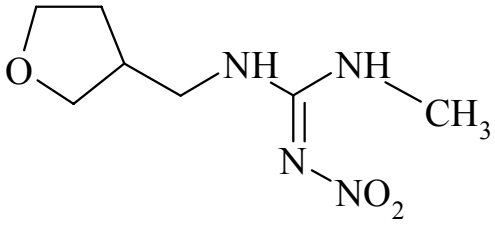
DINOTEFURAN
749



ISO common name	Dinotefuran
Chemical name	(RS)-1-methyl-2-nitro-3-(tetrahydro-3-furylmethyl)-guanidine (IUPAC); N-methyl-N'-nitro-N''-[(tetrahydro 3-furyl)methyl]guanidine (CA; 165252-70-0)
Empirical formula	$C_7H_{14}N_4O_3$
RMM	202.2
m.p.	107.5 °C
v.p.	Less than 1.7×10^{-6} Pa at 30 °C
Solubility	In water: 40 g/l; acetone: 58 g/l; hexane: 9.0×10^{-6} g/l; xylene: 72×10^{-3} g/l; methanol: 57 g/l; all at 20 °C
Description	White powder
Stability	Stable at room temperature
Formulations	Wettable powders and water soluble granules

DINOTEFURAN TECHNICAL
*749/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The retention time of dinotefuran for the sample solution should not deviate by more than 1% from that for the calibration solution.

2.2 Infrared. Prepare potassium bromide discs from the sample and from pure dinotefuran. Scan the discs from 4000 to 400 cm^{-1}. The spectrum produced from the sample should not differ significantly from that of the standard.

3 Dinotefuran

OUTLINE OF METHOD Dinotefuran is determined by reversed phase high performance liquid chromatography using UV detection at 270 nm and external standardisation.

REAGENTS

Methanol HPLC grade
Water HPLC grade
Dinotefuran analytical standard of certified purity. Store refrigerated.
Diluting solution methanol - water, 200 + 800 (v/v)
Mobile phase methanol - water, 200 + 800 (v/v)
Calibration solution. Prepare calibration solutions in duplicate. Weigh (to the nearest 0.1 mg) 50 mg (*s* mg) of dinotefuran analytical standard into a volumetric flask (50 ml). Add methanol (10 ml), fill to the mark with water and mix well. Pipette 5.0 ml of these solutions into separate volumetric flask (50 ml) and fill to the mark with diluting solution.

APPARATUS

High performance liquid chromatograph equipped with a constant flow pump, an auto automatic injector, a column oven and a UV spectrometric detector capable of measuring at 270 nm.
Column 250 × 4.6 mm (i.d.) Waters SymmetryShield RP_8, 5 μm
Electric integrator or *data system*
Ultrasonic bath

* CIPAC method 2005. Prepared by the Japanese PAC (JAPAC). Chairman: N.Tamori. Based on a method supplied by Mitsui Chemical Company, Japan.

PROCEDURE

(a) Chromatographic conditions (typical):

Flow rate	1.0 ml/min
Column temperature	40 °C
Injection volume	10 µl
Detector wavelength	270 nm
Run time	approximately 15 min
Retention time	dinotefuran: approximately 6 min

(b) Linearity check. Check the linearity of the detector response by injecting 10 µl of solutions with dinotefuran concentrations 0.5, 1 and 2 times that of the calibration solution before conducting the analysis.

(c) System equilibration. Inject 10 µl portions of the first calibration solution until the response factors obtained for two consecutive injections differ by less than 2%. Then inject 10 µl portions of the second solution. The response factor for this solution should not deviate by more than 2% from that for the first calibration solution, otherwise prepare new calibration solutions.

(d) Preparation of sample solution. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) sufficient sample to contain 50 mg (*w* mg) of dinotefuran into a volumetric flask (50 ml). Add methanol (10 ml) and water (35 ml) Swirl to suspend the powder and place the flasks in an ultrasonic bath for about 5 min. Allow to cool to room temperature and fill to the mark with water. Pipette 5.0 ml of these solutions into separate volumetric flask (50 ml) and fill the mark with diluting solution.

(e) Determination. Inject in duplicate 10 µl portions of each sample solution bracketing them by injections of the calibration solutions as follows:
calibration solution A, sample solution A, sample solution A, calibration solution B, sample solution B, sample solution B, calibration solution A, and so on. Measure the relevant peak areas.

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

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

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

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

Repeatability r = 12 g/kg at 995 g/kg active ingredient content
Reproducibility R = 12 g/kg at 995 g/kg active ingredient content

DINOTEFURAN WETTABLE POWDERS
*749/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity tests
2.1 HPLC. As for dinotefuran technical **749**/TC/M/2.1.
2.2 Infrared. Extract the sample with a suitable solvent, filter and evaporate the solvent with a stream of clean dry air. Proceed as for dinotefuran technical **749**/TC/M/2.2.

* CIPAC method 2005. Prepared by the Japanese PAC (JAPAC). Chairman: N.Tamori. Based on a method supplied by Mitsui Chemical Company, Japan.

3 Dinotefuran. As for dinotefuran technical **749**/TC/M/3 except:
(d) Preparation of sample solution. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) sufficient sample to contain 50 mg (*w* mg) of dinotefuran into a volumetric flask (50 ml). Add methanol (10 ml) and water (35 ml). Swirl to suspend the powder and place the flasks in an ultrasonic bath for about 5 min. Allow to cool to room temperature and fill to the mark with water. Pipette 5.0 ml of these solutions into separate volumetric flask (50 ml) and fill to the mark with diluting solution. Filter the supernatant through a 0.2 μm filter.

Repeatability r = 3 g/kg at 215 g/kg active ingredient content
= 4 g/kg at 212 g/kg active ingredient content
Reproducibility R = 3 g/kg at 215 g/kg active ingredient content
= 4 g/kg at 212 g/kg active ingredient content

DINOTEFURAN WATER SOLUBLE GRANULES
*749/SG/M/-

1 Sampling. Take at least 500 g.

2 Identity tests
2.1 HPLC. As for dinotefuran technical **749**/TC/M/2.1.
2.2 Infrared. As for dinotefuran wettable powders **749**/WP/M/2.2.

3 Dinotefuran. As for dinotefuran technical **749**/TC/M/3.

Repeatability r = 3 to 5 g/kg at 204 g/kg active ingredient content
Reproducibility R = 5 g/kg at 204 g/kg active ingredient content

* CIPAC method 2005. Prepared by the Japanese PAC (JAPAC). Chairman: N.Tamori. Based on a method supplied by Mitsui Chemical Company, Japan.

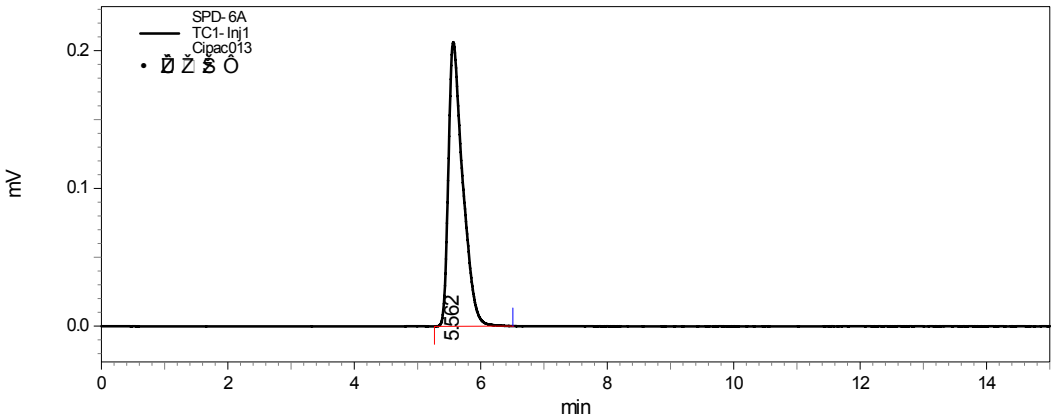


Fig. 26 HPLC chromatogram of dinotefuran TC

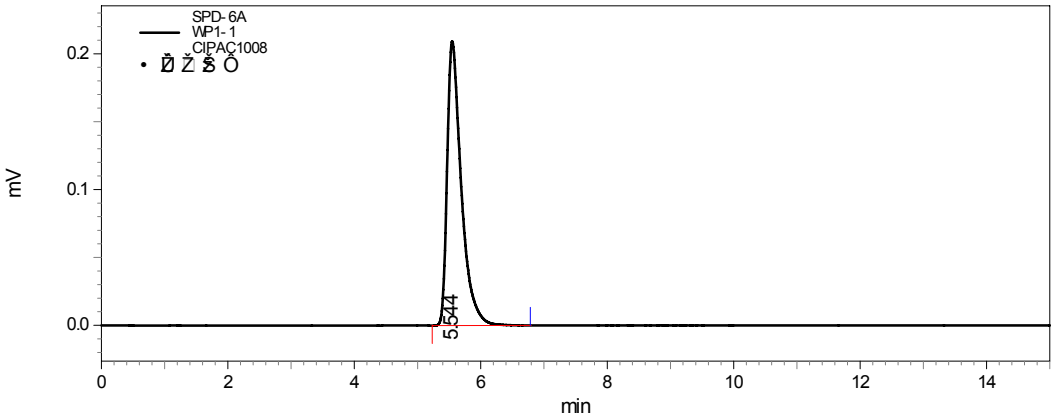


Fig. 27 HPLC chromatogram of dinotefuran 20% SG

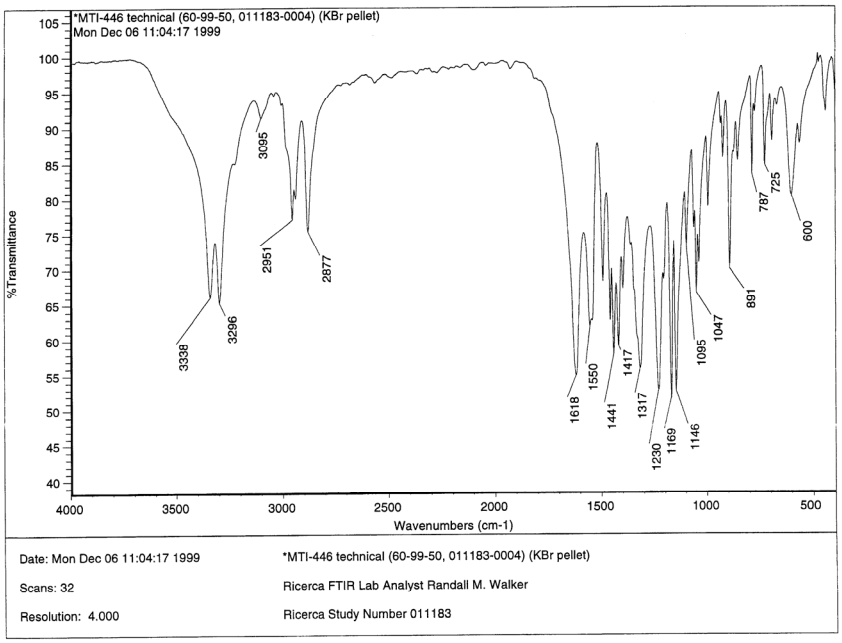


Fig. 28 FTIR spectrum of a dinotefuran KBr tablet