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Manish Kumar Verma
Junior Research Scholar, Department of Veterinary Pharmacology & Toxicology, G. B. Pant University of Agriculture & Technology, Pantnagar, Uttarakhand, India

Naveen Kumar
Ph.D. Scholar; Department of Veterinary Pharmacology & Toxicology, G. B. Pant University of Agriculture & Technology, Pantnagar, Uttarakhand, India

Correspondence
Manish Kumar Verma
Junior Research Scholar, Department of Veterinary Pharmacology & Toxicology, G. B. Pant University of Agriculture & Technology, Pantnagar, Uttarakhand, India

Determination of insecticide imidacloprid in soil by HPLC

Manish Kumar Verma and Naveen Kumar

Abstract
Imidacloprid is a new generation insecticide and highly active to protect the various vegetable crops, by controlling mites and insect pest. Imidacloprid is a systemic insecticide that enters the target pest via direct contact or ingestion. It is applied to seeds, soil and crops for controlling insects. Imidacloprid, a neurotoxic insecticide, belongs to family of the neonicotinoids. The analytical method using quick, easy, and effective for determination of insecticides in soil was developed using High Performance Liquid Chromatography (HPLC). 86 samples were collected from different region of U. S. Nagar, Uttarakhand to determine imidacloprid level, and positive samples were detected. The insecticide was extracted from soil using mobile phase acetonitrile and water (90:10, v/v). The determination of the target compounds were achieved in 8.0 min at flow rate of 1.0 ml/minute using reverse phase UV-detector for imidacloprid. The method showed that the limits of quantification (LOQ) ranged from 0.17- 0.25µg in all positive samples. A linear calibration curve was obtained by plotting area against concentration with a correlation coefficient of 0.995, while average recoveries were greater than 81 to 87 % in different samples. Out of 86 soil samples analysed 12 samples (10%) had detectable amount of residue levels for imidacloprid and 3 samples had residue levels above the recommended Maximum Residue Limit (MRL).

Keywords: insecticide imidacloprid, HPLC

Introduction
In modern agriculture practices for maximization of production increase in use of pesticides and fertilizers which results in contamination of the environment. Imidaclorprid pesticides are still common and authentic drug for the farmers to control pest. However, their indiscriminate use may lead to many adverse effects, such as toxicity in animals and human beings, bioaccumulation, pesticide resistance and the destruction of aquatic and other ecosystem etc (Baig *et al.,* 2012) [1]. It is used for controlling of termites, soil insects sucking insects and some chewing type insects (Meister, 2000) [7]. It is applied to seeds, soil, crops, and structures, and is used as a topical flea control treatment on domestic pets (Ishii *et al.,* 1994) [3]. Imidacloprid is rapidly moved through plant tissues after applications, and can be present in detectable concentrations in tissues such as leaves, vascular fluids, and pollen. Many non-target beneficial arthropods such as honeybees, parasitic wasps, and predaceous ground beetles are sensitive to imidacloprid. (Decourtye *et al.,* 2004) [2].
Imidacloprid is a systemic chloronicotinyl drug that enters in to the pest through direct contact or ingestion and causes disruption of nicotinic acetylcholine receptors in central nervous system of the insect (Tomizawa and john, 2005) [4]. The half life of the Imidacloprid in soil is reported to range from 29–190 days (Sarkar *et al.,* 2001) [11] and in case of wateris greater than 30 days depends on pH and temperature. High solubility and mobility of imidacloprid has the potential to leach into the ground water (Cox, 2001) [5]. The primary imidacloprid breakdown products in soil are imidacloprid urea, 6-hydroxynicotinic acid and 6-chloronicotinic acid all are similar toxic potential to their parent compound (Rouchaud *et al.,* 1996; Scholz, 1992) [6, 9]. The absorption level of imidacloprid is depending on soil properties such as organic carbon and minerals. As the organic carbon levels and laminar silicate clay content in the soil increase, the potential for imidacloprid to leach would decrease. Organic fertilizers, such as chicken and cow manure, increased the pesticide adsorption to the organic matter and increased its half-life (Cox *et al.,* 1997) [6]. The present study was chosen to know the level of imidacloprid residue in soil samples of Udham Singh Nagar district, Uttarakhand, India.

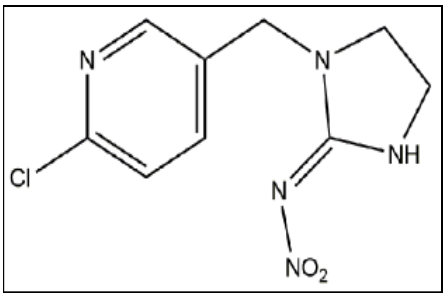

Fig 1: Chemical structure of Imidacloprid

Experimental method

Sample collection

The soil sample was collected from a pasture and agriculture land at a depth of 20 to 30 cm from the different spots of the land at pre monsoon period. The Samples was send to the laboratory on the collection day, after manual removal of stones and raw plant materials. The soil was sieved properly and the moisture content was maintained 40 to 60% using distilled water. The samples were stored in refrigerator at 4.0 ± 2.0 °C under aerobic condition.

Extraction procedure

A volume of 50 mL methanol was transferred into the conical flask containing (25g) fortified soil sample and allowed to stand for 2 hour. The conical flask was placed onto orbital shaker for 30 minutes. After shaking, the solutions were filtered through Whatman filter paper No.1 bearing a bed of anhydrous sodium sulphate. Solvent was evaporated through vacuum evaporator. The residue was re-extracted twice. The methanol extracts were collected, pooled and concentrated to smaller volume (5 to 10 mL) using vacuum evaporator at ≤ 40 °C. The concentrated extract was subjected to further clean up by column chromatography. A glass column packed with florisil asadsorbent placed in between two layer of anhydrous sodium sulphate was employed. The column was pre-conditioned with methanol and concentrated extracts were loaded onto the top of the column and eluted with 100mL acetonitrile @ 2 mL /minute. Eluate was concentrated to dryness using rotary vacuum evaporator at ≤ 40 °C and residue redissolved in 5mL acetonitrile. The samples were filter through Whatman No. 1 filter paper and final volume was made up to the mark with acetonitrile (Baruah and Barthakur, 1997) [10].

HPLC condition for Imidacloprid

Oven temp: 28ºC
Flow rate: 1.0 ml /min
Retention time: 8 minute
Wavelength: 280 nm
Detector: UV-VIS

Table 1: Data for linearity imidacloprid standard

Conc. (µg/ml)	Peak area
0.01	1018.35
0.1	8836.33
1	65264.32
2	112128.6
4	198157.4
8	364514.8

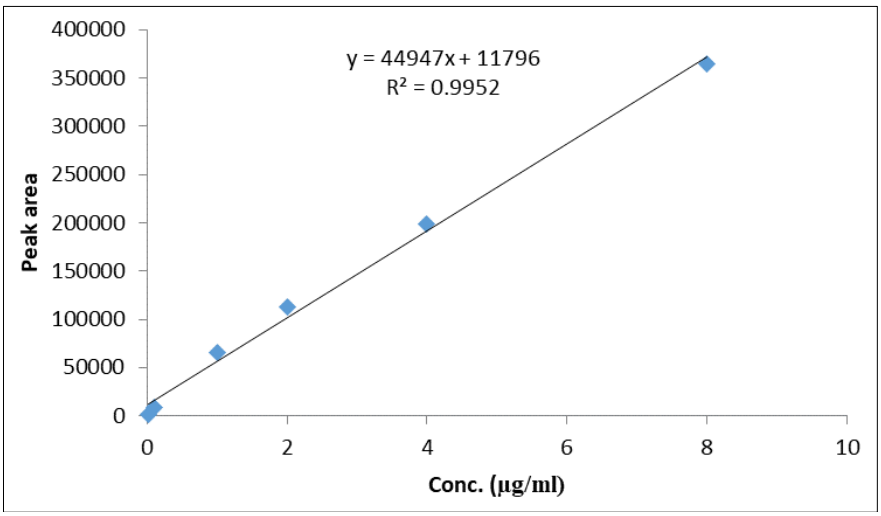

Fig 1: Linear graph of Imidaclorprid standard

Result and Discussion

In soil sample Imidacloprid estimated by high performance liquid chromatography with reverse phase column (RP-HPLC). The chromatogram (Figures 2 and 3) showed a well resolved peak of imidacloprid and the retention time was at 8.0 minutes under the operating condition of the chromatogram. Under the above experimental conditions, out of 86 samples found 12 samples are positive and 3 samples were above maximum residue level (MRL) for imidacloprid residues in soil. The concentration ranges from 0.01-5.1 µg/kg. These results suggest that imidacloprid has potential to persist in soil. In soils that are very porous or gravelly imidacloprid may move into ground water.

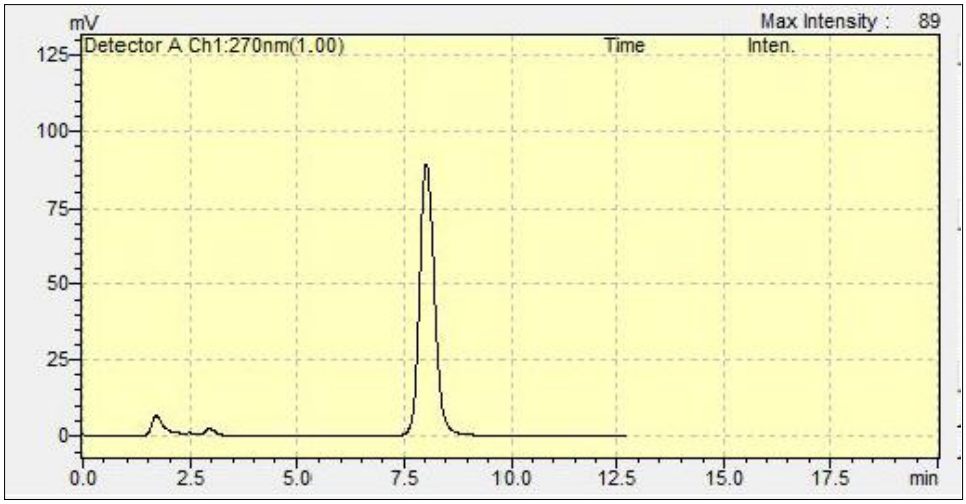

Fig 2: Chromatogram of standard Imidacloprid

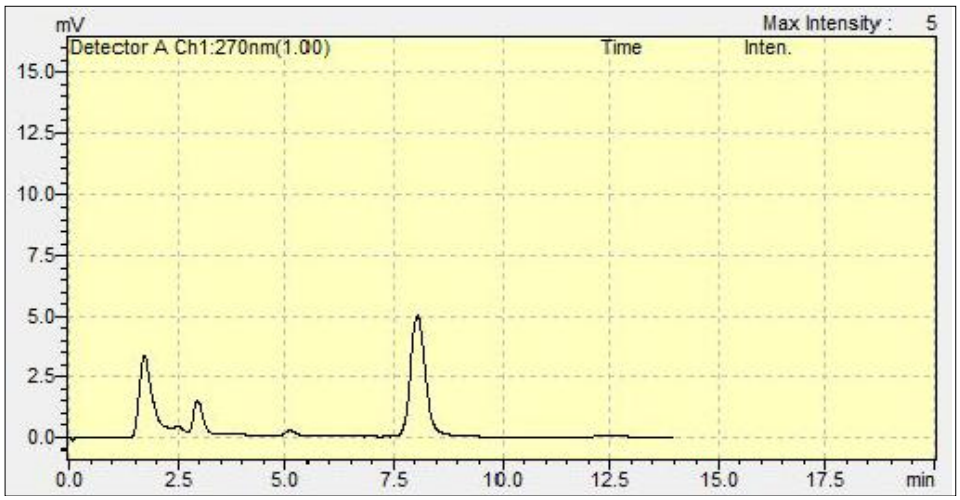

Fig 3: Chromatogram of Imidacloprid in soil sample

Conclusion

The method is satisfactory and subsequently used for extraction, clean up and estimation of imidacloprid from soil Limit of detection (LOD) was 0.01µg/ml that proves the excellent sensitivity, selectivity and precision of the method. Therefore, this method may be adapted to determine the imidacloprid residue level in soil. The study reveals that the extraction of samples with acetonitrile is suitable for imidacloprid analysis. The separation and quantification of imidacloprid by RP-HPLC is better at wavelength of 280 nm. The method is best for rapid precise analysis of imidacloprid in soil sample.

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