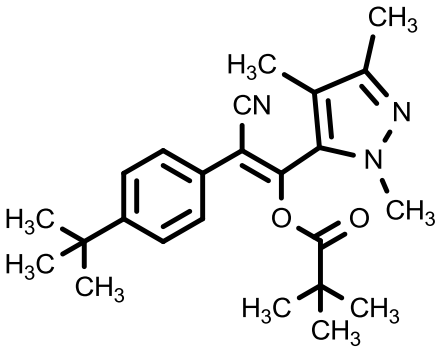
CYENOPYRAFEN
983



ISO common name	Cyenopyrafen
Chemical name	(*E*)-2-(4-*tert*-butylphenyl)-2-cyano-1-(1,3,4-trimethylpyrazol-5-yl)vinyl 2,2-dimethylpropionate (IUPAC) (1*E*)-2-cyano-2-[4-(1,1-dimethylethyl)phenyl]-1-(1,3,4-trimethyl-1*H*-pyrazol-5-yl)ethenyl 2,2-dimethylpropanoate (CA)
CAS No.	560121-52-0
Empirical formula	$C_{24}H_{31}N_3O_2$
RMM	393.52
m.p.	106.7 – 108.2 ºC
v.p.	5.2×10^{-7} Pa (25 ºC)
Solubility	In water: 0.30 mg/l, *n*-hexane: 21.08 g/l, *n*-octanol: 68.68 g/l, methanol: 169.0 g/l, toluene, acetone and ethyl acetate: 500 - 1000 g/l, dichloromethane: >1000 g/l at 20 ºC
Description	White solid
Stability	DT50 in water: 166.4 d (pH 4), 25.7 d (pH 7), 0.9 d (pH 9) at 25 ºC
Formulation	Suspension concentrates

CYENOPYRAFEN
983/TC/m/-

1. Sampling. Take at least 100 g.

2. Identity tests.

2.1 HPLC. Use the HPLC method described below. The retention time of cyenopyrafen for the sample solution should not deviate by more than 0.2 min from that of the calibration solution.

2.2 Infrared. Apply sample to IR spectrophotometer and scan the sample directly from 4000 to 450 cm^{-1} to determine by ATR method. The spectrum produced from the sample should not differ significantly from that of the standard (Figure 1).

3. Cyenopyrafen

OUTLINE OF METHOD Cyenopyrafen is determined by reversed phase high performance liquid chromatography using UV detection at 280 nm and external standardisation.

REAGENTS

Acetonitrile HPLC grade

Water HPLC grade

Cyenopyrafen standard of known purity

Mobile phase Acetonitrile – Water (80+20) (v/v)

Calibration solution. Weigh in duplicate (to the nearest 0.1 mg) 100 mg (*s* mg) of cyenopyrafen standard into separate volumetric flasks (100 ml). Add mobile phase (about 90 ml) and place the flask in an ultrasonic bath for 5 min. Fill to the mark with mobile phase. Mix thoroughly (calibration solutions C_1 and C_2).

APPARATUS

High performance liquid chromatograph equipped with a detector suitable for operation at 280 nm, constant-temperature column compartment and an injector capable of delivering 5 µl.

Column stainless steel, 250 × 4.6 mm (i.d.) packed with YMC Pack

Pro C18 5 µm, or equivalent.
Electronic integrator or data system
Filtration unit equipped with a PFTE membrane, 0.45 µm.
Ultrasonic bath

PROCEDURE

(a) Liquid chromatographic conditions (typical):

Column	stainless steel, 250 × 4.6 mm (i.d.), packed with YMC Pack Pro C18 5 µm, or equivalent.
Mobile phase	acetonitrile – Water, 80+20 (v/v)
Temperature	40 °C
Flow rate	1.0 ml/min
Detection wavelength	280 nm
Injection volume	5 µl
Retention time	cyenopyrafen: about 12 min.

(*b) Linearity check.* Check the linearity of the detector response by injecting 5 µl of cyenopyrafen reference standard solutions at concentrations of 0.5, 1 and 2 times that of the calibration solution.

(c) System equilibration. Inject 5 µl portions of the calibration solution C_1 and repeat the injections until peak areas deviate by less than ±1.0% of the mean for two consecutive injections. Then inject consecutively two 5 µl portions of the second calibration solution (C_2). The mean response factor for this solution should not deviate by more than 1.0% from that of the first calibration solution (C_1), otherwise prepare new calibration solutions.

(d) Sample preparation. Prepare sample solutions in duplicate. Weigh (to the nearest 0.1 mg) 100 mg of cyenopyrafen technical into separate 100 ml volumetric flasks. Add mobile phase (about 90 ml) and place the flask in an ultrasonic bath for 5 min. Fill to the mark with mobile phase. Mix thoroughly (sample solutions S_{TC1}-1, S_{TC1}-2, S_{TC2}-1 and S_{TC2}-2).

(e) Determination. Inject in duplicate 5 µl portions of each sample solution bracketing them by injections of the calibration solutions as follows;

C_1, S_{TC1}-1, S_{TC1}-1, C_2, S_{TC1}-2, S_{TC1}-2, C_1, S_{TC2}-1, S_{TC2}-1, C_2 ...
Determine 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 cyenopyrafen contents of the bracketed sample injections.

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

$$\text{Cyenopyrafen content} = \frac{f \times H_w}{w} \text{ (g/kg)}$$

Where,

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

Repeatability r = 9 to 10 g/kg at 996 to 997 g/kg active ingredient content
Reproducibility R = 12 to 13 g/kg at 996 to 997 g/kg active ingredient content

CYENOPYRAFEN SUSPENSION CONCENTRATES
983/SC/m/-

1. Sampling. Take at least 500 ml.

2. Identity tests.

2.1 HPLC. As for cyenopyrafen technical **983**/TC/m/2.1.

2.2 Infrared. Take 1 ml of sample and shake vigorously for 2 minutes with acetone (2 ml). Filter to remove any residue. To 1 ml of the filtrate add water (9 ml) and mix in a centrifuging tube. Centrifuge the precipitate of cyenopyrafen. Remove the supernatant and add 10 ml of water to the residue. Shake the mixture and centrifuge. Remove the supernatant and add 1 ml of methanol and 9 ml of water to the residue. Place it in ultrasonic bath for 2 minutes and centrifuge. Remove the supernatant and dry the residue at 60 °C under vacuum for three hours. Proceed as for cyenopyrafen technical **983**/TC/m/2.2.

3. Cyenopyrafen. As for **983**/TC/m/3 except:

Change 'PROCEDURE *(d) Sample preparation.*' as follows:

Homogenise each sample by vigorous shaking for 5 min. Prepare sample solutions in duplicate. Weigh (to the nearest 0.1 mg) 500 mg of SC formulation into separate 100 ml volumetric flasks. Add water (20 ml) to emulsify and then add acetonitrile (about 70 ml). Place the flask in an ultrasonic bath for 5 min. Allow to cool to ambient temperature and fill to the mark with acetonitrile. Mix thoroughly. Clear each solution from the formulated products by filtration through a 0.45 PTFE (polytetrafluoroethylene) filter (sample solutions S_{SC1}-1, S_{SC1}-2, S_{SC2}-1, S_{SC2}-2, S_{SC3}-1 and S_{SC3}-2).

Repeatability r	= 3 to 4 g/kg at 296 to 298 g/kg active ingredient content
Reproducibility R	= 6 to 9 g/kg at 296 to 298 g/kg active ingredient content

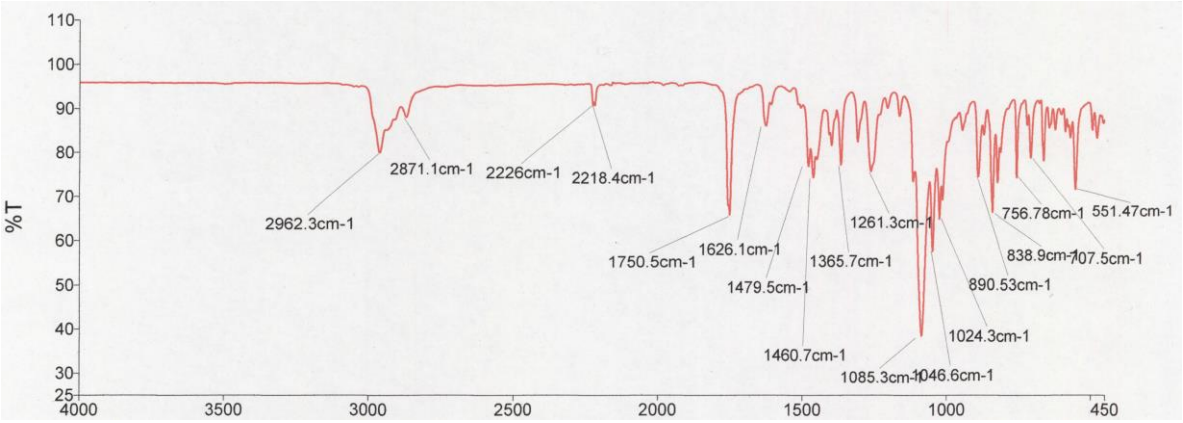


Figure 1 Infrared Spectrum of cyenopyrafen

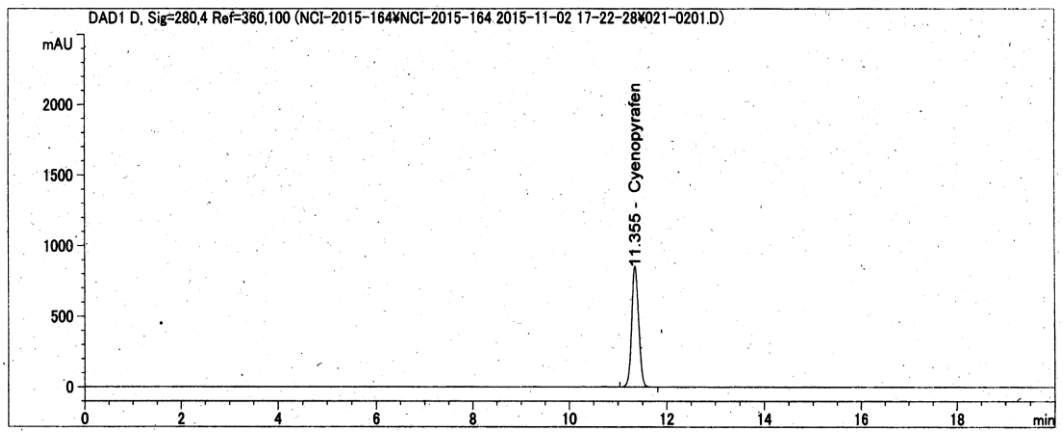


Figure 2 Chromatogram of cyenopyrafen TC

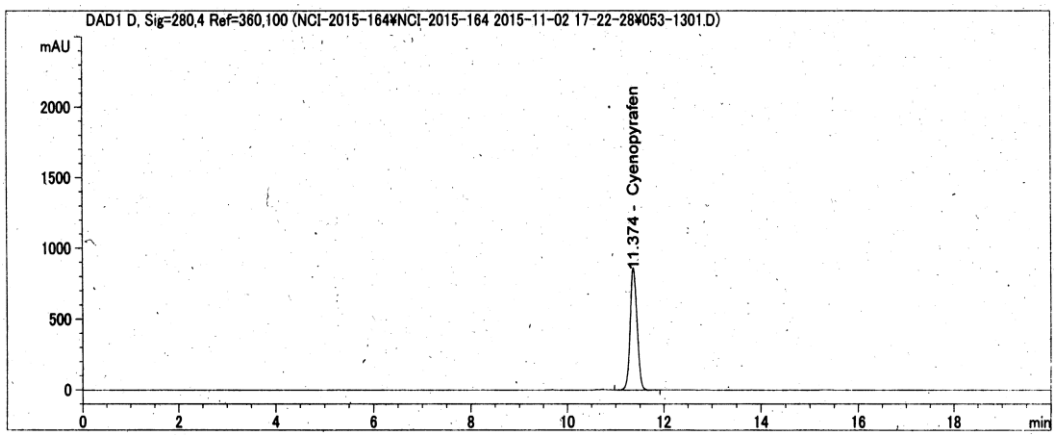


Figure 3 Chromatogram of cyenopyrafen SC