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Development and Validation of HPLC Analytical Method for Chlorantraniliprole in Bulk and in the Formulation

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Background: Chlorantraniliprole is widely used as a pesticide. It is only soluble in dimethyl formamide. However, most of the reported methods used acetonitrile and other solvents.

Aim: To develop rapid, accurate, precise, and sensitive HPLC method for chlorantraniliprole.

Materials and methods: 10 µg/mL of chlorantraniliprole containing solution was injected into the HPLC system and run in different solvent systems. 10 µg/mL solutions of chlorantraniliprole was injected in a column with 20 µL microsyringe. The chromatogram was run for appropriate minutes with mobile phase water. The flow rate was set to 1 mL/min and detection was carried out at wavelength 270 nm. The method is validated by measuring the limit of quantitation, limit of detection, repeatability, intraday precision, inter-day precision, and accuracy. Analysis of marketed formulation, bringle, and tomato for chlorantraniliprole content was also made for the developed analytical method.

Results: There was linearity of chlorantraniliprole for calibration curve in between 1 and 5 µg/mL concentration. The perfect sharp peak observed in water at a retention time of 6.28 min. The limit of detection and the limit of quantitation were 0.0050 µg/mL and 0.0152 µg/mL, respectively. The study reported 99.55% repeatability, 99.49% intraday precision, 99.65% interday precision, and 99.27% accuracy.

Conclusion: The rapid, accurate, precise, and sensitive HPLC method for the detection of chlorantraniliprole using dimethyl formamide was developed.

INTRODUCTION

Pesticide can be defined as a substance or a mixture of compounds intended for destroying, preventing, or controlling any pest, including human or animal disease, or insect attack on plants or animals, causing harm during production, related processing, storage of that, transportation of food, or marketing of any food, or substances and related agricultural commodities, or animal feedstuffs, that may be administered to animals for the control of insects or other pests in their bodies. The term includes substances intended for use as a plant growth promoting agent for retreating fruit and vegetables or stopping the premature reduction of produce. Also used as elements applied to crops either before or after harvest to protect the commodity from deterioration during packing and transference.^{1,2} Algaecides, antimicrobials, bio pesticides, desiccants, defoliant, disinfectants, fungicides, herbicides, illegal and

counterfeit pesticides, insecticides, insect growth regulators, minimum risk pesticides, miticides, molluscicides, plant growth regulators, ovicides, pheromones, natural and biological pesticides, rodenticides, repellents, wood preservatives are used as pesticides.³

Chemically, chlorantraniliprole (CNL) is 5-Bromo-2-(3-chloro-2-pyridine-2-yl)-2H-pyrazole-3-carboxylic acid (4-chloro-2-methyl-phenyl)-amide. It has a molecular formula of $C_{18}H_{14}N_5O_2BrCl_2$, the molecular weight of 483.14606 g/mol. The melting point range is 200-210°C, pKa is 10.88 ± 0.71 . It is soluble in a solvent like hexane, chloroform, ethyl acetate, methanol, acetone, acetonitrile, and water but practically, it is only soluble in dimethyl formamide (DMF).^{4,5}

CNL is widely used as a pesticide on vegetables, fruits and major crop for larval control. Its excessive use on vegetables and fruits produces a very

toxic effect on children and also on adults. Major toxic effect reported are aberration and polyploidy.

One study conducted field trial on cabbage and cauliflower for the study of residues of CNL and suggested that the washing samples with tap water removed 17-40% of the CNL while by boiling samples it removed 100% of CNL residues.⁶

A number of studies reported on the detection of CNL by HPLC and UV analytical methods. It is only soluble in DMF as per theoretical and practical solubility data. But most of the reported method used acetonitrile and another solvent except DMF. So it was an interesting point to focus on the solubility of it for method development and discover the suitable sample preparation condition.

Proposed method for increasing solubility of chlorantraniliprole is dissolved it in one or combination of solvent(s) like methanol, ethyl acetate, DMF with or without sonication.

AIM

To develop rapid, accurate, precise, and sensitive HPLC method for chlorantraniliprole.

MATERIALS AND METHODS

MATERIALS

CNL was procured as gift sample from Dupont, Mumbai, India. Methanol, acetonitrile, DMF was purchased from Merck laboratories, India.

METHODS

Sample of CNL was subjected to measure melting point by melting point apparatus (Remi equipment, Mumbai, India) and FTIR scan by Infra 3000A FTIR Shimadzu, Japan⁷ in the scanning range of 400-4000 cm^{-1} and resolution 1 cm^{-1} .

MOBILE PHASE SELECTION

10 $\mu\text{g/mL}$ of CNL containing solution was injected into the HPLC system (1600 EX, Cyber Lab, India, Column: Inertsil C18, 4.6 mm I.D. $\times$ 250 mm, 5 μm , Injector: Rheodyne 7725i, Pump: EX 1600 HP, UV deuterium, detector) and run in different solvent systems.⁸

PREPARATION OF MOBILE PHASE

HPLC grade water was filtered through 0.45 μm , 47 mm membrane filter paper then ultrasonicated for 20 minutes on ultrasonicator (Ultrasonic bath, 1.6 L, Remi equipment, Mumbai, India).

CHROMATOGRAPHIC SEPARATION

10 $\mu\text{g/mL}$ solutions of CNL were injected in a

column with 20 μL microsyringe. The chromatogram was run for appropriate minutes with mobile phase water. The flow rate was set to 1 mL/min . and detection was carried out at wavelength 270 nm (LT-2900, UV-Visible Spectrophotometer, Labtronics (I) Pvt. Ltd, Ambala, India). The chromatogram was stopped after peak achieved completely. Data related to peak like area, height, retention time, resolution etc. were recorded using software.

CALIBRATION CURVE

The calibration curves was derived for concentration against area under the curve. The Limit of Detection (LOD) and Limit of Quantification (LOQ) values were determined as per ICH guideline.⁹

VALIDATION OF METHOD

LINEARITY

CNL solution having concentration range having 1, 2, 3, 4, 5 $\mu\text{g/mL}$, and 20 μL of the concentration of drug was injected into the HPLC system two times separately and chromatography under the conditions was performed as described above. Chromatogram of the drugs was performed with UV detector (Dual Silicon Photodiode) at 270 nm. Peak areas were recorded. Standard calibration curves for CNL were plotted separately with concentration against the respective peak area.

PRECISION

REPEATABILITY

Standard solution containing CNL (3 $\mu\text{g/mL}$) was injected six times and area of peak was measured and % RSD was calculated.

INTRA-DAY PRECISION

A standard solution containing CNL (3 $\mu\text{g/mL}$) was analyzed six times on the same day and % relative standard deviation (% RSD) was calculated.

INTER-DAY PRECISION

A standard solution containing CNL (3 $\mu\text{g/mL}$) was analyzed six times on different days and % RSD was calculated.

LIMIT OF DETECTION (LOD) AND LIMIT OF QUANTIFICATION (LOQ)

Calibration curve was repeated six times and the standard deviation of the intercepts was calculated. Then LOD and LOQ were calculated as per **equation 1 and 2.**

$$\text{LOD} = 3.3 \times \frac{SD}{\text{slope of calibration curve}} \quad (1)$$

$$\text{LOQ} = 10 \times \frac{SD}{\text{slope of calibration curve}} \quad (2)$$

Where SD = Standard Deviation of intercepts of calibration curves.

ACCURACY

Recovery studies were carried out by applying the method to drug sample present in pesticide formulation to which known amount of CNL corresponding to 80%, 100%, and 120% of label claim was added (standard addition method). In 80% recovery study, 148 mg of CNL was added. In 100% recovery study, 185 mg of CNL added. In 120% recovery study, 222 mg of CNL was added. After the addition of the standards, 3 µg/mL concentrated solutions were prepared. The solutions were analyzed by the RP-HPLC method. At each level, six determinations were performed.

ROBUSTNESS

The evaluation of robustness should be considered during the development phase and depends upon the type of procedure under study. Robustness was performed by a small variation in the chromatographic conditions. The solution containing 10 µg/mL of CNL was injected into sample injector of HPLC three times.

ANALYSIS OF MARKETING FORMULATION

A quantity of pesticide equivalent to 185 mg of CNL accurately weighed (BL-220H, Shimadzu, Japan) and dissolved in 2 mL of DMF. The volume was made up to 100 mL with methanol and filtered through Whatman filter. The above solution sonicated for 10 minutes and diluted serially with methanol. The final solution obtained had a concentration of 3 µg/mL of CNL. This solution was analyzed using the developed method.

STABILITY STUDY OF DRUG

For photometric degradation, 1 g drug was put into UV light chamber on the wavelength of 250–400 nm and for oven degradation, 1 g drug was put into the oven at 50°C temperature.

From that 100 mg of the sample taken after a specific interval of time 1, 3 and 5 h respectively. The collected sample was added into 10 mL volumetric flask and drug dissolved with 2 mL DMF and

further volume make up to 10 mL with methanol and serial dilution did respectively to get 3 µg/mL concentrated solution of CNL. The sample was injected for a chromatographic run at 270 nm for 10 min.

SAMPLING

A total of 3 samples of various vegetables i.e. brinjal and tomato were commercially purchased from the locally marketed vegetables of Rajkot, India and some were collected from near villages of Saurashtra region, Gujarat, India, during December 2015 to March 2016 and served as blank or spiked samples. All samples were extracted fresh. The unit was generally 250 g.

EXTRACTION OF SAMPLES

All samples were extracted fresh. Each vegetable chopped into small pieces and macerate blender to make a fine paste. g of the paste was completely dry by placing into poetry plate in the oven (Yorko, USA) at 50°C temperature. The Dried paste was extracted with 10 mL DMF by sonicating sample for 10 min. and filter the extract through 0.45 µm membrane filter paper. From the above solution, 1 mL was taken and diluted up to 10 mL with methanol in a volumetric flask.

ANALYSIS OF EXTRACTION

All extracted samples were analyzed using RP-HPLC with UV detector at 270 nm wavelength.

RESULTS

A sample of CNL had observed the melting point of 202°C. The sample had FTIR peaks at 3257 cm⁻¹ (for N-H), 1635 cm⁻¹ (for C=O), 636 cm⁻¹ (for C-Cl), and 561 cm⁻¹ (for C-Br; **Fig 1**). Methanol is the best initial choice of organic solvent for the mobile phase. Acetonitrile-methanol mixture also has lower viscosity resulting in a somewhat higher number of plates and lower column back pressure than methanol-water (**Fig. 2A**). Chromatograms of CNL sample and marketed formulation in DML were showed in **Fig. 2 B** and **C**. The perfect sharp peak observed at a retention time of 6.28 min. The calibration curve (**Fig. 3**) showed linearity in the range of 1–5 µg/mL.

Statistical Validation for repeatability studies of CNL was reported as 99.55% mean, 0.688% standard deviation, 0.691% RSD, and 0.281 standard error. Statistical Validation for Intra-day Precision of CNL was reported as 99.49% mean, 0.616% standard deviation, 0.619% RSD, and 0.251 standard error.

Statistical Validation for Inter-day Precision of CNL was reported as 99.65% mean, 0.462% standard deviation, 0.463% RSD, and 0.188 standard error (table 1). LOD and LOQ of CNL were reported as 0.0050 $\mu\text{G/mL}$ and 0.0152 $\mu\text{G/mL}$.

Results of accuracy/recovery, robustness, analysis of marketed formulation by the developed method, stability studies, and detection of CNL residue in vegetables are presented in **Tables 2, 3, 4, 5, and 6**, respectively.

DISCUSSION

CNL sample had melting point range of 200–210°C. The test of melting point and FTIR confirmed the

purity of the sample.

It was found that methanol and water give satisfactory results as compared to other mobile phases. This mobile phase system was tried with different proportions and using different flow rates. Finally, the optimal composition water was taken as the mobile phase.

Method development was started with 100% methanol but the peak of the CNL was found poor. Then the mobile phase was adjusted by mixing acetonitrile with methanol in the ratio of 20:80. But the peak of CNL was found poor. To optimize the better peak, again phosphate buffer with pH 2.75 and water was tried. And finally, water was selected as mobile phase with a flow rate of 1 mL/min.

In previously developed LC–UV analytical method for determination of CNL, residues of CNL was extracted with acetonitrile and raw extracts were cleaned up with the same solvent. The LOD and the LOQ were 200 pG and <0.01 mg kg⁻¹.¹⁰ In availed developed HPLC–MS/MS analytical method for determining CNL, acetonitrile was used to extract CNL residues. The mixture was homogenized with some magnesium sulfate and sodium chloride at high speed. Centrifugation was done and the upper layer was collected and analyzed directly after serial dilution. LOQ was 0.01 mg/kg respectively.¹¹ In previously developed liquid

Figure 1. FTIR spectra of chlorantraniliprole.

Figure 2. Chromatogram A: Development of mobile phase. Concentration: 10 $\mu\text{G/mL}$, Mobile Phase: water, flow rate, 1mL/min; B: Chromatogram for chlorantraniliprole; C: Chromatogram of marketed formulation.

Figure 3. Calibration curve of chlorantraniliprole. 0.9916 correlation coefficient (R^2), 0.03 slope, and 20636 Y-intercept.

Table 1. Results of repeatability, Intra-day precision

Parameters	S. No.	Area	% Chlorantraniliprole found
Repeatability	1	35895.09	99.33
	2	35946.30	99.66
	3	35741.48	98.33
	4	35997.50	100.00
	5	36048.71	100.30
	6	35946.30	99.66
Intra-day Precision	1	36048.71	100.30
	2	35843.89	99.00
	3	35792.68	98.66
	4	35946.30	99.66
	5	35895.09	99.33
	6	35997.50	100.00
Inter-day Precision	1	35895.09	99.33
	2	36048.71	100.3
	3	35843.89	99.00
	4	35946.30	99.66
	5	35997.50	100.0
	6	35946.30	99.66

n = 6; Label Claim was 3 $\mu\text{G/mL}$ for all observations.

chromatographic analytical method for determining CNL, the CNL residue was extracted with methanol, then extracted with dichloromethane, and determined by HPLC–DAD. LOD was found to be 1.0×10^{-7} g and the lowest concentrations detected in soil and tomato were 0.005 mg/kg.¹² An additional LC/MS/MS for the analysis of residues of CNL in diverse

Table 2. Data of accuracy / recovery

Concentration Level (%)	Amount Spiked ($\mu\text{G/mL}$).	Area	Amount Recovered ($\mu\text{G/mL}$)	Recovery (%)
80	148	35792.68	2.96	98.66
80	148	35741.48	2.95	98.33
80	148	35843.89	2.97	99.00
100	185	35997.50	3.00	100.0
100	185	35792.68	2.96	98.66
100	222	35946.30	2.99	99.66
120	222	35895.09	2.98	99.33
120	222	35946.30	2.99	99.66
120	222	35997.50	3.00	100.0

Statistical Validation for recovery studies:

At 80% recovery 98.66% mean, 0.335% SD, 0.339% RSD, and 0.193 standard error,

At 100% recovery 99.44% mean, 0.696% SD, 0.699% RSD, and 0.402 SE,

At 120% recovery 99.66% mean, 0.335% SD, 0.336% RSD, and 0.193 SE.

SD: standard deviation, RSD: relativestandard deviation, SE: standard error.

Amount Present in all formulation were 185 $\mu\text{G/mL}$

Concentration of all solution was 3 $\mu\text{G/mL}$.

crops including citrus (fruit and juice) by liquid chromatography with mass selective detection using a triple quadrupole mass spectrometer (LC/MS/MS). In this technique CNL was extracted from the samples in two stages: first by soaking the samples in water for 20 minutes, followed by the addition of acetonitrile. The first extract was decanted and

Table 3. Data of robustness

Flow Rate (mL/min)	Change in Flow Rate		
	Level	Retention Time (min.)	Recovery (%)
0.9	-1	6.36	99.00
1	0	6.27	100.0
1.1	+1	6.15	98.99

Robustness was performed with $\pm 1\%$ variation in the volume and ± 0.1 mL/min in the flow rate of the mobile phase.

Table 5. Stability studies

Degradation	Sr. no.	Sampling time (h)	Area	Amount De- grade (μ G/mL)	Degradation (%)
Photometric	1	1	35639.07	0.07	2.30
	2	3	34307.74	0.33	11.0
	3	5	31337.85	0.91	30.3
Thermo*	1	1	35536.66	0.02	0.66
	2	3	33642.07	0.05	1.66
	3	5	31389.05	0.07	2.33

*Oven Degradation

Concentration of chlorantraniliprole in all sample was 3 μ G/mL

Table 4. Analysis of marketed formulation by developed method

Sr. No.	Area	Amount Recovered (μ G/mL)	Recovery (%)
1	35997.50	3.00	100.0
2	35895.09	2.98	99.33
3	35741.48	2.95	98.33
4	35895.09	2.98	99.33
5	35946.30	2.99	99.66
6	35843.89	2.97	99.00

Statistical Validation of marketed formulation (SC 20) was reported as 99.27% mean recovery, 0.574% SD, 0.578% RSD, and 0.234 SE

SD: standard deviation, RSD: relative standard deviation, SE: standard error.

Amount of chlorantraniliprole present in all formulation was 3 μ G/mL

Table 6. Detection of chlorantraniliprole residue from vegetables

Sr. no.	Name of vegetables.	Area	Amount of chlorantraniliprole found (μ G)
1	Bringle	23503.48	0.56
2		24476.38	0.75
3		24220.35	0.70
4		20646.24	0.76
5	Tomato	20641.12	0.58
6		20651.36	0.68

further acetonitrile was added for a second extraction. The two extracts were combined. An aliquot was taken, evaporated to dryness and re-dissolved in acetonitrile and water. The resulting solution was filtered by using a 0.22- μ m filter. Quantification was performed through LC-MS/MS. Recoveries for samples of citrus fruit and juice fortified at 0.01–0.1 mg/kg were acceptable.¹³ Standardized and validated analytical method for estimation of CNL residues present in different vegetables. Extraction was done with ethyl acetate and then reconstituted

with HPLC grade acetonitrile. Estimation was done using HPLC–PDA detector system, C18 column, and results were confirmed by a liquid chromatograph mass spectrometer (LCMS/MS), and high-performance thin layer chromatography (HPTLC). Acetonitrile: water (80:20, v/v) of HPLC grade was used as mobile phase, flow rate 0.4 mL/min. Retention time was 9.82 min. The limit of quantification of this method was 0.10 mg/kg.¹⁴ A novel developed an analytical method for residue analysis of CNL and its dissipation in grape. Ethyl acetate used for

extraction of pesticide and the extract was cleaned up with QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method and determined by HPLC–PAD technique. The LOD and LOQ were found to be 0.02 and 0.06 mg kg⁻¹, respectively.¹⁵ A developed analytical method for detection of CNL in brown rice, paddy water, rice straw, and soil, the residues of CNL pesticide was extracted with acetonitrile and determined by HPLC–ESI–MS/MS. LODs were 0.012 µg/L for paddy water, 0.15 µg/kg for soil, brown rice and rice straw.¹⁶ In the present study residues of CNL was extracted with DMF. Detection and quantification of CNL were performed by HPLC. LOD and LOQ were 0.0050 µg/mL and 0.0152 µg/mL.

CONCLUSION

The present study developed a rapid, accurate, precise, and sensitive HPLC method for the detection of chlorantraniliprole pesticide residue in the vegetables using dimethyl formamide. The method was validated by means of LOD, LOQ, % repeatability, % intraday precision, % interday precision, and percentage accuracy.

CONFLICT OF INTEREST

All authors declare that they have neither conflict of interest nor funding source.

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Разработка и валидация HPLC аналитического метода для обнаружения хлорантранилипрола в объёме и в формуле

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Введение: Применение хлорантранилипрола в качестве пестицида широко распространено. Его растворение возможно единственно при помощи диметилформамида. Независимо от этого, в большинстве известных методов используются ацетонитрил и другие растворители.

Цель: Целью настоящего исследования является разработка быстрого, надёжного, точного и чувствительного HPLC метода для обнаружения хлорантранилипрола.

Материалы и методы: 10 µg/mL хлорантранилипрола, содержащихся в растворе, были инъецированы в HPLC систему и введены в различные растворительные системы. 10µg/mL растворы хлорантранилипрола были инъецированы в колонку при помощи 20 µL микрошприца. Хроматограмму проводили в течение требуемых минут с водой в подвижной фазе. Поток воды был настроен на 1 мл/мин. Измерение осуществилось при длине волны 270 nm. Метод был валидирован по следующим параметрам: измерение предела количественного определения, предел обнаружения, повторяемость, прецизионность в рамках дня, воспроизводимость в рамках лаборатории и точность. Был проведён и анализ предлагаемых на рынке формул, баклажана и помидора на содержание хлорантранилипрола с целью усовершенствования аналитического метода.

Результат: Установлена линейность хлорантранилипрола для калибровочной кривой в концентрации между 1 и 5 µg/mL. Идеальный острый пик наблюдается в воде при времени удерживания 6,28 минуты. Предел обнаружения и предел количественного определения: 0.0050 µg/mL и 0.0152 µg/mL. Исследование установило 99.55% повторяемости, 99.49% прецизионности в рамках дня, 99.65% воспроизводимости в рамках лаборатории и 99.27% точности.

Заключение: Был разработан быстрый, надёжный, точный и чувствительный HPLC метод для обнаружения хлорантранилипрола при помощи диметилформамида.