SHIMADZU
Solutions for Science
since 1875

GCMS-QP2010 Application Data

Analysis of Polybrominated Biphenyl (PBBs) using GCMS-QP2010

Analytical System

GCMS : Shimadzu GCMS-QP2010

Analytical Conditions

■ Autosampler

# of Rinses with Sample	:1	# of Rinses with Solvent (Pre-run)	:1
# of Rinses with Solvent (Post-run)	:3	Viscosity Com. Time	:0.2sec
Plunger Speed (Injection)	:High	Syringe Injection Speed	:High
Inj. Port Dwell Time	:0.3sec	Injection Mode	:1
Pumping Times	:3 times		

■ GC

Column	:DB-5MS [0.25mm (i.d.) x 30m (length), Film thickness 0.1μm]
Injection Temp.	:350 ºC
Oven Temperature	:100 ºC (1min) → (15 ºC/min) → 180 ºC → (10 ºC/min) → 350 ºC (8min)
Carrier Gas	:He
Flow Control Mode	:Constant Linear Velocity (43.1cm/sec)
Injection Mode	:Splitless (Sampling Time :1min)
Injection Volume	:2μL

■ MS

Interface Temp.	:350 ºC	Ion Source Temp	:260 ºC

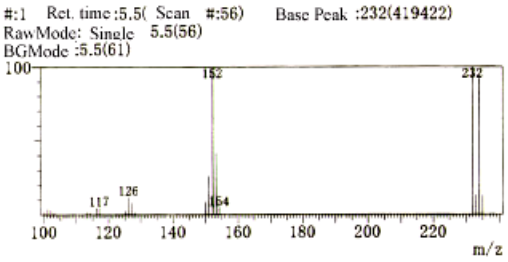
SCAN

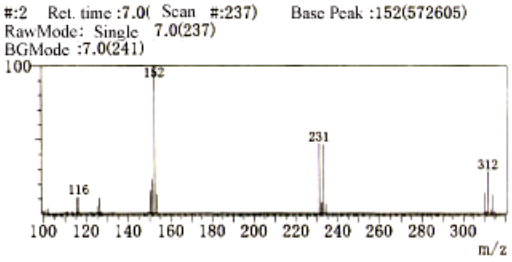
Interval	:0.5 sec	Scan Range	:m/z 45~350

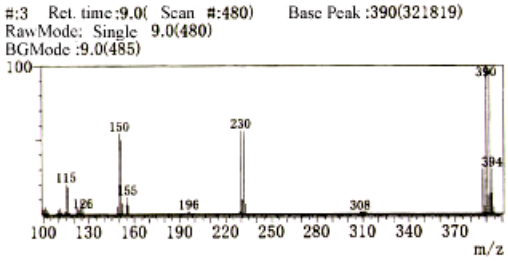
SIM

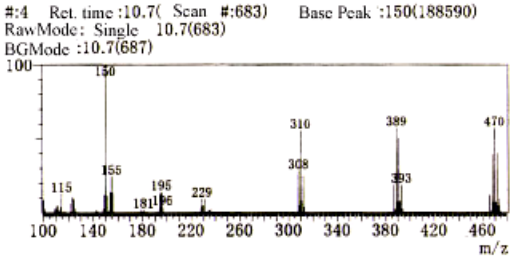
Interval :0.2 sec

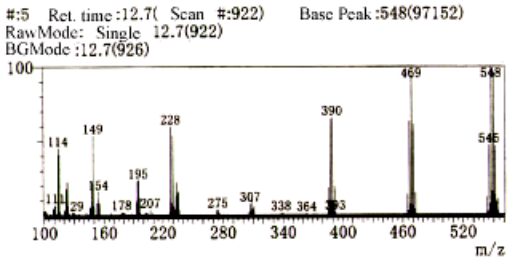
Compound Name		Monitor m/z
Monobromobiphenyl	PBB1	232.0, 152.0
Dibromobiphenyl	PBB2	311.9, 152.0
Tribromobiphenyl	PBB3	389.8, 229.9
Tetrabromobiphenyl	PBB4	469.7,150.1
Pentabromobiphenyl	PBB5	547.6, 468.7, 310.0
Hexabromobiphenyl	PBB6	627.5, 629.6, 467.7
Devabromobiphenyl	PBB10	943.3, 941.4

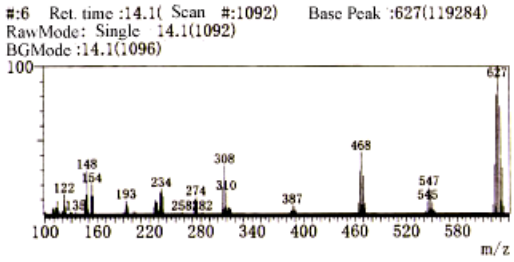


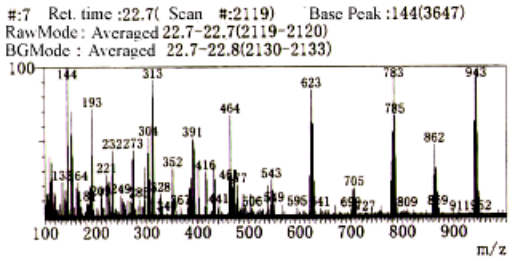








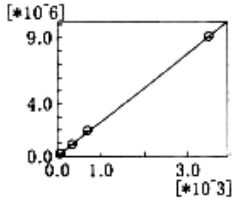




#1	Monobromobiphenyl	ID#1
#2	Dibromobiphenyl	ID#4
#3	Tribromobiphenyl	ID#9
#4	Tetrabromobiphenyl	ID#13
#5	Pentabromobiphenyl	ID#17
#6	Hexabromobiphenyl	ID#19
#7	Devabromobiphenyl	ID#22

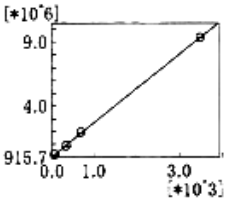
Calibration Curves of PBBs (0 ~ 3500µg/L)

ID#:1 m/z:232.00 Name:PBB1-1
f(x)=2578.549774*x+42201.410865
R^2 =0.999743



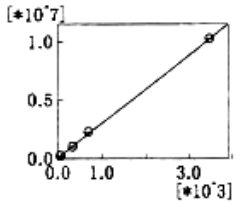
#	Conc. (ppm)	Area / Height
3	70.000	186002.60
4	350.000	899152.00
5	700.000	1943375.00
6	3500.000	9053176.00

ID#:2 m/z:232.00 Name:PBB1-2
f(x)=2666.797603*x-23059.281392
R^2 =0.999860



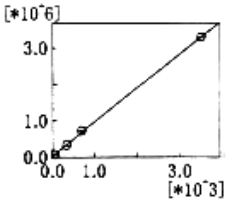
#	Conc. (ppm)	Area / Height
3	70.000	166337.80
4	350.000	851347.00
5	700.000	1906710.00
6	3500.000	9303973.00

ID#:3 m/z:232.00 Name:PBB1-3
f(x)=2921.144785*x+55694.073520
R^2 =0.999531



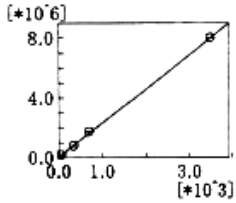
#	Conc. (ppm)	Area / Height
3	70.000	198903.20
4	350.000	1013648.00
5	700.000	2248055.00
6	3500.000	10257859.00

ID#:4 m/z:311.90 Name:PBB2-1
f(x)=937.244506*x+15922.395576
R^2 =0.999726



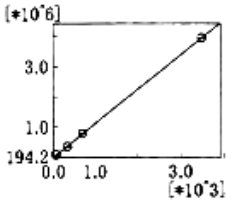
#	Conc. (ppm)	Area / Height
3	70.000	67503.20
4	350.000	327124.00
5	700.000	708114.00
6	3500.000	3291018.00

ID#:5 m/z:311.90 Name:PBB2-2
f(x)=2281.070267*x+48833.341900
R^2 =0.999648



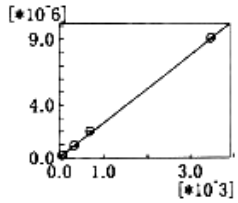
#	Conc. (ppm)	Area / Height
3	70.000	170257.00
4	350.000	800274.00
5	700.000	1745241.00
6	3500.000	8018106.00

ID#:6 m/z:311.90 Name:PBB2-3
f(x)=1126.689830*x-21754.653546
R^2 =0.999904



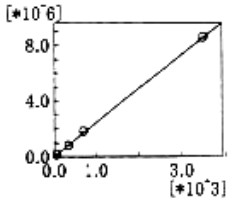
#	Conc. (ppm)	Area / Height
3	70.000	74092.40
4	350.000	348403.00
5	700.000	773336.00
6	3500.000	3922457.00

ID#:7 m/z:311.90 Name:PBB2-4
f(x)=2573.325593*x+74380.639558
R^2 =0.999403



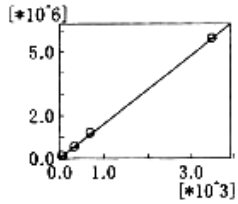
#	Conc. (ppm)	Area / Height
3	70.000	186827.80
4	350.000	918405.00
5	700.000	2022343.00
6	3500.000	9058711.00

ID#:8 m/z:311.90 Name:PBB2-5
f(x)=2443.039919*x+2571.443396
R^2 =0.999682



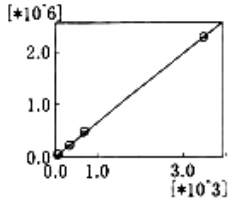
#	Conc. (ppm)	Area / Height
3	70.000	155578.20
4	350.000	791133.00
5	700.000	1809572.00
6	3500.000	8540847.00

ID#:9 m/z:389.80 Name:PBB3-1
f(x)=1602.629189*x+17075.087183
R^2 =0.999757



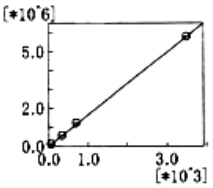
#	Conc. (ppm)	Area / Height
3	70.000	116298.20
4	350.000	541947.00
5	700.000	1195347.00
6	3500.000	5618855.00

ID#:10 m/z:389.80 Name:PBB3-2
f(x)=646.342299*x+7883.195185
R^2 =0.999716



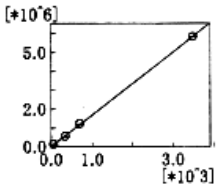
#	Conc. (ppm)	Area / Height
3	70.000	47122.20
4	350.000	218678.00
5	700.000	485032.00
6	3500.000	2266802.00

ID#:11 m/z:389.80 Name:PBB3-3
f(x)=1647.223254*x+20635.691607
R^2 =0.999657



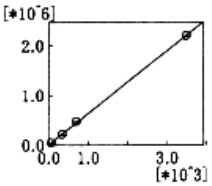
#	Conc. (ppm)	Area / Height
3	70.000	118914.20
4	350.000	554180.00
5	700.000	1242907.00
6	3500.000	5776713.00

ID#:12 m/z:389.80 Name :PBB3-4
f(x)=1663.065204*x+8480.539362
R^2 =0.999776



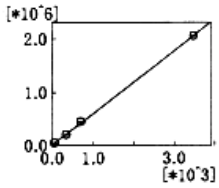
#	Conc. (ppm)	Area / Height
3	70.000	119866.40
4	350.000	548639.00
5	700.000	1225940.00
6	3500.000	5822838.00

ID#:13 m/z:469.70 Name:PBB4-1
f(x)=625.212878*x+10749.876383
R^2 =0.999581



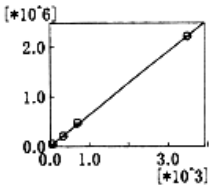
#	Conc. (ppm)	Area / Height
3	70.000	45703.00
4	350.000	213028.00
5	700.000	477808.00
6	3500.000	2194944.00

ID#:14 m/z:469.70 Name:PBB4-2
f(x)=584.525440*x+9946.566688
R^2 =0.999622



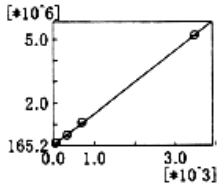
#	Conc. (ppm)	Area / Height
3	70.000	42869.80
4	350.000	199978.00
5	700.000	445278.00
6	3500.000	2052168.00

ID#:15 m/z:469.70 Name:PBB4-3
f(x)=631.501492*x+5596.527001
R^2 =0.999713



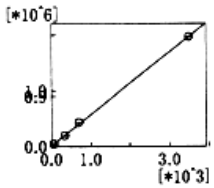
#	Conc. (ppm)	Area / Height
3	70.000	44760.00
4	350.000	210779.00
5	700.000	471647.00
6	3500.000	2212737.00

ID#:16 m/z:469.70 Name :PBB4-5
f(x)=1478.793325*x-186.189980
R^2 =0.999741



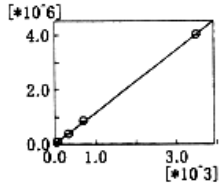
#	Conc. (ppm)	Area / Height
3	70.000	101600.40
4	350.000	475244.00
5	700.000	1084503.00
6	3500.000	5169933.00

ID#:17 m/z:547.60 Name:PBB5-1
f(x)=559.445421*x+10443.639232
R^2 =0.999581



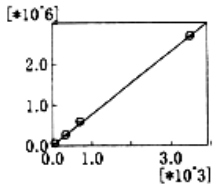
#	Conc. (ppm)	Area / Height
3	70.000	41337.40
4	350.000	191774.00
5	700.000	428468.00
6	3500.000	1964833.00

ID#:18 m/z:469.60 Name :PBB4-1
f(x)=1152.530365*x+1151.217892
R^2 =0.999551



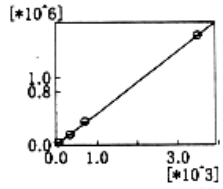
#	Conc. (ppm)	Area / Height
3	70.000	70507.16
4	350.000	368207.00
5	700.000	862662.00
6	3500.000	4027919.00

ID#:19 m/z:627.50 Name:PBB6-1
f(x)=765.562642*x+12520.297983
R^2 =0.999605



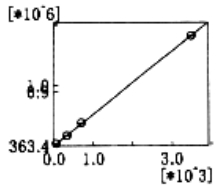
#	Conc. (ppm)	Area / Height
3	70.000	56009.60
4	350.000	260451.00
5	700.000	583305.00
6	3500.000	2687215.00

ID#:20 m/z:627.50 Name :PBB6-2
f(x)=463.695448*x+4218.658100
R^2 =0.999676



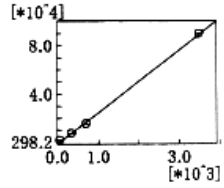
#	Conc. (ppm)	Area / Height
3	70.000	33463.60
4	350.000	153571.00
5	700.000	347301.00
6	3500.000	1624812.00

ID#:21 m/z:627.50 Name:PBB6-3
f(x)=518.810453*x-3429.713663
R^2 =0.999845



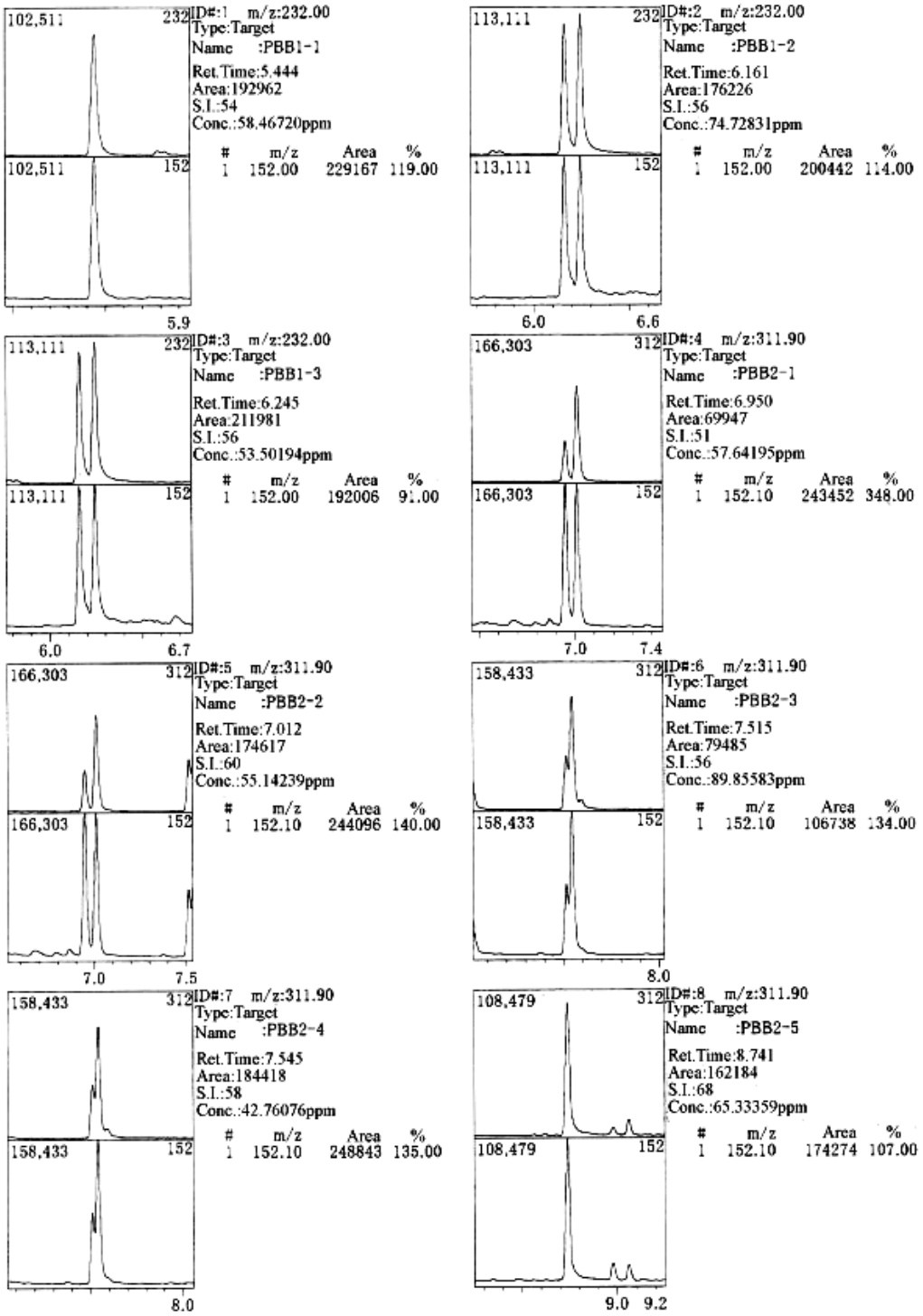
#	Conc. (ppm)	Area / Height
3	70.000	31396.44
4	350.000	167406.00
5	700.000	373655.00
6	3500.000	1810728.00

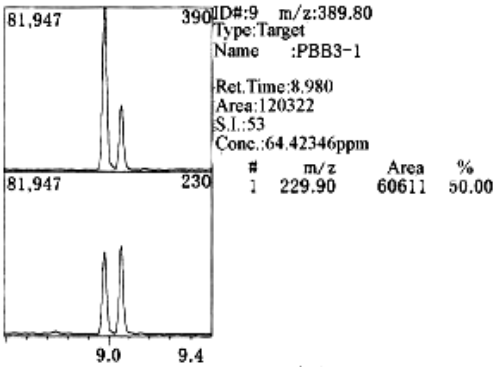
ID#:22 m/z:943.30 Name :PBB10-1
f(x)=25.888021*x-1740.554262
R^2 =0.999240



#	Conc. (ppm)	Area / Height
3	70.000	1444.44
4	350.000	7013.00
5	700.000	15045.00
6	3500.000	89138.00

SIM Chromatgram of PBBs (0.007μg/L)

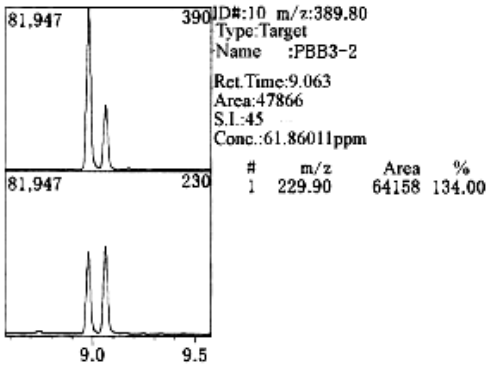




ID#:9 m/z:389.80
Type:Target
Name :PBB3-1

Ret.Time:8.980
Area:120322
S.I.:53
Conc.:64.42346ppm

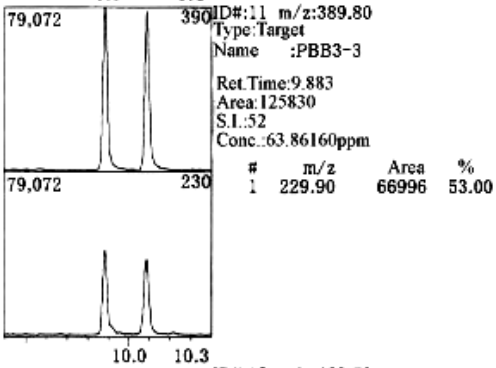
#	m/z	Area	%
1	229.90	60611	50.00



ID#:10 m/z:389.80
Type:Target
Name :PBB3-2

Ret.Time:9.063
Area:47866
S.I.:45
Conc.:61.86011ppm

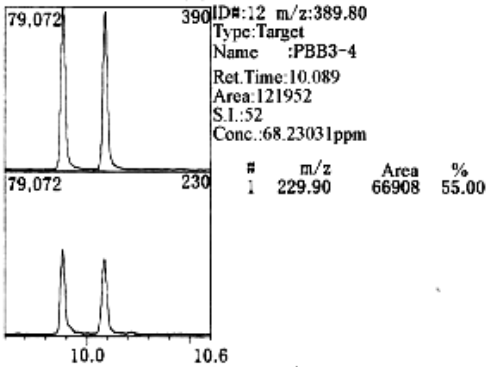
#	m/z	Area	%
1	229.90	64158	134.00



ID#:11 m/z:389.80
Type:Target
Name :PBB3-3

Ret.Time:9.883
Area:125830
S.I.:52
Conc.:63.86160ppm

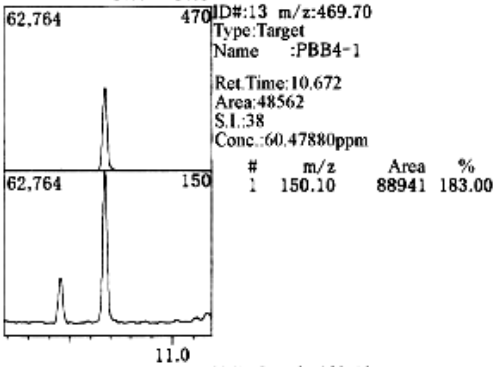
#	m/z	Area	%
1	229.90	66996	53.00



ID#:12 m/z:389.80
Type:Target
Name :PBB3-4

Ret.Time:10.089
Area:121952
S.I.:52
Conc.:68.23031ppm

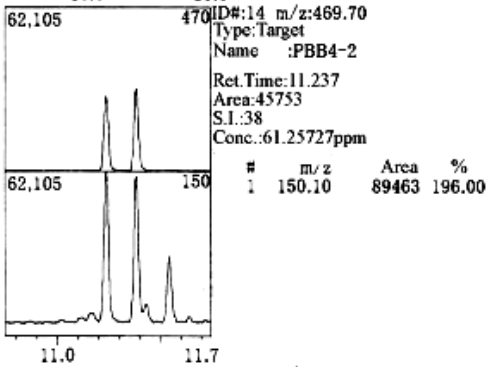
#	m/z	Area	%
1	229.90	66908	55.00



ID#:13 m/z:469.70
Type:Target
Name :PBB4-1

Ret.Time:10.672
Area:48562
S.I.:38
Conc.:60.47880ppm

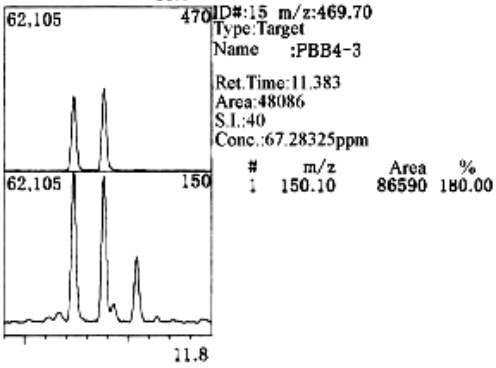
#	m/z	Area	%
1	150.10	88941	183.00



ID#:14 m/z:469.70
Type:Target
Name :PBB4-2

Ret.Time:11.237
Area:45753
S.I.:38
Conc.:61.25727ppm

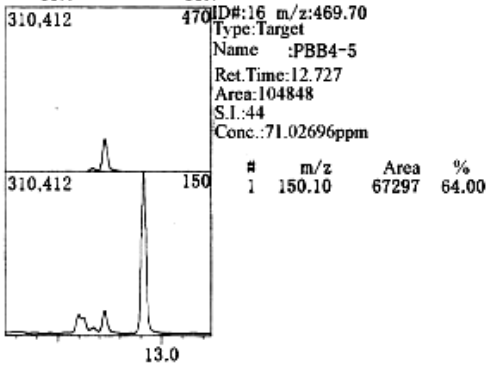
#	m/z	Area	%
1	150.10	89463	196.00



ID#:15 m/z:469.70
Type:Target
Name :PBB4-3

Ret.Time:11.383
Area:48086
S.I.:40
Conc.:67.28325ppm

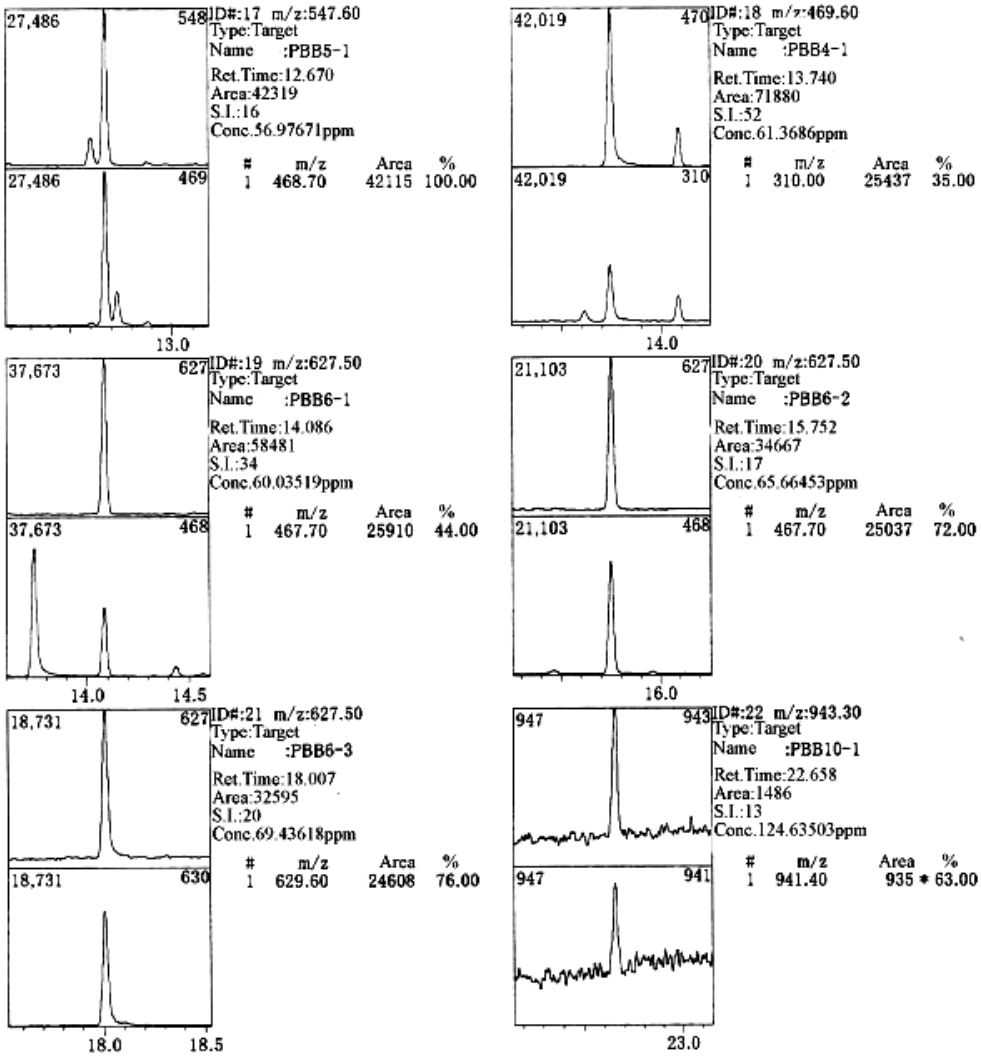
#	m/z	Area	%
1	150.10	86590	180.00



ID#:16 m/z:469.70
Type:Target
Name :PBB4-5

Ret.Time:12.727
Area:104848
S.I.:44
Conc.:71.02696ppm

#	m/z	Area	%
1	150.10	67297	64.00


27,486
548
27,486
469
13.0
ID#:17 m/z:547.60
Type:Target
Name :PBB5-1
Ret.Time:12.670
Area:42319
S.I.:16
Conc.56.97671ppm
m/z Area %
1 468.70 42115 100.00
42,019
470
42,019
310
14.0
ID#:18 m/z:469.60
Type:Target
Name :PBB4-1
Ret.Time:13.740
Area:71880
S.I.:52
Conc.61.3686ppm
m/z Area %
1 310.00 25437 35.00
37,673
627
37,673
468
14.0
14.5
ID#:19 m/z:627.50
Type:Target
Name :PBB6-1
Ret.Time:14.086
Area:58481
S.I.:34
Conc.60.03519ppm
m/z Area %
1 467.70 25910 44.00
21,103
627
21,103
468
16.0
ID#:20 m/z:627.50
Type:Target
Name :PBB6-2
Ret.Time:15.752
Area:34667
S.I.:17
Conc.65.66453ppm
m/z Area %
1 467.70 25037 72.00
18,731
627
18,731
630
18.0
18.5
ID#:21 m/z:627.50
Type:Target
Name :PBB6-3
Ret.Time:18.007
Area:32595
S.I.:20
Conc.69.43618ppm
m/z Area %
1 629.60 24608 76.00
947
943
947
941
23.0
ID#:22 m/z:943.30
Type:Target
Name :PBB10-1
Ret.Time:22.658
Area:1486
S.I.:13
Conc.124.63503ppm
m/z Area %
1 941.40 935 * 63.00

Repeatability (70μg/L, n=5)

	1	2	3	4	5	Average	σ	CV
PBB1-1	181216	177887	186926	191022	192962	186002.2	6388.559994	3.43
PBB1-2	161507	156165	169600	168191	176226	166337.8	7724.559644	4.64
PBB1-3	184026	187834	212289	198386	211981	198903.2	13175.11156	6.62
PBB2-1	66052	64174	67997	69346	69947	67503.2	2387.543863	3.54
PBB2-2	164056	164118	174061	174433	174617	170257	5636.016191	3.31
PBB2-3	75201	69905	73248	72623	79485	74092.4	3560.744136	4.81
PBB2-4	184236	183598	201143	180744	184418	186827.8	8137.778333	4.36
PBB2-5	148429	153224	156442	157612	162184	155578.2	5127.679904	3.30
PBB3-1	115641	109831	118514	117183	120322	116298.2	4004.378691	3.44
PBB3-2	47804	44320	50446	45175	47866	47122.2	2434.627733	5.17
PBB3-3	112628	112788	123613	119712	125830	118914.2	6074.333972	5.11
PBB3-4	116837	114759	121952	123834	121952	119866.4	3864.082595	3.22
PBB4-1	44340	43237	46863	45513	48562	45703	2091.321711	4.58
PBB4-2	40403	41002	44750	42441	45753	42869.8	2324.093522	5.42
PBB4-3	42579	42782	45765	44588	48086	44760	2278.767759	5.09
PBB4-5	100199	95657	106252	101046	104848	101600.4	4176.708429	4.11
PBB5-1	40923	39163	43350	40932	42319	41337.4	1587.308193	3.84
PBB4-1	69630	66851	71798	72376	71880	70507	2301.110384	3.26
PBB6-1	54441	53709	57915	55502	58481	56009.6	2106.495383	3.76
PBB6-2	32178	32050	35855	32586	34667	33463.6	1704.413183	5.09
PBB6-3	29929	30321	31990	32148	32595	31396.6	1189.921552	3.79
PBB10-1	1476	1354	1415	1491	1486	1444.4	59.02795948	4.09

Notes for Analysis of Polybrominated Biphenyl

1. Pay attention to the following points, because boiling point of Devabromobiphenyl is high.
   (1) Use the column with 0.1 μm film thickness to shorten retention time.
   (2) Set the injector temperature 350 ºC
   (3) Set the interface temperature 350 ºC
   (4) Set the ion source temperature 260 ºC
2. Pay attention to the following points, because mass number of Devabromobiphenyl is 933.184 and is not a round figure.
   (1) GC/MS system enabling to measure ions up to about m/z 950 is requied.
   (2) Set monitor mass to decimal places.
   (3) Acquire data with low resolution (FWHM 0.8)

   [Note]
   The mass of Bromine:
   79Br=78.9184
   81Br=80.9164
   The mass of Devabromobiphenyl:
   12.0000x12+78.9184x10=933.184