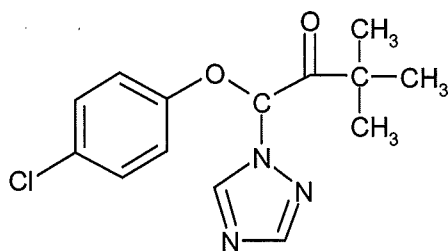


Bayer CropScience



# Triadimefon

## 352



*ISO common name:* triadimefon

*CAS index name:* 2-butanone, 1-(4-chlorophenoxy)-3,3-dimethyl-1-(1H-1,2,4-triazol-1-yl)- (9Cl)

*IUPAC Name:* 1-(4-chlorophenoxy)-3,3-dimethyl-1-(1H-1,2,4-triazol-1-yl)butan-2-one

*CAS-No.:* 43121-43-3

*Empirical formula:* C<sub>14</sub>H<sub>16</sub>ClN<sub>3</sub>O<sub>2</sub>

*RMM:* 293.7 g/mol

*Remark:* Racemat

*m.p.:* 82.3 °C

*Solubility at 20 °C:* In water: 0.07 g/l, 2-propanol: 100 – 200 g/l; toluene: 400 – 600 g/l

*Description:* Form: white powder

*Formulations:* WP, EC, WG, GR

**Triadimefon**

## 352/TC/(M)/-

**1 Sampling.** Take at least 100 g.

**2 Identity tests.**

**2.1 Infrared spectroscopy.**

(a) Technique: KBr disc

Prepare KBr disc using 1 mg of sample and 200 mg of KBr and also using a standard triadimefon. Scan the disc from 4000 to 400  $\text{cm}^{-1}$  (2.50 to 25  $\mu\text{m}$ ). The spectrum produced from the sample disc should not differ significantly from that from the standard

*Reference spectrum:*

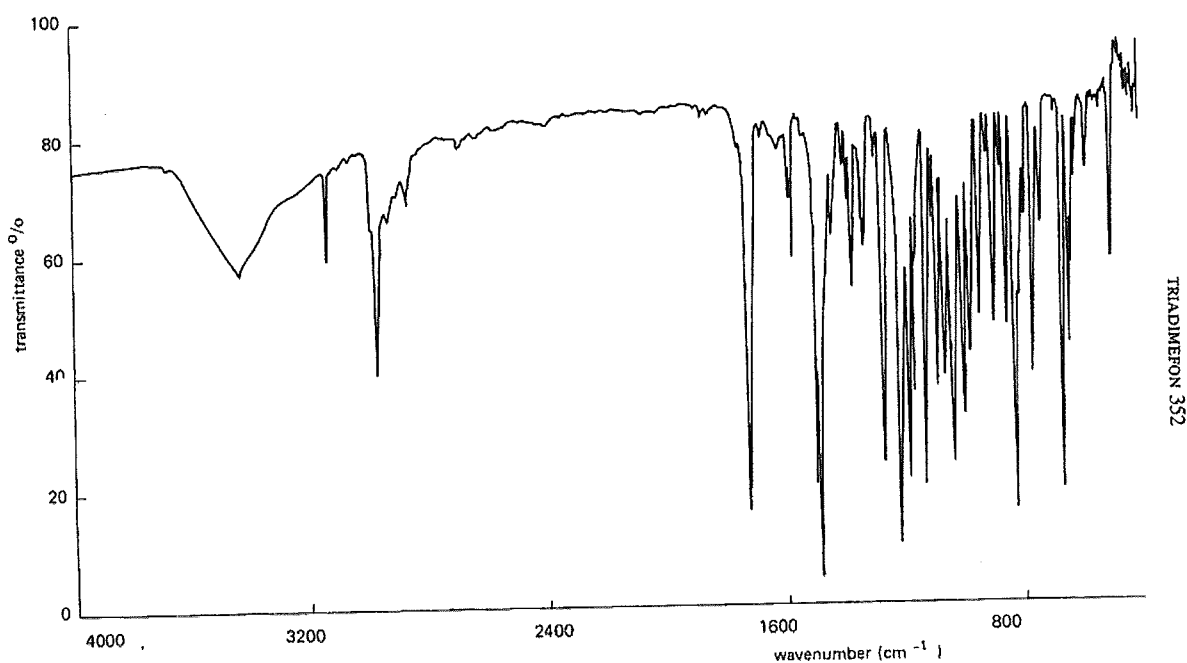
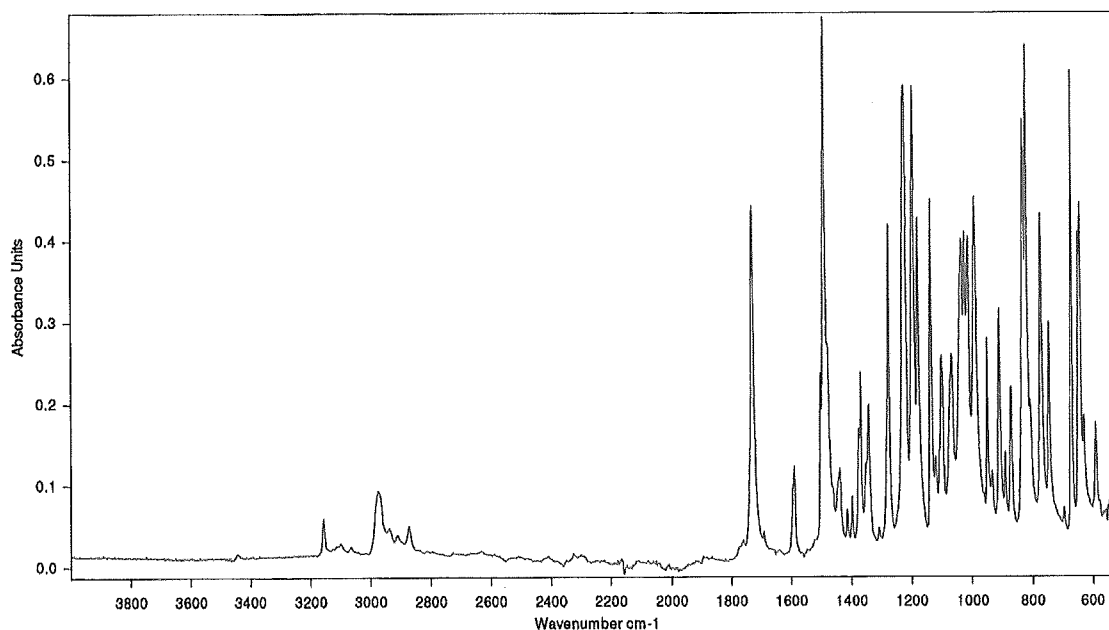


Fig. 138 IR spectrum of triadimefon technical (KBr disc).

(b) Technique: ATR, single reflection

Record the IR spectrum of the test substance in the range 4000 - 600  $\text{cm}^{-1}$  and 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:*

**2.2 HPLC.** The identity is checked simultaneously with the determination of triadimefon and is confirmed if the retention time of triadimefon for the sample solution does not deviate by more than 1.5 % from that for the calibration solution.

### 3 Triadimefon

#### OUTLINE OF METHOD

Triadimefon is dissolved in acetonitrile-water and separated by high performance chromatography on a 5  $\mu\text{m}$  Spherisorb-ODS-column with acetonitrile-water (49+51) (v/v) and UV-detection (276 nm). The active ingredient content is determined from peak areas using an external standard.

#### REAGENTS

*Acetonitrile* HPLC grade

*Water* HPLC grade

*Eluant* 49 % (v/v) acetonitrile / 51 % (v/v) water (degas before use)

*Triadimefon* standard of known purity

*Calibration solution.* Weigh (to the nearest 0.1 mg) about 60 mg of the of triadimenol standard into a 50 mL volumetric flask, dissolve and made up to volume with the eluant.

#### APPARATUS

*High performance liquid chromatograph* equipped with an ultraviolet spectrophotometric detector suitable for operation at 276 nm and an injection system capable to inject 20  $\mu\text{l}$ .

*Electronic integrator or data system*

*Column.* Stainless steel, length 125 mm, internal diameter 4 mm, packed with Spherisorb ODS 5  $\mu\text{m}$  or any commercially available column with at least 5000 theoretical plates (Hypersil ODS was also tested) .

*Ultrasonic bath*

#### PROCEDURE

**(a) Chromatographic conditions (typical)**

<i>Eluant flow rate:</i>	2 ml/min.
<i>Column temperature</i>	40 °C
<i>Injection volume</i>	20 µL
<i>Detector wavelength</i>	276 nm
<i>Run time</i>	approximately 8 min
<i>Retention time</i>	approximately 2.7 min (adjust the operating parameters to a retention time of triadimefon between 2 and 3 min).

**(b) Equilibration of the system.** Pump sufficient eluant through the column to equilibrate the system. Inject 20 µl portions of the calibration solution and repeat the injections until retention times and peak areas vary by less than ± 1.5 % of the mean for 5 successive injections.

**(c) Linearity check.** Check the linearity of the response by injecting solutions with half and two times the concentration of the calibration solution. *The linear range must not be exceeded otherwise the sample weights have to be reduced accordingly.*

**(d) Sample preparation.** Weigh (to the nearest 0.1 mg) about 60 mg (*w* mg) of the technical sample into a 50 ml volumetric flask, dissolve and make up to volume with the eluant.

**(e) Determination.** Inject 20 µl portions of two calibration solutions ( $C_1$ ,  $C_2$ ) of which the triadimefon content differs approximately by 10 %. Inject each calibration solution at least twice and calculate the average quotient of the peak area and the corresponding mass. The individual values should not deviate from the mean by more than 0.4 % otherwise repeat the calibration. Then inject in duplicate 20 µl portions of each sample solution ( $S_n$ ). A series of more than 4 sample runs requires a repetition of the calibration test at the end of the series. The following injection sequence is proposed:

$C_1, C_1, C_2, C_2, S_1, S_1, S_2, S_2, C_1, S_3, S_3, S_4, S_4, C_1, \dots, S_n, C_2$

Measure the relevant peak areas.

**(f) Calculation**

$$\text{The content of triadimefon} = \frac{F \times s \times P}{F_1 \times w} \text{ g/kg}$$

Where:

- F1 = peak area of triadimefon standard
- s = mass of triadimefon standard, in mg
- P = purity of triadimefon standard, in g/kg
- F = peak area of triadimefon sample
- W = mass of sample, in mg

Repeatability  $r_{95} = 0.97 \%$  at 90 – 100 % a.i. (ISO 5725)

Reproducibility  $R_{95} = 1.59 \%$  at 90 – 100 % a.i. (ISO 5725)

**Triadimefon Wettable Powder**  
352/WP/(M)/-

**1 Sampling.** Take at least 500 g.

**2 Identity test.** As for 352/TC/(M)/2.

### **3 Triadimefon**

Reagents and Apparatus. As for 352/TC/(M)/3 together with  
Ultrasonic bath  
Centrifuge 3000 rpm

Procedure. As for 352/TC/(M)/3, except:

*(b) Sample preparation.* Homogenize the sample and weigh (to the nearest 0.1 mg) sufficient sample ( $w$  mg) to contain about 60 mg of triadimefon into a 50 ml volumetric flask. Add about 40 ml of eluant and extract the active ingredient by treating the sample in an ultrasonic bath for about 15 min. Allow to equilibrate to room temperature, make up to volume with eluant and homogenize. Before injecting centrifuge in order to eliminate inert ingredients.

Repeatability  $r_{95} = 0.55$  % at 25 % a.i. (ISO 5725)

Reproducibility  $R_{95} = 0.80$  % at 25 % a.i. (ISO 5725)

### **4 Suspensibility**

*(a) Preparation of suspension according MT 15.1, MT 184.*

*(b) Determination of sedimentation according MT 15.1/ MT 184*

*(c) Determination of triadimefon in the bottom 25 ml of suspension. After removal of the top 225 ml of suspension transfer the remaining 25 ml of the suspension to a 50 ml volumetric flask. Fill up to the mark with eluant. Determine the mass of triadimefon as described in the method procedure*

### **Triadimefon Emulsifiable Concentrate**

352/EC/(M)/-

**1 Sampling.** Take at least 500 g.

**2 Identity test.** As for 352/TC/(M)/2.

### **3 Triadimefon**

Reagents and Apparatus. As for 352/TC/(M)/3 together with  
Ultrasonic bath

Procedure. As for 352/TC/(M)/3, except:

*(b) Sample preparation.* Thoroughly shake the sample container to ensure that the emulsion is homogeneous. Immediately weigh (to the nearest 0.1 mg) sufficient sample ( $w$  mg) to contain about 60 mg of triadimefon into a 50 ml volumetric flask. Add about 40 ml of eluant and dissolve the active ingredient by treating the sample in an ultrasonic bath for about 15 min. Allow to equilibrate to room temperature, make up to volume with eluant.

**Triadimefon Water Dispersible Granules**  
352/WG/(M)-

**1 Sampling.** Take at least 500 g.

**2 Identity test.** As for 352/TC/(M)/2.

**3 Triadimefon**

Reagents and Apparatus. As for 352/TC/(M)/3 together with  
Ultrasonic bath  
Centrifuge 3000 rpm

Procedure. As for 352/TC/(M)/3, except:

*(b) Sample preparation.* Homogenize the sample and weigh (to the nearest 0.1 mg) sufficient sample (*w* mg) to contain about 60 mg of triadimefon into a 50 ml volumetric flask. Add about 40 ml of eluant and extract the active ingredient by treating the sample in an ultrasonic bath for about 15 min. Allow to equilibrate to room temperature, make up to volume with eluant and homogenize. Before injecting centrifuge in order to eliminate inert ingredients.

**4 Suspensibility**

*(a) Preparation of suspension according MT 161, MT 184.*

*(b) Determination of triadimefon in the bottom 25 ml of suspension. After removal of the top 225 ml of suspension transfer the remaining 25 ml of the suspension to a 50 ml volumetric flask. Fill up to the mark with eluant. Determine the mass of triadimefon as described in the method procedure.*

**Triadimefon Granules**  
352/GR/(M)-

**1 Sampling.** Take at least 500 g.

**2 Identity test.** As for 352/TC/(M)/2.

**3 Triadimefon**

Reagents and Apparatus. As for 352/TC/(M)/3 together with  
Ultrasonic bath  
Centrifuge 3000 rpm

Procedure. As for 352/TC/(M)/3, except:

*(b) Sample preparation.* Homogenize the sample and weigh (to the nearest 0.1 mg) sufficient sample (*w* mg) to contain about 60 mg of triadimefon into a 50 ml volumetric flask. Add about 40 ml of eluant and extract the active ingredient by treating the sample in an ultrasonic bath for about 15 min. Allow to equilibrate to room temperature, make up to volume with eluant and homogenize. Before injecting centrifuge in order to eliminate inert ingredients.

