3 RESULTS

3.1 Milk Samples Analysis

3.1.1 Optimization of gas-chromatographic condition

In this work, temperature program was optimized and the other parameters were held constant .

Column : HP-608 (30m × 0.53mm I.D., 0.53 μm film thickness)

Injector : 250°C

Detector : 320°C

Injection : 1 μl of 10 μg l^{-1} standard PCBs mixture (PCBs No. 10, 28, 52, 138, 101, 153 and 180) in splitless mode

Carrier gas : Helium with flow rate 2.4 ml min^{-1}

Auxillary gas : Nitrogen

Temperature program :

EPA 608 : 80°C – 80°C (1 min) – 190°C (30°C min^{-1}) – 280°C (6°C min^{-1}) – 280°C (1 min) – 300°C (20°C min^{-1}) - 300°C (5 min)

Method A : 150°C – 150°C (1 min) – 280°C (7°C min^{-1}) – 280°C (7 min)

Method B : 160°C – 160°C (1 min) – 270°C (6°C min^{-1}) – 280°C (20°C min^{-1}) – 280°C (7 min)

Method C : 170°C – 170°C (1 min) – 265°C (5°C min^{-1}) – 280°C (20°C min^{-1}) – 280°C (7 min)

Method D: 180°C – 180°C (1 min) – 215°C (4°C min^{-1}) – 275°C (6°C min^{-1}) – 280°C (20°C min^{-1}) - 280°C (7 min)

Method E : 190°C – 190°C (4 min) – 215°C (5°C min^{-1}) – 280°C (6°C min^{-1}) – 280°C (7 min)

Retention times, peak areas and peak heights of PCBs obtained with various temperature program methods are tabulated in Tables 3.1, 3.2 and 3.3, respectively.

Table 3.1 Retention times (min) of PCBs obtained with various temperature program methods

PCBs No.	Retention time (min)					
	EPA 608	Method A	Method B	Method C	Method D	Method E
10	7.52	7.04	6.21	5.31	4.49	4.03
28	10.09	-	10.22	8.76	7.69	7.28
52	10.46	-	10.55	9.66	8.71	8.30
101	12.43	13.54	13.16	12.45	11.61	11.07
138	15.79	16.77	16.90	16.85	15.89	15.19
153	14.55	15.63	15.58	15.31	14.42	13.76
180	17.29	18.13	18.49	18.74	17.65	16.93

Table 3.2 Peak areas of PCBs obtained with various temperature program methods

PCBs No.	Peak area					
	EPA 608	Method A	Method B	Method C	Method D	Method E
10	12407	9601	11468	11126	11525	11574
28	6029	-	6488	9612	10338	13095
52	15956	-	15122	14826	17817	19622
101	13164	16322	18186	19639	22149	21932
138	12133	14874	15992	19261	19439	19239
153	14950	18384	18037	20011	21478	21280
180	15909	19921	22383	23460	23594	24031

Table 3.3 Peak heights of PCBs obtained with various temperature program methods

PCBs No.	Peak height					
	EPA 608	Method A	Method B	Method C	Method D	Method E
10	421	1463	1657	1736	1698	1520
28	256	-	226	320	665	1070
52	666	-	1045	1705	2111	2346
101	763	750	813	1072	1781	2463
138	622	883	863	985	1217	1608
153	732	919	943	1007	1387	1956
180	928	1289	1290	1311	1594	1902

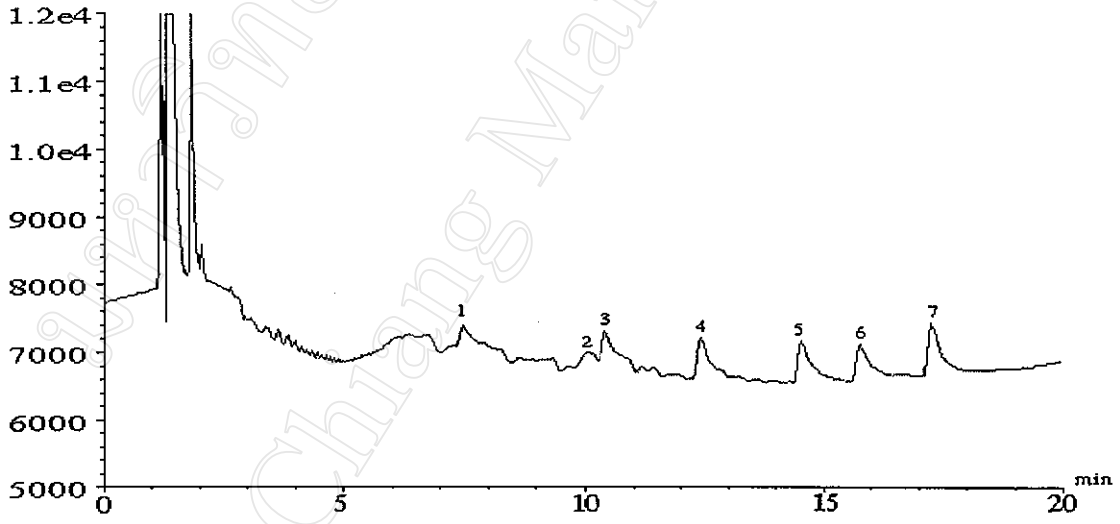

Figure 3.1 Chromatogram of 10 μg l^{-1} of PCBs mixture obtained with the EPA 608; 1, 2, 3, 4, 5, 6, 7 = PCBs No. 10, 28, 52, 101, 153, 138 and 180, respectively.

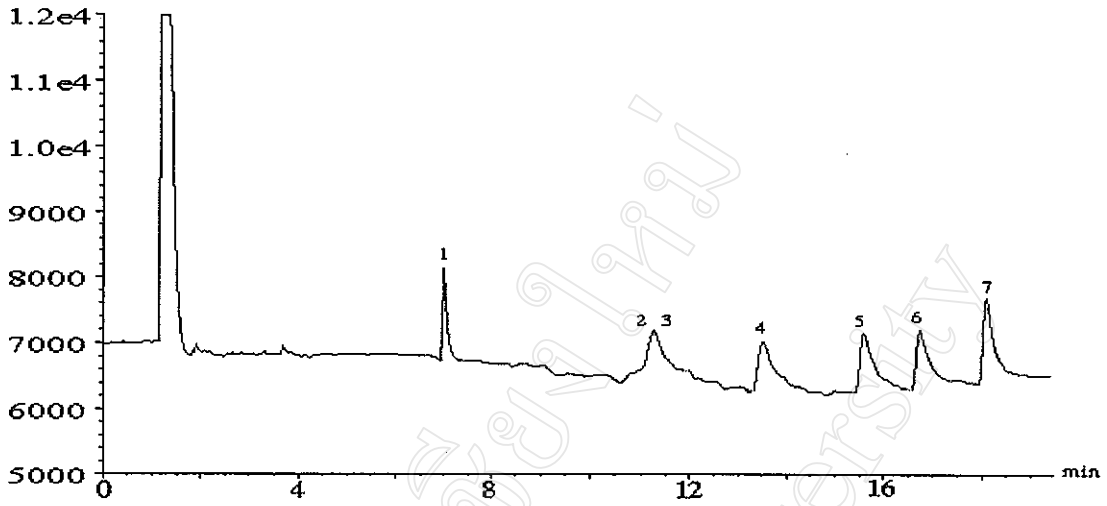

Figure 3.2 Chromatogram of 10 μg l^{-1} of PCBs mixture obtained with the temperature program method A; 1, 2, 3, 4, 5, 6, 7 = PCBs No. 10, 28, 52, 101, 153, 138 and 180, respectively.

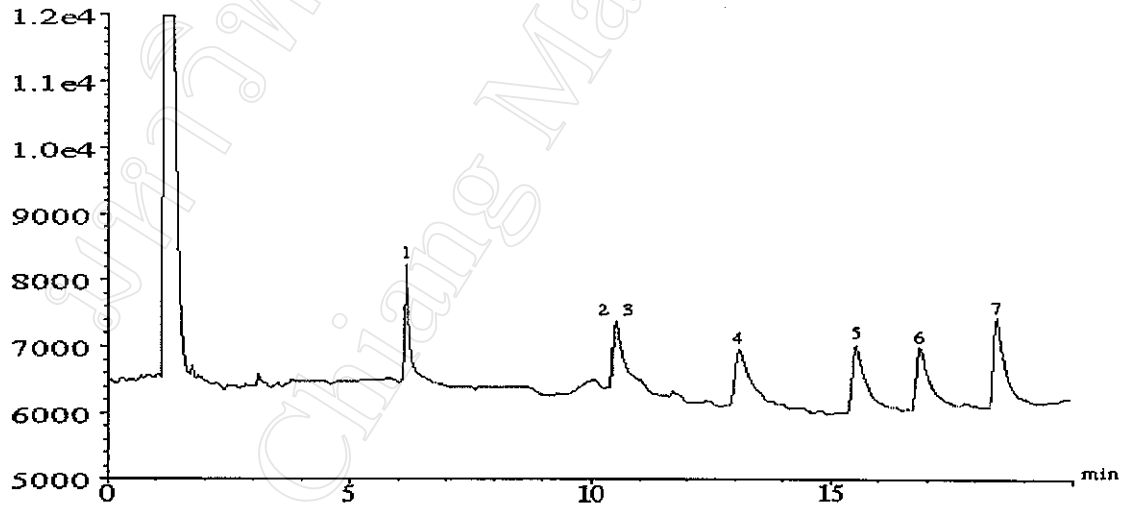

Figure 3.3 Chromatogram of 10 μg l^{-1} of PCBs mixture obtained with the temperature program method B ; 1, 2, 3, 4, 5, 6, 7 = PCBs No. 10, 28, 52, 101, 153, 138 and 180, respectively.

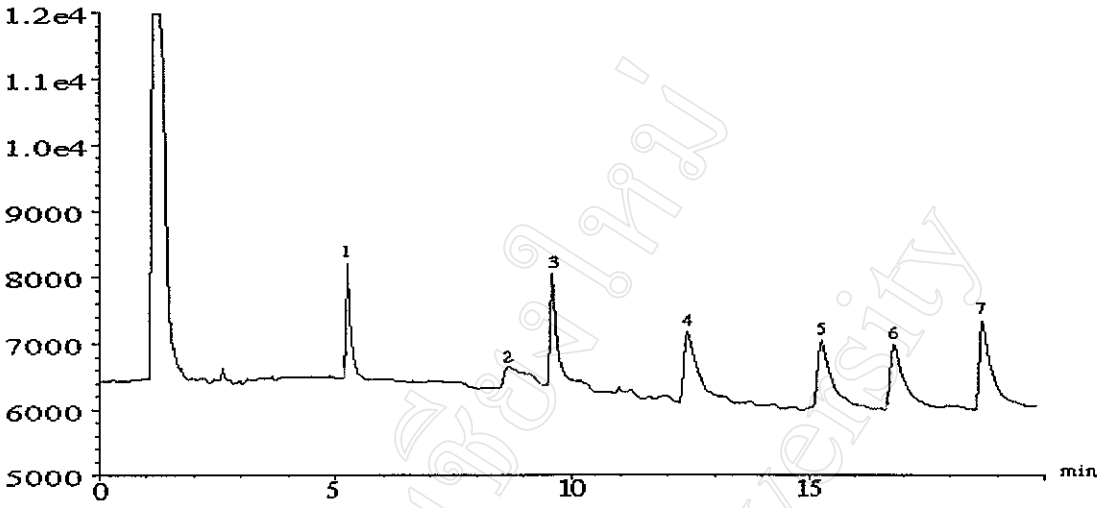

Figure 3.4 Chromatogram of 10 μg l^{-1} of PCBs mixture obtained with the temperature program method C ; 1, 2, 3, 4, 5, 6, 7 = PCBs No. 10, 28, 52, 101, 153, 138 and 180, respectively.

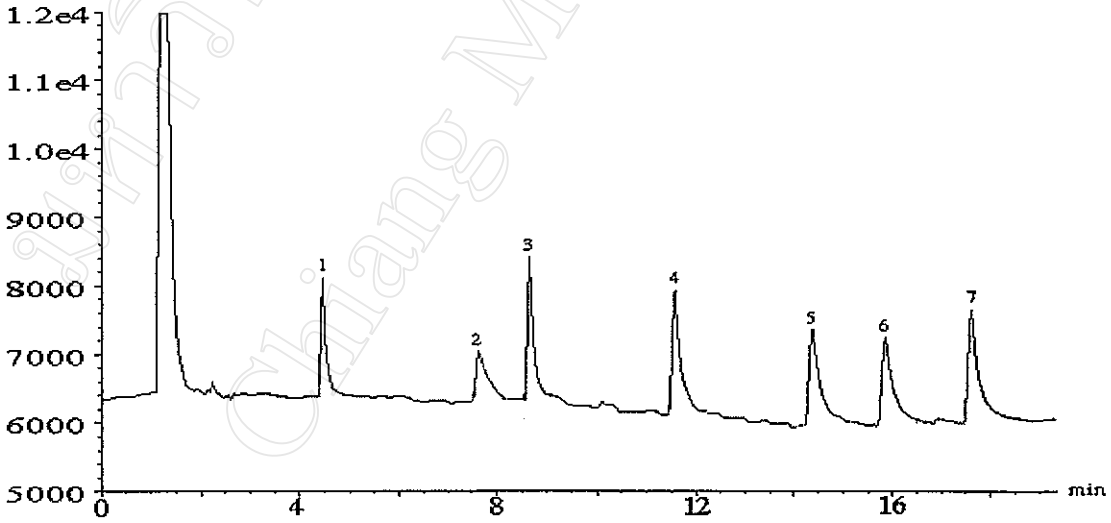

Figure 3.5 Chromatogram of 10 μg l^{-1} of PCBs mixture obtained with the temperature program method D; 1, 2, 3, 4, 5, 6, 7 = PCBs No. 10, 28, 52, 101, 153, 138 and 180, respectively.

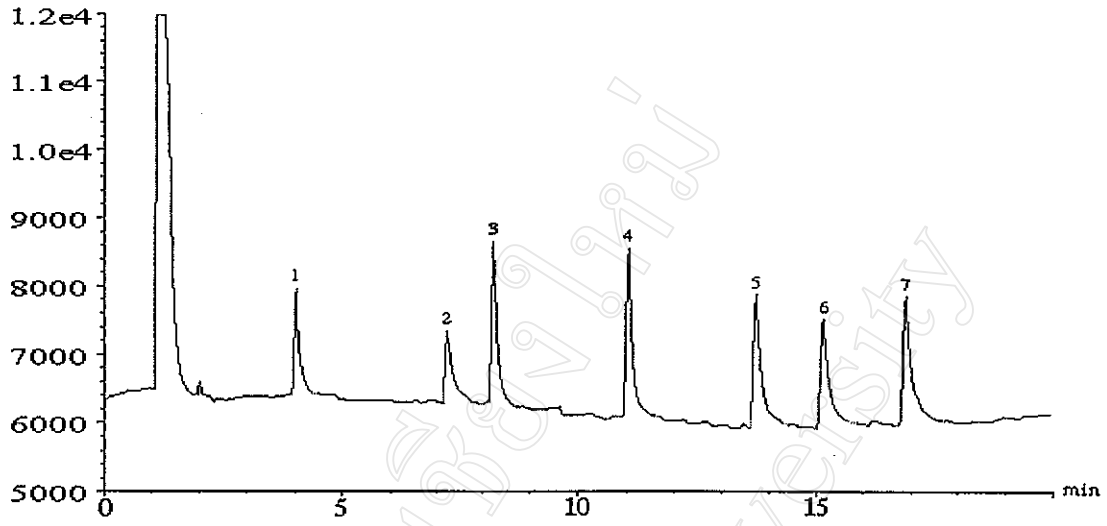

Figure 3.6 Chromatogram of 10 μg l^{-1} of PCBs mixture obtained with the temperature program method E; 1, 2, 3, 4, 5, 6, 7 = PCBs No. 10, 28, 52, 101, 153, 138 and 180, respectively.

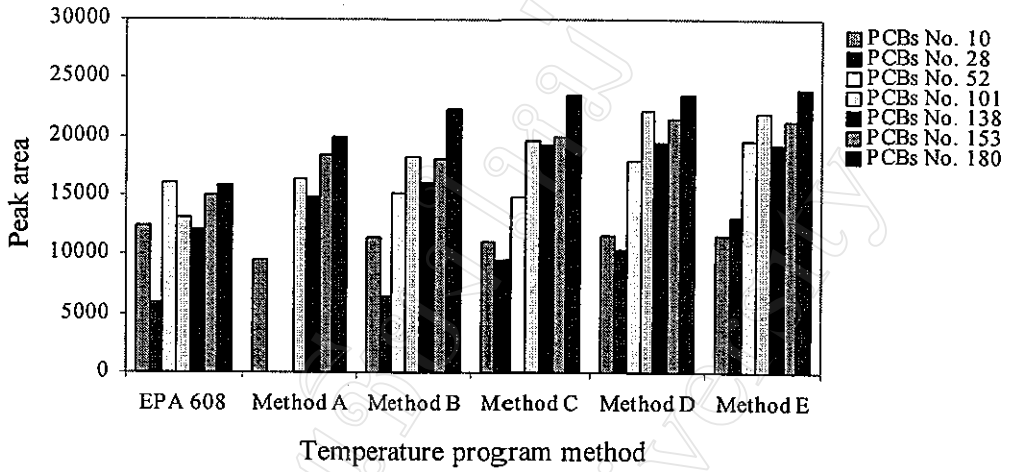

Figure 3.7 Effect of temperature program on peak areas of PCBs.

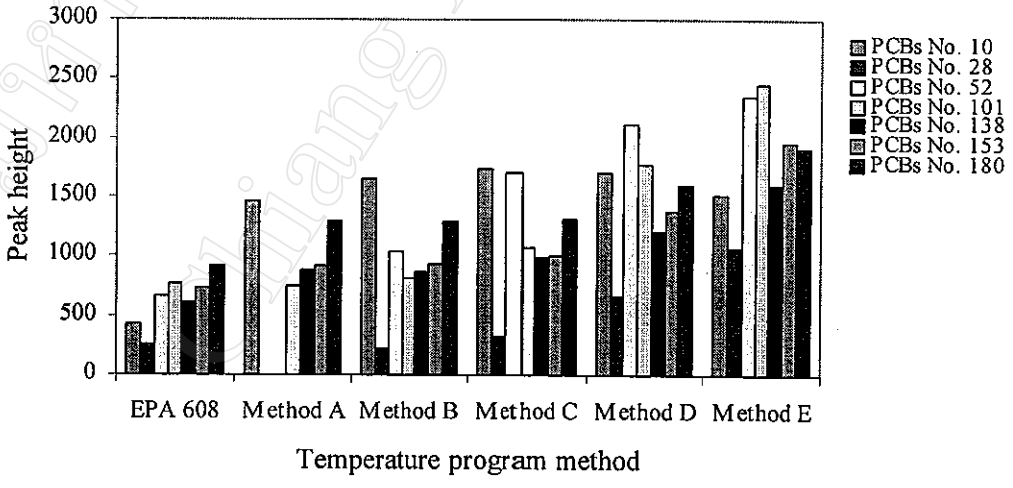

Figure 3.8 Effect of temperature program on peak heights of PCBs.

3.1.1.1 Limit of detection

Calculation of the limit of detection was based on a method of Miller and Miller.[104] The external calibration curve was constructed from peak areas and PCBs concentrations in the range of 0.5 – 7 μg l^{-1}.

The temperature program method E was used. The injection volume was 1 μl in splitless mode. Calibration curves of PCBs No. 10, 28, 52, 101, 138, 153 and 180 are shown in Figures 3.9, 3.10, 3.11, 3.12, 3.13, 3.14 and 3.15, respectively.

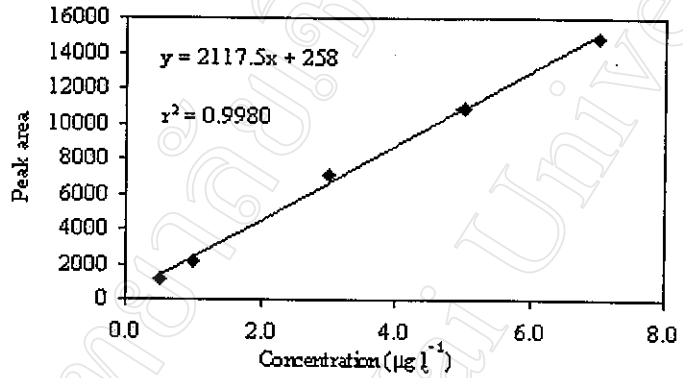

Figure 3.9 Calibration curve of PCB No. 10 in the range of 0.5 – 7 μg l^{-1}.

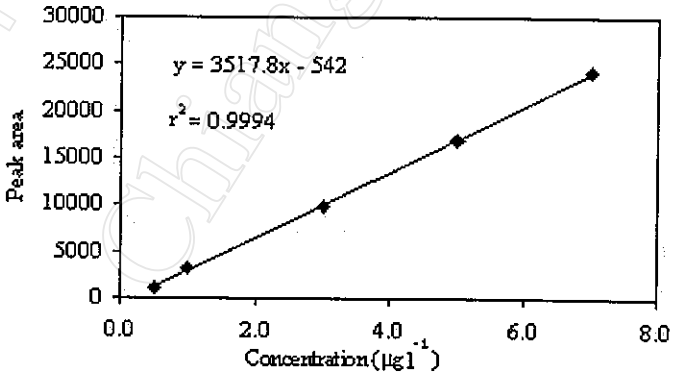

Figure 3.10 Calibration curve of PCB No. 28 in the range of 0.5 – 7 μg l^{-1}.

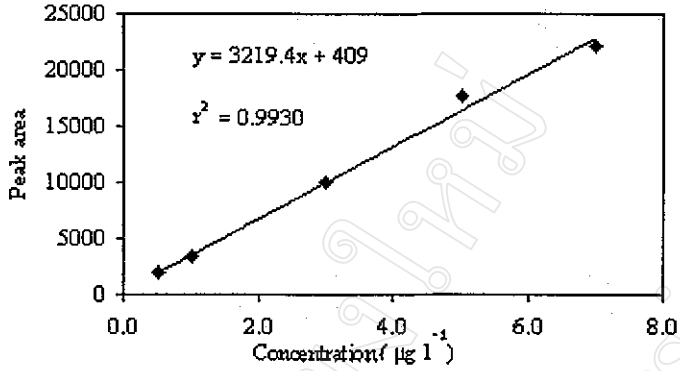

Figure 3.11 Calibration curve of PCB No. 52 in the range of 0.5 – 7 µg l^{-1}.

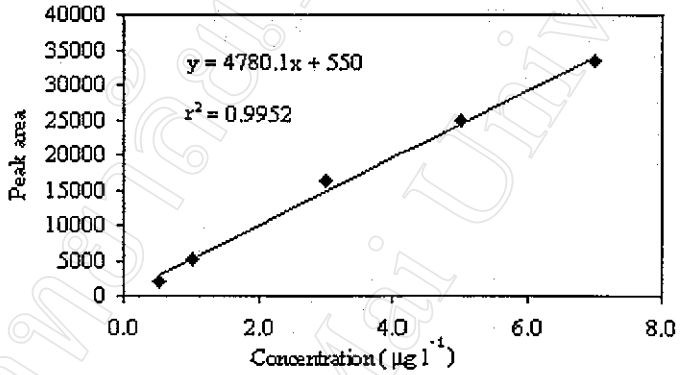

Figure 3.12 Calibration curve of PCB No. 101 in the range of 0.5 – 7 µg l^{-1}.

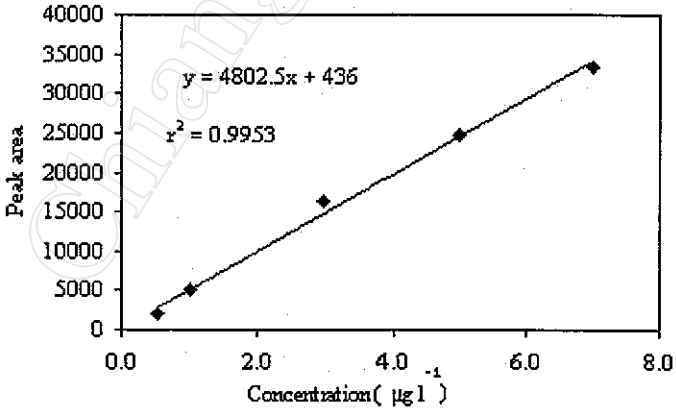

Figure 3.13 Calibration curve of PCB No. 138 in the range of 0.5 – 7 µg l^{-1}.

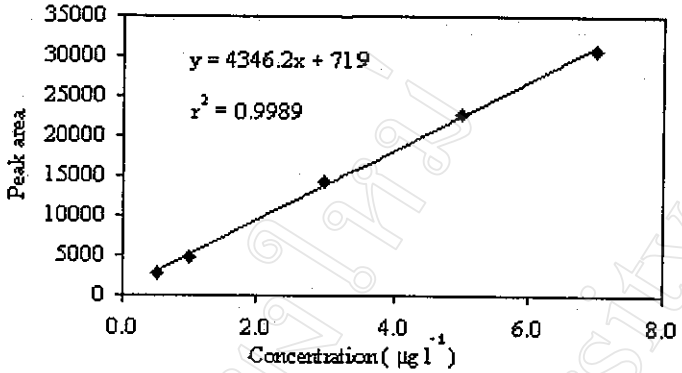

Figure 3.14 Calibration curve of PCB No. 153 in the range of 0.5 – 7 µg l^{-1}.

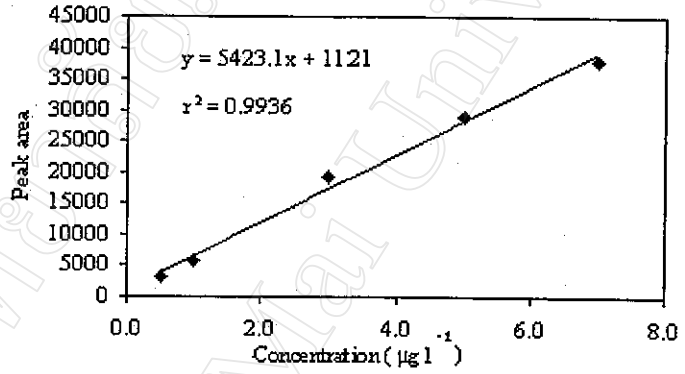

Figure 3.15 Calibration curve of PCB No. 180 in the range of 0.5 – 7 µg l^{-1}.

Table 3.4 Limit of detection of PCBs in milk sample analysis

PCBs No.	Limit of detection (µg l^{-1})
10	0.42
28	0.22
52	0.79
101	0.90
138	0.65
153	0.31
180	0.76

3.1.1.2 Summary of optimized GC conditions

The optimized GC conditions established in this work are listed in Table 3.5.

Table 3.5 Optimized GC conditions with HP-608, 30m × 0.53mm I.D., 0.53μm film thickness

Operation	Optimal conditions
Temperature program	190°C – 190°C (4 min) – 215°C (5°C min^{-1}) – 280°C (6°C min^{-1}) – 280°C (7 min)
Carrier gas	Helium with flow rate 2.4 ml min^{-1}
Auxilliary gas	Nitrogen
Injector temperature	250°C
Detector temperature	320°C
Injection mode	Splitless, 1 μl injection volume

3.1.2 Optimization of solid phase extraction

3.1.2.1 Study of the volume of water added to the standard solution PCBs

The solutions A, B and C containing 10, 20 and 30 ml of water respectively, were added to each C_{18} SPE column. The percent recovery of each set of experiment was calculated, with an assumption that 100% recovery is equal to 10 μg l^{-1} of each PCB. The results are shown in Table 3.6.

Table 3.6 Effect of water added to the standard solution on the percent recoveries of PCBs, based on three determinations

PCBs No.	Solution A Recovery(%), (R.S.D.%)	Solution B Recovery(%), (R.S.D.%)	Solution C Recovery(%), (R.S.D.%)
10	50<	51(25.5)	50<
28	83(4.0)	82(10.0)	77(5.3)
52	91(4.0)	98(4.7)	89(3.1)
101	101(3.6)	105(6.1)	98(5.1)
138	103(2.6)	103(4.8)	103(2.7)
153	99(5.4)	100(3.7)	99(3.3)
180	102(4.7)	102(3.2)	98(1.8)

3.1.2.2 Study of the effect of conditioned solution to the percent recoveries of PCBs

The solvent mixtures of the solution A, B and C were initially used in the conditioning step of SPE. The percent recovery of each set of experiment was calculated (100% recovery = 10 $\mu g\ l^{-1}$ of each PCB). The results are shown in Table 3.7.

Table 3.7 Effect of using solvent mixture of the solution A, B and C additionally in the conditioning step of SPE on the percent recoveries of PCBs, based on three determinations

PCBs No.	Solvent mixture of Solution A Recovery(%), (R.S.D.%)	Solvent mixture of Solution B Recovery(%), (R.S.D.%)	Solvent mixture of Solution C Recovery(%), (R.S.D.%)
10	<50	78(4.2)	69(11.1)
28	82(1.0)	95(1.7)	83(2.2)
52	93(2.5)	98(5.3	90(1.2)
101	95(2.3)	100(2.0)	92(1.0)
138	103(1.2)	106(1.6)	95(0.8)
153	102(0.5)	100(1.9)	94(0.3)
180	103(0.9)	104(1.6)	89(2.8)

3.1.2.3 Study of the drying step to the percent recoveries of PCBs

The standard PCBs mixture was directly loaded onto the C_{18} SPE column and dried for about 30 min. The percent recovery was calculated (100%recovery = 10 $\mu g\ l^{-1}$ of each PCB). The results are shown in Table 3.8.

Table 3.8 Effect of drying step on the percent recoveries of PCBs, based on three determinations

PCBs No.	Recovery(%), (R.S.D. %)
10	101(4.4)
28	98(7.4)
52	100(5.3)
101	100(2.5)
138	101(4.1)
153	98(2.9)
180	100(4.3)

3.1.2.4 Investigation of optimal elution volume

The solution B was loaded onto the C_{18} SPE and eluted with 4×1 ml of isooctane. Then each fraction was injected into GC/ECD and peak area was recorded. The results are shown in Table 3.9.

Table 3.9 Results on the investigation of optimal elution volume, based on three determinations

PCBs No.	Peak area			
	Fraction 1	Fraction 2	Fraction 3	Fraction 4
10	39536	2878	-	-
28	84871	3061	-	-
52	90012	4688	-	-
101	132214	6627	-	-
138	148519	5607	-	-
153	128219	6017	-	-
180	174059	6786	-	-

3.1.2.5 Summary of optimized SPE conditions

The optimized extraction conditions established in this work are shown in Table 3.10.

Table 3.10 Optimized SPE conditions for milk samples analysis in this work

Operation	**Optimal extraction conditions**
1. SPE column	8 ml glass column with polyethylene frits
2. Sorbent	500 mg of C_{18}
3. Conditioning solvent	5 ml of dichrolomethane, 5 ml of methanol and 5 ml of solvent mixture, water : ethyl acetate : acetone : methanol (10:1:2:2 v/v), loaded with flow rate about 1-2 ml min^{-1}
4. Loading	Flow rate about 2-3 ml min^{-1}
5. Drying	30 min with air pulled
6. Elution	2 ml of isooctane with gravity flow rate

3.1.3 Investigation of the number of extraction in milk sample

The number of extraction was investigated with using of spiked cow's milk at 10 μg l^{-1}. The extracting solvent was ethylacetate : acetone : methanol (1:2:2 v/v). A volume of 10 ml of this solvent mixture was used in a single time extraction and a volume of 10 ml (1^{st} extraction) and 5 ml (2^{nd} extraction) of this solvent mixture was used in the subsequent double extraction. The percent recoveries of PCBs were calculated and the results are shown in Table 3.11.

Table 3.11 The effect of a single and double extraction of spiked cow's milk sample (10 μg l^{-1}) on the percent recoveries of PCBs, based on three determinations

PCBs No.	Recovery(%), (R.S.D.%)	
	One extraction	Double extraction
10	<50	63(4.7)
28	76(13.5)	88(6.9)
52	78(3.5)	94(2.9)
101	65(1.2)	84(3.7)
138	78(1.9)	100(3.3)
153	75(3.7)	91(2.3)
180	75(1.8)	91(1.3)

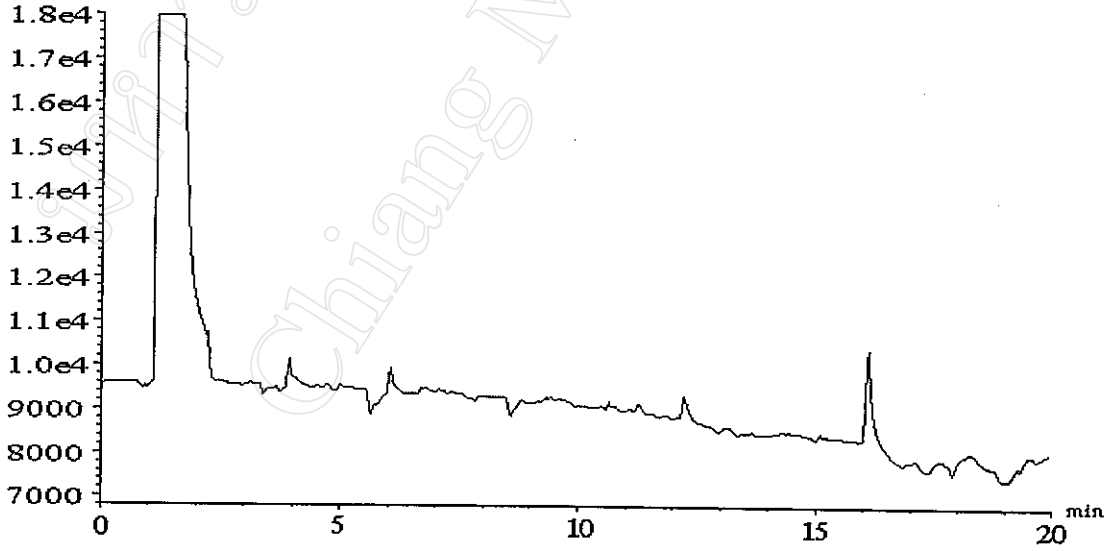

Figure 3.16 Chromatogram of nonspiked cow's milk used as blank solution for double extraction of 10 μg l^{-1} spiked cow's milk.

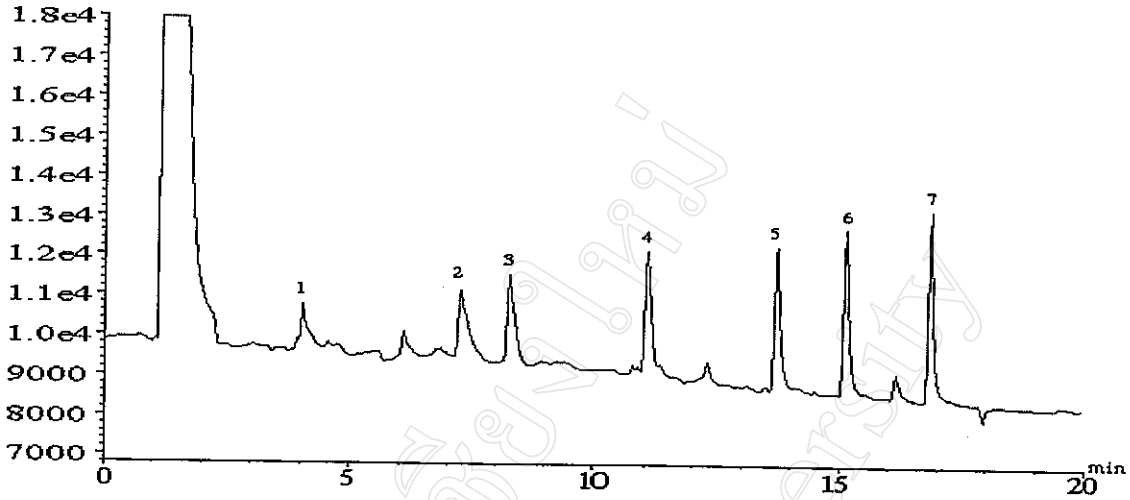

Figure 3.17 Chromatogram of spiked cow's milk at 10 μg l^{-1} for double extraction; 1, 2, 3, 4, 5, 6, 7 = PCBs No. 10, 28, 52, 101, 153, 138 and 180, respectively.

3.1.4 Summary of optimal extraction condition in milk sample analysis

The milk samples were subjected to double extraction with solvent mixtures, ethylacetate: acetone: hexane (1:2:2 v/v). Then the extract was mixed with water and introduced to C_{18} SPE. Isooctane was used as the eluent. The eluate was injected into GC/ECD.

3.1.5 Determination of fat content

The fat of cow's and powdered milk used in this work was extracted with hexane: acetone (1:1 v/v). The fat contents were calculated as percent weight by volume and weight by weight as shown in Table 3.12.

Table 3.12 The percent of fat content in cow's and powdered milk, based on three determinations

Milk	Percent (%R.S.D.)
Cow's milk	3.47(0.7) (w/v)
Powdered milk	25.76(4.4) (w/w)

3.1.6 Recovery assay

The percent recoveries were studied in both cow's and powdered milk at two levels. In cow's milk, the spiked level was 10 and 1.4 μg l^{-1}. In powdered milk, the spiked level was 74.1 and 10.4 μg kg^{-1}. Both samples were subjected to double extraction. The results are shown in Table 3.13.

Table 3.13 The percent recoveries of spiked cow's and powdered milk, based on three determinations

PCBs No.	Recoveries (%),(R.S.D.%)			
	Cow's milk spiked level (μg l^{-1})		Powdered milk spiked level (μg kg^{-1})	
	10	1.4	74.1	10.4
10	63(4.7)	ND	ND	ND
28	88(6.9)	<36	<50	<36
52	94(2.9)	36(11.2)	<50	<36
101	84(3.7)	53(5.7)	<50	57(3.7)
138	100(3.3)	54(5.0)	74(6.4)	59(7.6)
153	91(2.3)	57(2.8)	67(7.3)	62(6.8)
180	92(1.3)	64(5.5)	77(7.0)	65(10.5)

ND = not detected

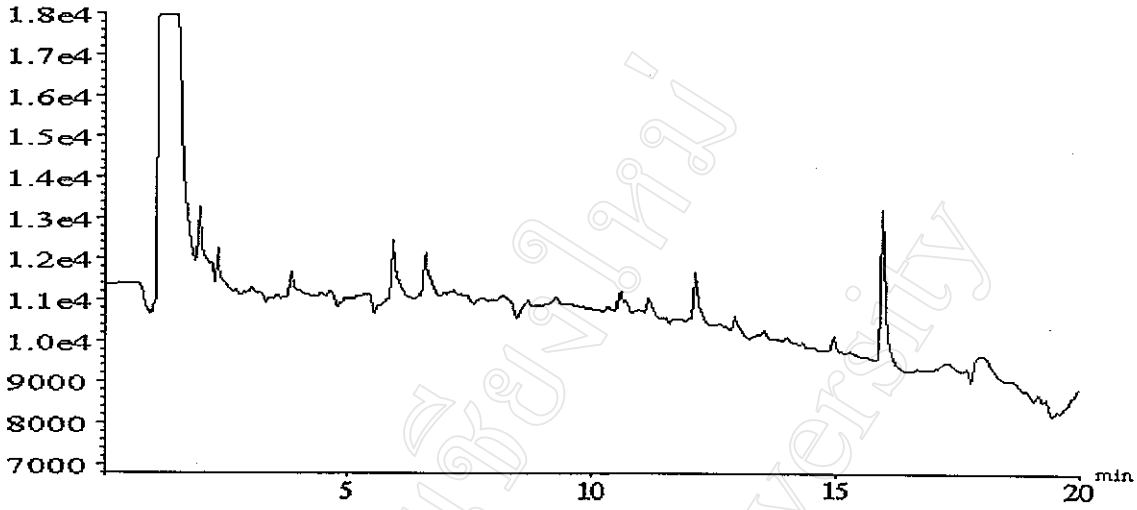

Figure 3.18 Chromatogram of nonspiked cow's milk used as blank solution for double extraction of 1.4 μg l^{-1} spiked cow's milk.

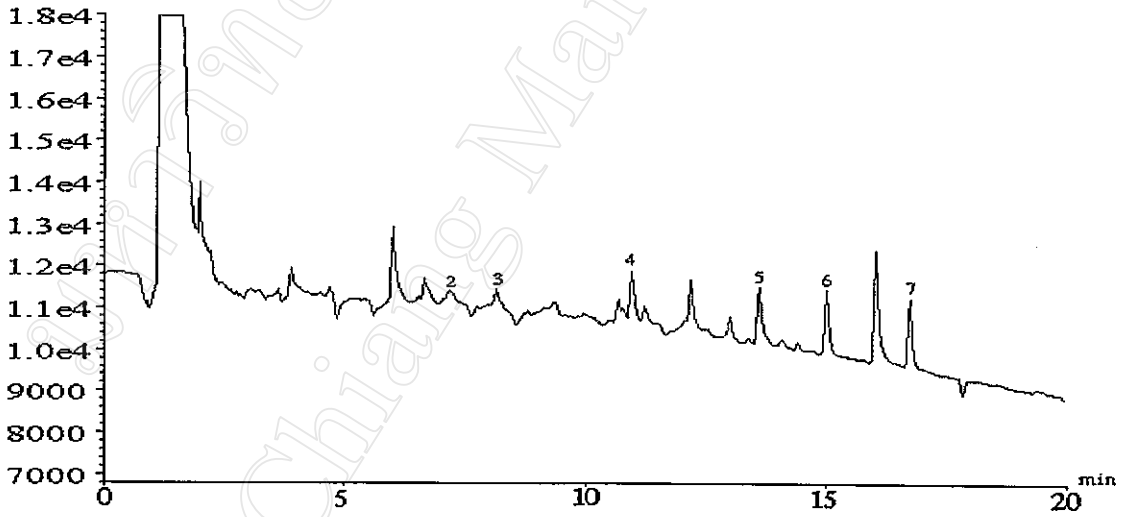

Figure 3.19 Chromatogram of spiked cow's milk at 1.4 μg l^{-1} in double extraction; 2, 3, 4, 5, 6, 7 = PCBs No. 28, 52, 101, 153, 138 and 180, respectively.

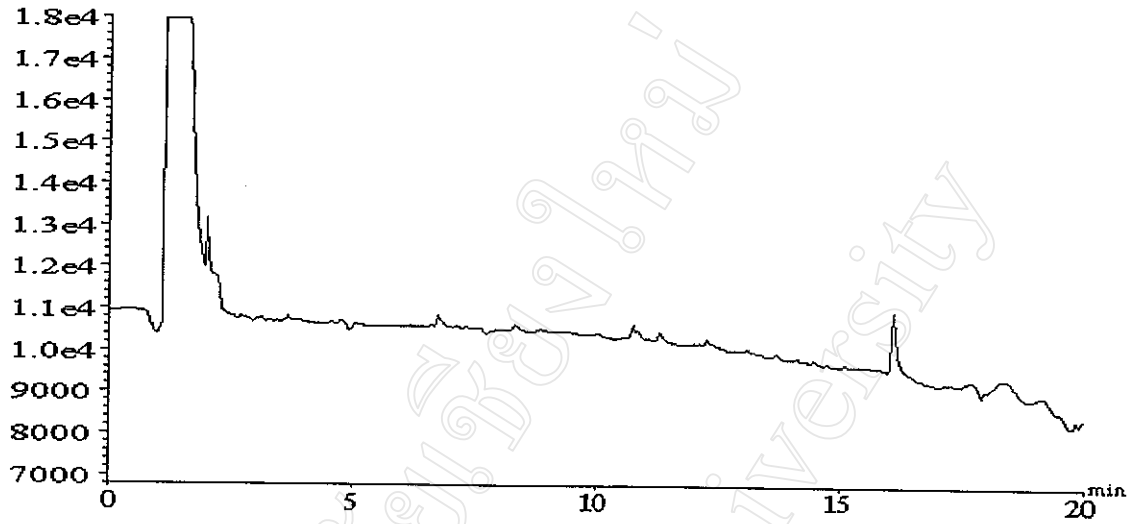

Figure 3.20 Chromatogram of nonspiked powdered milk used as blank solution for double extraction of 74.1 μg kg^{-1} spiked powdered milk.

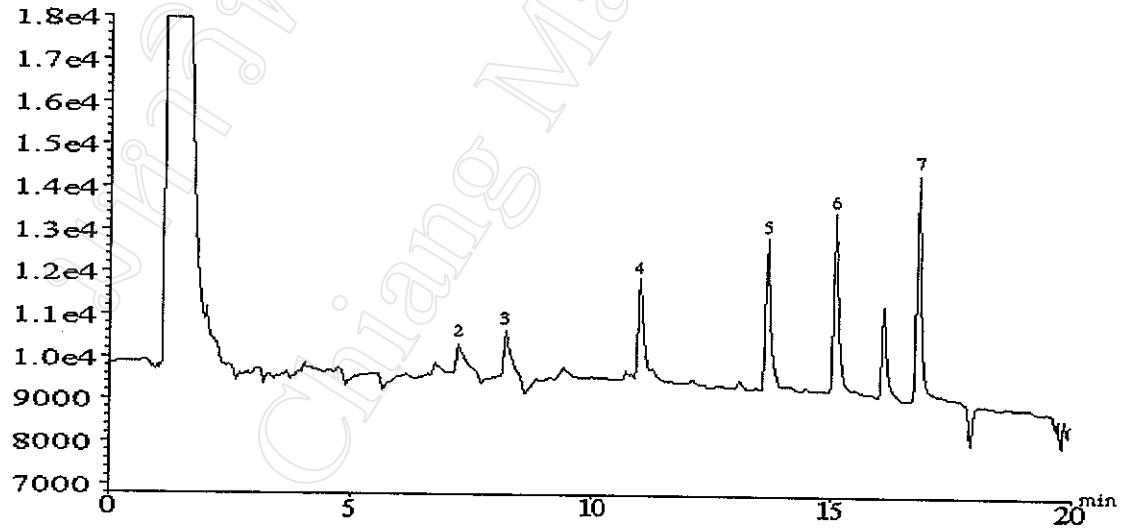

Figure 3.21 Chromatogram of spiked powdered milk at 74.1 μg kg^{-1} in double extraction; 2, 3, 4, 5, 6, 7 = PCBs No. 28, 52, 101, 153, 138 and 180, respectively.

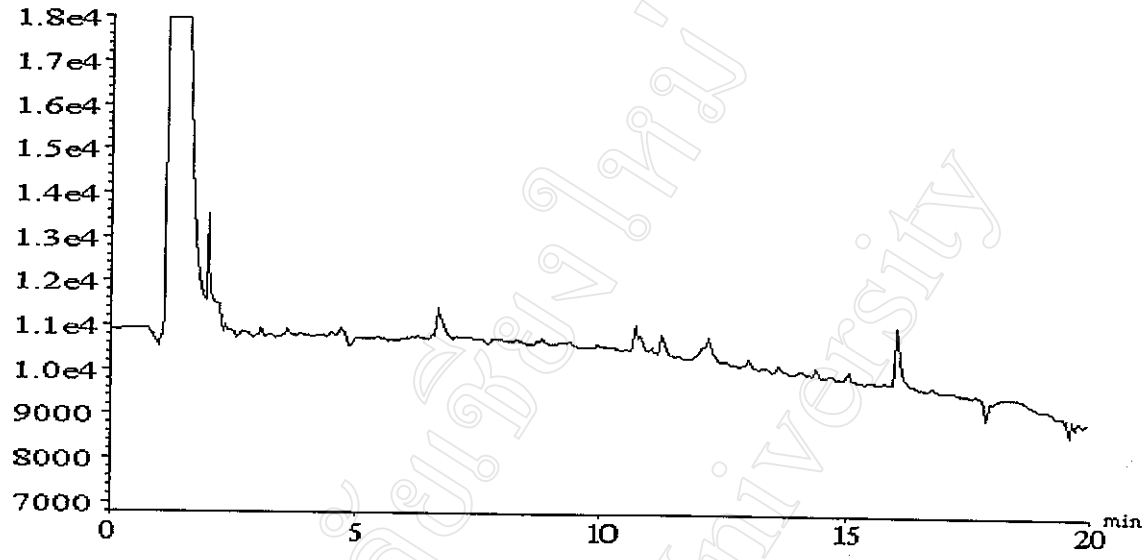

Figure 3.22 Chromatogram of nonspiked powdered milk used as blank solution for double extraction of 10.4 μg kg^{-1} spiked powdered milk.

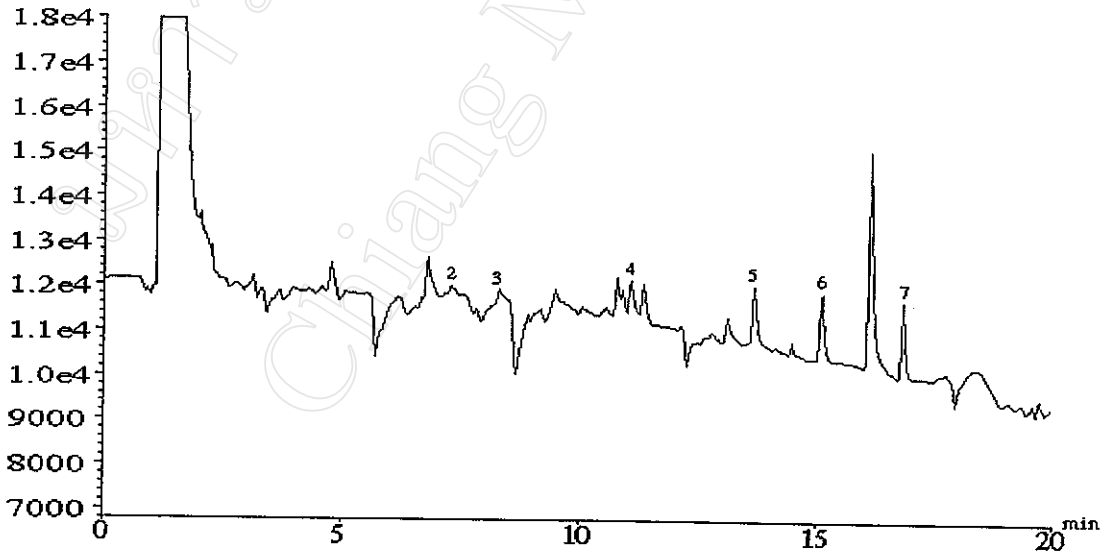

Figure 3.23 Chromatogram of spiked powdered milk at 10.4 μg kg^{-1} in double extraction; 2, 3, 4, 5, 6, 7 = PCBs No. 28, 52, 101, 153, 138 and 180, respectively.

3.1.7 Analysis of samples

In this work, one brand of cow's milk and powdered milk was chosen. There was no PCBs detection study in each sample. The chromatogram of cow's and powdered milk samples are shown in Figure 3.18 and 3.22, respectively.

3.2 Water Sample Analysis

3.2.1 Optimization of gas chromatographic condition

The temperature program was slightly optimized and other parameters were held constant.

Column : DB-1701 (30m × 0.25mm. I.D., 0.25 μm film thickness)

Injector : 250°C

Detector 320°C

Injection : 2 μl of 1 μg l^{-1} standard PCB mixture (PCBs No. 10, 28 52, 101, 138, 153 and 180) in splitless mode

Carrier gas : Helium with flow rate 1.0 ml min^{-1}

Auxillary gas : Nitrogen

Temperature program :

Method F : 100°C - 100°C (1 min) – 250°C (6°C min^{-1}) – 250°C (10 min)

Method G: 100°C - 100°C (1 min) – 235°C (10°C min^{-1}) - 235°C (10 min) - 250°C (5°C min^{-1}) – 250°C (7 min)

Method H: 100°C - 100°C (1 min) – 225°C (10°C min^{-1}) - 225°C (15min) - 250°C (5 °C min^{-1}) – 250°C (7 min)

Method I: 100°C - 100°C (1 min) – 190°C (10°C min^{-1}) - 250°C (5°C min^{-1}) – 250°C (10 min)

Retention times of PCBs obtained with various temperature program methods are listed in Table 3.14. A typical chromatogram of 1.0 μg l^{-1} of PCBs mixture obtained with the temperature program method I is given in Figure 3.24.

Retention times of PCBs obtained with various temperature program methods are listed in Table 3.14. A typical chromatogram of 1.0 μg l^{-1} of PCBs mixture obtained with the temperature program method I is given in Figure 3.24.

Table 3.14 Retention times (min) of PCBs obtained with various temperature program methods

PCBs No.	Retention time (min)			
	Method F	**Method G**	**Method H**	**Method I**
10	15.83	11.66	11.66	11.73
28	20.37	14.45	14.52	15.42
52	21.51	15.16	15.40	16.47
101	24.21	17.37	18.29	19.20
138	28.07	22.49	25.45	23.23
153	26.82	20.63	22.81	21.99
180	30.99	26.75	31.48	26.15

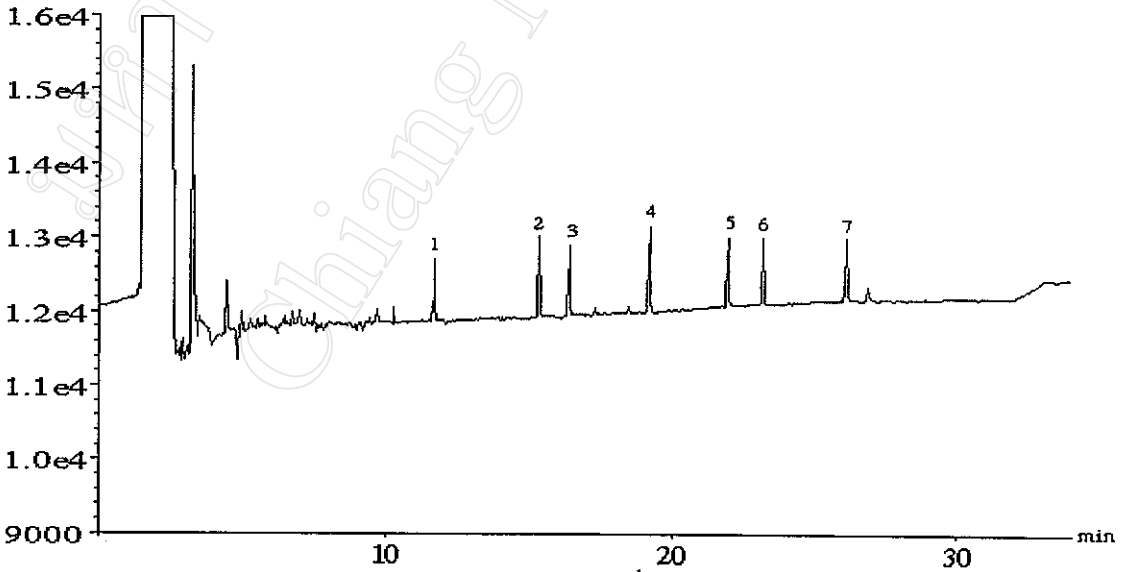

Figure 3.24 Chromatogram of 1.0 μg l^{-1} of PCBs mixture obtained with the temperature program method I; 1, 2, 3, 4, 5, 6, 7 = PCBs No. 10, 28, 52, 101, 153, 138 and 180, respectively.

3.2.1.1 Limit of detection

Calculation of the limit of detection was based on a method of Miller and Miller.[104] The external calibration curve was constructed from peak areas and PCBs concentrations in the range of 0.1-1.5 μg l^{-1}. The temperature program method I was used. The injection volume was 2 μl in splitless mode. Calibration curves of PCBs No. 10, 28, 52, 101, 138, 153 and 180 are shown in Figures 3.25, 3.26, 3.27, 3.28, 3.29, 3.30 and 3.31, respectively. Limit of detection of PCBs in water sample analysis are listed in Table 3.15.

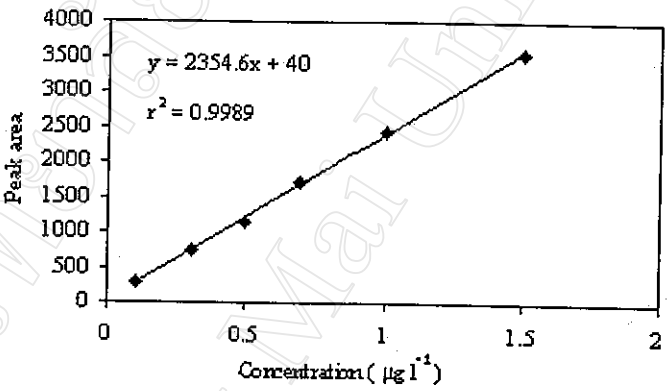

Figure 3.25 Calibration curve of PCB No. 10 in the range of 0.1 –1.5 μg l^{-1}.

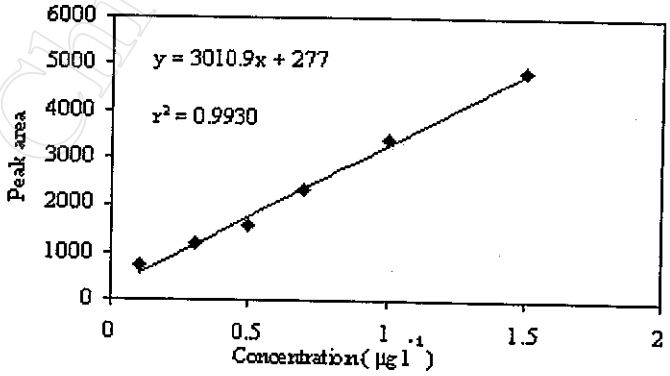

Figure 3.26 Calibration curve of PCB No. 28 in the range of 0.1 – 1.5 μg l^{-1}.

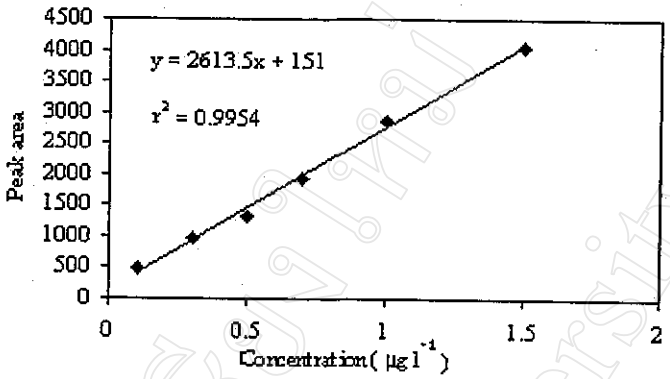

Figure 3.27 Calibration curve of PCB No. 52 in the range of 0.1 – 1.5 µg l^{-1}.

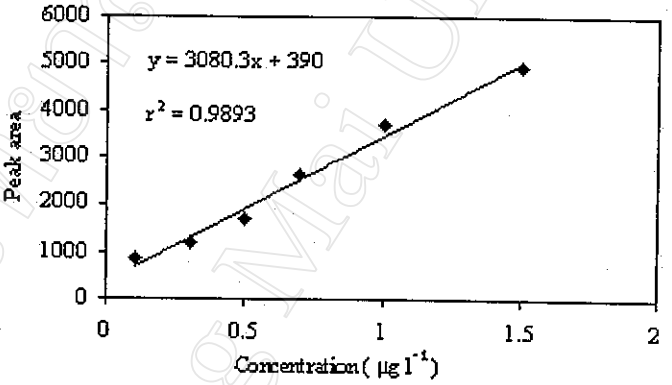

Figure 3.28 Calibration curve of PCB No. 101 in the range of 0.1 –1.5 µg l^{-1}.

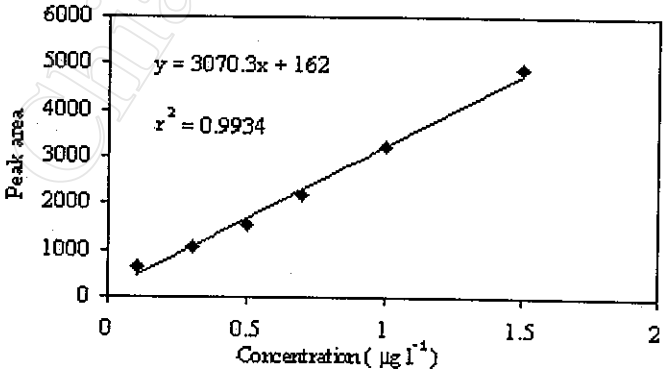

Figure 3.29 Calibration curve of PCB No. 138 in the range of 0.1 –1.5 µg l^{-1}.

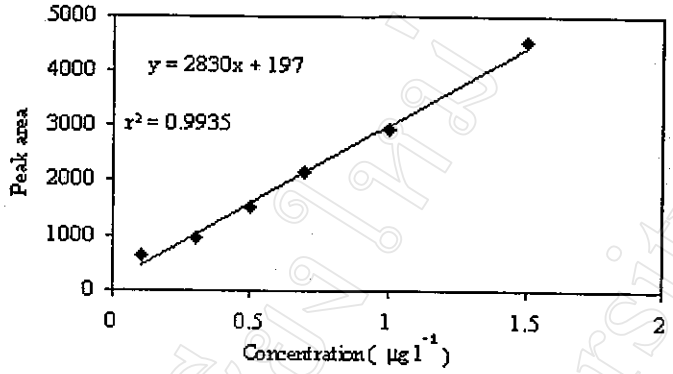

Figure 3.30 Calibration curve of PCB No. 153 in the range of 0.1 –1.5 µg l^{-1}.

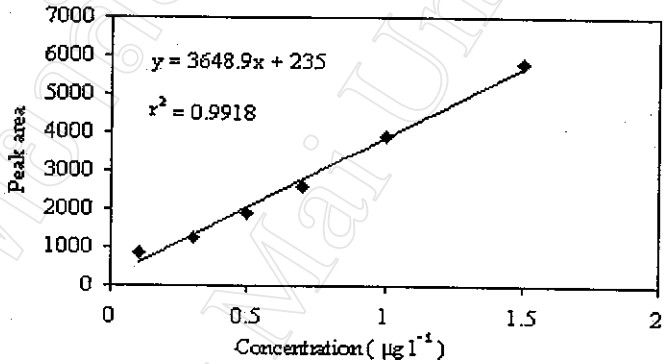

Figure 3.31 Calibration curve of PCB No. 180 in the range of 0.1 –1.5 µg l^{-1}.

Table 3.15 Limit of detection of PCBs in water sample analysis

PCBs No.	Limit of detection (µg l^{-1})
10	0.06
28	0.14
52	0.12
101	0.18
138	0.16
153	0.14
180	0.15

3.2.1.2 Summary of optimized GC conditions

The optimized GC conditions in water analysis are listed in Table 3.16.

Table 3.16 Optimized GC conditions with DB-1701, 30m × 0.25mm. I.D., 0.25μm film thickness

Operation	Optimal conditions
Temperature program	100°C –100°C(1min) – 190°C(10°C min^{-1}) –250°C (5° C min^{-1}) –250°C(10 min)
Carrier gas	Helium with flow rate 1.0 ml min^{-1}
Auxilliary gas	Nitrogen
Injector temperature	250°C
Detector temperature	320°C
Injection mode	Splitless, 2 μl injection volume

3.2.2 Optimization of solid phase extraction

3.2.2.1 Study of the effect of type and amount of wetting solvents and sample volume to percent recovery of PCBs

The solutions D-K were loaded onto C_{18} SPE and percent recoveries were studied. The details of solutions D-K are summarized in Table 3.17.

Table 3.17 Composition of solutions D-K

Solution	Composition (ml)					Spiked level ($\mu g\ l^{-1}$)
	Water	Acetone	Methanol	Isopropanol	Ethylacetate	
D	50	-	-	-	-	0.1
E	50	5	-	-	-	0.1
F	50	-	5	-	-	0.1
G	50	-	-	5	-	0.1
H	50	-	20	-	-	0.1
I	50	8	8	-	4	0.1
J	20	-	10	-	-	0.1
K	20	4	4	-	2	0.1

Table 3.18 Percent recoveries of solutions D-G, based on three determinations

PCBs No.	Recoveries(%), (R.S.D.%)			
	Solution D	Solution E	Solution F	Solution G
10	<50	<50	<50	50(2.6)
28	<50	56(4.7)	67(5.0)	64(6.5)
52	<50	64(2.7)	77(10.0)	86(9.2)
101	<50	70(3.4)	64(5.5)	63(16.8)
138	<50	<50	63(1.8)	55(16.6)
153	<50	<50	62(10.0)	63(13.5)
180	<50	<50	58(8.5)	<50

Table 3.19 Percent recoveries of solutions H and I, based on three determinations

PCBs No.	Recoveries(%), (R.S.D.%)	
	Solution H	Solution I
10	ND	ND
28	67(7.5)	72(12.5)
52	80(3.7)	90(6.3)
101	82(7.4)	88(8.2)
138	86(12.3)	94(6.5)
153	86(10.4)	92(7.6)
180	77(22.5)	83(1.2)

ND = not detected

Table 3.20 Percent recoveries of solutions J and K, based on three determinations

PCBs No.	Recoveries(%), (R.S.D.%)	
	Solution J	Solution K
10	57(21.4)	70(7.8)
28	89(0.5)	91(11.9)
52	87(1.1)	95(8.4)
101	92(3.5)	94(5.2)
138	82(2.2)	90(8.2)
153	79(2.8)	90(6.3)
180	75(6.4)	91(6.2)

3.2.2.2 Summary of optimized SPE conditions

The optimized extraction conditions established in this work are shown in Table 3.21.

Table 3.21 Optimized extraction conditions

Operation	Optimal extraction conditions
1. SPE column	8 ml glass column with polyethylene frits
2. Sorbent	500 mg of C_{18}
3. Conditioning solvent	5 ml of dichloromethane, 5 ml of methanol and 5 ml of solvent mixtures, water : ethylacetate : acetone : methanol (10:1:2: 2 v/v) loaded with flow rate 1-2 ml min^{-1}
4. Loading	Flow rate about 2-3ml min^{-1}
5. Drying	30 min with air pulled
6. Elution	3 ml of isooctane with gravity flow rate
7. Sample volume	20 ml

3.2.3 The percent recoveries of spiked drinking and natural surface water

The drinking and natural surface water samples was spiked at 0.1 and 1.0 μg l^{-1}, respectively. They were extracted with SPE by using the optimal conditions listed in Table 3.21. The percent recoveries of spiked drinking and natural surface water samples are shown in Tables 3.22 and 3.23, respectively.

Table 3.22 Percent recoveries of spiked drinking water with PCBs 0.1 μg l^{-1}, based on three determinations

PCBs No.	Recoveries (%), (R.S.D.%)
10	<50
28	79(4.5)
52	89(3.0)
101	96(1.9)
138	91(9.5)
153	86(3.1)
180	81(1.7)

Table 3.23 Percent recoveries of spiked natural surface water at 1.0 μg l^{-1}, based on three determinations

PCBs No.	Recoveries (%), (R.S.D.%)
10	<50
28	63(13.8)
52	75(14.5)
101	76(7.2)
138	87(6.0)
153	85(6.2)
180	85(6.9)

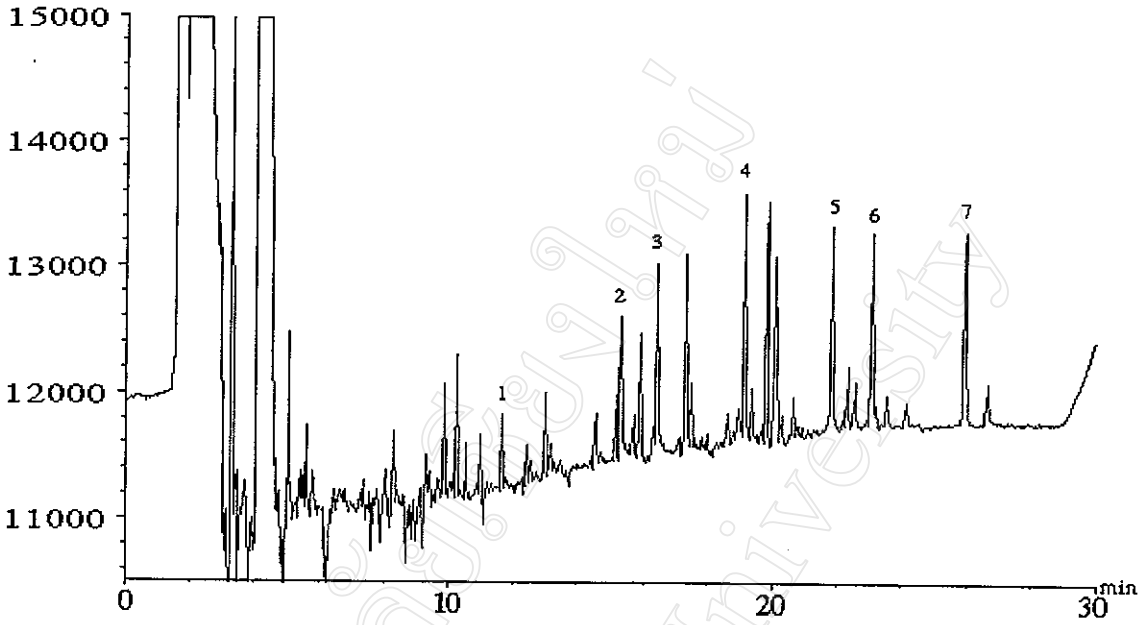

Figure 3.32 Chromatogram of spiked drinking water at 0.1 μg l^{-1}; 1, 2, 3, 4, 5, 6, 7 = PCBs No. 10, 28, 52, 101, 153, 138 and 180, respectively.

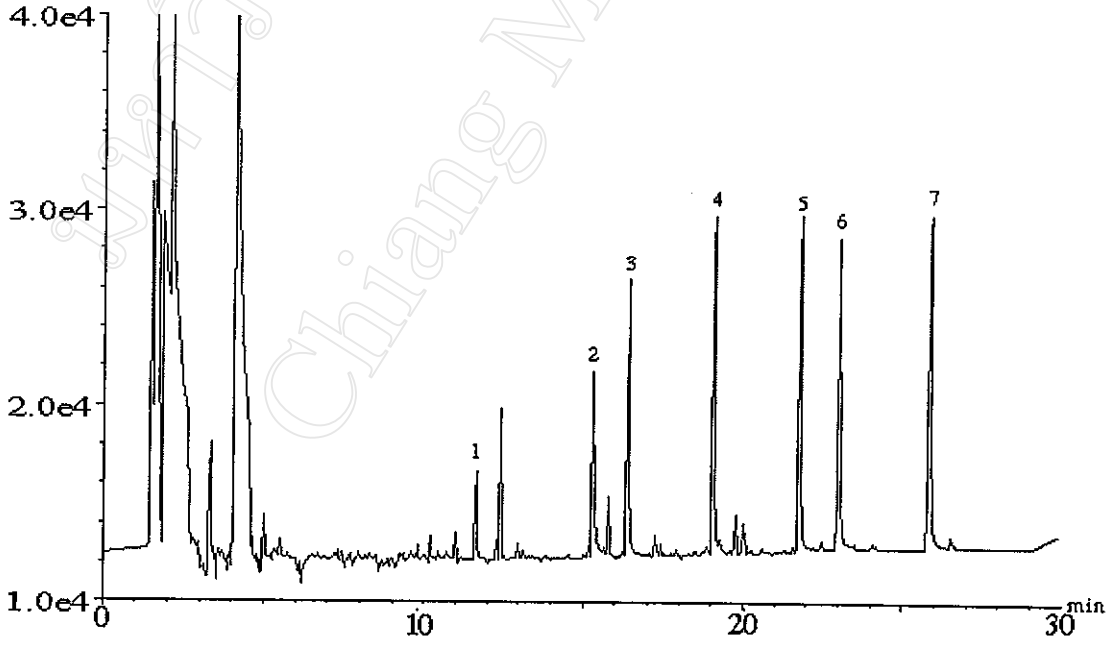

Figure 3.33 Chromatogram of spiked natural surface water at 1.0 μg l^{-1}; 1, 2, 3, 4, 5, 6, 7 = PCBs No. 10, 28, 52, 101, 153, 138 and 180, respectively.

3.2.4 Analysis of samples

Three brands of drinking water were analyzed and no PCBs was detected. The resultant chromatograms are shown in Figures 3.34 – 3.36.

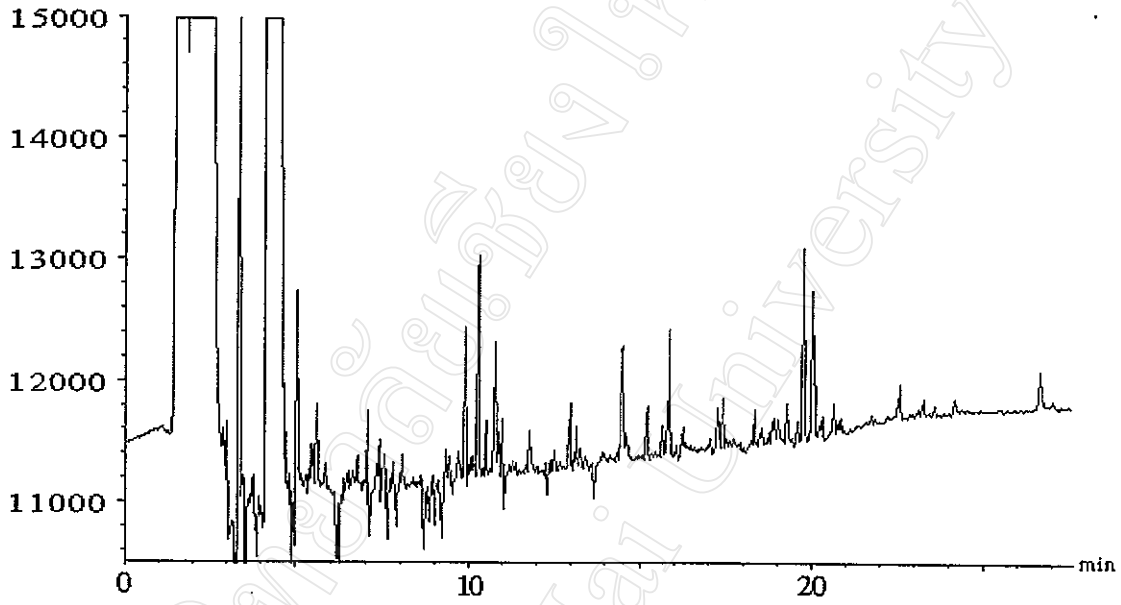

Figure 3.34 Chromatogram of drinking water brand A.

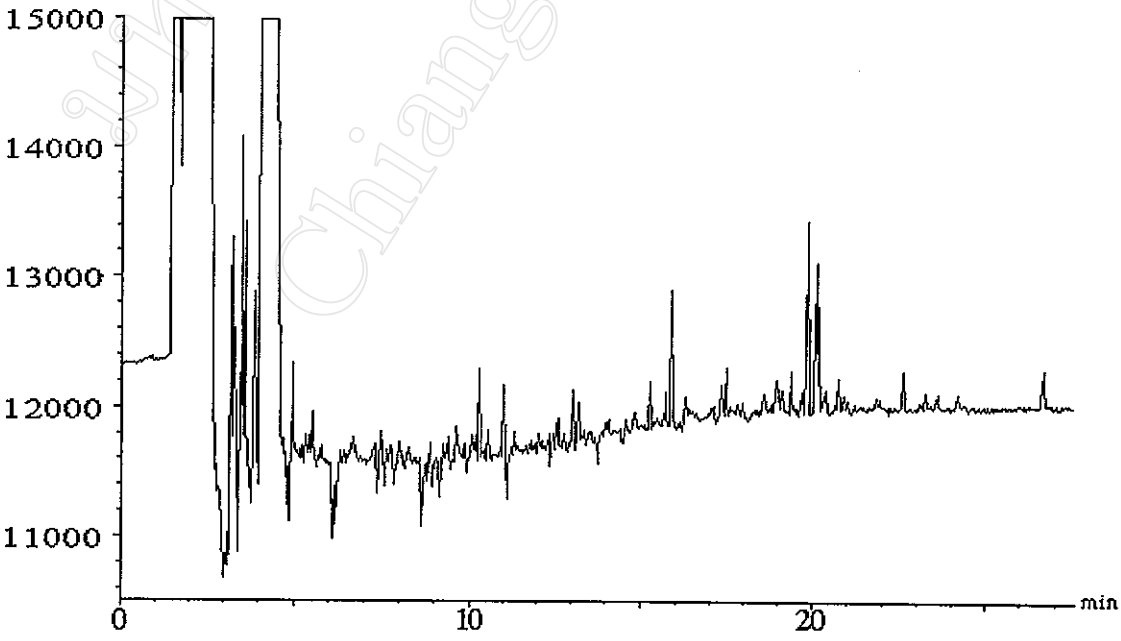

Figure 3.35 Chromatogram of drinking water brand B.

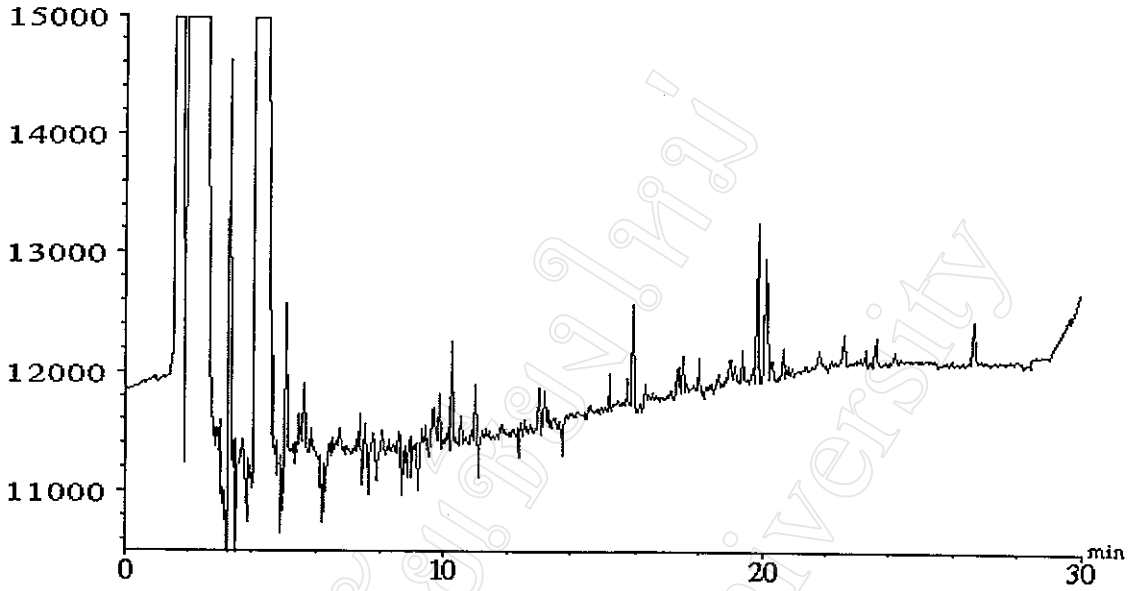

Figure 3.36 Chromatogram of drinking water brand C.

Natural surface water was sampled from four water resources in the northern of Thailand, namely Mae-ping, Mae-ngud, Mae-kaung and Hauyhong-krai. No PCBs were detected in all of samples except water from Mae-ping which indicated large interferences. The chromatograms of natural surface water are shown in Figures 3.37-3.40.

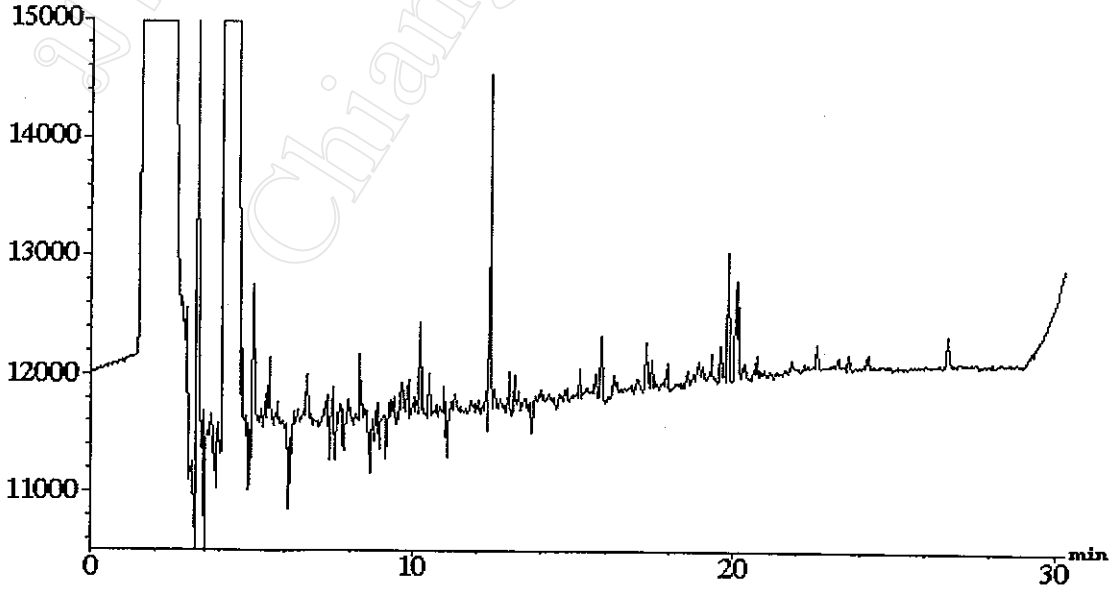

Figure 3.37 Chromatogram of Mae-ngud water sample.

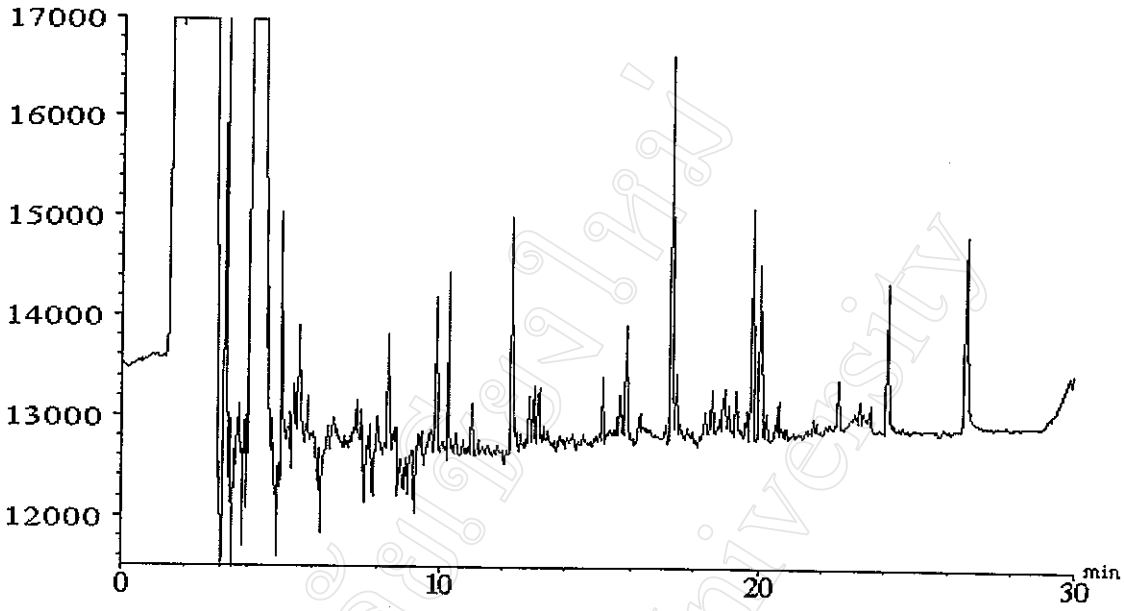

Figure 3.38 Chromatogram of Mae-kaung water sample.

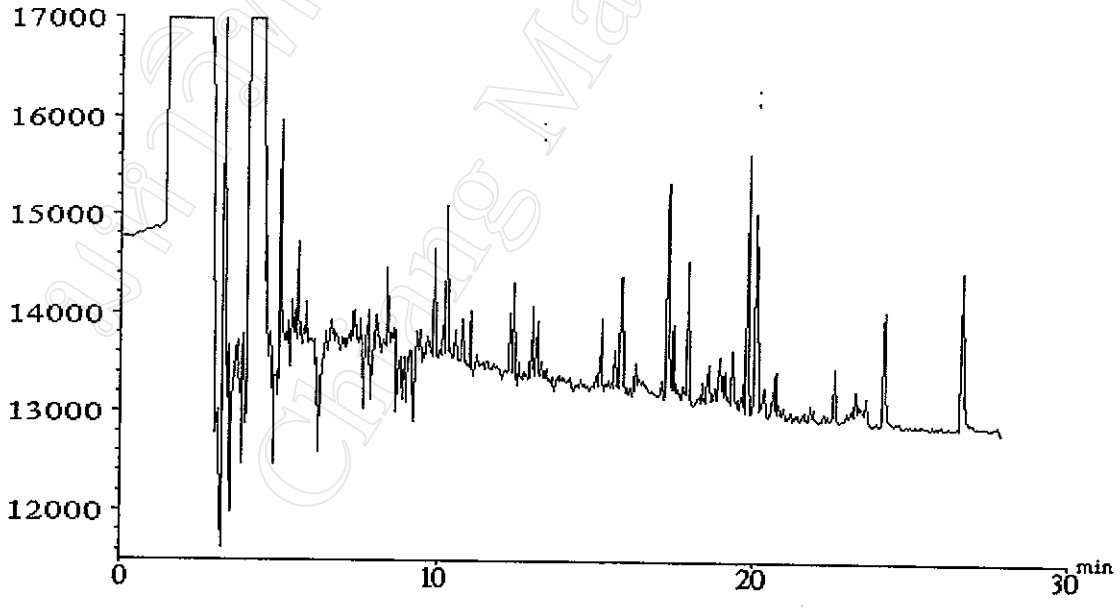

Figure 3.39 Chromatogram of Hauyhong-krai water sample.

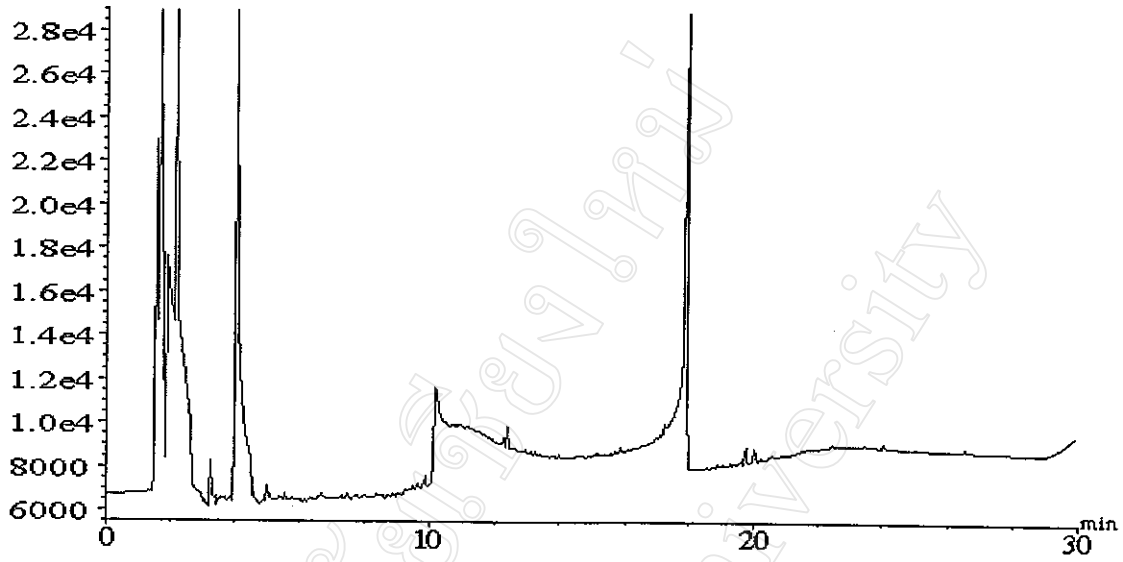

Figure 3.40 Chromatogram of Mae-ping water sample.

3.2.5 GC/MS of sulfur standard and Mae-ping water sample

To identify the type of interferences in Mae-ping water sample, GC/MS was used. The chromatograms of Mae-ping water sample and sulfur standard are shown in Figures 3.41 and 3.44. Mass spectra of Mae-ping sample are shown in Figures 3.42 and 3.43 and those of sulfur standard are shown in Figures 3.44 and 3.45.

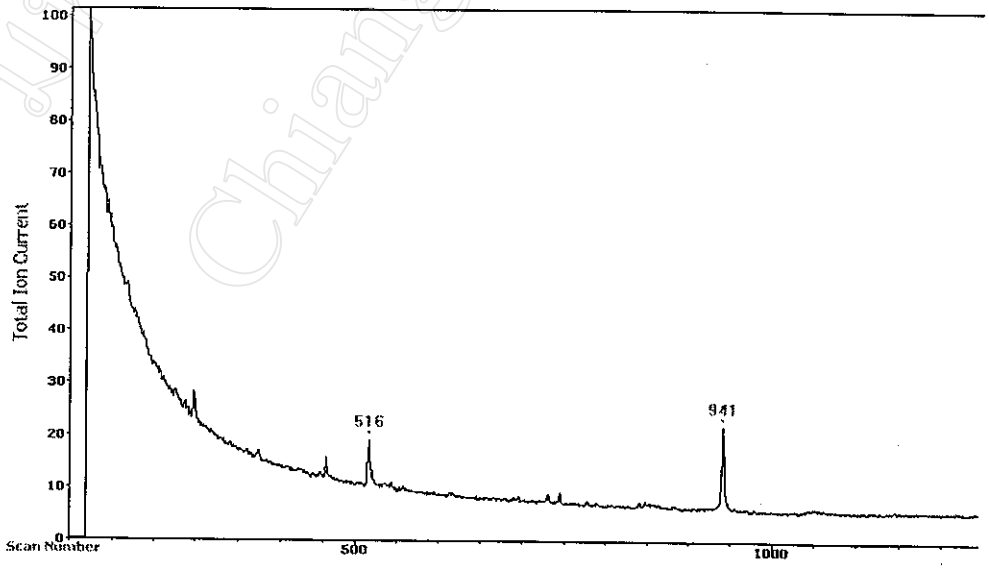

Figure 3.41 GC/MS chromatogram of Mae-ping water sample.

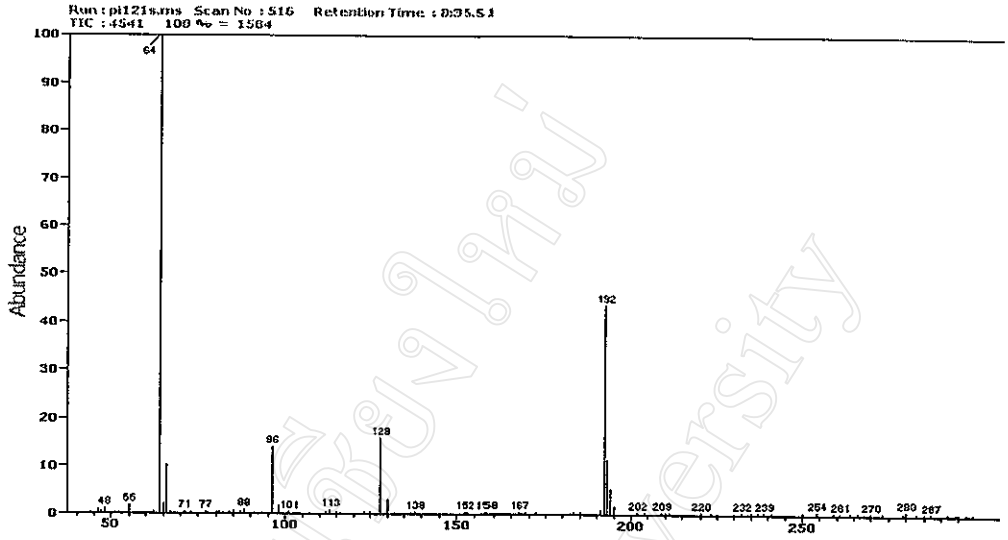

Figure 3.42 Mass spectrum of Mae-ping water sample at scan 516.

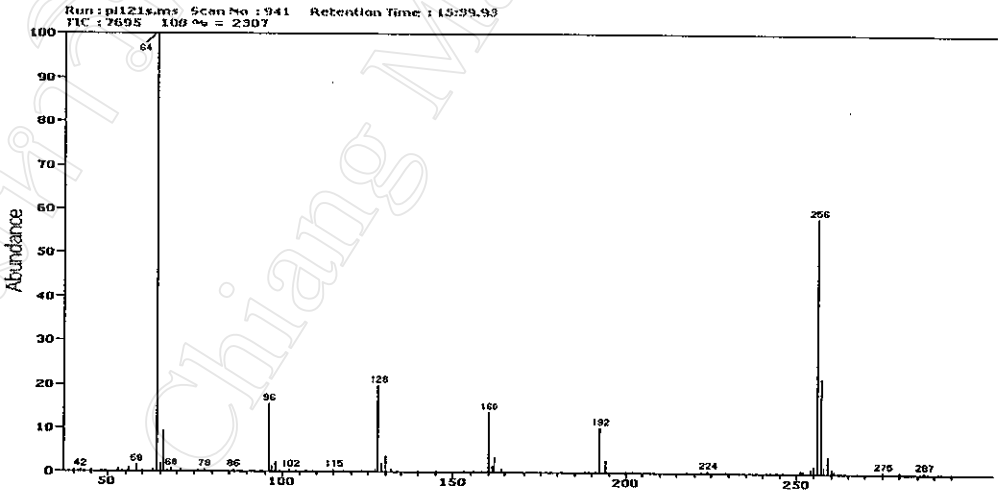

Figure 3.43 Mass spectrum of Mae-ping water sample at scan 941.

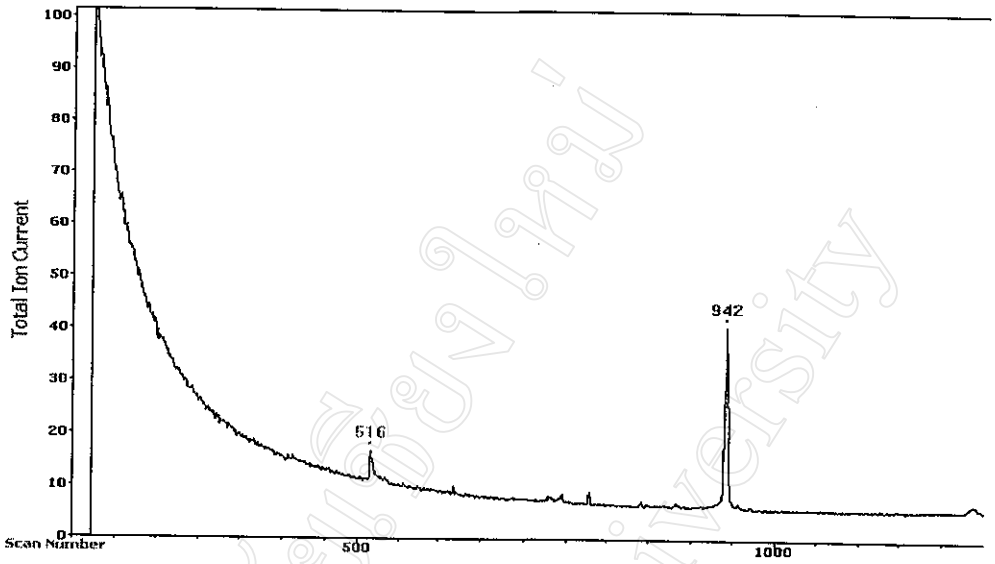

Figure 3.44 GC/MS chromatogram sulfur standard at 10 mg l^{-1}.

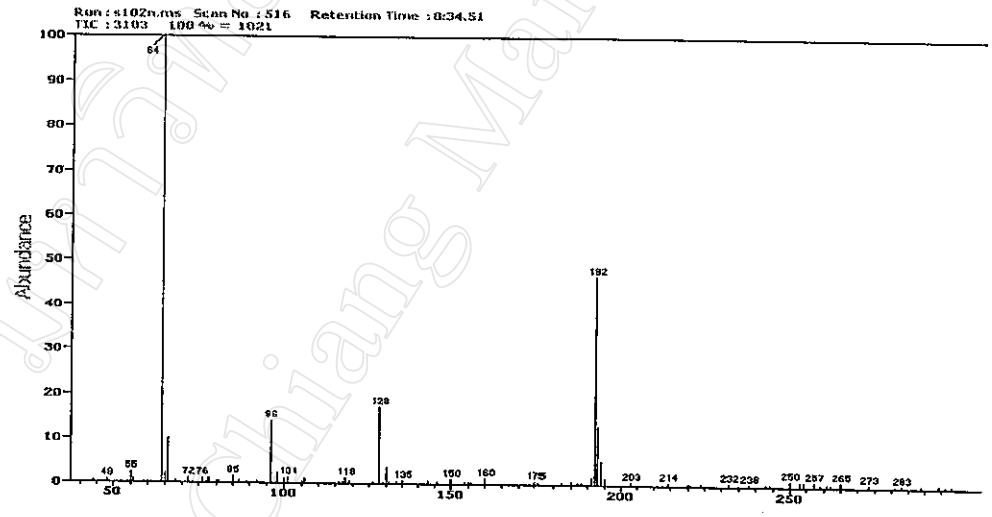

Figure 3.45 Mass spectrum of sulfur standard at scan 516.

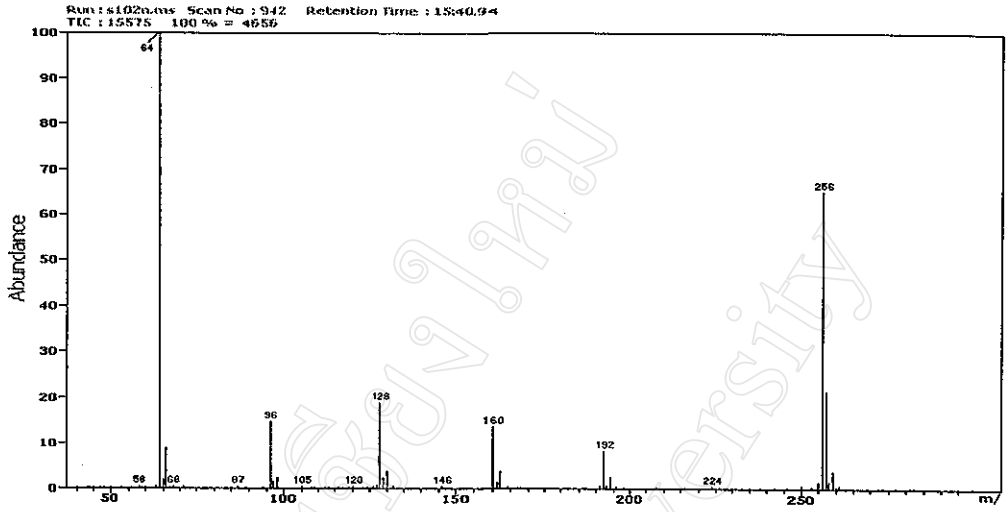

Figure 3.46 Mass spectrum of standard sulfur at scan 942.