CHAPTER IV

RESULTS AND DISCUSSIONS

The Study of Temperature

The effect of temperature on the distribution coefficient (K) and the sensitivity (S) of each volatile organic compound i.e., methylene chloride, chloroform, benzene, trichloroethylene and 1,4-dioxane are studied. The results of the study on the distribution coefficient (K) are presented in Tables 4.1 and 4.2 and the graphs showing the relationship of the distribution coefficient of each organic volatile compound with temperature are shown in Figures 4.1 and 4.2. The results as in the Figure show that the distribution coefficient of each volatile organic compound decreases when the temperature of system increases and thus the temperature has the effect on the distribution coefficient of each compound this can be explained by the fact that raising temperature will increase the vapor pressure of each compound also decrease their solubility in the solution as shown in Tables 4.1 and 4.2.

The result of the effect of the temperature on sensitivity of each volatile organic compound are shown in Tables 4.3 and 4.4 and the graphs plotted the sensitivity of each compound against temperature are shown in Figures 4.1 and 4.2. It demonstrates that the temperature has the effect on sensitivity of each volatile organic compound and therefore increasing temperature of the system will

result in the enhancement of the sensitivity of headspace analysis technique. According to the results in Tables 4.3 and 4.4 , it can be seen that the highest sensitivity of the headspace analysis technique is obtained at the temperature of 90 °C which is different from the temperature used in the study. The reason is that increasing the temperature of the system build up the pressure in it and is causing the leak of the components from headspace sample vials. Moreover, the water vapor in the headspace gas will be increased at high temperature resulting in the decrease in the detector response. Therefore, the temperature of 70 °C is selected as an optimum temperature for this headspace analysis due to it gives a , high precision as shown the percent relative standard deviation (%RSD) in Table 4.3 and 4.4 and is a sufficient sensitivity for the determination of each volatile organic compound.

Table 4.1 The effect of temperature on the distribution coefficient and the equilibrium concentration of each volatile organic compound in gas phase with concentration of aqueous standard solution in lower level of ppm.

Compound	Temperature (°C)	Peak area	K	C_g (ppm)
Methylene chloride	60.0	5756	34.08	0.12
(4.21 ppm)	70.0	6476	29.07	0.14
	80.0	6636	27.07	0.15
	90.0	7246	25.31	0.16
Chloroform	60.0	4544	25.50	0.16
(4.24 ppm)	70.0	5323	21.32	0.19
	80.0	5664	20.20	0.20
	90.0	6208	18.27	0.22
Benzene	60.0	59600	22.28	0.18
(4.19 ppm)	70.0	68982	18.95	0.21
	80.0	83539	15.76	0.25
	90.0	87661	15.12	0.26
Trichloroethylene	60.0	10014	22.39	0.18
(4.21 ppm)	70.0	11581	19.05	0.21
	80.0	15872	13.52	0.29
	90.0	17937	13.03	0.30
1,4-Dioxane	60.0	3507	2480.50	0.04
(99.26 ppm)	70.0	5814	1653.33	0.06
	80.0	9119	1101.89	0.09
	90.0	13428	708.00	0.14

Triplicate analyses

Table 4.2 The effect of temperature on the distribution coefficient and the equilibrium concentration of each volatile organic compound in gas phase with concentration of aqueous standard solution in higher level of ppm.

Compound	Temperature (^{o}C)	Peak area	K	C_g (ppm)
Methylene chloride	60.0	16111	35.09	0.35
(12.63 ppm)	70.0	17913	31.38	0.39
	80.0	19144	29.80	0.41
	90.0	19631	29.07	0.42
Chloroform	60.0	12224	27.93	0.44
(12.73 ppm)	70.0	14754	22.57	0.54
	80.0	14849	22.57	0.54
	90.0	16191	20.58	0.59
Benzene	60.0	134591	30.45	0.40
(12.58 ppm)	70.0	161598	25.21	0.48
	80.0	174589	23.19	0.52
	90.0	185036	21.87	0.55
Trichloroethylene	60.0	29611	25.31	0.48
(12.63 ppm)	70.0	33063	22.39	0.54
	80.0	45044	16.54	0.72
	90.0	45435	16.07	0.74
1,4-Dioxane	60.0	13810	2126.07	0.14
(297.79 ppm)	70.0	22309	1352.59	0.22
	80.0	34815	874.85	0.34
	90.0	50645	605.73	0.49

Triplicate analyses

Table 4.3 The effect of temperature on the sensitivity of each volatile organic compound with concentration of aqueous standard solution in lower level of ppm.

Compound	Temperature (°C)	Sensitivity	%RSD
Methylene chloride	60.0	1367	1.11
(4.21 ppm)	70.0	1538	3.45
	80.0	1576	1.21
	90.0	1721	1.11
Chloroform	60.0	1071	2.89
(4.24 ppm)	70.0	1255	1.25
	80.0	1335	1.50
	90.0	1463	2.17
Benzene	60.0	14205	2.80
(4.19 ppm)	70.0	16444	0.92
	80.0	19903	2.00
	90.0	20896	2.07
Trichloroethylene	60.0	2379	3.22
(4.21 ppm)	70.0	2751	2.52
	80.0	4007	2.93
	90.0	4260	2.62
1,4-Dioxane	60.0	35	3.05
(99.26 ppm)	70.0	59	2.06
	80.0	92	2.86
	90.0	135	3.05

Triplicate analyses

Table 4.4 The effect of temperature on the sensitivity of each volatile organic compound with concentration of aqueous standard solution in higher level of ppm.

Compound	Temperature (°C)	Sensitivity	%RSD
Methylene chloride	60.0	1276	3.95
(12.63 ppm)	70.0	1418	2.34
	80.0	1515	3.07
	90.0	1554	3.29
Chloroform	60.0	961	3.19
(12.73 ppm)	70.0	1159	0.39
	80.0	1167	3.77
	90.0	1272	1.99
Benzene	60.0	10692	2.55
(12.58 ppm)	70.0	12840	0.07
	80.0	13867	3.00
	90.0	14702	2.55
Trichloroethylene	60.0	2344	2.09
(12.63 ppm)	70.0	2618	1.49
	80.0	3565	0.71
	90.0	3574	3.02
1,4-Dioxane	60.0	46	2.22
(297.79 ppm)	70.0	75	0.54
	80.0	117	1.44
	90.0	170	0.80

Triplicate analyses

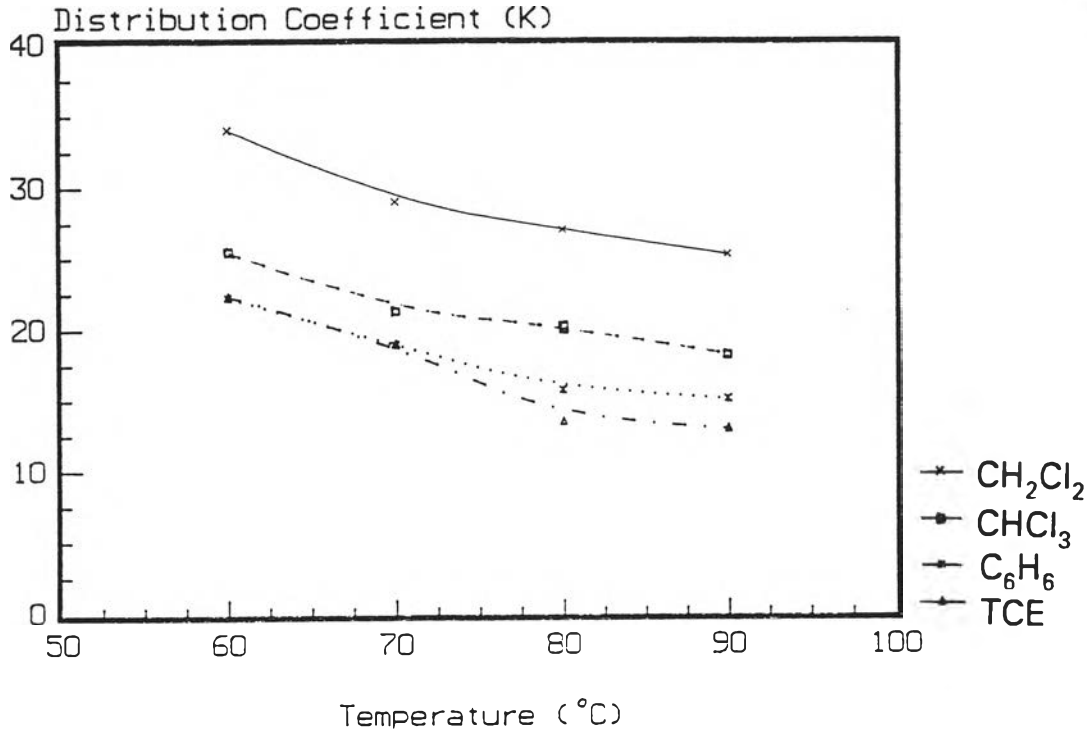


Figure 4.1 The effect of temperature on the distribution coefficient of each volatile organic compound with concentration (except 1,4-dioxane) of aqueous standard solution in lower level of ppm.

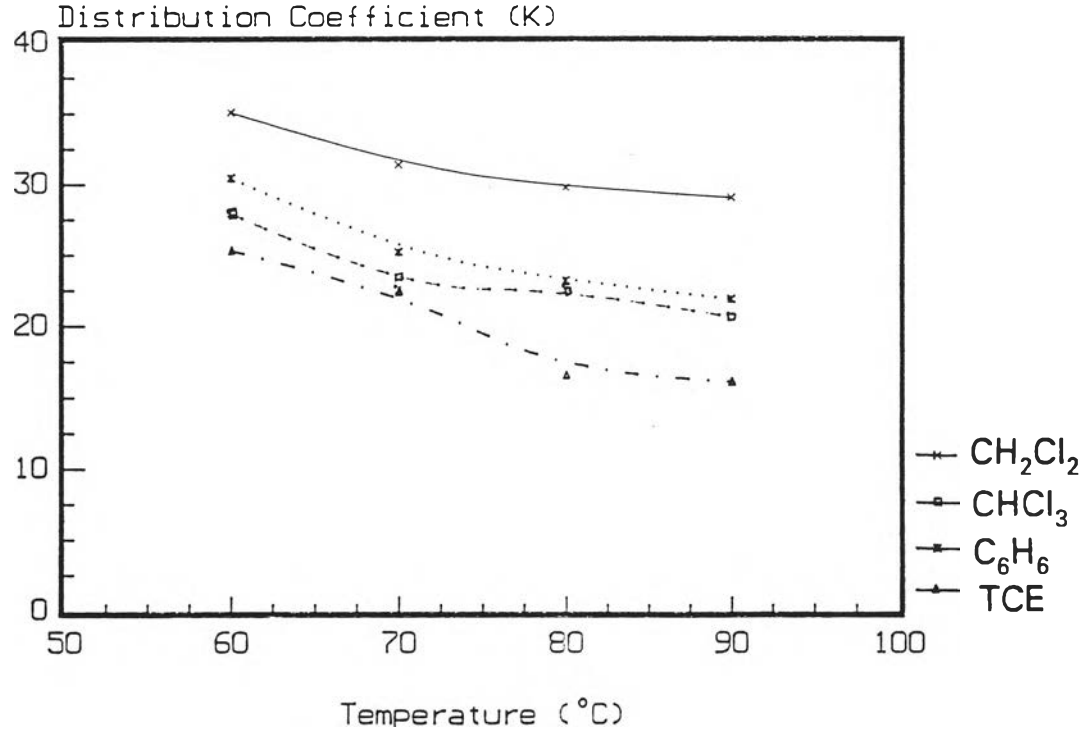


Figure 4.2 The effect of temperature on the distribution coefficient of each volatile organic compound (except 1,4-dioxane) with concentration of aqueous standard solution in higher level of ppm.

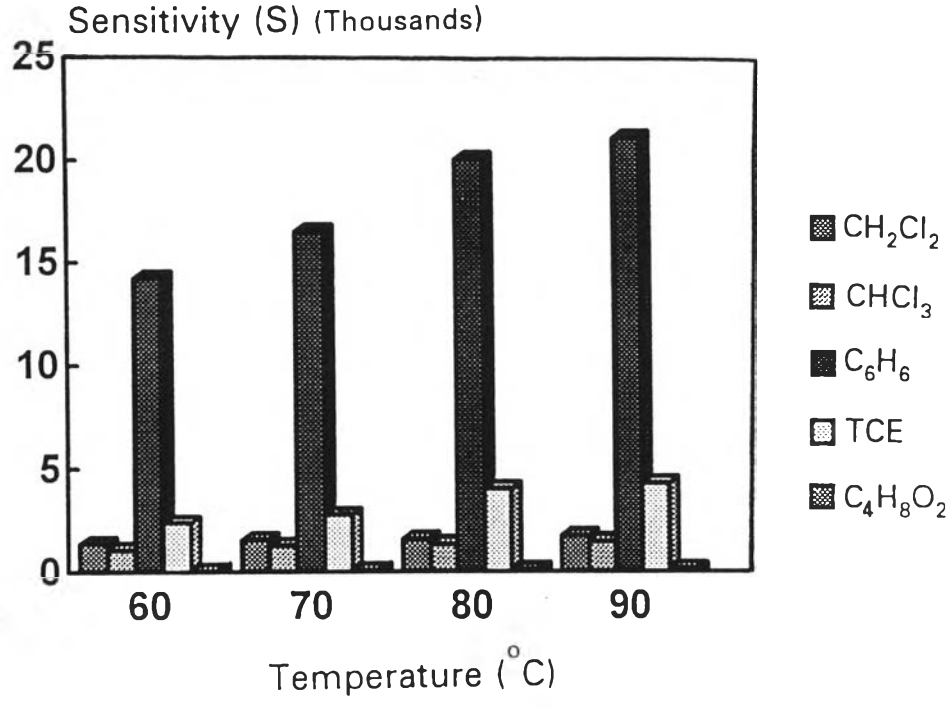


Figure 4.3 The effect of temperature on the sensitivity of each volatile organic compound with concentration of aqueous standard solution in lower level of ppm.

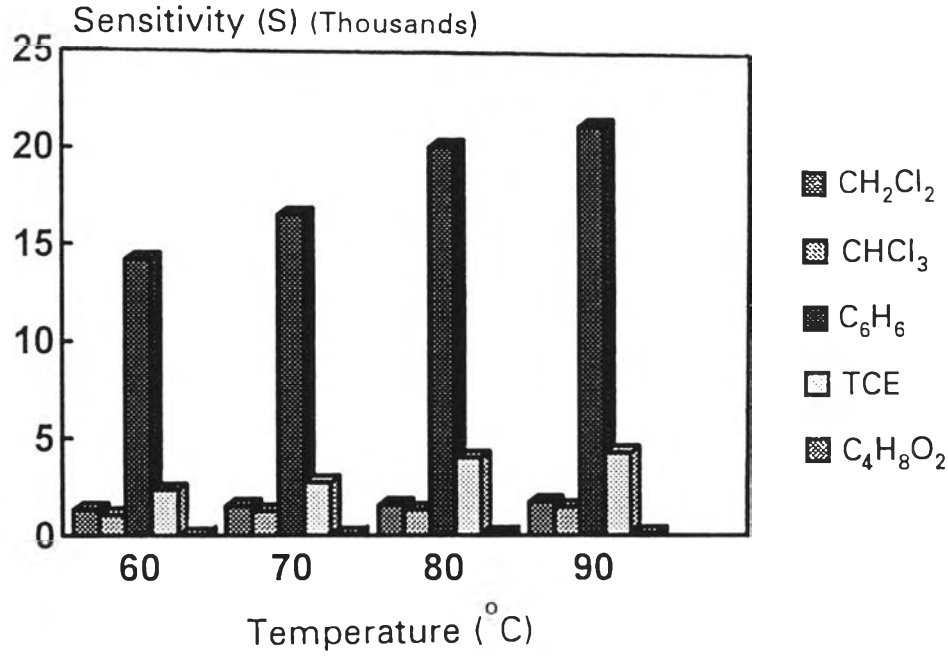


Figure 4.4 The effect of temperature on the sensitivity of each volatile organic compound with concentration of aqueous standard solution in higher level of ppm.

The Study of Equilibration Time

The results of the study of equilibration time for each volatile organic compound i.e., methylene chloride, chloroform, benzene, trichloroethylene and 1,4-dioxane obtained from the procedure in the experimental section 3.5.2 are given in Tables 4.5- 4.14. The graphs plotted the peak area (A) of each volatile organic compound against time are shown in the Figures 4.5-4.14. It is found that the equilibration time obtained from the study is 40 minutes for methylene chloride at both 4.21 and 12.63 ppm , 40 minutes for chloroform at both 4.24 and 12.73 ppm , 40 minutes for benzene at both 4.19 and 12.58 ppm, 40 minutes for trichloroethylene at both 4.21 and 12.63 ppm , and 50 minutes and 40 minutes for 1,4-dioxane at 99.26 and 297.79 ppm, respectively .Therefore , the time interval of 40 minutes is chosen as the optimum equilibration time for the studied compounds and it is used for the entire studies to ensure that the system is in the equilibrium.

Table 4.5 The result of the effect of equilibration time on the peak area of 4.21 ppm methylene chloride.

Time (min)	Peak Area	%RSD
20	5286	3.78
30	5395	1.62
40	6195	1.46
50	5904	0.45
60	6038	1.67
70	6154	0.43
80	6013	1.87
100	6054	1.27

Triplicate analyses

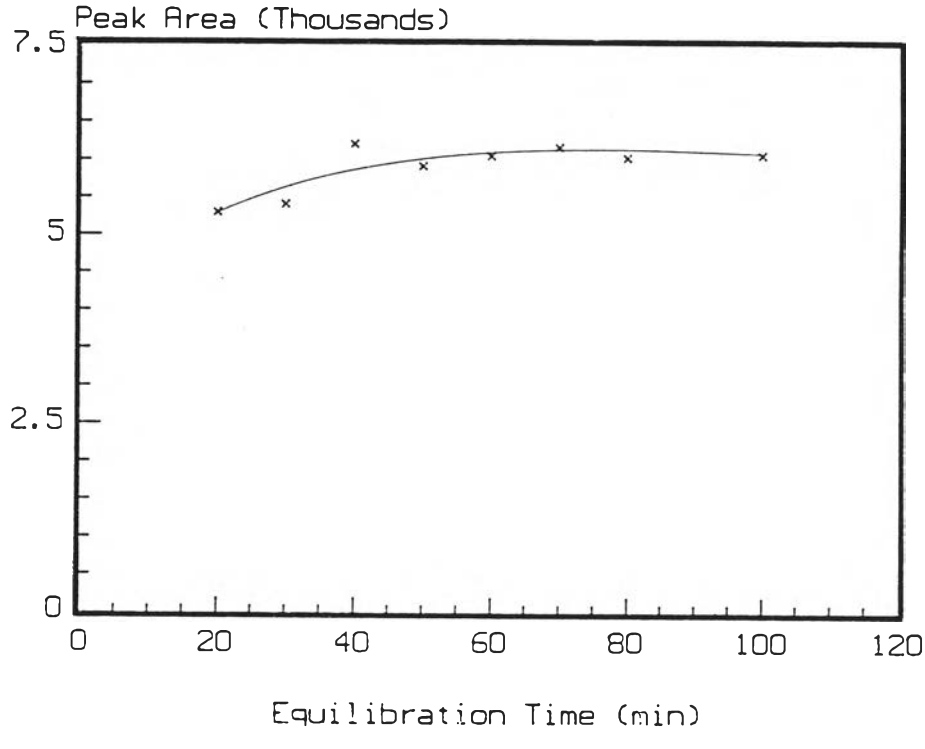


Figure 4.5 The effect of equilibration time on the peak area of 4.21ppm methylene chloride.

Table 4.6 The result of the effect of equilibration time on the peak area of 12.63 ppm methylene chloride.

Time (min)	Peak Area	%RSD
20	15021	5.31
30	15473	4.68
40	17558	3.53
50	15831	2.16
60	16174	6.99
70	16092	4.07
80	15832	6.45
100	15417	4.11

Triplicate analyses

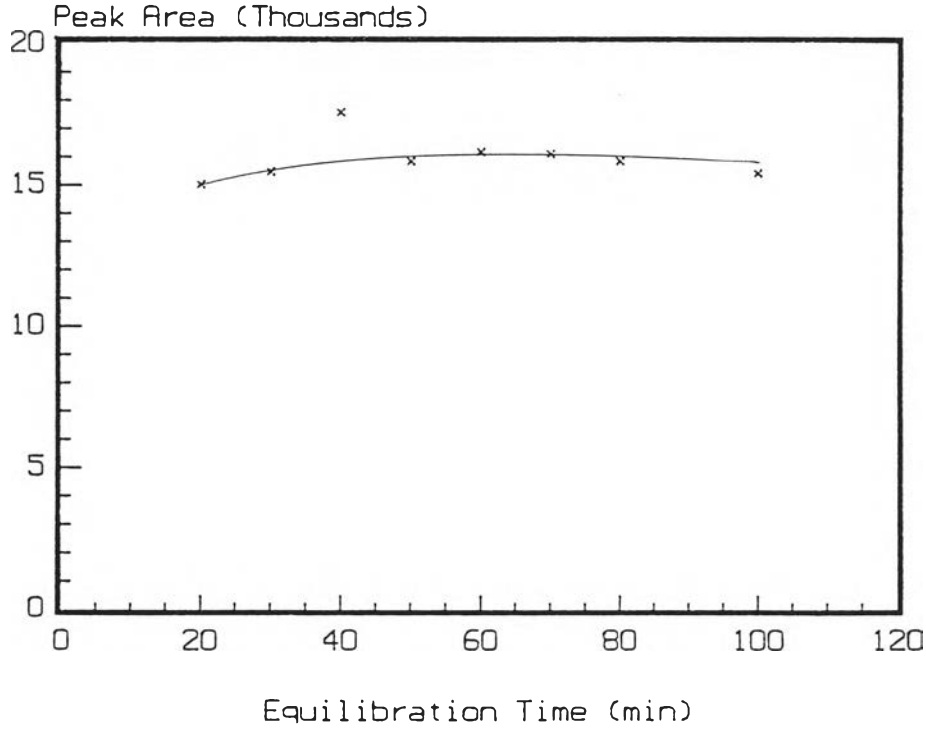


Figure 4.6 The effect of equilibration time on the peak area of 12.63 ppm methylene chloride.

Table 4.7 The result of the effect of equilibration time on the peak area of 4.24 ppm chloroform

Time (min)	Peak Area	%RSD
20	4072	2.95
30	4127	3.12
40	4542	0.97
50	4393	0.59
60	4413	3.19
70	4577	5.37
80	4378	1.57
100	4473	1.85

Triplicate analyses

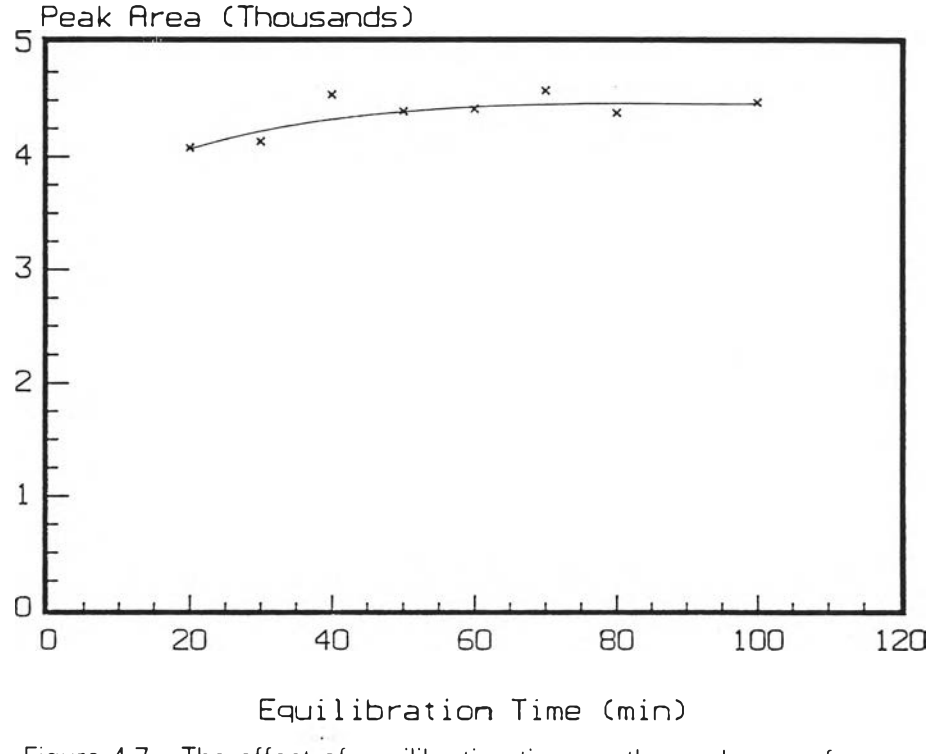


Figure 4.7 The effect of equilibration time on the peak area of 4.24ppm chloroform.

Table 4.8 The result of the effect of equilibration time on the peak area of 12.73 ppm chloroform.

Time (min)	Peak Area	%RSD
20	11046	7.50
30	11347	7.58
40	13854	3.71
50	12430	1.78
60	12698	6.71
70	12681	5.53
80	12199	6.80
100	12328	2.59

Triplicate analyses

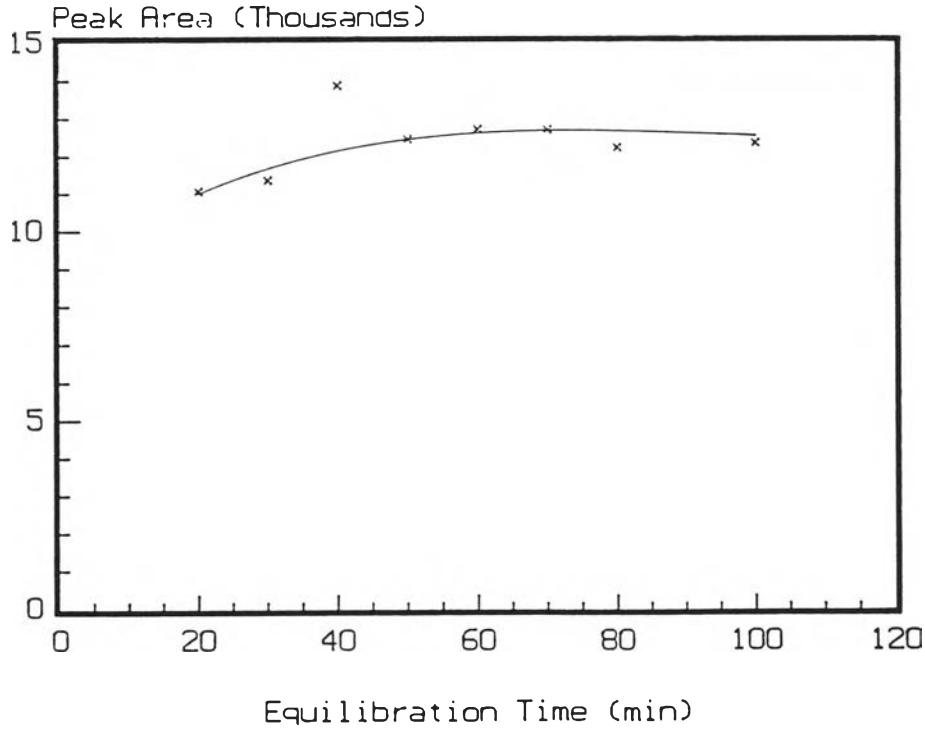


Figure 4.8 The effect of equilibration time on the peak area of 12.73 ppm chloroform.

Table 4.9 The result of the effect of equilibration time on the peak area of 4.19 ppm benzene.

Time (min)	Peak Area	%RSD
20	52909	1.21
30	56835	1.07
40	60211	0.60
50	57364	3.06
60	57254	2.96
70	58458	8.64
80	58198	1.64
100	59687	8.99

Triplicate analyses

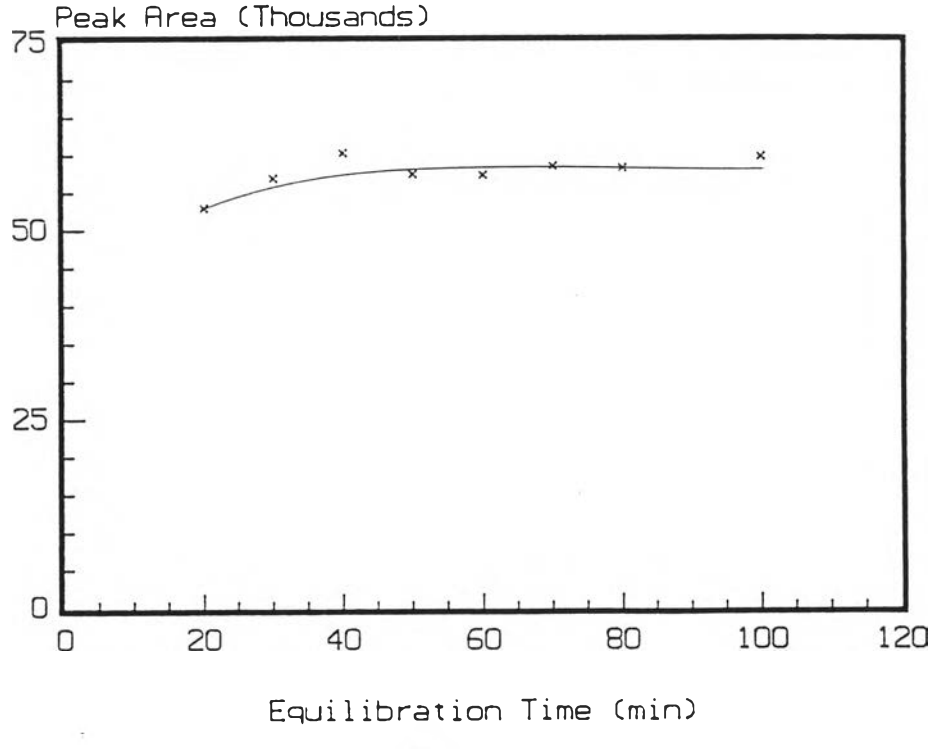


Figure 4.9 The effect of equilibration time on the peak area of 4.19 ppm benzene.

Table 4.10 The result of the effect of equilibration time on the peak area of 12.58 ppm benzene.

Time (min)	Peak Area	%RSD
20	126604	6.73
30	127365	8.52
40	140960	1.94
50	135414	2.02
60	131293	8.03
70	134651	2.94
80	131321	2.63
100	133871	4.99

Triplicate analyses

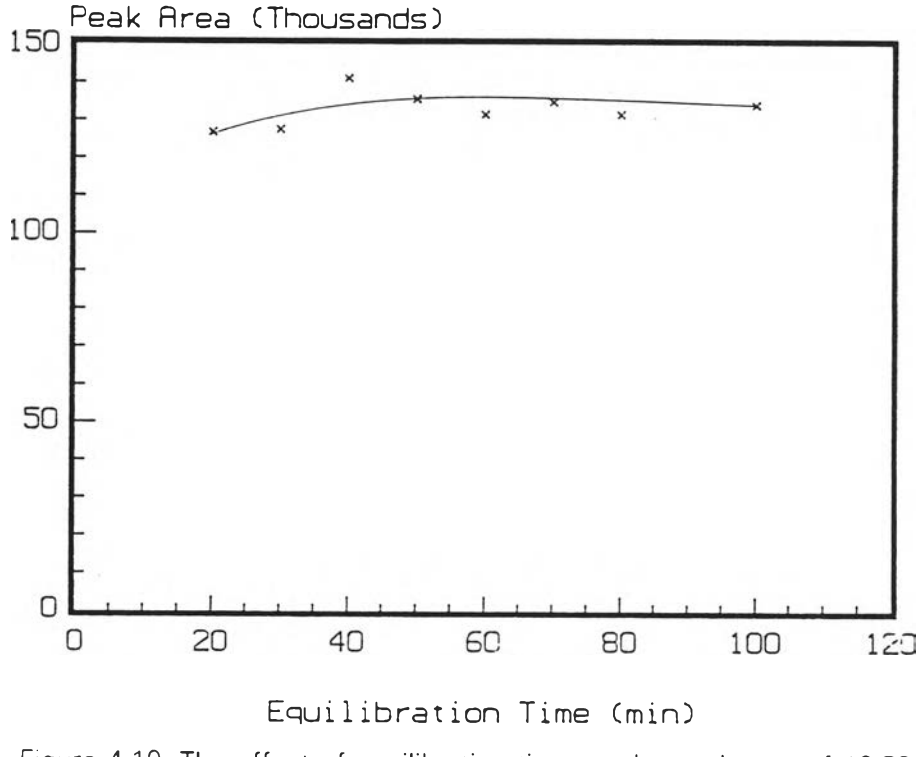


Figure 4.10 The effect of equilibration time on the peak area of 12.58 ppm benzene.

Table 4.11 The result of the effect of equilibration time on the peak area of 4.21 ppm trichloroethylene.

Time (min)	Peak Area	%RSD
20	8340	2.01
30	8573	7.55
40	8998	4.59
50	8716	5.25
60	8804	7.47
70	8841	0.11
80	8798	3.91
100	8777	6.26

Triplicate analyses

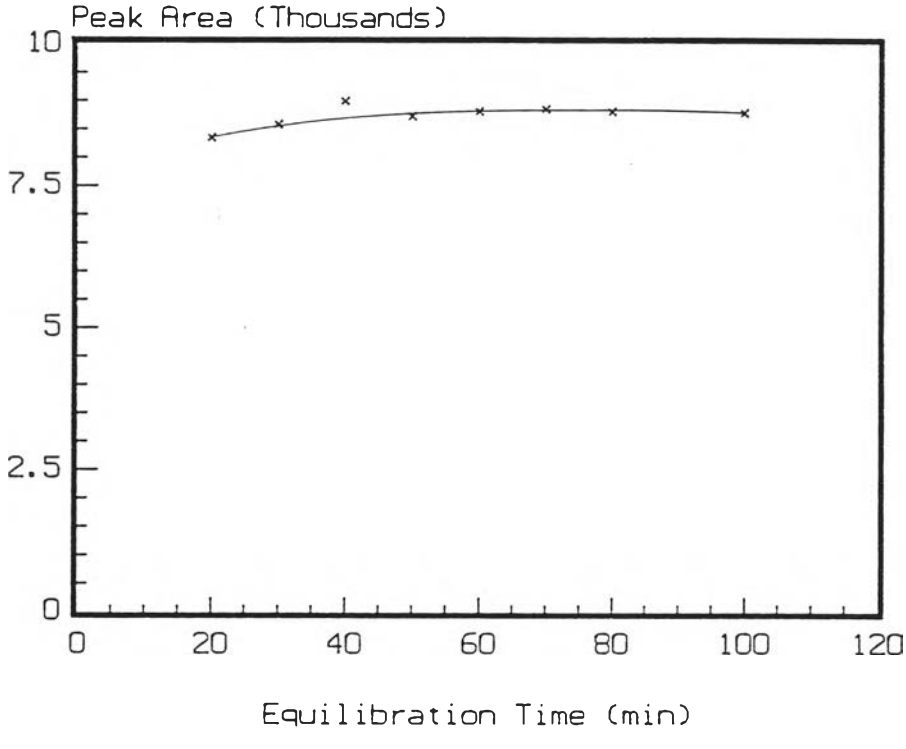


Figure 4.11 The effect of equilibration time on the peak area of 4.21ppm trichloroethylene.

Table 4.12 The result of the effect of equilibration time on the peak area of 12.63 ppm trichloroethylene.

Time (min)	Peak Area	%RSD
20	22912	3.12
30	24479	2.65
40	28388	2.40
50	26993	7.24
60	27234	4.51
70	27212	1.98
80	27613	6.21
100	27084	1.30

Triplicate analyses

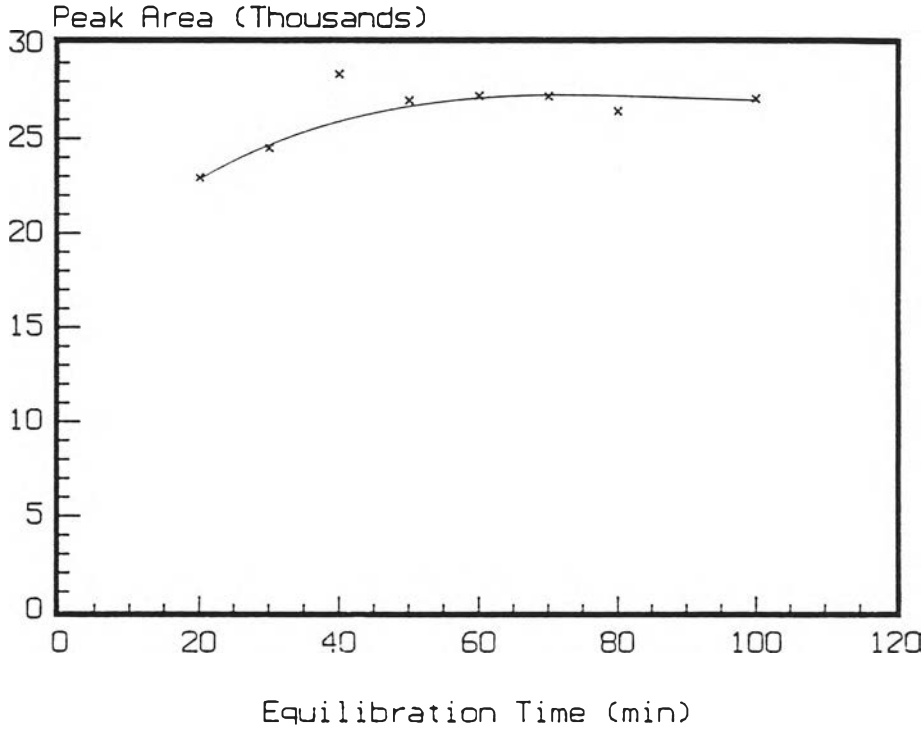


Figure 4.12 The effect of equilibration time on the peak area of 12.63 ppm trichloroethylene.

Table 4.13 The result of the effect of equilibration time on the peak area of 99.26 ppm 1,4-dioxane.

Time (min)	Peak Area	%RSD
20	5753	2.88
30	6101	7.88
40	6307	4.13
50	6557	5.07
60	6455	5.96
70	6473	2.05
80	6427	5.72
100	6366	0.04

Triplicate analyses

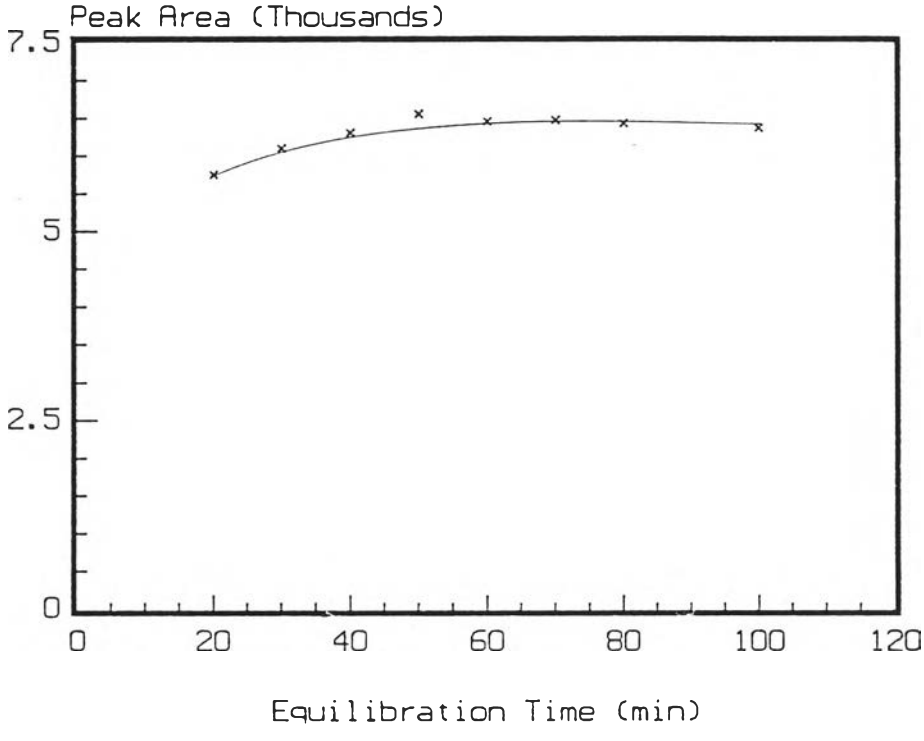


Figure 4.13 The effect of equilibration time on the peak area of 99.26 ppm 1,4-dioxane.

Table 4.14 The result of the effect of equilibration time on the peak area of 297.79 ppm 1,4-dioxane.

Time (min)	Peak Area	%RSD
20	22530	1.18
30	23359	2.63
40	24972	4.73
50	22685	5.06
60	22118	4.09
70	22897	0.55
80	22277	2.03
100	22891	2.90

Triplicate analyses

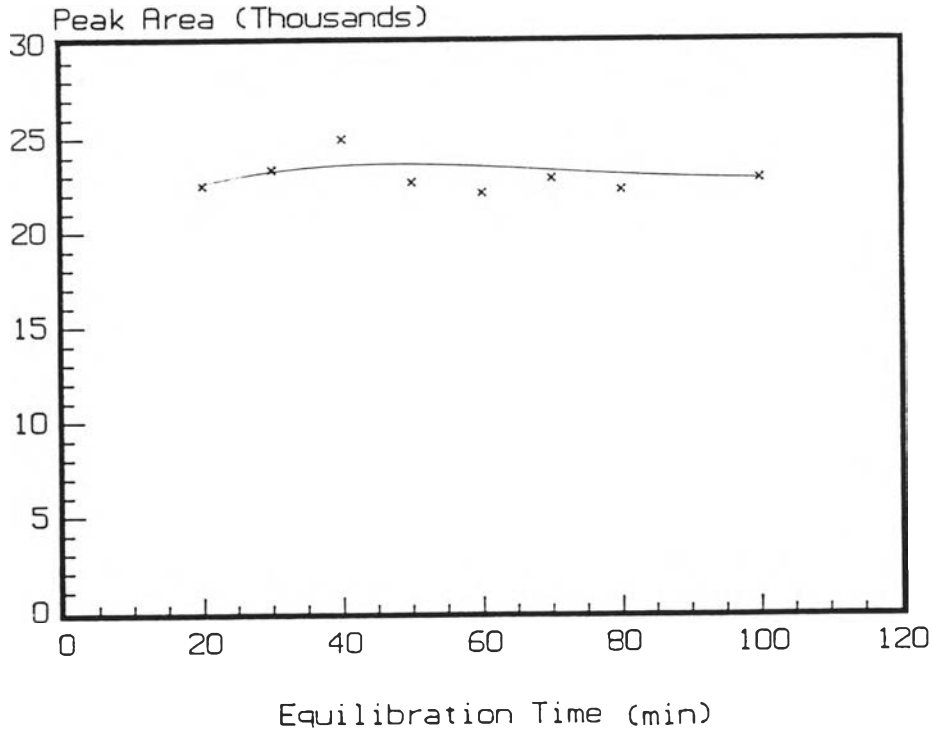


Figure 4.14 The effect of equilibration time on the peak area of 297.79 ppm 1,4-dioxane.

The Study of Liquid to Gas Phase Volume Ratio

The factor affecting the sensitivity of headspace analysis technique is also the liquid to gas phase volume ratio. The selection of the correct liquid to gas phase volume ratio will result in enhancing sensitivity and accuracy of the analysis. Therefore, its effect on the distribution coefficient and the sensitivity of each volatile compound are studied in order to determine the optimum liquid to gas phase volume ratio for the headspace analysis.

The results of liquid to gas phase volume ratio on the distribution coefficient and the sensitivity of each volatile organic compound i.e., methylene chloride, chloroform, benzene, trichloroethylene and 1,4-dioxane are presented in Tables 4.15-4.16 and 4.17-4.18, respectively. The graph plotted the distribution coefficient (K) and the sensitivity (S) of each volatile compound against the liquid to gas phase volume ratio are shown in Figures 4.15-4.16 and 4.17-4.18, respectively. It shows that the distribution coefficient of each volatile organic compound decreases when the value of liquid to gas phase volume ratio increases (except 1,4-dioxane is constant because of its is high solubility in water and high distribution coefficient). The decrease in the value of distribution coefficient will continue until the ratio of V_l/V_g reaches 1.0 (5.5) and it will remain constant up to higher phase ratios. Therefore, the sensitivity of each volatile compound is not much different in the liquid to gas phase volume ratios ranging from 5:5 to 8:2 as seen in Figures 4.17 and 4.18 and increasing the phase volume ratio of V_l/V_g tends to decrease the precision of the analysis as shown in Tables 4.17 and 4.18 due to the high concentration of each interested compound in gas phase (C_g) as shown in Tables 4.15 and 4.16. Hence, the liquid to gas phase volume ratio of 5:5 is chosen as a suitable ratio for a headspace analysis.

Table 4.15 The effect of liquid to gas phase ratio on the distribution coefficient and the equilibrium concentration of each volatile organic compound in gas phase with concentration of aqueous standard solution in lower level of ppm.

Compound	V_l / V_g	Peak Area	K	C_g(ppm)
Methylene chloride	2:8	4872	38.10	0.10
(4.21 ppm)	4:6	7058	26.57	0.15
	5:5	8125	22.39	0.18
	6:4	8576	21.49	0.19
	8:2	9027	20.80	0.20
Chloroform	2:8	6511	14.43	0.23
(4.24 ppm)	4:6	9602	12.18	0.31
	5:5	9924	11.11	0.35
	6:4	10157	11.10	0.36
	8:2	10721	10.91	0.38
Benzene	2:8	31051	37.90	0.10
(4.19 ppm)	4:6	49177	26.43	0.15
	5:5	56810	23.65	0.17
	6:4	57443	23.98	0.18
	8:2	64841	20.70	0.20
Trichloroethylene	2:8	51742	34.27	0.11
(4.21 ppm)	4:6	95317	23.26	0.17
	5:5	11375	20.05	0.20
	6:4	12088	19.38	0.21
	8:2	12283	18.87	0.22
1,4-Dioxane	2:8	7661	1236.75	0.08
(99.26 ppm)	4:6	7546	1238.25	0.08
	5:5	7603	1239.75	0.08
	6:4	7569	1240.08	0.08
	8:2	7550	1240.50	0.08

Triplicate analyses

Table 4.16 The effect of liquid to gas phase ratio on the distribution coefficient and the equilibrium concentration of each volatile organic compound in gas phase with concentration of aqueous standard solution in higher level of ppm.

Compound	V_l / V_g	Peak Area	K	C_g(ppm)
Methylene chloride	2:8	11446	46.52	0.25
(12.63 ppm)	4:6	19723	27.87	0.43
	5:5	21347	26.46	0.46
	6:4	22277	25.64	0.48
	8:2	24005	24.04	0.52
Chloroform	2:8	11543	26.31	0.42
(12.73 ppm)	4:6	13870	23.96	0.50
	5:5	15007	22.57	0.54
	6:4	15598	21.66	0.57
	8:2	16990	20.28	0.62
Benzene	2:8	76938	50.70	0.23
(12.58 ppm)	4:6	143593	27.76	0.43
	5:5	160081	25.77	0.47
	6:4	167304	25.00	0.49
	8:2	181402	23.05	0.54
Trichloroethylene	2:8	12620	53.41	0.22
(12.63 ppm)	4:6	32657	22.33	0.53
	5:5	35569	21.16	0.57
	6:4	39664	19.06	0.64
	8:2	42106	18.52	0.68
1,4-Dioxane	2:8	26871	1141.35	0.26
(297.79 ppm)	4:6	27720	1101.43	0.27
	5:5	27472	1101.93	0.27
	6:4	27353	1102.26	0.27
	8:2	27884	1102.68	0.27

Triplicate analyses

Table 4.17 The effect of liquid to gas phase ratio on the sensitivity of each volatile organic compound with concentration of aqueous standard solution in lower level of ppm.

Compound	V_l / V_g	Sensitivity	%RSD
Methylene chloride	2:8	1157	1.52
(4.21 ppm)	4:6	1676	2.28
	5:5	1929	0.82
	6:4	1966	1.29
	8:2	2145	2.13
Chloroform	2:8	2053	2.21
(4.24 ppm)	4:6	2062	2.75
	5:5	2113	0.96
	6:4	2198	2.80
	8:2	2240	3.60
Benzene	2:8	7401	2.03
(4.19 ppm)	4:6	11720	2.67
	5:5	13541	0.46
	6:4	13689	1.04
	8:2	14457	1.04
Trichloroethylene	2:8	1229	2.00
(4.21 ppm)	4:6	2264	0.64
	5:5	2702	3.51
	6:4	2734	3.56
	8:2	2818	3.68
1,4-Dioxane	2:8	77	0.44
(99.26 ppm)	4:6	76	0.83
	5:5	77	0.51
	6:4	76	0.75
	8:2	76	1.36

Triplicate analyses

Table 4.18 The effect of liquid to gas phase volume ratio on the sensitivity of each volatile organic compound with coefficient of aqueous solution in higher level of ppm.

Compound	V_l / V_g	Sensitivity (S)	%RSD
Methylene chloride	2:8	906	3.74
(12.63 ppm)	4:6	1561	0.97
	5:5	1863	1.63
	6:4	1900	1.49
	8:2	1929	1.89
Chloroform	2:8	907	4.54
(12.73 ppm)	4:6	1090	2.62
	5:5	1179	0.80
	6:4	1203	2.28
	8:2	1335	1.28
Benzene	2:8	6112	4.03
(12.58 ppm)	4:6	11405	2.79
	5:5	12716	2.66
	6:4	13290	2.83
	8:2	14409	2.41
Trichloroethylene	2:8	999	6.86
(12.63 ppm)	4:6	2585	3.56
	5:5	2816	1.29
	6:4	3140	3.16
	8:2	3378	0.86
1,4-Dioxane	2:8	90	1.53
(297.79 ppm)	4:6	93	1.81
	5:5	92	0.32
	6:4	92	1.32
	8:2	94	1.78

Triplicate Analyses

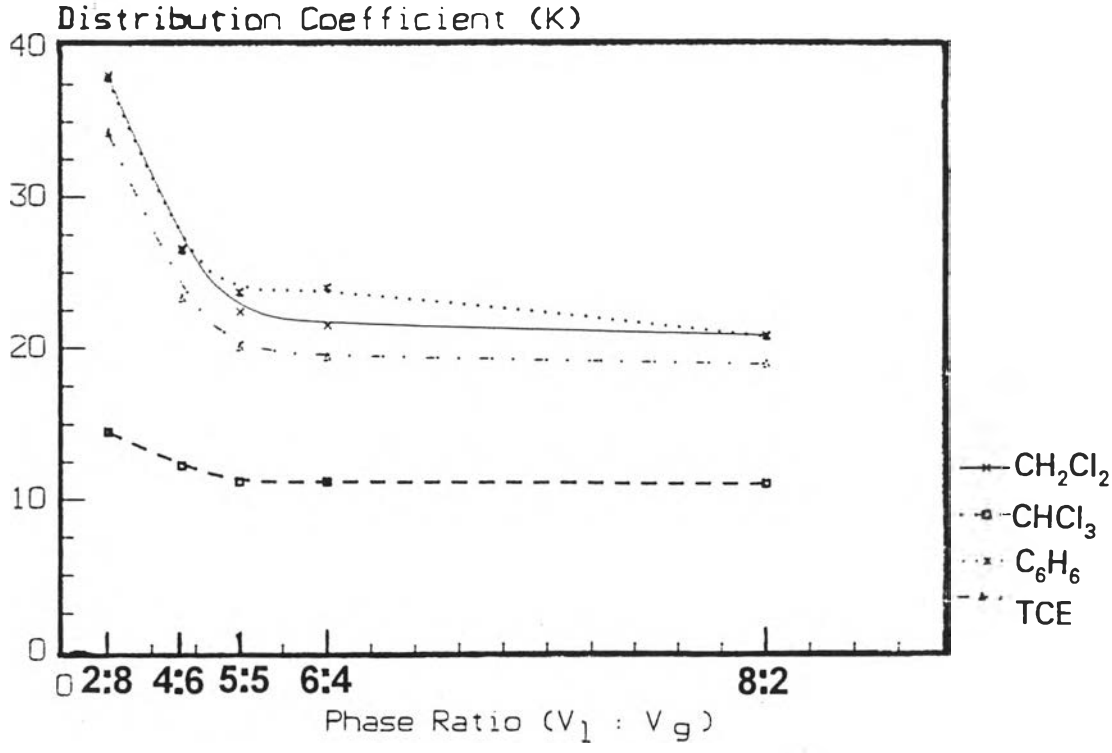


Figure 4.15 The distribution coefficient of each volatile organic compound (except 1,4-dioxane) with concentration of aqueous standard solution in lower level of versus liquid to gas phase volume ratio.

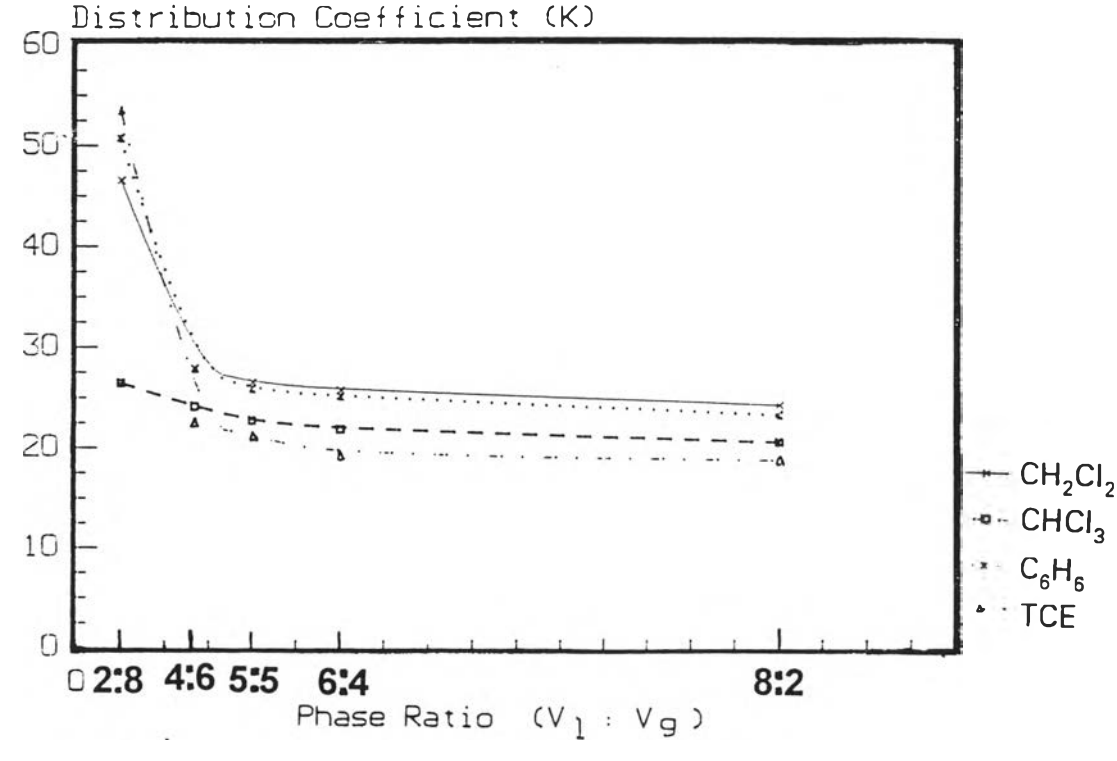


Figure 4.16 The distribution coefficient of each volatile organic compound (except 1,4-dioxane) with concentration of aqueous standard solution in higher level of versus liquid to gas phsae volume volume ratio.

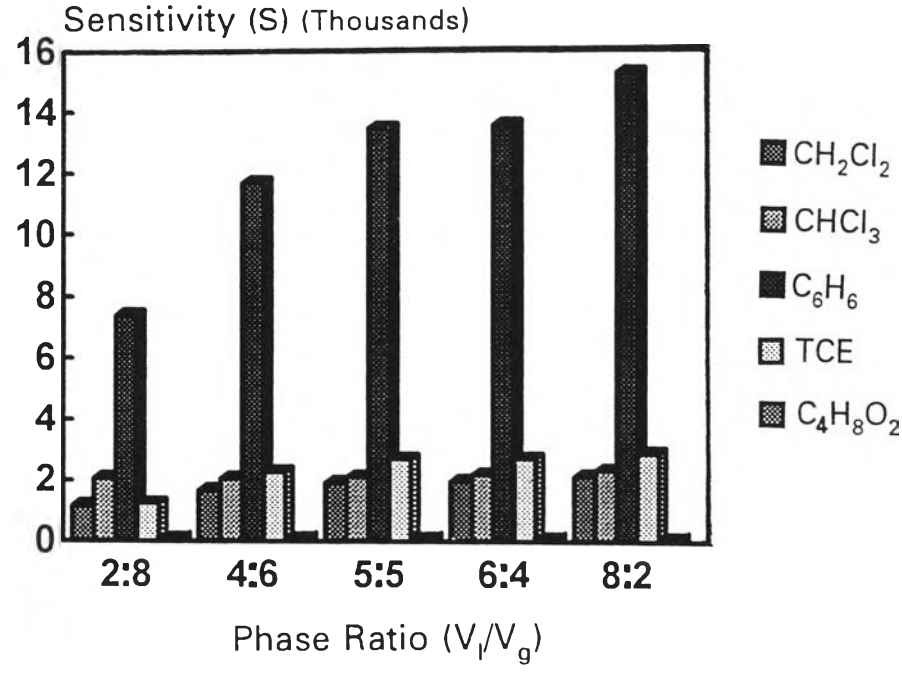


Figure 4.17 The effect of liquid to gas phase volume ratio on the sensitivity of each volatile organic compound with concentration of aqueous standard solution in lower level .

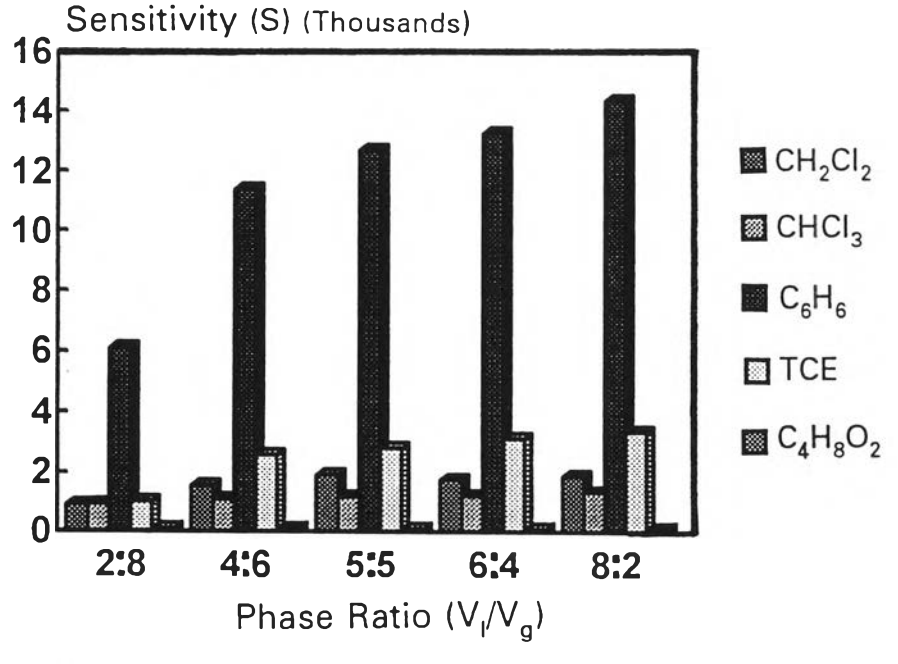


Figure 4.18 The effect of liquid to gas phase volume ratio on the sensitivity of each volatile organic compound with concentration of aqueous standard solution in higher level .

The study of Salting Out Effect

The results of adding 1.00g of anhydrous sodium sulfate on the distribution coefficient, the sensitivity and the percent recovery of each volatile compound i.e., methylene chloride, chloroform, benzene, trichloroethylene and 1,4-dioxane in mixture compound solution at two concentration levels using FID and ECD (except two volatile compounds that low sensitive using ECD i.e., benzene and 1.4-dioxane) as the detector as shown in Table 4.19-4.22 respectively and the graphs plotted the distribution coefficient, the sensitivity and the percent recovery against salt used are shown in Figures 4.19-4.22 and 4.23-4.26 and 4.27-4.30, respectively. It is found that the results of adding salt into the solutions give the lower distribution coefficient and the higher sensitivity and percent recovery for each volatile compound than not used salt. Therefore, the anhydrous sodium sulfate can be used to increase the sensitivity and the percent recovery of each volatile compound in both mixture compounds solution.

Table 4.19 The results of salting out effect on the percent recovery of each volatile organic compound in mixture solution with concentration of aqueous standard in lower level of ppm using FID as a detector.

Compound	Salt	K	S	%E	%RSD
Methylene chloride	No salt	28.00	1601	34.50	0.78
(4.21 ppm.)	Na_2SO_4	16.04	2717	58.72	0.55
Chloroform	No salt	22.11	1219	43.16	1.45
(4.24 ppm.)	Na_2SO_4	8.00	3070	71.20	3.39
Benzene	No salt	24.36	12791	40.48	1.41
(4.19 ppm.)	Na_2SO_4	14.57	21349	65.82	0.50
Trichloroethylene	No salt	20.51	2590	46.50	4.95
(4.21 ppm.)	Na_2SO_4	14.92	3656	62.82	2.66
1,4-Dioxane	No salt	487.26	61	0.68	1.70
(99.26 ppm.)	Na_2SO_4	154.99	207	2.16	2.89

Triplicate analyses

Table 4.20 The results of salting out effect on the percent recovery of each volatile organic compound in mixture solution with concentration of aqueous standard in higher level of ppm using FID as a detector.

Compound	Salt	K	S	%E	%RSD
Methylene chloride	No salt	33.18	1353	29.26	0.25
(12.63 ppm.)	Na_2SO_4	19.95	2200	47.74	3.06
Chloroform	No salt	27.21	979	35.44	7.41
(12.73 ppm.)	Na_2SO_4	14.85	1731	63.06	3.81
Benzene	No salt	32.53	10021	30.58	5.16
(12.58 ppm.)	Na_2SO_4	18.92	17055	51.46	5.81
Trichloroethylene	No salt	27.53	2137	35.06	5.71
(12.63 ppm.)	Na_2SO_4	17.59	3356	53.80	7.45
1,4-Dioxane	No salt	1385.77	73	0.72	1.04
(297.79 ppm.)	Na_2SO_4	484.09	213	2.06	1.56

Triplicate analyses

Table 4.21 The results of salting out effect on the percent recovery of each volatile organic compound in mixture solution with concentration of aqueous standard in lower level of ppb using ECD as a detector.

Compound	Salt	K	S	%E	%RSD
Methylene chloride	No salt	39.83	146216	34.48	1.76
(20.21 ppb)	Na_2SO_4	19.25	242765	62.00	4.28
Chloroform	No salt	20.66	2271607	46.32	3.50
(20.36 ppb)	Na_2SO_4	8.43	3997225	76.28	1.40
Trichloroethylene	No salt	12.94	8826901	51.80	5.53
(20.20 ppb)	Na_2SO_4	7.56	12962150	75.84	2.58

Triplicate analyses

Table 4.22 The results of salting out effect on the percent recovery of each volatile organic compound in mixture solution with concentration of aqueous standard in higher level of ppb using ECD as a detector.

Compound	Salt	K	S	%E	%RSD
Methylene chloride	No salt	21.99	200348	43.50	3.38
(200.03 ppb)	Na_2SO_4	11.49	352362	80.08	0.62
Chloroform	No salt	17.25	1988100	44.80	3.60
(201.51 ppb)	Na_2SO_4	10.50	3055810	73.94	1.13
Trichloroethylene	No salt	16.06	6264947	46.60	4.50
(200.00 ppb)	Na_2SO_4	10.58	9165889	70.34	1.57

Triplicate analyses

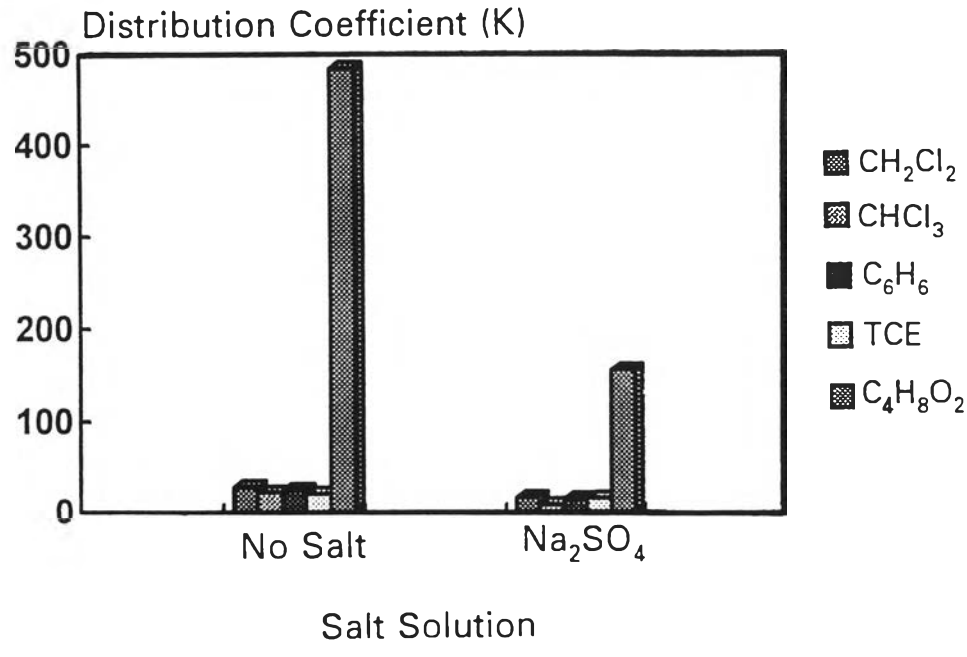


Figure 4.19 The effect of salting out on the distribution coefficient of each volatile compound with concentration of aqueous standard solution in lower level using FID as a detector.

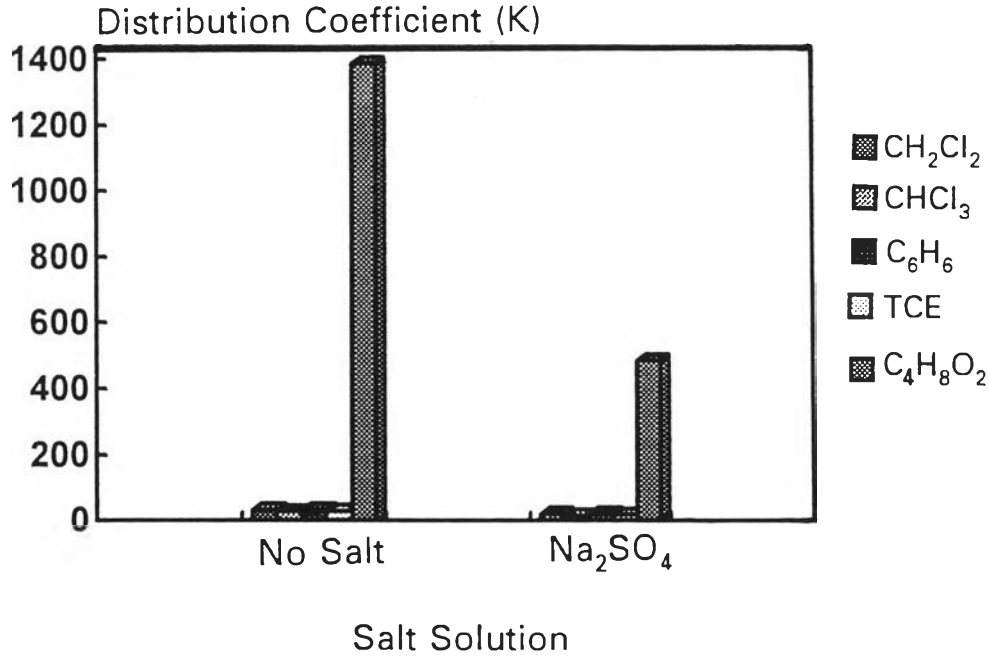


Figure 4.20 The effect of salting out on the distribution coefficient of each volatile compound with concentration of aqueous standard solution in higher level using FID as a detector.

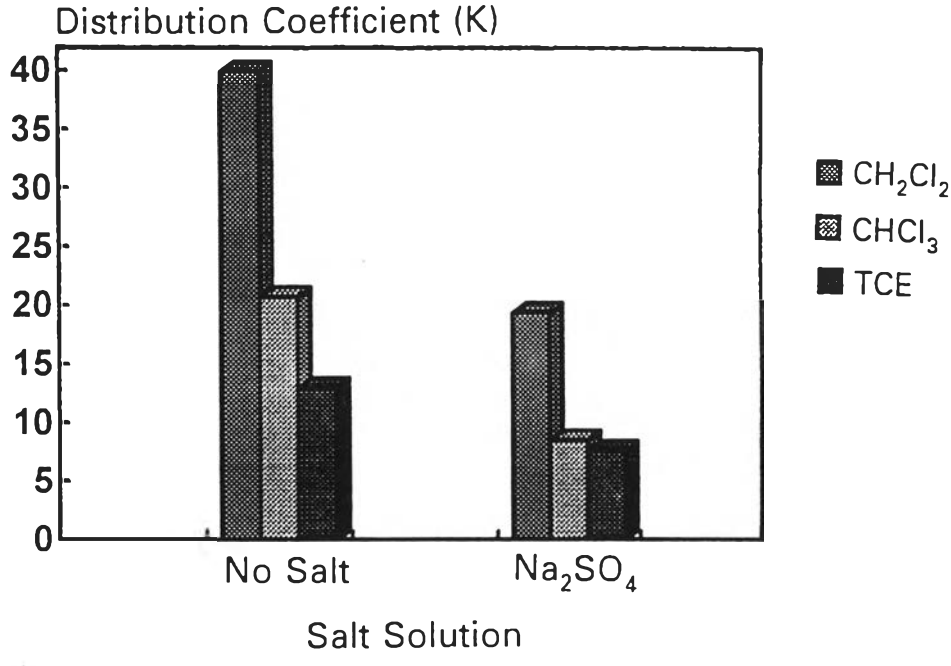


Figure 4.21 The effect of salting out on the distribution coefficient of each volatile compound with concentration of aqueous standard solution in lower level using ECD as a detector.

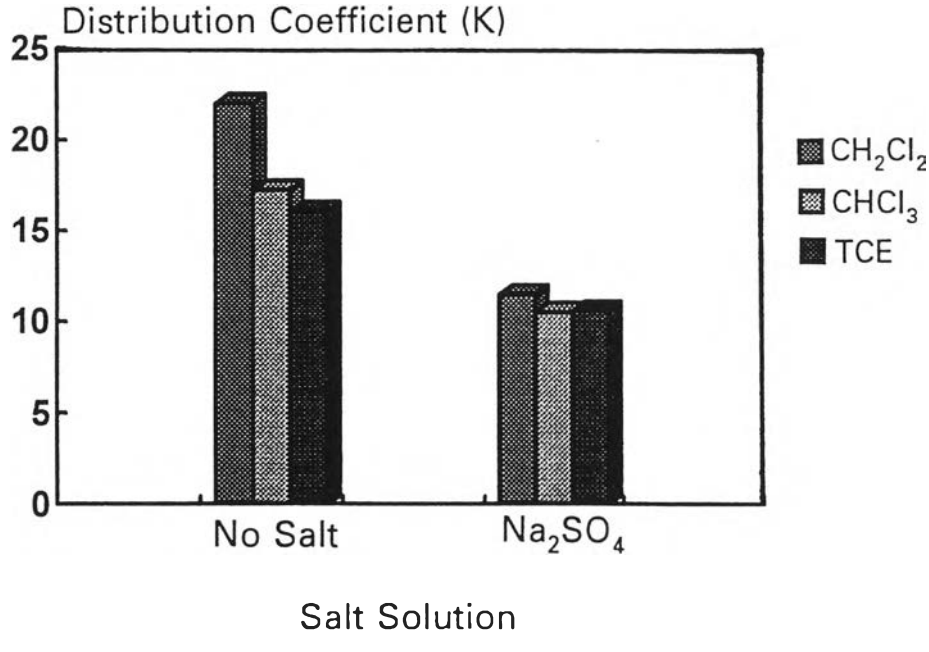


Figure 4.22 The effect of salting out on the distribution coefficient of each volatile compound with concentration of aqueous standard solution in higher level using ECD as a detector.

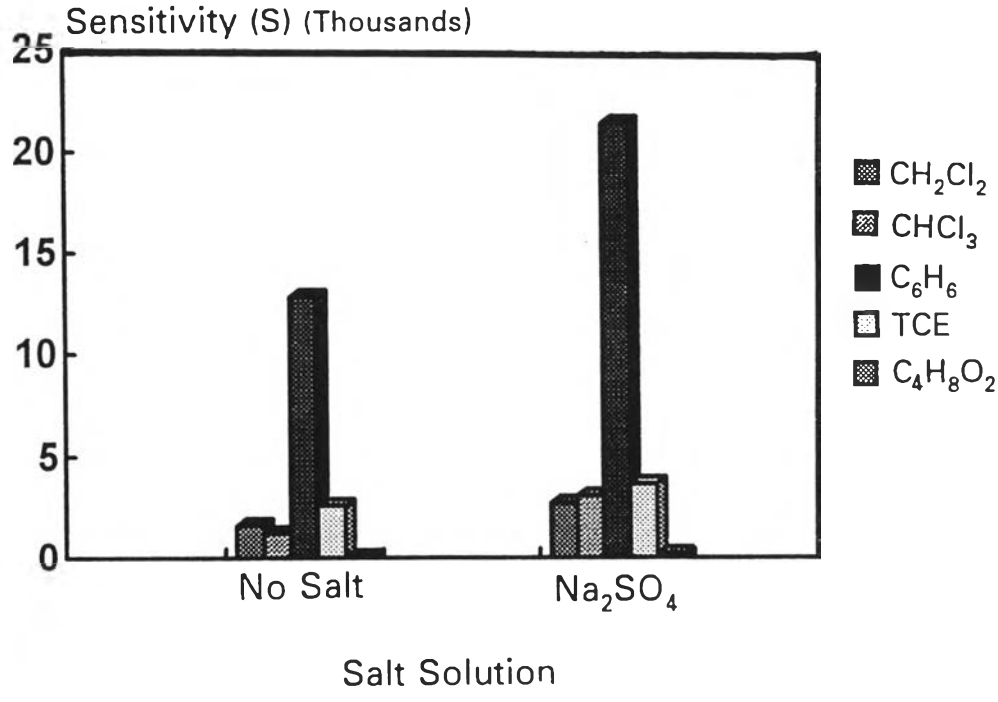


Figure 4.23 The effect of salting out on the sensitivity of each volatile organic compound with concentration of aqueous standard solution in lower level using FID as a detector.

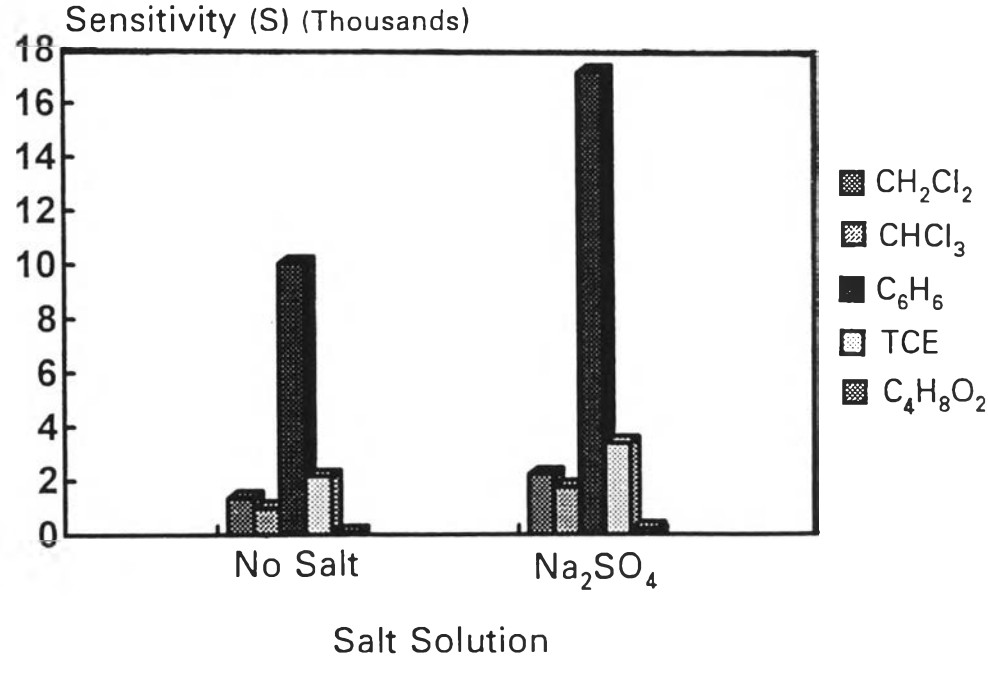


Figure 4.24 The effect of salting out on the sensitivity of each volatile organic compound with concentration of aqueous standard solution in higher level using FID as a detector.

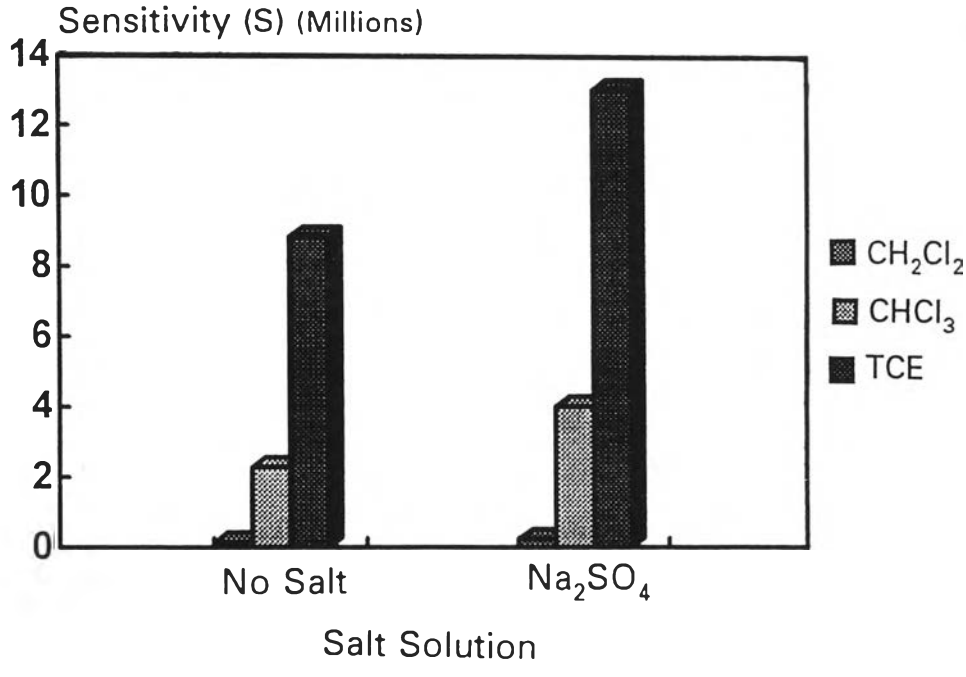


Figure 4.25 The effect of salting out on the sensitivity of each volatile organic compound with concentration of aqueous standard solution in lower level using ECD as a detector.

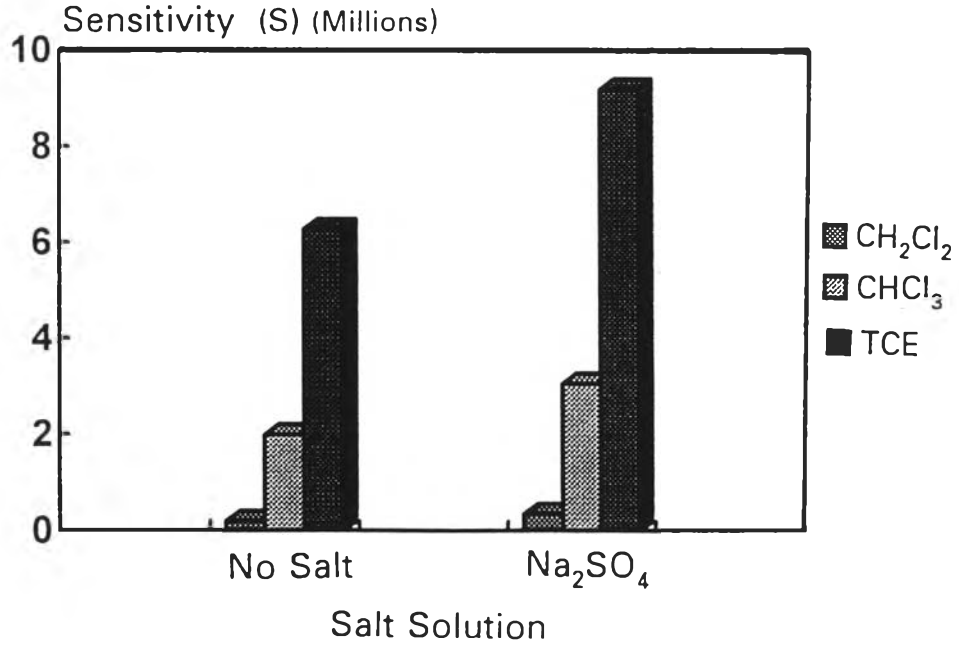


Figure 4.26 The effect of salting out on the sensitivity of each volatile organic compound with concentration of aqueous standard solution in higher level using ECD as a detector.

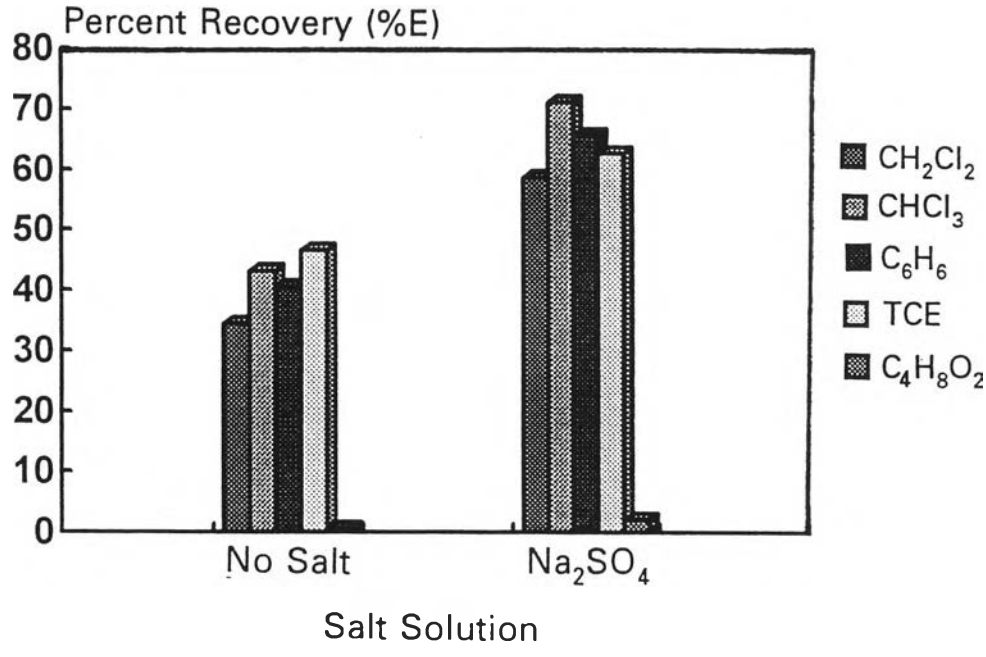


Figure 4.27 The effect of salting out on the percent recovery of each volatile organic compound with concentration of aqueous standard solution in lower level using FID as a detector.

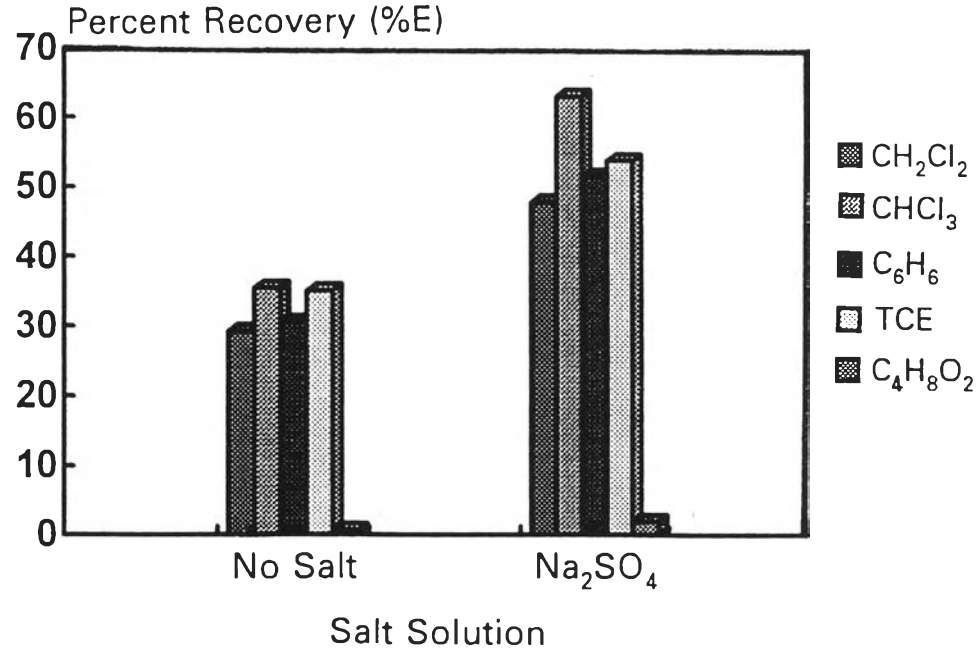


Figure 4.28 The effect of salting out on the percent recovery of each volatile organic compound with concentration of aqueous standard solution in higher level using FID as a detector.

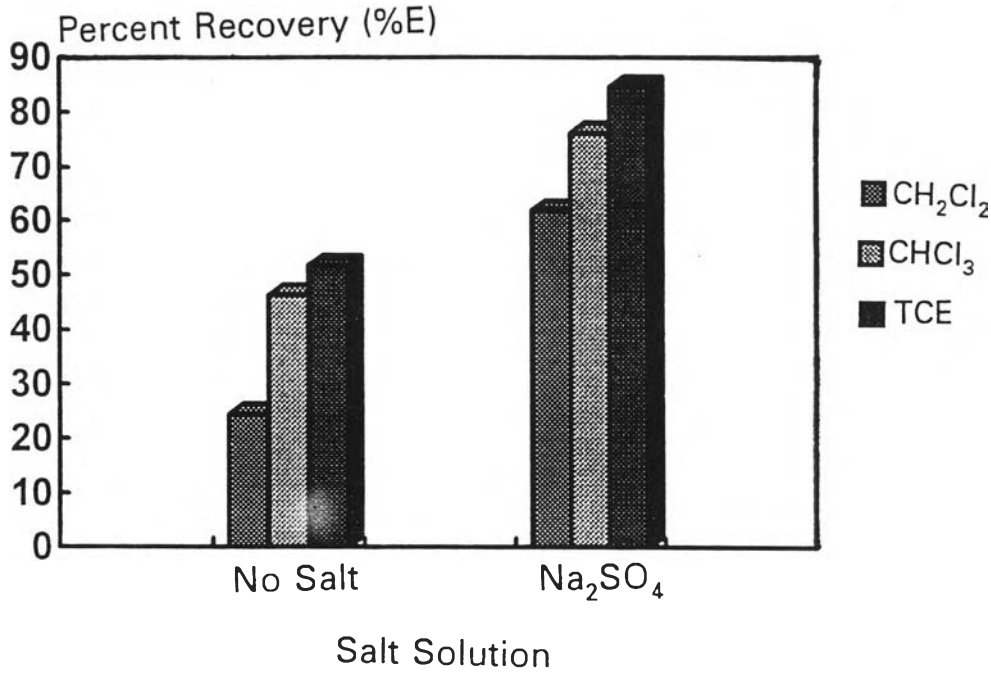


Figure 4.29 The effect of salting out on the percent recovery of each volatile organic compound with concentration of aqueous standard solution in lower level using ECD as a detector.

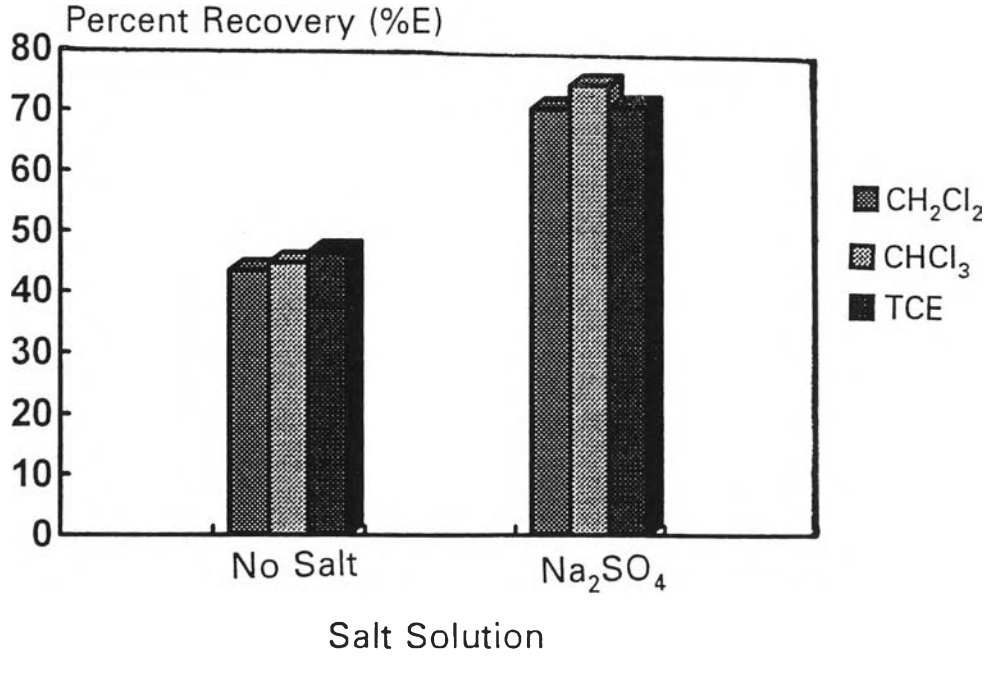


Figure 4.30 The effect of salting out on the percent recovery of each volatile organic compound with concentration of aqueous standard solution in higher level using ECD as a detector.

The result in Table 4.23 shows that the percent recovery of methylene chloride ranges from 47.74 to 70.08 with ±0.55-4.28%RSD, chloroform ranges from 63.06 to 76.28 with ± 1.13 - 3.81 %RSD, benzene ranges from 51.46 to 65.82 with ±0.50 - 5.81 %RSD, trichloroethylene ranges from 53.80 to 75.84 with ±1.57 - 7.45 %RSD and 1,4-dioxane ranges from 2.06 to 2.16 with ±1.56 - 2.89 %RSD. It can be summarized that the percent recovery of each volatile compound in solution at the two different concentrations is moderately different and the percent recovery of each volatile chlorinated hydrocarbon i.e., methylene chloride, chloroform and trichloroethylene using FID as a detector is different from the percent recovery of each volatile chlorinated hydrocarbon using ECD as a detector. This indicates that the percent recovery of each volatile organic compound is depend on the concentration of the compound and the detector of gas chromatagraph.

The sensitivity of each volatile chlorinated hydrocarbon i.e., methylene chloride, chloroform and trichloroethylene at two concentration levels in mixture compound solution with anhydrous sodium sulfate is shown in Table 4.24. It shows that the sensitivity of each compound at two concentration levels is slighted different. Hence, the concentration of each interested compound has not any effect on the sensitivity of each compounds. However, the sensitivity of each interested compound i.e., methylene chlorides, chloroform and trichloroethylene studied using ECD as a detector of gas chromatograph is higher than the one using FID as a detector but the sensitivity of each volatile compound i.e., benzene and 1,4-dioxane is lower than the one using FID as a detector. Therefore, FID is chosen as the detector of gas chromatograph for this headspace technique and using ECD as a detector when the interested compound is a chlorinated hydrocarbon.

Table 4.23 The percent recovery of each volatile compound at two concentration levels in mixture solution with anhydrous sodium sulfate.

Compound	The percent recovery (%E) (%RSD)			
	FID		ECD	
	Concentration level		Concentration level	
	lower	higher	lower	higher
Methylene chloride	58.72 (± 0.55)	47.74 (± 3.06)	62.00 (± 4.28)	70.08 (± 0.62)
Chloroform	71.60 (± 3.39)	63.06 (± 3.81)	76.28 (± 1.40)	73.94 (± 1.13)
Benzene	65.82 (± 0.50)	51.46 (± 5.81)	ND	ND
Trichloroethylene	62.82 (± 2.66)	53.80 (± 7.45)	75.84 (± 2.58)	70.34 (± 1.57)
1,4-Dioxane	2.16 (± 2.89)	2.06 (± 1.56)	ND	ND

Triplicate analyses

Table 4.24 The sensitivity of each volatile compound at two concentration levels in mixture solution with anhydrous sodium sulfate.

Compound	Sensitivity (S) (%RSD)			
	FID		ECD	
	Concentration level		Concentration level	
	lower	higher	lower	higher
Methylene chloride	2717 (± 0.55)	2200 (± 3.06)	242765 (± 4.28)	176221 (± 0.62)
Chloroform	3070 (± 3.39)	1731 (± 3.81)	3997225 (± 1.40)	3055810 (± 1.13)
Benzene	21349 (± 0.50)	17055 (± 5.81)	ND	ND
Trichloroethylene	3656 (± 2.66)	3359 (± 7.45)	12962150 (± 2.58)	9165889 (± 1.57)
1,4-Dioxane	207 (± 2.89)	213 (± 1.56)	ND	ND

Triplicate analyses

Table 4.25 The optimum headspace analysis condition used in the investigation of the minimum detectable level, the precision, the accuracy and analyses of the real drug samples using FID and ECD as a detector.

Headspace parameter	Headspace condition
Equilibration Time	40 minutes
Bath Temperature	70 °C
Valve/Loop Temperature	100 °C
Sampling Interval	40 minutes
Valve Timing	min:sec
Probe (Start)	0:01
Pressurize (Start)	0:03
Pressurize (Stop)	0:23
Vent (Start)	0:24
Vent (Stop)	0:29
Inject (Start)	0:30
Inject (Stop)	0:50
Probe (Standby)	0:51
Carrier Pressure (He)	0.3 Bar
Aux Pressure (N_2)	1.5 Bar
Air Pressure (Air)	3.7 Bar
Liquid to gas phase volume ratio	5:5 in 10 mL
Injection volume	1.00 mL
Salt used	1.00 g of anhydrous Na_2SO_4

Triplicate analyses

The Precision of Headspace Analysis Technique

The results of the precision of headspace analysis technique of each volatile compound using FID and ECD as a detector are shown in Tables 4.26 and 4.27, respectively . The average concentration and the percent relative standard derivation of methylene chloride , chloroform , benzene , trichloroethylene and 1,4-dioxane using FID as a detector are 20712 ± 1.83 , 14933± 2.48 , 161689 ± 3.04 , 40499 ± 3.00 and 31158 ± 2.02 , respectively . The average peak area and the percent relative standard derivation of methylene chloride , chloroform and trichloroethylene using ECD as a detector are 3888 ±1.97 , 105057 ± 2.43 and 291748 ± 2.24 , respectively.

Table 4.26 The precision of automated headspace analysis technique by using FID as a detector.

Compound	1	2	3	4	5	6	7	8	9	10	Average Concentration (ppm)	%RSD
Methylene chloride	4.22	4.19	4.02	4.13	4.25	4.24	4.18	4.11	4.25	4.12	4.17	±1.80
Chloroform	4.20	4.30	3.92	4.07	4.41	4.31	4.23	4.14	4.32	4.17	4.21	±2.48
Benzene	4.32	4.21	4.02	4.09	4.37	4.30	4.13	4.05	4.30	4.11	4.19	±3.04
Trichloroethylene	4.38	4.36	4.12	4.07	4.17	4.34	4.15	4.06	4.30	4.15	4.21	±3.00
1,4-Dioxane	95.95	97.17	97.56	99.94	101.15	98.58	101.27	99.18	102.36	99.44	99.26	±2.02

Table 4.27 The precision of automated headspace analysis technique by using ECD as a detector.

Compound	1	2	3	4	5	6	7	8	9	10	Average Concentration (ppb)	%RSD
Methylene chloride	20.74	20.13	20.14	20.10	19.94	19.64	20.27	20.77	19.94	20.76	20.23	±1.97
Chloroform	20.78	21.06	20.79	20.42	20.27	20.76	20.55	20.01	19.78	19.58	20.40	±2.11
Trichloroethylene	20.84	20.22	20.53	19.69	20.82	20.60	19.86	19.75	19.92	19.85	20.21	±2.24

Method Detection limit (MDL)

The method detection limit is defined as the smallest amount of solute required to produce a signal that is twice the noise level. The optimum headspace analysis condition used in the investigation of the method detection limit of each volatile compound in aqueous solution under GC condition (as described in Tables 3.4 and 3.5) is shown in Table 4.25 . This condition would be used in the investigation of precision and accuracy. The results obtained from the method detection limit study of each interested compound using FID and ECD as a detector are shown in Tables 4.28 and 4.29, respectively.

Table 4.28 The method detection limit of each volatile organic compound in aqueous solution using FID as the detector.

Compound	Method detection limit (MDL) (ppm)
Methylene chloride	0.10
Chloroform	0.20
Benzene	0.08
Trichloroethylene	0.20
1,4-Dioxane	12.41

Triplicate analyses

Table 4.29 The mrthod detection limit of each volatile organic compound in aqueous solution using ECD as the detector.

Compound	Method Detection Limit (MDL) (ppb)
Methylene chloride	2.5
Chloroform	0.4
Trichloroethylene	0.2

Triplicate analyses

The Linearity of Headspace Analysis Technique

The linearity of headspace analysis technique of each volatile compound using FID and ECD as a detector are shown in Tables 4.30 and 4.31 , respectively and the graph showing in Figures 4.21-4.25 and Figures 4.26-4.28 ,respectively. The linearity ranges of volatile organic compound using FID and ECD are 0.38-223.34 ppm and 4.21-1060.55 ppb, respectively. the correlation coefficient range are 0.9988-0.9997 and 0.9984-0.9990, respectively.

Table 4.30 The result of linearity of the concentration ranges and correlation coefficient of each volatile organic compound using FID as a detector.

Compound	Concentration Range (ppm)	Correlation Coefficient
Methylene chloride	0.42 - 151.60	0.9988
Chloroform	0.42 - 152.72	0.9994
Benzene	0.38 - 148.90	0.9993
Trichloroethylene	0.42 - 151.58	0.9997
1,4-Dioxane	12.41 - 223.34	0.9973

Triplicate analyses

Table 4.31 The result of linearity of the concentration ranges and correlation coefficient of each volatile organic compound using ECD as a detector

Compound	Concentration Range (ppb)	Correlation Coefficient
Methylene chloride	4.21 - 1052.80	0.9990
Chloroform	4.42 - 1060.55	0.9989
Trichloroethylene	4.21 - 1052.65	0.9984

Triplicate analyses

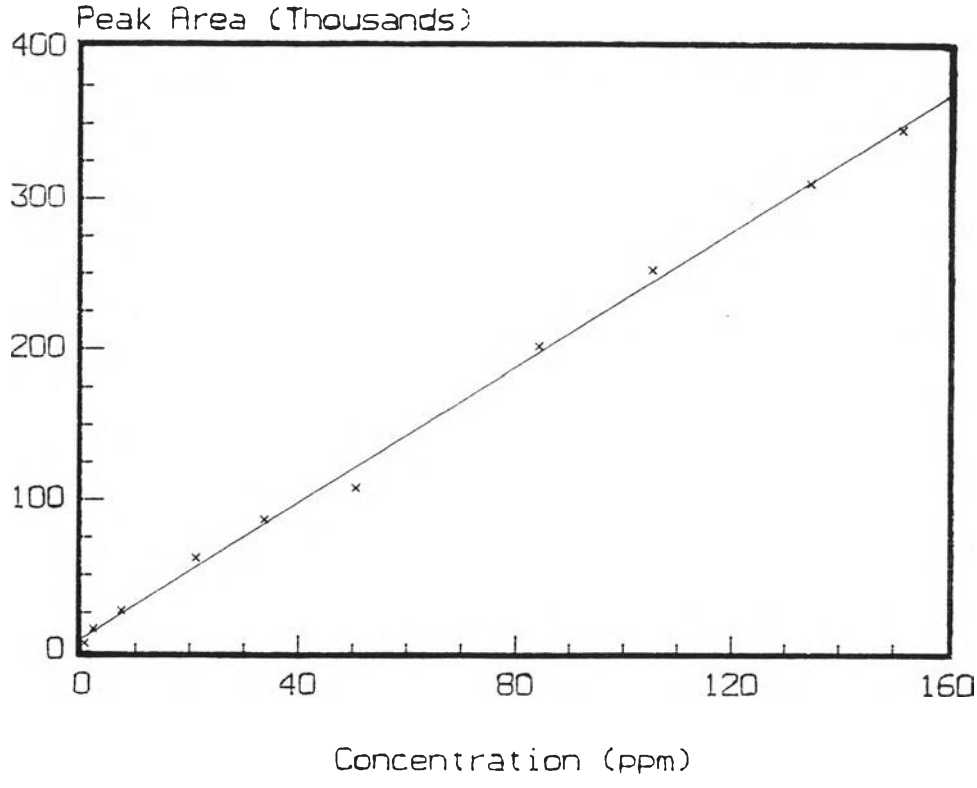


Figure 4.31 The linearity concentration ranges of methylene chloride in aqueous solution by using FID as a detector

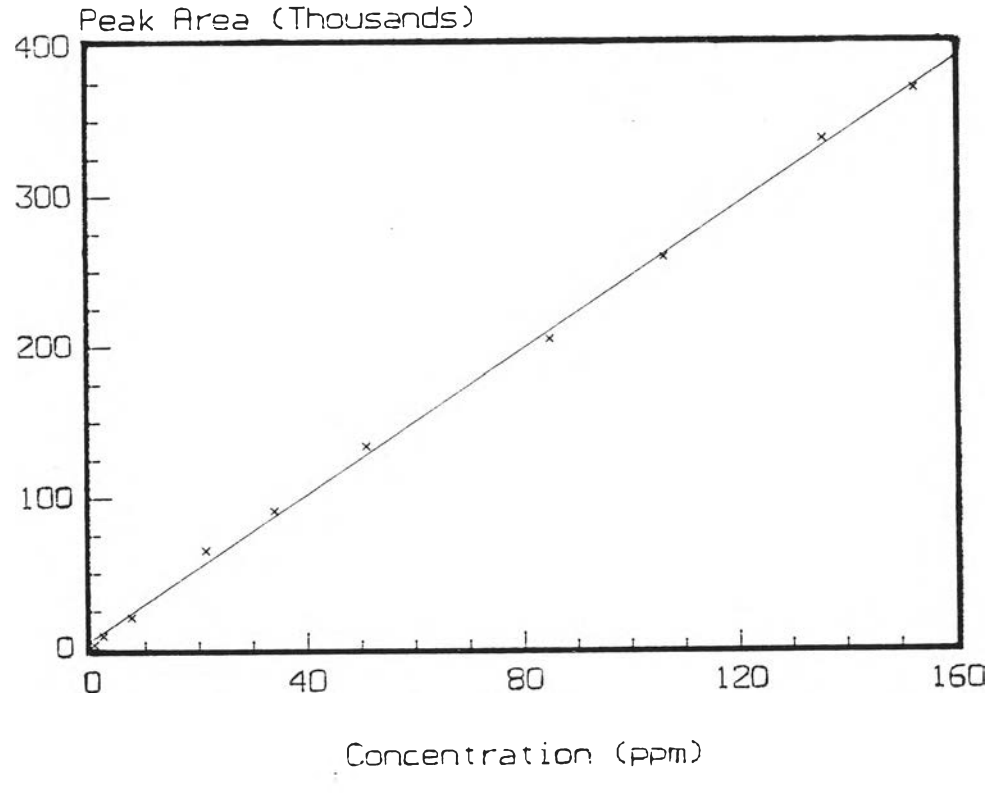


Figure 4.32 The linearity concentration ranges of chloroform in aqueous solution by using FID as a detector

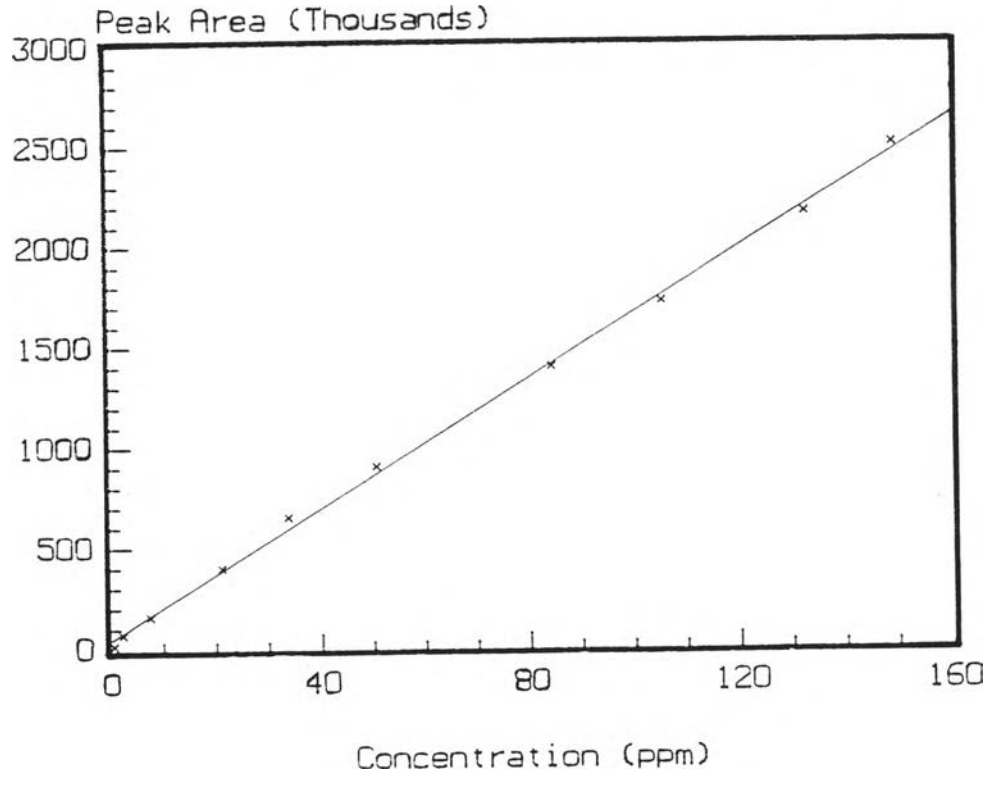


Figure 4.33 The linearity concentration ranges of benzene in aqueous solution by using FID as a detector

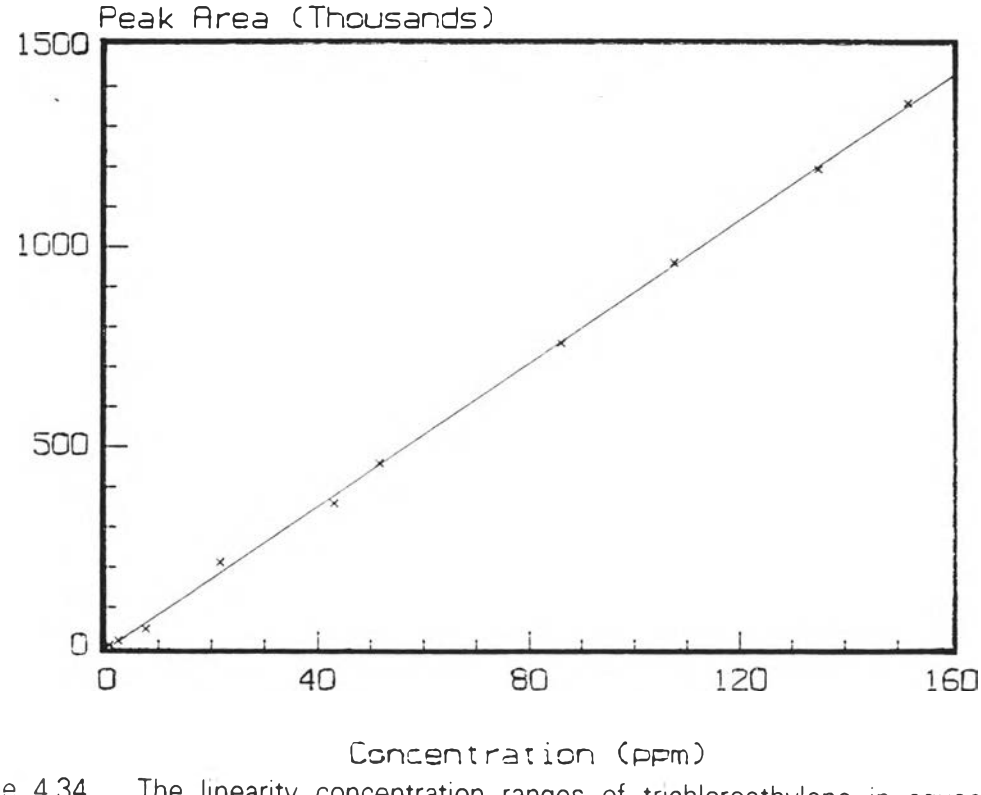


Figure 4.34 The linearity concentration ranges of trichloroethylene in aqueous solution by using FID as a detector

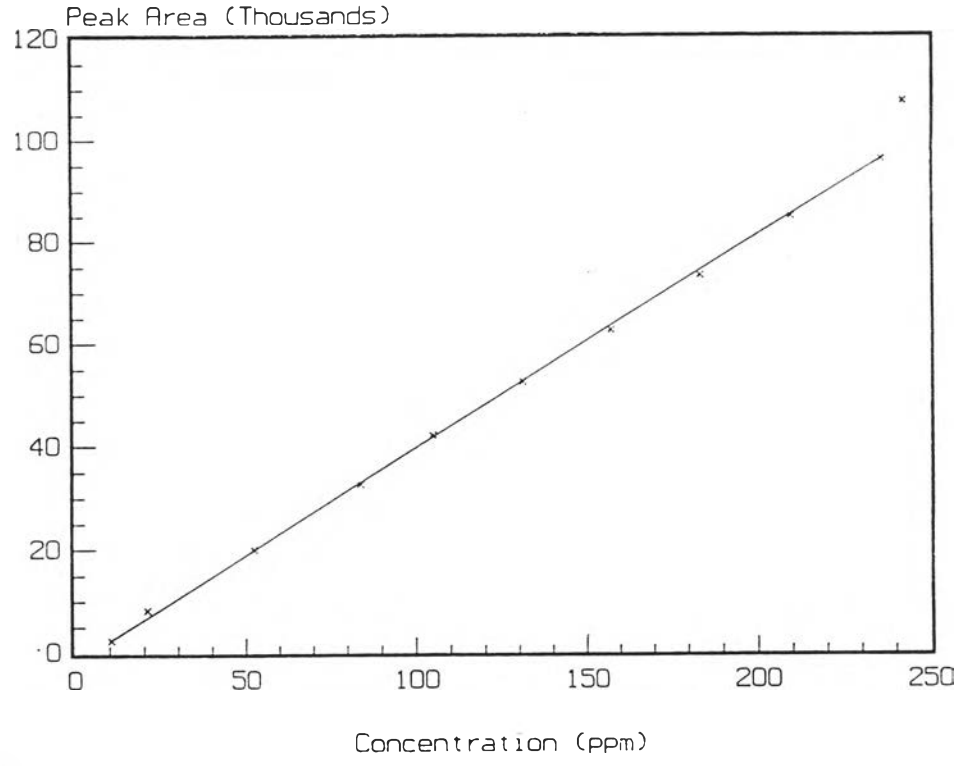


Figure 4.35 The linearity concentration ranges of 1,4-dioxane in aqueous solution by using FID as a detector

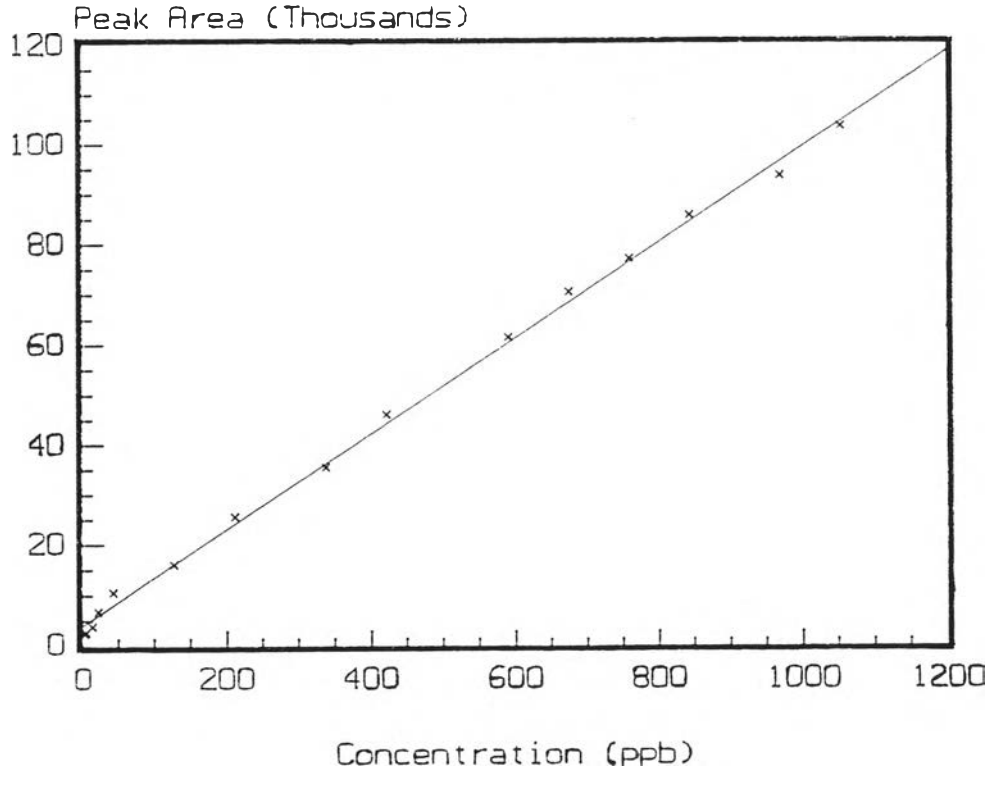


Figure 4.36 The linearity concentration ranges of methylene chloride in aqueous solution by using ECD as a detector

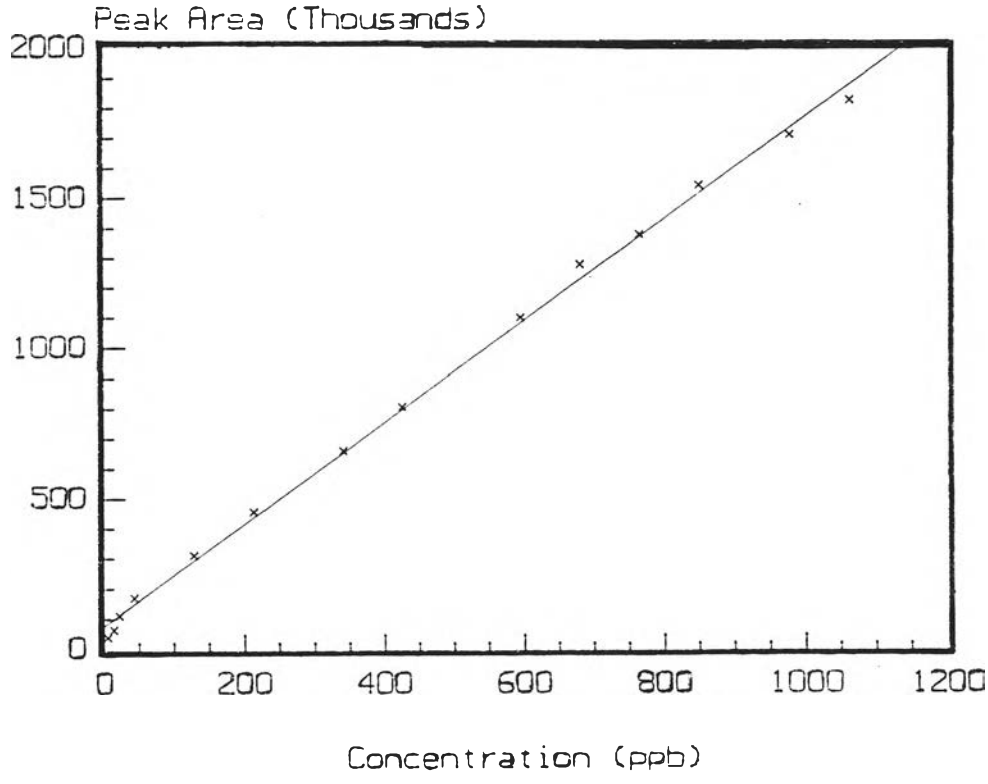


Figure 4.37 The linearity concentration ranges of chloroform in aqueous solution by using ECD as a detector

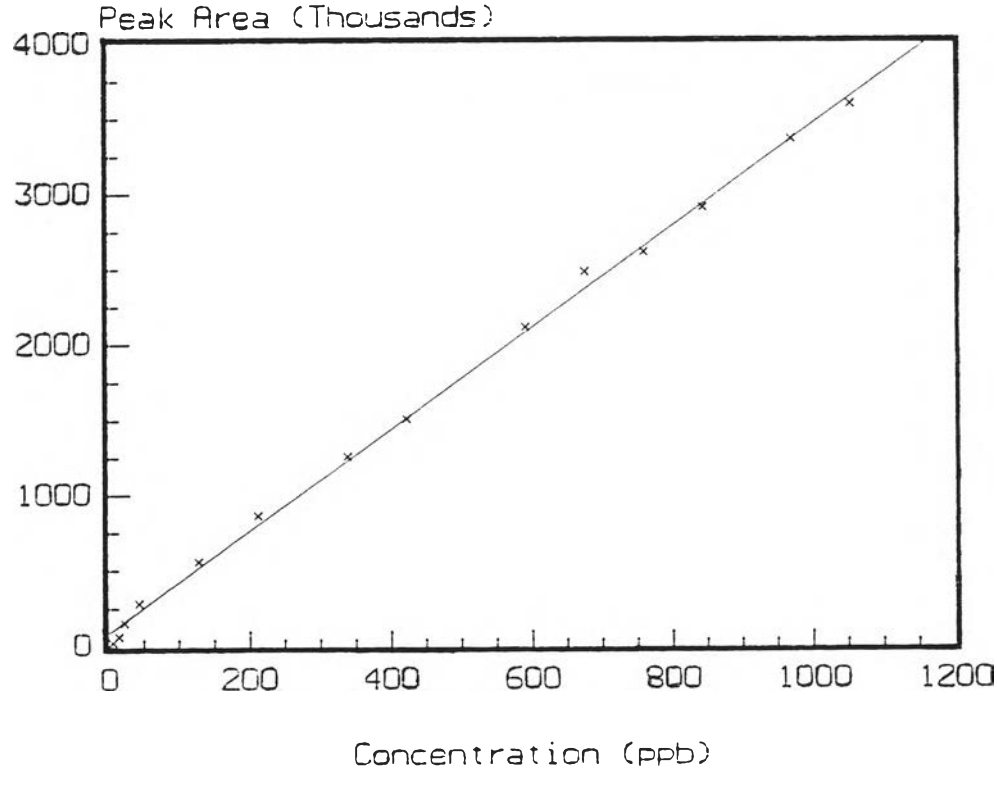


Figure 4.38 The linearity concentration ranges of trichloroethylene in aqueous solution by using ECD as a detector

The Accuracy of Headspace Analysis Technique

The accuracy of headspace analysis technique is investigated by comparing the results of concentration of each volatile organic compound obtained from the analysis with the true concentration of each compound in synthetic unknown mixture solution prepared by thesis advisor. The unknown is prepared in DMSO and it is diluted with water. The concentration of each volatile organic compound in the unknown solution is determined by means of external standardization method .

The results obtained from the study are presented in Table 4.32 The percent error of each volatile compound in synthetic unknown mixture is in the range of ±0.42 - 5.06 .

Table 4.32 The results of the analysis of synthetic unknown solution by external standard method and using FID as a detector.

Compound	Concentration (ppm)		%Error	%RSD
	True	Experiment		
Methylene chloride	0.2790	0.2814	± 0.86	± 2.38
Chloroform	0.3553	0.3568	± 0.42	± 2.20
Benzene	0.1293	0.1255	± 2.94	± 3.89
Trichloroethylene	0.3895	0.4092	± 5.06	± 5.02
1,4-Dioxane	20.9700	21.2885	±1.52	± 3.51

Triplicate analyses

All of the results obtained from the above studies indicated that the headspace analysis technique seems to be the best alternative method for determination of volatile organic compounds in drug or water samples. The reasons for this are that:

(1) This technique gives the good precision and good accuracy as showing in Tables 4.26-4.27 and 4.32, respectively.

(2) It requires no preconcentration step for the determination of trace volatile organic compound in drug.

(3) No interference peaks of uninterested nonvolatile organic compounds appear on the chromatogram, so the chromatographic analysis time is short.

(4) The method detection limit is found to be in mid ppb level (except 1,4-dioxane) using FID as a detector and sub-ppb level using ECD as a detector.

(5) It is comfortable to used because it can be performed analysis automatically.

(6) It can analyse 36 sample/day.

The Determination of Volatile Organic Compounds in Drug Samples

Fifteen drug samples i.e., Amoxycillin from the Unichem Co.,Ltd. , Aspirin and Aspent A.D. from the Unichem Co.,Ltd. , B1,6,12 from Merck Co.,Ltd. , Olic Co.,Ltd. , P.P.Lab Co.,Ltd. and Takeda Co.,Ltd. , Brofen and Ibuprofen from Pake-Davis Co.,Ltd. , Clamosin from Biolab Co.,Ltd. ,Erythromycin from Pharmaceutical Organization, Abbott Co.,Ltd. and Servipharm Co.,Ltd. , Fuben 500 from TO Co.,Ltd. and Stresstab 600+zinc from F.E. Zuellig Co., Ltd. are analyzed by the automated headspace analysis technique under the optimal headspace analysis condition as shown in Table 4.25 and under GC condition as described in Table 3.4 for FID as a detector and Table 3.5 for ECD as a detector, The chromatograms of drug samples are shown in Figures 4.40(A) - 4.54(A) by using FID as a detector and the chromatogram of some drug samples that can not be detected by FID in ppm level and however, it can be detected in ppb level by using ECD as shown in Figure 4.56 (A).

The retention time (t_r) of the unknown peaks obtained from the sample chromatogram are compared with the retention time of the standard mixture of volatile organic compound peaks as shown in the chromatogram in Figure 4.39 and 4.55 for FID and ECD as a detector, respectively. It found that the peak in the sample have the same retention times of methylene chloride chloroform by using ECD as a detector . Moreover, it seem to be that there are high ethanol and methanol peaks appeared in a lot of samples by using FID as a detector and it can be seen that there are a small CS_2 peaks and other peak appeared in all samples chromatograms by using ECD as a detector. To confirm this result , all samples are spiked with the standard mixture solution of the interested compounds and it

analysed under the identical analysis condition. The chromatograms of the spiked samples are shown in Figures 4.50(B) - 4.54(B) for FID as a detector and Figure 4.56(B) for ECD as a detector. It can be seen that all fifteen samples having the eluted at the same peaks of standard methylene chloride (t_r 2.83 min) and chloroform (t_r 5.11 min) by using FID and ECD as a detector, respectively. Moreover, to confirm this result, all sample are analysed by using MSD (mass selective detector) as shown in Figure 4.57-4.64. Three samples have 83 percent matching for methylene chloride and 4 percent matching for chloroform. Chloroform has low percent matching because of it has a low quantity in drug samples.

Methylene chloride and chloroform in all drug samples are then determined by means of external standardization methods as mentioned in section 3.7.1 and the concentration of the compound are indicated in Table 4.33 and other organic compounds are indicated in Table 4.34.

Table 4.33 The concentration of the interested volatile organic compounds in fifteen drug samples using gas chromatograph equipped with ionization detector and electron capture detector.

No.	Sample	Concentration of interested volatile organic compounds	
		CH_2Cl_2	$CHCl_3$
1	Amoxycillin (Beechem Co., Ltd.)	202.10 μg/g ± 0.31	ND
2	Aspirin (Unichem Co., Ltd.)	ND	ND
3	Aspent (Unichem Co., Ltd.)	ND	ND
4	B1,6,12 (Merck Co., Ltd.)	ND	ND
5	B1,6,12 (Olic Co., Ltd.)	ND	ND
6	B1,6,12 (P.P. Lab Co., Ltd.)	ND	957.10 ng/g ±2.05
7	B1,6,12 (Takeda Co., Ltd.)	ND	ND
8	Brofen (Parke-Davis Co., Ltd.)	ND	ND
9	Ibuprofen (Parke-Davis Co., Ltd.)	ND	ND

No.	Sample	Concentration of interested volatile organic compounds	
		CH_2Cl_2	$CHCl_3$
10	Clamosin (Biolab Co., Ltd.)	38.10 μg/g ±0.56	ND
11	Erythromycin (Pharmaceutical Organization)	40.10 μg/g ±2.73	ND
12	Erythromycin (Abbott Co., Ltd.)	ND	ND
13	Erythromycin (Servipharm Co., Ltd.)	ND	ND
14	Fuben 500 (TO Co., Ltd.)	ND	ND
15	Stresstab 600+ Zinc (F.E. Zuellig Co., Ltd.)	ND	ND

Remark : ND = Not Detectable

CH_2CL_2 = Methylene Chloride

$CHCl_3$ = Chloroform

Table 4.34 The other volatile organic compounds in fifteen drug samples using gas chromatograph equipped with ionization detector and electron capture detector and mass selective detector.

No.	Sample	Other organic volatile compound
1	Amoxycillin (Beechem Co., Ltd.)	cyclohexane[a*], methylamine[a*], pentanone[a*]
2	Aspirin (Unichem Co., Ltd.)	ethanol[a*], methanol[a*]
3	Aspent (Unichem Co., Ltd.)	acetamine[a*],carbondisulfide[b*]
4	B1,6,12 (Merck Co., Ltd.)	methanamine[a*],carbondisulfide[b*]
5	B1,6,12 (Olic Co., Ltd.)	tetrachloroethylene[a*],carbondisulfide[b*]
6	B1,6,12 (P.P. Lab Co., Ltd.)	carbondisulfide[b*]
7	B1,6,12 (Takeda Co., Ltd.)	carbondisulfide[b*]
8	Brofen (Parke-Davis Co., Ltd.)	cyclohexane[a*],carbondisulfide[b*]
9	Ibuprofen (Parke-Davis Co., Ltd.)	carbondisulfide[b*]

No.	Sample	Other organic volatile compound
10	Clamosin (Biolab Co., Ltd.)	methanol[a*], ethanol[a*]
11	Erythromycin (Pharmaceutical Organisation)	ethanol[a*], carbondisulfide[b*]
12	Erythromycin (Abbott Co., Ltd.)	methanol[a*], ethanol[a*]
13	Erythromycin (Servipharm Co., Ltd.)	methanol[a*], carbondisulfide[b*]
14	Fuben 500 (TO Co., Ltd.)	ethanol[a*], carbondisulfide[b*]
15	Stresstab 600+ Zinc (F.E. Zuellig Co., Ltd.)	carbondisulfide[b*]

Remark : a* = FID as a detector and confirming by GC/MSD

b* = ECD as a detector and confirming by GC/MSD

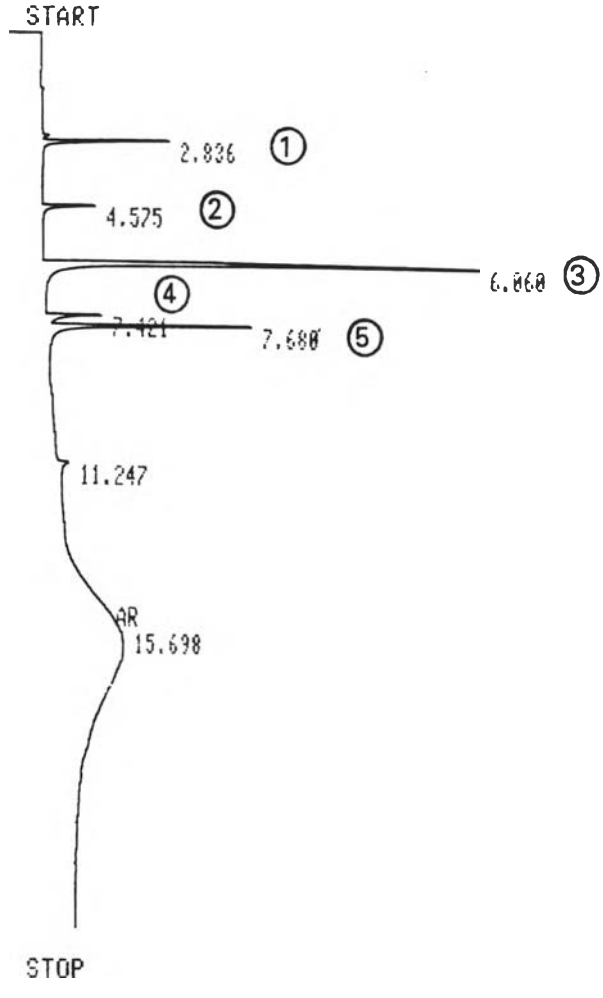


Figure 4.39 The gas chromatogram of standard mixture in aqueous solution.

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1

Concentration of the component :

(1)	Methylene chloride (CH_2Cl_2)	2.11 ppm
(2)	Chloroform ($CHCl_3$)	2.12 ppm
(3)	Benzene (C_6H_6)	2.10 ppm
(4)	Trichloroethylene (TCE)	2.11 ppm
(5)	1,4-Dioxane ($C_6H_8O_2$)	49.63 ppm

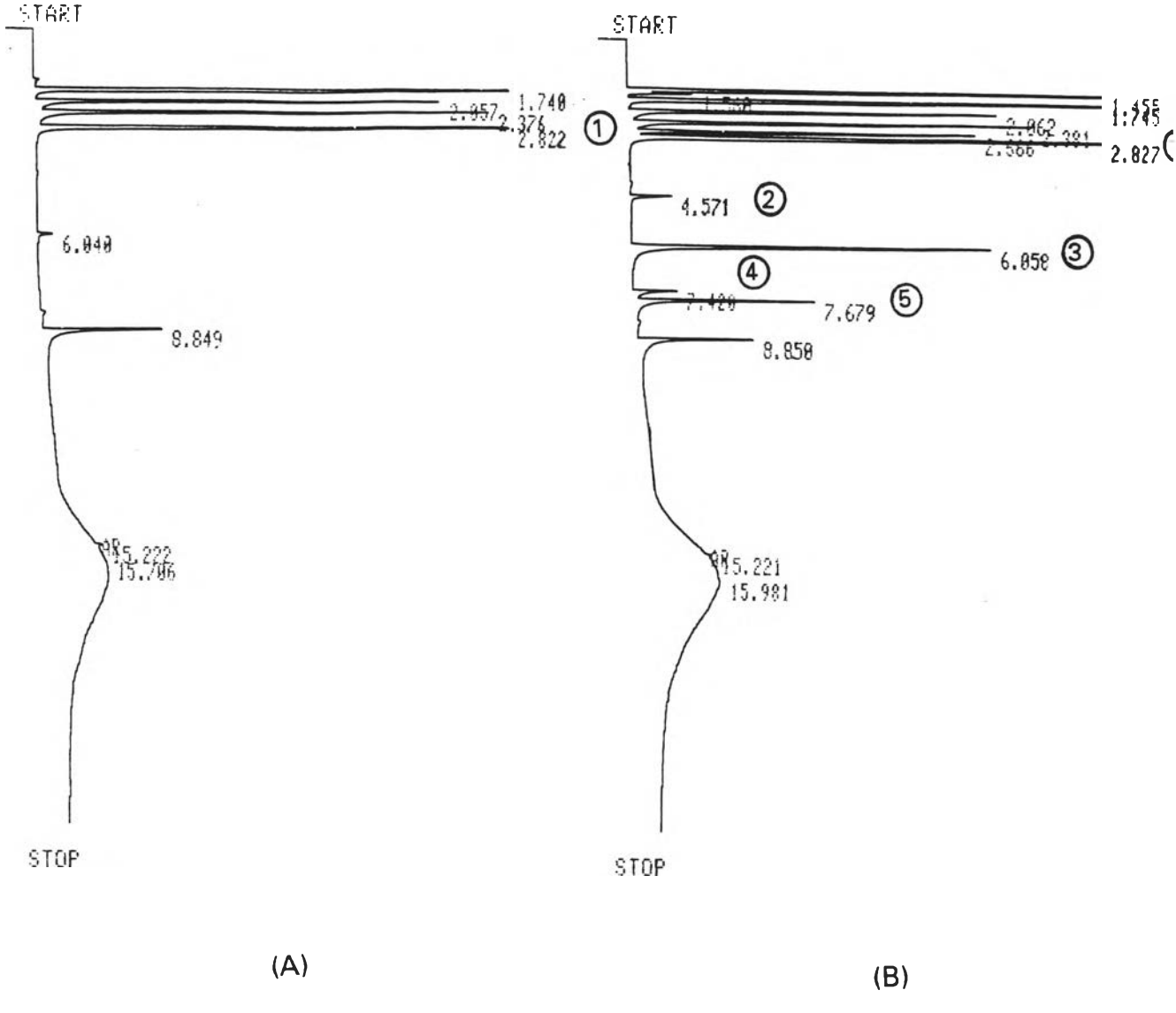


Figure 4.40 The gas chromatogram of

(A) Amoxycillin (Beechem Co.,Ltd.)

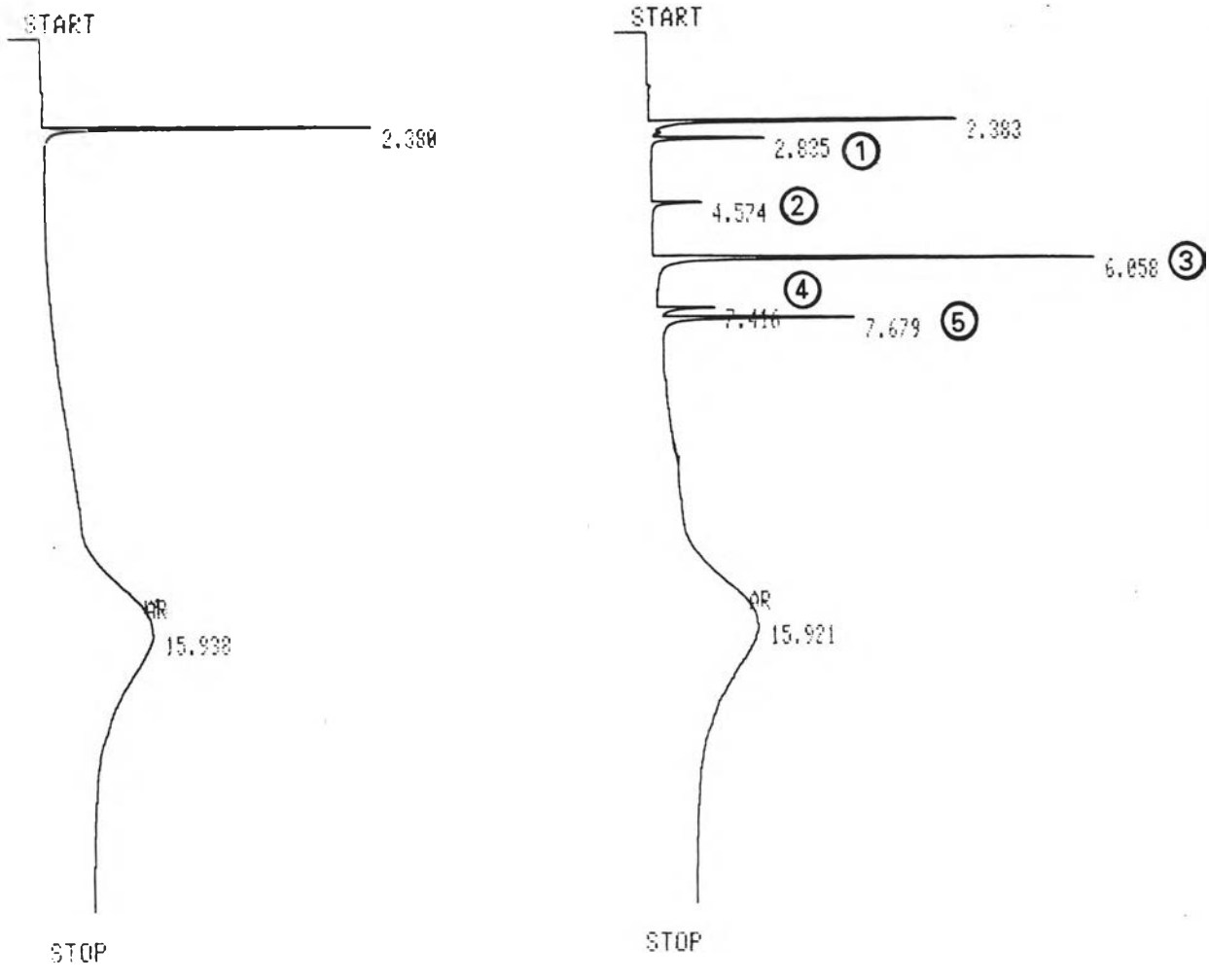
(B) Amoxycillin + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1



(A) (B)

Figure 4.41 The gas chromatogram of

(A) Aspirin (Unichem Co.,Ltd.)

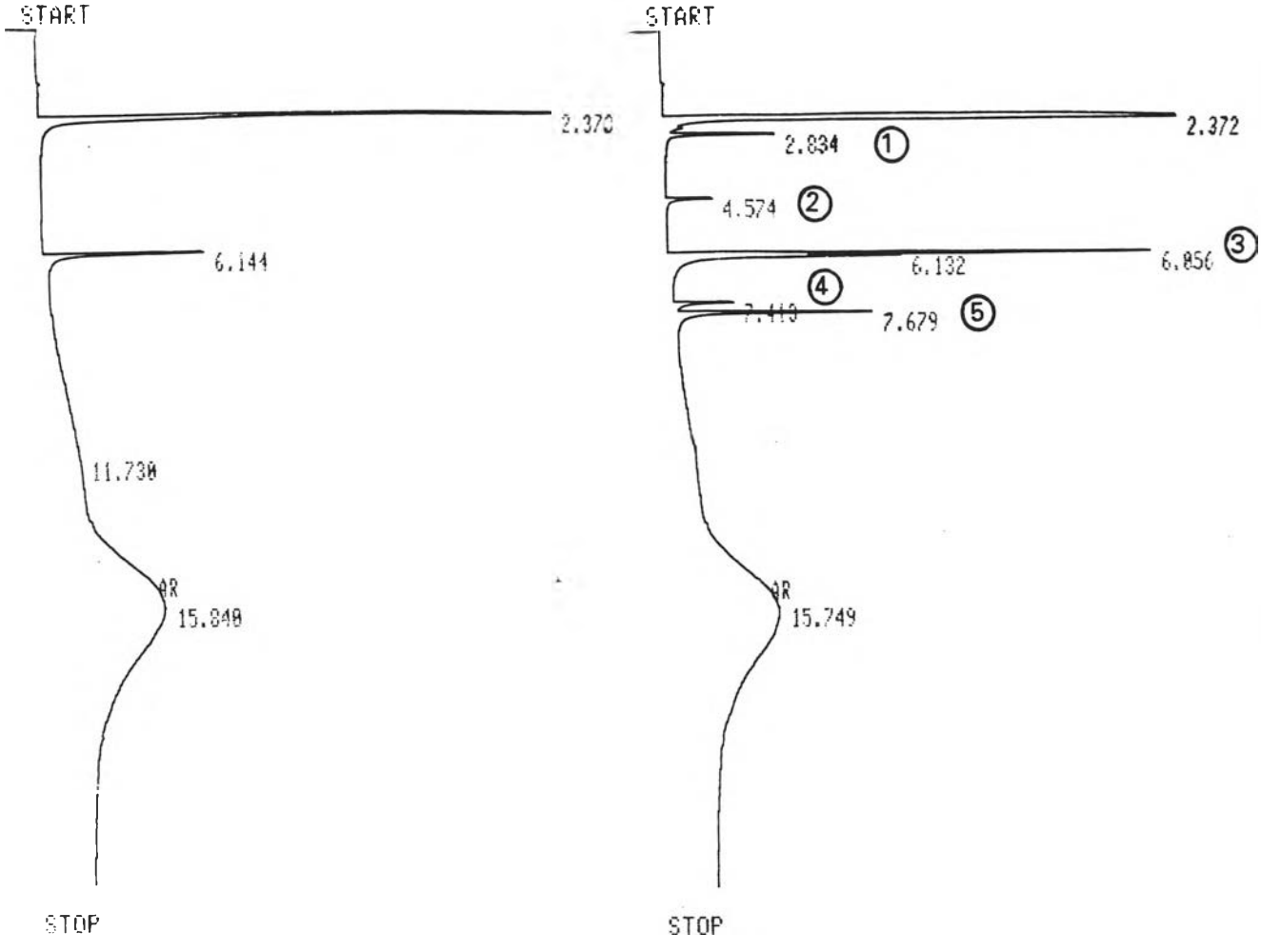
(B) Aspin + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1



(A)

(B)

Figure 4.42 The gas chromatogram of

(A) Aspent A.D. (Unichem Co.,Ltd.)

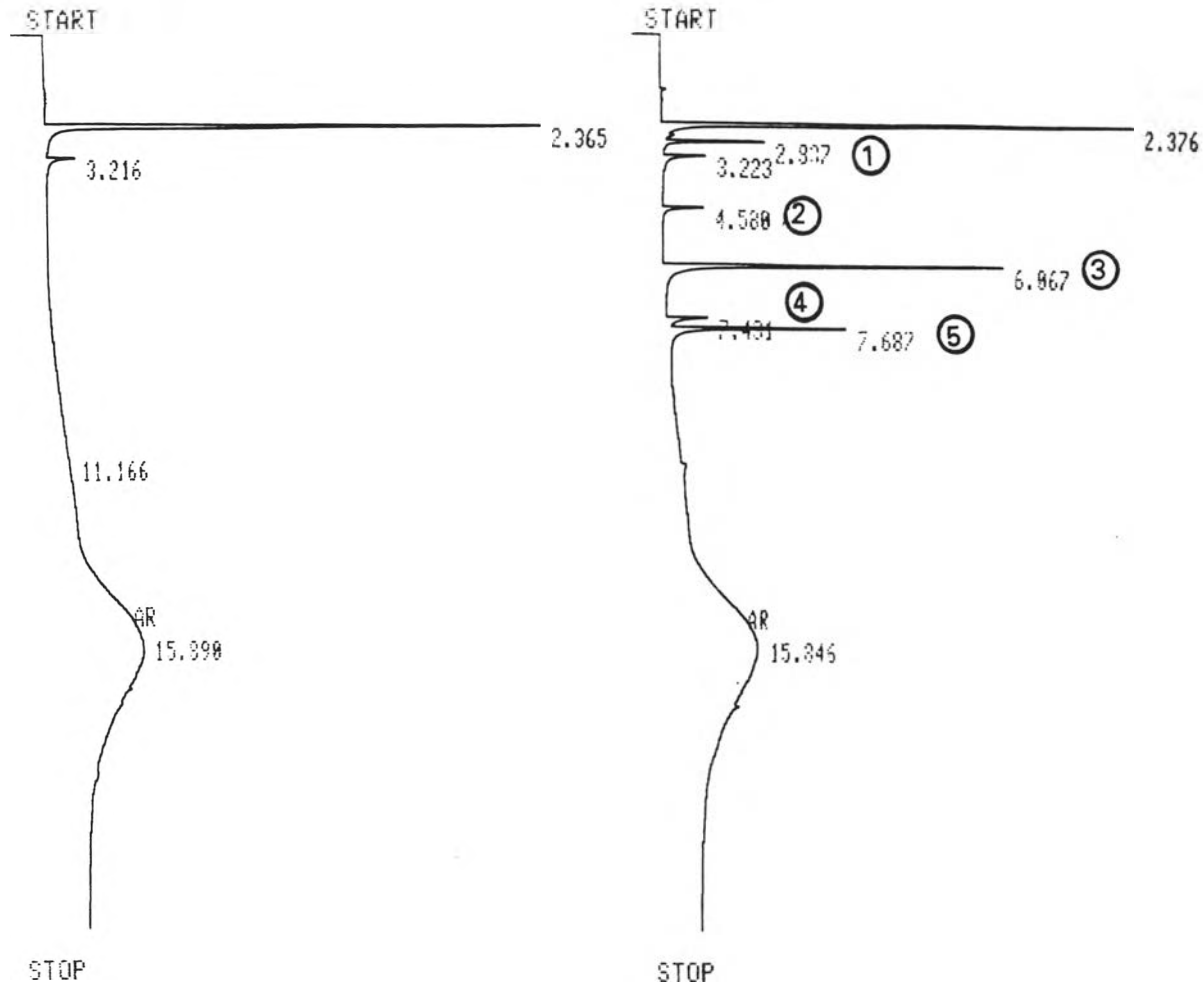
(B) Aspent A.D. + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1



(A) (B)

Figure 4.43 The gas chromatogram of

(A) Vitamin B1,6,12 (Merck Co.,Ltd.)

(B) Vitamin B1,6,12 + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1

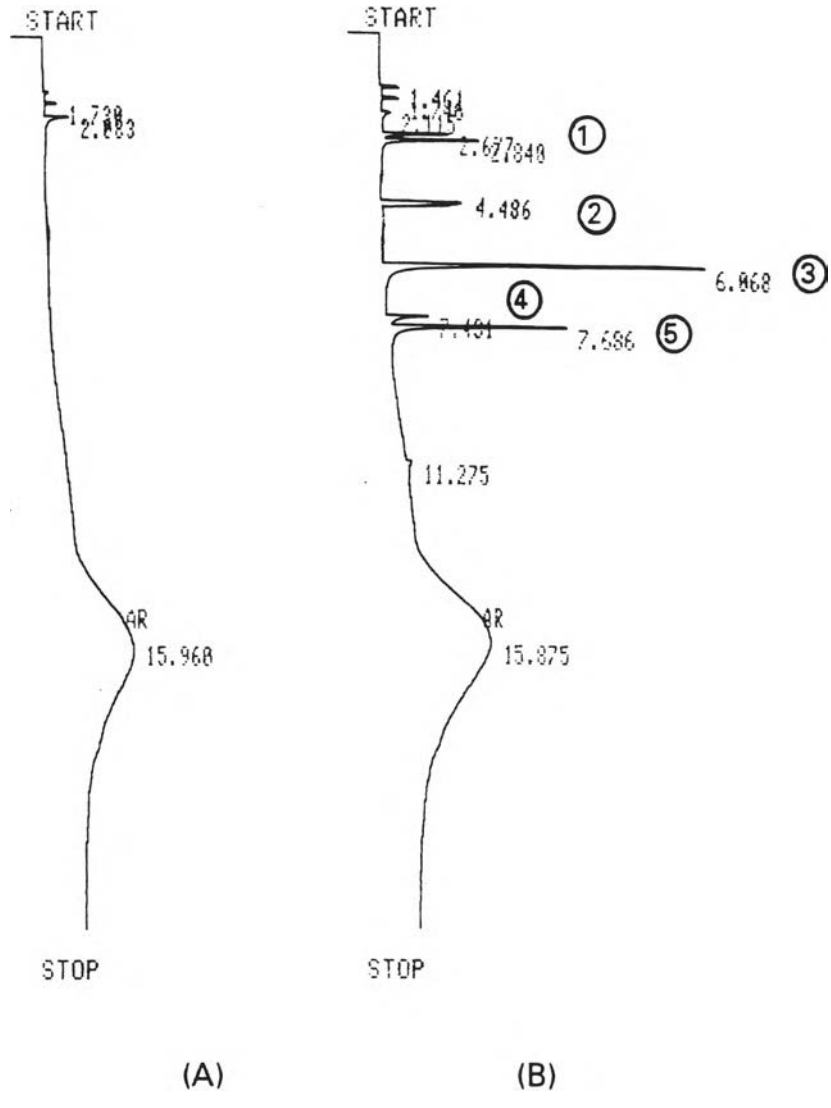


Figure 4.44 The gas chromatogram of

(A) Vitamin B1,6,12 (Olic Co.,Ltd.)

(B) Vitamin B1,6,12 + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1

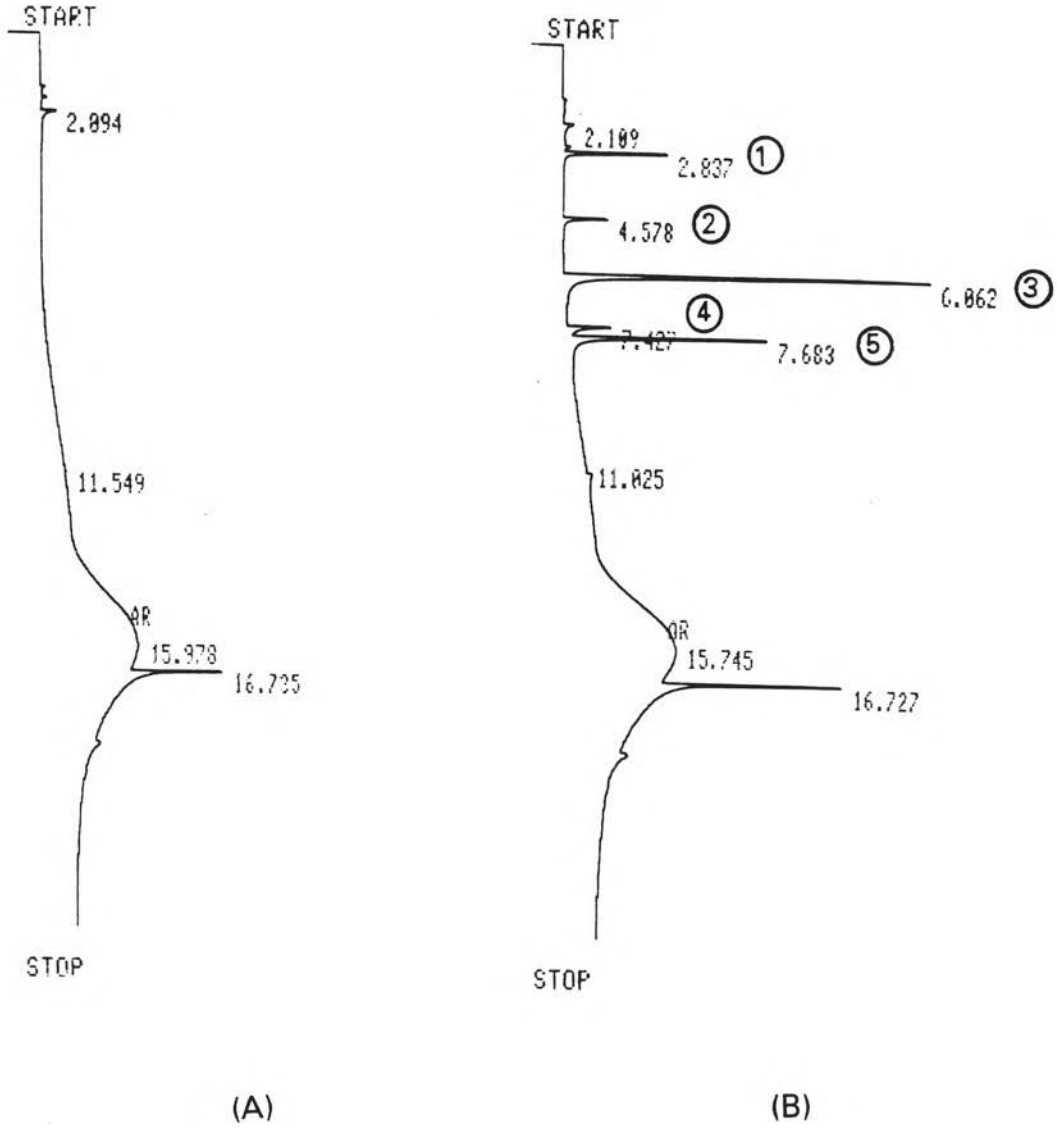


Figure 4.45 The gas chromatogram of

(A) Vitamin B1,6,12 (P.P. Lab Co.,Ltd.)

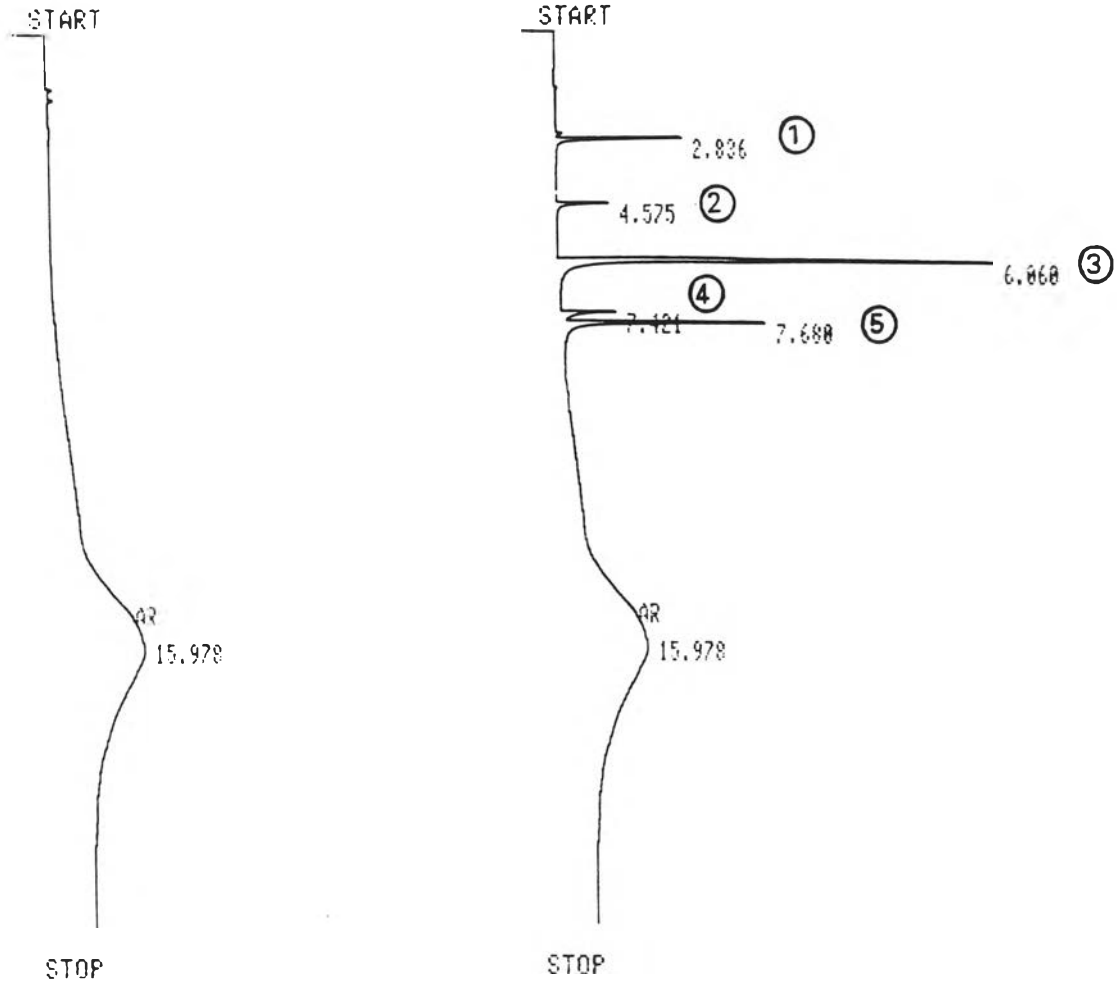
(B) Vitamin B1,6,12 + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1



(A) (B)

Figure 4.46 The gas chromatogram of

(A) Vitamin B1,6,12 (Takada Co.,Ltd.)

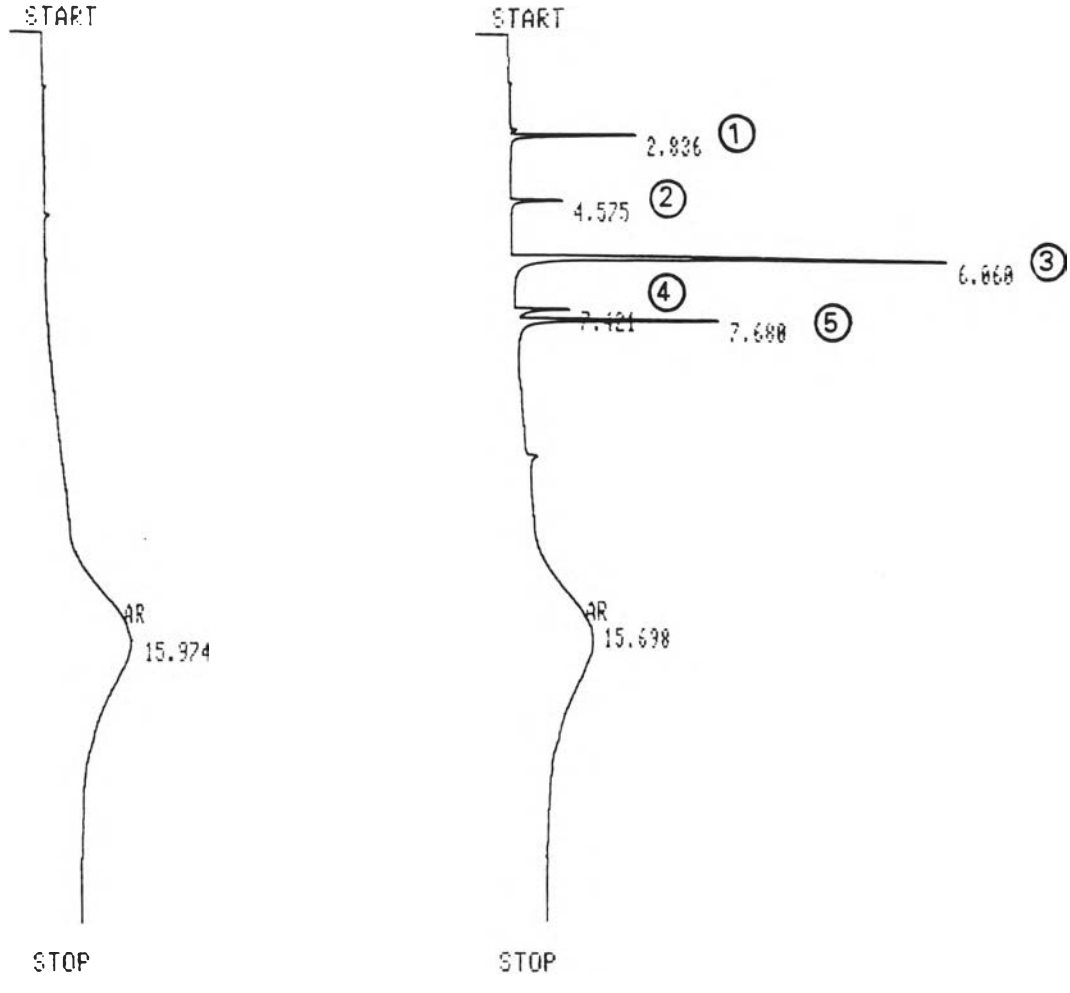
(B) Vitamin B1,6,12 + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1



(A) (B)

Figure 4.47 The gas chromatogram of

(A) Brofen (Parke-Davis Co.,Ltd.)

(B) Brofen + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1

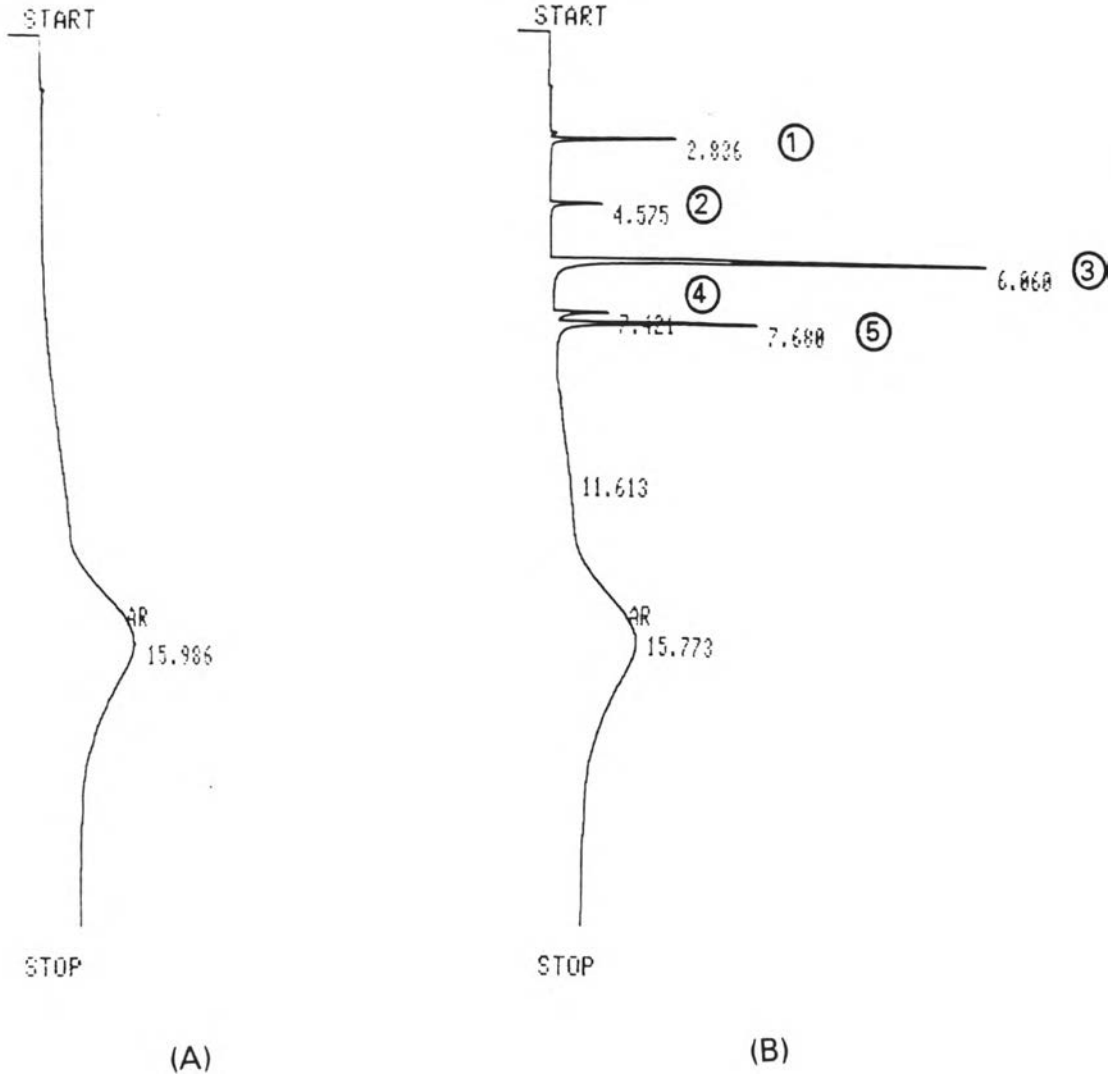


Figure 4.48 The gas chromatogram of

(A) Ibuprofen (Parke-Davis Co.,Ltd.)

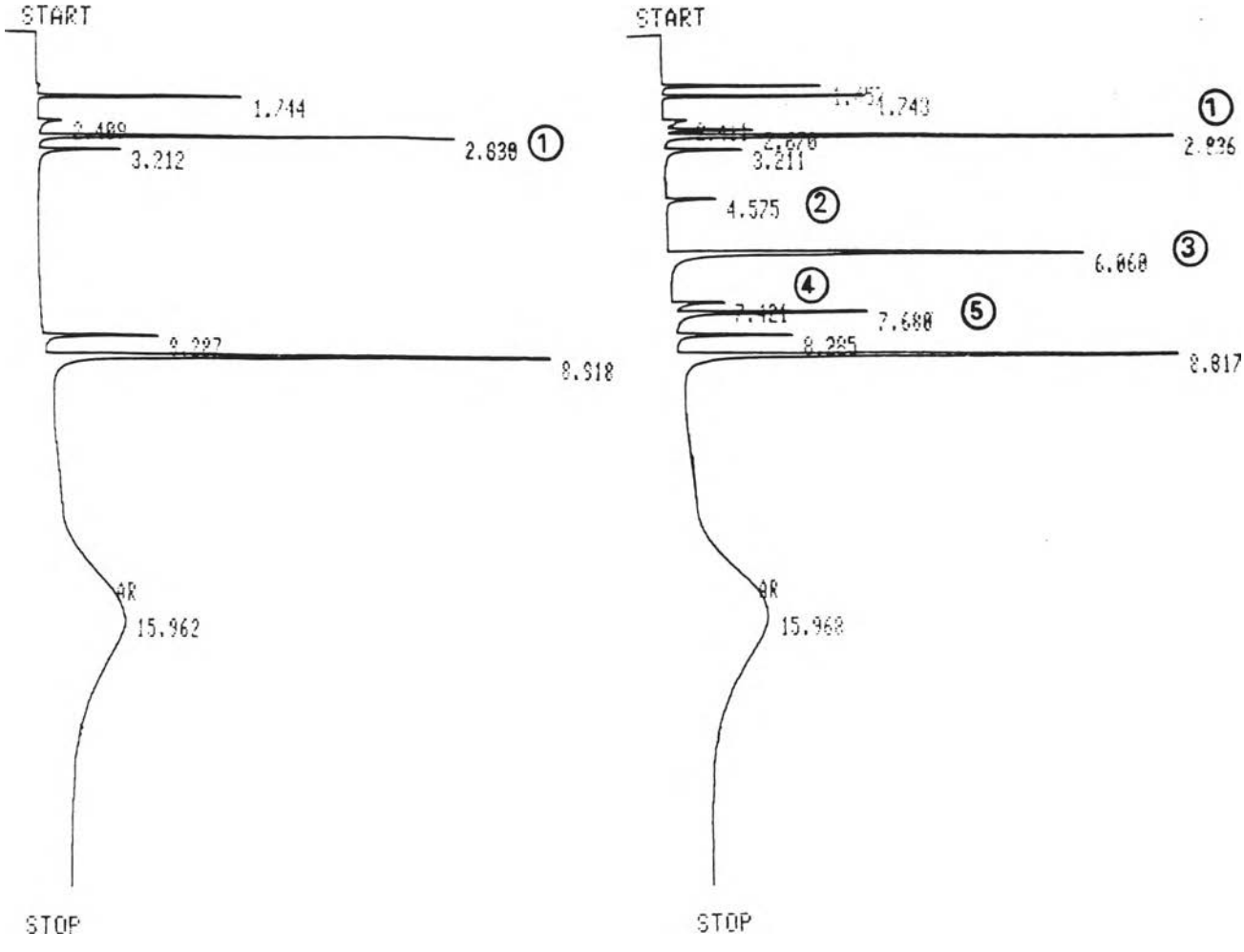
(B) Ibuprofen + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1



(A) (B)

Figure 4.49 The gas chromatogram of

(A) Clamosin (Biolab Co.,Ltd.)

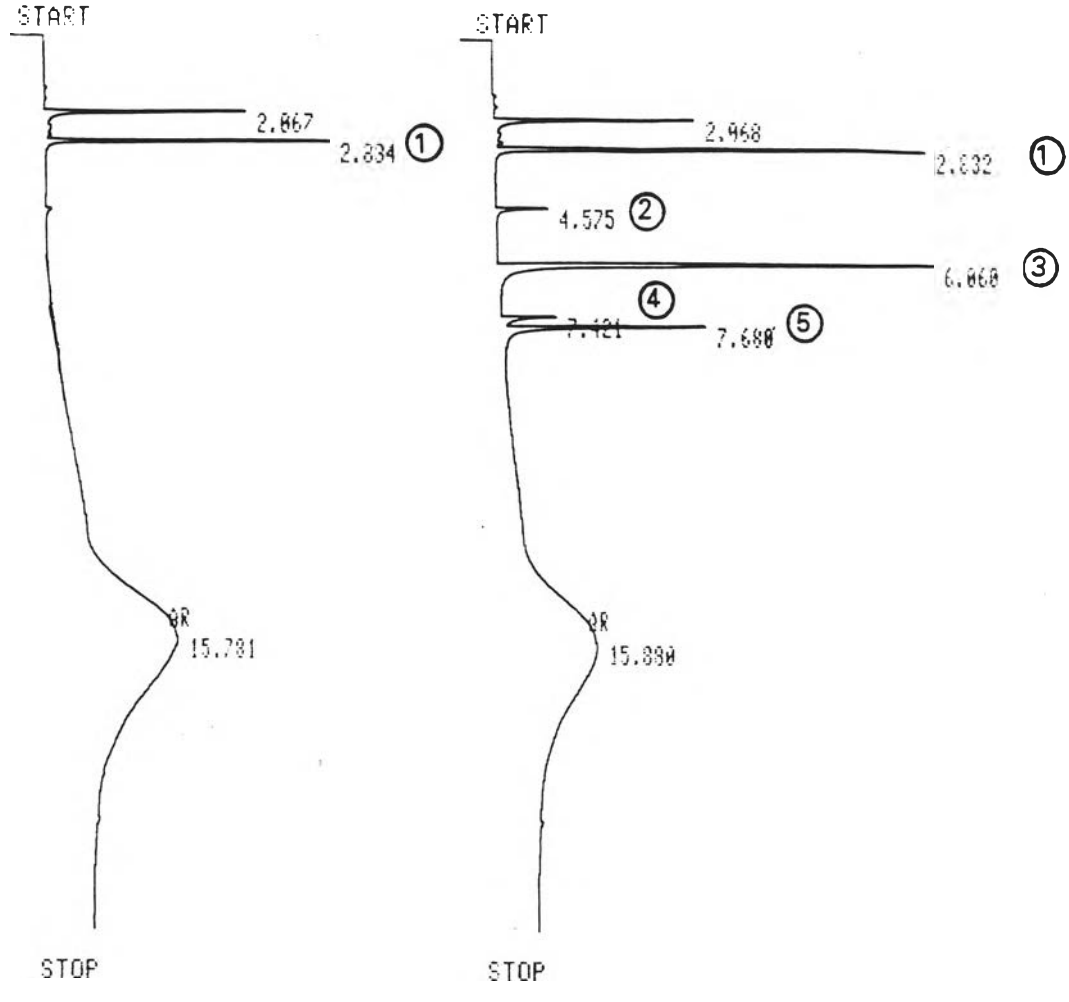
(B) Clamosin + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1



(A) (B)

Figure 4.50 The gas chromatogram of

(A) Erythromycin (Pharmaceutical Organization)

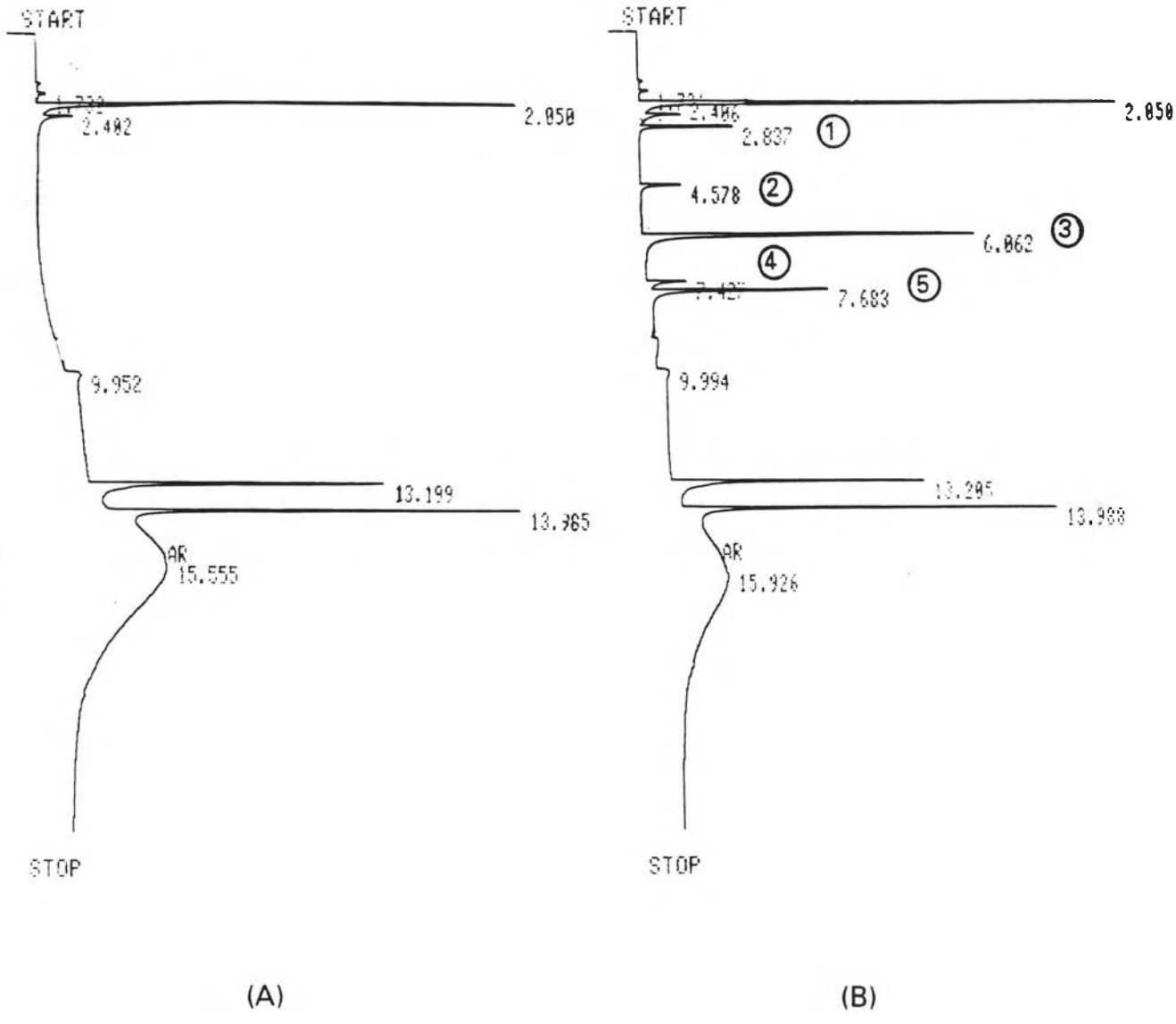
(B) Erythromycin + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1



(A) (B)

Figure 4.51 The gas chromatogram of

(A) Erythromycin (Abbott Co.,Ltd.)

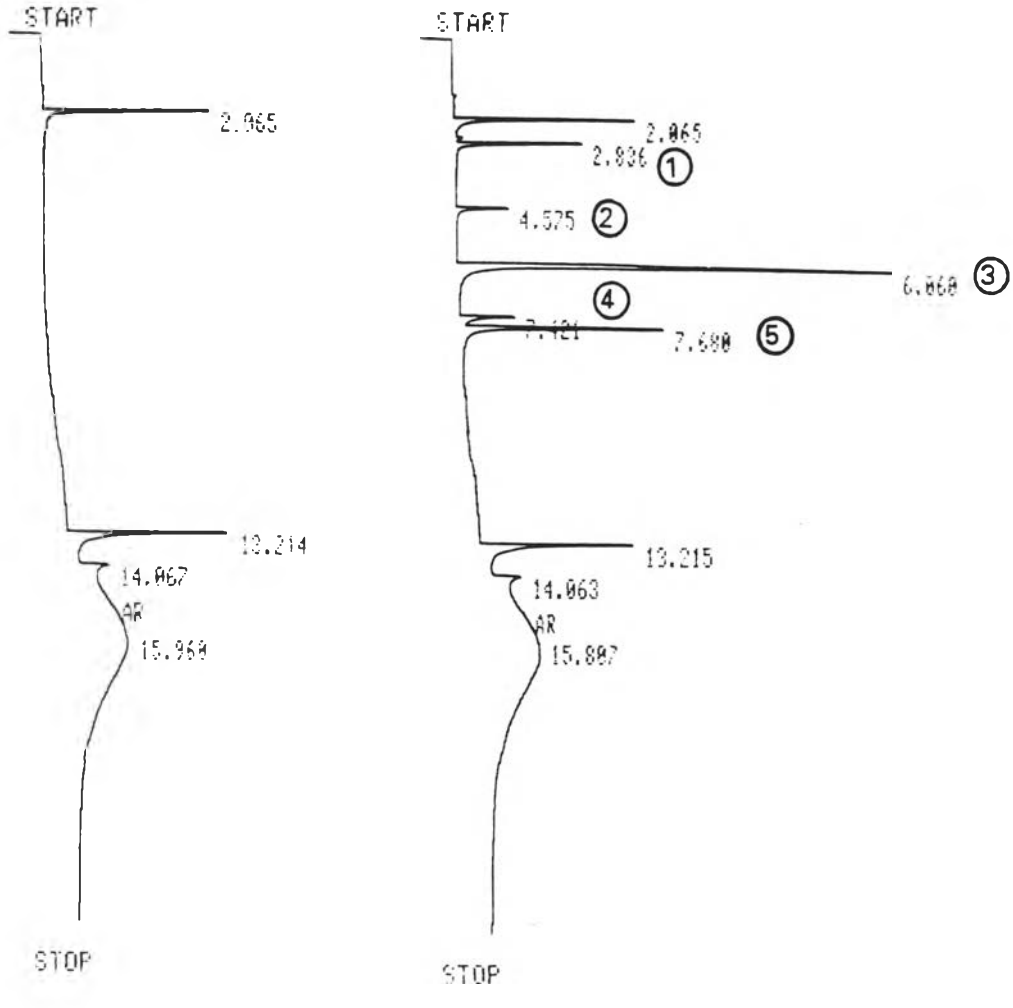
(B) Erythromycin + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1



(A) (B)

Figure 4.52 The gas chromatogram of

(A) Erythromycin (Servipharm Co.,Ltd.)

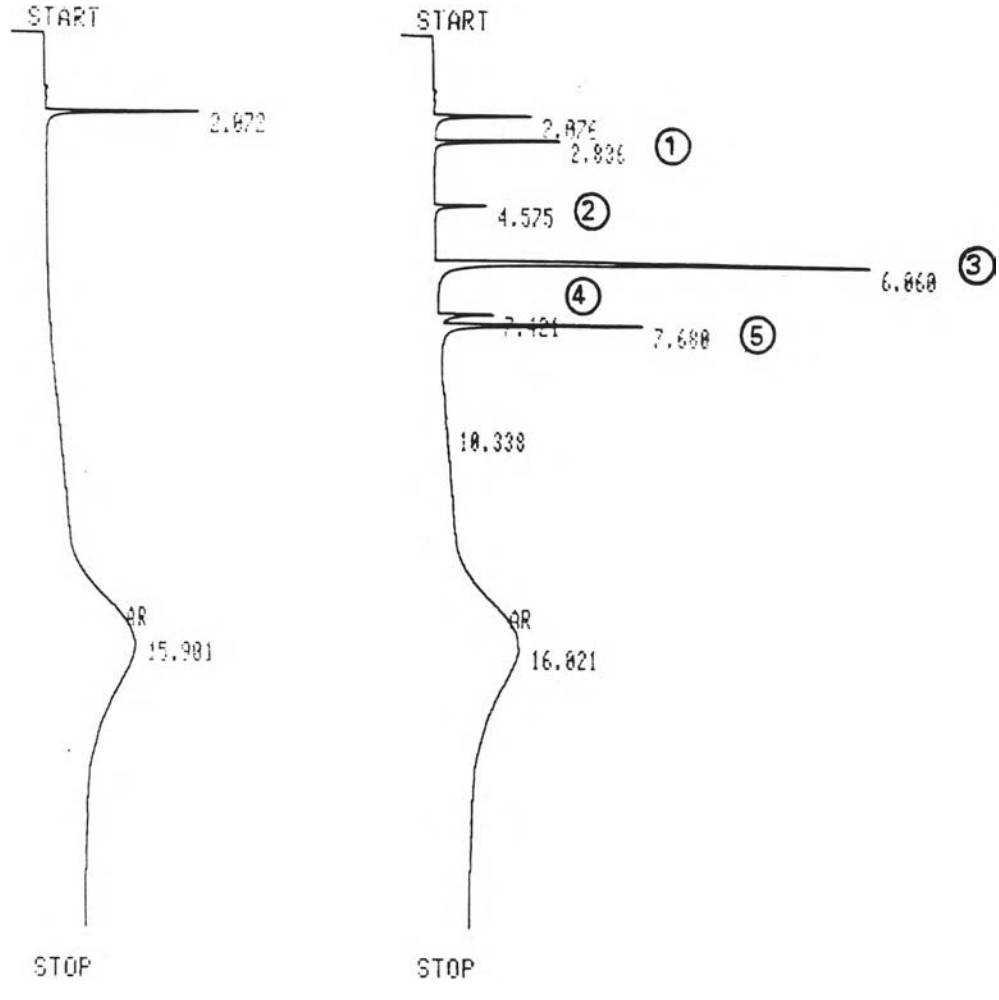
(B) Erythromycin + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1



(A) (B)

Figure 4.53 The gas chromatogram of

(A) Fuben 500 (TO Co.,Ltd.)

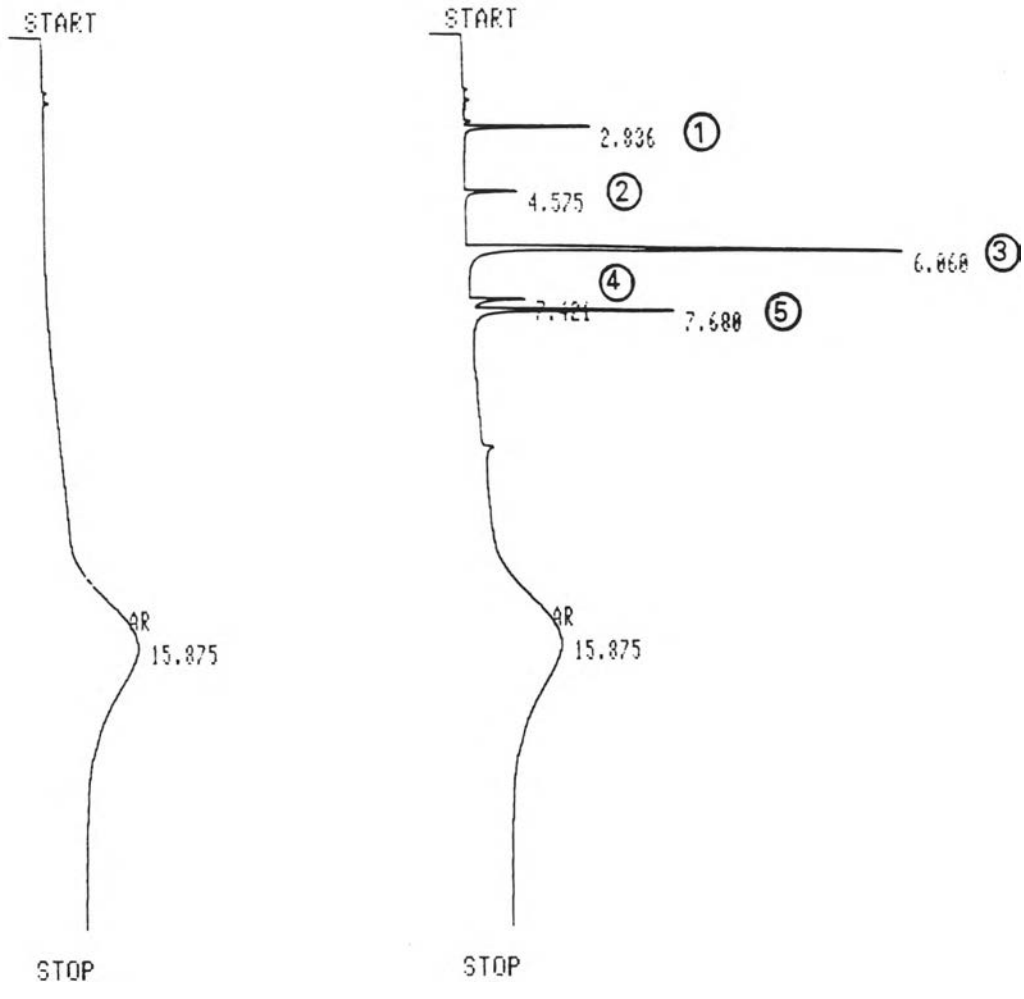
(B) Fuben 500 + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1



(A) (B)

Figure 4.54 The gas chromatogram of

(A) Stresstab 600 + zinc (F.E. Zuellig Co.,Ltd.)

(B) Stresstab 600 + zinc + standard mixture in aqueous solution

Condition

GC/FID : described in Table 3.4

Headspace : described in Table 4.25

Integrator : att 1

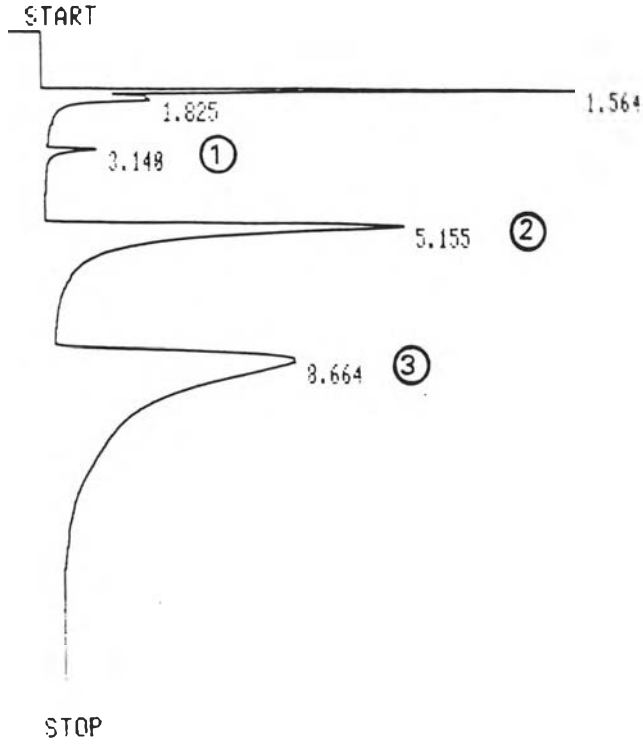


Figure 4.55 The gas chromatogram of standard mixture in aqueous solution.

Condition

GC/ECD : described in Table 3.5

Headspace : described in Table 4.25

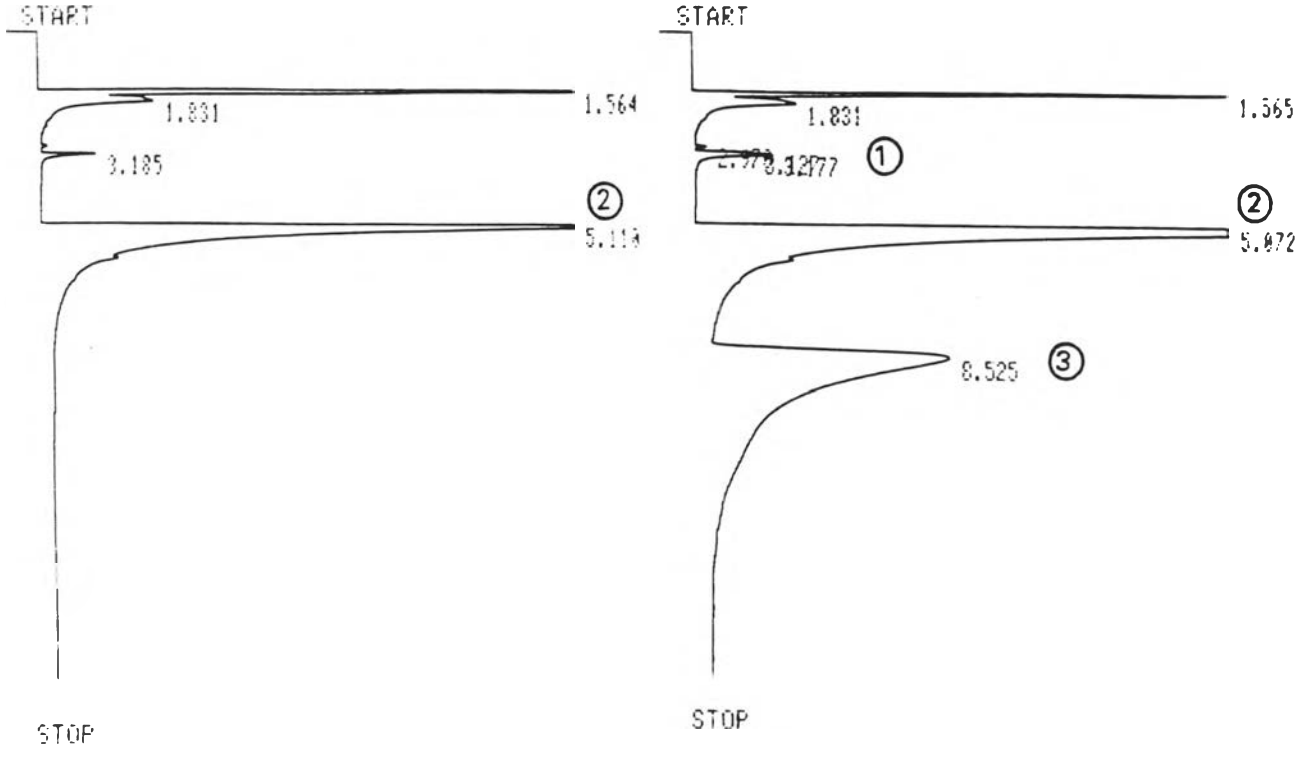
Integrator : att 1

Concentration of the component :

(1) Methylene chloride (CH_2Cl_2) 67.38 ppb

(2) Chloroform ($CHCl_3$) 65.99 ppb

(3) Trichloroethylene (TCE) 65.50 ppb



(A) (B)

Figure 4.56 The gas chromatogram of

(A) Vitamin B1,6,12 (P.P. Lab Co.,Ltd.)

(B) Vitamin B1,6,12 + standard mixture in aqueous solution

Condition

GC/ECD : described in Table 3.5

Headspace : described in Table 4.25

Integrator : att 1

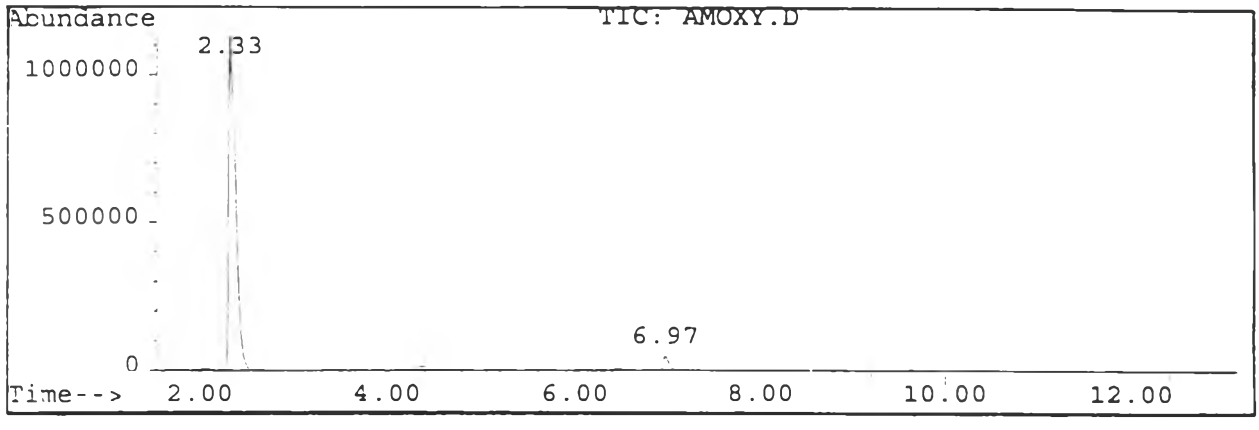


Figure 4.57 The gas chromatogram with MSD detection for headspace of Amoxycillin drug sample in aqueous solution.

GC/MSD Condition : described in Table 3.6

Library Searched : C:\DATABASE\PMW_TOXR.L
Quality : 83
ID : DICHLOROMETHANE

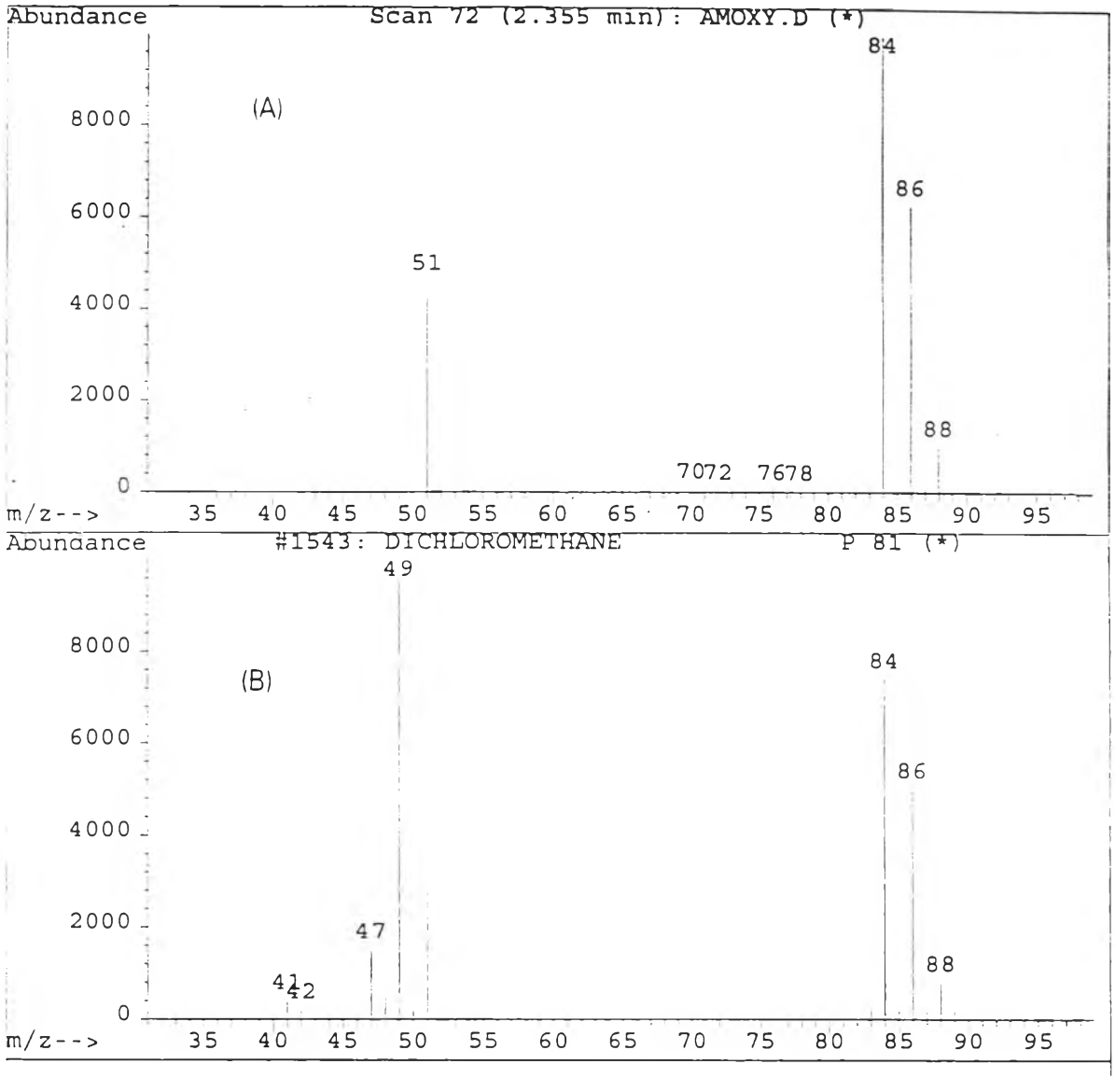


Figure 4.58 The mass spectrum for headspace of Amoxycillin drug sample with the retention time of 2.35 min. from the gas chromatogram in Figure 4.57

(A) Scan

(B) Library searched

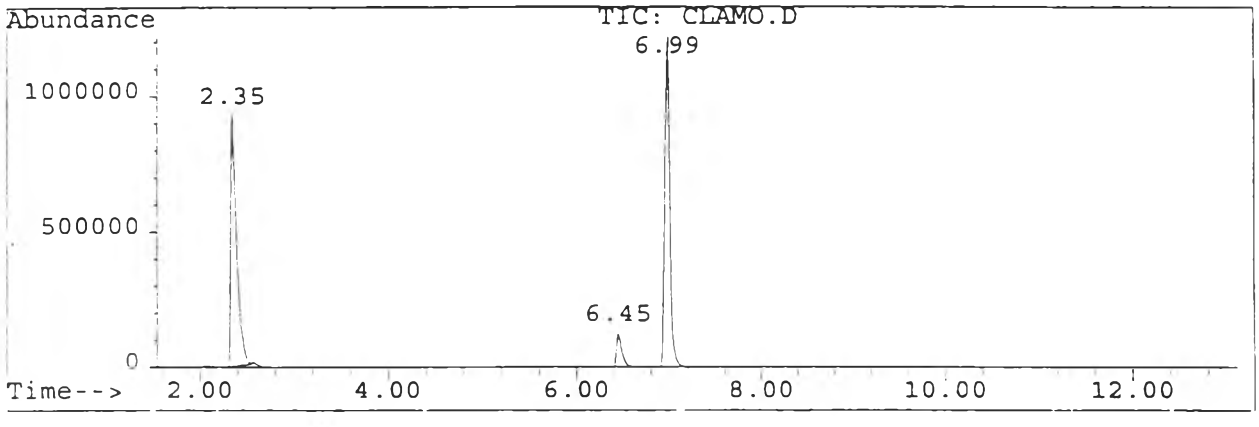


Figure 4.59 The gas chromatogram with MSD detection for headspace of Clamosin drug sample sample in aqueous solution.

GC/MSD Condition : described in Table 3.6

Library Searched : C:\DATABASE\WILEY138.L
Quality : 83
ID : Methane, dichloro-

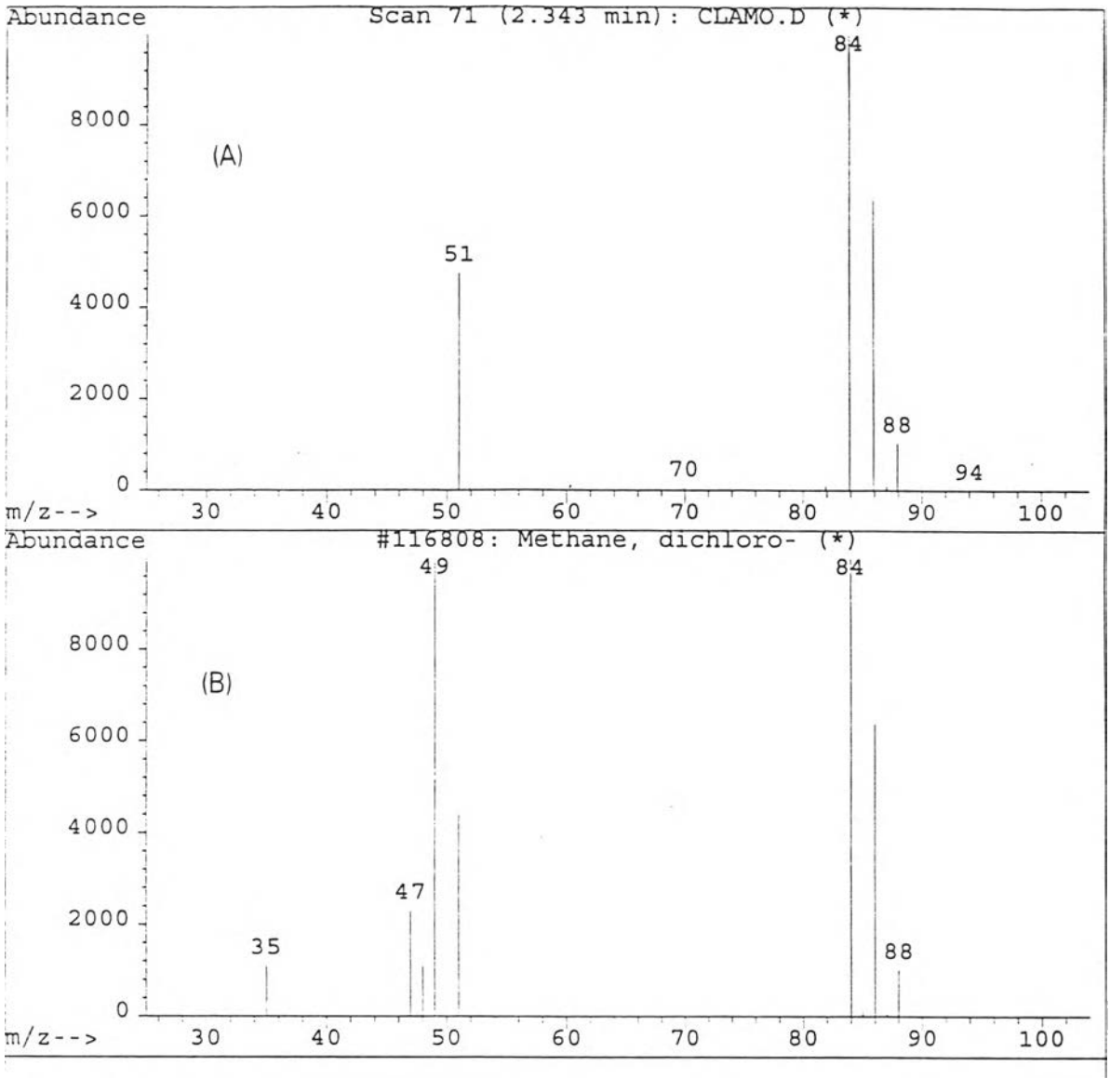


Figure 4.60 The mass spectrum for headspace of Clamosin drug sample with the retention time of 2.34 min. from the gas chromatogram in Figure 4.59

(A) Scan

(B) Library searched

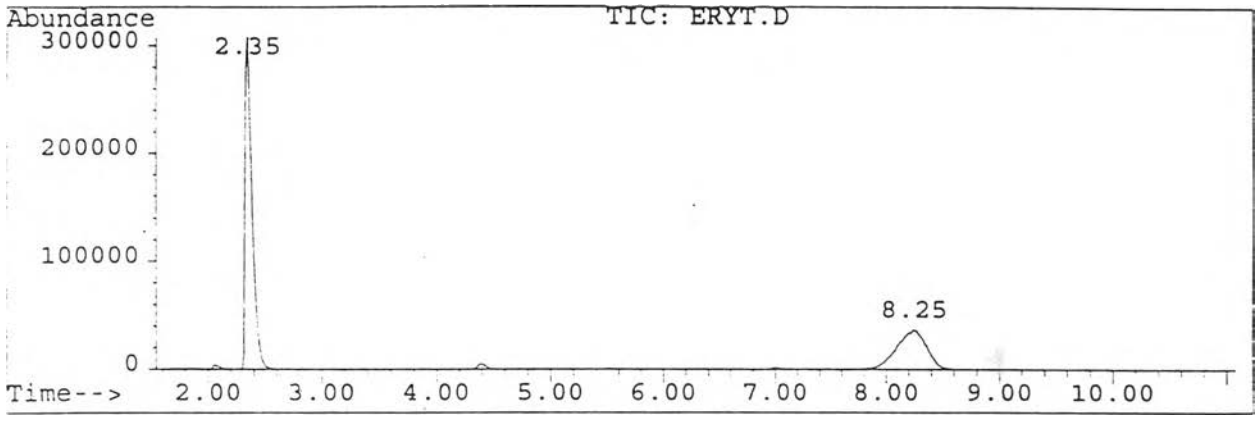


Figure 4.61 The gas chromatogram with MSD detection for headspace of Erythromycin (Pharmaceutical Organization) drug sample in aqueous solution.

GC/MSD Condition : described in Table 3.6

Library Searched : C:\DATABASE\WILEY138.L
Quality : 83
ID : Methane, dichloro-

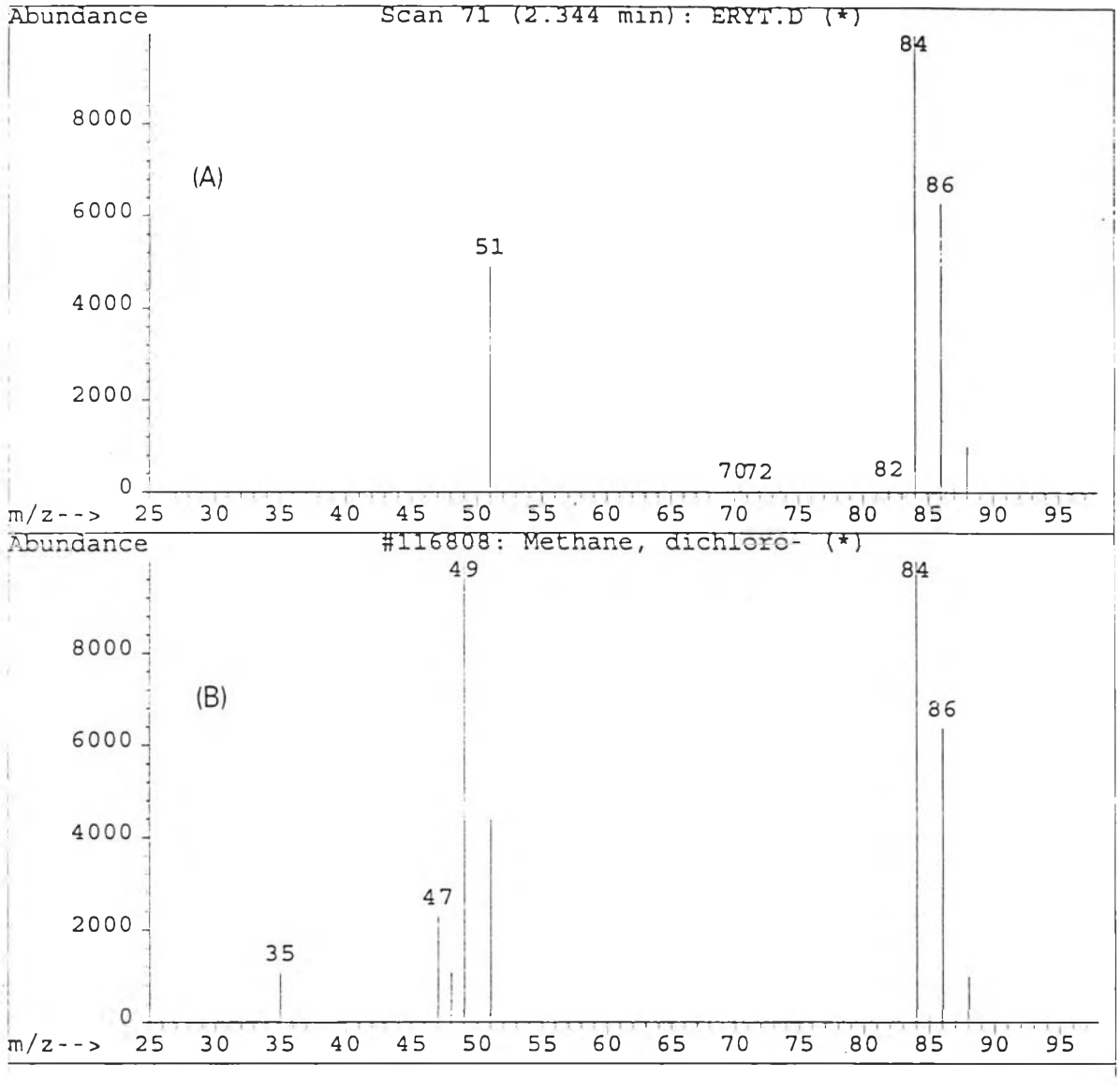


Figure 4.62 The mass spectrum for headspace of Erytromycin (Pharmaceutical Organization) drug sample with the retention time of 2.34 min. from the gas chromatogram in Figure 4.61

(A) Scan

(B) Library searched

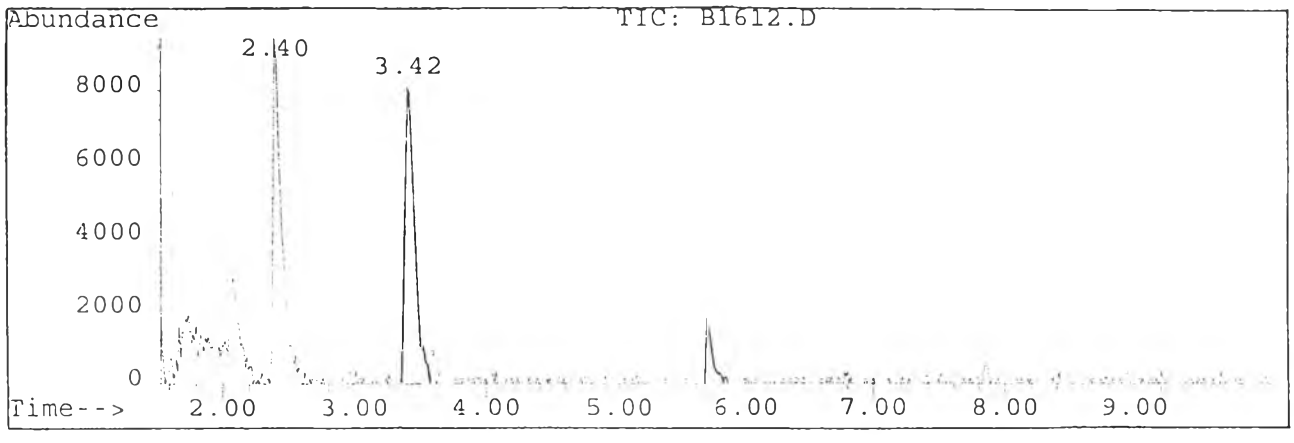


Figure 4.63 The gas chromatogram with MSD detection for headspace of Erytromycin (P.P. Lab Co.,Ltd.) drug sample in aqueous solution.

GC/MSD Condition : described in Table 3.6

Library Searched : C:\DATABASE\WILEY138.L
Quality : 4
ID : Chloroform

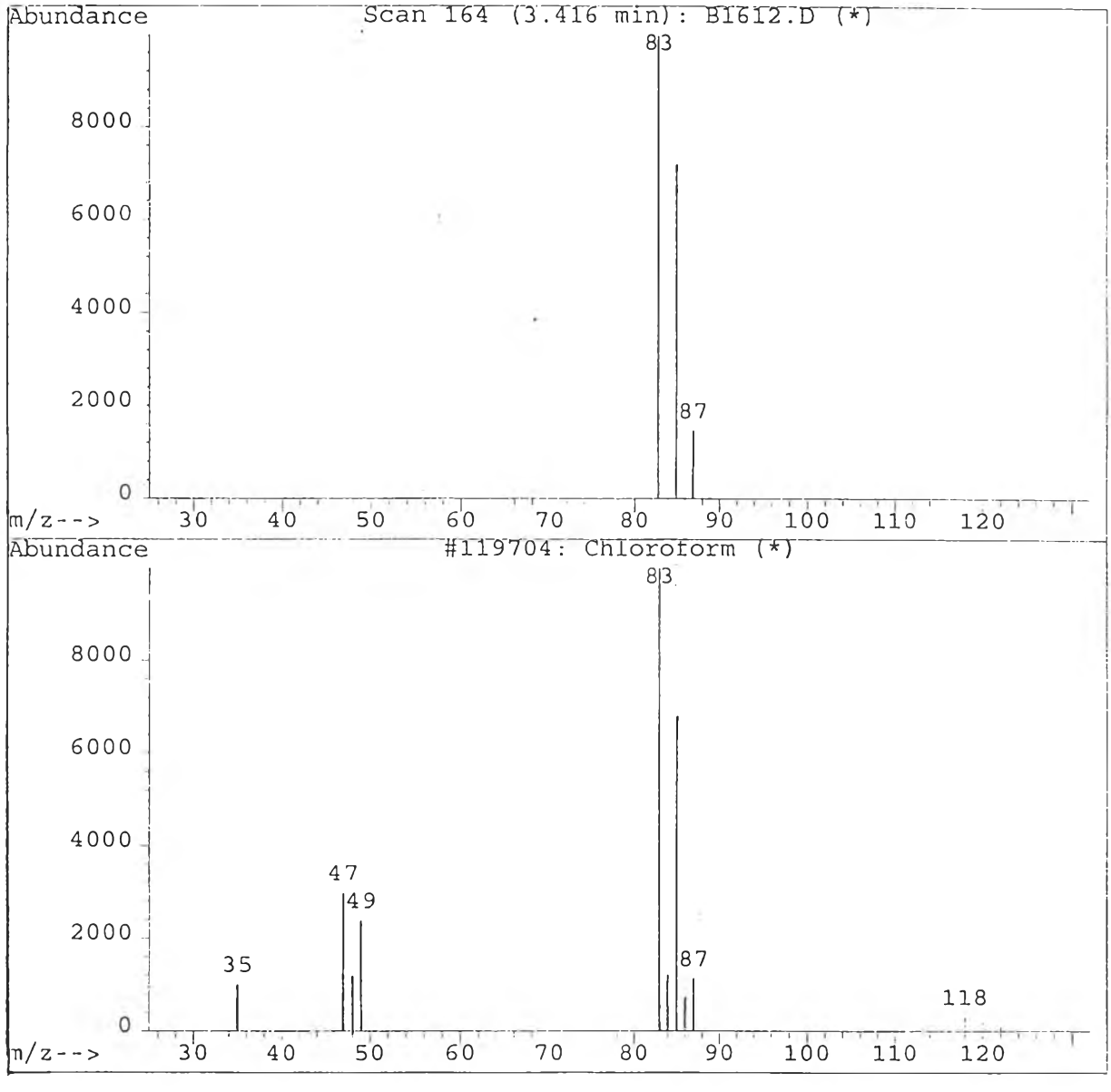


Figure 4.64 The mass spectrum for headspace of Erytromycin (P.P. Lab Co.,Ltd) drug sample with the retention time of 3.42 min. from the gas chromatogram in Figure 4.63.

(A) Scan

(B) Library searched