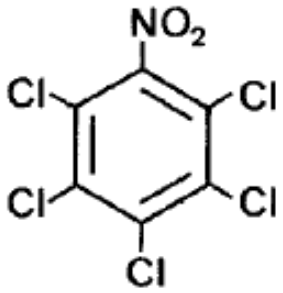
QUINTOZENE

78



ISO common name	Quintozene
Chemical name	pentachloronitrobenzene (IUPAC, CA; *82-68-8*)
Other name	PCNB
Empirical formula	$C_6Cl_5NO_2$
RMM	295·3
m.p.	146°C
v.p.	1·77 Pa (0·0133 mm Hg) at 25°C
d^{25}	1·718
Solubility	Practically insoluble in water. About 20 g/kg ethanol at 25°C. Soluble in benzene, carbon disulphide, chloroform.
Description	Colourless needles
Formulations	As dustable and wettable powders, emulsifiable concentrates and granules for seed and soil treatment.

QUINTOZENE TECHNICAL

***78/TC/M/–**

1 Sampling. Take at least 50 g.

2 Identity tests. Use the GLC method 3 below. The identity is confirmed if the difference between the retention times of quintozene and internal standard for the sample solution does not deviate by more than 10 seconds from that for the standard solution.

3 Quintozene

OUTLINE OF METHOD Dissolve the sample in chloroform, add o-terphenyl as internal standard and separate quintozene on SE 30 column by gas-liquid chromatography fitted with a flame ionization detector.

* AOAC–CIPAC method 1983.

REAGENTS

Chloroform RE 64
o-terphenyl internal standard
Solution: weigh about 0·2 g *o*-terphenyl into a 250 ml volumetric flask, dissolve and dilute to volume with chloroform, and mix thoroughly.
Quintozene. Standard of known purity.
Solution: weigh (to the nearest 0·1 mg) about 0·2 g of quintozene (*s* g) into a 100 ml volumetric flask, dissolve and dilute to volume with chloroform and mix thoroughly.

APPARATUS

Gas chromatograph with flame ionization detector
Column. Glass column 1·8 m × 4 mm (id) packed with 5% SE 30 on 80–100 mesh Chromosorb W (dimethylchlorosilane treated). Condition a newly packed column for 24 h at 285°C under a low N flow.
Recorder 1 mV full scale sensitivity and 1 second response
25 ml pipettes
10 ml pipettes

PROCEDURE

(*a*) *Preparation of the calibration solution*. Pipet 25 ml of the quintozene solution into a 100 ml glass-stoppered conical flask. Add by pipette 25 ml of the internal standard solution (Note 1) and mix thoroughly.

(*b*) *Preparation of sample solution*. Weigh (to the nearest 0·1 mg) about 0·2 g of the sample (*w* g) into a 100 ml volumetric flask, dissolve and dilute to volume with chloroform and mix thoroughly. Pipet 25 ml of the solution into a 100 ml glass-stoppered conical flask. Add by pipette 25 ml of the internal standard solution (Note 1) and mix thoroughly.

(*c*) *Conditions of chromatography*
Column temperature: 175–180°C
Injection port temperature: 200°C
Detector temperature: 250°C
Flow rate carrier gas: Nitrogen. Adjust the gas flow rate to elute quintozene at about 4·5 min.
Flow rate air and hydrogen: adjust as recommended by manufacturer
Attenuation: adjust so that the peak heights are 60–80% full scale
Approximate retention times: quintozene: about 4·5 min
internal standard: about 7·1 min

(*d*) *Chromatography of quintozene*. Inject 4 µl aliquots of the calibration solution until the variation in the response ratio (area or peak height) for quintozene (first peak) to o-terphenyl (second peak) is about 1%.

Then, inject the calibration solution, inject the sample solution twice and repeat the injection of the calibration solution. The retention times must be the same for the sample and the calibration solutions.

Calculate the average area or peak height ratio of quintozene to o-terphenyl for the two calibration and sample injections.

$$\text{Quintozene content} = \frac{R \times s \times P}{R' \times w} \text{ g/kg}$$

where: R = average response ratio for the sample solution
R' = average response ratio for the calibration solution
w = mass (g) of sample
s = mass (g) of quintozene in the calibration solution
P = purity (g/kg) of the quintozene standard

Note 1 The same pipette must be used for measuring the internal standard for both sample and calibration solutions.

QUINTOZENE DUSTABLE POWDERS

***78/DP/M/–**

1 Sampling. Take at least 500 g.

2 Identity tests. As for the technical **78**/TC/M/2.

3 Quintozene. As for the technical **78**/TC/M/3, except:

PROCEDURE

(*b*) *Preparation of the sample*. Weigh (to the nearest 0·1 mg) sufficient sample (w g) to contain about 0·2 g quintozene into a 250 ml glass-stoppered conical flask. Add 100 ml chloroform, stopper and shake for 2 h on a rotary shaker. Let insoluble material settle. Pipet 10 ml of the sample extract into a 50 ml glass-stoppered conical flask, add 10 ml internal standard solution, and mix.

QUINTOZENE WETTABLE POWDERS

***78/WP/M/–**

1 Sampling. Take at least 500 g.

2 Identity tests. As for the technical **78**/TC/M/2.

3 Quintozene. As for the dustable powder **78**/DP/M/3.

* AOAC–CIPAC method 1983.

QUINTOZENE EMULSIFIABLE CONCENTRATES

*78/EC/M/–

1 Sampling. Take at least 500 ml.

2 Identity tests. As for the technical **78**/TC/M/2.

3 Quintozene As for the technical **78**/TC/M/3, except:

PROCEDURE

(*b*) *Preparation of the sample.* Weigh (to the nearest 0·1 mg) sufficient sample (*w* g) to contain about 0·2 g quintozene into a 100 ml volumetric flask, dilute to volume with chloroform and mix thoroughly. Pipet 10 ml of this solution into a 50 ml glass-stoppered conical flask. Add 10 ml internal standard solution, and mix.

QUINTOZENE GRANULES

*78/GR/M/–

1 Sampling. Take at least 500 g.

2 Identity tests. As for the technical **78**/TC/M/2.

3 Quintozene. As for the technical **78**/TC/M/3, except:

PROCEDURE

(*b*) *Preparation of the sample.* Mix well the sample. Grind 100 g to pass 1 mm sieve. Mix well and weigh (to the nearest 1 mg) sufficient sample (*w* g) to contain about 0·2 g quintozene into a 250 ml glass-stoppered conical flask. Add 100 ml chloroform, stopper and shake for 2 h on a rotary shaker. Let insoluble material settle. Pipet 10 ml of the sample extract into a 50 ml glass-stoppered conical flask. Add 10 ml internal standard solution and mix.

* AOAC–CIPAC method 1983.