

## Simple, selective and sensitive spectrophotometric determination of fenitrothion using novel chromogenic reagent

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Received 8 October 2006; received in revised form 23 November 2006; accepted 23 November 2006

Available online 28 November 2006

### Abstract

A facile, rapid and sensitive spectrophotometric method was developed for the determination of fenitrothion in its formulations, water, food grain and agriculture soil samples with newly synthesised reagent. The method was based on the alkaline hydrolysis of fenitrothion pesticide and resultant hydrolysed product of fenitrothion was coupled with diazotised 4,4'-methylene-bis-(*p*-amino-3'-hydroxybenzanilide) in basic medium to give orange coloured product having  $\lambda_{\text{max}}$  480 nm. The formation of coloured derivative with the coupling agent is instantaneous and stable for 48 h. Beer's law was obeyed in the concentration range of 0.1–19.0  $\mu\text{g ml}^{-1}$ . The proposed method is sensitive, easy to operate and permitted the determination of fenitrothion with a detection limits of 0.08  $\mu\text{g ml}^{-1}$ . The experimental results indicate that the procedure can eliminate the fundamental interferences caused by other pesticides and non-target residues which made these methods more sensitive and selective. The method was applicable to the determination of fenitrothion residue in water, food grain and soil samples up to ng level.

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**Keywords:** Fenitrothion; 4,4'-Methylene-bis-(*p*-amino-3'-hydroxybenzanilide); Diazotization method; Spectrophotometry; Water; Food grain and agriculture soil samples

### 1. Introduction

Fenitrothion (*o,o*-dimethyl-*o,p*-nitro-3-methylphenyl phosphorothionate) belongs to an organophosphorus pesticide, extensively applied on crops. Fenitrothion is a contact and stomach poison and it destroys insects, beetles, caterpillars, cockroaches and other pests in all stages. In agriculture it is used for seed, dressing and granular formulations for the control of soil, pests and some foliar pests, particularly effective against rice stem borers and with some effect against red spider mites. The insecticide activity of fenitrothion has been summarized by schmitt [1]. It is slightly toxic to mammals [2] and also wild birds [3]. Burt [4] studied the biophysical aspects of nervous activity in relation to the mode of action of organophosphate, which effect the transmission of nerve impulses from one cell to another. Due to indiscriminate application, it finds a way into surface

water bodies, through agriculture runoff and municipal waste water systems by ingestion and inhalation. Finally, it reaches the human system. Many methods have been developed for the determination of fenitrothion [5–15]. The spectrophotometric methods based on the using diazotization in the presence of oxidising agents and its metabolites by various alkaline hydrolysis of the pesticides have been determined [16–21].

In view of its wide applicability still there is a need for the development of sensitive and reliable methods for the assessment of quality of insecticide formulations and the quantitative residual analysis of fenitrothion. The present paper deal with a simple, sensitive and economical spectrophotometric method for the determination of fenitrothion in its formulations, water and Agriculture soil samples. Here authors successfully synthesised a new reagent, 4,4'-methylene-bis-(*p*-amino-3'-hydroxy benzanilide) for determination of fenitrothion based on the hydrolysed product followed by diazotization. These methods offers better advantages of sensitivity, selectivity and reproducibility than the reported methods [22]. The quantitative and qualitative estimation of these pesticides in their formulations,

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water (spiked), food grains (wheat and rice) and agriculture soil samples were determined.

## 2. Experimental

### 2.1. Synthesis of 4,4'-methylene-bis-(*p*-amino-3'-hydroxybenzanilide)

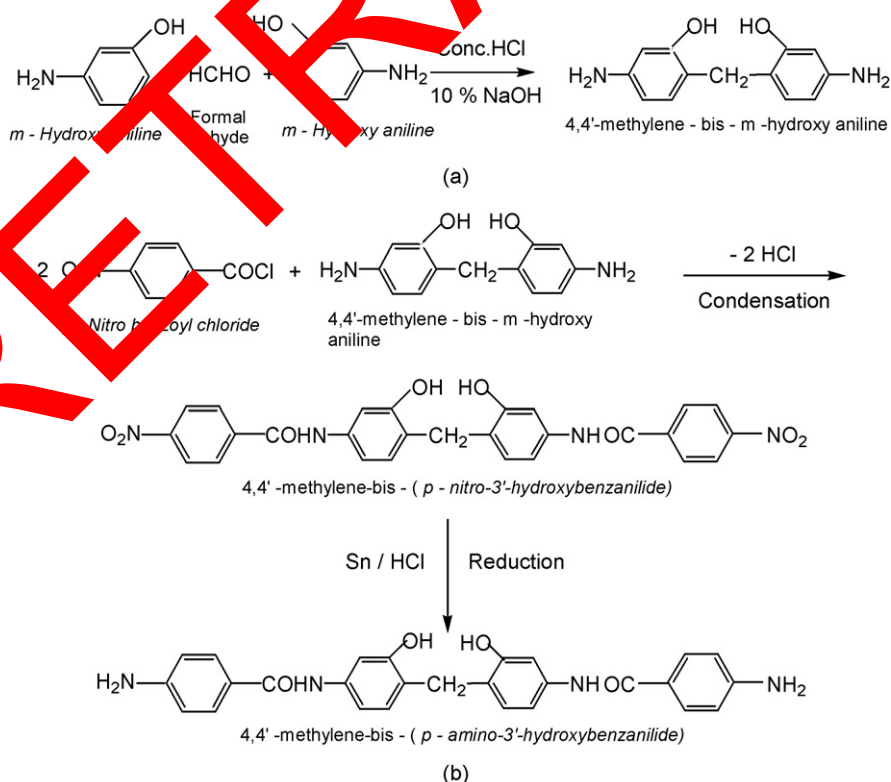
10.91 g of 3-hydroxy aniline (*m*-hydroxy aniline) was dissolved in 125 ml aqueous methanol and 25 ml of 36.5% hydrochloric acid at 50 °C. The reaction mixture was then treated with 35 ml of 3% aqueous formaldehyde solution at 60 °C with stirring for 1 h and neutralised with 10% sodium hydroxide. The solid obtained was filtered, washed with hot water, dried and recrystallized from acetic acid (mp 132–134 °C; yield, 87%) MF: C<sub>13</sub>H<sub>14</sub>N<sub>2</sub>O<sub>2</sub> and elemental analysis of Calculated(found): C-67.82 (67.79), H-6.08 (6.04), N-12.17(12.13), O-13.91(13.87) as shown in Scheme 1a.

2.3 g (0.01 mol) of 4,4'-methylene-bis-*m*-hydroxyaniline was dissolved in 20 ml of DMF in 250 ml round bottomed flask. To this add 3.7 g (0.02 mol) of *p*-nitro benzylchloride. Heat the reaction mixture on water bath maintaining the temperature at 80–90 °C for 2–3 h. Then reaction mixtures was cooled and pour the liquid residue with stirring into cold water by simultaneous addition of 1%(w/v) sodium carbonate solution. The product was filtered off and washed thoroughly. It was then recrystallized from glacial acetic acid in yellowish brown needles. Yield is 86% and melting point is 225–228 °C. MF: C<sub>27</sub>H<sub>20</sub>N<sub>4</sub>O<sub>8</sub> elemental analysis of calculated (found): C-61.36(61.31)%, H-3.78(3.70)%, N-10.60(10.54)%, O-24.24(24.23)% as shown in Scheme 1b.

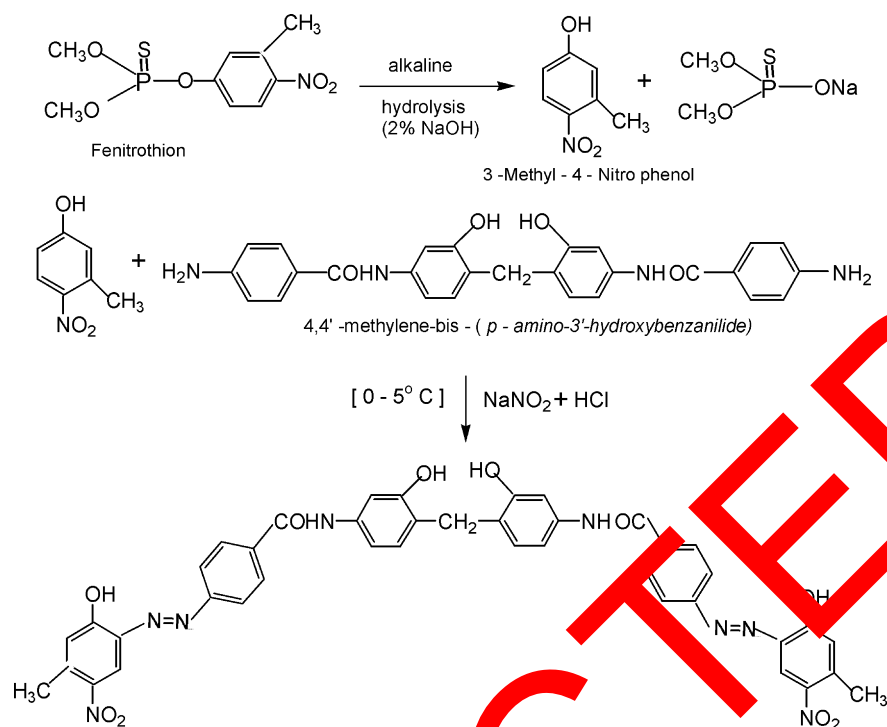
4.49 g (0.0085 mol) of 4,4'-methylene-bis-*m*-hydroxyaniline was taken in a round bottom flask, fitted with a reflux condenser. 11.57 g (0.019 mol) of powdered tin and 25 ml of HCl was introduced to the above. Heat the mixture gently until the reaction commences, and then the heating was stopped. Shake well, and after about 25 min, most of the tin will have reacted and clear solution remains. It was allowed to cool and decant the liquid into beaker and it was washed with 15 ml of water, the washings are added the content of beaker. Concentrated ammonia solution was added to the above, to alkaline to litmus and digest the suspension of precipitated hydrated tin oxide on a steam bath for 20 min. The filtrate was transferred into beaker and heated on water bath with 200 ml of water to ensure extraction of product and refilter. Concentrate the combined filtrate and washings until the volume has been reduced to 175–200 ml filter off any solids, which separates. Acidify the liquid to litmus with glacial acetic acid and evaporate on a water bath until crystals commence to separate, cool in ice, filter the crystals with the pump and dry in the steam oven. The yield of 4,4'-methylene-bis-(*p*-amino-3'-hydroxybenzanilide) is 84%, mp 198–202 °C MF: C<sub>27</sub>H<sub>24</sub>N<sub>4</sub>O<sub>4</sub> elemental analysis of calculated(found): C-69.23(69.21)%, H-5.12(5.09)%, N-11.96(11.94)%, O-13.67(13.63)% as shown in Scheme 2.

### 2.2. Apparatus and reagents

A HITACHI U 2001 spectrophotometric with 1.0 cm matched glass cells were used for all absorbance measurement. An Elico Li-29 model pH metre with combined glass electrode was used for pH measurements.



Scheme 1. (a) Synthesis of 4,4'-methylene-bis-*m*-hydroxy aniline. (b) Synthesis of 4,4'-methylene-bis-(*p*-amino-3'-hydroxybenzanilide).



Scheme 2. Reaction mechanism of the 4,4'-methylene-bis-(p-amino-3'-hydroxybenzanilide) with fenitrothion.

All the chemicals and reagents used were of analytical reagent grade and double-distilled water was used throughout experiments. The reagents like sodium hydroxide, sodium nitrate, potassium carbonate, anhydrous sodium phosphate and HCl were purchased from S.D. fine chemicals, Mumbai, India. 3-hydroxy aniline, acetone, formaldehyde, p-nitrobenzyl chloride, methanol, chloroform and tin were purchased from Merck Chemicals, Mumbai, India. The technical grade samples of fenitrothion pesticide in the form of wettable powder and 75% EC were obtained from Bayer India Pvt. Ltd., Mumbai, India. The reagents like sodium hydroxide (2%), sodium nitrate (0.3%), 1N HCl were prepared. The solvents like methanol, chloroform, acetone were purified and employed for the present investigation.

#### 2.2.1. Stock fenitrothion solution ( $1 \text{ mg ml}^{-1}$ )

A stock solution ( $1 \text{ mg ml}^{-1}$ ) of fenitrothion (Rallis India Limited, Bangalore, India) was dissolved in methanol and working standard solution ( $1 \text{ mg ml}^{-1}$ ) was prepared by appropriate dilution with distilled water.

#### 2.2.2. 4,4'-Methylene-bis-(p-amino-3'-hydroxybenzanilide) (2%)

4,4'-methylene-bis-(p-amino-3'-hydroxybenzanilide) (2%) was prepared by dissolving 2 g of 4,4'-methylene-bis-(p-amino-3'-hydroxybenzanilide) was dissolved in 20 ml of 5 M HCl and then made up to 100 ml with distilled water.

#### 2.2.3. Potassium carbonate solution (0.1 M)

Potassium carbonate solution (0.1 M) was prepared by dissolving 1.3821 g of Potassium carbonate in distilled water and diluted to 100 ml.

#### 2.2.4. Sodium hydroxide solution (2%)

Sodium hydroxide solution (2%) was prepared by dissolving 2 g of sodium hydroxide in distilled water and diluted to 100 ml.

#### 2.2.5. Sodium nitrate solution (0.3%)

Sodium nitrate solution (0.3%) was prepared by dissolving 0.3 g of sodium nitrate in distilled water and diluted to 100 ml.

#### 2.2.6. 1N HCl solution

1N HCl solution was prepared by diluting concentrated HCl with distilled water.

#### 2.2.7. Buffer (pH 7.8–10.5)

An amount of 3.4 ml concentrated sulphuric acid is added to 250 ml distilled water in a 500 ml flask. Twenty-five grams of mono potassium dihydrogen phosphate is added to this, shaken until dissolution is complete and diluted to 500 ml for pH 7.8–10.5.

#### 2.3. Procedure for the determination of fenitrothion residues

The coloured derivatives of fenitrothion were prepared by azo coupling reaction, 20 ml of fenitrothion standard solution was taken in a clean dry 100 ml beaker, 5 ml of 2% NaOH solution was added and kept for 20 min for complete hydrolysis to yield 3-methyl-4-nitrophenol. Diazonium salt 4,4'-methylene-bis-(p-amino-3'-hydroxybenzanilide) was prepared using 4 ml of 0.3%  $\text{NaNO}_2$  and 4 ml of 1N HCl for coupling reaction at 0–5 °C. The reaction mechanism of the colour compounds was shown in Scheme 2. The pH of the

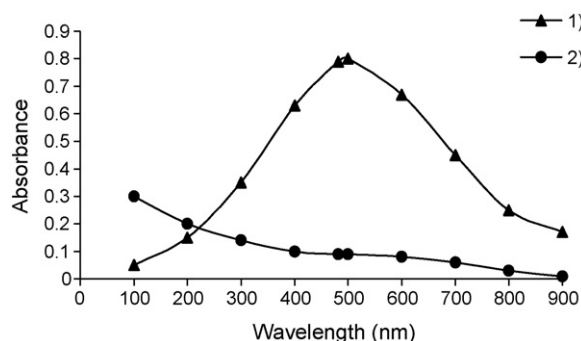


Fig. 1. Absorption spectra of the fenitrothion. (1) Fenitrothion + 4,4'-methylene-bis-(*p*-amino-3'-hydroxybenzanilide), (2) reagent blank.

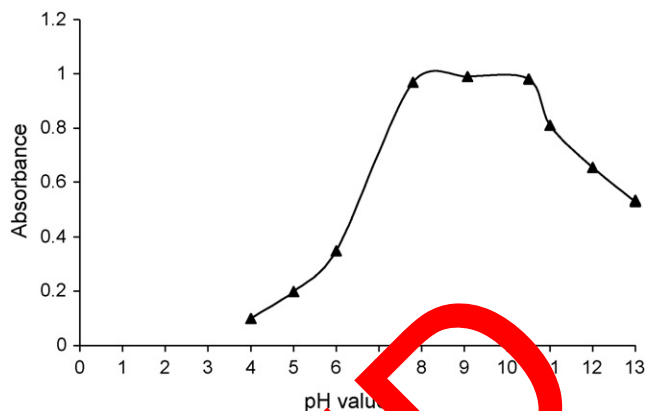


Fig. 2. Effect of pH on diazotization method for determination of fenitrothion.

Table 1  
Optical characteristics, precision and accuracy of the method with coupling reagent 4,4'-methylene-bis-(*p*-amino-3'-hydroxybenzanilide)

Optical characteristics	Diazotization method
Concentration range ( $\mu\text{g ml}^{-1}$ )	0.1–19.0
$\lambda_{\text{max}}$ (nm)	480
Color	Orange
Limit of Detection ( $\mu\text{g ml}^{-1}$ )	0.085
Limit of Quantification ( $\mu\text{g ml}^{-1}$ )	3.027
Stability of the color (h)	48 h
Molar absorptivity ( $1 \text{ mol}^{-1} \text{ cm}^{-1}$ )	$1.959 \times 10^4$
Sandell's sensitivity ( $\mu\text{g cm}^{-2}$ )	0.0197
Regression equation ( $Y = bx + a$ )	
Slope ( $b$ )	0.0219
Intercept ( $a$ )	0.0112
Standard error (%)	0.0010
Standard deviation (S.D.) <sup>a</sup>	0.267
Correlation coefficient	0.999
Relative error (%)	1.0

<sup>a</sup> Calculation for five samples containing same amount of fenitrothion, where  $x$  is the concentration in  $\mu\text{g ml}^{-1}$ .

reaction was maintained between 7.8 and 10.5. The spectra for the above reaction were recorded in the UV–vis region as shown in Fig. 1 and optical, precision and accuracy data was shown in Table 1. Beer's law was obeyed over the range from 0.1 to 19.0  $\mu\text{g ml}^{-1}$ . This data was used for the analysis of water, food grain and agriculture soil samples (Fig. 2).

Table 3  
Recovery of fenitrothion residue from spiked water samples

Sample number	Diazotisation method		Recovery $\pm$ S.D. <sup>a</sup> (%)	$F$ -test	$t$ -Test	Reported method [22] recovery $\pm$ S.D. <sup>a</sup> (%)
	Taken ( $\mu\text{g ml}^{-1}$ )	Found ( $\mu\text{g ml}^{-1}$ )				
1	25.70	25.48	99.14 $\pm$ 0.08	0.95	0.24	99.10 $\pm$ 0.09
2	50.50	50.40	99.80 $\pm$ 0.09	1.00	0.25	97.60 $\pm$ 0.08
3	75.60	75.36	99.68 $\pm$ 0.08	0.88	0.45	99.50 $\pm$ 0.09
4	100.30	100.10	99.80 $\pm$ 0.07	0.72	0.18	99.70 $\pm$ 0.06
5	125.40	125.20	99.84 $\pm$ 0.09	0.54	0.28	99.67 $\pm$ 0.05
6	150.50	150.30	99.86 $\pm$ 0.06	0.53	0.11	99.80 $\pm$ 0.08

<sup>a</sup>  $n = 5$ .

Table 2  
Determination of fenitrothion in various pesticidal formulation using coupling agent 4,4'-methylene-bis-(*p*-amino-3'-hydroxybenzanilide)

Formulation	4,4'-methylene-bis-( <i>p</i> -amino-3'-hydroxy benzanilide)
5% dust	4.92 $\pm$ 0.04
25% wettable powder	24.89 $\pm$ 0.03
75% EC	74.91 $\pm$ 0.01

#### 2.4. Procedure for the determination of fenitrothion in its formulations

Fenitrothion in 50% wettable powder, 75% EC and 98.7% technical grade were analysed using the aforesaid procedure by coupling with diazotized compound of 4,4'-methylene-bis-(*p*-amino-3'-hydroxybenzanilide). The method was compared with already reported methods in the literature [22]. Percentage recovery of the fenitrothion in the above formulations was shown in Table 2.

#### 2.5. Procedure for the determination of fenitrothion in water samples

The water samples were spiked with concentrations in the ranges from 30.70 to 150. Fifty micrograms per milliliter in methanol in two methods under study which are given in the Table 3. The spiked water samples were extracted with chloroform. The combined extracts were washed with 0.1 M potassium carbonate solution to break any emulsion formed

during the extraction and dried over anhydrous sodium sulphate. Finally, chloroform was evaporated to dryness on a steam bath and the residue was dissolved in methanol and the amount was determined using the procedures described earlier.

### 2.6. Procedure for the determination of fenitrothion in grain samples (wheat and rice)

The grain samples (rice and wheat) of 50 g each were taken in warming blender and blended for 5 min with 100 ml of chloroform. The samples were spiked with different concentrations of insecticide ranges from 45.94 to 304.20  $\mu\text{g ml}^{-1}$  in methanol and blended for 3 min chloroform was filtered and the residue was retained. The residue was washed twice with 10 ml of chloroform and blended for 3 min. The chloroform extracts were evaporated on a steam bath and the residue was dissolved in methanol and the amount was determined by the procedure outlined earlier. The results are summarised in Table 4.

### 2.7. Procedure for the determination of fenitrothion in agriculture wastewater and soil samples

Agriculture wastewater samples (10 ml) were collected from S.V. Agricultural Research Station, Tirupati, India and the water samples were extracted with chloroform (2 ml  $\times$  10 ml). The chloroform extracts was then evaporated to dryness under reduced pressure and the residue was dissolved in 25 ml of 50% methanol and amount of the fenitrothion was determined by the described procedure earlier. Ten grams of agricultural soil samples was extracted according to the reported method [22] and the amount of the fenitrothion was analysed by the aforesaid procedure.

## 3. Results and discussions

Table 1. Shows that the formation of coloured products to the coupling reaction of the pesticide sample with the reagents under study are instantaneous and stable for a reasonable period of time indicates its advantage over reported methods [22]. Beer's law was obeyed in the concentration range from 0.1 to 19  $\mu\text{g ml}^{-1}$  indicating low Sandell's sensitive values. The results obtained for fenitrothion (Table 1) was reproducible with low relative standard deviations ranges from 0.257. Limit of quantification (LOQ) is determined by taking the ratio of standard deviation ( $\sigma$ ) of the blank with respect to water and the slope of calibration curve ( $s$ ) multiplied by a factor of 3. This means that LOQ is approximately three times limit of detection (LOD). Naturally, the LOQ slightly crosses the lower limit of Beer's law range. However, LOD is well below the lower limit of Beer's law range. The upper limit of the Beer-Lambert's range is determined by a plot of absorbance against concentration at the value of  $\lambda_{\text{max}}$ . Beyond this limit the correlation results were strongly affected. The correlation coefficient values obtained for this reaction was very close to unity suggesting that the absorbance depends upon the concentration of the fenitrothion. The values

Table 4  
Recovery of fenitrothion residue from grain samples (rice and wheat)

Sample number	Diazotisation method						Reported method [22] recovery (%)			
	Rice			Wheat			Rice	Wheat		
	Taken ( $\mu\text{g ml}^{-1}$ )	Found ( $\mu\text{g ml}^{-1}$ )	Recovery $\pm$ S.D. <sup>a</sup> (%)	F-test	t-Test	Taken ( $\mu\text{g ml}^{-1}$ )	Found ( $\mu\text{g ml}^{-1}$ )	Recovery $\pm$ S.D. <sup>a</sup> (%)	F-test	t-Test
1.	50.10	50.00	99.80 $\pm$ 0.182	1.000	3.516	48.95	48.86	99.80 $\pm$ 0.05	0.001	2.49
2.	100.20	100.12	99.92 $\pm$ 0.182	0.583	1.170	88.12	86.94	98.79 $\pm$ 0.560	0.013	2.74
3.	150.30	150.15	99.90 $\pm$ 0.150	0.754	0.322	137.42	136.84	99.60 $\pm$ 0.182	0.284	2.474
4.	200.40	200.28	99.70 $\pm$ 0.182	0.284	0.504	184.81	184.17	99.67 $\pm$ 0.070	0.677	0.290
5.	250.50	250.40	98.50 $\pm$ 0.567	0.094	0.952	302.14	300.30	99.40 $\pm$ 0.182	0.001	0.394
6.	300.60	302.04	99.30 $\pm$ 0.182	1.000	0.216	303.14	301.30	99.40 $\pm$ 0.091	1.000	0.241

<sup>a</sup> n = 5.



Table 5  
Comparison of fenitrothion in its formulations and environmental samples

Samples	Fenitrothion added ( $\mu\text{g ml}^{-1}$ )	Proposed method				Reported method [22] recovery $\pm$ S.D <sup>a</sup> (%)
		Found ( $\mu\text{g ml}^{-1}$ )	Recovery $\pm$ S.D <sup>a</sup> (%)	F-test	t-Test	
<b>Formulation</b>						
5% dust	–	4.91	98.90 $\pm$ 0.14	0.286	4.29	94.50 $\pm$ 0.08
25% wettable powder	–	24.25	99.28 $\pm$ 0.13	0.321	4.71	95.50 $\pm$ 0.08
75% EC	–	74.21	98.77 $\pm$ 0.08	0.929	4.22	97.62 $\pm$ 0.07
<b>Water samples<sup>b</sup></b>						
Spiked water	9.0	8.86	98.77 $\pm$ 0.08	0.929	4.22	97.62 $\pm$ 0.07
Natural water	9.0	8.92	99.44 $\pm$ 0.11	0.754	0.32	97.62 $\pm$ 0.182
<b>Food grains</b>						
Rice	15.0	14.81	98.00 $\pm$ 0.14	0.013	2.21	97.70 $\pm$ 0.182
Wheat	15.0	14.75	98.73 $\pm$ 0.09	0.094	0.952	98.50 $\pm$ 0.567
<b>Agricultural soil samples<sup>c</sup></b>						
Sample 1	25.0	24.38	98.40 $\pm$ 0.09	0.002	0.522	98.73 $\pm$ 0.13
Sample 2	25.0	24.31	98.10 $\pm$ 0.07	0.002	0.950	97.70 $\pm$ 0.182

Recovery in%, amounts of insecticide in  $\mu\text{g}$ .

<sup>a</sup> Average values for  $n = 6$ .

<sup>b</sup> Agricultural samples collected from S.V. Agricultural Research Institute, Tirupati, AP, India.

<sup>c</sup> Agricultural samples collected from mallam, Chittamuru Mandal, Nellore District, AP, India.

obtained for the relative standard deviation and percentage error suggest that these new procedures offer a good precision and accuracy.

The data included in Table 2 shows that the active ingredient present in the formulations of fenitrothion can be successfully determined spectrophotometrically using the new reagent. The results of these recoveries reveal that the amounts close to the manufacturer's specifications and these are favorable compared with the method reported in the literature [22] was shown in Table 5. These observations suggested that the other ingredients present in these formulations do not interfere hence, the methods can be adopted as an additional method for a routine control of the purity of the commercial insecticide formulations. The data presented in Tables 3 and 4 suggested that the percentage of pesticide recovery from fortified water and grain samples range from 97.70 to 99.9%. The Tables 3 and 4 indicates that the percentage recovery of the fenitrothion from water and grain samples is as follows: water samples > wheat samples; wheat samples > rice samples; water samples > rice samples.

These methods reveal that the percentage recovery of the fenitrothion in food grains was observed as rice > wheat.

It is evident from the results that the proposed methods are simple, convenient, rapid and sensitive. More over, these methods do not involve elaborate cleanup procedures as is required by the other methods. Hence, our proposed methods describe here would serve as additional techniques for the estimation of the fenitrothion in spiked water, food grain and agriculture soil samples.

### 3.1. Analytical application

The proposed methods were applied to the determination of fenitrothion in spiked water, food grain (rice and wheat) and agriculture soil samples. The results obtained were in good agreement with the reported method [22] was given in Table 5.

## 4. Conclusion

The present methods describes simple analytical procedure for the determination of fenitrothion in its formulations, fortified water and food grains. The comparison of the proposed method with the other methods or the determination of fenitrothion by spectrophotometry was given in Table 5. The preparation of the coupling reagent 4,4'-methylene-bis-(*p*-amino-3'-hydroxybenzanilide) is simple and convenient to synthesis in any ordinary laboratory. The color derivatives of the fenitrothion are more stable than the other reported methods [22].

The proposed method has the following distinct advantages:

- Simplicity in synthesis of color forming reagent at ordinary laboratories for determination of fenitrothion.
- The results of the fenitrothion recovery from spiked water and food grain samples with the reagents have good agreement results than the reported methods in the literature [22].
- The proposed methods can be applied routine control for the determination of fenitrothion in its commercial formulations and environmental samples.
- All the non-target species do not interfere with the present method.
- The statistical analysis of the results indicate that the present method have good precision, accuracy and the present method do not involve elaborated clean up procedure as required by the other methods.

## Acknowledgements

The authors are grateful to Bayer (India) Ltd., Bombay, for the supply of technical grade fenitrothion and the Head of the Department, Department of Biotechnology, S.V. University, Tirupati for providing instrumental facility to carry out this work.

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