**AI**

**XXXX**



*ISO common name* “AI”

*Chemical name* (Z)-2-(4-tert- …..

*CAS No.* XXX

*Empirical formula* C4H3…

*Molecular mass* XX

*m.p.* XX °C ~ XX ℃

*v.p.* x.x × 10-8 Pa at 20 °C

*Solubility* water xx mg/L at 20 °C; methanol xx g/l;
 hexane xx g/l; acetone, dichloromethane, xylene,
 ethyl acetate xx g/l

*Stability* stable in neutral acidic or alkaline conditions

*Description* The pure material is a white, odourless powder

*Formulation* Suspension concentrates (SC)

# AI TECHNICAL

# [[1]](#footnote-1)\*XXXX/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 1.5% from that of the calibration solution.

**2.2 Infrared.** …. from 4000 to 400 cm-1. The spectrum produced from the sample should not differ significantly from that of the standard.

**3 AI**

**OUTLINE OF METHOD**

AI is determined by reversed phase high performance liquid chromatography using UV detection at XXX nm and external standardization.

**REAGENTS**

*AI* reference standard of known purity.

*Acetonitrile* HPLC grade

*Phosphoric acid* puriss 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.**Weigh in duplicate about XX mg (to the nearest 0.1 mg) of AI reference standard (s mg) into a volumetric flask (XX ml). Add acetonitrile (about XX ml) and place the flask in an ultrasonic bath for 3 min…. (calibration solutions CA and CB).

**APPARATUS**

*High performance liquid chromatograph* equipped with a UV detector suitable for operation at XXX nm and an injection system capable of injecting XX µl.

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

*Ultrasonic bath*

**PROCEDURE**

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

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

 *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: approximately XX.X min

**(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 1.5%.....

**(c)** **Sample preparation.**Prepare sample solutions in duplicate for each sample.Weigh (to the nearest 0.1 mg) sufficient sample (*w*mg) to contain about XX mg of AI into a volumetric flask (XX ml). Add acetonitrile (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 acetonitrile. Mix thoroughly. (sample solutions S1 and S2).

**(d) Determination.** Inject in duplicate each sample solution bracketing them by injections of the calibration solutions as follows: calibration solution CA, sample solution S1, sample solution S1, calibration solution CB, sample solution S2, sample solution S2, calibration solution CA, and so on.

**(e) Calculation.**Measure the relevant peak areas. Average the values of the duplicate sample injections. Calculate the mean values of the response factors of the calibration solution bracketing two sample solutions and use this value to calculate the AI content of the bracketed samples. The AI content is the mean value of two sample solutions.

$$f\_{i}=\frac{s×P}{H\_{s}}$$

$$AI content= \frac{f × H\_{w}}{w} (g/kg)$$

where:

*fi =* individual response factor

*f =* mean response factor

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

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

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

*w =* 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)\*XXXX/SC/M/-

**1 Sampling.** Take at least 600 ml.

**2 Identity tests**

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

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

**(c) Preparation of sample.**Homogenize the sample by vigorous shaking. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) sufficient sample (w mg) to contain about XXX mg of AI (s mg) into a volumetric flask (XXX ml). Add water (about X ml) to emulsify the sample, then add acetonitrile (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 acetonitrile. Mix thoroughly. Filter an aliquot of each prepared solution through a XXX µm syringe filter (e.g. RC) 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 184.1.

**PROCEDURE**

1. **Preparation of suspension and determination of sedimentation.** MT 184.1.
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 acetonitrile (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 acetonitrile. Mix thoroughly. Filter through a XXX µm filter prior to analysis. Determine the mass of AI (Q g) by **XXX**/TC/M/3.

**(c) Calculation**

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

where:

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

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

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