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Residue and Risk Assessment of Spirotetramat in/on Apple

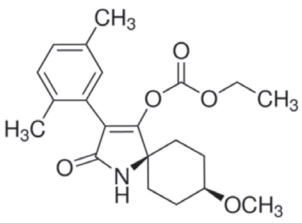
SK Patyal*, JK Dubey, Sapna Katna, Avinash Chauhan, Nisha Devi and Alam Ashraf Wani[1]
Department of Entomology, Dr Y.S. Parmar University of Horticulture and Forestry, Nauni, Solan 173 230, Himachal Pradesh (H.P.), India
[1]Research Centre for Residue and Quality Analysis (RCRQA) Laboratory of SKAUST, Kashmir, Jammu and Kashmir (J. & K.), India

Persistence of spirotetramat and its metabolites is reported on apple fruit under field conditions at different locations of Himachal Pradesh and Jammu and Kashmir during 2015. Experiments conducted in four orchards in the two apple growing states revealed that at Matiana (2000 m above sea level), and Thanedhar (2200 m above sea level), both in H.P., spirotetramat sprayed twice at 20 d interval @ 150g (standard dose) and 300 (double dose) g a.i ha^{-1} left initial deposits of 0.799-0.996 and 1.596-1.786 mg kg^{-1} respectively on apple fruit. At harvest, the residues in fruits as well as the soil were below determination level of 0.05 mg kg^{-1}. Half-life (RL_{50}) was 1.90-2.02 and 2.00-2.32 d at the standard and double dose, respectively. No residues of spirotetramat-enol metabolite was detected. In J. & K., at Youngora (1600 m above sea level), and Lassipora (1800 m above sea level) harvest time residues were below determination level with no enol metabolite being detected. The data will enable establishment of maximum residue limits by the FSSAI and suggest a safe waiting period for harvesting apple fruit.

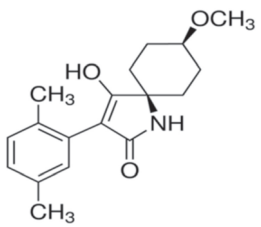
Key words: Spirotetramat, residues, half-life, apple

Apple (*Malus domestica*) is commercially cultivated in temperate zone and is fourth in order of fruit production in the world after banana, orange and grape. China is the leading nation in its production. In India, apple crop is mainly grown in the states of Jammu and Kashmir, Himachal Pradesh, Arunachal Pradesh, Mizoram, Sikkim, Tamil Nadu and Uttarakhand. Its production wise cultivation in Himachal Pradesh is mainly confined to eight districts viz. Shimla, Kullu, Mandi, Chamba, Kinnaur, Lahul and Spiti, Kangra and Sirmour (Saxena and Gandhi, 2014) or in the areas which experience 1000-1500 chilling hours, i.e. the number of hours during which temperature remains at or below 7^0C during the winter season. Low chilling cultivars of apple are becoming very popular amongst the farmers of the lower altitudes due to their early maturity and remunerative price in the market, but warmer climate aggravates insect-pest incidence including San Jose scale (*Quadraspidiotus perniciosus*), white scale (*Pseudoulacaspis* sp.), woolly apple aphid (*Eriosoma lanigerum*) and blossom thrips (*Thrips rhopalantennalis*) (Gupta, 2010, 2013; Gupta and Gupta, 2013; Sherwani *et al*., 2016) causing severe damage to the crop leading to reduction in fruit yield.

Spirotetramat (*cis*-3-(2,5-dimethylphenyl)-8-methoxy-2-oxo-1-azaspiro[4.5]dec-3-en-4-yl ethyl carbonate, *cis*-4-(ethoxycarbonyloxy)-8-methoxy-3-(2,5-xylyl)-1-azaspiro[4.5]dec-3-en-2-one) is a tetramic acid derivative insecticide (Fig. 1). It exhibits an excellent systemic and translaminar property, gets translocated within the entire vascular system, moves upwards and downwards through its xylem and phloem bundles, controls the hidden pests



Spirotetramat (Empirical formula $C_{21}H_{27}NO_5$; Molecular weight 373.44)



Spirotetramat-enol (Empirical formula $C_{18}H_{23}NO_3$; Molecular weight 301.38)

Figure 1. Chemical structures of spirotetramat and spirotetramat-enol

*Corresponding author E-mail: patyalsk@yahoo.co.in

like root aphids and protects new shoots or leaves appearing after its foliar application (Nauen *et al*., 2008; Schoevaerts *et al*., 2011). It inhibits lipid biosynthesis, affecting incomplete ecdysis, reduction in fecundity and fertility (Fischer and WeiB, 2008; Kuhnhold *et al*., 2008; Schoning, 2008). Psylla on pear is resistant to other group of chemicals but can be managed easily by spirotetramat (Jaworska *et al*., 2012). Spirotetramat is a proven effective chemical against species of whiteflies, aphids, scales, mealy bugs, psyllids and thrips on vegetable crops, cotton, soybean, pome and stone fruits, grapes, hops, citrus, nut trees, and bananas (Bruck *et al*., 2009). In apple orchards, application of miticides has been approved (Gupta *et al*., 2005) against population exceeding 2 mites leaf^{-1} threshold value which otherwise can easily be managed by using summer oil.

Apple fruits are consumed mostly as fresh fruit, juice, jam and wine. Pesticides applied during the fruit growth period can dissipate at a faster rate because of dilution effect due to plant growth (Sundaram, 1995; Xia *et al*., 1992). Appearance of mite infestation after growth pushes the growers to use spirotetramat as an alternative effective chemical, but its harmful residues are likely to be carried to the harvested produce and processed products, if applied close to harvest on fruits. Dissipation rate of the pesticides is one of the most important parameters in assessing their fate in the environment. Therefore, the present investigation was carried out to reduce the pesticide load below the maximum residues limit (MRL) through monitoring of persistence data and residue risk assessment of spirotetramat from the point of view of new molecule registration for inclusion in the list of Central Insecticide Board and Registration Committee (CIBRC).

MATERIALS AND METHODS

Chemicals and reagents

Movento 150 OD formulation, the certified reference spirotetramat (99.20% purity) along with its metabolite, spirotetramat-enol (99.80% purity) were supplied by Bayer CropScience Limited, India. Primary secondary amine (PSA), particle size 40 µm, was purchased from Agilent Technologies, USA. Anhydrous magnesium sulphate, sodium sulphate, sodium chloride and HPLC grade water were procured from Merck Specialties Pvt. Ltd. Worli, Mumbai. HPLC grade acetonitrile was purchased from Genetix Biotech Asia Pvt. Ltd., New Delhi. Nylon membrane filters of dimensions 13 and 47 mm (0.45 µm pore size) were procured from Rankem, New Delhi.

Preparation of standard solutions

Stock solutions of spirotetramat and its metabolite spirotetramat-enol were prepared in HPLC grade acetonitrile and suitably diluted to obtain the working standards of 0.03 to 0.50 ppm. Reagent blank was run along with the actual samples to ensure non-interference by solvents and reagents.

Instrumentation

The Shimadzu high performance liquid chromatograph (HPLC LC-20AT) was used for the estimation of spirotetramat and its metabolite spirotetramat–enol residues. HPLC alongwith degasser DGU-20A5, autoinjector SIL-20 AHT and SPD-M20A photo diode array detector (PDA) was supplied by Shimadzu Corporation, Kyoto, Japan. BONDAPAK™ Reverse phase (RP) C_{18} column (2.0 mm X 30 cm) was procured from Waters India Pvt Ltd. Robot Coupe high volume homogenizer (Blixer® 6V.V) and low volume high speed homogenizer (Silent Crusher M) were from Heidolph, Germany. Rotospin centrifuge tube shaker was procured from Tarson Products Pvt. Ltd., Kolkata. Centrifuge (Model 5804) was procured from Eppendorf India Ltd., Chandigarh. Turbo evaporator (Turbo Vap® LV) was from Caliper Life Sciences.

Experiment layout

Persistence behaviour of spirotetramat (Movento 150 OD) on apple was studied as supervised field trial in four different major apple growing areas *viz*. Location I (Matiana, 31.2102°N, 77.4056°E 2000 m, above sea level), Shimla and Location II (Thanedhar, 31.3183°N, 77.4458°E, 2200 m above sea level), Shimla, of Himachal Pradesh, India and two locations in Jammu and Kashmir *viz*. Location III (Youngora, 34.2164°N, 74.7719° E, 1600 m above sea level), Ganderbal, and Location IV (Lassipors, 33.8718°N, 74.8993°E, 1800 m above sea level), Pulwama. Spirotetramat (Movento 150 OD) was sprayed on fruit bearing apple trees at the rate @ 150 g a.i. ha^{-1} (standard dose) and 300 g a.i. ha^{-1} (double dose) @ 1000 L volume, two times with power sprayer at an interval of 10-25 d during 2015 crop season depending upon the environmental condition of the location. First application was given at fruit development stage. Two apple trees comprised single

Table 1. Meteorological conditions during experiment period (from 20th July to 2nd October 2015) at the four test locations

S. no.	Weather parameter	Location I (July 26 to Sept 7, 2015)	Location II (July 26 to Sept 7, 2015)	Location III (July 20 to Sept 20, 2015)	Location IV (Aug 07 to Oct 02, 2015)
1	Maximum temperature (°C)	22.03	22.03	29.35	26.09
2	Minimum temperature (°C)	12.81	12.81	15.31	12.23
3	Relative humidity (%)	77.63	77.63	69.00	55.27
4	Rainfall (mm)	336.00	336.00	320.00	149.50

replication and each treatment was replicated thrice. Untreated control plants were kept for comparison.

Meteorological conditions

Weather parameters like temperature, relative humidity (%) and rainfall (mm) were recorded during the experimental period from first spray till completion of experiment (Table 1).

Sample collection

Fruit samples (2 kg) for persistence study were collected from location I and II for residue estimation including dissipation behaviour at regular intervals of 0, 1, 3, 5, 7, 10, 15, 20, 25 d. However, harvest time fruit (2 kg) and soil (1kg from each replication of tree basin soil, depth 5 cm) samples at 30 d interval (post last spray) were collected and processed for residue analysis.

Sample preparation

The samples were processed and analysed in the Pesticide Residue Laboratory, Department of Entomology, Dr YSP University of Horticulture and Forestry, Nauni, Solan (H.P.). Residues of spirotetramat and its metabolite spirotetramat–enol were extracted and purified from apple fruits using QuEChERS (Sharma, 2013).

Fruits. Apple fruits (2 kg) were homogenized in Robot Coupe high volume homogenizer. The 15 g homogenized sample was transferred into 50 mL polypropylene centrifuge tube containing 30 mL HPLC grade acetonitrile and homogenized at 15000 rpm for 2 min in high speed homogenizer. Then 3 g sodium chloride was added into the tube and shaken at 50 rpm for 3 min in Rotospin followed by centrifugation at 3000 rpm for 3 min. Eighteen milliliter of upper organic layer was taken in 50 mL centrifuge tube containing 9 g anhydrous sodium sulphate to remove moisture content. From this 11 mL extract was transferred into 15 mL polypropylene centrifuge tube containing 0.4 g PSA sorbent and 1.15 g anhydrous magnesium sulphate for dispersive clean up. The tube was capped, shaken for 5 min at 50 rpm in Rotospin mixer and then centrifuged at 3000 rpm for 3 min. Six milliliter extract was transferred to the turbo glass test tube and evaporated to dryness in the Turbovap concentrator at 45°C in the presence of nitrogen. Residues were dissolved in 3 mL acetonitrile : water mixture (80:20 v/v), filtered through 13 mm nylon filter (0.45 µm pore size) and 20 µL was injected into HPLC.

Soil. Soil samples were extracted and cleaned up by using QuEChERS technique. Apple tree basin soil (1 kg) was air dried and passed through 2 mm sieve. Representative soil sample of 10 g was taken in 50 mL polypropylene centrifuge tube containing 20 mL HPLC grade acetonitrile, 4 g magnesium sulphate and 1 g of sodium chloride. Tube was shaken at 50 rpm for 1 min in Rotospin followed by centrifugation at 3000 rpm for 3 min. Approximately 10 mL of upper organic layer was taken in 15 mL centrifuge tube containing 1.5 g anhydrous magnesium sulphate and 0.25 g PSA. The tube was capped, shaken for 1 min at 50 rpm in Rotospin mixer and then centrifuged at 4000 rpm for 10 min. Transferred 4 mL extract to the test tube and evaporated to dryness in the Turbovap concentrator at 45°C in the presence of nitrogen. Residue was dissolved in 2 mL acetonitrile : water mixture (80:20 v/v), filtered through 13 mm nylon filter (0.45 µm pore size) and injected 20 µL into HPLC.

HPLC-PDA analysis

Spirotetramat and its metabolite spirotetramat-enol residues were estimated on liquid chromatograph equipped with photodiode array detector (PDA) and RP C_{18} column. The mobile phase acetonitrile : water (80:20 v/v) was run at a flow rate of 0.50 mL min^{-1}. Spirotetramat and its enol metabolite were detected at 229 nm. With these operating

parameters, the retention time of spirotetramat-enol and spirotetramat was 6.2 and 7.6 min, respectively.

Validation of analytical method

The efficiency and validity of an analytical method are determined by the agreement between the true value of analyte in the sample and the value obtained by its analysis. Measure of accuracy is expressed as percentage of the analyte recovered of the true value of the amount of spiking (Tiryaki, 2016). To validate the method / establish efficiency of the method and see the linearity of the pesticides, recovery studies were carried out by fortifying apple fruit and soil samples with spirotetramat and its metabolite spirotetramat-enol at 0.03, 0.15, 0.30 and 0.50 mg kg^{-1} and each spiking level was replicated five times employing QuEChERS multiresidue method. The average recoveries of spirotetramat and its metabolite spirotetramat-enol were more than 75 per cent in apple fruit and soil, in agreement with the guidelines of SANTE (SANTE, 2015). The accuracy values were evaluated by using four low to high concentrations of pesticides in apple fruits and soil. The precision of the method was expressed as the per cent relative standard deviation (% RSD) for replicate analysis of the spiked samples.

Data analysis

The residue data were subjected to statistical analysis according to Hoskin (1961) to compute the residue half life. Log residues were regressed on time interval to half-life of residues. Residue half life (RL_{50}) was calculated as $T_{1/2}$ = Log2/b, whereas $T_{1/2}$ = Residue half life (RL_{50}) in days; b = slope.

RESULTS AND DISCUSSION

Method validation

Spirotetramat and its metabolite spirotetramat-enol gave a good linear response at all proportionate concentrations with correlation coefficients (R^2) higher than 0.999. The linear response of all the standards is evident from chromatograph of matrix matched calibrations (Fig. 2). The data on the per cent recovery of spirotetramat and its metabolite spirotetramat–enol from fortified apple fruits and soil samples at 0.03, 0.15, 0.30 and 0.5 mg kg^{-1} levels are presented in Table 2. In fruits, the recovery of spirotetramat was in the range of 96.00-104.47 per cent and in soil, it was in the range of 103.93-117.87 per cent. The recovery of spirotetramat-enol from fruit was in the range of 94.53-

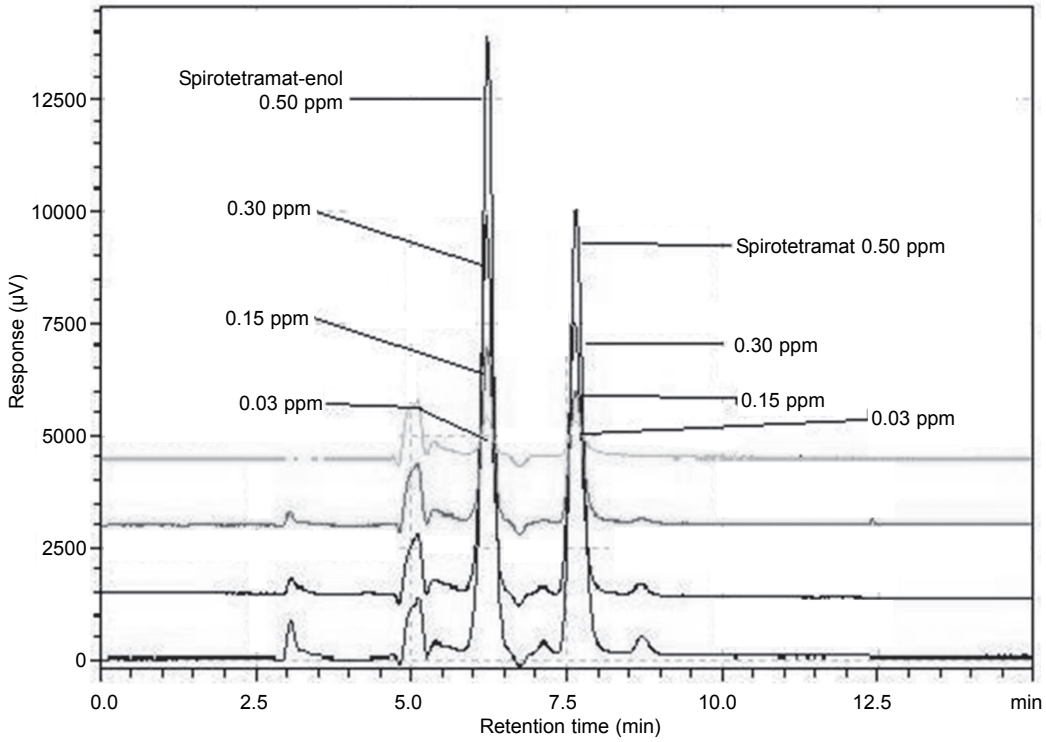


Figure 2. Overlaid chromatographic view of spirotetramat and spirotetramat-enol in spiked apple fruit

Table 2. Recovery of spirotetramat and spirotetramat-enol from apple fruit and tree basin soil

Fortification level (mg kg^{-1})	Mean (%) recovery* (%RSD)			
	Apple fruits		Soil	
	Spirotetramat	Spirotetramat-enol	Spirotetramat	Spirotetramat-enol
0.03	96.00 (7.86)	101.33 (3.80)	115.33 (3.80)	96.66 (7.17)
0.15	104.24 (2.80)	94.53 (3.50)	117.87 (3.50)	100.27 (3.60)
0.30	100.47 (1.90)	99.87 (3.34)	103.93 (3.33)	88.00 (6.77)
0.50	101.24 (7.19)	101.76 (4.07)	117.56 (4.08)	100.00 (6.84)

*Average of five replicates; RSD = Relative standard deviation

101.76 per cent and in soil, 88.00-100.27 per cent. The results are similar to Salazar-Lopez *et al*. (2015) in grape fruits and leaves (92.00-97.00 %); Schoning (2008) in tomato, avocado, citrus, potato and hops (84.00-104.0 %) and Singh *et al*. (2013) in okra, eggplant and pepper (82.00-97.00%). The precision of the method determined by per cent relative standard deviation (RSD) values for repeatability ranged from 1.90-7.86 for spirotetramat and spirotetramat-enol at different spiking levels in fruit and soil (Table 2). The limit of determination (LOD) of both spirotetramat and spirotetramat- enol in apple fruit and soil was 0.03 mg kg^{-1}. The calibration curves were linear over the test ranges of spirotetramat and spirotetramat-enol and correlation coefficients (R^2) were > 0.99 for both the analytes. The clean up procedure for this method was found to be efficient and as such no significant matrix effect was observed (Fig. 2).

Persistence of spirotetramat residues on apple fruits at Matiana and Thanedhar

The data on the persistence of spirotetramat on apple fruits at Matiana and Thanedhar along with its spirotetramat-enol metabolite, their degradation kinetics, residue half-life and pre-harvest intervals at the limit of quantitation (LOQ) of 0.05 mg kg^{-1} are presented in Table 3.

The fruits from both the locations were analysed for spirotetramat and its metabolite spirotetramat-enol residues. The initial deposits of spirotetramat were in the range of 0.799-0.996 and 1.596-1.786 mg kg^{-1} on apple fruits at both locations at the standard and double doses, respectively (Table 3). The residues of spirotetramat reached below its LOD of 0.03 mg kg^{-1} in 10 and 15 d at the respective two doses.

The residues were below LOD in both the apple fruits and soil samples collected at fruit harvest time from all the four locations. No residue of spirotetramat-enol was detected in any sampling up to the detection of parent spirotetramat molecule.

Two applications of spirotetramat at 144 g a.i. ha^{-1} in North Europe resulted in residues in the range of 0.14-0.17 mg kg^{-1} on apricot after a period of 7 d, while in Southern Europe from similar treatment, residues in the range of 0.05-0.07 mg kg^{-1} were reported. On peaches, spirotetramat yielded residues to the tune of 0 .14 mg kg^{-1} at the application rate of 180 g a.i. ha^{-1} in North Europe after 7 d. In Southern Europe same treatment reported residues in the range of 0.09-0.15 mg kg^{-1} after 7 d (Anonymous, 2008). The present results also showed similar trend in residue level after 7 d in the range of 0.051-0.075 and 0.139-0.190 mg kg^{-1} from the treatments at the standard and double doses, respectively (Table 3).

Metabolic study of spirotetramat in plants has shown that the major residues in apple fruits and leaves remained

Table 3. Persistence of spirotetramat in/on apple fruits at Matiana and Thanedhar

Interval (d)	*Spirotetramat residues (mg kg^{-1}±SD)			
	150 g a.i ha^{-1}		300 g a.i ha^{-1}	
	Location I (Matiana)	Location II (Thanedhar)	Location I (Matiana)	Location II (Thanedhar)
0	0.996±0.044	0.799±0.011	1.786±0.004	1.596±0.006
1	0.510±0.003	0.451±0.003	1.342±0.014	1.174±0.002
3	0.342±0.010	0.288±0.002	0.714±0.012	0.630±0.026
5	0.179±0.003	0.144±0.001	0.446±0.015	0.329±0.005
7	0.075±0.003	0.051±0.005	0.190±0.007	0.139±0.003
10	BDL	BDL	0.096±0.004	0.048±0.002
15	-	_	BDL	BDL
Control	ND	ND	ND	ND

*Mean of three replications; SD = Standard deviation; ND= Not detected; BDL = Below determination limit (0.03 mg kg^{-1})

Table 4. Statistical constants for spirotetramat on apple fruits

Location	Dose (g a.i. ha^{-1})	Correlation coefficient (r)	Regression equation (Y= a+bx)	$t_{1/2}$ (d)	WP (d)
Location I	150	-0.989	-0.051 -0.149 X	2.02	1.0
	300	-0.997	0.252 -0.129 X	2.32	3.0
Location II	150	-0.990	-0.112 -0.159 X	1.90	1.0
	300	-0.998	0.233 -0.153 X	2.00	3.0

WP= Waiting period based on MRL of 0.7 mg kg^{-1}

unchanged from the parent compound (Sur, 2008). The metabolite spirotetramat-enol is oxidized to BY108330-ketohydroxy and subsequently to BY108330-MA- amide (Babczinski and Hellpointer, 2008). Spirotetramat is also known to degrade quickly in soil. The metabolites generated from spirotetramat are not expected to accumulate in the environment since they are further degraded to form non-extractable residues and CO_2. Results of present study on persistence of spirotetramat on apple fruits and soil are are in agreement with faster degradation rate of spirotetramat-enol metabolite with the findings in grapes and soil (Vermuri *et al*., 2014), chilli fruits (Chahil *et al*., 2015; Mathew *et al*., 2012) and in mango fruit and soil (Mohapatra *et al*., 2012). However, Salazar-Lopez *et al*. (2015) reported spirotetramat-enol residues in leaves and grape fruits to an extent of 0.03 and 0.04 mg kg^{-1}, respectively on 1 d and 0.005 and 0.008 mg kg^{-1} after 5 d of its application @ 90 g a.i. ha^{-1} and thereafter, there was no significant difference in enol residues upto 48 d after application. Shukla *et al*. (2006) did not observe spirotetramat-enol residues in brinjal fruits on d 0 (1 h) but recorded 0.06 µg g^{-1} residues at double dose which reduced below the LOQ of 0.05 mg kg^{-1} on 3rd day.

Harvest time residues of spirotetramat in apple fruits and tree basin soil

No residues of spirotetramat and its metabolites were detected in apple fruits and tree basin soil at the LOQ level of 0.03 mg kg^{-1} at all the above four locations (H.P. and J. & K.) including at harvest.

Half-life and pre-harvest intervals of spirotetramat

The kinetic equation, half-lives, and coefficient (R^2) of the spirotetramat residue dissipation were calculated from the experimental data summarized in Table 4. The data revealed statistical conformity of the dissipation data of spirotetramat due to strong correlation coefficient (r= 0.990, 0.998) in apple. It followed first order kinetics which is based on the first order rate equation: $C=C_o\, e^{-kt}$ and $t_{1/2}=ln\ 2/k$, where C (mg kg^{-1}) is the pesticide concentration at time t, C_o is apparent initial concentration (mg kg^{-1}), k is rate constant, and $t_{1/2}$ the pesticide half-life in apple. The half life values varied between 1.90-2.02 and 2.00-2.32 d for spirotetramat at standard and double dose levels, respectively (Table 4). Faster half – lives of 1.91 and 1.30 d on green chilli fruits were observed by Chahil *et al.* (2015) following application rate of 120 and 240 g a.i. ha^{-1}. The correlation coefficient (r= 0.990, 0.998) indicated statistical conformity of the dissipation data to the first order kinetics. A pre-harvest interval (PHI) was calculated to be 3 d at both the single and double doses, which is also suggested as waiting period for safe consumption of apple fruits if MRL is set at the value of 0.7 mg kg^{-1} (Codex, 2017).

CONCLUSION

QuEChERS can be used for the analysis of spirotetramat and its enol metabolite in apple fruit and tree basin soil as all the validation parameters and the linearity for the compounds were in the acceptable range. On the basis of dissipation kinetics, half-life ranging from 1.9 to 2.32 d is suggested. Waiting period of 3 d is suggested at the standard dose (150 g ai ha^{-1}).

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