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RESEARCH ARTICLE

Determination of Diethyl Amine by Stop Flow Technique

Dakhil Nassir Taha[1], Sadiq Jaafer Baqir[2], Ahmed Shaalan Khlaif[3]

[1.] *Department of Chemistry, College of Science for Women, University of Babylon, 51002 Babylon, Iraq.*

[2.] *Al-Mustaqbal University College, 51002 Babylon, Iraq.*

[3.] *Department of Chemistry, College of Science, University of Babylon, 51002 Babylon, Iraq.*

Abstract

The aim of this research is determination of diethyl amine by stop flow injection analysis technique. The method is based on the reaction between diethyl amine with NQS reagent. This method involves a homemade new valve, which is manufactured from a cheap available material. Optimization of the conditions of the reaction includes, flow rate, sample volume, reagent volume, reagent concentration and reaction coil length were done. Linearity was in the range of (1-10 v/v %), correlation coefficient (R^2) was 0.9967. The method was applied successfully to the determination of diethyl amine in aqueous solution.

Keywords: *Diethyl amine, FIA, Stop flow technique, Dispersion.*

Introduction

Flow Injection (FI) was first introduced in 1975 by Ruzicka and Hansen in Denmark and Stewart in USA [1, 2, 3]. This technique is used for the determination tracing [4, 5, 6]. It is an automated or semi-automated [7] analytical sample processing technique which is based on injection of definite volume of liquid sample solution into a continuously flowing unsegmented carrier stream, followed by the quantization of species of interest at a downstream detection area. The injected sample forms a zone, which is then transported toward a detector that continuously records the changes in absorbance, electrode potential, or other physical parameter resulting from the passage of the sample material through the flow cell.

This technique has high ability and in importance than other techniques which are: Analysis of very low volumes of samples , High speed of sampling , Low detection limit and concentration zone broadening and Small equipment [8] and has many applications For example used in the determination of hydrogen peroxide [9]. Iron (II) [10], Aniline blue [11], Malachite green dye [12]. In this paper, FI methods using spectrophotometric detection at 480 nm are described for the determination of diethyl amine with NQS via exchange reaction.

The proposed methods have been successfully applied to the determination of diethyl amine in aqueous solution.

Experimental

Apparatus

Balance, UV-visible spectrophotometer, Kompensog Graph, Reaction coil (made from glass and Teflon), Quaternary valve (homemade), loops (made from Teflon).

Preparation of Stock Solutions

Materials

Stock solutions (20v/v %) of diethyl amine is prepared by tacking 10ml from diethyl amine and diluting to 50 ml in a volumetric flask. Working solutions were prepared by diluting the above solution by distilled water. -(0.04 w/v %) of (NQS) was prepared by dissolving (0.02g) in a sufficient amount of water then completing the volume to the marking a 50ml volumetric flask.

Methodology

A new valve was manufactured according to the primary valve which designed from cheap materials [13]. According to the flow diagram shown in Figure 1, loop1 is loaded with NQS (157.00 µL), loop 2 is loaded with diethyl amine (157.00 µL).

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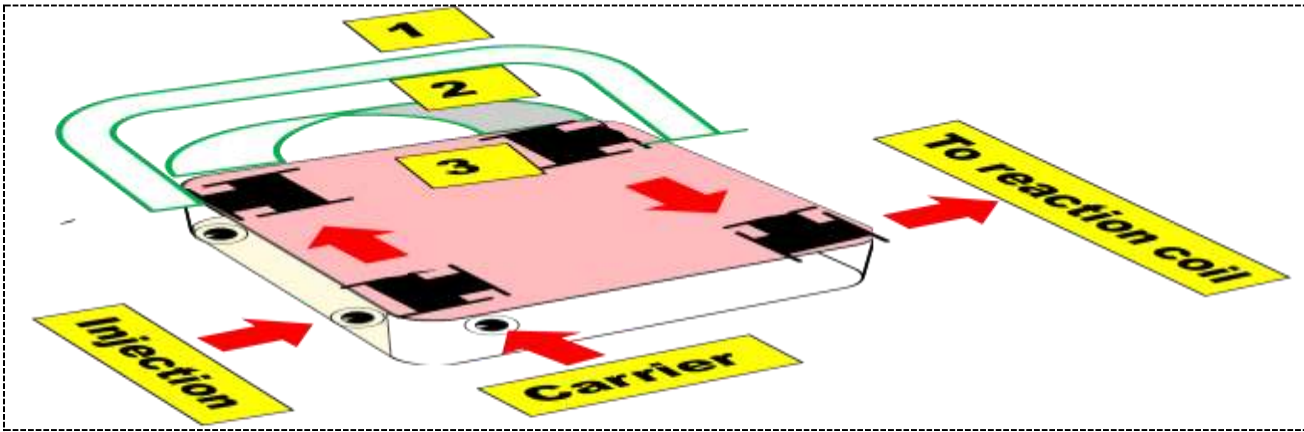

Fig. 1: Valve (1-Reagent loop, 2- sample loop, 3- sub valve)

The simplest FIA unit consists of a pump, an injection valve, reaction coil, detector and recorder. As shown in Figure (2).

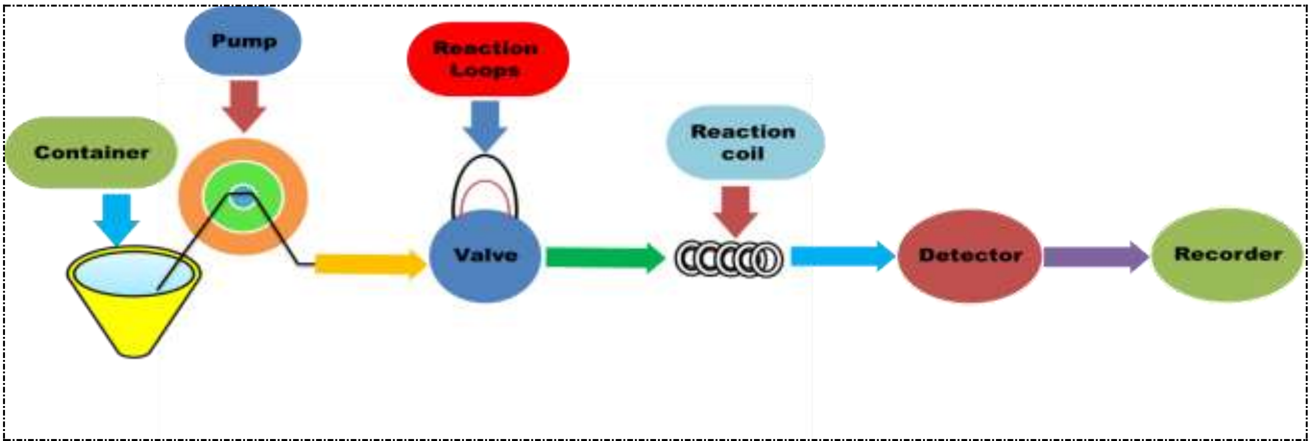

Fig. 2: FIA unit design

Figure (3) shows the response obtained which clearly indicate to the absorption spectrum of a mixture.

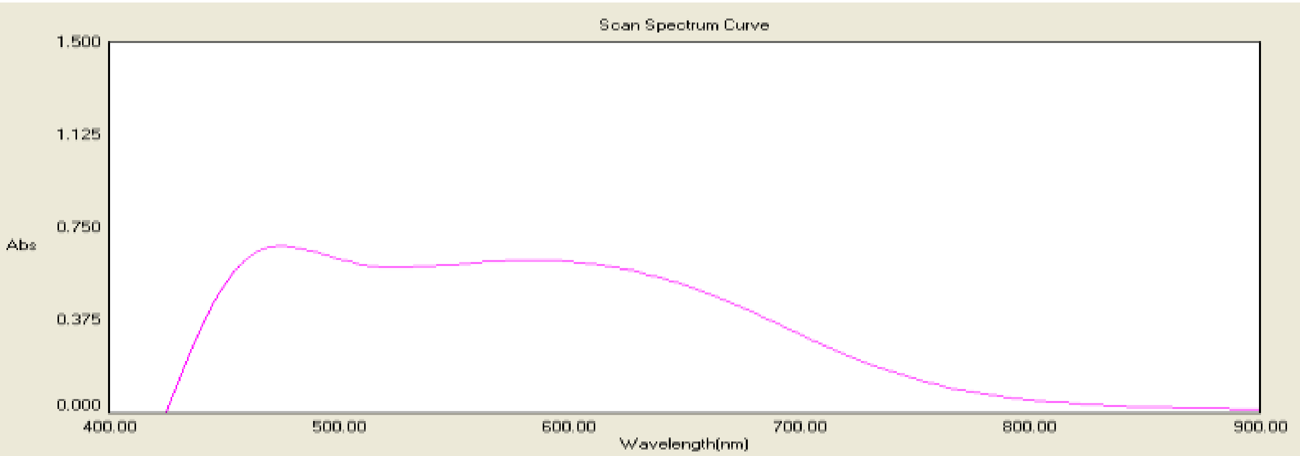

Fig. 3: the absorption spectra of mixture

Results and Discussion

Stop Time of Reaction

The reaction time for the system was studied through stop time method after completion of mixing of the reactants, the time range used (1-6 min) at: Flow rate (12.3ml/min), reaction coil (100 cm), sample volume (157µl) reagent volume (157µl), order of addition (NQS+ amine). Sample concentration (6v/v %), reagent concentration (0.01w/v %).

The results were appearing that the response is increased (1.516, 1.783, 2.300 cm) with increasing the time from (1-5 min) while mixing time (6 min) the response which decrease. According to the result in the Table (1) and Figure (4), the best response was set at the mixing time (5min).

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Table 1: stop time of reaction

stop time(min)	Response(cm)			Mean	S.D	R.S.D%
1	1.450	1.500	1.600	1.516	0.074	4.881
3	1.700	1.850	1.800	1.783	0.074	4.150
5	2.300	2.300	2.300	2.300	0.000	0.000
6	2.000	2.100	2.000	2.033	0.056	0.027

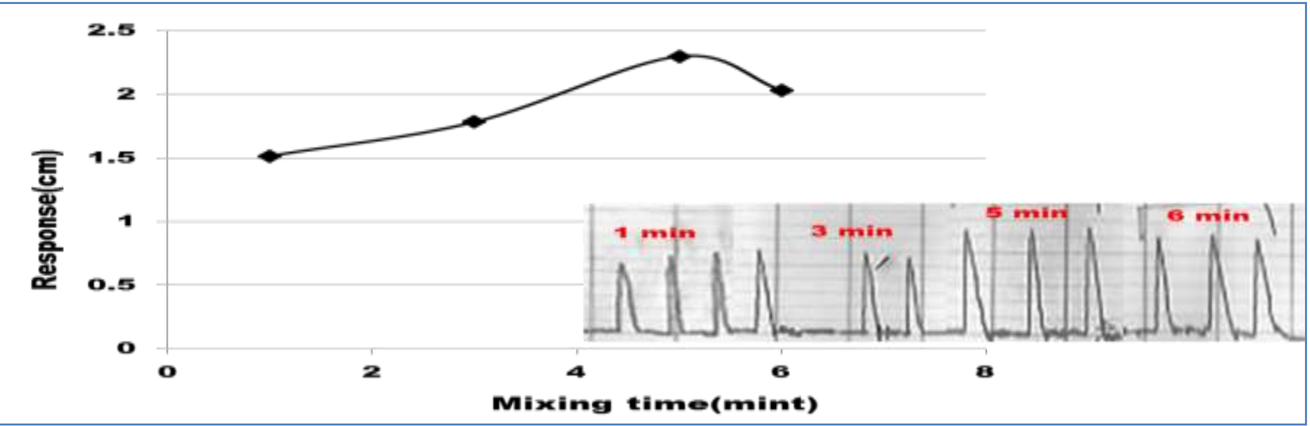

Fig. 4: stop time of reaction

Effect of the Order of Addition

The effect of the order of addition was studied at: Flow rate (12.3ml/min), reaction coil (100 cm), sample volume (157µl) reagent volume (157µl), mixing time (5min), sample concentration (6v/v %) reagent concentration (0.01w/v %).

The preferred response was at the order of addition (NQS + Diethyl amine) according to the result in the Table (2) and Figure (5) as it gives higher mean response (2.333 cm) than other order of addition.

Table 2: effect of the order of addition

Mixing time(min)	Response(cm)			Mean	S.D	R.S.D%
NQS+Diethyl amin	2.400	2.300	2.300	2.333	0.056	2.400
((NQS+Diethyl amin)+NH3)	1.600	1.600	1.600	1.600	0.000	0.000
((Diethyl amin+NH_3)+NQS)	1.600	1.600	1.600	1.600	0.000	0.000

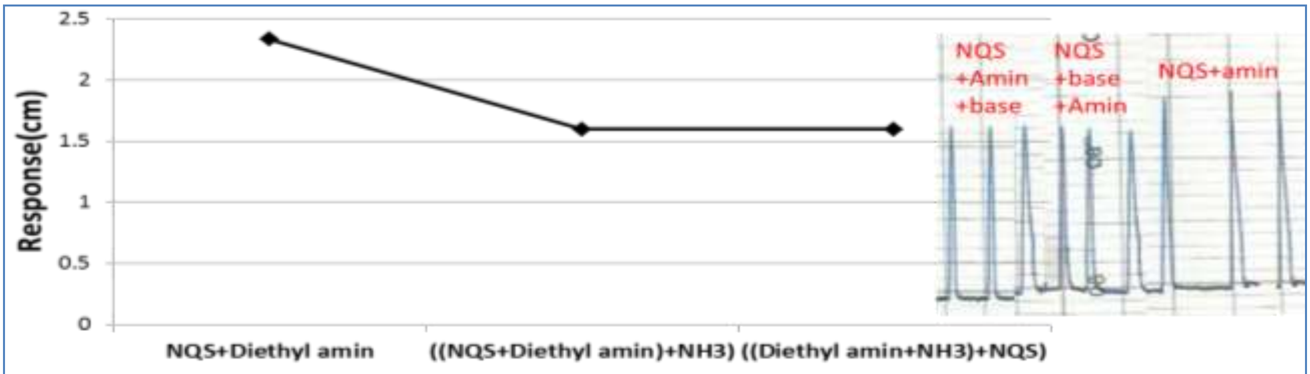

Fig. 5: Effect of the order of addition

Effect of the Reaction coil Length

In this study, different reaction coil length (30-150 cm) was used at: Flow rate (12.3ml/min), order of addition (NQS+ amine), sample volume (157µl) reagent volume (157 µl), mixing time (5min), sample concentration (6v/v %) reagent concentration (0.01w/v %).

From the result of the Table (3) and Figure (6), the best response was at the reaction coil length (100cm).

Table 3: Effect of reaction coil length

Reaction coil length (cm)	Peak length(cm)			Mean	S.D	R.S.D%
0	0.800	0.800	0.800	0.800	0.000	0.000
30	1.200	1.250	1.200	1.213	0.030	2.473
50	1.200	1.200	1.100	1.166	0.057	4.888
100	2.300	2.300	2.300	2.300	0.000	0.000
115	1.900	1.900	2.000	1.933	0.056	2.897

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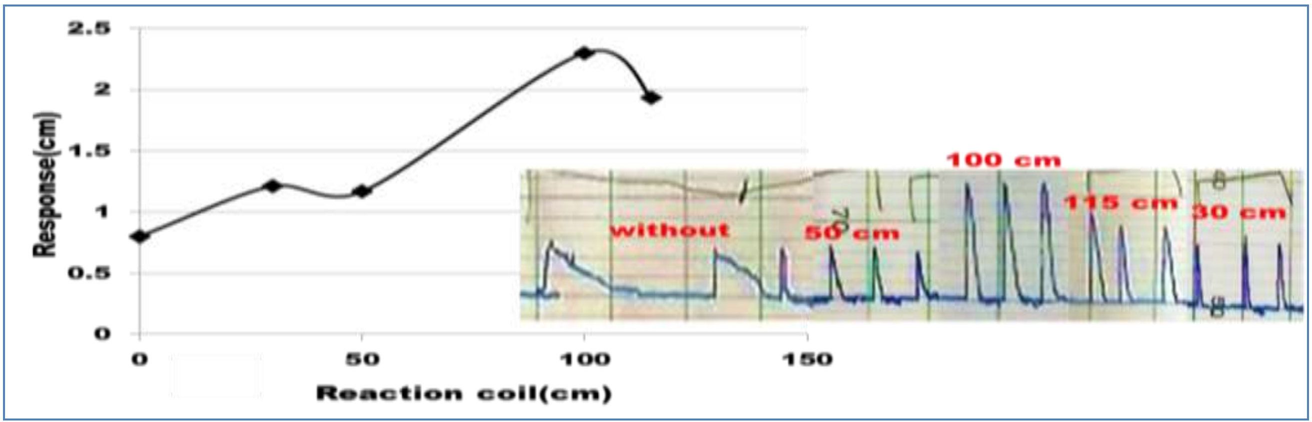

Fig. 6: Effect of reaction coil

Effect of Flow Rate

The effect of flow rate was studied in the range of(3.3-12.3 ml/ min) and choosing the best response at: reaction coil (100 cm), order of addition (NQS+ amine), sample volume (157µl) reagent volume (157µl), mixing time (5min), sample concentration (6v/v%) reagent concentration (0.01w/v%).

The result in Figure (7) and Table (4) shows the effect of flow rate on the response value at the above condition. The flow rate (10 ml/min) has response mean (2.850 cm) and this flow rate is favored over the other flow rate (3.3,4.9,6.6,8.1.12.3) because it has higher value of the response mean.

Table 4: Effect of Flow rate

R.S.D%	S.D	Mean	Peak length(cm)			Flow rate(ml/min)	Speed pump
0.000	0.000	0.800	0.800	0.800	0.800	3.3	20
2.657	0.027	1.016	1.050	1.000	1.000	4.9	30
2.899	0.057	1.966	2.000	1.900	2.000	6.6	40
0.906	0.022	2.426	2.400	2.440	2.440	8.1	50
0.000	0.000	2.850	2.850	2.850	2.850	10	60
0.000	0.000	2.300	2.300	2.300	2.300	12.3	70

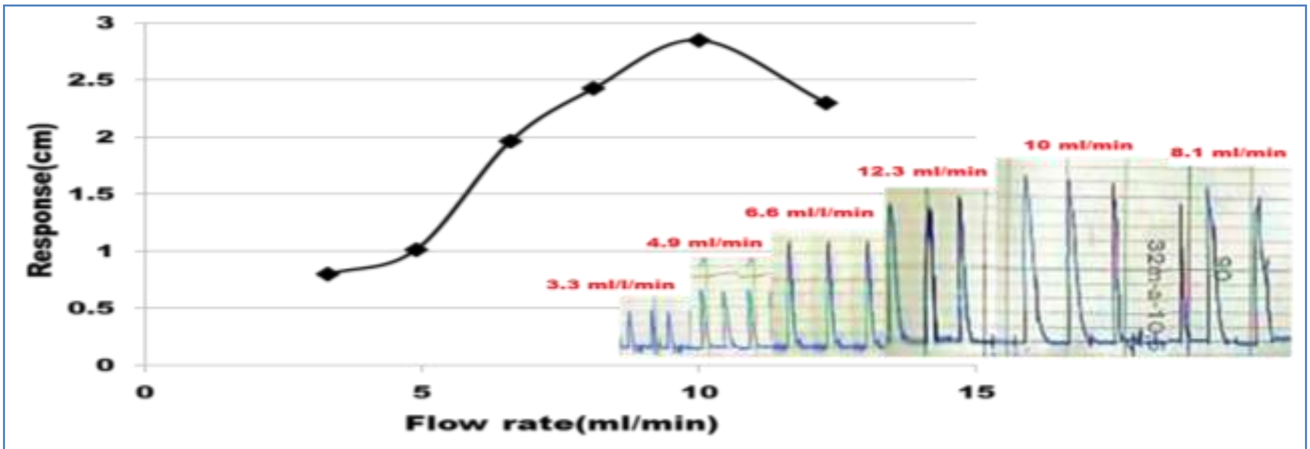

Fig. 7: Effect of Flow rate

Effect of Sample Volume

The effect of sample volume was investigated in the range(78.50 - 314 µL) the following parameters: Reaction coil (100 cm), order of addition (NQS+ amine), flow rate (10 ml/min) reagent volume (157µl), mixing time (5min), sample concentration (6v/v%) Reagent Concentration (0.01w/v%).

The best volume of sample that gives the best response for the reaction between diethyl amine and NQS is 157 µL as shown in Table(5) Figure(8).

Table 5: Effect of sample volume

Sample volume(µl)	Peak length(cm)			Mean	S.D	R.S.D%
078.500	1.800	1.800	1.800	1.800	0.000	0.000
157.000	2.800	2.800	2.800	2.800	0.000	0.000
196.250	1.500	1.500	1.550	1.516	0.027	1.781
235.500	1.200	1.200	1.200	1.200	0.000	0.000
314.000	1.800	1.770	1.800	1.790	0.017	0.949

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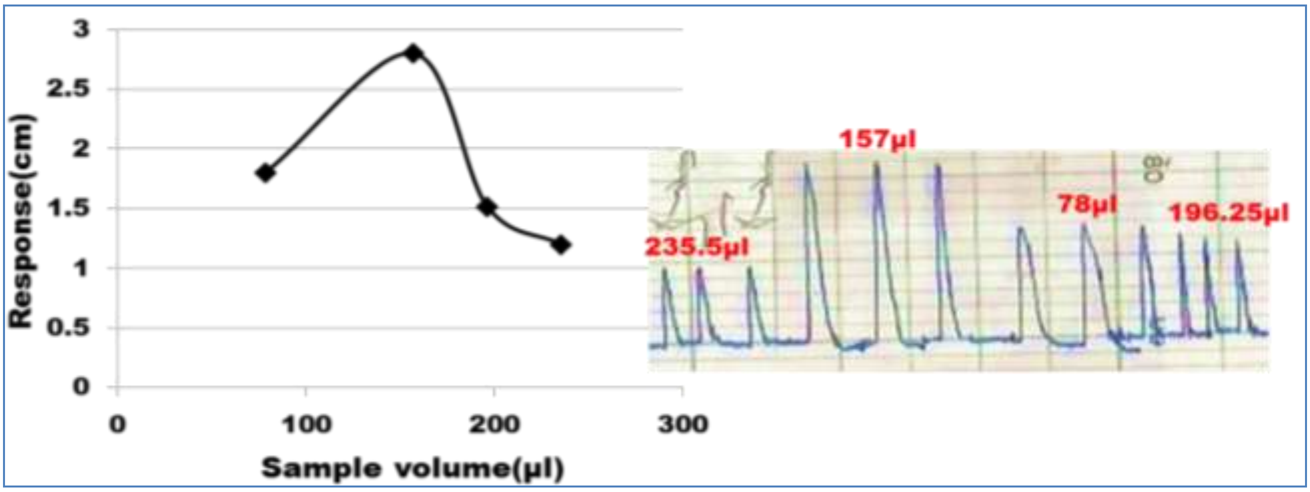

Fig 8: Effect of sample volume

Effect of Reagent Volume

The effect of reagent volume was investigated in the range (117.75- 314µL). The results in (Table (6) and Fig (9) showed that the reagent volume of 157µL gave the best response at the constant parameters: reaction coil (100 cm), order of addition (NQS+ amine), flow rate (10 ml/min) sample volume (157µl), mixing time (5min), sample concentration (6v/v %) reagent concentration (0.01w/v %).

Table6: Effect of reagent volume

Reagent volume(µl)	Peak length(cm)			Means	S.D	R.S.D%
117.750	1.800	1.800	1.800	1.800	0.000	0.000
157.000	2.750	2.800	2.750	2.766	0.027	0.976
235.500	2.750	2.700	2.650	2.700	0.050	1.851
314.000	2.000	2.080	2.080	2.053	0.045	2.191

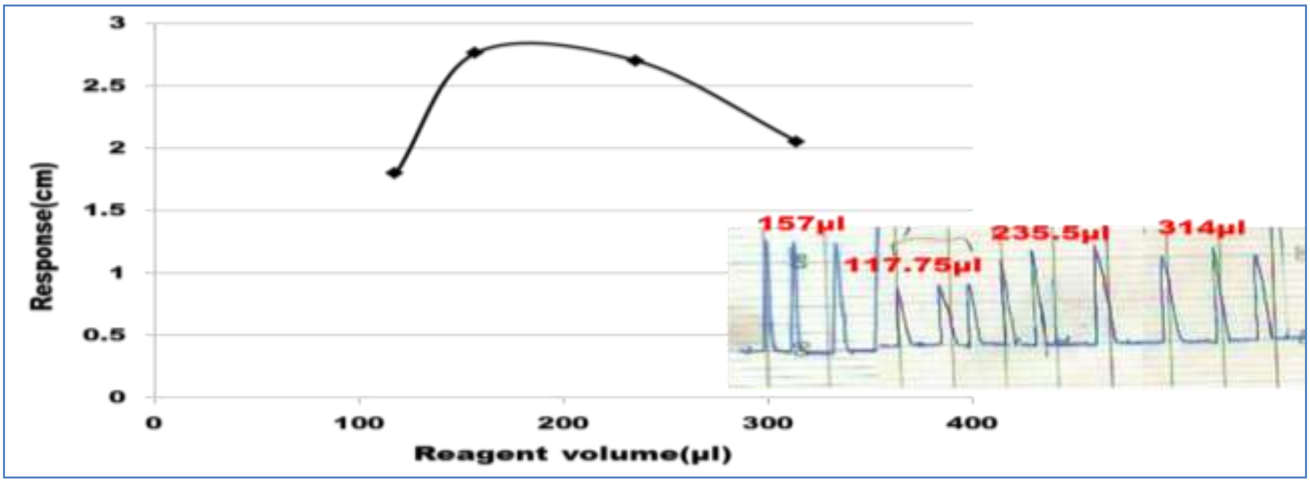

Fig 8: Effect of reagent volume

Effect of Reagent Concentration

The effect of reagent concentration was studied in the range (0.01-0.03 w/v %) at: reaction coil length (100 cm), order of addition (NQS+ amine), flow rate (10 ml/min) sample volume (157µl), mixing time (5min), sample concentration (6v/v %) reagent volume (157µl). The results obtained showed that the concentration of (0.02w/v %) gave the best response as shown in Table (7) and Fig (9).

Table 7: Effect of reagent concentration

Reagent concentration (w/v %)	Peak length(cm)			Means	S.D	R.S.D%
0.01	2.850	2.800	2.800	2.816	0.027	0.958
0.02	3.800	3.850	3.900	3.850	0.050	1.298
0.03	3.100	3.140	3.140	3.126	0.020	0.639

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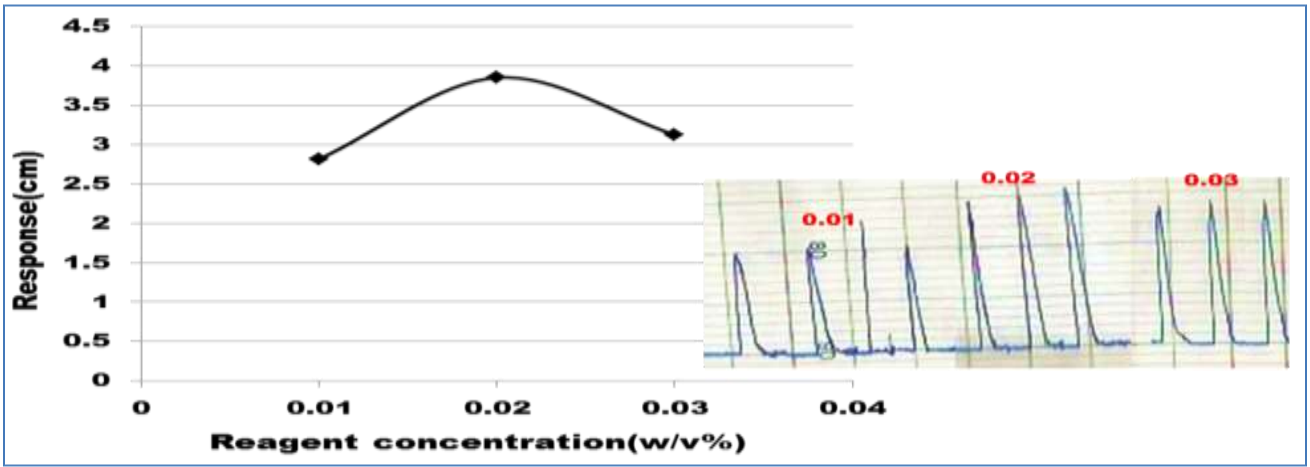

Fig 9: Effect of reagent concentration

Calibration Curve

A series of concentrations of diethyl amine was taken to construct the calibration curve. A linear Calibration graph is shown in (Figure (10) and Table (8) with correlation coefficient of 0.9967 for the range (1-10v/v %) of diethyl amine.

Table 8: calibration curve

Concentration (v/v %)	Peak length(cm)			Means	S.D	R.S.D%
1	0.6	0.650	0.600	0.616	0.027	4.383
2	1.150	1.200	1.200	1.183	0.026	2.197
3	1.400	1.400	1.400	1.400	0.000	0.000
4	2.000	2.000	2.000	2.000	0.000	0.000
5	2.500	2.500	2.500	2.500	0.000	0.000
6	2.900	2.800	2.800	2.833	0.056	1.976
7	3.250	3.200	3.300	3.250	0.050	1.538
8	3.700	3.700	3.700	3.700	0.000	0.000
9	4.300	4.200	4.200	4.233	0.056	1.322
10	4.500	4.500	4.500	4.500	0.000	0.000

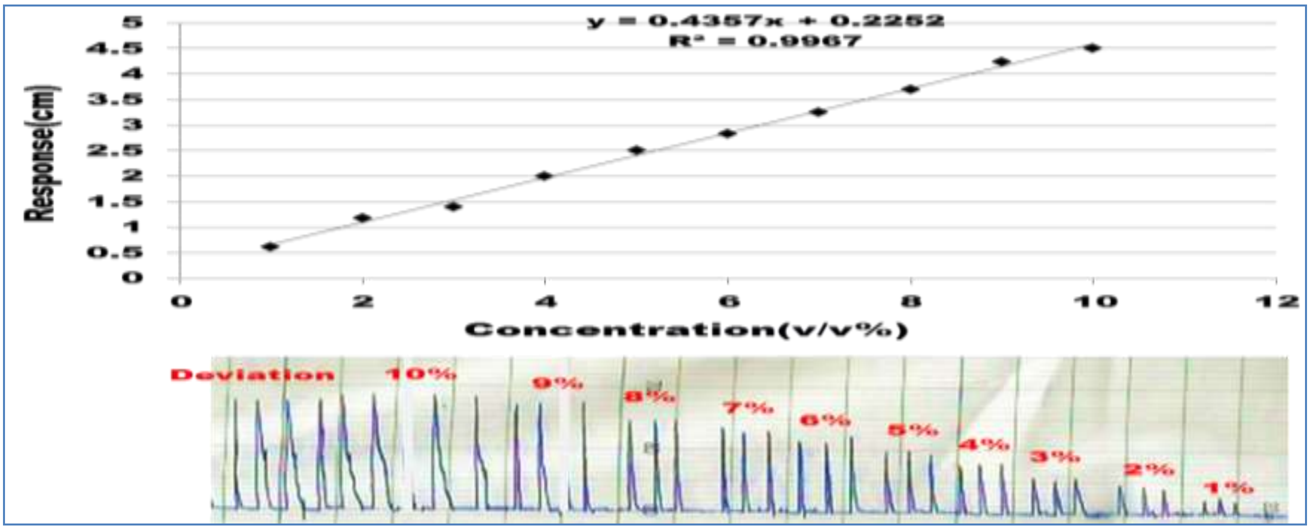

Fig 10: calibration curve

Table 9: Factors value of calibration curve

Parameter	Value
Linearity range (v/v%)	1-10
Linear regression equation	Y= 0.4357x + 0.2252
R^2	0.9967
Correlation coefficient(r)	0.9983
Relative Standard Deviation of 7v/v%	1.538
Slope (m)	0.4357
Intercept (i)	0.2252

Reproducibility

The reproducibility is repeating of measurements at the same concentration (10v/v %) and condition for many times. The purpose of this experiment is to know the accuracy and efficiency of the system. the results of Figure (11) and Table (9) show good reproducibility of the measurements.

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Table 10: Reproducibility

R.S.D%	S.D	means	Response				concentration
1.381	0.062	4.487	4.5	4.4	4.5	4.55	10v/v%

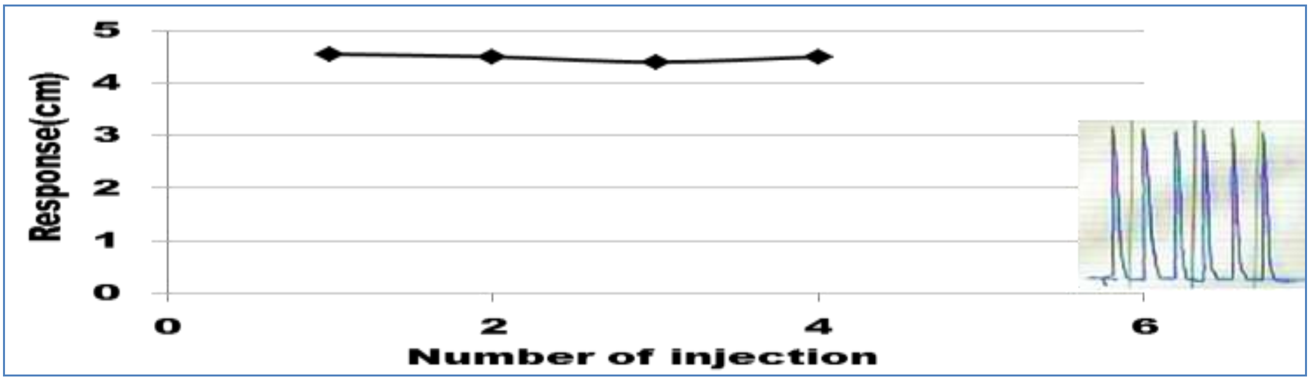

Fig 11: Reproducibility

The Dispersion

The dispersion was studied to determine the degree of dilution of the sample from injection point until passage through flow cell before the recorder. The results (Figure (12) show that the dispersion coefficient is (1.44) which is in the desired limit.

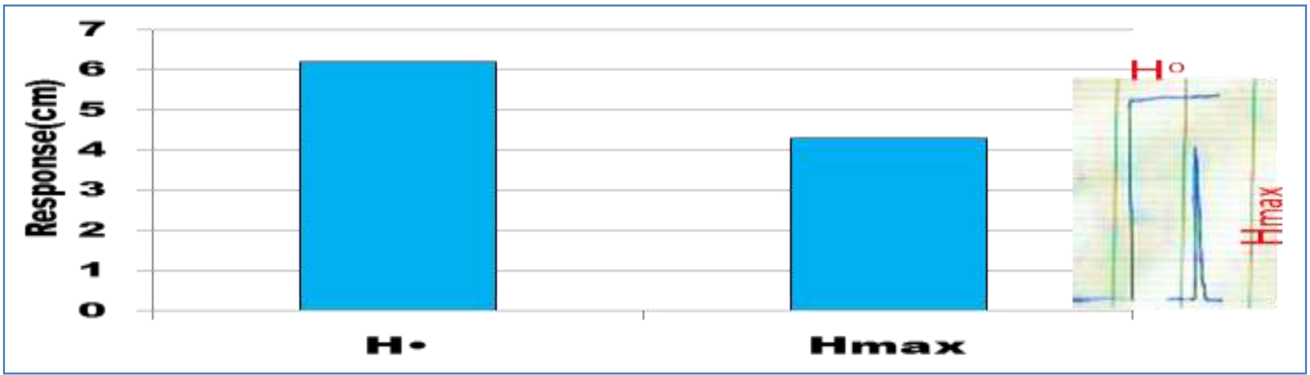

Fig 12: The dispersion

Dead Volume

The purpose of this study is to know the accuracy of the manufactured valve by doing two experiments. The first experiment including the injection of NQS (reagent) only without injection of diethyl amine, then in the next experiment diethyl amine is injected alone without injection of NQS. The dead volume was equal to zero.

Analytical Application

The proposed method was successfully applied to an aqueous solution, as shown in Table (11) and Fig (13).

Table 11: Application of the proposed methods to the determination of diethyl amine in aqueous solution

Sample	Reagent	Amount of sample (v/v %)		Proposed method							
		Taken	Found	Response(cm)			Means	S.D	RSD%	Error%	Rec%
aqueous solution	NQS	4.000	4.500	2.200	2.200	2.200	2.200	0.000	0.000	1.250	101.250
		5.000	5.000	2.550	2.4500	2.500	2.500	0.050	2.000	0.000	100.000

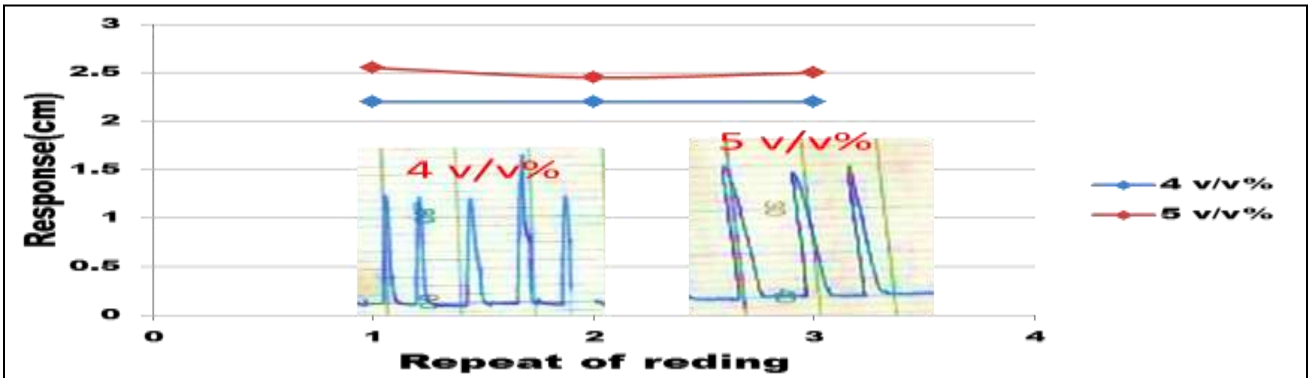

Fig 13: application of the proposed methods to the determination of diethyl amine in aqueous solution

Conclusions

The proposed method results in a simple, fast, inexpensive, precise and accurate analytical technique to determine diethyl amine.

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