

FATE OF GLYPHOSATE IN A SANDY LOAM SOIL AND ANALYSIS FOR RESIDUES IN FIELD-GROWN CROPS¹

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Following application in the spring to a sandy loam soil, residues of glyphosate (N-phosphonomethyl glycine) decreased rapidly and degraded into aminomethylphosphonic acid which also was short lived. Analysis for glyphosate and its metabolite as their methyl trifluoroacetate derivatives was by the flame specific phosphorus gas chromatography. Glyphosate and its metabolite in the soil at 1, 10, 19, 29, 52, 86 and 122 days after application was 69.5, 29.6, 22.1, 14.1, 11.7, 4.2 and 0.0% and 2.3, 31.0, 35.2, 24.0, 15.0, 9.4 and 6.1%, respectively, of the 4.25 kg a.i./ha applied. Neither compound was found in field-grown barley, oat, wheat, sweet corn, beans, peas, red beet or carrots. No injury symptoms were observed on any of the crops during the growing season.

La concentration des résidus de glyphosate (N-phosphonométhyl glycine) décroissant rapidement après d'application à un terreau sablonneux au printemps. Il se dégradait en acide aminométhylphosphonique qui disparaissait rapidement. Le glyphosate et son métabolite sous forme de dérivés méthyltrifluoroacétate furent analysés par chromatographie en phase gazeuse à flamme spécifique au phosphore. Les concentrations dans le sol, des glyphosate et son métabolite 1, 10, 19, 29, 52, 86 et 122 jours après une application de 4.25 kg a.i./ha étaient de 69.5, 29.6, 22.1, 14.1, 11.7, 4.2 et 0.0% et de 2.3, 31.0, 35.2, 24.0, 15.0, 9.4 et 6.1% respectivement. Aucun composé ne fut retrouvé dans l'orge, l'avoine, le blé, le maïs, les haricots, les pois, les belles rouges ou les carottes cultivés dans ces champs. Aucune lésion n'est apparue chez les plants pendant la saison de croissance.

Introduction

Glyphosate (N-phosphonomethyl glycine) is the parent acid formulated as monoisopropylamine salt in the water-soluble herbicide Roundup®. Glyphosate is highly effective as a foliar treatment to weeds and will eradicate many hard to control perennial and annual weeds in a wide variety of cropping practices and non-crop usage. This unique herbicide is useful where herbicides with soil persistence, high mammalian toxicity, or hormone-like activity should not be used, or where the weed population contains a broad spectrum of species.

The initial rapid inactivation of glyphosate in soil is by adsorption (Sprankle et al. 1975; Hance 1976) but degradation occurs by microbial action (Sprankle et al. 1975; Rueppel 1977).

Glyphosate has been used successfully for weed control in numerous horticultural and field crops in Atlantic Canada for several years. However, no studies on residue persistence have been reported. The present study documents the extent of glyphosate residues in several crops and the rate of degradation in soil.

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Materials and Methods

Field Experiment. The experimental sites were at Canard, Nova Scotia, on a sandy loam soil. The soil in site 1 had a pH value of 5.4, organic matter content of 2.7% and a particle-size distribution of 81% sand, 8% silt, and 11% clay. Glyphosate at the rate of 2.75 kg a.i./ha in 300 l water was applied to the whole area in October to control mature weeds that are killed by this treatment. In the following spring the area was rototilled to a depth of 15 cm and divided into 16 plots of 4.5 x 10.5 m with 1.2-m strips separating them and arranged in 4 blocks. About 5 weeks later, when the weed regrowth was about 10 cm high, three plots from each block were sprayed with glyphosate at the rate of 2 kg a.i./ha in 300 l water with a hand sprayer then each plot was randomly seeded with barley (*Hordeum vulgare* L.), oats (*Avena sativa* L.) and wheat (*Triticum aestivum* L.) individually arranged in rows. Site 2 was a fallow area of pH value of 4.8, organic carbon content of 2.9%, and a particle-size distribution of 70% sand, 11% silt, and 19% clay and was divided into four plots of 5 x 7 m with 1-m strips separating them. In late May, three of the four plots were sprayed with glyphosate at a rate of 4.25 kg a.i./ha in 300 l water with a hand sprayer. Ten days later, the plots were rototilled to a depth of 15 cm and each was seeded with oat, sweet corn (*Zea mays* L.), beans (*Phaseolus vulgaris* L.), peas (*Pisum sativa* L.), carrots (*Daucus carota* L. cv. Nantes), and red beets (*Beta vulgaris* L. cv. Detroit dark red) individually arranged randomly in rows. The check plot was weeded by hand. At the appropriate harvest time, samples (about 300 g each) of corn kernels, barley, wheat and oat grains, bean and pea pods, and carrots and beet roots were taken at random from each row. Carrots and beet roots were washed with a brush and warm water to remove soil particles adhering to them, then wiped. Soil samples in site 2 to a depth of 15 cm were taken at random with a soil auger at intervals given in Table I. Samples were stored in plastic bags at -20°C until analysed.

The basic procedures described by Kramer (1976) and the Pesticide Analytical Manual (PAM) (1977) for the extraction and clean up of glyphosate and aminomethylphosphonic acid metabolite residues in soil and crop samples were employed. Analysis of the two compounds as their methyl trifluoroacetate derivatives was by flame photometric gas chromatography (Kramer 1976 and PAM 1977). Identity confirmation was made by concentrating appropriate aliquots of original extracts and employing thin-layer chromatography (Ragab 1978). The analytical technique was

Table I Dissipation and degradation of glyphosate in the upper 15 cm depth of a sandy loam soil.¹

Days after application	Glyphosate (remaining) ³		Aminomethylphosphonic acid ² (produced) ³	
	ppm ⁴	% ⁵	ppm ⁴	% ⁵
1	1.48	69.5	0.05	2.3
10	0.63	29.6	0.66	31.0
19	0.47	22.1	0.75	35.2
29	0.30	14.1	0.51	24.0
52	0.25	11.7	0.32	15.0
86	0.09	4.2	0.20	9.4
122	0.00	0.0	0.13	6.1

¹ Applied in the spring at 4.25 kg a.i./ha

² Glyphosate major metabolite

³ Dry weight basis

⁴ Assuming the weight of soil in the upper 15 cm depth of a hectare = 2x10⁶ kg

⁵ Of the originally applied 4.25 kg a.i. glyphosate/ha (2.13 ppm).

Residues of glyphosate and its aminomethylphosphonic acid metabolite from crops pre-emergent field treated with

Amount added (μg)	Glyphosate ²			Field-treated Amount added (μg)	Aminomethylphosphonic acid Fortified Amount found (μg)
	Fortified Amount found (μg)	Recovery (%)	(ppm)		
3.0	2.40	80.0	< 0.05	3.0	2.05
3.0	2.43	81.0	< 0.05	3.0	2.10
3.0	2.38	79.3	< 0.05	3.0	2.02
3.0	2.41	80.3	< 0.05	3.0	2.01
3.0	2.33	77.7	< 0.05	3.0	2.06
1.5	1.25	83.3	< 0.05	1.5	0.96
1.5	1.20	80.0	< 0.05	1.5	1.06
3.0	1.91	63.7	< 0.05	3.0	1.95
3.0	2.02	67.3	< 0.05	3.0	2.22

crops 30 g except beans and peas 15 g.

and ppm were on actual (wet) basis. Results were average of 4 determinations (fortified) and 6 determinations (field-treated) for its metabolite were found in crop checks. Detection limit of the method 0.05 ppm and < 0.05 denotes 0.005 ppm.

1 kg a.i. and 2 kg a.i. glyphosate/ha in October and following April, respectively.

1 kg a.i. glyphosate/ha in May.

verified using check soil and crop samples fortified at the extraction step with 0.1 ppm each of analytical standards of glyphosate and aminomethylphosphonic acid.

Results and Discussion

Table 1 shows that there was rapid disappearance of glyphosate in soil and no residues were left after 122 days of application. The same table shows also that glyphosate degraded to aminomethylphosphonic acid which, in turn, degraded rapidly. These results agree with those of other investigators (Sprankle et al. 1975 and Rueppel et al. 1977).

Table II indicates that there were no measurable concentrations of either glyphosate or its soil metabolite aminomethylphosphonic acid in any of the eight crops grown in the soil treated with glyphosate. The lowest concentration at which compounds could be reliably measured is 0.05 ppm. No injury symptoms such as wilting and yellowing of the plants as a result of glyphosate treatment were observed during the growing season.

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