

## Dissipation Studies of Metribuzin in Alfisol Soils and its Terminal Residues in Potato Tubers

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Paper No. 336

Received: 21 October 2015

Accepted: 26 May 2015

Published: 29 June 2015

### Abstract

A field investigation was conducted to determine dissipation behaviour in soil and terminal residues of metribuzin in potato crop applied at half the recommended dose ( $0.25 \text{ kg. ha}^{-1}$ ), recommended dose ( $0.50 \text{ kg. ha}^{-1}$ ) and double the recommended dose ( $1.00 \text{ kg. ha}^{-1}$ ). Soil samples at different time intervals after herbicide spray and potato tuber samples at the maturity of crop were collected, processed and subjected to metribuzin residue analysis by a validated gas chromatography method with an accepted recovery of above 80%. In all herbicide treatments, more than 90% of applied herbicide in soil dissipated within 45 days after herbicide application. Metribuzin residues in potato tubers at the time of harvest were below detectable levels. This indicated that the metribuzin did not leave any residues in potato tuber at any of the applied doses. From the study, it may be concluded that pre emergence application of metribuzin in potato for weed management could be considered safe, as its residues were below the prescribed MRL ( $0.05 \text{ mg. kg}^{-1}$ ).

### Highlights

- A method for extraction and analysis (by GC) of metribuzin residues in alfisol soil and potato tuber was standardized.
- Recovery efficiency of method was above 80%
- Dissipation of metribuzin at all three levels of application viz.  $0.25$ ,  $0.50$  and  $1.00 \text{ kg. ha}^{-1}$  in soil fitted first order kinetics decay curve.
- At the time of harvest metribuzin residues were below detectable level in potato tubers.

**Keywords:** Herbicide, recovery, half life, persistence, metribuzin

Potato, an important tuber crop which belongs to Solanaceae family is widely consumed in the world. It is fourth largest crop in world following wheat, rice and maize and rank fifth in terms of human consumption (Horton 1987). Potato crop occupies an important place both in nutrition and economic

well being of masses. It is high volume crop and yields substantially more edible energy, protein and dry matter per unit area and time because of short vegetative cycle than other crops. It is an excellent source of carbohydrates with low fat contents which makes it a balance food. It has wide adaptability

and can be grown in a varied range of agro climatic conditions. Severe infestation of weeds forms a negative factor in potato crop production because they compete with crop for space, water and nutrients and is responsible for marked losses in crop yield. Weed losses are more serious because fast early growth of potato is essential for high yield. The yield reduction due to weeds in potato is estimated to be as high as 10 to 80% (Lal and Gupta 1994, Kumar *et al.* 2007, 2008). In weed management practices, chemical weed control holds a great promise in dealing with effective and economic weed suppression. Though chemical weed control contribute gainful increment in crop production, yet it is important to make proper use of herbicides. The most important consideration in chemical weed control is persistence of herbicide in soil, and its residue in crop produce. The persistence of pesticide in the soil determines period of time for which it remains active. In case of herbicides, persistence is particularly important because on one hand it determines the period of time for which weeds can be controlled and on the other hand, it is related to the phytotoxic effects which can damage the subsequent crop (Weber 1993, Liu *et al.* 2005).

Metribuzin [4-amino-6-(1, 1-dimethylethyl)-3-(methylthio)-2, 5-triazin-5-(4H)-one], a triazine herbicide is recommended as both pre-emergence and early post-emergence to control of broad spectrum of weed flora in field and vegetable crops including potatoes (Majumdar and Singh 2007, Khoury *et al.* 2006). The site of action of this group of herbicide is PSII of photosynthesis and interferes with photosynthetic electron transport between primary and secondary acceptors of photosystem II. It is readily taken up by the roots and translocated to the shoots and leaves of treated plants in the apoplast (Walker *et al.* 1996, Sondhia 2005). Soils represent a complex biological, chemical and physical media. Different studies conducted to evaluate the degradation of metribuzin reported that most of degradation of metribuzin occurred as a result of aerobic microbial activity and followed pseudo first-order kinetics, and was influenced by

the temperature and organic substances in the soil (Savage 1977, Moorman and Harper 1989). Metribuzin is weakly basic which is protonated in acidic soil, and adsorbed by negative charges of colloids in the soil, decreasing as a consequence of its concentration in the soil solution. This means it is positively correlated with soil pH (Weber *et al.* 1993). Slow degradation of metribuzin in subsurface horizon soil is attributed to inherently lower microbial activity (Locke and Harper 1991, Locke 1992). However, Xu *et al.* (2000) reported rapid dissipation of metribuzin in sandy loam during the initial 10–15 days and concluded that leaching might be an important dissipation pathway for metribuzin under irrigated potato production. Similar results are also given by Khoury *et al.* (2003) who found very intense and rapid degradation and high mobility of metribuzin in sandy loam and clay soils. The persistence of herbicide in soil is the result of interaction of edaphic and climatic factors with herbicide. Herbicide residue estimation in soil and edible parts is very essential to determine herbicide activity in soil and its effect on crop quality. Field studies on persistence of metribuzin residues in potato crop in North western Himalayan region are very meager. The present study was undertaken with the objective to determine persistence of the metribuzin residues in soil and bioaccumulation in potato crop.

## Materials and Methods

### Reagents and chemicals

Metribuzin (99.8%) was obtained from AccuStandard, Inc., USA and purity checked by GC before use. Formulation of metribuzin (Sencor 70% WP) was purchased from local market. The solvents used in study were of analytical grade and purchased from Merck India Pvt. Ltd.

### Field experiment

Field experiment using a randomized block design with four replications pertaining to the residues of metribuzin in soil and potato tuber was conducted at the Research Farm of Department of Agronomy,



CSKHPKV, Palampur, (HP) India located at 32°06'N latitude, 76°03'E longitude and at an altitude of about 1290 meters above sea mean level. The experimental site falls in sub temperate mid hill zone of Himachal Pradesh. The soil of experiment field with silty clay loam in texture (25% sand, 41% silt and 33% clay) with acidic pH 5.22, CEC 12.9 centi-mol per kg (cmol. kg<sup>-1</sup>) and organic carbon 1.22%. The fertility status of experiment field was low in available N (205.1 kg. ha<sup>-1</sup>), P (25.0 kg. ha<sup>-1</sup>) and available K (230.9 kg. ha<sup>-1</sup>). Potato variety Kufri Jyoti was planted and metribuzin @0.25 kg. ha<sup>-1</sup>, 0.5 kg. ha<sup>-1</sup> and 1.0 kg. ha<sup>-1</sup> as pre-emergence application i.e. 48 hrs of potato sowing was applied by using a volume spray of 750 litre water ha<sup>-1</sup> with the help of Knapsack sprayer with flat fan nozzle. During growing crop season mean weakly maximum and minimum temperature was between 12.0-33.5°C and 2.4 to 19.2°C respectively. Crop experienced well distributed total rainfall of 445.4mm. The highest monthly total rainfall was recorded during month of February. The mean relative humidity during crop season was between 49.1-71.2%. The data on plant height, yield attributes and tuber yield were recorded and analysed following statistical analysis of variance procedure. Five soil cores were randomly taken from each of treated and untreated plots using auger up to the depth of 15cm at 0 (4hours), 3, 5, 7, 10, 15, 30, 45, 60, 75, 90 and 120 days after herbicide application. These cores were bulked together, air dried, powdered and passed through a 2 mm sieve to achieve uniform mixing. Potato tubers were collected at the maturity of the crop from metribuzin treated and untreated plots.

#### Extraction and cleanup

Metribuzin residues from soil and potato tubers were extracted as described by (Sondhia and Yaduraju 2005). Homogenized samples were taken in 200 ml conical flask and added 100 ml of acetonitrile and water (9:1, v/v). The contents were shaken for 1 hour by using rotary shaker. Thereafter soil was filtered through Whatmann filter paper and washed with an additional 25 ml acetonitrile: water solution.

The collected filtrate was transferred to separating funnel. Added 300 ml of water to it and extracted the solution thrice with 25 ml of dichloromethane. The dichloromethane fractions were pooled and passed through AR grade anhydrous sodium sulphate and were concentrated by using rotary flash evaporator at 40°C. Extract was cleaned by passing through selective adsorbent through a packed glass column (2 cm i.d. x 30 cm long). Packed the column in order, a small plug of glass wool at the bottom, 0.5 cm layer of anhydrous sodium sulphate, 5 cm layer of activated florisil and 0.5 cm layer of anhydrous sodium sulphate at the top. Washed the column with 25 ml dichloromethane. Loaded the column with dichloromethane extract obtained from sample and eluted the column twice each with 25 ml dichloromethane and concentrated the eluant by using flash evaporator. Dissolved the dry residues in hexane and the final volume was made up to 5 ml in graduated glass tube.

#### Residue analysis by gas chromatography

The gas chromatography (Perkin Elmer) equipped with 63Ni electron capture detector (ECD) and fitted with a column RTX 5 (30 m x 0.25 mm i.d.) with oven at isothermal conditions of 210°C was used for determination of residues. The injector and detector were maintained at 220°C and 240°C respectively. Nitrogen was used as a carrier gas with a flow of 25 ml /min.

#### Recovery experiments

The reference analytical standard of metribuzin was used for quantification, recovery and determination. In recovery experiments, the soil samples fortified with 0.05, 0.10 and 1.0 µg.g<sup>-1</sup> of metribuzin and potato tubers spiked at two levels of metribuzin i.e. 1.00 and 2.00 µg.g<sup>-1</sup> for assessing the recovery. Formulation sprayed in experiment was also tested for its active gradient.

#### Results and Discussion

The data presented in Table 1 revealed that plant height, plant population and haulms per plant were

not affected significantly by different application rates of metribuzin.

**Table 1.** Effect of different doses of metribuzin on plant height, yield attributes and tuber yield of potato

Treatments	Plant (cm)	Plant population (No /m)	No. of haulms/plant	Potato tuber yield (q. ha <sup>-1</sup> )
Metribuzin 0.25 kg. ha <sup>-1</sup>	55.3	4.7	3.5	200.9
Metribuzin 50 kg. ha <sup>-1</sup>	59.8	5.0	4.0	245.2
Metribuzin 1.00 kg. ha <sup>-1</sup>	62.9	5.0	3.7	216.7
Control	55.8	5.0	3.3	119.9
CD(P=0.05)	NS	NS	NS	35.2

Weeds in unweeded check (control) reduced the tuber yield by 51.1% over the best treatment i.e. metribuzin 0.5 kg. ha<sup>-1</sup>. The better tuber yield i.e. 245.2 q. ha<sup>-1</sup> owed to superior weed control due to metribuzin applied at rate of 0.5 kg. ha<sup>-1</sup> metribuzin 1 kg. ha<sup>-1</sup> being statistically at par with metribuzin 0.25 kg. ha<sup>-1</sup> was the next best treatment. However unweeded check (control) resulted into significantly lowest yield of potato tubers.

The method validated for metribuzin estimation by gas chromatography (GC) gave the retention time of reference material 5.9 minutes. The recovery experiment conducted with soil and potato tubers showed recovery percentage above 80%. The recoveries of metribuzin at different concentration levels were well within acceptable limits, confirming a good repeatability of the method. The assay results of commercial formulations Sencor 70% (WP) revealed that the active ingredient was 69.3% with  $\pm 0.3$  standard deviation. Metribuzin residues and % dissipation data in soil at different time intervals as detected by GC are presented in Table 2. The initial deposits of metribuzin in soil immediately after application of metribuzin 0.25, 0.50 and 1.00 kg. ha<sup>-1</sup> were 0.190, 0.328, 0.660 and after 3 days of herbicide application reached to 0.128, 0.247 and 0.585 g. g<sup>-1</sup> respectively. The corresponding per cent losses of

applied metribuzin at three levels i.e. 0.25 kg. ha<sup>-1</sup>, 0.50 kg. ha<sup>-1</sup> and 1.00 kg. ha<sup>-1</sup> were 32.63%, 24.69% and 11.36% respectively at three days after herbicide application. The data on % dissipation of herbicides revealed at three levels of metribuzin application i.e. 0.25, 0.50 and 1.00 kg. ha<sup>-1</sup> approximately 17.9, 19.21 and 27.73% of metribuzin remained in soil respectively at 15 days after herbicide application.

At 45 days after herbicide application, in all herbicide applied treatments more than 90% of herbicide dissipated. Per cent dissipation of metribuzin was 94.20% in metribuzin 0.50 kg ha<sup>-1</sup> followed by metribuzin 0.25 kg. ha<sup>-1</sup> (91.57%) and metribuzin 1.00 kg. ha<sup>-1</sup> (90.90%). The residues were below detectable level for metribuzin @ 0.25 kg. ha<sup>-1</sup> at 75 days after herbicide application indicating that applied metribuzin might have dissipated to 100% completely. However the concentration of metribuzin 0.50 kg. ha<sup>-1</sup> was 0.005 g. g<sup>-1</sup> and for metribuzin 1.00 kg. ha<sup>-1</sup> was 0.022 g. g<sup>-1</sup>. The corresponding % dissipation for metribuzin 0.25 kg. ha<sup>-1</sup>, 0.50 kg. ha<sup>-1</sup>, 1.00 kg. ha<sup>-1</sup> was 100, 98.47 and 96.60% respectively. The residues of metribuzin exhibited a declining pattern as a function of time. A perusal of data as evident in (Table 2) revealed that metribuzin at three application rates i.e. 0.25, 0.50 and 1.00 kg. ha<sup>-1</sup> persisted in soil up to 60, 75 and 120 days respectively. This indicates that metribuzin at higher doses persisted in soil for longer duration than lower doses. Metribuzin did not leave residues in soils beyond harvest of the crop at any of applied doses. Further, the examination of data (Table 2) depicts that rate of dissipation of metribuzin in soil was rapid which could be attributed to the favourable physico-chemical parameters like volatilization, vapour pressure, heavy rainfall, favourable temperature, high organic matter content and heavy texture of soil.

(Audus 1964, Sheets 1962, Khoury *et al.* 2003) also reported very intense and rapid degradation of metribuzin and concluded from the study that leaching by rainfall and degradation is important in disappearance of metribuzin from soil.



**Table 2. Residues of metribuzin in soil treated at different doses**

Days	Residues ( $\mu\text{g g}^{-1}$ )		
	Rates of metribuzin application ( $\text{kg. ha}^{-1}$ )		
	0.25	0.50	1.00
0	0.190 $\pm$ 0.003 (0)*	0.328 $\pm$ 0.07 (0)	0.660 $\pm$ 0.05 (0)
3	0.128 $\pm$ 0.002 (32.6)	0.247 $\pm$ 0.02 (24.6)	0.585 $\pm$ 0.06 (11.3)
5	0.098 $\pm$ 0.007 (48.4)	0.182 $\pm$ 0.03 (44.51)	0.433 $\pm$ 0.02 (34.3)
7	0.076 $\pm$ 0.007 (60.0)	0.154 $\pm$ 0.05 (53.0))	0.297 $\pm$ 0.02 (55.0)
10	0.052 $\pm$ 0.001 (72.6)	0.082 $\pm$ 0.02 (69.4))	0.192 $\pm$ 0.02 (70.9)
15	0.034 $\pm$ 0.009 (82.1)	0.063 $\pm$ 0.04 (80.7)	0.183 $\pm$ 0.02 (72.2)
30	0.025 $\pm$ 0.006 (86.8)	0.038 $\pm$ 0.05 (88.4)	0.122 $\pm$ 0.04 (81.5)
45	0.016 $\pm$ 0.003 (91.5)	0.019 $\pm$ 0.02 (94.2)	0.060 $\pm$ 0.02 (90.9)
60	0.008 $\pm$ 0.004 (95.7)	0.012 $\pm$ 0.01 (96.3)	0.042 $\pm$ 0.03 (93.6)
75	BDL**	0.005 $\pm$ 0.03 (98.4)	0.022 $\pm$ 0.02 (96.6)
90	BDL	BDL	0.014 $\pm$ 0.01 (97.8)
120	BDL	BDL	0.007 $\pm$ 0.05 (98.9)

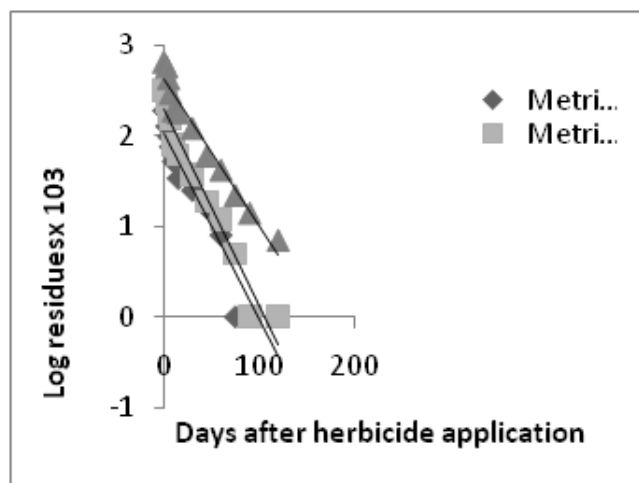
\*Values in parenthesis corresponds to % dissipation

\*\*BDL—Below detectable level

In general, rapid loss of herbicide applied at different rates i.e. 0.25, 0.50 and 1.00  $\text{kg. ha}^{-1}$  was noticed in initial 30 days. As the period of initial 30 days experienced high rainfall (191.2 mm) it is quite likely that metribuzin might have leached down or washed off due to rains. Similar results by (Sondhia 2005) of the rapid dissipation of metribuzin under irrigated potato production in sandy loam soil during initial 10-15 days in a study led to infer that leaching is an important dissipation pathway for metribuzin.

The logarithmic plots of herbicide residues vs time has been illustrated in Figure 1. These plots indicated that dissipation of metribuzin at all three levels of

application viz. 0.25, 0.50 and 1.00  $\text{kg. ha}^{-1}$  fitted first order kinetics decay curve.

**Figure 1.** First order dissipation curves of metribuzin at different doses in soil

The slope of curve, correlation coefficient and calculated half life of herbicides along with the regression equation are summarized in Table 3.

**Table 3. Slope of curve, correlation coefficient, half life and regression equation of metribuzin residues in soil**

Treatments	Slope of curve	half life days	Correlation coefficient	Regression equation
Metribuzin 0.25 $\text{kg. ha}^{-1}$	0.020	15.0	0.91	$Y = -0.020x + 2.063$
Metribuzin 0.50 $\text{kg. ha}^{-1}$	0.022	13.7	0.95	$Y = -0.022x + 2.327$
Metribuzin 1.00 $\text{kg. ha}^{-1}$	0.016	18.8	0.96	$Y = -0.016x + 2.624$

The correlation coefficient for all applied doses i.e. metribuzin 0.25  $\text{kg. ha}^{-1}$ , metribuzin 0.50  $\text{kg. ha}^{-1}$ , metribuzin 1.00  $\text{kg. ha}^{-1}$  were 0.91, 0.95 and 0.96 respectively indicating perfect fit. As time vs log residues plot is linear, dissipation followed first order kinetics reaction. These findings are in direct conformity with several other workers (Burgard *et al.* 1994, Workman *et al.* 1995, Gynor *et al.* 1998). The values for the half life of metribuzin 0.25, 0.50 and 1.00  $\text{kg. ha}^{-1}$  ranged from 13.7-18.8 days and were in direct conformity with findings of several other workers (Savage 1977, Johnson 2001).

At three levels of metribuzin application i.e. 0.25, 0.50 and 1.00 kg. ha<sup>-1</sup>, no peaks in chromatograms were observed corresponding to Rt value of metribuzin i.e. 5.9 minute clearly indicated that residues of applied herbicide were below detectable level in potato crop. The above findings are in conformity with results given by ( Jiyu *et al.* 2010, Al-mughrabi *et al.* 1999).

## Conclusion

On the basis of above findings, it can be concluded that metribuzin applied @ 0.25 kg. ha<sup>-1</sup>, 0.5 kg. ha<sup>-1</sup> and 1.00 kg. ha<sup>-1</sup> can be safely applied to the potato crop as pre-emergence as the residues were not detected at these application rates both in soil and potato tubers. However, metribuzin at above doses persisted in soil up to 60, 75 and 120 days respectively.

## Acknowledgements

Authors greatly acknowledge Head, Department of Agronomy Forages Grassland and management CSKHP Agricultural University, Palampur (India) for providing necessary facilities to conduct the work.

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