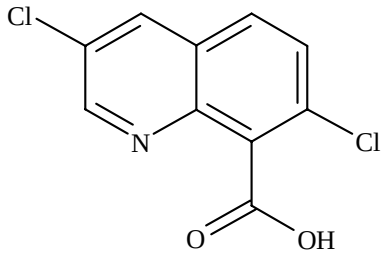
QUINCLORAC
493



ISO common name	Quinclorac
Chemical name	3,7-Dichloro-8-quinolinecarboxylic acid (IUPAC and CA; *84087-01-40*)
Empirical formula	$C_{10}H_5Cl_2NO_2$
RMM	242.1
m.p	274 °C
v.p.	Less than 1×10^{-9} Pa at 20 °C
Solubility	In water: 70 mg/l; acetone: 2 g/kg; ethanol: 2 g/kg; ethyl acetate: 1 g/kg; hexane: less than 1 g/kg; all at 20 °C
Description	Colourless crystals
Stability	Stable against hydrolysis, elevated temperatures and air
Formulations	Wettable powders, water dispersible granules and suspension concentrates

QUINCLORAC TECHNICAL
[*]**493**/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The retention time of quinclorac for the sample solution should not deviate by more than 10 s from that for the calibration solution.

2.2 Infrared. Prepare potassium bromide discs from the sample and from quinclorac standard taking 1.3 to 1.5 mg of material and 300 mg potassium bromide. Scan the discs from 400-4000 cm^{-1}. The spectrum obtained from the sample should not differ significantly from that from the standard.

3 Quinclorac

OUTLINE OF METHOD Quinclorac is dissolved in tetrahydrofuran and determined by high performance liquid chromatography on a reversed phase column using a tetrahydrofuran-water-sulphuric acid mixture as eluent, UV detection at 238 nm, and external standardisation.

REAGENTS

Quinclorac standard of known purity
Tetrahydrofuran HPLC grade
Water HPLC grade
Sulphuric acid solution $c(H_2SO_4)$ = 0.5 mol/l
Mobile phase. Add sulphuric acid (5 ml) and tetrahydrofuran (360 ml) to water (640 ml). Mix thoroughly and degas.
Calibration solution. Weigh (to the nearest 0.01 mg) in duplicate into two volumetric flasks (100 ml) 50 to 60 mg (*s* mg) of quinclorac standard. Add to each flask tetrahydrofuran (80 ml) and sulphuric acid (0.5 mol/l, 10 ml). Place the flasks in an ultrasonic bath for 10 min. Allow to cool to room temperature and fill to the mark with tetrahydrofuran. Transfer by pipette 5.0 ml of these solutions to separate volumetric flasks (50 ml). Dilute with tetrahydrofuran (20 ml) and fill to the mark with water. Mix well (solutions C_A, C_B).

[*] CIPAC method 1997. Prepared by the German Committee (DAPA). Chairman: W Dobrat. Based on a method supplied by BASF, Germany.

APPARATUS

High performance liquid chromatograph equipped with an automatic loop injector (5 µl) and a UV spectrophotometric detector operated at 238 nm
Column stainless steel, 125 × 4 or 4.6 (i. d.) mm, packed with Spherisorb ODS II (3 or 5 µm).
Electronic integrator or *data system*
Ultrasonic bath
Centrifuge

PROCEDURE

(a) Operating conditions (typical):

Mobile phase	tetrahydrofuran-water-sulphuric acid (0.5 mol/l), 360 + 640 + 5 v/v
Stationary phase	Spherisorb ODS II, particle size 3 to 5 µm.
Column	125 × 4.0 mm
Eluent flow rate	0.8 ml/min
Column temperature	ambient
Injection volume	5 µl
Detector wavelength	238 nm
Retention time	quinclorac: about 4 min

(b) Linearity check. Check the linearity of the detector response by injecting 20 µl of solutions with quinclorac concentrations 0.5, 1 and 2 times that of the calibration solution. Be sure that the concentrations of the solutions are in the linear range of the detector, otherwise alter the weighings or the dilutions accordingly. Inject each calibration solution at least twice and determine the mean peak area to mass ratios. The single values should differ by less than 0.5 % from the mean value, otherwise repeat the calibration. A plot of the mean peak area versus the mass should give a linear curve. The regression coefficient should be less than 0.999.

(c) Sample preparation. Weigh (to the nearest 0.01 mg) in duplicate into two volumetric flasks (100 ml) enough sample to contain 50 to 60 mg (*w* mg) of quinclorac. Add each flask tetrahydrofuran (80 ml) and sulphuric acid (0.5 mol/l, 10 ml). Place the flasks in an ultrasonic bath for 10 min. Allow to cool to room temperature and fill to the mark with tetrahydrofuran. Transfer by pipette 5.0 ml of these solutions to separate volumetric flasks (50 ml). Dilute with tetrahydrofuran (20 ml) and fill to the mark with water. Mix well (solutions S_1 and S_2). Before analysis centrifuge the solutions to remove any insoluble material.

(d) Determination. Inject in duplicate 5µl portions of the sample solution. Bracket the sample solutions injections by duplicate calibration solutions injections e.g.:

$$C_A, C_A, S_1, S_1, S_2, S_2, C_B, C_B, \text{.....etc.}$$

Record the areas of the peaks. Average the response factors of each double injection. Calculate the mean value of each pair of response factors bracketing the injections of two sample solution injections and use this value for calculating the quinclorac contents of the bracketed sample solution injections.

(e) Calculation

$$\text{Response factor } f_i = \frac{s \times P}{H_s} 1$$

$$\text{Quinclorac content} = \frac{H_w \times f}{w} 2\text{g/kg}$$

where:

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

The content of quinclorac is the mean value of the two sample solutions.

Repeatability r = 20 g/kg at 992 g/kg active ingredient content
Reproducibility R = 30 g/kg at 992 g/kg active ingredient content

Based on a study with 19 participants and 38 values.

QUINCLORAC WETTABLE POWDERS
[*]**493**/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity tests.
2.1 HPLC. As for **493**/TC/M/2.1.
2.2 Infrared. Extract the sample with a suitable solvent, filter and evaporate the solvent in a stream of clean, dry air. Continue as for **493**/TC/M/2.2.

3 Quinclorac. As for **493**/TC/M/3.

Repeatability r = 23 to 24 g/kg at 500 g/kg active ingredient content
Reproducibility R = 26 to 28 g/kg at 500 g/kg active ingredient content

Based on a study with 20 participants and 40 values.

4 Suspensibility (Draft method)

REAGENTS AND APPARATUS As for **493**/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 quinclorac in the bottom 25 ml of suspension. After removal of the top 225 ml of suspension transfer the 25 ml remaining quantitatively to a large evaporating dish, remove water in an oven at 100°C and determine the mass (*Q* g) of quinclorac with the residue by **493**/TC/M/3.

(d) Calculation

$$\text{Suspensibility} = \frac{111\,(c - Q)}{c}\ 3\%$$

where:
c = mass of active ingredient in sample actually taken
Q = mass of active ingredient in the 25 ml remaining in the cylinder

[*] CIPAC method 1997. Prepared by the German Committee (DAPA). Chairman: W Dobrat. Based on a method supplied by BASF AG, Germany.

QUINCLORAC WATER DISPERSIBLE GRANULES
*493/WG/M/-

1 Sampling. Take at least 1 kg.

2 Identity tests. As for **493**/WP/M/2.

3 Quinclorac. As for quinclorac technical **493**/TC/M/3.

Repeatability r = 26 g/kg at 762 g/l active ingredient content
Reproducibility R = 42 g/kg at 762 g/l active ingredient content

Based on a study with 26 participants and 42 values.

4 Suspensibility (Draft method). As for quinclorac wettable powders **493**/WP/M/4 and MT 168.

QUINCLORAC SUSPENSION CONCENTRATES
*493/SC/M/-

1 Sampling. Take at least 1 l.

2 Identity tests. As for **493**/WP/M/2.

3 Quinclorac. As for quinclorac technical **493**/TC/M/3.

Repeatability r = 12 g/kg at 219 g/kg active ingredient content
Reproducibility R = 18 g/kg at 219 g/kg active ingredient content

Based on a study with 21 participants and 42 values.

4 Suspensibility (Draft method). As for quinclorac wettable powders **493**/WP/M/4 and MT 161.

* CIPAC method 1997. Prepared by the German Committtee (DAPA). Chairman: W Dobrat. Based on a method supplied by BASF AG, Germany.