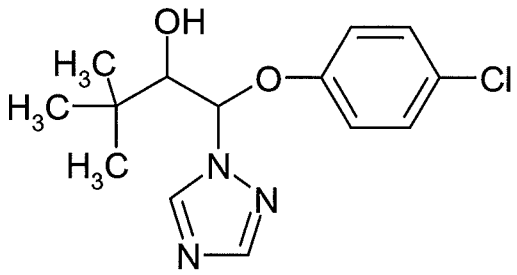
Bayer CropScience

Triadimenol
398
GLC method
Method Extension



ISO common name:	triadimenol
CAS index name:	1H-1,2,4-Triazole-1-ethanol, .beta.-(4-chlorophenoxy)-.alpha.-(1,1-dimethylethyl)- (9Cl)
IUPAC Name:	1-(4-Chlorophenoxy)-3,3-dimethyl-1-[1,2,4]triazol-1-yl-1butan-2-ol (unstated stereochemistry)
CAS-No.:	55219-65-3
Empirical formula:	$C_{14}H_{18}ClN_3O_2$
RMM:	295.8 g/mol
Note:	Triadimenol is a diastereomeric mixture (A(threo) : B(erythro) = 7 : 3); A = (1R,2S)- + (1S,2R)-enantiomer (CAS-No. 89482-17-7); B = (1R,2R)- + (1S,2S)-enantiomer (CAS-No. 82200-72-4)
m.p.:	diastereomeric form A: 138.2 °C, B: 133.5 °C
Solubility at 20 °C:	dichlormethane 100 - 200 g/L; acetone 190 g/L; toluene 10 – 30 g/L
Description:	Form: white powder
Formulations:	WP, EC, DC, GR, WG, SC, FS and EW.

Triadimenol
398/TC/(M)/-

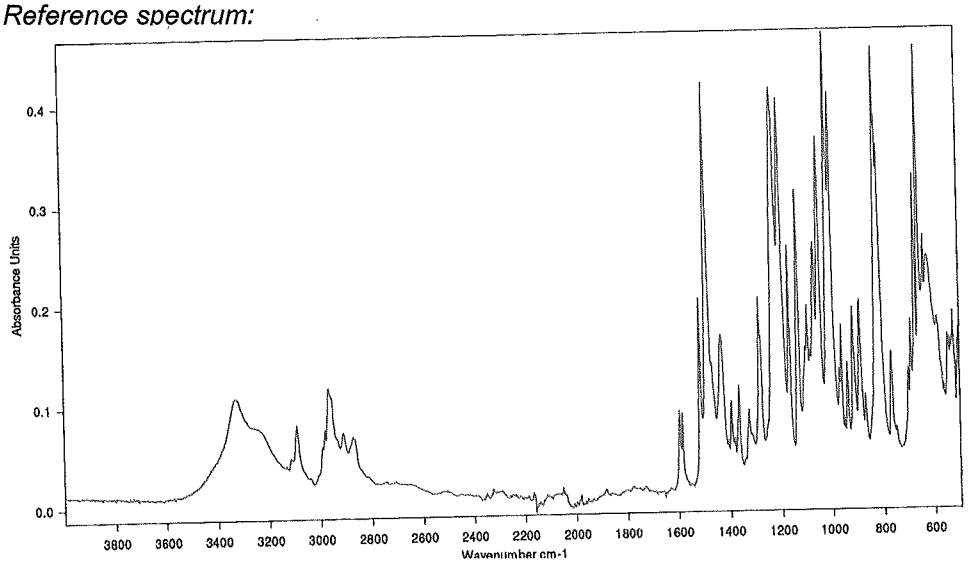
1 Sampling. Take at least 100 g.

2 Identity tests.

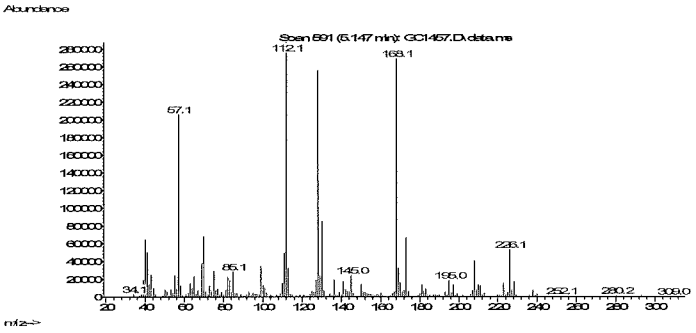
2.1 GLC.
Use the GLC method below. The difference between the retention times of triadimenol and the internal standard for the sample solution should not deviate by more than 10 s from that for calibration solution.

2.2 Infrared spectroscopy.
FT-IR Infrared Spectrometer with ATR accessories (e.g. Diamond ATR)
Disperse the test substance homogeneously on the crystal and record the IR spectrum in the range 4000 - 600 cm^{-1}. Compare it qualitatively with the reference spectrum. The test is considered to be positive when the spectrum is qualitatively identical with the reference spectrum.

Reference spectrum:



2.3 Mass spectrometry (GC-MS).
Use the sample preparation below. Record the MS spektrum using GC-MS and compare it qualitatively with the reference spectrum. The test is considered to be positive when the spectrum is identical with the reference spectrum.
Reference spectrum:



3 Triadimenol
OUTLINE OF METHOD
Triadimenol is dissolved in, or extracted with acetone, di-(2-ethylhexyl) phthalate is added as internal standard, and the content of triadimenol is determined by capillary gas chromatography with on-column cold injection. Alternatively to on-column cold injection split injection is applicable when the temperature program is adjusted appropriately.

REAGENTS
Acetone
Triadimenol standard of known purity, at least 950 g/kg
Di-(2-ethylhexyl) phthalate internal standard
Calibration solution. Prepare in duplicate calibration solutions. Weigh (to the nearest 0.1 mg) about 200 mg of the of triadimenol standard (s mg) and 200 mg of di-(2-ethylhexyl) phthalate (*r* mg) into a 20 ml volumetric flask, fill to the mark with acetone and dissolve. Transfer with a pipette exactly 1.0 ml of the solution to a 100 ml volumetric flask, fill to the mark with acetone and homogenize.

APPARATUS
Gaschromatograph with flame ionization detector and on-column capillary column accessory (alternatively split injector)
Electronic integrator or lab data system
Column Quartz capillary, 30 m x 0.53 (i.d.) mm, coated with silicone ODER 1.1 µm.
Ultrasonic bath

PROCEDURE
(a) Praparation of sample solution. Weigh (to the nearest 0.1 mg) about 200 mg of sample (*w* mg) into a 20 ml volurimetric flask, add 200 mg of internal standard di-(2-ethylhexyl) phthalate (*q* mg), fill to the mark with acetone and dissolve for 10 min using an ultrasonic bath. Transfer with a pipette exactly 1.0 ml of the solution to a 100 ml volumetric flask, fill to the mark with acetone and homogenize. Prepare two solutions for each sample.

(b) Operating conditions (typical)

Column temperature	2 min at 80°C, then 10°C/min up to 280 °C
Injection port temperature	*cold on column injection*
Detector temperature	280 °C
Flow rate carrier gas	*helium: 5 ml/ min*
Flow rate make up gas	*nitrogen: 30 ml/ min*
Flow rate air and hydrogen	*as recommended by the manufacturer*
Retention time	*triadimenol: about 18 min*
	di-(2-ethylhexyl) phthalate: about 23 min

(c) Determination. Inject at least two times 1 µl aliquots of each calibration solution and calculate the single response factors f_i using the formula below. The individual values should agree within 2 %, otherwise repeat the calibration. If the response is satisfactory, inject 1 µl aliquots of each sample solution bracketing them by injections of the calibration solutions as follows: calibration solution 1, sample solution1, sample solution 2, calibration solution 2. Measure the relevant peak areas. Calculate the mean value *f* of each pair of response factors bracketing two sample injections and use this value for evaluating the two bracketed sample runs.

(d) Calculation

$$\text{Response factor } f_i = \frac{I_r \times P \times s}{H_s \times r}$$

Where:

f_i	= single response factors of triadimenol
I_r	= peak area of the internal standard in the calibration solution i
P	= purity of triadimenol standard, in g/kg
s	= mass of triadimenol in the calibration solution i (mg)
H_s	= peak area of triadimenol in the calibration solution i
r	= mass of the internal standard in the calibration solution i (mg)

$$\text{Triadimenol content} = \frac{f \times H_w \times q}{I_q \times w} \text{ g/kg}$$

Where:

f	= average response factor
H_w	= *peak area of triadimenol in the sample solution*
q	= *mass of internal standard in the sample solution (mg)*
I_q	= *peak area of internal standard in the sample solution*
w	= *mass of sample taken (mg)*

The content of triadimenol (sum of both diastereometric forms) is the mean value of the results of the solutions.

Repeatability r_{95} = 12.4 g/kg at 967 g/kg active ingredient content
Reproducibility R_{95} = 26.5 g/kg active ingredient content

Triadimenol Wettable Powder

398/WP/(M)/-

1 Sampling. Take at least 500 g.

2 Identity tests.
2.1 GLC. As for 398/TC/(M)/2.1.
2.2 Infrared

REAGENTS and APPARATUS
Tetrachlormethan
Ultrasonic bath
Centrifuge 3000 rpm

PROCEDURE
Weigh an amount of the formulation containing about 50 mg of active ingredient into a 10 ml flask and add tetrachlormethane. Sonificate the suspension for 15 min then centrifuge the suspension and apply a portion of the clear supernatant uniformly to the crystal. Verify the identity by comparing the spectrum with those of triadimenol standard.

2.3 Mass spectrometry (GC-MS). As for 398/TC/(M)/2.3

3 Triadimenol. As for 398/TC/(M)/3 except:
(a) Preparation of sample solutions. Homogenize the sample and weigh (to the nearest 0.1 mg) into a 20 ml volumetric flask enough sample (*w* mg) to contain about 200 mg of pure triadimenol, add 200 mg of internal standard (*q* mg). Fill to the mark with acetone and extract for 30 min in an ultrasonic bath. Transfer with a pipette exactly 1.0 ml of the solution to 100 ml volumetric flask, fill to the mark with acetone and homogenize. Centrifuge the suspension and inject 1 µl aliquots of the supernatant liquid. The concentration of triadimenol should be similar to its concentration in the calibration solution. Prepare two solutions of each sample.

Repeatability r_{95}	= 3.29 g/kg at 148 g/kg active
Reproducibility R_{95}	= 5.09 g/kg at 148 g/kg active

4 Suspensibility.
(a) Preparation of suspension according MT 15.1 / MT 184 (i)
(b) Determination of sedimentation according MT 15.1/ MT 184 (ii)
(c) Determination of triadimenol in the bottom 25 ml suspension. After removal of the top 225 ml of suspension add to the remaining 25 ml in the cylinder 75 ml of N,N-dimethylacetamid containing an amount of internal standard that corresponds to the amount of active ingredient. Analyze according to the method 398/TC/M/3 using an injection port temperature of 160 °C.

Triadimenol Emulsifiable Concentrate
398/EC/(M)/-

1 Sampling. Take at least 500 ml.

2 Identity tests.
2.1 GLC. As for 398/TC/(M)/2.1.
2.2 Mass spectrometry (GC-MS). As for 398/TC/(M)/2.1.

3 Triadimenol. As for 398/TC/(M)/3 except:
(a) Preparation of sample solutions. Thoroughly shake the sample container to ensure that the emulsion is homogeneous. Immediately weigh (to the nearest 0.1 mg) enough sample (*w* mg) to contain about 200 mg of pure triadimenol into a 20 ml volumetric flask, add 200 mg of internal standard (*q* mg). Fill to the mark with acetone and dissolve for 10 min in an ultrasonic bath.. Transfer with a pipette exactly 1.0 ml of the solution to 100 ml volumetric flask, fill to the mark with acetone and homogenize. Inject 1 µl aliquots of the solution. The concentration of triadimenol should be similar to its concentration in the calibration solution. Prepare two solutions of each sample.

Repeatability r_{95} = 4.6 g/kg at 217 g/kg active ingredient content
Reproducibility R_{95} = 10.0 g/kg at 217 g/kg active ingredient content

Formatiert: Standard, Tabstopps: Nicht an 1 cm

Triadimefon Water Dispersable Granules
398/WG/(M)/-

1 Sampling. Take at least 500 g.

2 Identity tests.
2.1 GLC. As for 398/TC/(M)/2.1.
2.2 Infrared As for 398/WP/(M)/2.2
2.3 Mass spectrometry (GC-MS). As for 398/TC/(M)/2.1.

3 Triadimenol. As for 398/TC/(M)/3 except:
(a) Preparation of sample solutions. Homogenize the sample and weigh (to the nearest 0.1 mg) into a 20 ml volumetric flask enough sample (*w* mg) to contain about 200 mg of pure triadimenol, add 200 mg of internal standard (*q* mg). Fill to the mark with acetone and extract for 30 min in an ultrasonic bath. Transfer with a pipette exactly 1.0 ml of the solution to 100 ml volumetric flask, fill to the mark with acetone and homogenize. Centrifuge the suspension and inject 1 µl aliquots of the supernatant liqid. The concentration of triadimenol should be similar to its concentration in the calibration solution. Prepare two solutions of each sample.

4 Suspensibility.
(a) Preparation of suspension according MT 15.1 / MT 184 (i)
(b) Determination of sedimentation according MT 15.1/ MT 184 (ii)
(c) Determination of triadimenol in the bottom 25 ml suspension. After removal of the top 225 ml of suspension add to the remaining 25 ml in the cylinder 75 ml of N,N-dimethylacetamid containing an amount of internal standard that corresponds to the amount of active ingredient. Analyze according to the method 398/TC/M/3 using an injection port temperature of 160 °C.

Triadimenol Suspension Concentrates
398/SC/(M)/-

1 Sampling. Take at least 500 g.

2 Identity tests.
2.1 GLC. As for 398/TC/(M)/2.1.
2.2 Infrared. As for 398/WP/2.2
2.3 Mass spectrometry (GC-MS). As for 398/TC/(M)/2.1.

3 Triadimenol. As for 398/TC/(M)/3 except:
(a) Preparation of sample solutions. Thoroughly shake the sample container to ensure that the suspension is homogeneous. Immediately weigh (to the nearest 0.1 mg) into a 20 ml volumetric flask enough sample (*w* mg) to contain about 200 mg of pure triadimenol, add 200 mg of internal standard (*q* mg). Fill to the mark with acetone and extract for 30 min in an ultrasonic bath. Transfer with a pipette exactly 1.0 ml of the solution to 100 ml volumetric flask, fill to the mark with acetone and homogenize. Centrifuge the suspension and inject 1 µl aliquots of the supernatant liquid. The concentration of triadimenol should be similar to its concentration in the calibration solution. Prepare two solutions of each sample.

4 Suspensibility.
(a) Preparation of suspension according MT 161/MT 184 (i)
(b) Determination of sedimentation according MT 161 / MT 184 (ii)
(c) Determination of triadimenol in the bottom 25 ml suspension. After removal of the top 225 ml of suspension add to the remaining 25 ml in the cylinder 75 ml of N,N-dimethylacetamid containing an amount of internal standard that corresponds to the amount of active ingredient. Analyze according to the method 398/TC/M/3 using an injection port temperature of 160 °C.

Triadimenol Flowable Concentrate for Seed Treatment
398/FS/(M)/-

1 Sampling. Take at least 500 g.

2 Identity tests.
2.1 GLC. As for 398/TC/(M)/2.1.
2.2 Infrared As for 398/WP/2.2
2.3 Mass spectrometry (GC-MS). As for 398/TC/(M)/2.1.

3 Triadimenol. As for 398/TC/(M)/3 except:
(a) Preparation of sample solutions. Thoroughly shake the sample container to ensure that the suspension is homogeneous. Immediately weigh (to the nearest 0.1 mg) into a 20 ml volumetric flask enough sample (*w* mg) to contain about 200 mg of pure triadimenol, add 200 mg of internal standard (*q* mg). Fill to the mark with acetone and extract for 30 min in an ultrasonic bath. Transfer with a pipette exactly 1.0 ml of the solution to 100 ml volumetric flask, fill to the mark with acetone and homogenize. Centrifuge the suspension and inject 1 µl aliquots of the supernatant liqid. The concentration of triadimenol should be similar to its concentration in the calibration solution. Prepare two solutions of each sample.

4 Suspensibility.
(a) Preparation of suspension according MT 161/MT 184 (i)
(b) Determination of sedimentation according MT 161 / MT 184 (ii)
(c) Determination of triadimenol in the bottom 25 ml suspension. After removal of the top 225 ml of suspension add to the remaining 25 ml in the cylinder 75 ml of N,N-dimethylacetamid containing an amount of internal standard that corresponds to the amount of active ingredient. Analyze according to the method 398/TC/M/3 using an injection port temperature of 160 °C.

Triadimenol Emulsion, Oil in Water
398/EW/(M)/-

1 Sampling. Take at least 500 ml.

2 Identity tests.
2.1 GLC. As for 398/TC/(M)/2.1.
2.2 Mass spectrometry (GC-MS). As for 398/TC/(M)/2.1.

3 Triadimenol. As for 398/TC/(M)/3 except:
(a) Preparation of sample solutions. Thoroughly shake the sample container to ensure that the emulsion is homogeneous. Immediately weigh (to the nearest 0.1 mg) enough sample (*w* mg) to contain about 200 mg of pure triadimenol into a 20 ml volumetric flask, add 200 mg of internal standard (*q* mg). Fill to the mark with acetone and dissolve for 10 min in an ultrasonic bath. Transfer with a pipette exactly 1.0 ml of the solution to 100 ml volumetric flask, fill to the mark with acetone and homogenize. Inject 1 µl aliquots of the solution. The concentration of triadimenol should be similar to its concentration in the calibration solution. Prepare two solutions of each sample.