CHAPTER 2

OPTIMIZATION OF FLOW INJECTION SPECTROPHOTOMETRY FOR METHYL PARATHION DETERMINATION

2.1 Introduction

The colorimetric methods available for methyl parathion are time consuming and involve the use of drastic chemical procedures such as the reduction of the nitro group to an amine, its diazotization and coupling to a chromogenic agent (33,42,43,47), or its hydrolysis with hot alkali to liberate 4-nitrophenol(58) which is directly determined spectrophotometrically. These methods are not suitable when it is necessary to analyze a large number of samples.

In this research, a simple, rapid and milder reaction that allows the determination of methyl parathion was proposed. The method is base on the combination of flow injection with spectrophotometric detection by using alkaline hydroxylamine with ethanol for the decomposition of methyl parathion to 4-nitrophenol which is also determined spectrophotometrically at 410 nm.

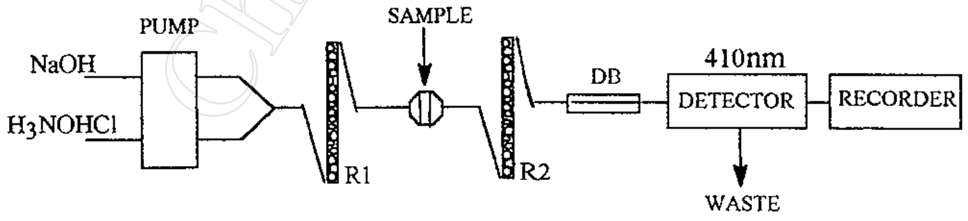

Figure 2.1 Schematic diagram of the flow system used for methyl parathion determination; R1=first reactor, R2=second reactor, DB=debubler with PTFE pipe thread tape as a gas diffusion membrane.

A flow injection system was employed for the determination of methyl parathion, and its manifold was designed and constructed as shown in Figure 2.1. The experimental set-up is shown in Figure 2.2.

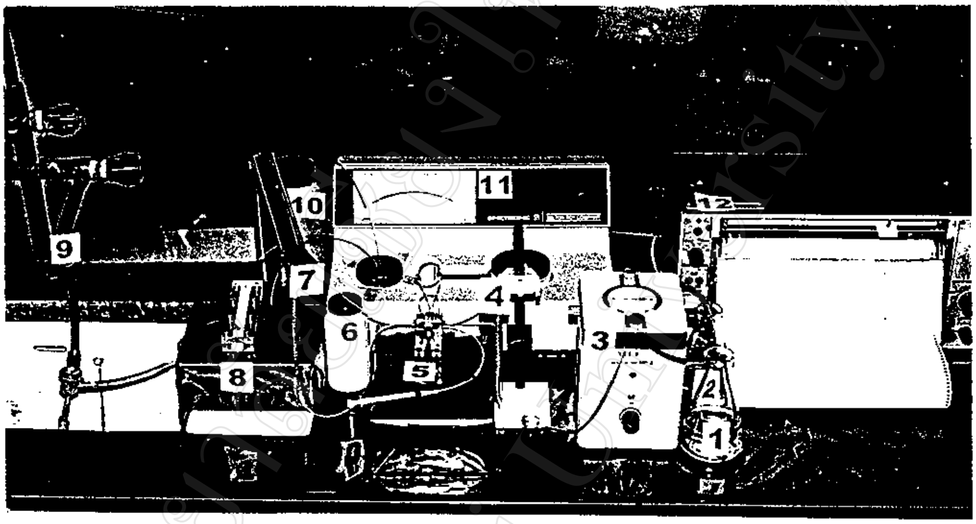

Figure 2.2 FIA system for methyl parathion determination; 1,2=reagent reservoir, 3=peristaltic pump, 4=reactor R1, 5=injection valve, 6=waste, 7=reactor R2, 8=deblubbler, 9=suction, 10=waste, 11=detector, 12= recorder.

2.2 Experimental

2.2.1 Apparatus and Instruments

1) UV-VIS Spectrophotometer, model U-2000 Spectrophotometer (Hitachi, Japan).
2) Cecil 1000 Series (Cecil Instruments, England).
3) Spectronic 21 (Milton Roy Company, USA).
4) 1 Pen recorder, Model 135A (National, Japan).
5) Peristaltic pump, Eyela SMP-23S (Tokyo Rikakikai, Japan).
6) Home-made injection valve and membrane separator(59,60).
7) Flow-through cell for spectrophotometer (Hellma, Germany).

8) Plastic tube (I.D. 0.0173-0.050 in).
9) Teflon tube (I.D. 0.050 in).
10) Test tube (O.D. 0.30-1.80 cm).
11) Disposable syringe, 1 ml (Nissho Nipro Corporation Ltd., Thailand).
12) Water bath (Sunvic, U.K.)
13) PTFE Pipe thread tape (Thai pipe, Thailand).

2.2.2 Chemicals

1) Methyl parathion, AR grade (Dr. Ehrenstorfer, Germany).
2) Methyl parathion, 50%w/v E.C. (Bayer Thai Ltd., Thailand).
3) Sodium hydroxide pellets (EKA Nobel, Sweden).
4) Hydroxylamine hydrochloride, AR grade (Ajax Chemicals, Australia).
5) Ethanol, absolute (E. Merck, Germany).

2.2.3 Preparation of Standard Solutions and Reagents

Deionized distilled water was used throughout the whole experiment.

1) Methyl parathion standard solution, AR grade (100 ppm).

   Stock solution was prepared by dissolving 0.01g of methyl parathion in 50% ethanol and diluted to 100 ml, this stock solution was stored in a refrigerator and then used to study in the analytical characteristics.

2) Methyl parathion standard solution, 50%E.C. (2500 ppm).

   0.5 ml of 50%E.C. methyl parathion was pipetted into a 100-ml volumetric flask and diluted to volume with 50% ethanol. This solution was stored in a refrigerator and then used to study in the optimized procedures.

3) Sodium hydroxide solution (3.5 M).

140g of pure sodium hydroxide pellets were dissolved approximately in water and diluted to 1 l. This solution was then standardized with standard sulphamic acid using methyl red as indicator(61).

4) Hydroxylamine hydrochloride solution (2.0 M).

139g of the pure substance was dissolved in water and diluted to 1 l. This solution was filtered and stored in a refrigerator.

5) Alkaline hydroxylamine solution.

Alkaline hydroxylamine solution was prepared freshly by mixing equal volumes of 2.0 M hydroxylamine hydrochloride solution and 3.5 M sodium hydroxide solution. These solutions were stored in a refrigerator in order to avoid generation of heat on mixing. The mixture is stable for not more than 1 hour.

2.3 Preliminary Studies of Absorption Spectrum of Methyl Parathion Using Alkaline Hydroxylamine.

2.3.1 Absorption Spectrum by Conventional method

1-ml aliquot of the methyl parathion standard solution was pipetted into a dry test tube and 4-ml of alkaline hydroxylamine solution was added into this test tube. The test tube was shaked gently and stood for at least 5-min. The absorbance of liberated 4-nitrophenol was scanned between 190 and 550 nm. The absorption spectrum of 4-nitrophenol solution is shown in Figure 2.3.

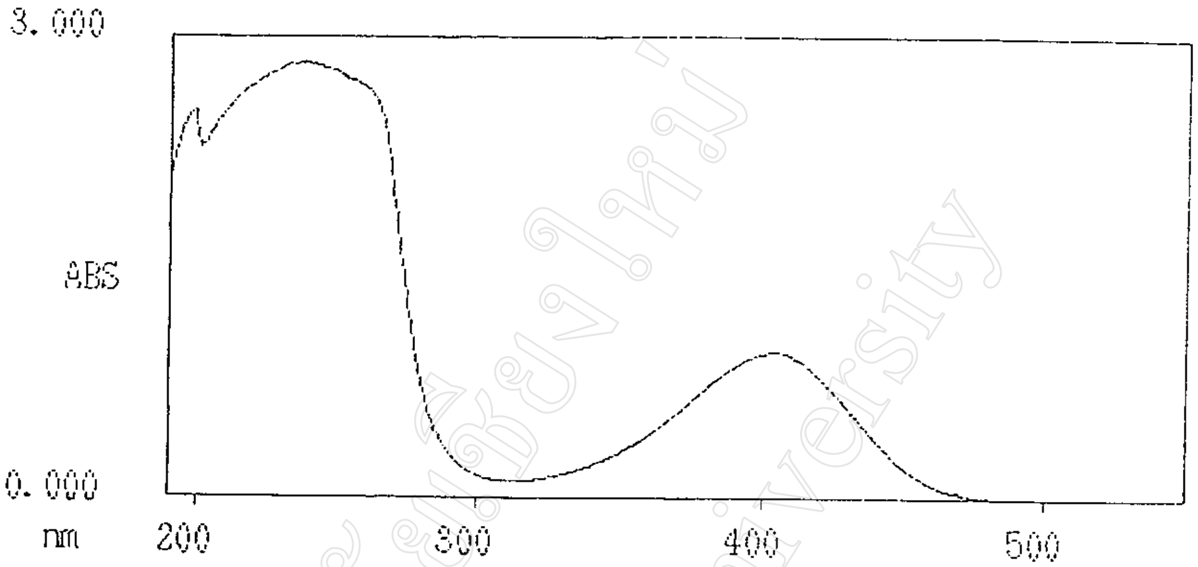

Figure 2.3 Absorption spectrum of 4-nitrophenol against reagent blank solution.

The spectrum shows maximum absorption at 403 nm and the reagent blank solution does not interfere in this region.

2.3.2 Absorption Spectrum by FIA-Spectrophotometric Method.

A comparative study of the absorption spectrum of methyl parathion standard solution based on the same chemical reaction as section 2.3.1 was also carried out by FIA with spectrophotometric detection using the experimental conditions (fixing by random) as shown in Table 2.1. The FIA manifold was shown in Figure 2.1. 300 ml of methyl parathion standard solution was injected into a reagent stream containing sodium hydroxide and hydroxylamine solution. The FIA signals were recorded at various wavelength (380-425nm). The absorption spectrum of the pesticide was obtained by plotting peak heights (mV) against various wavelengths (nm) results are shown in Table 2.2 and Figure 2.4.

Table 2.1 Experimental condition for methyl parathion determination

Experimental condition	Information
Sodium hydroxide concentration	1.5 M
Hydroxylamine concentration	0.2 M
Methyl parathion standard concentration	4 ppm
Injection volume	300 µl
Pump flow rate	3.0 ml/min
Inner diameter of mixing tubing, R1 and R2	0.02 and 0.02 in
Length of mixing tubing, R1 and R2	30 and 100 cm
Shape of mixing part	coil
Absorbance range	0-2
Chart speed	6 min/cm
Sensitivity	40 mV/cm

Table 2.2 Peak height at various wavelengths.

Wavelength (nm)	Peak height*	
	cm	mV
380	2.00	80
385	2.15	86
390	2.25	90
395	2.35	94
400	2.45	98
405	2.55	102
410	2.57	103
415	2.50	100
420	2.35	94
425	2.20	88

*average of triplicate results.

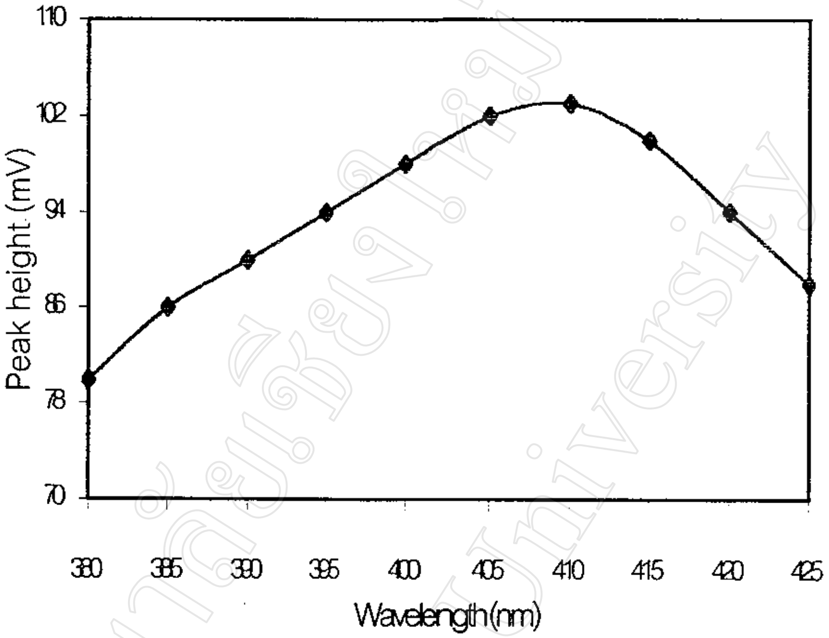

Figure 2.4 Peak height at various wavelengths.

2.4 Optimization of the Flow Injection (FI) System

As depicted in Figure 2.2, 300 μl of methyl parathion standard solution was injected into a reagent stream with the flow rate of 3.0 ml/min. The random analytical parameters were shown in Table 2.1. The reagent concentrations, the flow rate, length and type of mixing reactor, and sample volume should be optimized to achieve the suitable conditions.

2.4.1 Wavelength

The optimum wavelength for methyl parathion determination was studied over the range of 380 to 425 nm as described in section 2.3.2.

As depicted in Figure 2.4, a maximum peak height was observed at 410 nm. Thus this wavelength was chosen for the further FIA measurement.

2.4.2 Effect of Sodium Hydroxide (NaOH) Concentrations

To obtain the sensitivity of this method, five different concentrations of NaOH solution were studied. The standard solutions of methyl parathion were injected into the FI system under the experimental conditions as shown in Table 2.1. The results obtained are shown in Table 2.3 and Figure 2.5.

Table 2.3 Effect of NaOH concentrations on peak height

Methyl Parathion (ppm)	Peak height* (mV) at NaOH concentration (M)				
	1.5	2.0	2.5	3.0	3.5
0	84.0	64.0	70.8	76.0	76.0
2	91.2	75.6	88.8	90.4	92.0
4	96.8	87.2	102.8	106.0	108.0
6	108.0	96.8	115.2	120.0	120.0
Slope (mV/ppm)	3.88	5.50	7.36	7.38	7.40
Correlation coefficient	0.9892	0.9990	0.9963	0.9998	0.9978

*average of triplicate results.

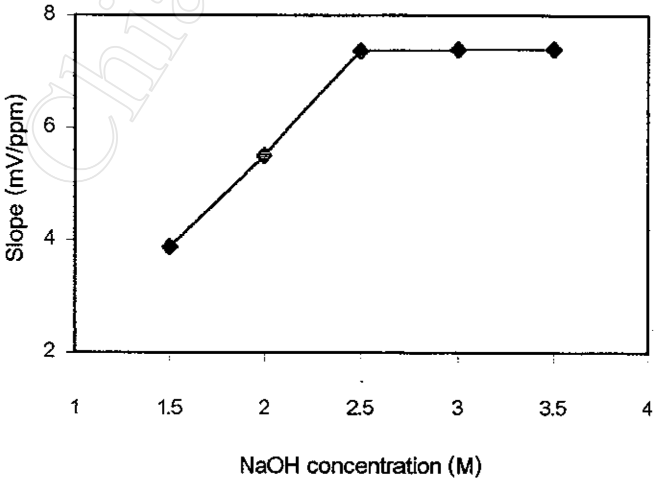

Figure 2.5 Relationship between slope (mV/ppm) and various NaOH concentrations (M).

It was seen that high sensitivity was obtained when NaOH concentration was increased, but the appropriate concentration is 2.5 M.

2.4.3 Effect of Hydroxylamine (H_3NOHCl) Concentrations

The effect of various hydroxylamine concentrations on peak height was studied by varying the concentration of hydroxylamine solutions in the range of 0.6-2.2 M. The results obtained are shown in Table 2.4 and Figure 2.6.

Table 2.4 Effect of hydroxylamine concentrations on peak height.

Methyl Parathion (ppm)	Peak height* (mV) at H_3NOHCl concentration (M)				
	0.6	1.0	1.4	1.8	2.2
0	104.0	104.0	94.0	80.0	74.0
2	120.0	123.2	114.0	94.8	82.8
4	136.0	144.0	132.8	115.2	92.0
6	154.8	160.0	152.0	132.0	102.0
Slope (mV/ppm)	8.42	9.44	9.64	8.82	4.66
Correlation coefficient	0.9992	0.9987	0.9999	0.9983	0.9996

*average of triplicate results.

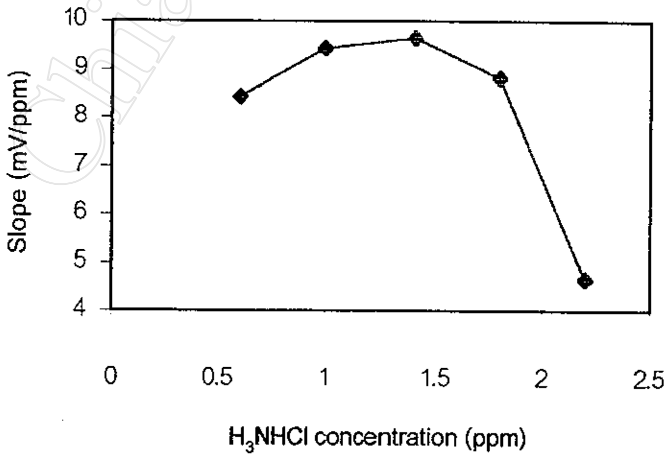

Figure 2.6 Relationship between slope (mV/ppm) and various H_3NOHCl concentrations (M).

The results indicate that the maximum sensitivity is obtained when the concentration of hydroxylamine solution is 1.4 M.

2.4.4 Effect of Temperature

The optimum temperature was studied in the range of 22.0-71.0°C, using sodium hydroxide and hydroxylamine concentrations as 2.5 and 1.4 M, respectively. The results obtained are summarized in Table 2.5 and Figure 2.7.

Table 2.5 Effect of temperature on peak height.

Methyl Parathion (ppm)	Peak height* (mV) at temperature (°C)				
	22.0	31.0	40.5	50.5	71.0
0	104.8	88.8	78.0	60.8	72.8
2	120.0	108.0	101.2	88.8	106.8
4	140.0	126.8	120.0	119.2	137.2
6	158.8	146.8	148.0	155.2	169.6
Slope (mV/ppm)	9.10	9.64	11.44	15.68	16.04
Correlation coefficient	0.9985	0.9999	0.9971	0.9983	0.9998

*average of triplicate results.

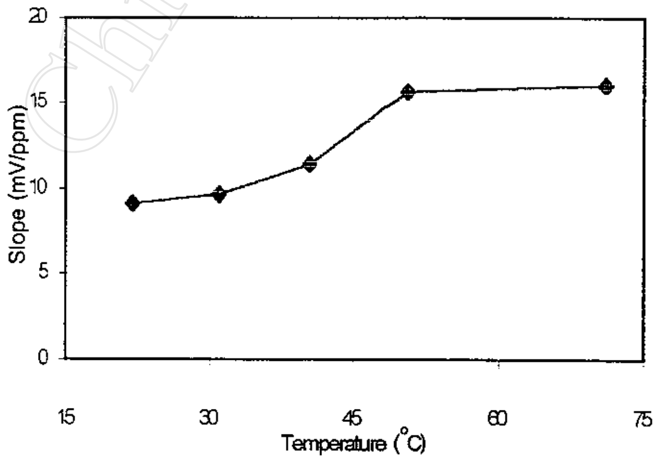

Figure 2.7 Relationship between slope (mV/ppm) and temperature (°C).

The results show that the maximum sensitivity can be attained at the higher temperature. Unfortunately, the bubbles frequently occurred when the temperature was higher. Thus the FI system should be operated at room temperature.

2.4.5 Effect of Flow Rates

The effect of flow rate was investigated by injecting a fixed volume of methyl parathion into the FI system at various flow rates as depicted in Table 2.6 and Figure 2.8.

Table 2.6 Effect of pump flow rates on peak height.

Methyl Parathion (ppm)	Peak height* (mV) at pump flow rate (ml/min)				
	2.0	3.0	4.0	5.0	6.0
0	112.0	112.0	64.0	97.2	108.0
2	124.0	136.0	85.2	117.6	126.0
4	152.0	162.0	110.0	134.0	143.2
6	186.0	187.2	131.2	150.0	156.0
Slope (mV/ppm)	12.50	12.58	11.32	8.74	8.06
Correlation coefficient	0.9804	0.9998	0.9995	0.9982	0.9972
t_{base} (sec)	72.0	43.2	24.0	21.6	19.2
Sample/hr	50	83	150	166	187

*average of triplicate results.

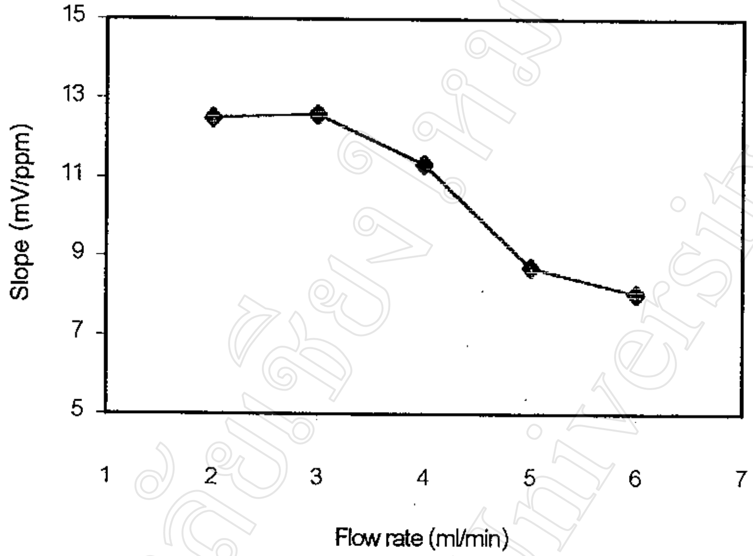

Figure 2.8 Relationship between slope (mV/ppm) and pump flow rate (ml/min).

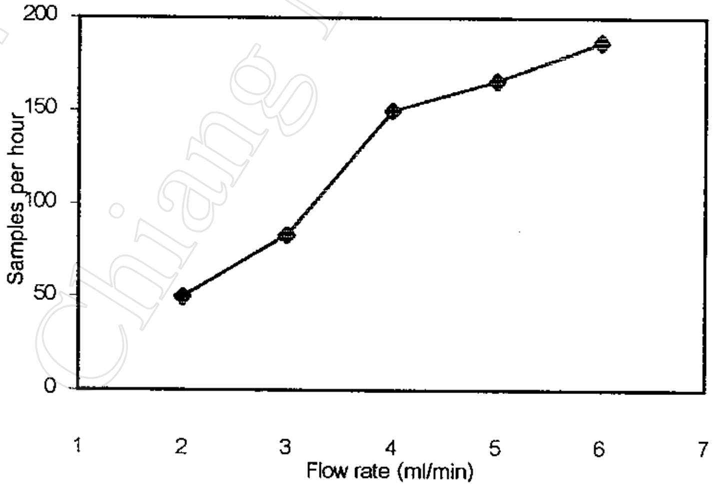

Figure 2.9 Relationship between sampling rate (samples/hr) and pump flow rate.

A flow rate of 3.0 ml/min was chosen in order to obtain a relatively high sensitivity and a reasonable of sample throughput.

2.4.6 Effect of Inner Diameter of Mixing Tubing R1.

The effect of inner diameter of the mixing tubing R1 was studied in the range of 0.0173-0.050 inches. Table 2.7 and Figure 2.10 show that the optimum inner diameter of mixing tubing R1 is 0.04 inches.

Table 2.7 Effect of the inner diameter of mixing tubing on peak height.

Methyl Parathion (ppm)	Peak height* (mV) at the I.D. of mixing tubing (in)				
	0.0173	0.02	0.0299	0.04	0.05
0	54.8	60.8	52.0	50.8	77.6
2	77.2	84.0	92.0	75.2	80.0
4	84.8	102.8	112.0	101.6	106.0
6	125.2	126.4	132.0	127.6	102.0
Slope (mV/ppm)	10.94	10.78	13.00	12.84	7.66
Correlation coefficient	0.9617	0.9991	0.9827	0.9998	0.9611

*average of triplicate results.

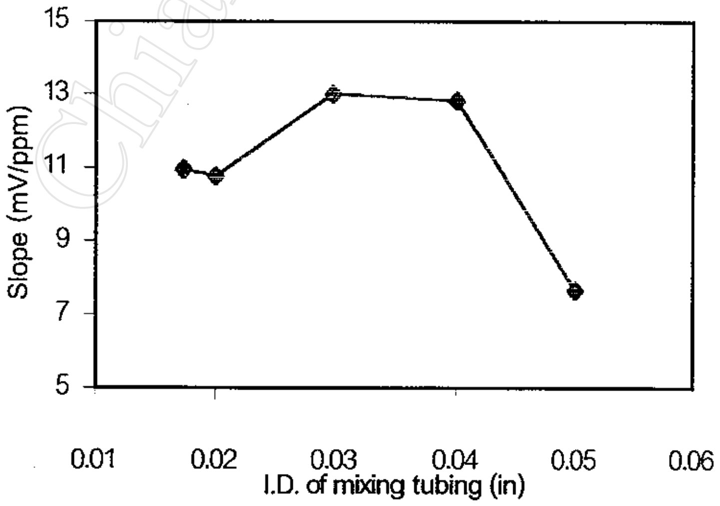

Figure 2.10 Relationship between slope (mV/ppm) and I.D. of mixing tubing (in).

2.4.7 Effect of the Length of Mixing Tubing R1

The effect of the mixing tubing length was studied by varying the length from 15.00 to 75.00 cm. The results in Table 2.8 and Figure 2.11 show that the optimum length of mixing tubing is 60.00 cm.

Table 2.8 Effect of the length of mixing tubing on peak height.

Methyl Parathion (ppm)	Peak height* (mV) at the length of mixing tubing (cm)			
	15.00	45.00	60.00	75.00
0	48.0	48.0	44.0	44.0
2	70.0	64.0	70.0	64.0
4	95.2	93.6	87.2	83.2
6	116.0	119.2	120.0	116.0
Slope (mV/ppm)	11.46	12.16	12.26	11.76
Correlation coefficient	0.9994	0.9935	0.9932	0.9908

*average of triplicate results.

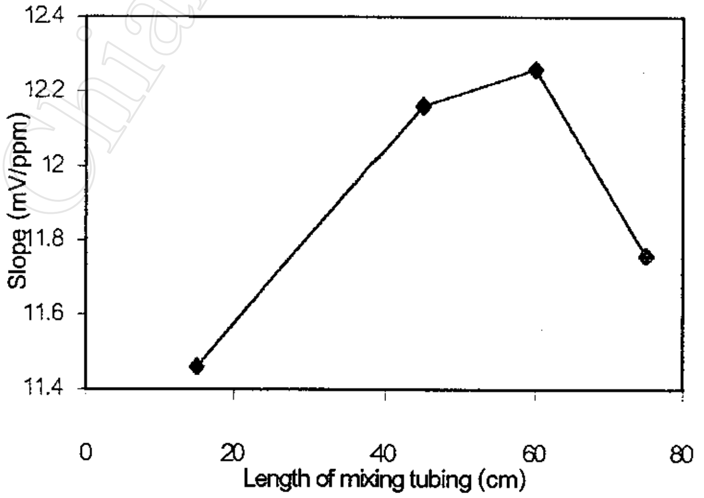

Figure 2.11 Relationship between slope (mV/ppm) and length of mixing tubing (cm).

2.4.8 Effect of the Size of Coiled Mixing Part R1

The effect of O.D. of coiled mixing part was studied by using various sizes of test tube over the range of 0.30-1.55 cm. The results in Table 2.9 and Figure 2.12 show that the optimum size of coiled mixing part is 0.80 cm.

Table 2.9 Effect of the size of coiled mixing part on peak height.

Methyl Parathion (ppm)	Peak height* (mV) at the O.D. of test tube (cm)				
	0.30	**0.50**	**0.80**	**1.20**	**1.55**
0	44.0	54.4	52.0	48.0	49.2
2	62.0	72.0	81.2	68.0	67.6
4	84.0	92.0	104.0	92.0	88.0
6	112.0	111.2	128.0	114.0	110.8
Slope (mV/ppm)	11.30	9.52	12.54	11.10	10.26
Correlation coefficient	0.9951	0.9997	0.9985	0.9994	0.9988

*average of triplicate results.

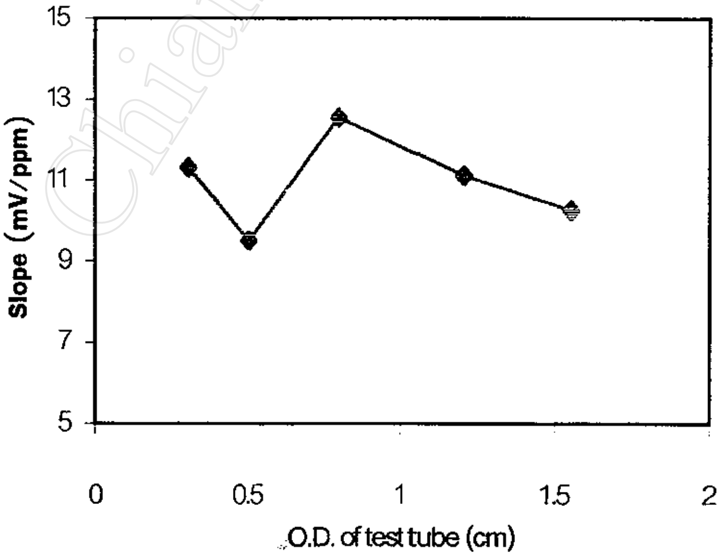

Figure 2.12 Relationship between slope (mV/ppm) and size of coiled mixing part.

2.4.9 Effect of Inner Diameter of Mixing Tubing R2

The I.D. of mixing tubing R2 was also studied over the range of 0.0173 to 0.05 inches. Table 2.10 and Figure 2.13 show that the optimum I.D. of mixing tubing R2 is 0.02 cm.

Table 2.10 Effect of the inner diameter of mixing tubing R2 on peak height.

Methyl Parathion (ppm)	Peak height* (mV) at the I.D. of mixing tubing (in)				
	0.0173	**0.02**	**0.0299**	**0.04**	**0.05**
0	52.0	50.0	56.0	52.0	52.0
2	69.2	72.0	74.0	69.2	71.2
4	90.4	94.0	96.0	92.0	92.0
6	113.0	122.0	120.0	116.0	116.0
Slope (mV/ppm)	10.24	11.90	10.70	10.74	10.64
Correlation coefficient	0.9981	0.9981	0.9979	0.9973	0.9987

*average of triplicate results.

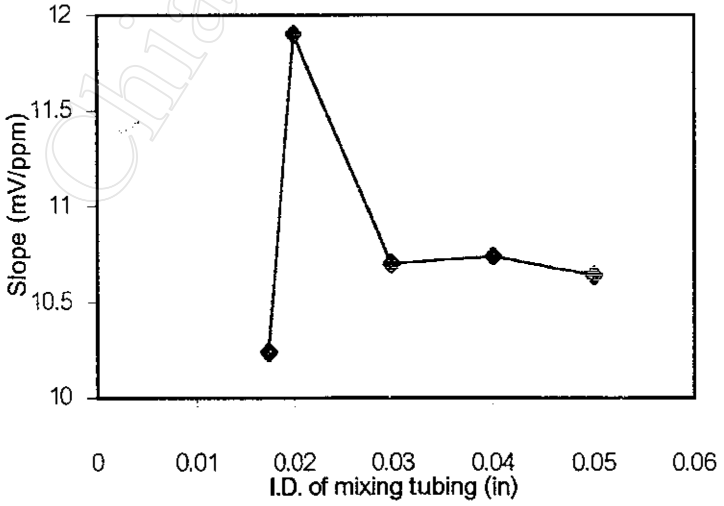

Figure 2.13 Relationship between slope (mV/ppm) and I.D. of mixing tubing R2 (in).

2.4.10 Effect of the Length of Mixing Tubing R2

The effect of the length of mixing tubing R2 on peak height was studied over the range of 50-200 cm. The results in Table 2.11 and Figure 2.14 indicate that the length of 100 cm should be the most appropriate as giving the best sensitivity.

Table 2.11 Effect of the length of mixing tubing R2 on peak height.

Methyl Parathion (ppm)	Peak height* (mV) at the length of mixing tubing (cm)				
	50.00	75.00	100.00	150.00	200.00
0	45.6	48.0	50.0	50.0	43.2
2	74.0	68.0	72.0	72.0	69.2
4	95.6	88.0	98.0	88.0	87.2
6	120.0	120.0	123.2	116.0	111.2
Slope (mV/ppm)	12.24	11.80	12.28	10.70	11.10
Correlation coefficient	0.9986	0.9923	0.9992	0.9945	0.9978

*average of triplicate results.

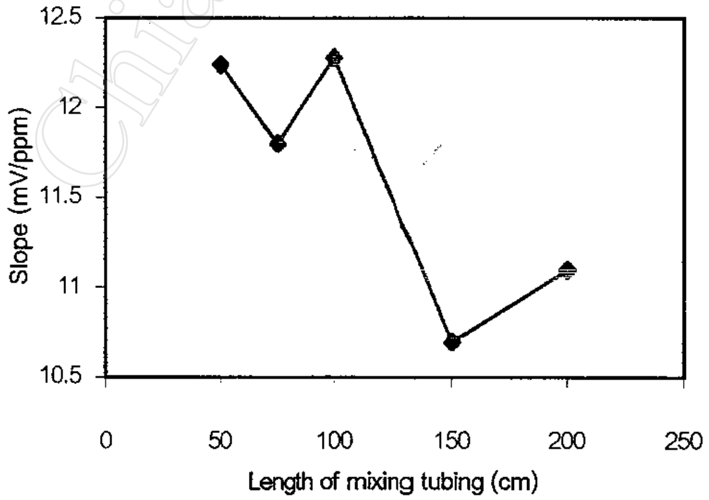

Figure 2.14 Relationship between slope (mV/ppm) and length of mixing tubing R2.

2.4.11 Effect of the size of coiled mixing part

The effect of O.D. of coiled mixing part was also tested. The results obtained in Table 2.12 and Figure 2.15 showed that the optimum size of coiled mixing part is 0.50 cm.

Table 2.9 Effect of the size of coiled mixing part R2 on peak height.

Methyl Parathion (ppm)	Peak height* (mV) at the O.D. of test tube (cm)				
	0.30	**0.50**	**0.80**	**1.20**	**1.80**
0	46.8	48.0	55.2	52.0	54.0
2	64.8	72.0	68.0	71.2	75.2
4	87.2	95.2	94.0	93.6	96.0
6	112.0	120.0	116.8	116.8	120.0
Slope (mV/ppm)	10.90	11.96	10.54	10.84	10.94
Correlation coefficient	0.9975	0.9999	0.9914	0.9991	0.9994

*average of triplicate results.

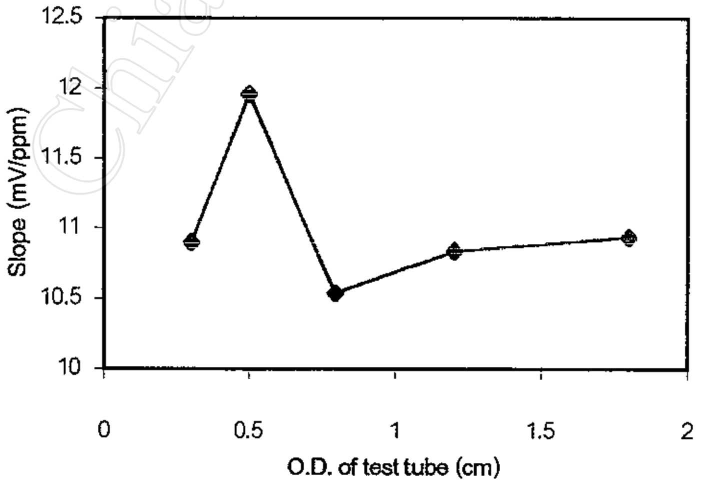

Figure 2.15 Relationship between slope (mV/ppm) and size of coiled mixing part R2.

2.4.12 Effect of Glass Bead Column as a Mixing Reactor R1

A glass bead column made from 3.2 mm I.D. silicone tubing, packed with glass bead (0.3 mm diameter in single straight line), namely single bead string reactor (s.b.s.r.) was used. Investigations were carried out under similar conditions as previously described. Results are shown in Table 2.13 and Figure 2.16. Maximum peak height and slope are obtained when the length of glass bead column R1 was 10.00 cm.

Table 2.13 Effect of the length of s.b.s.r. R1 on peak height.

Methyl Parathion (ppm)	Peak height* (mV) at the length of s.b.s.r. (cm)			
	4.00	7.00	10.00	13.00
0	25.2	33.2	29.6	14.8
2	44.0	56.0	53.2	31.2
4	72.0	80.0	80.0	53.2
6	92.8	106.0	106.4	72.0
Slope (mV/ppm)	11.54	12.12	12.86	9.68
Correlation coefficient	0.9973	0.9996	0.9996	0.9986

*average of triplicate results.

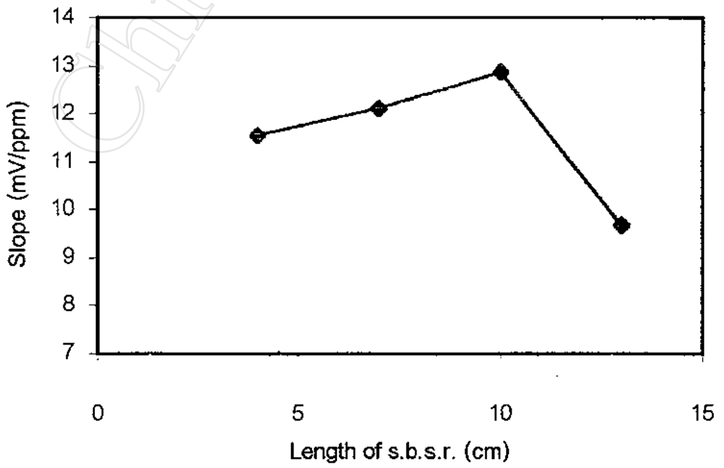

Figure 2.16 Relationship between slope (mV/ppm) and length of s.b.s.r. R1 (cm).

2.4.13 Effect of Glass Bead Column as a Mixing Reactor R2

The effect of the length of s.b.s.r. R2 on peak height was also studied over the range 7.00-20.00 cm. The results in Table 2.14 and Figure 2.17 indicate that the optimum length of s.b.s.r. R2 is 13.00 cm.

Table 2.14 Effect of the length of s.b.s.r. R2 on peak height.

Methyl Parathion (ppm)	Peak height* (mV) at the length of s.b.s.r. (cm)				
	7.00	10.00	13.00	16.00	20.00
0	31.2	30.8	29.6	32.8	29.2
2	52.4	56.0	54.0	57.2	52.0
4	78.4	83.6	82.0	84.4	80.0
6	104.0	112.0	115.2	112.0	108.0
Slope (mV/ppm)	12.22	13.56	14.24	13.24	13.22
Correlation coefficient	0.9989	0.9996	0.9976	0.9996	0.9988

*average of triplicate results.

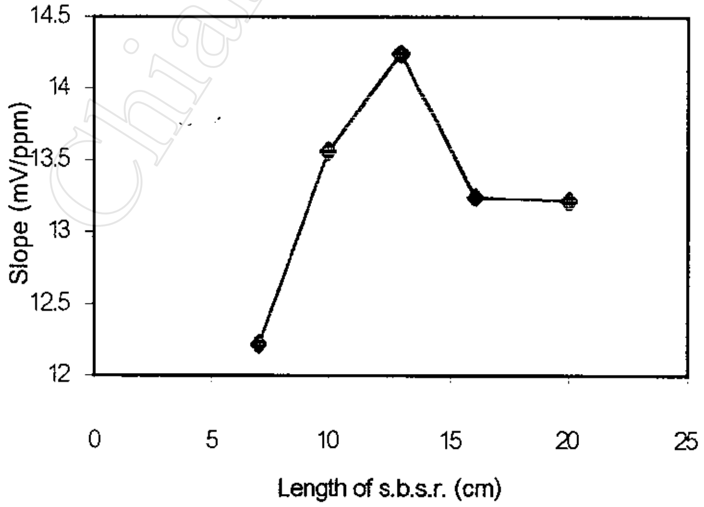

Figure 2.17 Relationship between slope (mV/ppm) and length of s.b.s.r. R2 (cm).

2.4.14 Effect of Various Shapes of Mixing Part R1 and R2

This effect was studied by comparing the peak heights obtained from the various shapes of the mixing parts. The results obtained are shown in Table 2.15 and Figure 2.18. It was found that the s.b.s.r. is the optimum shape, since it exhibits the best sensitivity.

Table 2.15 Effect of various shape of mixing part R1 and R2 on peak height.

Methyl Parathion (ppm)	Peak height* (mV) at the shape of mixing part as				
	s.b.s.r	knitted	zigzag 1×1cm	coil	straight
0	32.0	60.0	62.0	44.0	44.0
2	56.8	84.0	81.6	72.8	72.0
4	84.0	107.2	111.2	85.6	92.8
6	116.8	130.0	133.2	108.0	113.2
Slope (mV/ppm)	14.08	11.66	12.16	10.24	11.42
Correlation coefficient	0.9979	0.9999	0.9972	0.9899	0.9968

*average of triplicate results.

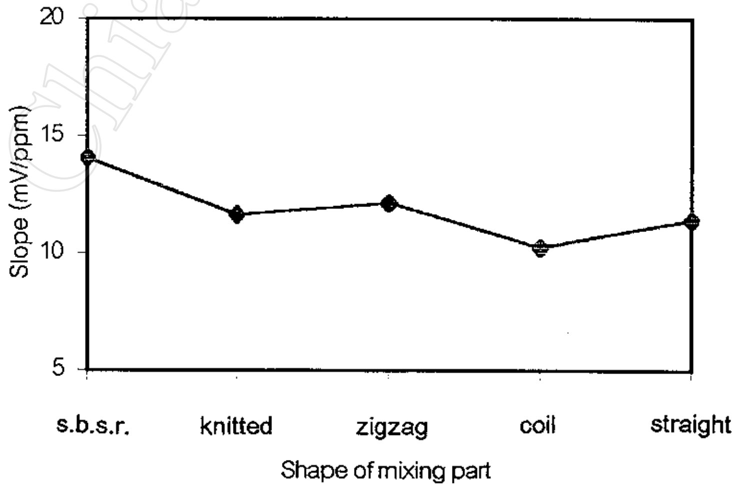

Figure 2.18 Relationship between slope (mV/ppm) and various shape of mixing part.

2.4.15 Effect of Injection Volume

The effect of injection volume was investigated by varying a sample loop length. The results are shown in Table 2.16 and Figure 2.19. It is shown that the greater peak height and sensitivity are obtained when the injection volume is increased. Thus a volume of 500 μl was chosen as optimum injection volume with a reasonable of sample consumption, good sensitivity, and sampling frequency of samples per hour.

Table 2.16 Effect of injection volume on peak height.

Methyl Parathion (ppm)	Peak height* (mV) at the injection volume (μl) as				
	300	400	500	600	700
0	33.6	44.0	48.8	60.0	72.0
2	54.0	71.2	80.0	92.0	102.0
4	80.0	92.8	108.0	122.4	136.0
6	101.2	128.0	146.0	160.0	177.2
Slope (mV/ppm)	11.44	13.68	15.98	16.52	17.48
Correlation coefficient	0.9989	0.9954	0.9980	0.9989	0.9974
t_{base} (sec)	72	90	97.2	108	126
Samples/hr	50	40	37	33	28

*average of triplicate results.

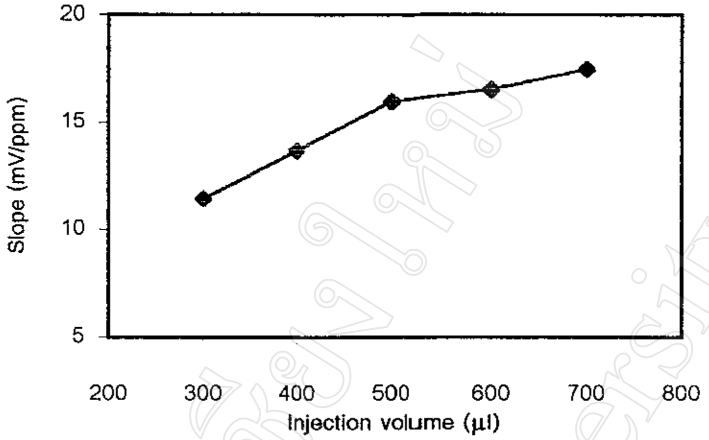

Figure 2.19 Relationship between slope (mV/ppm) and injection volume (µl).

2.5 Analytical Characteristics of the Method

2.5.1 Linear Range

The linear range of the proposed method was studied by injecting methyl parathion standard solutions (0-30 ppm) into FI system under the suitable conditions (Table2.1). The results obtained in Table 2.17 and Figure 2.20 indicated that a linear working calibration curve ranging from 0.5 to 15 ppm of methyl parathion solution with the correlation of 0.9996.

Table 2.17 Optimum conditions for methyl parathion determination.

Analytical characteristics	Information
Sodium hydroxide concentration	2.5 M
Hydroxylamine concentration	1.4 M
Injection volume	500 µl
Pump flow rate	3 ml/min
Shape of mixing part	s.b.s.r.
Length of mixing part (R1 and R2)	10 and 13 cm
Wavelength	410 nm
Chart speed	6 min/cm
Sensitivity	40 mV/cm

Table 2.18 Study of linear range

Methyl parathion (ppm)	Peak height (mV)	Net signal (mV)
0	40.0	-
0.5	44.0	4.0
1.0	51.2	11.2
1.5	56.0	16.0
2.0	64.0	24.0
3.0	79.2	39.2
4.0	92.0	52.0
6.0	120.0	80.0
8.0	148.0	108.0
10.0	179.2	139.2
15.0	244.0	204.0
20.0	305.6	265.6
30.0	404.0	364.0

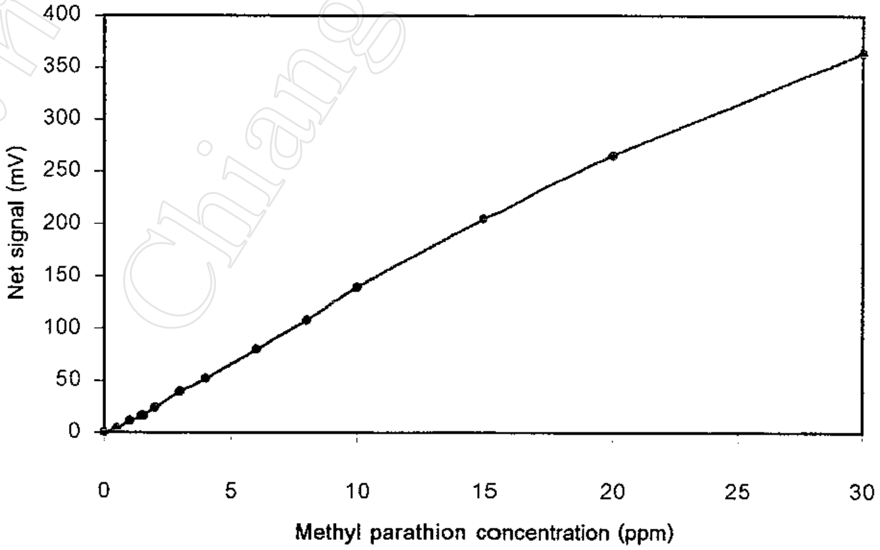

Figure 2.20 Relationship between net signal (mV) and various concentrations of methyl parathion (ppm).

2.5.2 Reproducibility of the Flow Injection System

The reproducibility of the flow injection (FI) system was determined by repeating injection of 2 ppm methyl parathion standard solution (500 μl) into the FI system for 12 replicates. The results are summarized in Table 2.19.

Table 2.19 Reproducibility of replicated determination of methyl parathion.

Experimental no.	Peak height* (mV)
1	68.0
2	68.0
3	70.4
4	69.6
5	69.6
6	69.6
7	68.0
8	69.2
9	68.0
10	68.0
11	68.8
12	68.8
$\overline{X}$	68.83
SD	0.84
%RSD	1.22

*average of triplicate results.

2.5.3 Detection Limit

The detection limit is defined as the concentration of the analyte which gives the signal equivalent to three times of the standard deviation (3σ) of the blank signals(62). The results are given in Tables 2.20 and 2.21. The detection limit of the proposed method was found to be 0.7 ppm of methyl parathion solution.

Table 2.20 Peak height obtained for blank solutions.

Experimental no.	Peak height* (mV)
1	44.0
2	44.8
3	44.8
4	45.2
5	45.2
6	44.0
7	44.0
8	44.0
9	44.0
10	45.2
11	44.8
12	44.4
$\overline{X}$	44.53
SD	0.52
Detection limit	46.09

*average of triplicate results.

Table 2.21 Peak heights obtained from low concentration of methyl parathion determination.

Methyl parathion (ppm)	Peak height* (mV)
0.5	44.0
0.7	46.8
1.0	51.2
1.5	56.0

*average of triplicate results.

2.5.4 Calibration Curve

As depicted in Figure 2.1, methyl parathion standard solutions (2-10 ppm) were injected into the FI system under the established optimum conditions (Table 2.17). The results are shown in Table 2.22 and Figure 2.21. The calibration curve as shown in Figure 2.22 was established by plotting peak height versus the various methyl parathion concentrations. A correlation coefficient (r) is 0.9994 and the calibration equation is

$$Y = 14X - 5.2$$

where Y is net signal in mV

X is concentration of methyl parathion in ppm

Table 2.22 Peak height for calibration curve

Methyl parathion (ppm)	Peak height (mV)	Net signal (mV)
0.0	42	-
2.0	66	24
4.0	92	50
6.0	120	78
8.0	148	106
10.0	178	136

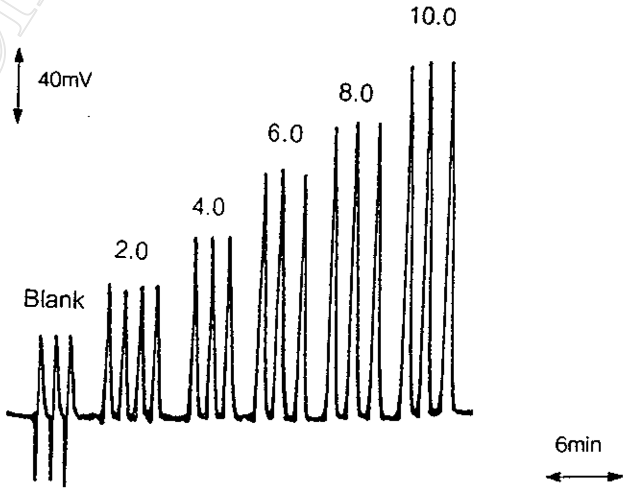

Figure 2.21 Calibration signal output of methyl parathion determination.

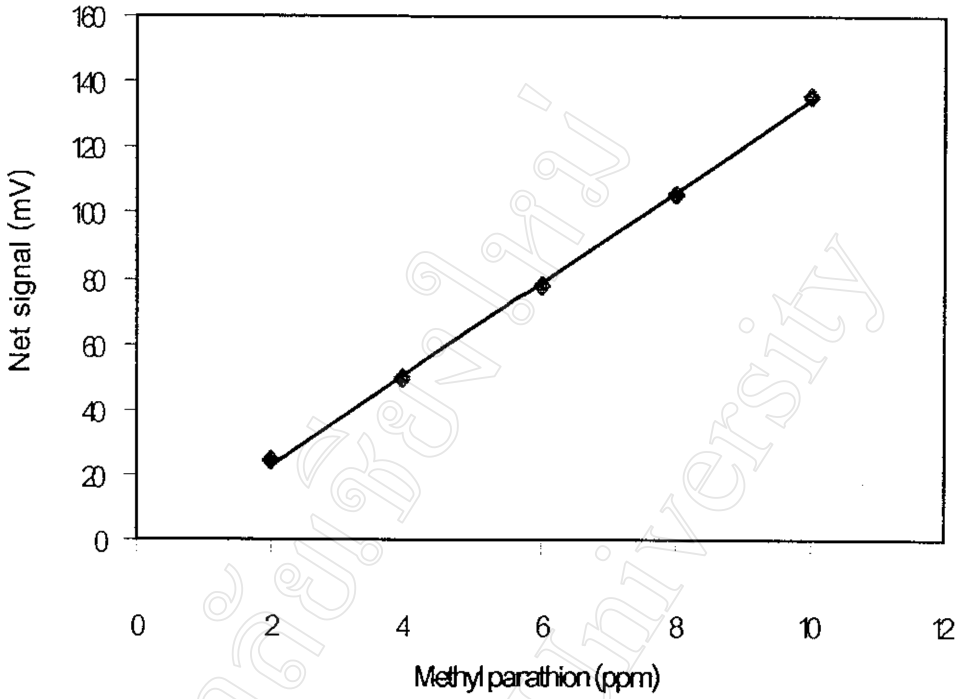

Figure 2.22 The calibration curve of methyl parathion determination.

2.5.5 Effect of Interferences

The effect of some possible interferences were studied by adding known amounts of each interference to 4 ppm of methyl parathion standard solution and injecting into the FIA system. The results are presented in Table 2.23.

Table 2.23 Effect of interferences

Interference	Methyl parathion : Interference	Peak height (mV)	%Relative error
Methyl parathion	-	100	-
Ca^{2+}	1 : 2	100	0
	1 : 5	100	0
	1 : 10	116	+16
	1 : 15	124	+24
	1 : 20	152	+52

Table 2.23 Continued

Interference	Methyl parathion : Interference	Peak height (mV)	%Relative error
Mg^{2+}	1 : 10	100	0
	1 : 20	100	0
	1 : 50	104	+4
	1 : 100	124	+24
Na^{+}	1 : 10	100	0
	1 : 20	100	0
	1 : 50	92	-8
	1 : 100	90	-10
K^{+}	1 : 10	100	0
	1 : 20	99	-1
	1 : 50	92	-8
	1 : 100	90	-10
	1 : 200	88	-12
SO_4^{2-}	1 : 10	100	0
	1 : 20	100	0
	1 : 50	100	0
	1 : 100	98	-2
	1 : 200	92.8	-7.2
PO_4^{3-}	1 : 10	100	0
	1 : 20	100	0
	1 : 50	100	0
	1 : 100	104	+4
	1 : 200	106	+6
Malathion	1 : 2	100	0
	1 : 5	100	0
	1 : 10	124	+24
	1 : 50	212	+112

Table 2.23 Continued

Interference	Methyl parathion : Interference	Peak height (mV)	%Relative error
Methamidophos	1 : 10	100	0
	1 : 50	100	0
	1 : 100	102	+2
	1 : 200	106	+6
Monocrotophos	1 : 5	100	0
	1 : 10	104	+4
	1 : 30	110	+10
	1 : 50	130	+30
Carbaryl	1 : 10	100	0
	1 : 30	100	0
	1 : 50	104	+4
	1 : 100	120	+20