Short Communication:

Determination of Deltamethrin Insecticide in Environmental Samples by Diazo-Coupling Reaction via Spectrophotometric Method

Fatimah Hameed Flayyih*, Abbas Noor Alshirifi, and Khudheyer Jawad Kadem

Department of Chemistry, College of Science, University of Babylon, Najaf–Hilla Road, Babil Province, Hilla 51002, Iraq

* ***Corresponding author:***

tel: +964-7810670125
email: fatima841110@gmail.com

Received: July 1, 2025
Accepted: August 5, 2025

DOI: 10.22146/ijc.108638

Abstract: *The aptitude to differentiate among various analytes is a vigorous feature of scientific exploration. In this study, a new azo dye exhibiting distinct sensing aptitudes was synthesized and characterized. Dye is the result of the interaction of deltamethrin insecticide with 5-amino-2-hydroxybenzoic acid in the alkaline medium at nearly pH 9 to form a stable yellow azo in the presence of acetone to enhance the solubility of deltamethrin insecticide. The formed compound was named Delta-P, and the maximum absorbance was detected at λ_{max} 402 nm. UV-vis, FTIR, ^{1}H-NMR, and ^{13}C-NMR spectra were used to characterize the diazo coupling reaction generation. The mechanism by which deltamethrin binds to 5-amino-2-hydroxybenzoic acid was explained. The Beer-Lambert law is obeyed in the 3–60 μg/mL range, which is considered excellent linearity over a wide concentration range, with high sensitivity and reproducibility. The detection and quantification limits gained highlight the significant sensitivity of the synthesized azo dye, ensuring the method's applicability for trace-level analysis. Environmental samples, including water and soil, are successfully analyzed, confirming the method's effectiveness. This finding indicates that the interaction can be used for colorimetric detection of deltamethrin in environmental samples and practical applications in quality control within agricultural and industrial settings.*

Keywords: *deltamethrin; coupling reaction; soil*

■ INTRODUCTION

The detection and quantification of insecticides in environmental and biological samples are critical for evaluating their impact on human health and ecosystems [1-2]. Deltamethrin (Delta) is a widely used synthetic pyrethroid insecticide known for its high efficacy against a broad spectrum of insect pests in both agricultural and public health applications [3-4]. It functions as a potent neurotoxin that disrupts the nervous systems of insects, leading to paralysis and death. Due to its powerful insecticidal properties, Delta has become a key component of pest control programs worldwide [5].

Delta is extensively applied across various sectors; however, it poses potential health risks to humans, particularly with prolonged or high-level exposure. Its toxicity is primarily linked to its action on voltage-gated sodium channels in the nervous system, resulting in symptoms such as headaches, dizziness, skin irritation, and, in severe cases, convulsions and respiratory distress [6]. The oral LD_{50} in rats ranges from 30 to 300 mg/kg, indicating moderate to high acute toxicity. Thus, understanding its toxicological profile is essential for assessing environmental and occupational safety [3-7].

In agriculture, Delta protects crops such as wheat, cotton, vegetables, and fruits from destructive insect infestations, thereby reducing economic losses and enhancing food security [8]. In public health, it is a key component of mosquito control programs aimed at preventing vector-borne diseases such as malaria and dengue fever [9]. Additionally, Delta is used in residential pest control treatments targeting cockroaches, ants, and bedbugs, and in veterinary medicine to control ectoparasites in pets and livestock [10].

Despite its benefits, Delta poses significant ecological and environmental concerns. Its widespread use and high toxicity to aquatic organisms, particularly fish and invertebrates, have raised concerns about pesticide residues in food, water, and soil [11]. Moreover, some insect populations have developed resistance due to frequent exposure, necessitating more careful management practices. Accurate determination of Delta residues in environmental samples is essential due to their extensive use and potential for ecological harm [12]. Recent modeling by the U.S. Environmental Protection Agency (EPA) indicates that peak environmental concentrations of Delta in both surface and groundwater can reach up to 0.20 μg/L, highlighting a potential risk to human health and aquatic ecosystems and reinforcing the necessity for sensitive and reliable detection methods [13].

Traditional analytical methods for detecting insecticides—such as gas chromatography (GC) [14] and high-performance liquid chromatography (HPLC) [15] are highly accurate but often expensive, time-consuming, and require sophisticated instruments [16-17]. A more cost-effective and efficient alternative is spectroscopic analysis, particularly ultraviolet (UV) spectrophotometry [18]. UV spectroscopy is a well-established method known for its speed, simplicity, and sensitivity in detecting various compounds [19-20]. Recent advances have improved UV spectrophotometric techniques by developing coupling reactions that enhance sensitivity and selectivity [21].

One approach involves combining Delta with 5-amino-2-hydroxybenzoic acid (5-A-2-HBA)—a reagent known for forming colored complexes with specific compounds [22-23]. This reaction produces a stable chromophore that can be identified and quantified using UV spectroscopy, offering a reliable method for Delta determination. The formation of the Delta–P complex improves the pesticide's UV absorbance characteristics, allowing for sensitive detection even in complex sample matrices [24-25].

Several studies have been conducted to determine Delta concentrations. For instance, one study used GC–mass spectrometry (GC-MS) to detect Delta residues in bovine milk following matrix solid-phase dispersion for extraction and cleanup [26]. Another investigation proposed ultrasound-assisted liquid microextraction combined with UV-vis spectrophotometry to determine Delta in food samples [27]. Additionally, Delta's immunotoxicity and the resulting tissue damage in the gills of gibel carp were assessed following acute exposure [28].

Therefore, this study introduces a novel, rapid, and cost-effective spectrophotometric method for the determination of Delta based on its diazotization–coupling reaction with 5-A-2-HBA. This reaction forms a stable, colored complex measurable by UV spectroscopy, eliminating the need for complex extraction procedures or advanced instrumentation. To the best of our knowledge, this specific coupling strategy has not been previously applied for Delta detection, marking a significant contribution to the development of accessible pesticide monitoring techniques.

■ EXPERIMENTAL SECTION

Materials

The following chemicals were used without further purification: Delta [(*S*)-cyano(3-phenoxyphenyl)methyl(1*R*,3*R*)-3-(2,2-dibromoethen-1-yl)-2,2-dimethylcyclopropane-1-carboxylate], 2.5% emulsifiable concentrate (EC, MEGHMANI ORGANICS Ltd.), 5-A-2-HBA (98%, Merck), sodium hydroxide (NaOH, 98%, LOBAChemie), hydrochloric acid (HCl, 35–38%, SDFCL), sodium nitrite ($NaNO_2$, 98%, CDH), acetone (99.8%, Sigma-Aldrich), and sulfamic acid (99%, Eisen-Golden Laboratories).

Instrumentation

The synthesized products were characterized by comparison with standard samples and spectral data, including UV-vis, FTIR, ^{1}H-NMR, and ^{13}C-NMR techniques. FTIR spectra were recorded using a Shimadzu FTIR 8400S spectrometer. UV-vis spectra were obtained with a Shimadzu UV-1800 Double Beam UV-visible spectrophotometer. ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a BRUKER 400 MHz spectrometer (Germany). Melting points were measured using a Stuart SMP30 apparatus (UK). The pH

measurements were conducted with a WTW pH meter, model 720 (Germany). Weighing was performed using a Sartorius sensitive digital balance, model BP 3015 (Germany).

Procedure

Diazotization of 5-A-2-HBA

In an ice bath (0–5 °C), 2.5 mL of 5-A-2-HBA (10^{-3} mol/L) was mixed with 0.5 mL of 1.0 mol/L HCl, followed by the addition of 2.0 mL of 0.5 mol/L $NaNO_2$. The mixture was stirred gently to allow the diazotization reaction to proceed. Then, 0.75 mL of 0.2 mol/L sulfamic acid was added to eliminate the excess nitrite, and the λ_{max} of 5-A-2-HBA was measured at 301 nm.

Preparation of the Delta solution

Delta solution (10^{-3} mol/L) was prepared by dissolving 0.050 g of Delta in 100 mL of distilled water. Then, 1.5 mL of this solution was mixed with 0.75 mL of 1.0 mol/L NaOH, and the λ_{max} of Delta was observed at approximately 266.5 nm.

Optimization of pH

A series of test solutions was prepared at varying pH levels (3, 5, 7, 9, and 11) to determine the optimal pH for the coupling reaction. The maximum absorbance was obtained at pH 9.

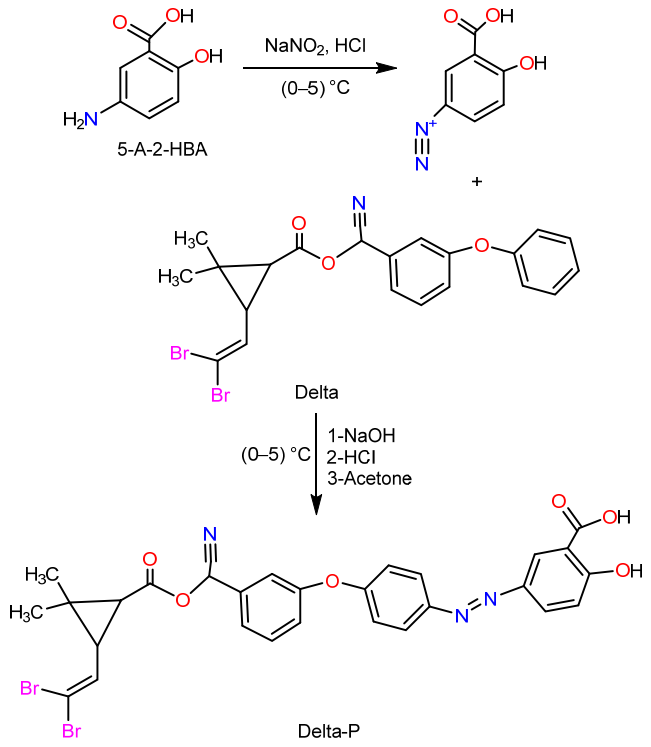
Coupling reaction and complex formation

The solution from step (2) was added to the diazonium salt solution from step (1), followed by the addition of 0.75 mL of acetone to enhance solubility. The final mixture was stirred briefly, and the total volume was adjusted to 25 mL with distilled water. The λ_{max} of the resulting Delta–P azo complex was measured at approximately 402 nm. Scheme 1 illustrates the synthesis of the azo compound, showing the initial and final expected reactions.

■ RESULTS AND DISCUSSION

Analytical Discussion

The synthesized azo compound was confirmed through spectroscopic techniques, including FTIR, ^{1}H-NMR, ^{13}C-NMR, and UV-vis spectroscopy. The absorbance of Delta-P was analyzed in the visible region, with a λ_{max} at 402 nm, which was used for the determination



Scheme 1. The proposed reaction between Delta and 5-A-2-HBA

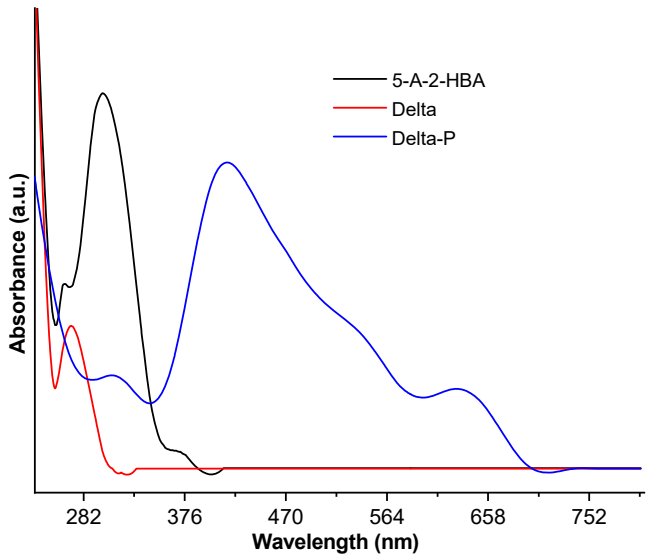


Fig 1. Absorption spectra of Delta at 266.5 nm, 5-A-2-HBA at 301 nm, and Delta-P at λ_{max} 402 nm

of Delta as depicted in Fig. 1. The formed precipitate was collected as a pure solid and colored material. FTIR signal at 3443 cm^{-1} indicates an O–H stretch in phenols, indicating the presence of a hydroxyl group; at 3077 cm^{-1} indicates a C–H stretch in unsaturated bonds

as alkenes; and at 2145 cm^{-1} indicates the presence of a C≡N (nitrile) group. At 1716 cm^{-1}, this strong absorption indicates a C=O group, found in ketones, aldehydes, or carboxylic acids. The bands at 1693 and 1678 cm^{-1} indicate additional vibrations of C=O groups. Two peaks of 1382 and 1189 cm^{-1}; these bands may be associated with C–H stretches in alkanes and C–N groups in amines. Lastly, 698 cm^{-1} indicates vibrations of aromatic C–H bonds, indicating the presence of an aromatic ring as explained in Fig. 2. The ^{1}H-NMR signals at 6.19–9.00 ppm indicate the presence of aromatic rings such as benzene or its derivatives, at ~3.49 ppm due to BrC=CH, peak at ~2.5 ppm due to DMSO-d_6, and at ~2.13 ppm due to protons attached to groups such as –CH_2– close to highly electronegative atoms such as ketones or amides. At 1.49–0.77 ppm, these multiple peaks indicate the presence of protons attached to alkyl groups (–CH_3, –CH_2–). Peaks at ~0.77 ppm indicate the presence of terminal methyl groups Signals at 1.0–1.5 ppm due to alkane groups such as –CH_2– or –CH_3 in an environment unaffected by highly electronegative atoms, as explained in Fig. 3. The ^{13}C-NMR signals at 160–180 ppm indicates the presence of C=O ester, at 118–140 ppm for aromatic carbonyl range, indicating the presence of a benzene ring and aromatic system (the multiplicity of signals indicates that the ring may contain different substituent groups), at 60–100 ppm due to C–O in ester and C–N in amines, at 39.82 ppm refers to aliphatic carbons (CH, CH_2, CH_3), and the signal at ~40 ppm indicates the presence of CH_2 and CH_3 groups, as explained in Fig. 4.

Optimization of Reaction Conditions

The optimum volume

The effect of different volumes of Delta (0.5–4.0 mL) with a concentration of 10^{-3} mol/L was studied. The results show that the best absorbance was obtained at a volume of 1.5 mL. Different volumes of 5-A-2-HBA, 10^{-3} mol/L, ranging from 0.5–4.0 mL, are used in the Delta spectrophotometric determination; the consequences show that the best absorbance was acquired at a volume of 2.5 mL. The effect of different volumes of 1.00 mol/L of NaOH solution, ranging from 0.25 to 1.5 mL, is used in the determination of Delta. The results mention that the best absorbance was at the optimum volume (0.75 mL). The effect of different volumes of HCl solution (1.00 mol/L) ranging from 0.25 to 1.5 mL was used in the Delta spectrophotometric determination. The results display that the top absorbance was found at 0.5 mL. An acidic medium is needed to protonate the amine group to make it more reactive to nitrous acid [29]. The effect of different volumes of 0.50 mol/L of $NaNO_2$ solution (0.5–3.5 mL) is used in the Delta determination.

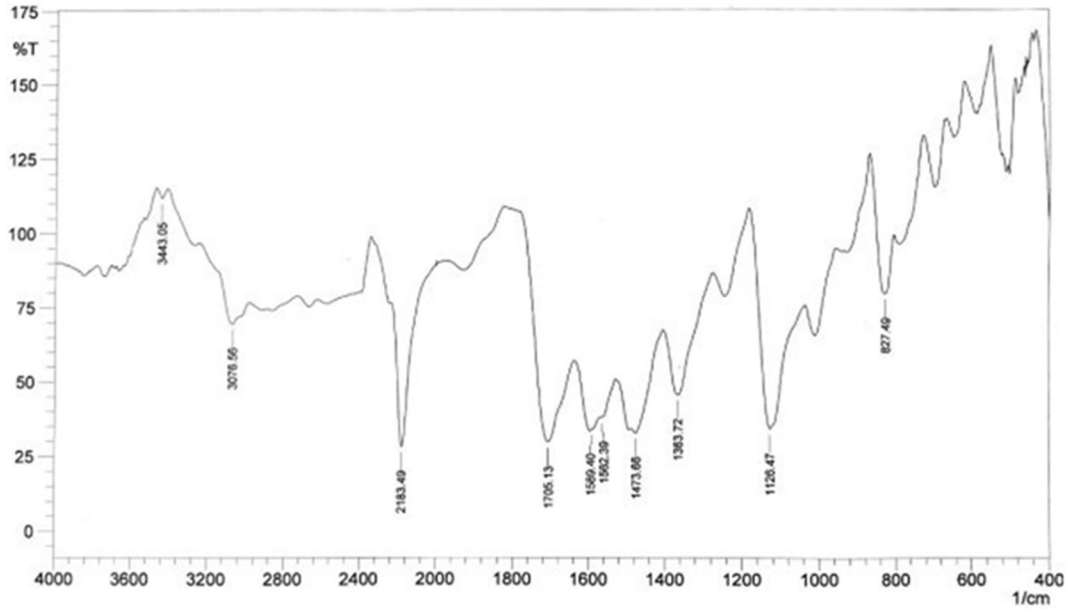


Fig 2. FTIR spectrum of the reaction of Delta with 5-A-2-HBA

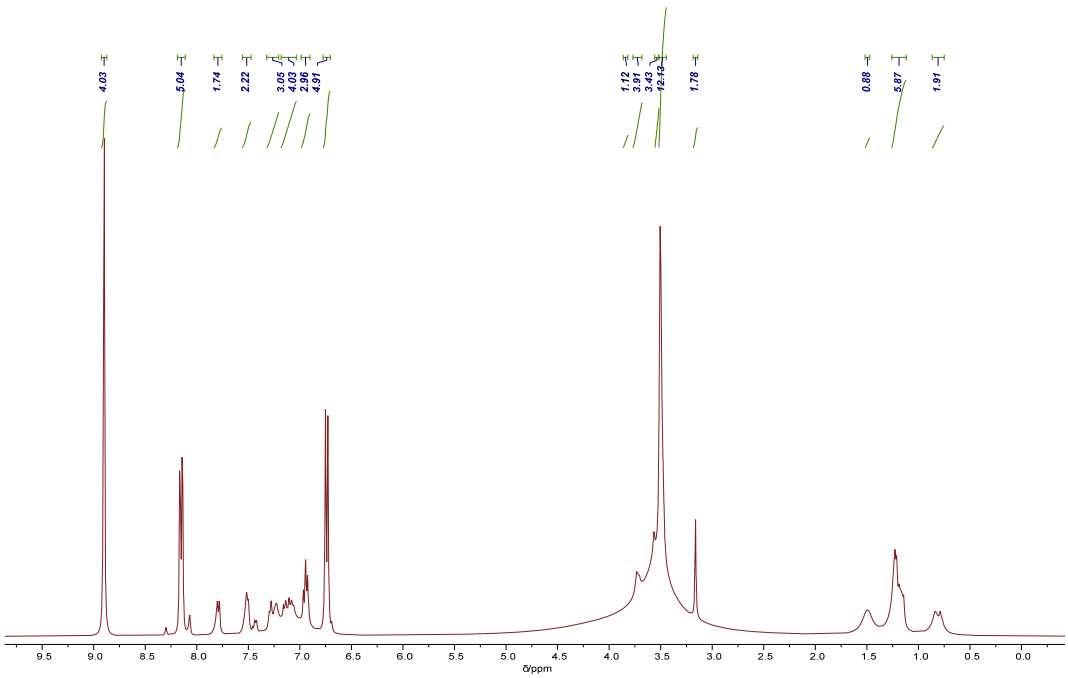


Fig 3. [1]H-NMR spectra of the product from Delta reaction with 5-A-2-HBA

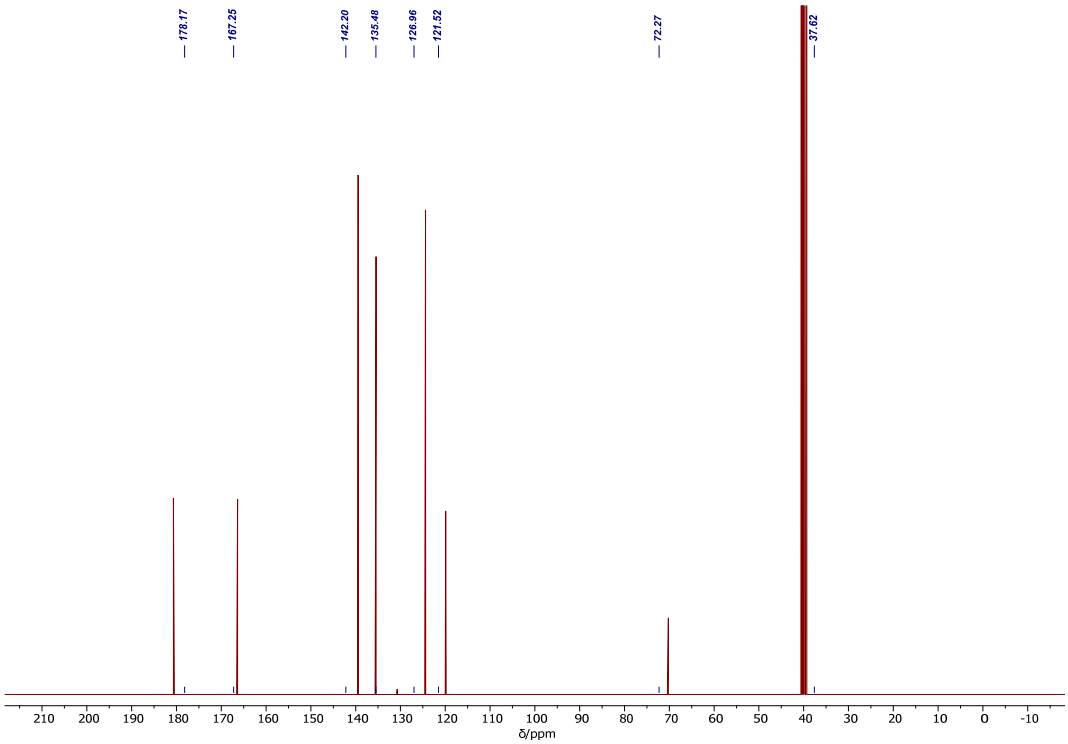


Fig 4. [13]C-NMR spectra of product from the reaction of Delta with 5-A-2-HBA

The best absorbance was obtained at a volume of 2.0 mL. $NaNO_2$ is used to simplify the conversion of the primary aromatic amines to diazonium salts [30]. The effect of different volumes of sulfamic acid (1.00 mol/L) ranges from 0.25 to 1.5 mL. Sulfamic acid plays an essential role in the diazo coupling reactions by providing the crucial acidic environment for the diazonium salt formation and acting as a coupling substance for azo compound

synthesis [31]. The best absorbance is 0.75 mL. The effect of acetone volume was also examined utilizing a range of acetone volumes. The spectrophotometric measurement of Delta uses 0.25–1.25 mL at various percentages (0.50–2.5%), respectively. The results indicate high absorption at 0.75 mL (1.5%). Since acetone is a polar aprotic solvent, it may dissolve a variety of polar and nonpolar compounds. It also improves clarity by increasing the solubility of Delta particles, which cause turbidity, in the solution [32]. Each volume recorded at specific concentrations has an influence on the new azo compound's absorption, as explained in Fig. 5 and 6.

The effect of time and temperature

The effect of the required time to complete the formation of diazonium salt for Delta was completed within 10 min, as indicated by the highest absorbance value observed at this time (0.463), with minimal variation in absorbance at longer durations, suggesting reaction stability. The formed azo compound revealed that it has somewhat good stability with the time passing; the results are explained in Fig. 7. The effect of temperature was studied by testing different temperatures on the absorbance and has been investigated in order to determine the Delta spectrophotometrically. The absorbance values are equal at 5 to 10 °C, then increase between 15 and 20 °C, after which they nearly stabilize at 20 °C, as appears in Fig. 8.

Optimization of pH

To determine the optimal pH for the reaction between Delta and 5-A-2-HBA, a series of buffer solutions with pH values ranging from 3.0 to 11.0 was prepared. The absorbance of the resulting dye was measured at each pH. The results showed that the maximum absorbance occurred at pH 9.0, indicating the

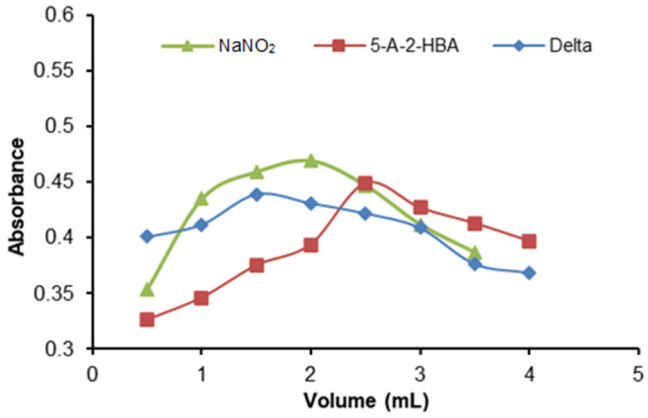


Fig 5. Volume effect of Delta, 5-A-2-HBA, and $NaNO_2$

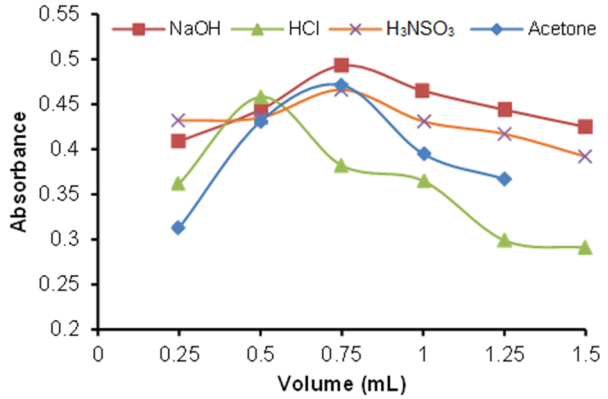


Fig 6. Volume effect of HCl, H_3NSO_3, NaOH, and acetone

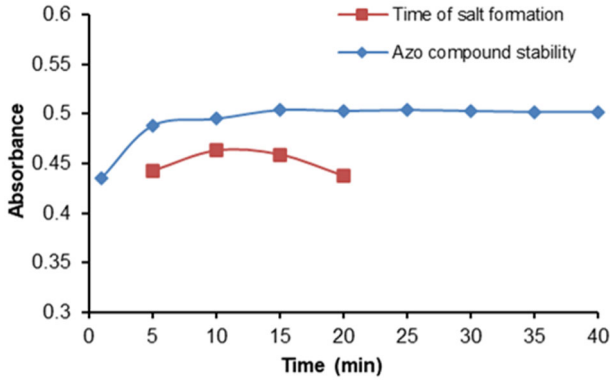


Fig 7. Effect of time on the diazotization salt formation and the stability of Delta-P

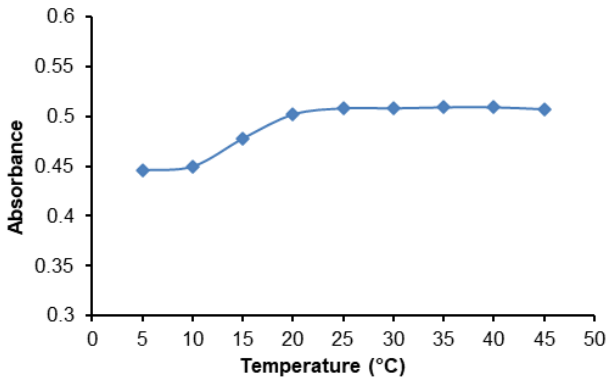


Fig 8. Temperature effect on the reaction of Delta and with 5-A-2-HBA

most favorable reaction conditions. At lower pH values, protonation of the amino group likely inhibited the diazotization–coupling reaction. In comparison, at higher pH values, possible degradation of reagents or instability of the colored product was observed. Therefore, pH 9.0 was selected as the optimal value for subsequent analyses [33], as appears in Fig. 9.

The Stoichiometric Chemical Reaction

The stoichiometry is calculated using molar ratio methods between 5-A-2-HBA and Delta. Diazotized 5-A-2-HBA solutions were prepared as a series of 25 mL volumetric flasks to provide 1.0 mL of 10^{-4} mol/L. In each flask, 0.50–4.00 mL of 10^{-4} mol/L Delta solution was added in increments, and the final volume was completed to the mark with water. At 402 nm, the absorbance of the resultant solutions was measured. As for Job's methods, Using a series of 25 mL volumetric flasks, increment volumes of 0.1–0.9 mL of 5-A-2-HBA solutions, and decrement volumes of Delta-P 0.9–0.1 mL with the same concentration (10^{-4} mol/L), the process was carried out as previously described, and the absorbance intensity data obtained at 402 nm were plotted against the mole fraction of insecticide for each indicator, this experiment is important to ensure that the method produces reliable results in the stoichiometry of azo-compound formation reactions. The results show that the ratio of Delta to 5-A-2-HBA was found to be nearly 1:1.

Analytical Parameters and the Suggested Method's Validity

In the calibration curve, different concentrations of Delta (3–60 µg/mL) were prepared at the optimum conditions with 5-A-2-HBA. The absorbance of the formed compound (Delta-P) was measured at 402 nm and plotted against the concentrations to acquire the calibration curve, as explained in Fig. 10. LOD and LOQ were determined by the plot obtained, and Table 1 shows analytical parameters of the proposed method.

Accuracy and Precision

For accuracy, three sample concentrations (10, 20, and 30 µg/mL) of the sample were used. In order to minimize the errors and ensure the results, the experiment has been repeated four times for all concentrations. The values of recovery percentage (rec%) show that the current method is accurate. Also for precision, three concentrations (40, 50, and 60 µg/mL) are created and

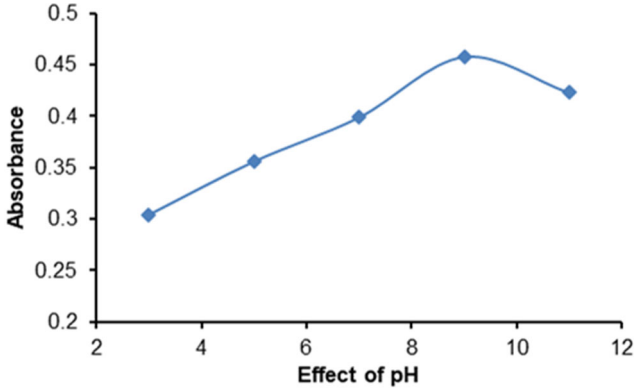


Fig 9. Effect of pH

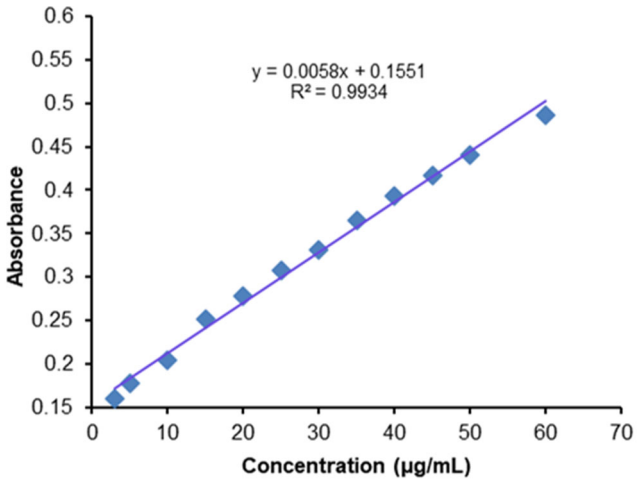


Fig 10. Calibration curve for the reaction between Delta and 5-A-2-HBA

Table 1. The analytical parameters of the proposed method

Parameter	Value
Linear range (µg/mL)	3–60
Slope	0.0058
Determination coefficient r^2	0.9934
Correlation coefficient r	0.9967
Intercept	0.1551
Molar absorptivity (L/mol cm)	2.930×10^3
λ_{max} (nm)	402
Limit of detection (LOD, µg/mL)	0.535
Limit of quantification (LOQ, µg/mL)	1.784

Table 2. The precision and accuracy details of the suggested method

The accuracy details of the suggested method				
Sample	Taken conc. (μg/mL)	Found conc. (μg/mL)	E% ± SD	Rec%*
1	10	09.29	−7.069 ± 0.0010	93.00
2	20	21.71	8.534 ± 0.0030	91.50
3	30	29.63	−1.207 ± 0.0035	98.80

The precision details of the suggested method					
Sample	Conc. (μg/mL)	Intra-day precision		Inter-day precision	
		Rec%	RSD%	Rec%	RSD%
1	10	90.04	1.259	92.00	2.349
2	20	96.00	1.002	98.00	1.759
3	30	93.40	0.208	95.30	0.517

*Mean of four determinations; E%: Relative error%, SD: Standard deviation, Rec%: Recovery, and RSD%: Relative standard deviation

Table 3. Details of Delta on the soil of the suggested method

Sample	Taken conc. (μg/mL)	Found conc. (μg/mL)	%Rec* after the application to the soil
1	40	13.086	32.72
2	50	15.845	31.69
3	60	21.017	35.03

*Mean of four determinations

Table 4. Comparative analysis of Delta determination methods

Technique	Linear range	RSD%	Rec%	LOD	LOQ	Ref.
GC + LC-MS/MS	0.003–0.1 μg/mL	< 20	71.69-117.9	0.003 μg/g	0.01 μg/g	[35]
GC –flame	-	< 5	93.0–97.0	0.03 μg/L	0.1 μg/L	[36]
LC-MS/MS	10–500 μg/kg	-	-	3 μg/kg	10 μg/kg	[37]
CFIA technique	0.5–18 μg/mL	0.88	98.30	0.14 μg/mL	0.48 μg/mL	[38]
GC-EI +MS/MS	-	≤ 20	60–102	2 μg/kg	10 μg/kg	[39]
GC-QTOF-MS	-	< 9	82–102	0.2–2 μg/kg	0.6–79 μg/kg	[40]
PADs-CL probe	0.3–10 μg/mL	-	-	0.15 μg/mL	-	[41]
CL probe	1.8–20 μg/L	-	-	0.3 μg/L	-	[42]
UV-visible	3–60 μg/mL	0.823	91.5–98.8	0.535 μg/mL	1.784 μg/mL	Current study

analyzed to verify the accuracy of the suggested method. The results show that the RSD% is less than 2, which means the precision is sufficient. The low rec% could be the result of Delta breaking down or evaporating during the analytical procedure [34]. All results of precision and accuracy are explained in Table 2.

Effect of Delta on Soil

After spraying the soil with the concentrations of 40, 50, and 60 μg/mL placed in plastic anvils, they were left for two weeks. As much as 1 g of the soil's surface has been skimmed, washed with 10 mL of distilled water, and filtered through the filter papers; then, all the steps required to prepare the resulting Delta-P compound were carried out, and the absorbance was measured. We note that the recovery values were very low. Such behavior is expected, as a large amount of the insecticide remained in the soil, which means an adequate quantity of Delta will seep into the soil, as displayed in Table 3. More than a statistical description of numerous essential methods used to determine Delta, and the suggested approach that was suitable for RSD% values and the recovery range is given in Table 4. This method can detect even the smallest pesticide quantities.

■ CONCLUSION

A new, low-cost, and simple technique for the determination of Delta based on the diazo coupling reaction mechanism has been suggested in this study. Amine compounds can be easily converted into aryl diazonium derivatives. The coupling reaction of Delta proceeds with high chemoselectivity and sensitivity, which is helpful for quality control and environmental monitoring. The identification and characterization of an insecticide's chemical structure might depend on the creation of a diazonium salt, which may expose the presence of specific functional groups or moieties in the Delta molecule. The toxicological results are one of the major aspects; diazonium salts may be more effective at combating insects than their parent molecule, so that they may change Delta's toxicity profile. The recommended approach for determining Delta is easy to use, quick, affordable, and sensitive.

■ ACKNOWLEDGMENTS

I want to express my gratitude to the Ministry of Agriculture for allowing me to complete my pesticide studies and research.

■ CONFLICT OF INTEREST

The authors have no conflict of interest.

■ AUTHOR CONTRIBUTIONS

Fatimah Hameed Flayyih: data curation, investigation, methodology, validation, visualization, writing-original draft, writing and review; Abbas Noor Alshirifi: validation and review; Khudheyer Jawad Kadem: data curation and formal analysis of data. All authors have approved the final version of the manuscript.

■ REFERENCES

[1] Zhou, W., Li, M., and Achal, V., 2025, A comprehensive review on environmental and human health impacts of chemical pesticide usage, *Emerging Contam.*, 11 (1), 100410.

[2] Singh, P.K., Kumar, U., Kumar, I., Dwivedi, A., Singh, P., Mishra, S., Seth, C.S., and Sharma, R.K., 2024, Critical review on toxic contaminants in surface water ecosystem: Sources, monitoring, and its impact on human health, *Environ. Sci. Pollut. Res.*, 31 (45), 56428–56462.

[3] Shi, T., Zhang, Q., Chen, X., Mao, G., Feng, W., Yang, L., Zhao, T., Wu, X., and Chen, Y., 2024, Overview of deltamethrin residues and toxic effects in the global environment, *Environ. Geochem. Health*, 46 (8), 271.

[4] Benavent-Albarracín, L., Pérez-Hedo, M., Alonso-Valiente, M., Catalán, J., Urbaneja, A., and González-Cabrera, J., 2025, Response of *Amblyseius swirskii* to deltamethrin, *Pest. Manage. Sci.*, 81 (6), 2800–2811.

[5] Davies, T.G.E., Field, L.M., Usherwood, P.N.R., and Williamson, M.S., 2007, DDT, pyrethrins, pyrethroids and insect sodium channels, *IUBMB Life*, 59 (3), 151–162.

[6] Oyovwi, M.O., Atere, A.D., Chimwuba, P., and Joseph, U.G., 2025, Implication of pyrethroid neurotoxicity for human health: A lesson from animal models, *Neurotoxic. Res.*, 43 (1), 1.

[7] World Health Organization, 2020, *The WHO Recommended Classification of Pesticides by Hazard and Guidelines to Classification 2019*, WHO, Geneva.

[8] Abubakar, M., Koul, B., Chandrashekar, K., Raut, A., and Yadav, D., 2022, Whitefly (*Bemisia tabaci*) management (WFM) strategies for sustainable agriculture: A review, *Agriculture*, 12 (9), 1317.

[9] Moreno Gómez, M., 2022, The Role of Insecticides and Repellents in Protecting Humans and as Tools in the Fight Against Vector-Borne Disease Transmission, *Dissertation*, University of the Balearic Islands, Spain.

[10] Montag, A., 2021, "Diseases Caused by Arthropods" in *Braun-Falco's Dermatology*, Eds. Plewig, G., French, L., Ruzicka, T., Kaufmann, R., and Hertl, M., Springer, Heidelberg, Berlin, 1–45.

[11] Lu, Q., Sun, Y., Ares, I., Anadón, A., Martínez, M., Martínez-Larrañaga, M.R., Yuan, Z., Wang, X., and Martínez, M.A., 2019, Deltamethrin toxicity: A review of oxidative stress and metabolism, *Environ. Res.*, 170, 260–281.

[12] Castells-Sierra, J., Guillem-Amat, A., López-Errasquín, E., Sánchez, L., and Ortego, F., 2023, First detection of resistance to deltamethrin in Spanish populations of the Mediterranean fruit fly, *Ceratitis capitata*, *J. Pest Sci.*, 96 (3), 1229–1242.

[13] U.S. Environmental Protection Agency, 2013, *Risks of Deltamethrin Use to Federally Threatened and Endangered Species: EPA Endangered Species Effects Determination*, Environmental Protection Agency, Washington, D.C., US, https://www3.epa.gov/pesticides/endanger/litstatus/effects/redleg-frog/2013/deltamethrin/analysis.pdf.

[14] Pico, Y., Alfarhan, A.H., and Barcelo, D., 2020, How recent innovations in gas chromatography-mass spectrometry have improved pesticide residue determination: An alternative technique to be in your radar, *TrAC, Trends Anal. Chem.*, 122, 115720.

[15] Fan, X., Tang, T., Du, S., Sang, N., Huang, H., Zhang, C., and Zhao, X., 2022, Simultaneous determination of 108 pesticide residues in three traditional Chinese medicines using a modified QuEChERS mixed sample preparation method and HPLC-MS/MS, *Molecules*, 27 (21), 7636.

[16] Dugheri, S., Mucci, N., Bonari, A., Marrubini, G., Cappelli, G., Ubiali, D., Campagna, M., Montalti, M., and Arcangeli, G., 2020, Solid phase microextraction techniques used for gas chromatography: A review, *Acta Chromatogr.*, 32 (1), 1–9.

[17] Zotou, A., 2012, An overview of recent advances in HPLC instrumentation, *Cent. Eur. J. Chem.*, 10 (3), 554–569.

[18] Shi, Z., Chow, C.W.K., Fabris, R., Liu, J., and Jin, B., 2022, Applications of online UV-vis spectrophotometer for drinking water quality monitoring and process control: A review, *Sensors*, 22 (8), 2987.

[19] Parmar, A., and Sharma, S., 2016, Derivative UV-vis absorption spectra as an invigorated spectrophotometric method for spectral resolution and quantitative analysis: Theoretical aspects and analytical applications: A review, *TrAC, Trends Anal. Chem.*, 77, 44–53.

[20] Qi, X., Lian, Y., Xie, L., Wang, Y., and Lu, Z., 2024, Water quality detection based on UV-vis and NIR spectroscopy: A review, *Appl. Spectrosc. Rev.*, 59 (8), 1036–1060.

[21] Ortiz-Martínez, M., Molina González, J.A., Ramírez García, G., de Luna Bugallo, A., Justo Guerrero, M.A., and Strupiechonski, E.C., 2024, Enhancing sensitivity and selectivity in pesticide detection: A review of cutting-edge techniques, *Environ. Toxicol. Chem.*, 43 (7), 1468–1484.

[22] Abd-El-Aziz, A., Li, Z., Zhang, X., Elnagdy, S., Mansour, M.S., ElSherif, A., Ma, N., and Abd-El-Aziz, A.S., 2025, Advances in coordination chemistry of Schiff base complexes: A journey from nanoarchitectonic design to biomedical applications, *Top. Curr. Chem.*, 383 (1), 8.

[23] Ceylan, B., Tekkeli, E.K., and Önal, C., 2022, Development of an HPLC method for the determination of mesalazine in human plasma by fluorimetric derivatization and application to a prototype pharmacokinetic study, *J. Fluoresc.*, 32 (1), 319–325.

[24] Faye, D., Lo, M., Seye, D., Diop, C.A., Sall, M.L., Diop, M.G., Gning, I., Bhakta, A.K., Koné, A., and Fall, M., 2024, Synergistic effect of copper oxide nanoparticles and diazonium-modified carbon nanotubes for colorimetric detection of dimethoate residues in food products, *Chem. Afr.*, 7 (10), 5317–5328.

[25] Han, E., Zhang, M., Pan, Y., and Cai, J., 2022, Electrochemical self-assembled gold nanoparticle SERS substrate coupled with diazotization for sensitive detection of nitrite, *J. Mater.*, 15 (8), 2809.

[26] Hernandes, T., Dores, E.F.G.C., Ribeiro, M.L., Rossignoli, P.A., and Malm, O., 2014, Simple method to determine residual cypermethrin and deltamethrin in bovine milk, *J. Braz. Chem. Soc.*, 25 (9), 1656–1661.

[27] Elik, A., Durukan, H., Sarac, H., Demirbas, A., and Altunay, N., 2022, Application of levulinic acid-based natural deep eutectic solvents for extraction and determination of deltamethrin in food samples, *Sustainable Chem. Pharm.*, 30, 100861.

[28] Wu, H., Gao, J., Xie, M., Xiang, J., Zuo, Z., Tian, X., Song, R., Yuan, X., Wu, Y., and Ou, D., 2022, Histopathology and transcriptome analysis reveals the gills injury and immunotoxicity in gibel carp following acute deltamethrin exposure, *Ecotoxicol. Environ. Saf.*, 234, 113421.

[29] Longsworth, O.M., Bready, C.J., and Shields, G.C., 2023, The driving effects of common atmospheric molecules for formation of clusters: The case of sulfuric acid, formic acid, hydrochloric acid, ammonia, and dimethylamine, *Environ. Sci.: Atmos.*, 3 (9), 1335–1351.

[30] Leyva, E., and Loredo-Carrillo, S.E., 2024, Synthesis of aromatic azides using different methodologies, *Curr. Org. Chem.*, 28 (9), 655–674.

[31] Ahmad, S.M., Al-Taie, Z.S., Hussein, M., Hammza, R.A., Rahmansyah, M., Bufaroosha, M., and Yousif, E., 2024, An overview of preparation for different azo compounds, *Al-Nahrain J. Sci.*, 27 (1), 1–13.

[32] Liu, D., Li, W., Gao, H., Huang, C., Xu, S., and Lıu, W., 2024, Organic water-soluble fertilizers enhance pesticide degradation: Towards reduced residues, *J. Agric. Sci.*, 30 (1), 179–192.

[33] Grahek, R., Drev, M., Zupančič, B., Hren, J., Ošlaj, M., Bastarda, A., Kocijan, A., and Časar, Z., 2023, Stability and degradation pathways of *N*-nitroso-hydrochlorothiazide and the corresponding aryl diazonium ion, *Org. Process Res. Dev.*, 27 (10), 1792–1811.

[34] Tadesse, B., Asrat, M., Zewge, B., and Tesfaw, A., 2025, Quantitative analysis of pesticides in vegetables: A study on tomatoes and onions from Ethiopian farms, *Meas.: Food*, 19, 100242.

[35] Tulasi, B., Kaithamalai, B., Angappan, S., Gurudevan, T., Padmanaban, G., Chellamuthu, S., Venkidusamy, M., Palanisamy, K., and Chelladurai, A.V., 2024, Standardization of an analytical technique for determination of pesticide residues in fresh and processed button mushroom *Agaricus bisporus* (Lange) Imbach, *Sci. Rep.*, 14 (1), 30747.

[36] Asadi, T., Rahimi Kakavandi, N., Nili Ahmadabadi, A., Heshmati, A., Ranjbar, A., Abdi, K., and Ezoddin, M., 2022, Development of effervescence-assisted switchable polarity solvent homogeneous liquid-phase microextraction for the determination of permethrin and deltamethrin in water samples prior to gas chromatography–flame ionization detection, *Biomed. Chromatogr.*, 36 (5), e5304.

[37] Xu, X., and Murphy, L.A., 2023, Simultaneous determination of pyrethrins, pyrethroids, and piperonyl butoxide in animal feeds by liquid chromatography-tandem mass spectrometry, *Toxins*, 15 (6), 401.

[38] Qassim, B.B., and Mohamed, M.R., 2022, A new indirect and simultaneous determination of deltamethrin insecticide in pure form, industrial and biological samples via a green method of CFIA technique, *Biochem. Cell. Arch.*, 22 (1), 43–51.

[39] Hamzawy, A.H., and Hussein, O.E., 2023, Validation of short run time GC-EI (+) MS/MS method for pesticide residues determination in dry *Hibiscus syriacus* L. via application of d-MRM and end-point backflush quantification techniques, *Int. J. Environ. Anal. Chem.*, 103 (18), 7168–7181.

[40] Jano, A., Fuente-Ballesteros, A., Bernal, J., Ares, A.M., and Valverde, S., 2025, Development and validation of an analytical methodology to determine deltamethrin residues and its metabolites in pine products using GC-QTOF-MS, *ACS Agric. Sci. Technol.*, 5 (4), 656–666.

[41] Al Yahyai, I., Hassanzadeh, J., and Al-Lawati, H.A., 2021, A novel and selective multi-emission chemiluminescence system for the quantification of deltamethrin in food samples, *Sens. Actuators, B*, 327, 128927.

[42] Azimi, M., Manzoori, J.L., Amjadi, M., and Abolhasani, J., 2021, Determination of deltamethrin in water samples using sulfur and nitrogen co-doped carbon quantum dots as a chemiluminescence probe, *J. Anal. Chem.*, 76 (10), 1217–1224.