

make-up gas helium: 25 mL/min

air 400 mL/min

hydrogen 40 mL/min

Running time 14 minutes

Retention time Tebuconazole: approx. 4.8 min

dicyclohexyl phthalate: approx. 6.3min

(b) System equilibration. Pump sufficient carrier gas through the column to equilibrate the system. Inject 0.2 µL portions of the calibration solution C1 (see below) and repeat the injections until retention times and peak areas deviate by less than ± 1 % from the mean for three successive injections.

(c) Preparation of sample solution. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.01 mg) sufficient sample (*w* in mg) (containing approximately 50 mg of Tebuconazole) into separate suitable vessels. Add by pipette internal standard solution (10 ml). Mix thoroughly. (Sample solutions S1, S2, chromatogram of S1 see Fig. 3)

(d) Determination. Inject in duplicate each sample solution and bracket a series of sample solution injections by injections of the calibration solution as follows: calibration solution C1, calibration solution C2, calibration solution C1, sample solution S1, sample solution S1, calibration solution C1, sample solution S2, sample solution S2, calibration solution C1 ... (C1, C2, C1, S1, S1, C1, S2, S2, C1 …)

Determine the peak areas of Tebuconazole and dicyclohexyl phthalate.

(e) Calculation. Calculate the mean value of each pair of calibration response factors, bracketing the two injections of a sample, and use this value for calculating the Tebuconazole contents of the bracketed sample injections.

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

Content of Tebuconazole = $\frac{f \times H_W}{I_q \times w}$ g/kg

where:

f_i = individual response factor

f = mean response factor

H_s = peak area of Tebuconazole in the calibration solution

H_w = peak area of Tebuconazole in the sample solution

I_r = peak area of the internal standard in the calibration solution

I_q = peak area of the internal standard in the sample solutions

s = mass of Tebuconazole reference standard in the calibration solution (mg)

w = mass of sample taken (mg)

P = purity of Tebuconazole reference standard (g/kg)

TEBUCONAZOLE WETTABLE POWDER
494/WP/（M）/-

1 Sampling. Take at least 100 g.

2 Identity tests.
2.1 GLC. As for tebuconazole 494/TC/M/-
2.2 GC-MS. As for tebuconazole 494/TC/M/-

3 tebuconazole.
Same approach as for tebuconazole 494/TC/M/-

3.1 Determination of tebuconazole by gas chromatography
As for tebuconazole 494/TC/M/- in addition:

Disposable PTFE syringe filter compatible with organic solvents and a 0.45 µm pore diameter or centrifuge.

PROCEDURE

(c) Preparation of sample solution. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.01 mg) sufficient sample (*w* in mg) (containing approximately 50 mg of tebuconazole) into separate suitable vessels. Add by pipette internal standard solution (10 ml). Mix thoroughly. Clarify a part of the solution by centrifugation or filtration prior to analysis.

(sample solutions S3, S4, chromatogram of S3 see Fig. 4).

4. Suspensibility

REAGENTS and APPARATUS as for CIPAC MT 184.1

PROCEDURE

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

(b) Determination of tebuconazole in the bottom 25 ml of suspension:

After removal of the top 225 ml of suspension, add 10 ml internal standard solution (dipentylphthalate) and 20 ml trichloromethane, homogenize well and let stand undisturbed for phase separation. Take the lower layer (subnatant) for centrifuge and further analysis to determine the content of Tebuconazole in the suspension.

Determine the mass of Tebuconazole, in [g]) by 494/M

*This is calculated for a 0.5% suspension; in case of other concentrations the volume has to be adjusted but it has to be ensured to work within the linear range.

TEBUCONAZOLE SUSPENSION CONCENTRATE
494/SC/M/-

1 Sampling. Take at least 500 mL. Shake the sample well before weighing.

2 Identity tests.
2.1 GLC. As for tebuconazole 494/TC/M/-
2.2 GC-MS. As for tebuconazole 494/TC/M/-

3 tebuconazole.
Same approach as for tebuconazole 494/TC/M/-

3.1 Determination of tebuconazole by gas chromatography
As for tebuconazole 494/TC/M/- in addition:

Calibration solutions C1 and C2. Weigh in duplicate (to the nearest 0.01 mg) approximately 50 mg (*s* in mg) of the tebuconazole reference standard into separate suitable vessels. Add by pipette internal standard solution (10 ml). Mix thoroughly. (calibration solutions C1, C2, chromatogram of C1 see Fig. 2).

Disposable PTFE syringe filter compatible with organic solvents and a 0.45 µm pore diameter or centrifuge.

PROCEDURE
(c) Preparation of sample solution. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.01 mg) sufficient sample (*w* in mg) (containing approximately 50 mg of tebuconazole) into separate suitable vessels. Add by pipette internal standard solution (10 ml).Mix thoroughly.
(sample solutions S5, S6, chromatogram of S5 see Fig. 5).

4. Suspensibility
Same approach as for Tebuconazole 494/WP/M

TEBUCONAZOLE EMULSIFIABLE CONCENTRATE
494/WP/M/-

1 Sampling. Take at least 500 mL. Shake the sample well before weighing.

2 Identity tests.
2.1 GLC. As for tebuconazole 494/TC/M/-
2.2 GC-MS. As for tebuconazole 494/TC/M/-

3 tebuconzaole.
Same approach as for tebuconazole 494/TC/M/-

3.1 Determination of tebuconazole by gas chromatography
As for tebuconazole 494/TC/M/- in addition:

Calibration solutions C1 and C2. Weigh in duplicate (to the nearest 0.01 mg) approximately 50 mg (*s* in mg) of the tebuconazole reference standard into separate suitable vessels. Add by pipette internal standard solution (10 ml).Mix thoroughly. (calibration solutions C1, C2, chromatogram of C1 see Fig. 2).

PROCEDURE
(c) Preparation of sample solution. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.01 mg) sufficient sample (*w* in mg) (containing approximately 50 mg of tebuconazole) into separate suitable vessels. Add by pipette internal standard solution (10 ml).Mix thoroughly.

(sample solutions S7, S8, chromatogram of S7 see Fig. 6).

4. Suspensibility
Same approach as for Tebuconazole 494/WP/M

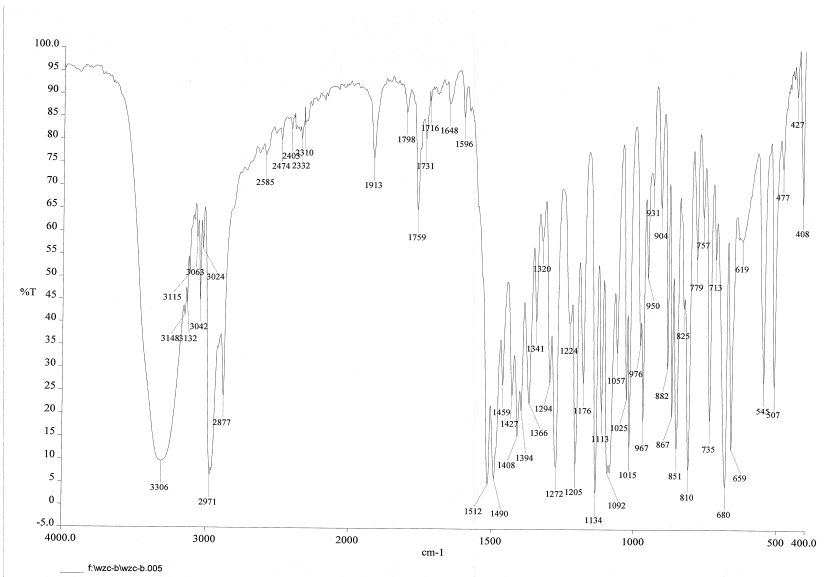


Fig. 1 Typical IR spectrum of Tebuconazole

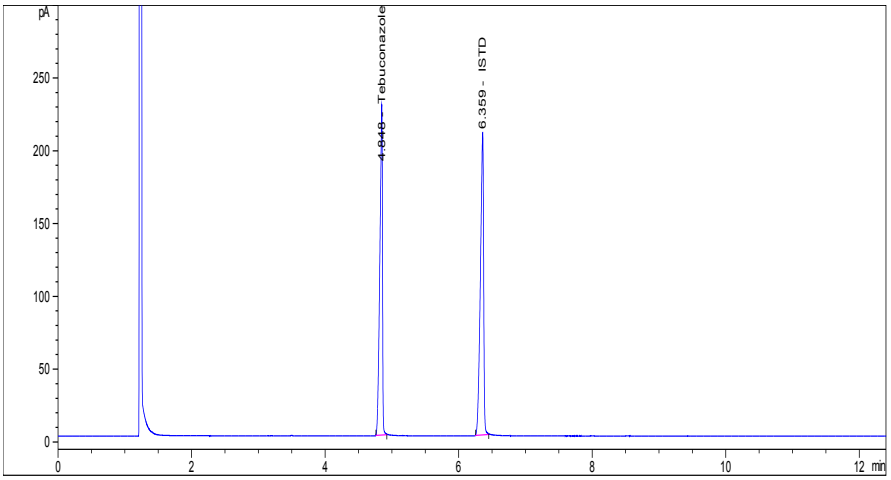


Fig. 2 Analytical Standard Tebuconazole

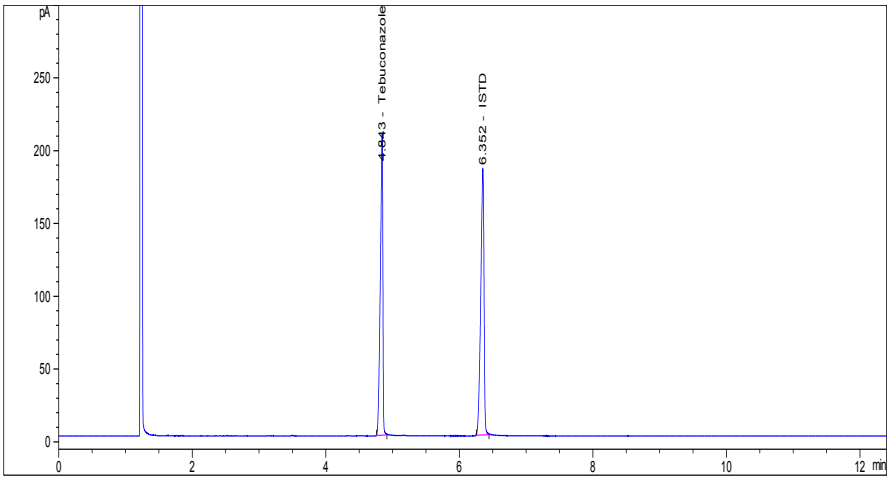


Fig. 3 Technical Material TC

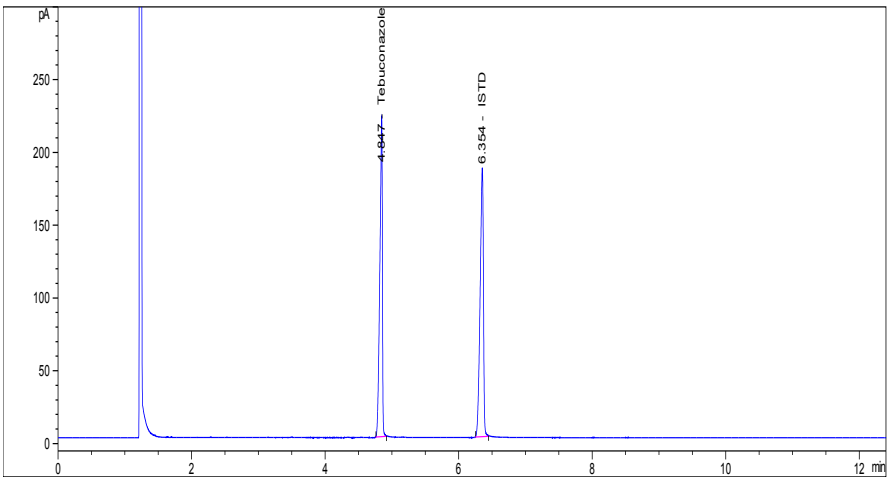


Fig. 4 Wettable powder WP

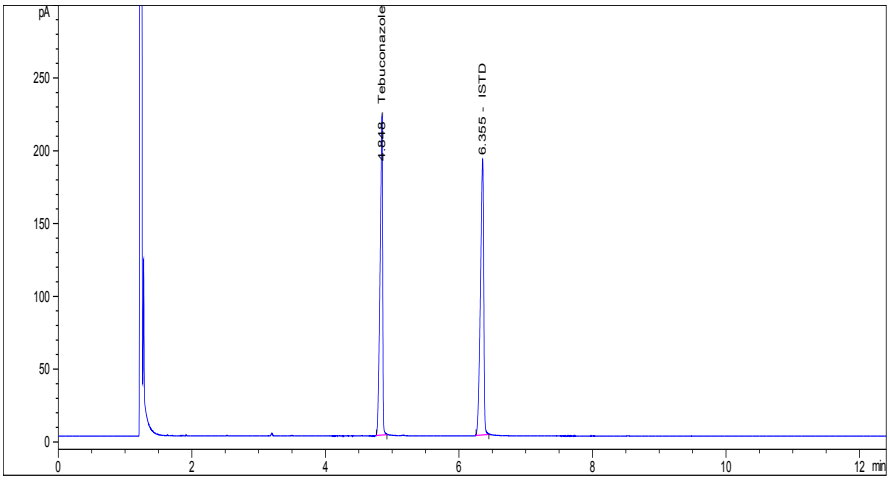


Fig. 5 Suspension Concentrate SC

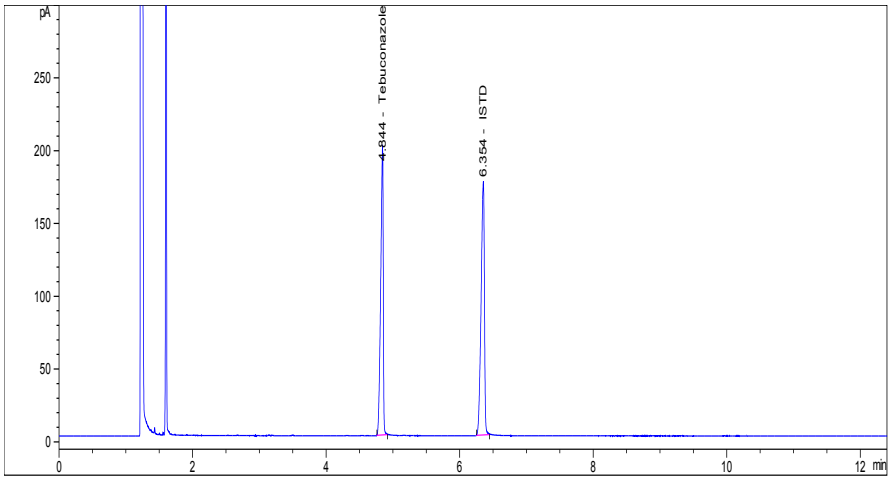


Fig. 6 Emulsifiable Concentrate EC