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**GCMS-QP2010 Application Data**


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**Analysis of Polybrominated Biphenyl (PBBs) using GCMS-QP2010**


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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 °C		
Oven Temperature	:100 °C (1min) → (15 °C/min) → 180 °C → (10 °C/min) → 350 °C (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 °C	Ion Source Temp	:260 °C
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**SCAN**

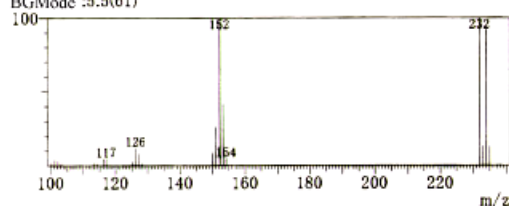
Interval	:0.5 sec	Scan Range	:m/z 45~350
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**SIM**

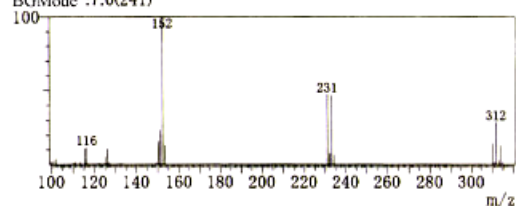
Interval	:0.2 sec
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Compound Name		Monitor m/z
Monobromobiphenyl	PBB1	232.0, 152.0
Dibromobiphenyl	PBB2	311.9, 152.0
Tribromobiphenyl	PBB3	389.8, 229.9
Tetrabromobiphenyl	PBB4	469.7, 150.1
Pentabromobiphenyl	PBB5	547.6, 468.7, 310.0
Hexabromobiphenyl	PBB6	627.5, 629.6, 467.7
Devabromobiphenyl	PBB10	943.3, 941.4

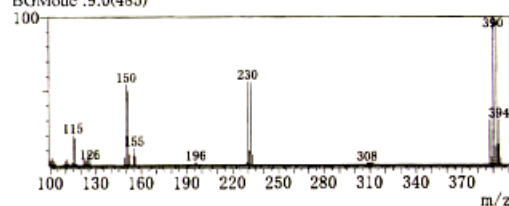
#1 Ret. time:5.5( Scan #:56) Base Peak :232(419422)  
RawMode: Single 5.5(56)  
BGMode :5.5(61)



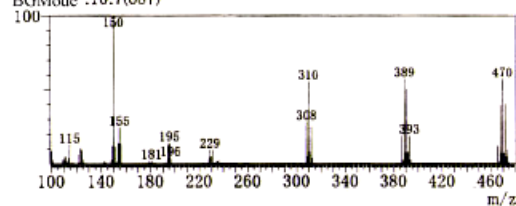
#2 Ret. time:7.0( Scan #:237) Base Peak :152(572605)  
RawMode: Single 7.0(237)  
BGMode :7.0(241)



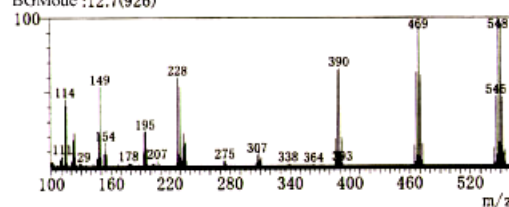
#3 Ret. time:9.0( Scan #:480) Base Peak :390(321819)  
RawMode: Single 9.0(480)  
BGMode :9.0(485)



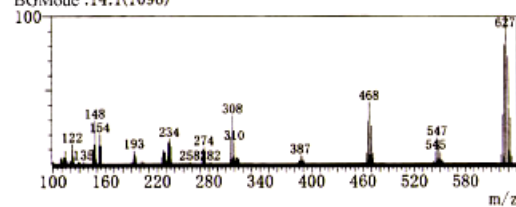
#4 Ret. time:10.7( Scan #:683) Base Peak :150(188590)  
RawMode: Single 10.7(683)  
BGMode :10.7(687)



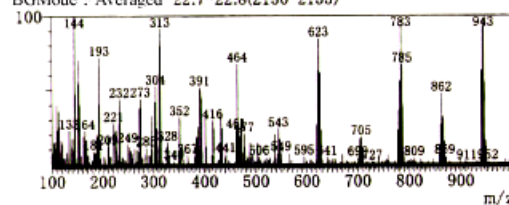
#5 Ret. time:12.7( Scan #:922) Base Peak :548(97152)  
RawMode: Single 12.7(922)  
BGMode :12.7(926)



#6 Ret. time:14.1( Scan #:1092) Base Peak :627(119284)  
RawMode: Single 14.1(1092)  
BGMode :14.1(1096)



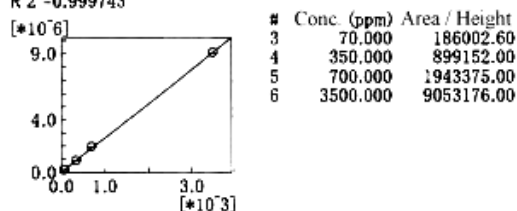
#7 Ret. time:22.7( Scan #:2119) Base Peak :144(3647)  
RawMode: Averaged 22.7-22.7(2119-2120)  
BGMode : Averaged 22.7-22.8(2130-2133)



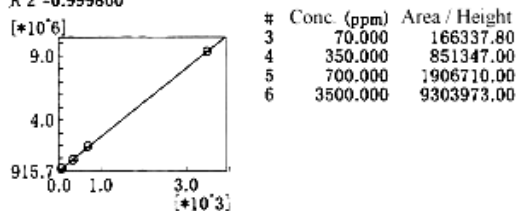
#1	Monobromobiphenyl	ID#1
#2	Dibromobiphenyl	ID#4
#3	Tribromobiphenyl	ID#9
#4	Tetrabromobiphenyl	ID#13
#5	Pentabromobiphenyl	ID#17
#6	Hexabromobiphenyl	ID#19
#7	Devabromobiphenyl	ID#22

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

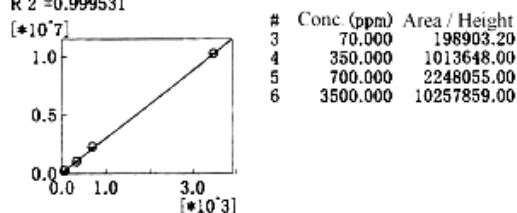
ID#:1 m/z:232.00 Name:PBB1-1  
 $f(x)=2578.549774*x+42201.410865$   
 $R^2=0.999743$



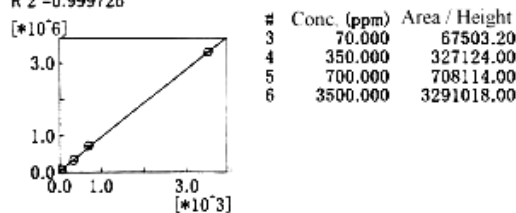
ID#:2 m/z:232.00 Name:PBB1-2  
 $f(x)=2666.797603*x-23059.281392$   
 $R^2=0.999860$



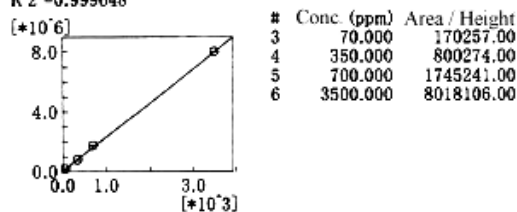
ID#:3 m/z:232.00 Name:PBB1-3  
 $f(x)=2921.144785*x+55694.073520$   
 $R^2=0.999531$



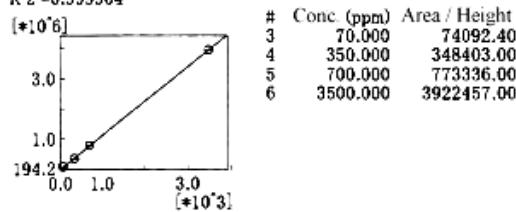
ID#:4 m/z:311.90 Name:PBB2-1  
 $f(x)=937.244506*x+15922.395576$   
 $R^2=0.999726$



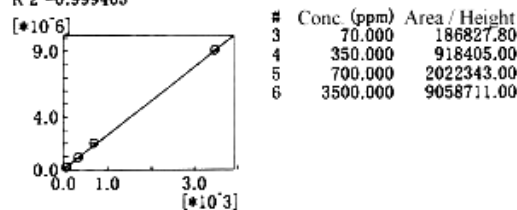
ID#:5 m/z:311.90 Name:PBB2-2  
 $f(x)=2281.070267*x+48833.341900$   
 $R^2=0.999648$



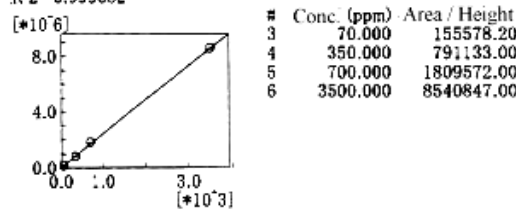
ID#:6 m/z:311.90 Name:PBB2-3  
 $f(x)=1126.689830*x-21754.653546$   
 $R^2=0.999904$



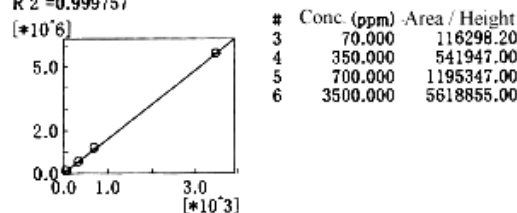
ID#:7 m/z:311.90 Name:PBB2-4  
 $f(x)=2573.325593*x+74380.639558$   
 $R^2=0.999403$



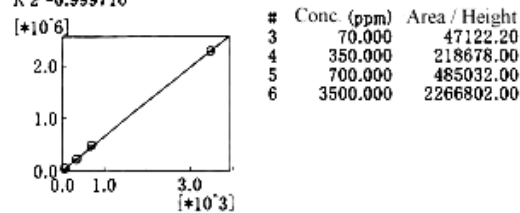
ID#:8 m/z:311.90 Name:PBB2-5  
 $f(x)=2443.039919*x+2571.443396$   
 $R^2=0.999682$



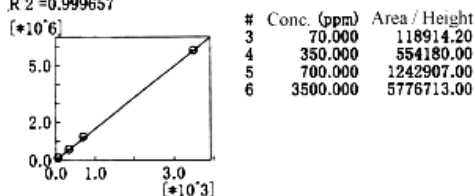
ID#:9 m/z:389.80 Name:PBB3-1  
 $f(x)=1602.629189*x+17075.087183$   
 $R^2=0.999757$



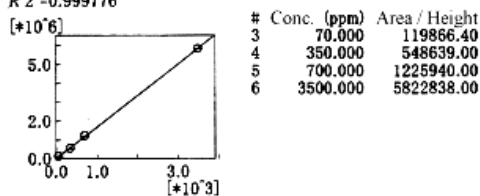
ID#:10 m/z:389.80 Name:PBB3-2  
 $f(x)=646.342299*x+7883.195185$   
 $R^2=0.999716$



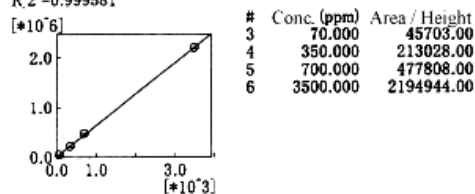
ID#:11 m/z:389.80 Name:PBB3-3  
 $f(x)=1647.223254*x+20635.691607$   
 $R^2=0.999657$



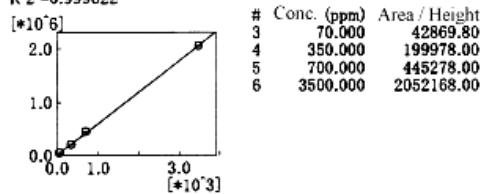
ID#:12 m/z:389.80 Name:PBB3-4  
 $f(x)=1663.065204*x+8480.539362$   
 $R^2=0.999776$



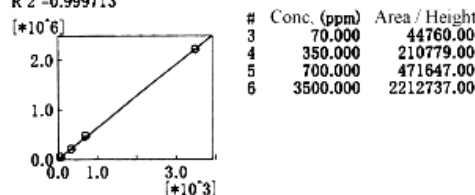
ID#:13 m/z:469.70 Name:PBB4-1  
 $f(x)=625.212878*x+10749.876383$   
 $R^2=0.999581$



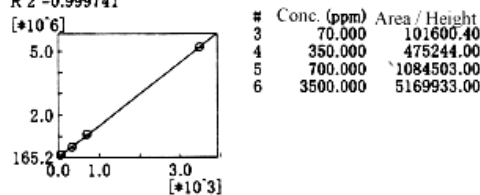
ID#:14 m/z:469.70 Name:PBB4-2  
 $f(x)=584.525440*x+9946.566688$   
 $R^2=0.999622$



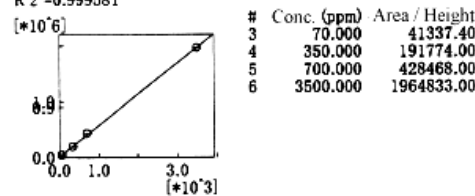
ID#:15 m/z:469.70 Name:PBB4-3  
 $f(x)=631.501492*x+5596.527001$   
 $R^2=0.999713$



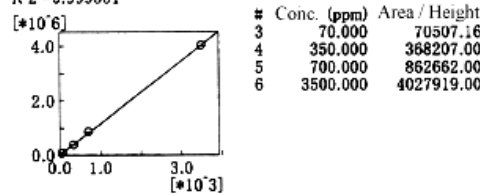
ID#:16 m/z:469.70 Name:PBB4-5  
 $f(x)=1478.793325*x-186.189980$   
 $R^2=0.999741$



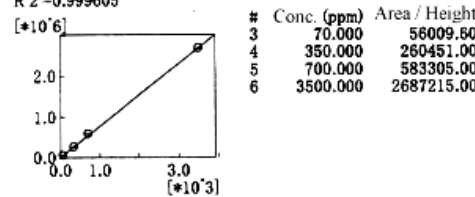
ID#:17 m/z:547.60 Name:PBB5-1  
 $f(x)=559.445421*x+10443.639232$   
 $R^2=0.999581$



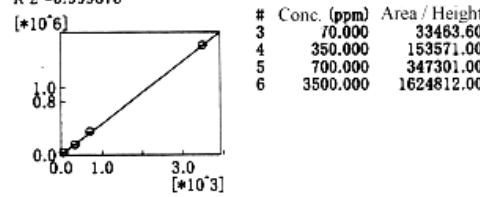
ID#:18 m/z:469.60 Name:PBB4-1  
 $f(x)=1152.530365*x+1151.217892$   
 $R^2=0.999551$



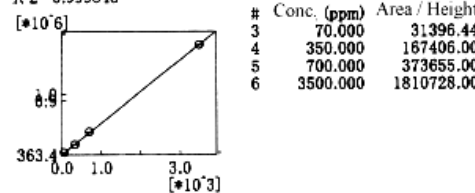
ID#:19 m/z:627.50 Name:PBB6-1  
 $f(x)=765.562642*x+12520.297983$   
 $R^2=0.999605$



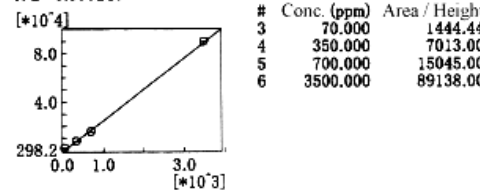
ID#:20 m/z:627.50 Name:PBB6-2  
 $f(x)=463.695448*x+4218.658100$   
 $R^2=0.999676$



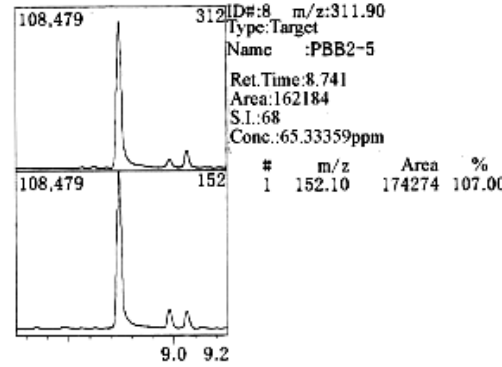
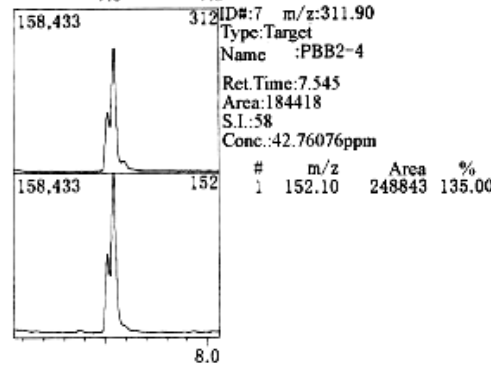
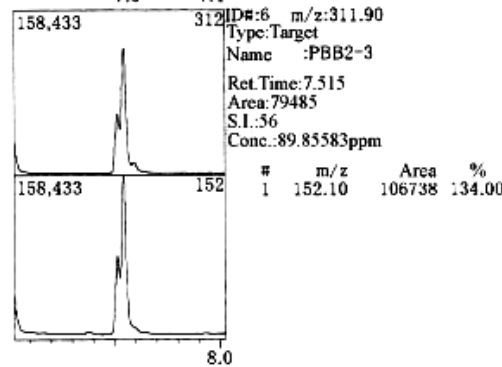
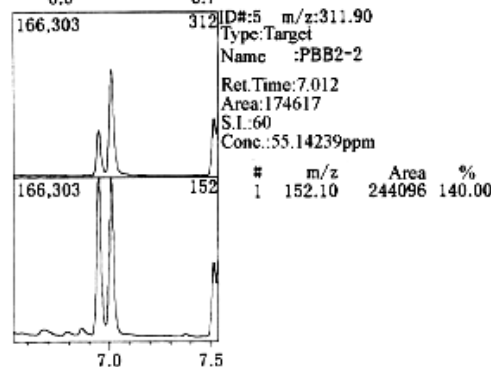
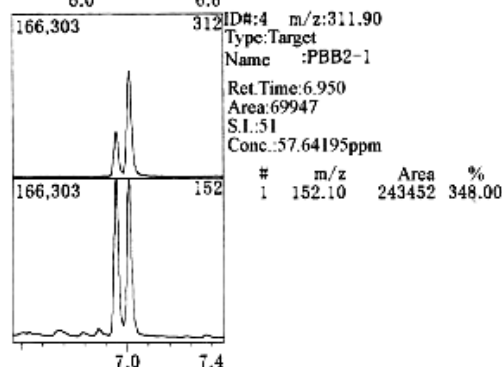
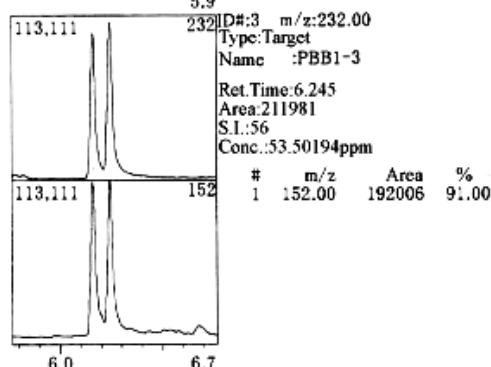
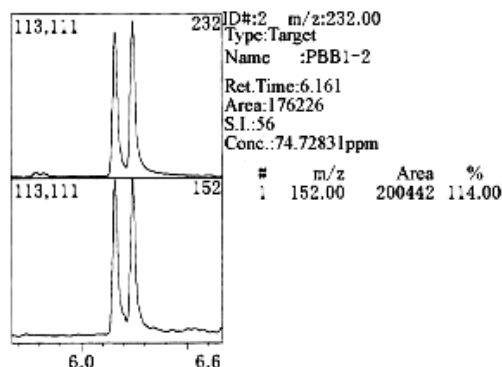
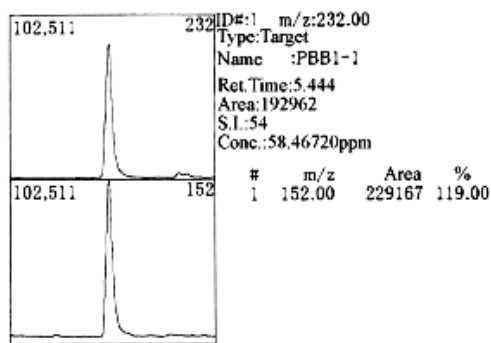
ID#:21 m/z:627.50 Name:PBB6-3  
 $f(x)=518.810453*x-3429.713663$   
 $R^2=0.999845$

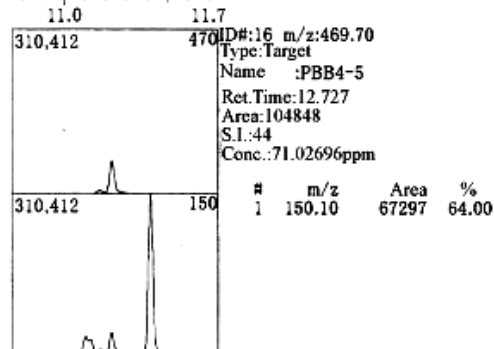
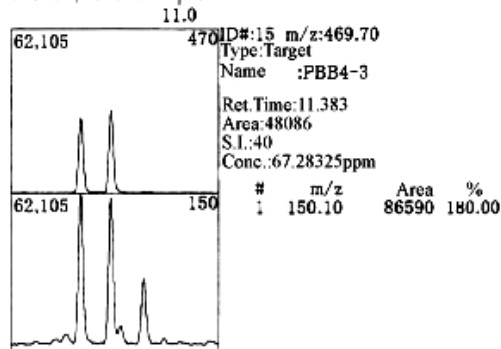
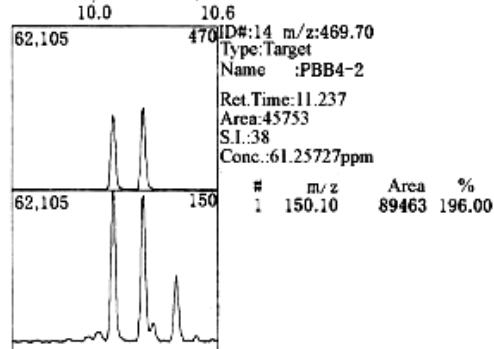
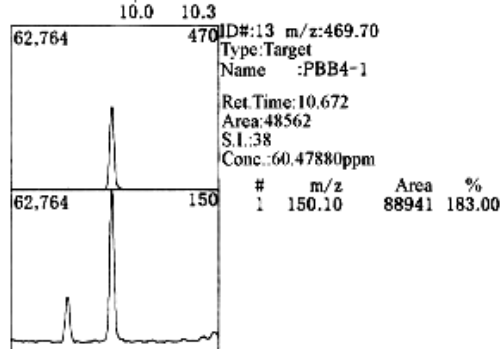
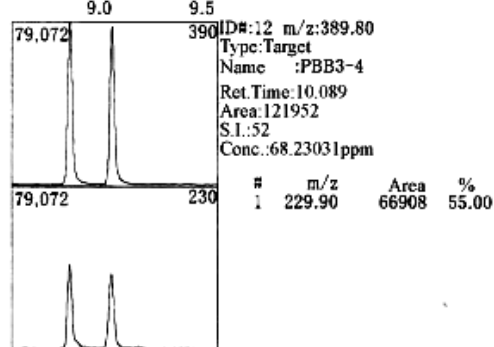
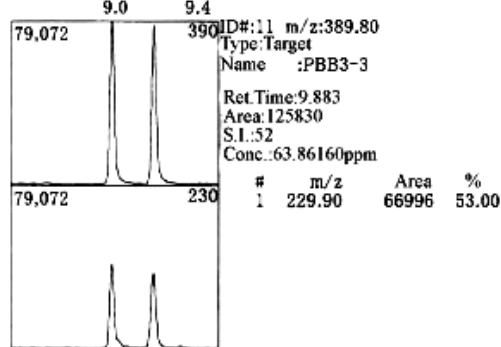
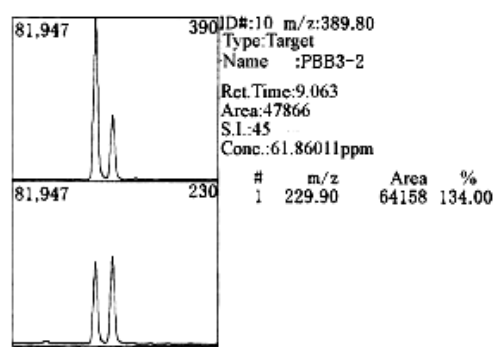
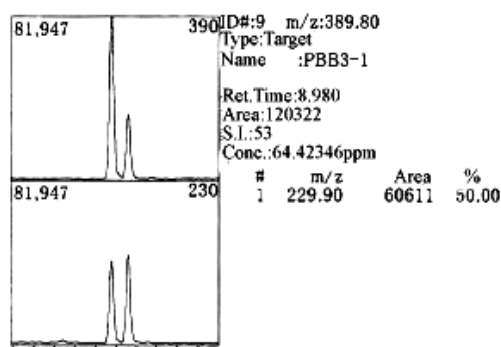


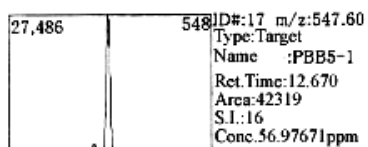
ID#:22 m/z:943.30 Name:PBB10-1  
 $f(x)=25.888021*x-1740.554262$   
 $R^2=0.999240$



**SIM Chromatogram of PBBs ( 0.007µg/L)**

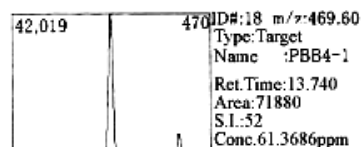






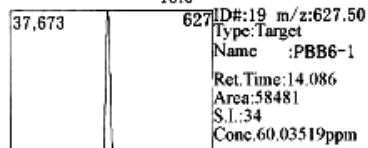
ID#17 m/z:547.60  
 Type:Target  
 Name :PBB5-1  
 Ret.Time:12.670  
 Area:42319  
 S.I.:16  
 Conc.56.97671ppm

#	m/z	Area	%
1	468.70	42115	100.00



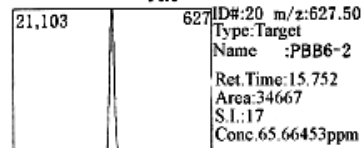
ID#18 m/z:469.60  
 Type:Target  
 Name :PBB4-1  
 Ret.Time:13.740  
 Area:71880  
 S.I.:52  
 Conc.61.3686ppm

#	m/z	Area	%
1	310.00	25437	35.00



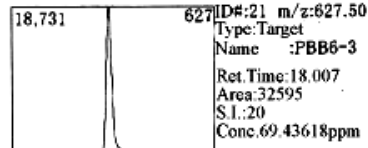
ID#19 m/z:627.50  
 Type:Target  
 Name :PBB6-1  
 Ret.Time:14.086  
 Area:58481  
 S.I.:34  
 Conc.60.03519ppm

#	m/z	Area	%
1	467.70	25910	44.00



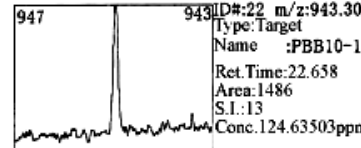
ID#20 m/z:627.50  
 Type:Target  
 Name :PBB6-2  
 Ret.Time:15.752  
 Area:34667  
 S.I.:17  
 Conc.65.66453ppm

#	m/z	Area	%
1	467.70	25037	72.00



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



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	$\sigma$	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  $\mu\text{m}$  film thickness to shorten retention time.
  - (2) Set the injector temperature 350  $^{\circ}\text{C}$
  - (3) Set the interface temperature 350  $^{\circ}\text{C}$
  - (4) Set the ion source temperature 260  $^{\circ}\text{C}$
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 required.
  - (2) Set monitor mass to decimal places.
  - (3) Acquire data with low resolution (FWHM 0.8)

[Note]

The mass of Bromine:

$$^{79}\text{Br}=78.9184$$

$$^{81}\text{Br}=80.9164$$

The mass of Devabromobiphenyl:

$$12.0000 \times 12 + 78.9184 \times 10 = 933.184$$