Concentrations of Quintozene at Different Depths in Bulb-growing Soils

by

M. LEISTRA and J. H. SMELT

Laboratory for Research on Insecticides
Wageningen, Netherlands

Soil has been treated occasionally with quintozene (pentachloronitrobenzene) against plant-parasitic fungi for some decades, particularly before planting flower bulbs, lettuce and potatoes. The fungicide is often applied to soils low in humus, that are particularly suitable for growing commercial bulbs. The areas concerned partly coincide with catchment areas where groundwater is pumped up for drinking purposes. In these areas, no substances may be used or stored, unless there is evidence that groundwater cannot be polluted. For the continuance of market gardens in these areas, data was needed on the concentration patterns of quintozene in the types of soil concerned.

Half-times of 4.7 to 9.7 months were found for quintozene added to three soil types incubated in flasks at 25 °C (WANG and BROADBENT 1972). An important conversion product is pentachloroaniline, a compound with a lower fungicidal activity than quintozene (KO and FARLEY 1969). When quintozene was mixed with wet soil in flasks through which air was conducted, it gradually evaporated (CASELEY 1968). Ultraviolet irradiation of quintozene solutions in various solvents decomposed the compound only very slowly (CROSBY and HAMADMAD 1971). For the solubility in water at 20 °C, a value is reported of 0.44 µg/cm^3 (ECKERT 1962). No data was available on concentrations of quintozene deeper in the soil. Therefore concentration was measured in a series of samples from fields where quintozene was occasionally applied to soil.

Procedures

Soil samples were taken in July 1972 from six fields near three different settlements in the bulb areas of the Province of North Holland. The samples from the upper decimeters were taken with a cylindrical auger equipped with a plunger. Layers to a depth of about 140 cm were sampled with Perspex tubes, equipped with a steel cylindrical knife at the bottom. Before pulling these tubes out of the soil, suction was applied at the upper end with an inverted cycle pump. This was necessary to prevent the loose wet subsoil from running out of the tubes. For each field or plot three points were sampled. The three samples from each layer were bulked and thoroughly mixed.

Subsamples of 30 g of soil were weighed into 200-ml screw flasks after which 25 ml of water and 24 ml of n-hexane were added. The flasks were closed with plug and screw cap and mechanically shaken for 2 hours. The concentrations in the n-hexane layer were measured with an Aerograph B550 gas chromatograph, equipped with Tritium electron-capture detector.

Bulletin of Environmental Contamination & Toxicology,
Vol. 11, No. 3 © 1974 by Springer-Verlag New York Inc.

TABLE 2

Concentrations of quintozene in bulb-growing soils expressed in µg/g dry soil.

Andyk				Breezand P			
Field 1		Field 2		Replicate 1		Replicate 2	
depth (cm)	concentration (µg/g)	depth (cm)	concentration (µg/g)	depth (cm)	concentration (µg/g)	depth (cm)	concentration (µg/g)
5- 10	3.9	5- 10	26	0- 20	0.32	0- 26	0.30
15- 20	18	15- 25	1.9	20- 46	0.14	26- 45	0.97
25- 30	0.032	25- 30	4.6	47- 72	0.003	45- 65	0.002
45- 50	0.004	45- 50	0.005	72- 98	<0.001	65- 84	<0.001
65- 70	<0.001	65- 70	0.002	98-117	<0.001	84-104	<0.001
85- 90	<0.001	85- 90	<0.001	117-137	<0.001	104-123	<0.001
110-120	<0.001	110-120	<0.001			123-132	<0.001

Breezand N				Beverwyk			
Replicate 1		Replicate 2		Field 1		Field 2	
depth (cm)	concentration (µg/g)	depth (cm)	concentration (µg/g)	depth (cm)	concentration (µg/g)	depth (cm)	concentration (µg/g)
0- 27	1.4	0- 27	0.80	0- 25	19	0- 27	17
27- 54	0.68	27- 54	0.024	25- 50	2.7	27- 48	1.6
54- 82	<0.001	54- 77	0.001	50- 63	2.7	48- 68	0.82
82-110	<0.001	77- 96	<0.001	63- 82	interfering substances	68- 89	interfering substances
110-137	<0.001	96-116	<0.001	82-101		89-119	
		116-136	<0.001				

The glass column 150 cm long had an inner diameter of 2 mm and the fill was 2% Silicone XE-60 on a support phase of 100 to 120 mesh Varaport-30. The carrier gas was pure nitrogen at a flow rate of 60 ml/min. The temperatures were: injection part 210 °C and oven part 180 °C. Retention time was 2.0 min and the lower limit of sensitivity with injection of 5 µl of the solution was 0.001 µg/ml.

Standard solutions in n-hexane of different concentrations were made with quintozene 99%. After injection of two unknown concentrations, a known concentration was injected, so that the unknown concentrations could be calibrated. Recovery percentage of the procedure was determined by adding 0.5 ml acetone with a known concentration of quintozene to blank samples of the soil types concerned. The flasks with soil and quintozene were sealed, occasionally mixed by turning while being stored for a few days at 2 °C. The same extraction procedure was used for these samples and recovery proved to be 96%.

For subsamples moisture content was measured by drying to constant weight at 105 °C. Soil composition was analysed by the Laboratory for Soil and Crop testing at Oosterbeek, Netherlands.

Results and Discussion

Data on the soils from the sites sampled are given in Table 1.

TABLE 1

Characteristics of the soils at the sites. Layer 0 to 50 cm. Contents in percentage by weight.

Field	Organic matter (%)	Clay (%)	$CaCO_3$ (%)	pH-KCl
Andyk 1	4.8	22	8.3	7.2
Andyk 2	4.5	20	5.6	7.3
Breezand P	0.8	2	0.8	7.1
Breezand N	1.0	2	0.8	6.9
Beverwyk 1	1.5	2	1.2	7.1
Beverwyk 2	2.0	3	0.5	7.1

The measurements for quintozene are given in Table 2. The fields near to the villages of Andyk and Breezand showed a similar pattern. To depths of about 70 cm, the compound could easily be detected. In the layer from zero to about 40 cm, significant amounts were present, while in the layer from about 40 to about 70 cm the concentrations were much lower In these areas, the depth of tillage after a crop was several decimeters, and it is likely that the distribution in the top 40 cm was mainly due to mechanical working. This is also shown by the irregular distribution of quintozene in this tilled surface layer. With predominant transport by leaching, a more regular pattern would be expected. The low concentration in the 40 to 70 cm layer could have resulted from some diffusion and leaching. As shown by these low concentrations, chromatographic transport was unimportant. At depths greater than 70 cm, no quintozene was detected. Other peaks appearing on the chromatograms for the top layer were very small for these depths. With the extracts of the samples for the deeper layers of the Beverwyk fields, there was strong interference in the chromatogram and quintozene could not be measured.

References

CASELEY, J.C.: Bull. Environ. Contam. Toxicol. 3, 180 (1968).

CROSBY, D.G., and N. HAMADMAD: J. Agric. Food Chem. 19, 1171 (1971).

ECKERT, J.W.: Phytopathology 52, 642 (1962).

KO, W.H. and J.D. FARLEY: Phytopathology 59, 64 (1969).

WANG, C.H. and F.E. BROADBENT: Soil Sci. Soc. Amer. Proc. 36, 742 (1972).