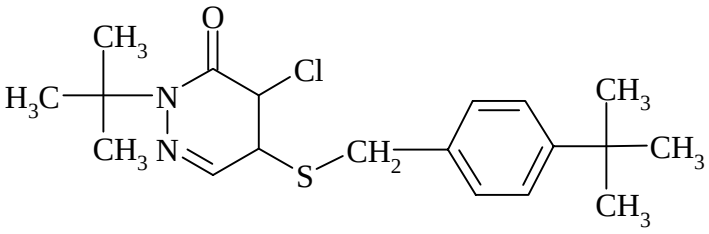
PYRIDABEN
583



ISO common name	Pyridaben
Chemical name	2-*tert*-Butyl-5-(4-*tert*-butylbenzylthio)-4-chloro-pyridazin-3(2*H*)-one (IUPAC); 4-chloro-2-(1,1-di-methyl)-5-[[4-(1,1-dimethylethyl) phenyl]methyl-thio]-3(2*H*)-pyridazinone CA;*96489-71-3*)
Empirical formula	$C_{19}H_{25}ClN_2OS$
RMM	364.9
m.p.	108-110 °C
v.p.	2.5×10^{-4} Pa at 20 °C
Solubility	In water: 1.2×10^{-5} g/l at 20 °C; soluble in organic solvents
Description	White crystalline odourless powder
Stability	Stable at pH 4 to 9, unstable under strong alkaline conditions
Formulations	Wettable powders and suspension concentrates

PYRIDABEN TECHNICAL
*583/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the method described below. The relative retention time of pyridaben for the sample solution should not deviate by more than 2% from that of the calibration solution.

2.2 Infrared. Prepare potassium bromide discs from the sample and from pure pyridaben with using 1.5 mg compound and 300 mg potassium bromide. Scan the discs from 4000 to 400 cm^{-1}. The spectrum produced from the sample should not differ significantly from that of the pure pyridaben (Fig. 28).

3 Pyridaben

OUTLINE OF METHOD The sample is dissolved in acetonitrile containing laurophenone as internal standard. Pyridaben is determined by reversed phase high performance liquid chromatography on a C_8 phase using acetonitrile - water (75 + 25) as mobile phase.

REAGENTS

Acetonitrile HPLC grade
Water HPLC grade
Pyridaben standard of known purity
Laurophenone
Internal standard solution. Weigh into a volumetric flask (200 ml) laurophenone (about 300 mg). Dissolve in acetonitrile and fill to the mark with acetonitrile. Mix thoroughly.
Mobile phase acetonitrile - water, 75 + 25 (v/v)
Calibration solution. Weigh (to the nearest 0.1 mg) into two volumetric flasks (100 ml) pyridaben reference material (120 mg, *s* mg). Add by pipette to each flask internal standard solution (10.0 ml). Swirl to dissolve, add acetonitrile (about 75 ml) and place the flasks in an ultrasonic bath for 5 min. Allow to cool to room temperature, fill to the mark with acetonitrile and mix thoroughly. Transfer by pipette 1.0 ml of each solution to separate volumetric flasks (20 ml), dilute to volume with acetonitrile and mix thoroughly (Solutions C_A and C_B). Filter the solutions through a 0.45 µm filter before injection.

* CIPAC method 1999. Prepared by the Japanese Committee (JAPAC). Chairman: N Tamori. Based on a method supplied by Nissan Chemicals Industries, Ltd Japan.

APPARATUS

Liquid chromatograph equipped with a constant flow pump, a constant temperature compartment, an automatic loop injector (10 µl) and a UV detector capable of measuring the absorbance at 240 nm

Liquid chromatographic column stainless steel, 250 × 4 mm (i.d.) packed with Nucleosil C_8, 5 µm, or equivalent

Electronic integrator or *data system*

Filtration system equipped with a PTFE membrane, 0.45 µm

Ultrasonic bath

PROCEDURE

(a) Operating conditions (typical):

Column	Stainless steel, 250 × 4 mm (i.d.) packed with Nucleosil C_8, 5 µm, or equivalent
Column temperature	40 °C
Injection volume	10 µl
Detector wavelength	240 nm
Flow rate	1.0 ml/min
Retention times	pyridaben: about 7 min laurophenone: about 9 min. The ideal retention time for laurophenone is 7.5 to 10 min. Adjust the mobile phase composition or the flow rate to maintain the above retention time.

(b) Equilibration of system. Inject in duplicate 10 µl portions of the calibration solutions and calculate the response factors. The individual values should not deviate by more than ± 0.5 %, otherwise repeat the calibration.

(c) Preparation of sample. Weigh (to the nearest 0.1 mg) into two volumetric flasks (200 ml) sufficient sample to contain 100 to 140 mg (*w* mg) pyridaben. Add by pipette to each flask internal standard solution (10.0 ml). Add acetonitrile (90 ml) and place the flasks in an ultrasonic bath for 5 min. Allow to cool to room temperature and mix thoroughly. Transfer by pipette 1.0 ml of each solution to separate volumetric flasks (20 ml), dilute to volume with acetonitrile and mix thoroughly (Solutions S_A and S_B). Filter the solutions through a 0.45 µm filter before injection.

(d) Determination. Inject in duplicate 10 µl portions of each sample solution bracketing them by injections of the calibration solutions as follows:

$$C_{A1}, S_{A1}, S_{A2}, C_{B1}, S_{B1}, S_{B2}, C_{A2}\ldots.$$

and so on for the following sample solutions.
Measure and record the relevant the peak areas.

(e) Calculation. Calculate the response factors (f_1, f_2, etc) for the pair of calibration solutions that bracket the sample solutions e.g. use C_{A1} and C_{B1} for sample injections S_{A1} and S_{A2}, etc and obtain the mean response factor *f.* Calculate for each sample solution injection e.g. S_{A1} the pyridaben content.

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

$$\text{Content of pyridaben} = \frac{H_w \times f}{I_r \times w} \text{ g/kg}$$

where:

f_i = response factor
f = average response factor
H_s = area of pyridaben peak in the calibration solution
H_w = area of pyridaben peak in the sample solution
I_q = area of laurophenone peak in the calibration solution
I_r = area of laurophenone peak in the sample solution
s = mass of pyridaben in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of pyridaben reference substance (g/kg)

Calculate the pyridaben content of the sample as the mean of the four determinations as follows:

Sample injection	Use response factor from	Content g/kg	
S_{A1}	C_{A1} and C_{B1}	Q)	
		)	X
S_{A2}	C_{A1} and C_{B1}	R)	
S_{B1}	C_{B1} and C_{A2}	S)	
		)	Y
S_{B2}	C_{B1} and C_{A2}	T)	

Take the mean of the values X and Y as the pyridaben content.

Repeatability r = 9 g/kg at 994 g/kg active ingredient content
Reproducibility R = 10 g/kg at 994 g/kg active ingredient content

PYRIDABEN WETTABLE POWDERS
*583/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity tests

2.1 HPLC. As for pyridaben technical **583**/TC/M/2.1.

2.2.UV spectrum. Use the HPLC method **583**/TC/M/3 and record the spectrum of the pyridaben peak with a diode array detector. The spectrum obtained from the sample should not differ significantly from that of the standard (Fig. 28).

3 Pyridaben. As for pyridaben technical **583**/TC/M/3.

Repeatability r	=	9 g/kg at 752 g/kg active ingredient content
	=	3 to 4 g/kg at 205 g/kg active ingredient content
Reproducibility R	=	11 g/kg at 752 kg active ingredient content
	=	4 to 5 g/kg at 205 g/kg active ingredient content

4 Suspensibility (Draft method)

REAGENTS AND APPARATUS As for **583**/TC/M/3 and MT 15.

PROCEDURE

(a) Preparation of suspension. MT 15.1 (*i*).

(b) Determination of sedimentation. MT 15.1 (*ii*).

(c) Determination of pyridaben in the bottom 25 ml of suspension. After removal of the top 225 ml of suspension add acetonitrile (75 ml) to the 25 ml remaining in the cylinder and mix thoroughly. Place the cylinder in an ultrasonic bath for 5 min. Allow to cool to room temperature and take a suitable aliquot of the solution. Determine the mass of pyridaben (*Q* g) by **583**/TC/M/3, except that the peak areas should be measured instead of the pyridaben – laurophenone peak area ratios, because of the absence of the internal standard

(d) Calculation

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

where:

c = mass of pyridaben in sample taken for the preparation of the suspension (g)

Q = mass of pyridaben in the bottom 25 ml of suspension (g)

* CIPAC method 1999. Prepared by the Japanese Committee (JAPAC). Chairman: N Tamori. Based on a method supplied by Nissan Chemicals Industries, Ltd Japan.

PYRIDABEN SUSPENSION CONCENTRATES
*583/SC/M/-

1 Sampling. Take at least 500 ml.

2 Identity tests

2.1 HPLC. As for pyridaben technical **583**/TC/M/2.1.

2.2.UV spectrum. Use the HPLC method **583**/TC/M/3 and record the spectrum of the pyridaben peak with a diode array detector. The spectrum obtained from the sample should not differ significantly from that of the standard.

3 Pyridaben. As for pyridaben technical **583**/TC/M/3 except:

(c) Preparation of sample. Shake the sample container thoroughly to homogenise the sample. Immediately weigh (to the nearest 0.1 mg) into two volumetric flasks (200 ml) sufficient sample to contain 100 to 140 mg, (*w* mg) pyridaben. Continue as for **583**/TC/M/3*(c).*

Repeatability r = 4 g/kg at 207 g/kg active ingredient content
Reproducibility R = 5 g/kg at 207 g/kg active ingredient content

4 Suspensibility (Draft method)

REAGENTS AND APPARATUS As for **583**/TC/M/3 and MT 161.

PROCEDURE

(a) Preparation of suspension and determination of sedimentation. MT 161.

(b) Determination of pyridaben in the bottom 25 ml of suspension. As for **583**/WP/M/4 *(c).*

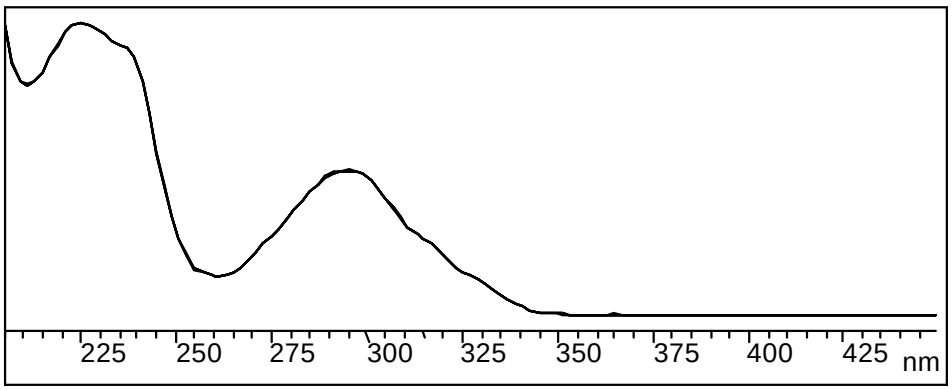
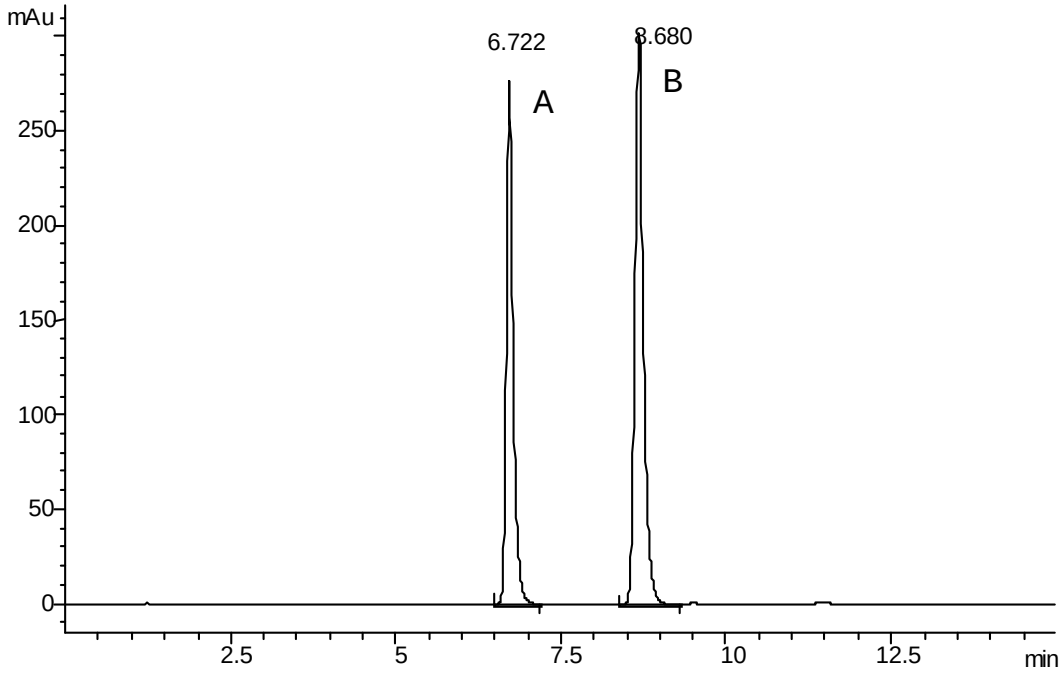


Fig. 26 UV spectrum of pyridaben

* CIPAC method 1999. Prepared y the Japanese Committee (JAPAC). Chairman: N Tamori.based on a method supplied by Nissan Chemical Industries, Ltd. Japan



A = pyridaben, B = laurophenone

Fig. 27 HPLC chromatogram of pyridaben technical

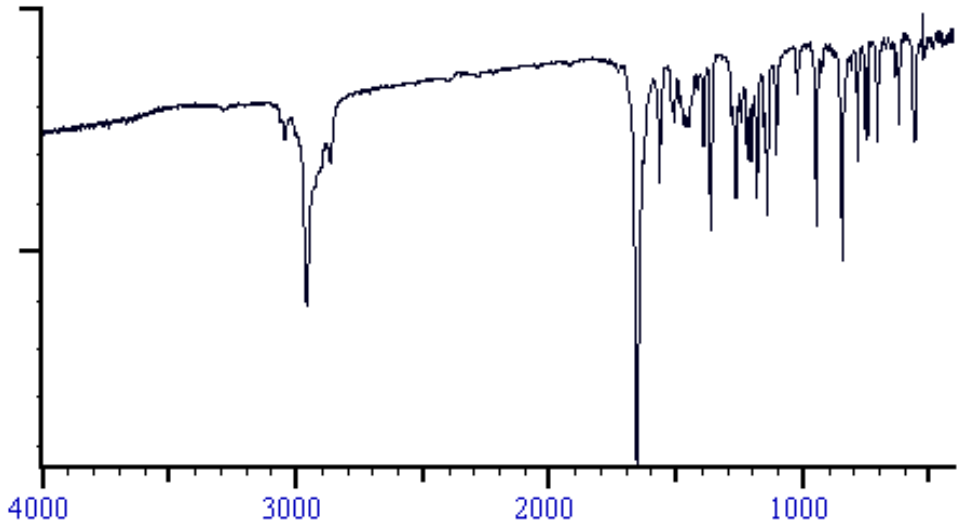


Fig. 28 IR spectrum of pyridaben