**AI**

**XXX**

*figure molecule*

*ISO common name* AI

*Chemical name* (*Z*)-2-(4-*tert*-butylphenyl)-2-cyano-1-(1-ethyl-3-
 methylpyrazol-5-yl)vinyl …..

*CAS No.* xxxx-xx-x

*Molecular formula* C4H3NOS…

*Molecular mass* xxx.x g/mol

*Melting point* xxx °C

*Vapor pressure* x.x x 10-8 Pa at 20 °C

*Solubility* water xx mg/l (20 °C); methanol xx g/l (20 °C);
 hexane xx g/l (20 °C) …

*Description* odorless, white powder

*Stability* stable in neutral acidic or alkaline conditions:
 ……

*Formulation(s)* Suspension concentrate (SC)

# AI TECHNICAL

# [[1]](#footnote-1)\*xxx/TC/M/-

**1 Sampling.** Take at least XXX g

**2 Identity tests**

**2.1 HPLC.** Use the reversed phase HPLC method below. The retention time of the AI peak in the sample solution should not deviate by more than xxx min from that of the calibration solution.

**2.2 Infrared.** Use either the ATR technique or prepare KBr discs from the sample and from AI reference substance. Scan from 4000 to 400 cm-1. The spectrum of the sample should not differ significantly from that of the reference substance.

**3 Active ingredient**

**OUTLINE OF METHOD**

The sample of XXX technical material is dissolved in ..., and the XXX content (g/kg) is determined by reversed phase high performance liquid chromatography using UV detection at XXX nm and external standardization.

**REAGENTS**

*XXX* reference standard of known purity

*Solvent,* HPLC grade

*Phosphoric acid,* purity p.a.

*Water,* HPLC grade

*0.05% Phosphoric acid solution,* add 0.5 ml phosphoric acid to water and dilute to 1000 ml

**Calibration solution.**Prepare calibration solutions in duplicate (CA and CB). Weigh to the nearest 0.1 mg about x mg (*s*) of reference standard into a volumetric flask (x ml). Add ... (about x ml) and place the flask in an ultrasonic bath for x min.

**APPARATUS**

*High performance liquid chromatograph,* equipped with a UV detector.

*Liquid chromatographic column,* stainless steel, 150 x 4.6 mm (i.d.), XXX C18, X μm, or equivalent with the same selectivity.

*Ultrasonic bath*

*Syringe filter* compatible with the solvent (for example, 0.45 µm pore diameter, RC)

**PROCEDURE**

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

 *Column* 150 x 4.6 mm (i.d.), XXX C18, X μm, or equivalent

 *Mobile phase* acetonitrile - phosphoric acid solution,
 XX + XX (v/v)

 *Column temperature* XX °C

 *Flow rate* XX ml/min

 *Detector wavelength* XX nm

 *Injection volume* XX µl

 *Retention time* AI: XX.X min (indicative)

**(b) System equilibration.**Pump sufficient mobile phase through the column to equilibrate the system. Inject X µl portion of calibration solution CA until the response obtained from two consecutive injections deviate by less than 1.5 %. Then inject X µl portion of calibration solution CB. The response factor for this solution should not deviate by more than x % from that of solution CA.....

**(c)** **Sample preparation.**Prepare sample solutions in duplicate for each sample.Weigh to the nearest 0.1 mg sufficient sample (*m*) to contain about XX mg of AI into a volumetric flask (XX ml). Add solvent (about XX ml) and place the flask in an ultrasonic bath for X min. Allow to cool to ambient temperature and fill to the mark with solvent. Mix thoroughly (sample solutions S1 and S2).

**(d) Determination.** Inject in duplicate x µl portions of each sample solution bracketing them by duplicate injections of the calibration solutions as follows: calibration solution CA, calibration solution CB, calibration solution CA, sample solution S1A, sample solution S1B, calibration solution CA, sample solution S2A, sample solution S2B, calibration solution CA, and so on for further samples. Measure the relevant peak areas.

**(e) Calculation.**Calculate the mean values of each pair of calibration response factors, bracketing two injections of a sample, and use this value for calculating the AI contents of the bracketed sample injections. The AI content is the mean value of two sample solutions.

$$f\_{i}=\frac{s×P}{A\_{c}}$$

$$AI content= \frac{f × A\_{s}}{m} (g/kg)$$

where:

*fi* =individual response factor

*f* =mean response factor

*Ac* =peak area of AI in the calibration solution

*As* =peak area of AI in the sample solution

*s =* mass of AI reference standard in the calibration solution (mg)

*m* = mass of sample taken (mg)

*P* *=* purity of AI reference standard (g/kg)

**Repeatability r** = X to X g/kg
at XX to XX g/kg active ingredient content

**Reproducibility R** =X to X g/kg
at XX to XX g/kg active ingredient content

**AI suspension concentrates**

# [[2]](#footnote-2)\*XXX/SC/M/-

**1 Sampling.** Take at least xxx ml. Mix thoroughly to obtain sample homogeneity.

**2 Identity tests**

**2.1 HPLC.** As for AI technical **XXX**/TC/M/2.1.

**3 Active ingredient.** As for AI technical **XXX**/TC/M/3 except:

**(c) Sample preparation.**Prepare sample solutions in duplicate for each sample. Weigh to the nearest 0.1 mg sufficient sample (*m*) to contain about XX mg of AI into a volumetric flask (XXX ml). Add water (about X ml) to emulsify the sample, then add solvent (about XX ml) and place the flask in an ultrasonic bath for X min. Allow to cool to ambient temperature and fill to the mark with solvent. Mix thoroughly and filter an aliquot of each prepared solution through a XXX µm syringe filter prior to analysis (sample solutions S1 and S2).

**Repeatability r** = X to X g/kg
at XX to XX g/kg active ingredient content

**Reproducibility R** =X to X g/kg
at XX to XX g/kg active ingredient content

**4 Suspensibility**

**REAGENTS AND APPARATUS.** As for **XXX**/TC/M/3 and MT 184 (actual version)

**PROCEDURE**

1. **Preparation of suspension and determination of sedimentation.** MT 184 (actual version)
2. **Determination of AI in the bottom 25 ml of suspension*.***After removal of the top 225 ml of suspension transfer the remaining 25 ml to a volumetric flask (XXX ml) and add solvent (about XX ml). Place the flask in an ultrasonic bath for X min. Allow to cool to ambient temperature and fill to the mark with solvent. Mix thoroughly and filter through a XXX µm filter prior to analysis. Determine the mass of AI (*Q* g) by **XXX**/TC/M/3.

**(c) Calculation**

See CIPAC method MT 184, actual version

**Fig. 1** Infrared spectrum of AI

**Fig. 2** HPLC chromatogram of AI standard

**Fig. 3** HPLC chromatogram of AI TC

**Fig. 4** HPLC chromatogram of AI SC

1. \* CIPAC method 20XX. Based on a method supplied by …… [↑](#footnote-ref-1)
2. \* CIPAC method 20XX. Based on a method supplied by… [↑](#footnote-ref-2)