Asymmetric semipinacol rearrangement of 2,3-allenols with *N*-bromo-1,8-naphthalimide

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Supporting Information

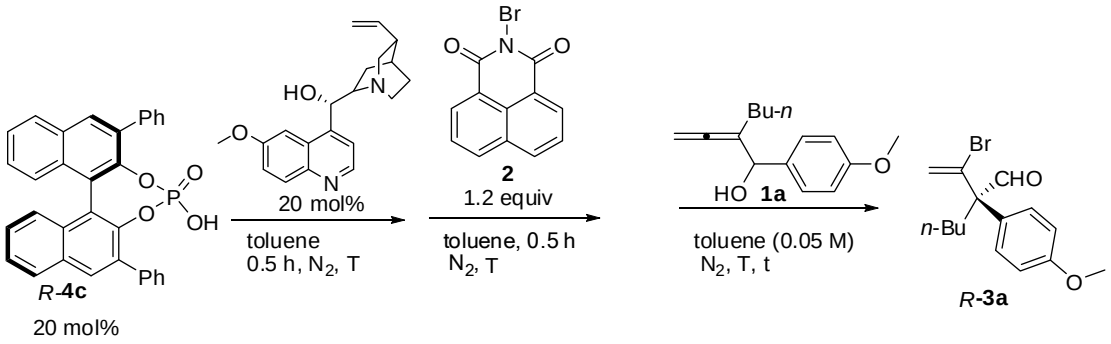
General methods:

^{1}H and ^{13}C NMR spectra were recorded with the an instrument operated at 300 and 75 MHz, respectively, in $CDCl_3$. Chemical shift (δ) are given in parts per million (ppm) with the residual peak of $CHCl_3$ at 7.260 ppm or TMS at 0.000 ppm as the internal standard. Infrared spectra were recorded with a Perkin–Elmer 983G instrument. Elemental analyses were recorded with a Carlo-Erba EA1110 elementary analysis instrument. Mass spectra were performed with an HP 5989A system. High-resolution mass spectra were recorded with a Finnigan MAT 8430 or Bruker APEXIII instrument. Flash column chromatography was performed on silica gel (10-40 μ). Toluene was refluxed over sodium wire using diphenyl ketone as indicator and distilled.

Part 1. Optimization of reaction conditions

All reactions in **Tables S1-6** were carried out with 0.1 mmol of **1a** unless otherwise noted. And the % ees of product **3a** were determined with the following HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ = 230 nm.

Table S1. The effect of temperature

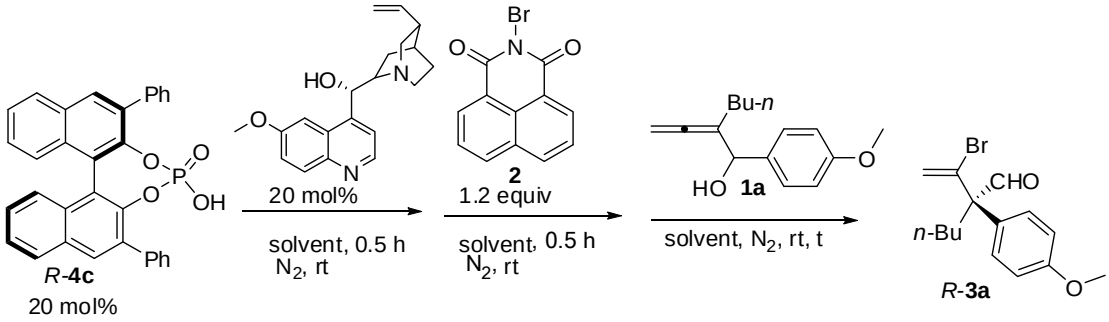


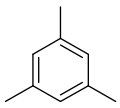
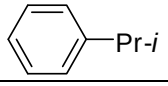
Entry	T (°C)	t (h)	Isolated yield of *R*-**3a** (%)	ee of *R*-**3a** (%)	No.
1[a]	-78	24	~30	30	7-37
2[a, b]	-78	24	~25	43	7-73
3	-30	25	~75	53	7-38
4[c]	0	13	90	65	6-196
5[c]	10	11	87	67	7-11
6[c]	15	3	84	68	7-13
7[c]	20	11.5	83	69	7-23
8	24	10.7	85	74	7-12
9	28	2.8	~83	68	7-39
10	40	2.4	79	59	7-78

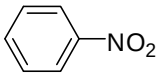
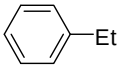
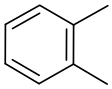
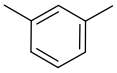
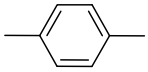
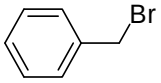
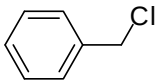
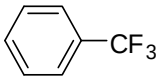
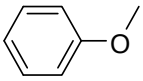
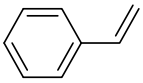
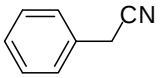
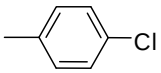
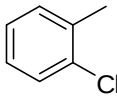
11[d]	24 (rt)	11	85	69	7-24
12[e]	23 (rt)	12	36	53	7-47
13[f]	23 (rt)	12.3	80	72	7-46
14[g]	24	5	80	68	7-25
15[h]	23	16	80	65	7-28

[a] Starting material **1a** was not completely converted. [b] The solution of quinidine and *R*-**4c** in 1 mL of toluene was stirred at rt for 0.5 h, **2** was then added and the resulting mixture was stirred for another 0.5 h at rt; then allenol **1a** and 1 mL of toluene were added; the resulting mixture was stirred at -78 ^{o}C; [c] The solution of quinidine and **2** in 1 mL of toluene was stirred for 0.5 h, *R*-**4c** was then added and the resulting mixture was stirred for another 0.5 h; then allenol **1a** and 1 mL of toluene were added; [d] **2** (0.2 equiv) was added every 1.5 h; [e] The solution of **1a** in 1 mL of toluene was added by syringe pump within 10 h; [f] The concentration was 0.025 M; [g] 4Å MS (40 mg) was added; [h] H_2O (3.0 equiv) of was added.

Table S2.The effect of solvent

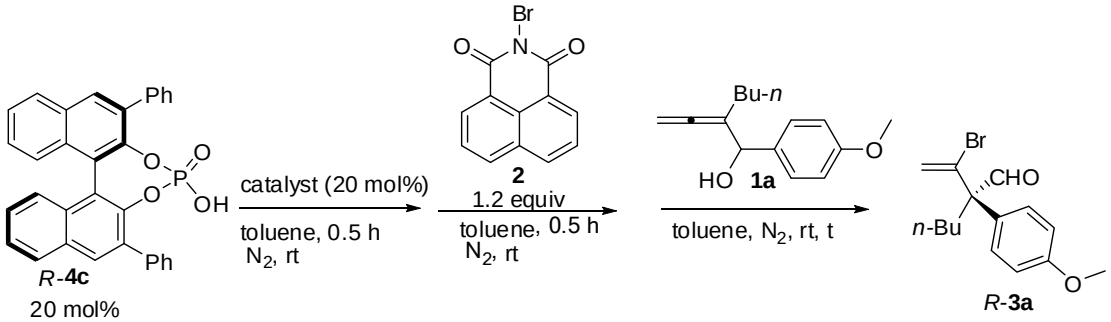


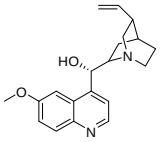
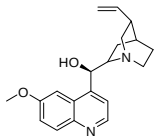
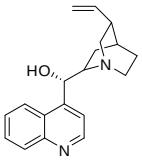
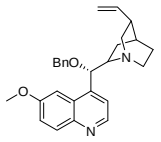
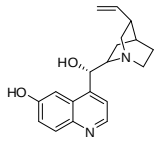
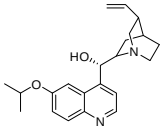
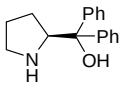
Entry	Solvent	t (h)	Isolated yield of *R*-**3a** (%)	ee of *R*-**3a** (%)	No.
1	DCE	1.8	92	27	7-31
2	$CHCl_3$	1.7	92	18	7-32
3	THF	21.7	88	0	7-33
4	MeCN	1.7	98	23	7-35
5[a]	$MeNO_2$	18	0	-	7-36
6	DMF	4.7	77	0	7-60
7	Et_2O	5	75	29	7-40
8[a]	*n*-hexane	24	~39	5	7-41
9	*c*-hexane	24	~67	10	7-44
10	benzene	11	~77	70	7-42
11	xylenes	4.3	~81	61	7-43
12	toluene	12.3	~80	72	7-46
13		11.3	76	52	7-93
14[a]		39.5	-	6	7-94

15		11.2	87	19	7-95
16	PhCl	11	77	55	7-104
17	CCl_4	3.5	85	56	7-97
18		11	75	3	7-126
19		5	73	60	7-128
20		5	74	51	7-129
21		11	75	64	7-127
22		5.5	~93	2	7-134
23		5.5	~86	0	7-135
24		2	~77	2	7-136
25		21.3	81	32	7-138
26		40.5	~58	17	7-139
27		21.3	~74	0	7-140
28		21.2	81	7	7-141
29		11	69	39	7-142

[a] Starting material **1a** was not completely converted.

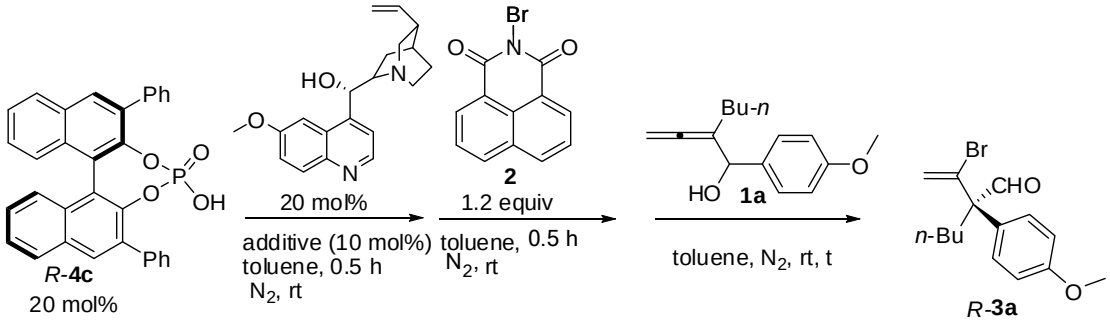
Table S3. The effect of quinidine derivatives

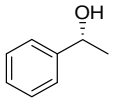
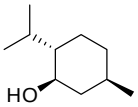
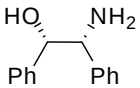
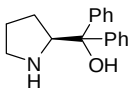


Entry	Catalyst (20 mol%)	t (h)	Isolated yield of *R*-**3a** (%)	ee of *R*-**3a** (%)	No.
1		12.3	80	72	7-46
2		1.5	75	-13	7-18
3		11	~76	2	7-49
4		7	~88	0	7-58
5		11	79	0	7-80
6		11.5	92	4	7-87
7		12.7	~85	0	9-23
8[a]	quinoline	11	80	-5	11-189
9[a]	PPh_3	12	80	0	11-188

[a] 15 mol% of catalyst and 5 mol% of *R*-**4c** were used.

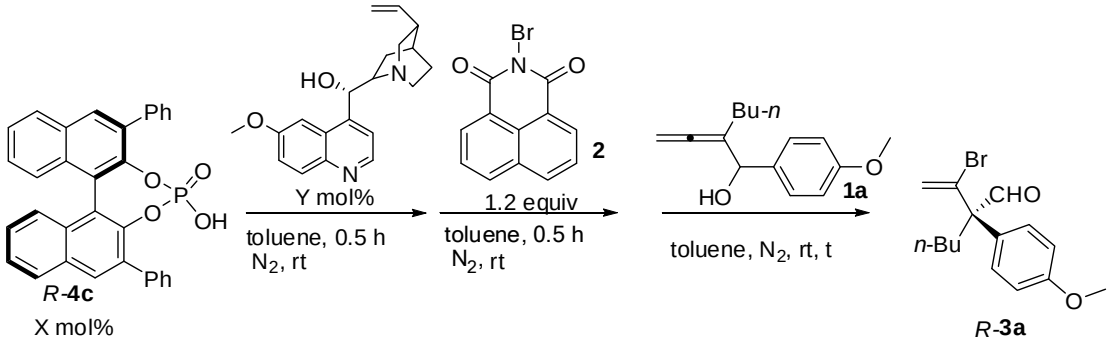
Table S4. The effect of additives



Entry	Additive (10 mol%)	t (h)	Isolated yield of *R*-**3a** (%)	ee of *R*-**3a** (%)	No.
1	-	11	73	71	8-38
2[a]	MeOH	3	82	74	8-95
3	EtOH	14	78	72	8-41
4	*i*-PrOH	6.5	75	72	8-49
5	*t*-BuOH	5.4	77	70	8-50
6[a]	*s*-Butanol	3	79	73	8-101
7[a]	CH_2OHCH_2OH	3	78	69	8-102
8		5	82	70	8-63
9		12	80	72	8-79
10		15.3	84	-2	8-78
11[a]		3.6	~69	72	8-183

[a] 5 mol% of *R*-**4c** and 15 mol% of quinidine were used.

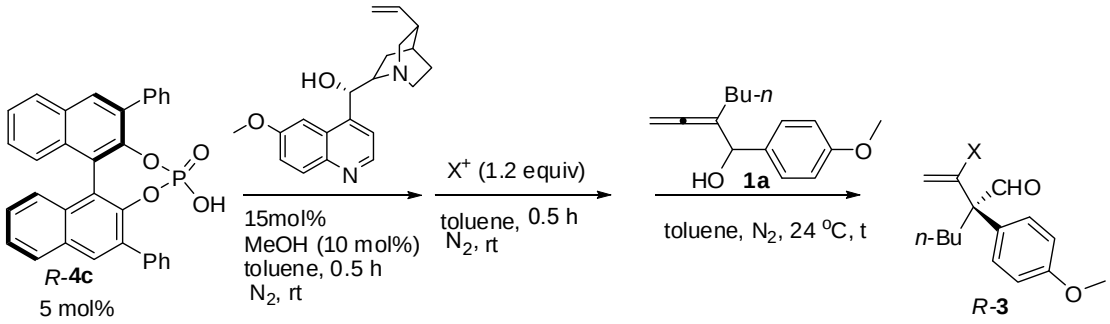
Table S5. The effect of the ratio of each component in the catalyst

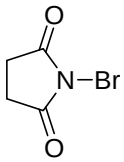
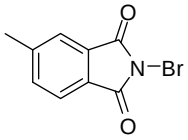
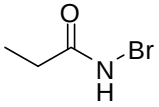
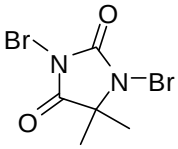
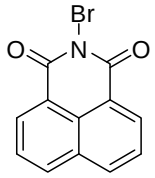
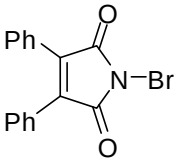
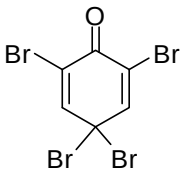


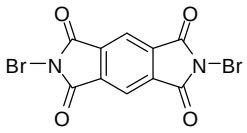
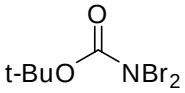
Entry	X (mol%)	Y (mol%)	t (h)	Isolated yield of *R*-**3a** (%)	ee of *R*-**3a** (%)	No.
1	30	30	1.3	83	70	7-48
2	20	20	12.3	80	72	7-46
3	15	20	3	79	72	7-66
4	10	20	3	79	71	7-67
5	5	20	3	81	68	7-68
6	5	15	1.3	~84	69	7-79
7[a]	5	15	3	82	74	8-95
8	5	10	4.3	79	61	6-179

[a] MeOH (10 mol%) was added.

Table S6. The effect of X^+ sources



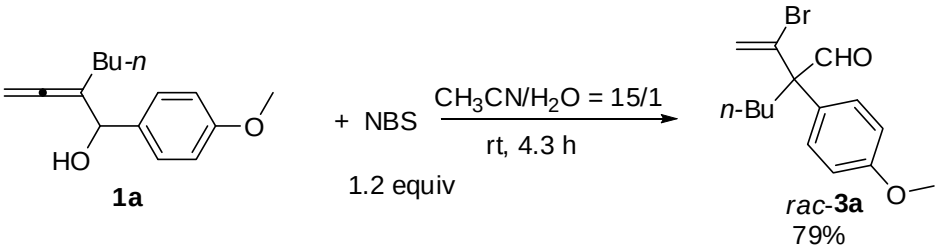
Entry	X^+	t (h)	Isolated yield of *R*-**3** (%)	ee of *R*-**3** (%)	No.
1		2.7	73 (*R*-**3a**)	27	8-107
2		2.6	76 (*R*-**3a**)	39	8-108
3		2.8	78 (*R*-**3a**)	28	8-110
4		1	68 (*R*-**3a**)	8	8-111
5		3	82 (*R*-**3a**)	74	8-95
6		1.3	~79 (*R*-**3a**)	32	8-112
7		1	~84 (*R*-**3a**)	0	8-119

8		11	~67 (*R*-**3a**)	28	8-117
9		2.3	~61 (*R*-**3a**)	43	9-9
10	NIS	25	~50 (*R*-**3a'**, X= I)[a]	11	8-123
11	NCS	49	~45(*R*-**3a"**, X = Cl)[a]	0	8-125

[a] These two compounds have not been fully characterized due to the issue of purity.

Part 2. Synthesis of racmic products *rac*-**3a~3q**

1. Synthesis of 3-bromo-2-butyl-2-(4-methoxyphenyl)-3-butenal *rac*-**3a** (Gbj-8-165)

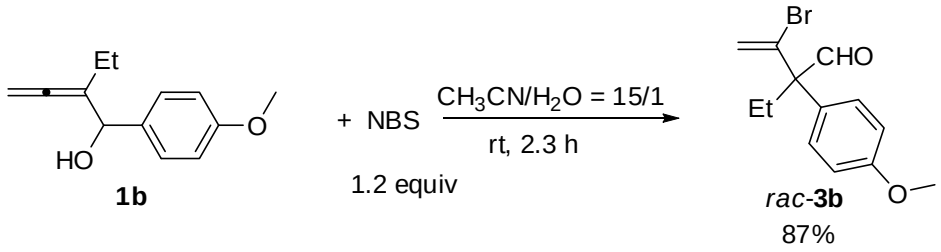


Typical Procedure I: To a solution of **1a** (116.3 mg, 0.5 mmol) in MeCN (4.5 mL) and H_2O (0.3 mL) was added NBS (106.9 mg, 0.6 mmol). The resulting mixture was stirred at room temperature. After the reaction was complete as monitored by TLC, the mixture was then quenched with a saturated aqueous solution of $Na_2S_2O_3$ (4 mL), which was followed by the addition of 10 mL of water. This resulting mixture was extracted with diethyl ether (3×15 mL), washed with a saturated aqueous solution of NaCl, and dried over Na_2SO_4. Filtration, evaporation, and column chromatography on silica gel (petroleum ether/ethyl acetate = 50:1) afforded *rac*-**3a**[1] (123.2 mg, 79%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H, CHO), 7.25 (d, *J* = 8.1 Hz, 2H, ArH), 6.93 (d, *J* = 8.4 Hz, 2H, ArH), 6.02 (d, *J* = 1.8 Hz, 1H, =CH), 5.94 (d, *J* = 2.1 Hz, 1H =CH), 3.81 (s, 3H, OCH_3), 2.28-2.14 (m, 1H, one proton of CH_2), 2.13-1.97 (m, 1H one proton of CH_2), 1.50-1.09 (m, 4H, 2×CH_2), 0.94 (t, *J* = 7.4 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.8, 159.2, 134.3, 129.3, 128.5, 121.1, 114.2, 65.0, 55.2, 31.4, 26.9, 23.1, 13.9; IR (neat) *v* (cm^{-1}) 2957, 2933, 2871, 2837, 2719, 1728, 1607, 1580, 1511, 1464, 1442, 1417, 1380, 1298, 1254, 1184, 1094, 1037; MS (70 eV, EI) *m/z* (%): 312 (M^+(^{81}Br), 2.48), 310 (M^+(^{79}Br), 3.31), 160 (100).

The following compounds (*rac*-**3b~3q**) were prepared according to **Typical**

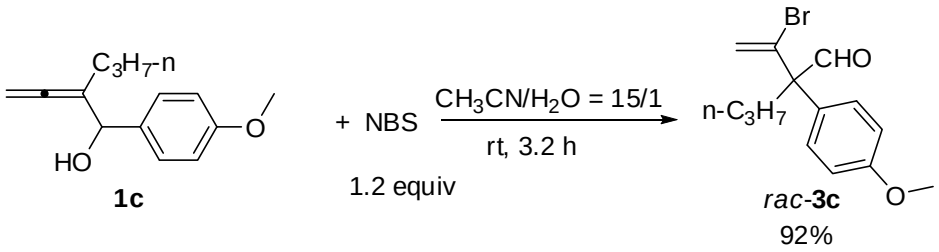
Procedure I.

2. Synthesis of 3-bromo-2-ethyl-2-(4-methoxyphenyl)-3-butenal *rac*-**3b** (gbj-8-181)



The reaction of **1b** (102.6 mg, 0.5 mmol) and NBS (106.3 mg, 0.6 mmol) in 4.5 mL of MeCN and 0.3 mL of H_2O at rt for 2.3 h afforded *rac*-**3b**[1] (123.3 mg, 87%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H, CHO), 7.25 (d, *J* = 9.0 Hz, 2H, ArH), 6.93 (d, *J* = 9.3 Hz, 2H, ArH), 6.02 (d, *J* = 2.7 Hz, 1H, =CH), 5.96 (d, *J* = 3.0 Hz, 1H, =CH), 3.80 (s, 3H, OCH_3), 2.40-2.22 (m, 1H, one proton of CH_2), 2.19-2.01 (m, 1H one proton of CH_2), 0.93 (t, *J* = 7.4 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.6, 159.1, 133.9, 129.3, 128.3, 121.3, 114.2, 65.4, 55.1, 24.4, 9.2; IR (neat) *v* (cm^{-1}) 2971, 2936, 2881, 2837, 2723, 1728, 1608, 1580, 1511, 1463, 1442, 1416, 1383, 1299, 1255, 1185, 1153, 1086, 1034; MS (70 eV, EI) *m/z* (%): 284 (M^+(^{81}Br), 3.51), 282 (M^+(^{79}Br), 2.54), 174 (100).

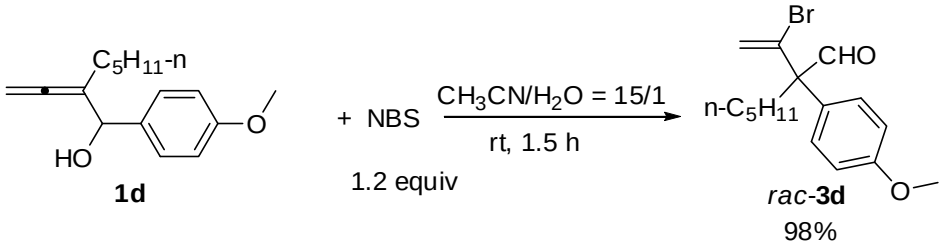
3. Synthesis of 3-bromo-2-propyl-2-(4-methoxyphenyl)-3-butenal *rac*-**3c** (gbj-8-179)



The reaction of **1c** (109.4 mg, 0.5 mmol) and NBS (107.2 mg, 0.6 mmol) in 4.5

mL of MeCN and 0.3 mL of H_2O at rt for 3.2 h afforded *rac*-**3c** (136.9 mg, 92%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H, CHO), 7.24 (d, *J* = 8.7 Hz, 2H, ArH), 6.92 (d, *J* = 8.7 Hz, 2H, ArH), 6.01 (d, *J* = 2.7 Hz, 1H, =CH), 5.93 (d, *J* = 2.4 Hz, 1H, =CH), 3.80 (s, 3H, OCH_3), 2.27-2.12 (m, 1H, one proton of CH_2), 2.11-1.95 (m, 1H, one proton of CH_2), 1.45-1.12 (m, 2H, CH_2), 1.00 (t, *J* = 7.4 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.7, 159.1, 134.2, 129.3, 128.5, 121.0, 114.2, 65.1, 55.1, 33.8, 18.2, 14.5; IR (neat) *v* (cm^{-1}) 3000, 2960, 2933, 2873, 2837, 2719, 1726, 1608, 1580, 1511, 1464, 1442, 1417, 1380, 1302, 1255, 1184, 1091, 1035; MS (70 eV, EI) *m/z* (%): 298 (M^+(^{81}Br), 3.76), 296 (M^+(^{79}Br), 4.68), 188 (100); HRMS calcd for $C_{14}H_{17}O_2{}^{79}Br$ (M^+): 296.0412. Found: 296.0408.

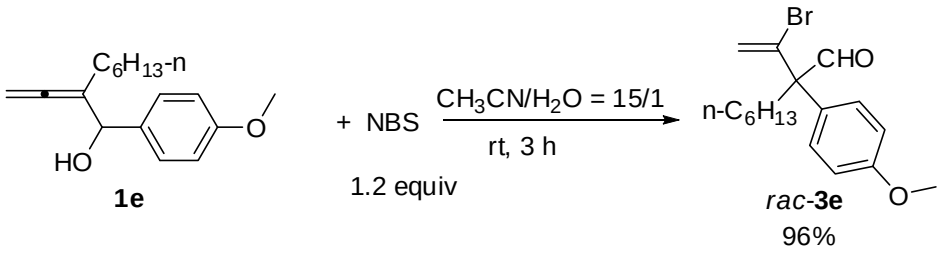
4. Synthesis of 3-bromo-2-pentyl-2-(4-methoxyphenyl)-3-butenal *rac*-**3d** (Gbj-8-176)



The reaction of **1d** (123.1 mg, 0.5 mmol) and NBS (107.2 mg, 0.6 mmol) in 4.5 mL of MeCN and 0.3 mL of H_2O at rt for 1.5 h afforded *rac*-**3d** (158.9 mg, 98%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H, CHO), 7.24 (d, *J* = 8.7 Hz, 2H, ArH), 6.92 (d, *J* = 8.7 Hz, 2H, ArH), 6.02 (d, *J* = 2.7 Hz, 1H, =CH), 5.93 (d, *J* = 2.7 Hz, 1H, =CH), 3.79 (s, 3H, OCH_3), 2.28-2.13 (m, 1H, one proton of CH_2), 2.12-1.95 (m, 1H, one proton of CH_2), 1.47-1.12 (m, 6H, 3×CH_2), 0.90 (t, *J* = 6.6 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.7, 159.1, 134.3, 129.3, 128.5, 121.0, 114.2, 65.0,

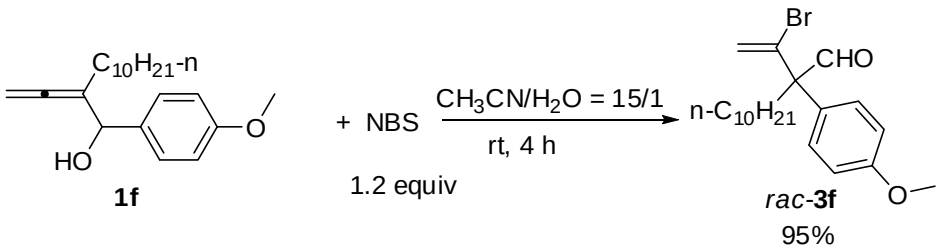
55.1, 32.1, 31.6, 24.4, 22.4, 14.0; IR (neat) ν (cm^{-1}) 3000, 2955, 2929, 2870, 2834, 2719, 1732, 1607, 1580, 1514, 1463, 1438, 1417, 1379, 1299, 1255, 1184, 1096, 1036; MS (70 eV, EI) *m/z* (%): 326 (M^+(^{81}Br), 5.28), 324 (M^+(^{79}Br), 5.51), 160 (100); HRMS calcd for $C_{16}H_{21}O_2{}^{79}Br$ (M^+): 324.0725. Found: 324.0728.

5. Synthesis of 3-bromo-2-hexyl-2-(4-methoxyphenyl)-3-butenal *rac*-**3e** (gbj-8-184)



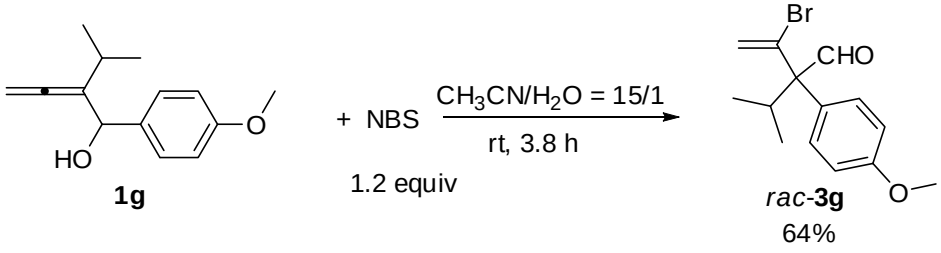
The reaction of **1e** (130.5 mg, 0.5 mmol) and NBS (107.5 mg, 0.6 mmol) in 4.5 mL of MeCN and 0.3 mL of H_2O at rt for 3 h afforded *rac*-**3e** (163.8 mg, 96%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H, CHO), 7.24 (d, J = 8.4 Hz, 2H, ArH), 6.92 (d, J = 8.7 Hz, 2H, ArH), 6.02 (d, J = 2.7 Hz, 1H, =CH), 5.93 (d, J = 2.7 Hz, 1H, =CH), 3.80 (s, 3H, OCH_3), 2.29-2.14 (m, 1H, one proton of CH_2), 2.13-1.97 (m, 1H, one proton of CH_2), 1.48-1.10 (m, 8H, 4×CH_2), 0.89 (t, J = 6.3 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.7, 159.1, 134.3, 129.3, 128.5, 121.0, 114.2, 65.0, 55.1, 31.6, 31.5, 29.6, 24.7, 22.6, 14.0; IR (neat) ν (cm^{-1}) 2997, 2953, 2929, 2856, 2719, 1727, 1607, 1580, 1511, 1464, 1442, 1417, 1378, 1299, 1255, 1184, 1144, 1097, 1035; MS (70 eV, EI) *m/z* (%): 340 (M^+(^{81}Br), 4.69), 338 (M^+(^{79}Br), 4.50), 160 (100); HRMS calcd for $C_{17}H_{23}O_2{}^{79}Br$ (M^+): 338.0881. Found: 338.0887.

6. Synthesis of 3-bromo-2-decyl-2-(4-methoxyphenyl)-3-butenal *rac*-**3f** (Gbj-8-174)



The reaction of **1f** (158.6 mg, 0.5 mmol) and NBS (107.4 mg, 0.6 mmol) in 4.5 mL of MeCN and 0.3 mL of H_2O at rt for 4 h afforded *rac*-**3f** (187.8 mg, 95%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H CHO), 7.24 (d, *J* = 8.7 Hz, 2H, ArH), 6.92 (d, *J* = 8.7 Hz, 2H, ArH), 6.02 (d, *J* = 2.7 Hz, 1H, =CH), 5.93 (d, *J* = 2.4 Hz, 1H, =CH), 3.80 (s, 3H, OCH_3), 2.28-2.13 (m, 1H, one proton of CH_2), 2.13-1.96 (m, 1H, one proton of CH_2), 1.46-1.12 (m, 16H, 8×CH_2), 0.88 (t, *J* = 6.6 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.8, 159.2, 134.3, 129.3, 128.6, 121.1, 114.2, 65.1, 55.2, 31.9, 31.7, 30.0, 29.6, 29.4, 29.3, 24.8, 22.7, 14.1; IR (neat) *v* (cm^{-1}) 3000, 2953, 2925, 2853, 2718, 1731, 1608, 1580, 1512, 1464, 1442, 1417, 1378, 1299, 1255, 1184, 1142, 1101, 1037; MS (70 eV, EI) *m/z* (%): 396 (M^+(^{81}Br), 2.29), 394 (M^+(^{79}Br), 2.63), 160 (100); HRMS calcd for $C_{21}H_{31}O_2{}^{79}Br$ (M^+): 394.1507. Found: 394.1517.

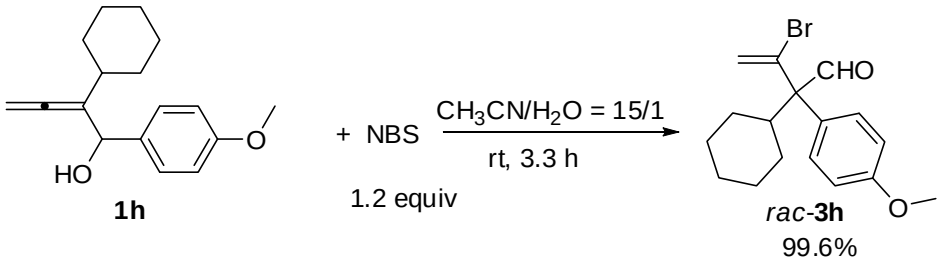
7. Synthesis of 3-bromo-2-isopropyl-2-(4-methoxyphenyl)-3-butenal *rac*-**3g** (gbj-9-16)



The reaction of **1g** (109.2 mg, 0.5 mmol) and NBS (106.7 mg, 0.6 mmol) in 4.5

mL of MeCN and 0.3 mL of H_2O at rt for 3.8 h afforded *rac*-**3g** (95.4 mg, 64%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H, CHO), 7.17 (d, *J* = 8.7 Hz, 2H, ArH), 6.93 (d, *J* = 9.0 Hz, 2H, ArH), 6.02 (d, *J* = 2.7 Hz, 1H, =CH), 5.98 (d, *J* = 2.4 Hz, 1H, =CH), 3.82 (s, 3H, OCH_3), 2.92 (heptet, *J* = 6.8 Hz, 1H, CH), 0.97 (d, *J* = 6.9 Hz, 3H, CH_3), 0.92 (d, *J* = 6.9 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.8, 158.8, 133.0, 130.7, 127.3, 123.4, 113.6, 69.2, 55.2, 30.4, 18.5, 18.2; IR (neat) *v* (cm^{-1}) 3036, 2967, 2936, 2877, 2837, 2720, 1727, 1610, 1579, 1513, 1490, 1464, 1442, 1389, 1370, 1296, 1255, 1186, 1124, 1097, 1073, 1035; MS (70 eV, EI) *m/z* (%): 298 (M^+(^{81}Br), 14.21), 296 (M^+(^{79}Br), 13.37), 188 (100); HRMS calcd for $C_{14}H_{17}O_2{}^{79}Br$ (M^+): 296.0412. Found: 296.0422.

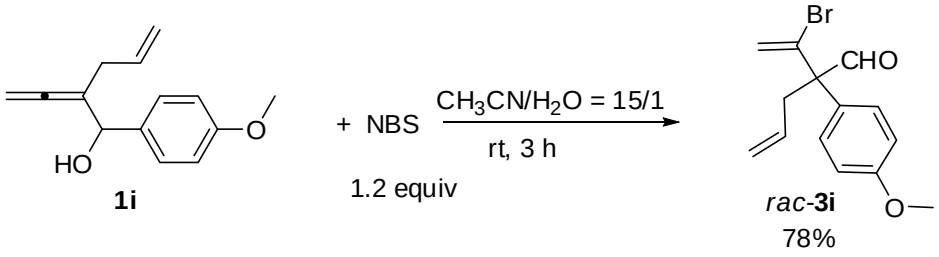
8. Synthesis of 3-bromo-2-cyclohexyl-2-(4-methoxyphenyl)-3-butenal *rac*-**3h** (gbj-9-14)



The reaction of **1h** (129.1 mg, 0.5 mmol) and NBS (107.4 mg, 0.6 mmol) in 4.5 mL of MeCN and 0.3 mL of H_2O at rt for 3.3 h afforded *rac*-**3h** (167.9 mg, 99.6%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.57 (s, 1H, CHO), 7.14 (d, *J* = 8.7 Hz, 2H, ArH), 6.92 (d, *J* = 9.0 Hz, 2H, ArH), 6.00 (d, *J* = 2.4 Hz, 1H, =CH), 5.96 (d, *J* = 2.4 Hz, 1H, =CH), 3.82 (s, 3H, OCH_3), 2.51 (t, *J* = 11.7 Hz, 1H, CH), 1.92-1.53 (m, 5H, 2 $\times CH_2$ and one proton of CH_2), 1.47-1.22 (m, 2H, CH_2), 1.16-0.74 (m, 3H, CH_2 and

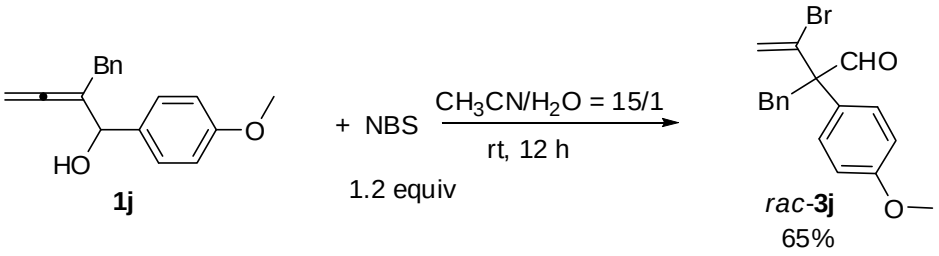
one proton of CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.6, 158.7, 133.1, 130.7, 127.5, 123.3, 113.6, 69.2, 55.1, 41.2, 28.9, 28.6, 26.8, 26.7, 26.4; IR (neat) *v* (cm^{-1}) 3001, 2932, 2853, 2714, 1727, 1609, 1579, 1512, 1462, 1453, 1417, 1296, 1255, 1185, 1154, 1123, 1037; MS (70 eV, EI) *m/z* (%): 338 (M^+(^{81}Br), 10.28), 336 (M^+(^{79}Br), 10.30), 307 (100); HRMS calcd for $C_{17}H_{21}O_2{}^{79}Br$ (M^+): 336.0725. Found: 336.0728.

9. Synthesis of 2-allyl-3-bromo-2-(4-methoxyphenyl)-3-butenal *rac*-**3i** (gbj-8-198)



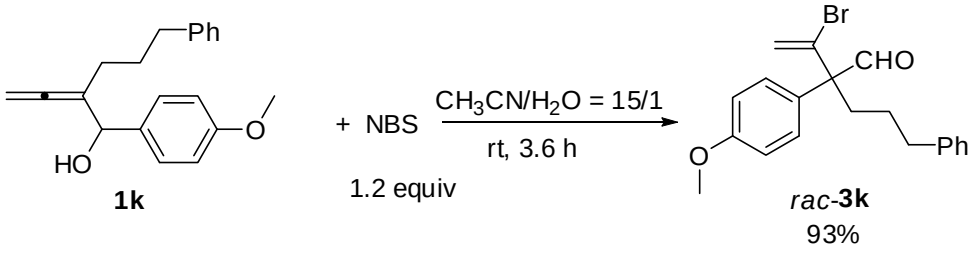
The reaction of **1i** (107.9 mg, 0.5 mmol) and NBS (107.1 mg, 0.6 mmol) in 4.5 mL of MeCN and 0.3 mL of H_2O at rt for 3 h afforded *rac*-**3i**[1] (114.4 mg, 78%): Liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 9.64 (s, 1H, CHO), 7.24 (d, *J* = 8.7 Hz, 2H, ArH), 6.93 (d, *J* = 9.0 Hz, 2H, ArH), 6.06-5.85 (m, 2H, $=CH_2$), 5.82-5.60 (m, 1H, =CH), 5.19 (dd, *J* = 17.1 Hz and 0.9 Hz, 1H, one proton of $=CH_2$), 5.11 (d, *J* = 10.2 Hz, 1H, one proton of $=CH_2$), 3.80 (s, 3H, OCH_3), 3.05 (dd, *J* = 14.1 and 6.6 Hz, 1H, one proton of CH_2), 2.86 (dd, *J* = 14.3 and 7.4 Hz, 1H, one proton of CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.7, 159.2, 133.6, 132.7, 129.4, 127.9, 121.5, 118.8, 114.2, 64.7, 55.2, 36.4; IR (neat) *v* (cm^{-1}) 3078, 3036, 3005, 2977, 2957, 2934, 2911, 2837, 2720, 1727, 1640, 1608, 1580, 1512, 1463, 1442, 1417, 1299, 1255, 1185, 1098, 1033; MS (70 eV, EI) *m/z* (%): 296 (M^+(^{81}Br), 14.33), 294 (M^+(^{79}Br), 15.50), 145 (100).

10. Synthesis of 2-benzyl-3-bromo-2-(4-methoxyphenyl)-3-butenal *rac*-**3j** (gbj-10-78)



The reaction of **1j** (133.2 mg, 0.5 mmol) and NBS (107.3 mg, 0.6 mmol) in 4.5 mL of MeCN and 0.3 mL of H_2O at rt for 12 h afforded *rac*-**3j** (112.3 mg, 65%): Oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.66 (s, 1H, CHO), 7.34-7.05 (m, 7H, ArH), 6.91 (d, *J* = 9.0 Hz, 2H, ArH), 5.82 (s, 2H, =CH_2), 3.81 (s, 3H, OCH_3), 3.65 (d, *J* = 13.2 Hz, 1H, one proton of $ArCH_2$), 3.42 (d, *J* = 13.2 Hz, 1H, one proton of $ArCH_2$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.4, 159.3, 136.2, 133.3, 130.7, 129.6, 128.5, 127.8, 126.7, 122.2, 114.2, 66.7, 55.2, 38.3; IR (neat) *v* (cm^{-1}) 3060, 3032, 3006, 2956, 2931, 2838, 2722, 1722, 1620, 1604, 1581, 1511, 1455, 1438, 1418, 1294, 1259, 1185, 1123, 1078, 1033; MS (70 eV, EI) *m/z* (%): 346 (M^+(^{81}Br), 13.11), 344 (M^+(^{79}Br), 11.84), 174 (100); HRMS calcd for $C_{18}H_{17}O_2{}^{79}Br$ (M^+): 344.0412. Found: 344.0410.

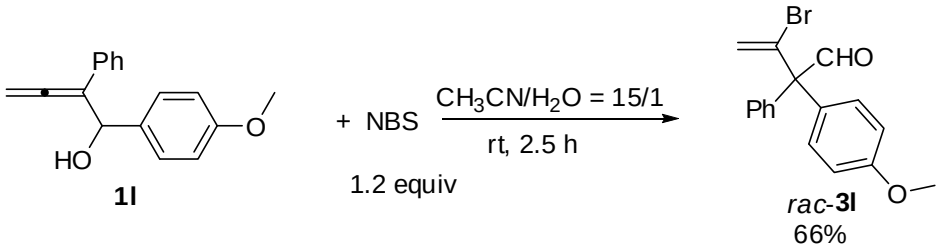
11. Synthesis of 3-bromo-2-(3-phenylpropyl)-2-(4-methoxyphenyl)-3-butenal *rac*-**3k** (gbj-9-1)



The reaction of **1k** (147.5 mg, 0.5 mmol) and NBS (107.3 mg, 0.6 mmol) in 4.5 mL of MeCN and 0.3 mL of H_2O at rt for 3.6 h afforded *rac*-**3k** (173.6 mg, 93%):

solid; mp. 68.7~71.0 °C (Et_2O/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.58 (s, 1H, CHO), 7.38-7.08 (m, 7H, ArH), 6.90 (d, *J* = 9.0 Hz, 2H, ArH), 5.95 (d, *J* = 2.7 Hz, 1H, =CH), 5.91 (d, *J* = 2.4 Hz, 1H, =CH), 3.79 (s, 3H, OCH_3), 2.84-2.60 (m, 2H, CH_2), 2.37-2.19 (m, 1H, one proton of CH_2), 2.17-1.98 (m, 1H, one proton of CH_2), 1.74-1.46 (m, 2H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.4, 159.1, 141.7, 134.0, 129.2, 128.3, 128.2, 128.1, 125.8, 121.2, 114.2, 64.9, 55.1, 36.0, 31.0, 26.5; IR (KBr) *v* (cm^{-1}) 3084, 3061, 3026, 3002, 2950, 2935, 2836, 2721, 1727, 1606, 1580, 1511, 1454, 1442, 1417, 1299, 1256, 1185, 1122, 1082, 1034; MS (70 eV, EI) *m/z* (%): 374 (M^+(^{81}Br), 2.25), 372 (M^+(^{79}Br), 2.48), 160 (100); Anal. calcd. for $C_{20}H_{21}BrO_2$: C 64.35, H 5.67; found: C 64.42, H 5.70.

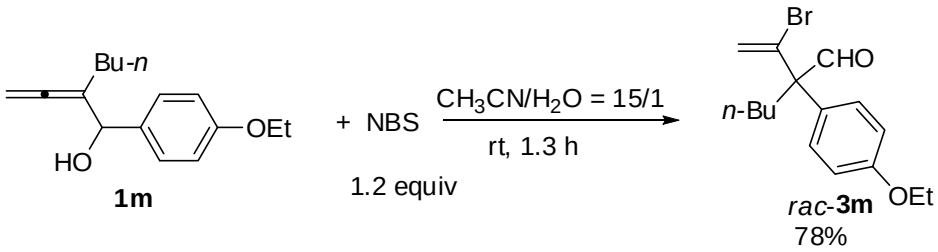
12. Synthesis of 3-bromo-2-phenyl-2-(4-methoxyphenyl)-3-butenal *rac*-**3l** (gbj-11-144)



The reaction of **1l** (125.3 mg, 0.5 mmol) and NBS (107.4 mg, 0.6 mmol) in 4.5 mL of MeCN and 0.3 mL of H_2O at rt for 2.5 h afforded *rac*-**3l** (108.5 mg, 66%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.11 (s, 1H, CHO), 7.44-7.30 (m, 3H, ArH), 7.28-7.19 (m, 2H, ArH), 7.17-7.08 (m, 2H, ArH), 6.94-6.87 (m, 2H, ArH), 6.00 (d, *J* = 2.4 Hz, 1H, one proton of $=CH_2$), 5.69 (d, *J* = 2.4 Hz, 1H, one proton of $=CH_2$), 3.82 (s, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.4, 159.2, 137.2, 133.4, 131.4, 130.1,

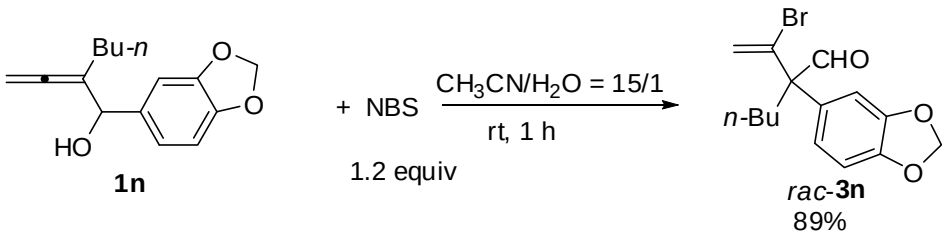
128.6, 128.5, 128.0, 124.4, 113.9, 71.0, 55.3; IR (neat) ν (cm^{-1}) 3059, 3033, 3003, 2956, 2932, 2837, 2729, 1732, 1608, 1580, 1505, 1463, 1446, 1417, 1299, 1255, 1185, 1116, 1088, 1035; MS (70 eV, EI) *m/z* (%): 332 (M^+(^{81}Br), 0.51), 330 (M^+(^{79}Br), 0.66), 303 (M^+(^{81}Br)-CHO, 35.54), 301 ((M^+(^{79}Br)-CHO, 37.30), 222 (100); HRMS calcd for $C_{17}H_{15}O_2{}^{79}Br$ (M^+): 330.0255. Found: 330.0264.

13. Synthesis of 3-bromo-2-butyl-2-(4-ethoxyphenyl)-3-butenal *rac*-**3m** (gbj-9-29)



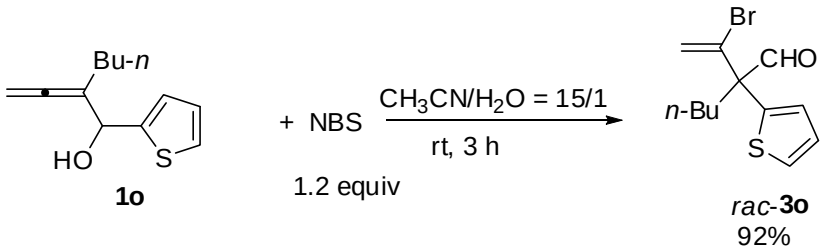
The reaction of **1m** (123.4 mg, 0.5 mmol) and NBS (106.5 mg, 0.6 mmol) in 4.5 mL of MeCN and 0.3 mL of H_2O at rt for 1.3 h afforded *rac*-**3m** (126.5 mg, 78%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.59 (s, 1H, CHO), 7.23 (d, J = 8.7 Hz, 2H, ArH), 6.91 (d, J = 8.7 Hz, 2H, ArH), 6.01 (d, J = 2.4 Hz, 1H, =CH), 5.92 (d, J = 2.7 Hz, 1H, =CH), 4.02 (q, J = 6.9 Hz, 2H, CH_2), 2.30-2.13 (m, 1H, one proton of CH_2), 2.13-1.93 (m, 1H, one proton of CH_2), 1.52-1.06 (m, 7H, CH_3 and 2×CH_2), 0.93 (t, J = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.7, 158.5, 134.3, 129.3, 128.3, 121.0, 114.7, 65.0, 63.3, 31.3, 26.9, 23.1, 14.7, 13.9; IR (neat) ν (cm^{-1}) 2957, 2931, 2872, 2819, 2719, 1727, 1608, 1579, 1511, 1477, 1393, 1296, 1253, 1185, 1117, 1093, 1047, 1011; MS (70 eV, EI) *m/z* (%): 326 (M^+(^{81}Br), 2.78), 324 (M^+(^{79}Br), 2.77), 174 (100); HRMS calcd for $C_{16}H_{21}O_2{}^{79}Br$ (M^+): 324.0725. Found: 324.0726.

14. Synthesis of 3-bromo-2-butyl-2-(3,4-methylenedioxyphenyl)-3-butenal *rac*-**3n** (gbj-8-190)



The reaction of **1n** (122.6 mg, 0.5 mmol) and NBS (107.4 mg, 0.6 mmol) in 4.5 mL of MeCN and 0.3 mL of H_2O at rt for 1 h afforded *rac*-**3n**[1] (144.8 mg, 89%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.58 (s, 1H, CHO), 6.88-6.73 (m, 3H, ArH), 6.04 (d, J = 2.4 Hz, 1H, =CH), 5.97 (s, 2H, OCH_2O), 5.93 (d, J = 2.7 Hz, 1H, =CH), 2.26-2.10 (m, 1H, one proton of CH_2), 2.10-1.93 (m, 1H one proton of CH_2), 1.50-1.08 (m, 4H, 2×CH_2), 0.93 (t, J = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.4, 148.2, 147.3, 134.0, 130.4, 121.7, 121.1, 108.4, 108.3, 101.3, 65.1, 31.5, 26.9, 23.0, 13.9; IR (neat) v (cm^{-1}) 2957, 2931, 2872, 2774, 2716, 1727, 1618, 1504, 1487, 1438, 1380, 1351, 1243, 1166, 1112, 1094, 1040; MS (70 eV, EI) *m/z* (%): 326 (M^+(^{81}Br), 25.41), 324 (M^+(^{79}Br), 24.65), 115 (100).

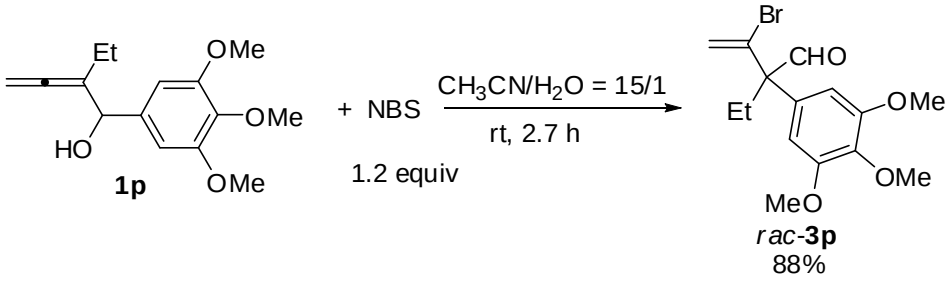
15. Synthesis of 3-bromo-2-butyl-2-thienyl-3-butenal *rac*-**3o** (gbj-9-51)



The reaction of **1o** (103.5 mg, 0.5 mmol) and NBS (106.9 mg, 0.6 mmol) in 4.5

mL of MeCN and 0.3 mL of H_2O at rt for 3 h afforded *rac*-**3o** (132.0 mg, 92%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.58 (s, 1H, CHO), 7.36 (dd, *J* = 5.0 Hz and 1.4 Hz, 1H, ArH), 7.12-6.96 (m, 2H, ArH), 6.03 (d, *J* = 3.0 Hz, 1H, =CH), 5.93 (d, *J* = 2.7 Hz, 1H, =CH), 2.32-2.07 (m, 2H, CH_2), 1.52-1.17 (m, 4H, 2×CH_2), 0.93 (t, *J* = 7.1 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 193.8, 140.5, 133.1, 127.2, 127.1, 126.4, 121.1, 63.6, 33.4, 26.6, 22.9, 13.9; IR (neat) *v* (cm^{-1}) 3108, 3069, 2957, 2931, 2871, 2816, 2714, 1732, 1619, 1466, 1429, 1380, 1238, 1156, 1095, 1047; MS (70 eV, EI) *m/z* (%): 288 (M^+(^{81}Br), 3.43), 286 (M^+(^{79}Br), 2.48), 257 (100); HRMS calcd for $C_{12}H_{15}O_2S^{79}Br$ (M^+): 286.0027. Found: 286.0023.

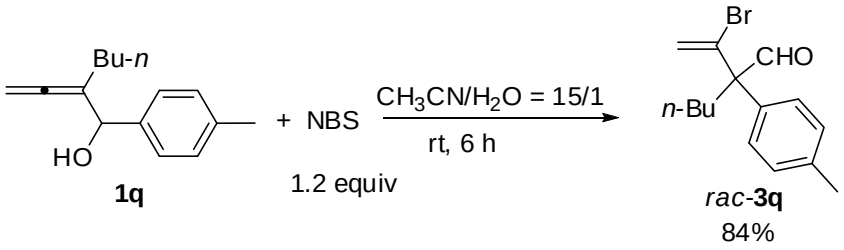
16. Synthesis of 3-bromo-2-ethyl-2-(3,4,5-trimethoxyphenyl)-3-butenal *rac*-**3p** (gbj-11-84)



The reaction of **1p** (132.1 mg, 0.5 mmol) and NBS (107.3 mg, 0.6 mmol) in 4.5 mL of MeCN and 0.3 mL of H_2O at rt for 2.7 h afforded *rac*-**3p** (150.6 mg, 88%) (petroleum ether/ethyl acetate = 10/1): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.62 (s, 1H, CHO), 6.53 (s, 2H, ArH), 6.06 (d, *J* = 1.8 Hz, 1H, =CH), 6.01 (d, *J* = 2.4 Hz, 1H, =CH), 3.86 (s, 9H, 3×OCH_3), 2.40-2.22 (m, 1H, one proton of CH_2), 2.20-2.02 (m, 1H, one proton of CH_2), 0.94 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.3, 153.3, 137.8, 133.3, 131.9, 121.7, 105.4, 66.1, 60.8, 56.1, 24.6, 9.3; IR (neat) *v*

(cm^{-1}) 2970, 2938, 2881, 2835, 2724, 1727, 1620, 1588, 1510, 1455, 1416, 1382, 1321, 1247, 1187, 1128, 1007; MS (70 eV, EI) *m/z* (%): 344 (M^+(^{81}Br), 3.02), 342 (M^+(^{79}Br), 3.58), 195 (100); HRMS calcd for $C_{15}H_{19}O_4{}^{79}Br$ (M^+): 342.0467. Found: 342.0473.

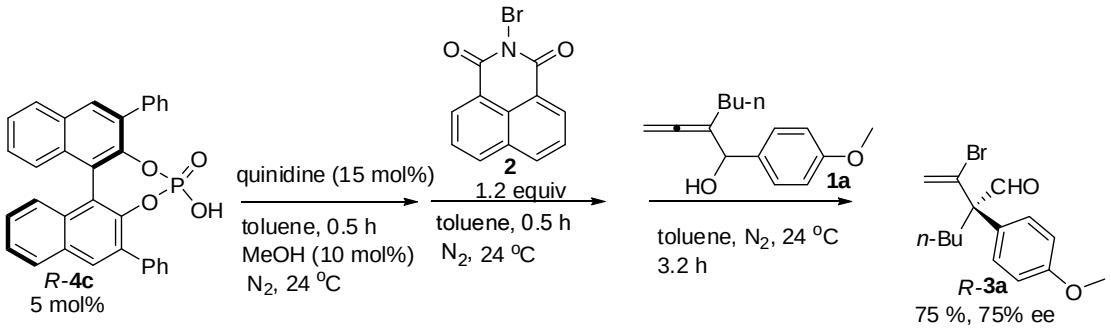
17. Synthesis of 3-bromo-2-butyl-2-(4-methylphenyl)-3-butenal *rac*-**3q** (gbj-11-198)



The reaction of **1q** (107.5 mg, 0.5 mmol) and NBS (107.1 mg, 0.6 mmol) in 4.5 mL of MeCN and 0.3 mL of H_2O at rt for 6 h afforded *rac*-**3q**[1] (124.0 mg, 84%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.63 (s, 1H, CHO), 7.21 (s, 4H, ArH), 6.03 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 5.95 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 2.35 (s, 3H, CH_3), 2.29-2.16 (m, 1H, one proton of CH_2), 2.15-2.00 (m, 1H one proton of CH_2), 1.48-1.08 (m, 4H, 2×CH_2), 0.93 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.1, 137.9, 134.1, 133.7, 129.6, 128.0, 121.2, 65.4, 31.5, 26.9, 23.1, 21.1, 13.9; IR (neat) *v* (cm^{-1}) 3024, 2957, 2930, 2871, 2718, 1729, 1619, 1510, 1466, 1412, 1376, 1160, 1093, 1019; MS (70 eV, EI) *m/z* (%): 296 (M^+(^{81}Br), 1.55), 294 (M^+(^{79}Br), 1.68), 143 (100).

Part 3. Synthesis of optically active product *R*-**3a~3n**, *S*-**3o** and *R*-**3p~3q**

1. Synthesis of (+)-3-bromo-2-butyl-2-(4-methoxyphenyl)-3-butenal *R*-**3a** (gbj-8-170)

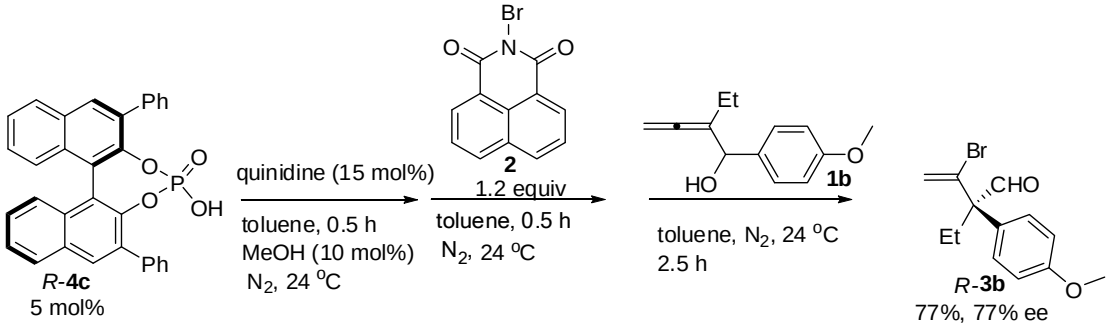


Typical procedure II: To a dried 50 mL rubber-capped round bottomed Schlenk vessel were added quinidine (24.5 mg, 0.075 mmol), *R*-**4c** (12.5 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), and 5 mL of toluene. After being stirred at 24 °C for 0.5 h, *N*-bromo-1,8-naphthalimide **2** (165.5 mg, 0.6 mmol) was added and the resulting mixture was stirred at 24 °C for another 0.5 h, which was followed by the squential addition of allenol **1a** (116.5 mg, 0.5 mmol) and 5 mL of toluene. The resulting mixture was stirred at 24 °C until the reaction was complete as monitored by TLC after 3.2 h. A saturated aqueous solution of $Na_2S_2O_3$ (4 mL) and 10 mL of H_2O were added to quench the reaction. This resulting mixture was extracted with diethyl ether (3×15 mL), washed with a saturated aqueous solution of NaCl and dried over anhydrous Na_2SO_4. Filtration, evaporation and purification by chromatography (petroleum ether/ethyl acetate = 50/1) on silica gel afforded *R*-**3a** (117.5 mg, 75%, 75% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ= 230 nm, t_R (major) = 8.5 min, t_R (minor) = 15.3 min) as a liquid: $[\alpha]^{20}_D$ = +76.6 ° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H, CHO), 7.24 (d, *J* = 8.7 Hz, 2H, ArH), 6.92 (d, *J* = 8.7 Hz, 2H, ArH), 6.02 (d, *J* = 2.4 Hz, 1H, =CH), 5.94 (d, *J* = 2.4 Hz, 1H, =CH), 3.81 (s, 3H, OCH_3), 2.29-2.13 (m, 1H, one proton of CH_2), 2.13-1.97 (m, 1H, one proton of CH_2), 1.50-1.08 (m, 4H, 2×CH_2), 0.94 (t, *J* = 7.4 Hz,

3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.8, 159.2, 134.3, 129.3, 128.6, 121.1, 114.2, 65.0, 55.2, 31.4, 26.9, 23.1, 13.9; IR (neat) *v* (cm^{-1}) 2957, 2933, 2871, 2837, 2719, 1728, 1607, 1580, 1511, 1464, 1442, 1417, 1380, 1298, 1254, 1184, 1094, 1037; MS (70 eV, EI) *m/z* (%): 312 (M^+(^{81}Br), 3.45), 310 (M^+(^{79}Br), 3.31), 160 (100); HRMS calcd for $C_{15}H_{19}O_2{}^{79}Br$ (M^+): 310.0568. Found: 310.0565.

The following compounds (*R*-**3b**~**3n**, *S*-**3o** and *R*-**3p~3q**) were prepared according to **Typical Procedure II**.

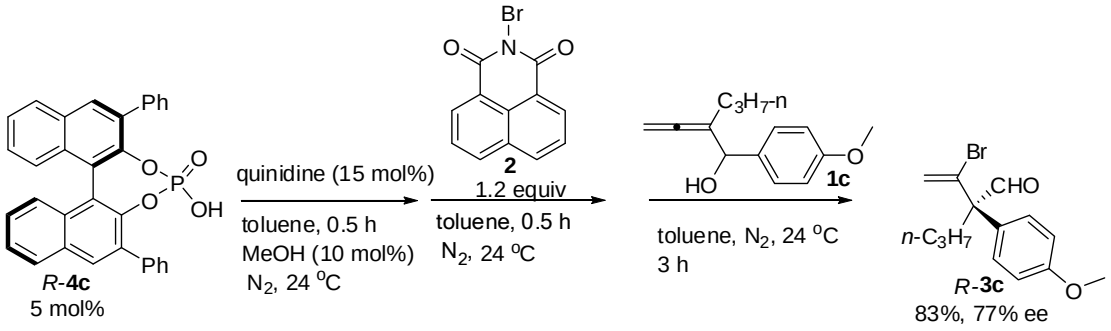
2. Synthesis of (+)-3-bromo-2-ethyl-2-(4-methoxyphenyl)-3-butenal *R*-**3b** (gbj-8-182)



The reaction of quinidine (24.5 mg, 0.075 mmol), *R*-**4c** (12.4 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (165.5 mg, 0.6 mmol), and allenol **1b** (101.1 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 2.5 h afforded *R*-**3b** (107.9 mg, 77%, 77% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ= 230 nm, t_R (major) = 22.7 min, t_R (minor) = 19.1 min) as a liquid: $[\alpha]^{20}_D$ = +94.5 ° (*c* = 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H, CHO), 7.25 (d, *J* = 9.0 Hz, 2H, ArH), 6.93 (d, *J* = 8.7 Hz, 2H, ArH), 6.02 (d, *J* = 2.7 Hz, 1H, =CH), 5.96 (d, *J* = 2.7 Hz, 1H, =CH), 3.81 (s, 3H, OCH_3), 2.42-2.22 (m, 1H, one proton of CH_2), 2.20-2.02 (m, 1H, one proton of CH_2), 0.93 (t, *J* = 7.4 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.7, 159.2, 133.9, 129.4, 128.4, 121.4, 114.2, 65.5, 55.2, 24.5, 9.2; IR (neat) *v*

(cm^{-1}) 2971, 2936, 2881, 2836, 2723, 1728, 1608, 1580, 1511, 1462, 1442, 1416, 1383, 1299, 1255, 1185, 1153, 1086, 1034; MS (70 eV, EI) *m/z* (%): 284 (M^+(^{81}Br), 2.68), 282 (M^+(^{79}Br), 2.53), 174 (100); HRMS calcd for $C_{13}H_{15}O_2{}^{79}Br$ (M^+): 282.0255. Found: 282.0259.

3. Synthesis of (+)-3-bromo-2-propyl-2-(4-methoxyphenyl)-3-butenal ***R*-3c** (gbj-8-180)

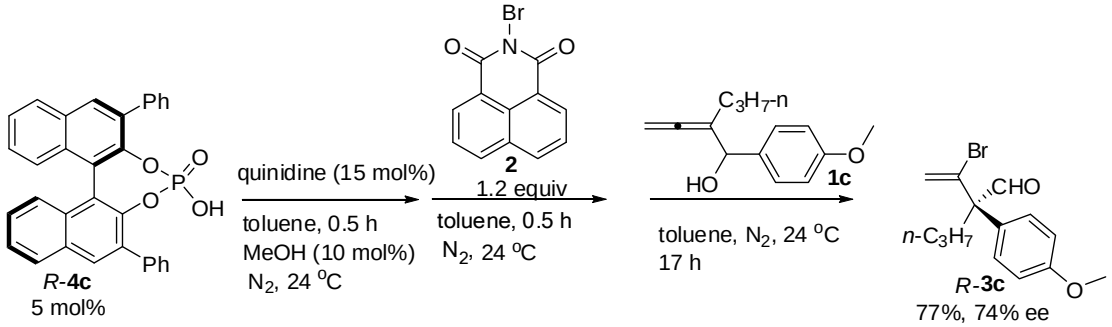


The reaction of quinidine (24.2 mg, 0.075 mmol), *R*-**4c** (12.6 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (165.5 mg, 0.6 mmol), and allenol **1c** (109.5 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 3 h afforded *R*-**3c** (124.4 mg, 83%, 77% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ = 230 nm, t_R (major) = 8.7 min, t_R (minor) = 12.8 min) as a liquid: $[\alpha]^{20}_D$ = +82.9 ° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H, CHO), 7.24 (d, *J* = 9.3 Hz, 2H, ArH), 6.93 (d, *J* = 9.0 Hz, 2H, ArH), 6.01 (d, *J* = 2.4 Hz, 1H, =CH), 5.93 (d, *J* = 2.7 Hz, 1H, =CH), 3.80 (s, 3H, OCH_3), 2.28-2.12 (m, 1H, one proton of CH_2), 2.03 (td, *J* = 12.5 Hz and 5.0 Hz, 1H, one proton of CH_2), 1.42-1.13 (m, 2H, CH_2), 1.00 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.7, 159.2, 134.2, 129.3, 128.5, 121.0, 114.2, 65.1, 55.2, 33.8, 18.2, 14.5; IR (neat) *v* (cm^{-1}) 2994, 2960, 2933, 2873,

2836, 2719, 1726, 1608, 1580, 1511, 1464, 1442, 1417, 1302, 1255, 1184, 1090, 1035; MS (70 eV, ESI) *m/z*: 501 [M(^{81}Br) + Na]$^+$, 499[M(^{79}Br) + Na]$^+$, 479 [M(^{81}Br) + H]$^+$, 477 [M(^{79}Br) + H]$^+$; HRMS calcd for $C_{14}H_{17}O_2{}^{79}Br$ (M^+): 296.0412. Found: 296.0409.

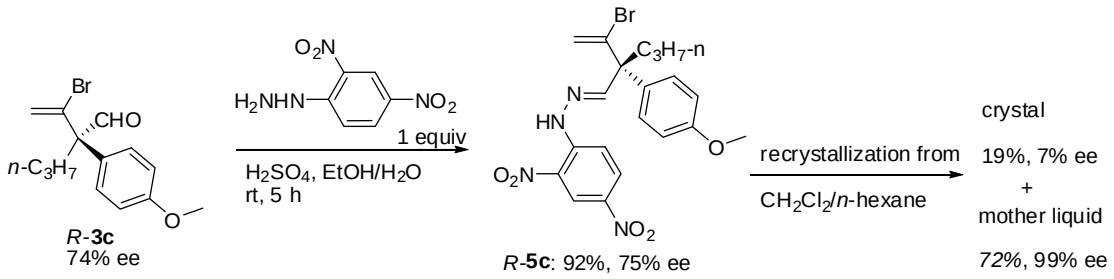
Synthesis of *R*-3c in 98% ee:

(1) A gram scale reaction of *R*-**3c** (gbj-10-17)



The reaction of quinidine (243.3 mg, 0.75 mmol), *R*-**4c** (125.1 mg, 0.25 mmol), MeOH (16.1 mg, 0.5 mmol), **2** (1.6562 g, 6 mmol), and allenol **1c** (1.0908 g, 5 mmol) in 100 mL of toluene at 24 °C for 17 h afforded *R*-**3c** (1.1428 g, 77%, 74% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ= 230 nm, t_R (major) = 7.3 min, t_R (minor) = 9.7 min) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.61 (s, 1H, CHO), 7.24 (d, *J* = 9.0 Hz, 2H, ArH), 6.92 (d, *J* = 9.0 Hz, 2H, ArH), 6.01 (d, *J* = 2.4 Hz, 1H, =CH), 5.93 (d, *J* = 2.7 Hz, 1H, =CH), 3.80 (s, 3H, OCH_3), 2.27-2.12 (m, 1H, one proton of CH_2), 2.04 (td, *J* = 12.6 Hz and 4.8 Hz, 1H, one proton of CH_2), 1.45-1.13 (m, 2H, CH_2), 1.00 (t, *J* = 7.1 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.8, 159.2, 134.3, 129.3, 128.6, 121.0, 114.3, 65.1, 55.2, 34.0, 18.2, 14.5.

(2) Synthesis of hydrazone *R*-**5c** (gbj-10-23)



To a 100 mL round bottomed flask were added 2,4-dinitrophenylhydrazine (198.2 mg, 1 mmol) and concentrated H_2SO_4 (1.5 mL). After dissolution of hydrazine, EtOH (15 mL), H_2O (35 mL), *R*-**3c** (297.0 mg, 1 mmol, 74% ee), and EtOH (5 mL) were added with stirring and the resluting mixture was stirred at rt until the reaction was complete after 5 h as monitored by TLC. This resulting mixture was extracted with ethyl acetate (3×30 mL), washed with a saturated aqueous solution of NaCl and dried over anhydrous Na_2SO_4. Filtration, evaporation and purification by chromatography (petroleum ether/ethyl acetate = 10/1) on silica gel afforded *R*-**5c** (437.8 mg, 92%, 75% ee). After recrystalization from the solution of CH_2Cl_2 and *n*-hexane, the crystal (92.5 mg, 19%, 7% ee) was obtained leaving the mother liquid containing *R*-**5c** (345.2 mg, 72%, 99% ee).

R-**5c**: 75% ee (HPLC conditions: OD-H column, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t_R (major) = 40.6 min, t_R (minor) = 47.6 min); ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.11 (s, 1H, NH), 9.15-9.06 (m, 1H, ArH), 8.33 (dd, *J* = 9.6 Hz and 2.4 Hz, 1H, ArH), 7.86 (d, *J* = 9.6 Hz, 1H, ArH), 7.75 (s, 1H, N=CH), 7.23 (d, *J* = 8.7 Hz, 2H, ArH), 6.92 (d, *J* = 9.0 Hz, 2H, ArH), 5.90 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 5.88 (d, *J* = 2.4 Hz, 1H, one proton of $=CH_2$), 3.82 (s, 3H, OCH_3), 2.27 (t, *J* = 8.3 Hz, 2H, CH_2), 1.50-1.20 (m, 2H, CH_2), 1.03 (t, *J* = 7.4 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.8, 152.3, 145.0, 138.2, 137.5, 132.3, 130.1, 129.1, 128.6,

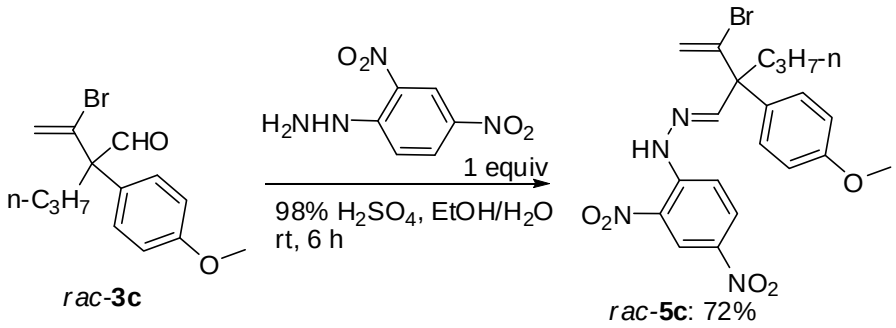
123.3, 120.0, 116.5, 114.0, 57.7, 55.2, 37.3, 18.4, 14.7; IR (neat) v (cm^{-1}) 3298, 3107, 2961, 2932, 2872, 2838, 1614, 1590, 1505, 1462, 1426, 1335, 1284, 1254, 1223, 1184, 1139, 1081, 1035; MS (70 eV, ESI) *m/z* (%): 501 $[M^+(^{81}Br) + Na]^+$, 499 $[M^+(^{79}Br) + Na]^+$, 479 $[M^+(^{81}Br) + H]^+$, 477 $[M^+(^{79}Br) + H]^+$; Elemental analysis calcd for $C_{20}H_{21}BrN_4O_5$: C, 50.33; H, 4.43; N, 11.74. Found: C, 50.40; H, 4.50; N, 11.35.

R-**5c**: 7% ee (HPLC conditions: OD-H column, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t_R (major) = 39.9 min, t_R (minor) = 45.2 min); Solid, mp. 143.8~145.5 ^oC; ^1H NMR (300 MHz, $CDCl_3$) δ 11.11 (s, 1H, NH), 9.09 (d, J = 2.4 Hz, 1H, ArH), 8.32 (dd, J = 9.6 Hz and 2.4 Hz, 1H, ArH), 7.86 (d, J = 9.6 Hz, 1H, ArH), 7.77 (s, 1H, N=CH), 7.24 (d, J = 8.7 Hz, 2H, ArH), 6.91 (d, J = 8.7 Hz, 2H, ArH), 5.90 (d, J = 2.4 Hz, 1H, one proton of $=CH_2$), 5.88 (d, J = 2.4 Hz, 1H, one proton of $=CH_2$), 3.81 (s, 3H, OCH_3), 2.28 (t, J = 8.0 Hz, 2H, CH_2), 1.52-1.13 (m, 2H, CH_2), 1.03 (t, J = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.7, 152.4, 145.0, 138.0, 137.5, 132.3, 130.0, 129.1, 128.5, 123.3, 120.0, 116.4, 113.9, 57.7, 55.2, 37.3, 18.4, 14.6.

R-**5c**: 99% ee (HPLC conditions: OD-H column, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t_R (major) = 40.8 min, t_R (minor) = 48.7 min) $[\alpha]^{20}_D$ = +58.1 ^{o} (*c* = 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 11.10 (s, 1H, NH), 9.10 (d, J = 2.4 Hz, 1H, ArH), 8.33 (dd, J = 9.5 Hz and 2.3 Hz, 1H, ArH), 7.86 (d, J = 9.6 Hz, 1H, ArH), 7.75 (s, 1H, N=CH), 7.23 (d, J = 8.7 Hz, 2H, ArH), 6.92 (d, J = 9.0 Hz, 2H, ArH), 5.90 (d, J = 2.4 Hz, 1H, one proton of $=CH_2$), 5.88 (d, J = 2.7 Hz, 1H, one proton of

=CH_2), 3.82 (s, 3H, OCH_3), 2.27 (t, J = 8.1 Hz, 2H, CH_2), 1.55-1.18 (m, 2H, CH_2), 1.03 (t, J = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.8, 152.3, 145.0, 138.1, 137.5, 132.3, 130.1, 129.1, 128.6, 123.3, 120.0, 116.5, 114.0, 57.7, 55.2, 37.4, 18.4, 14.7.

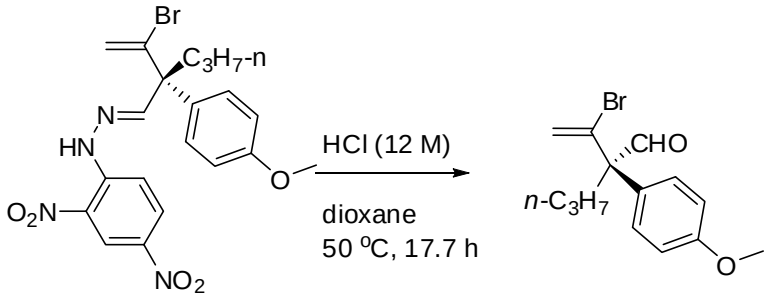
(3) Synthesis of hydrazone *rac*-**5c** (gbj-10-10)



According to the above procedure for the synthesis of *R*-**5c**, the reaction of 2,4-dinitrophenylhydrazine (198.2 mg, 1 mmol), H_2SO_4 (98%, 1.5 mL), EtOH (15 mL + 5 mL), H_2O (35 mL), and *rac*-**3c** (297.5 mg, 1 mmol) at rt for 6 h afforded *rac*-**5c** (344.4 mg, 72%): Solid, mp. 145.6~146.8 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.11 (s, 1H, NH), 9.09 (d, J = 2.4 Hz, 1H, ArH), 8.32 (dd, J = 9.6 Hz and 2.1 Hz, 1H, ArH), 7.86 (d, J = 9.6 Hz, 1H, ArH), 7.76 (s, 1H, N=CH), 7.24 (d, J = 8.4 Hz, 2H, ArH), 6.91 (d, J = 8.4 Hz, 2H, ArH), 5.90 (s, 1H, one proton of =CH_2), 5.88 (s, 1H, one proton of =CH_2), 3.82 (s, 3H, OCH_3), 2.28 (t, J = 8.0 Hz, 2H, CH_2), 1.52-1.18 (m, 2H, CH_2), 1.03 (t, J = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.7, 152.3, 145.0, 138.1, 137.5, 132.3, 130.1, 129.1, 128.6, 123.3, 120.0, 116.4, 114.0, 57.7, 55.2, 37.3, 18.4, 14.7; IR (KBr) v (cm^{-1}) 3308, 3110, 3003, 2957, 2927, 2865, 2838, 1618, 1590, 1511, 1421, 1332, 1249, 1218, 1185, 1134, 1073, 1053, 1029; MS (70 eV, ESI) *m/z*: 501 [M(^{81}Br) + Na]$^{+}$, 499 [M(^{79}Br) + Na]$^{+}$, 479 [M(^{81}Br) + H]$^{+}$, 477 [M(^{79}Br) +

H]$^+$; Elemental analysis calcd for $C_{20}H_{21}BrN_4O_5$: C, 50.33; H, 4.43; N, 11.74. Found: C, 50.56; H, 4.53; N, 11.50.

(4) The hydrolysis of hydrazone *R*-**5c** to form *R*-**3c** (gbj-10-53)



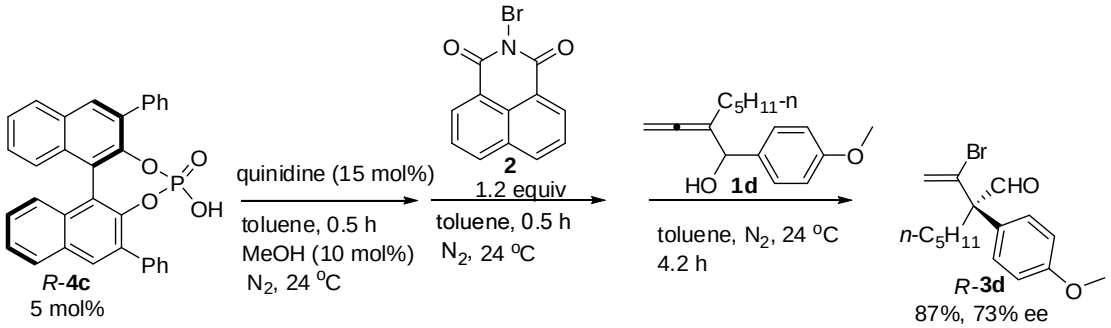
R-**5c**, 99% ee

R-**3c**: 81% yield, 98% ee

To a round bottomed flask were added *R*-**5c** (238.1 mg, 0.5 mmol), 10 mL of dioxane, and 5 mL of HCl (12 M). The resulting mixture was stirred at 50 ^oC until the reaction was complete after 17.7 h as monitored by TLC. After the reaction mixture cooled to rt, a saturated aqueous solution of $NaHCO_3$ was added to quench the reaction. This mixture was extracted with ethyl acetate (3×15 mL), washed with a saturated aqueous solution of NaCl and dried over anhydrous Na_2SO_4. Filtration, evaporation and purification by chromatography (petroleum ether/ethyl acetate = 50/1) on silica gel afforded *R*-**3c** (120.7 mg, 81%, 98% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ= 230 nm, t_R (major) = 7.6 min, t_R (minor) = 10.3 min) as a liquid: $[\alpha]^{20}_D$ = +108.1 ^{o} (*c* = 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H, CHO), 7.24 (d, *J* = 9.0 Hz, 2H, ArH), 6.92 (d, *J* = 9.0 Hz, 2H, ArH), 6.01 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 5.93 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 3.80 (s, 3H, OCH_3), 2.27-2.12 (m, 1H, one proton of CH_2), 2.04 (td, *J* = 12.6 Hz and 4.8 Hz, 1H, one proton of CH_2), 1.45-1.14 (m, 2H, CH_2), 1.00 (t, *J* = 7.2 Hz,

3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.8, 159.2, 134.3, 129.3, 128.6, 121.0, 114.2, 65.1, 55.2, 33.9, 18.2, 14.4.

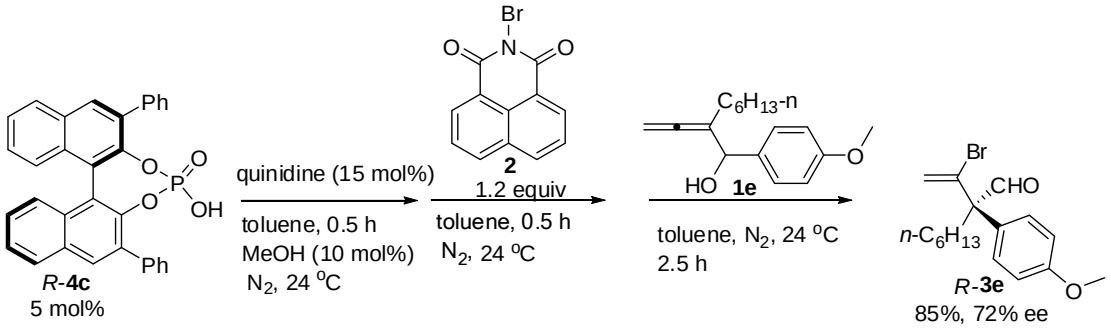
4. Synthesis of (+)-3-bromo-2-pentyl-2-(4-methoxyphenyl)-3-butenal *R*-**3d** (Gbj-8-177)



The reaction of quinidine (24.4 mg, 0.075 mmol), *R*-**4c** (12.5 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (165.3 mg, 0.6 mmol), and allenol **1d** (123.2 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 4.2 h afforded *R*-**3d** (142.5 mg, 87%, 73% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ = 230 nm, t_R (major) = 8.8 min, t_R (minor) = 13.7 min) as a liquid: $[\alpha]^{20}_D$ = +72.3 ° (*c* = 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 9.59 (s, 1H, CHO), 7.24 (d, *J* = 8.7 Hz, 2H, ArH), 6.91 (d, *J* = 9.0 Hz, 2H, ArH), 6.01 (d, *J* = 2.4 Hz, 1H, one proton of $=CH_2$), 5.92 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 3.79 (s, 3H, OCH_3), 2.32-2.13 (m, 1H, one proton of CH_2), 2.13-1.97 (m, 1H, one proton of CH_2), 1.48-1.06 (m, 6H, 3× CH_2), 0.90 (t, *J* = 6.3 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.6, 159.1, 134.3, 129.3, 128.4, 121.0, 114.2, 65.0, 55.1, 32.1, 31.5, 24.4, 22.4, 14.0; IR (neat) *v* (cm^{-1}) 3003, 2955, 2931, 2870, 2837, 2719, 1728, 1608, 1580, 1511, 1464, 1442, 1417, 1379, 1299, 1255, 1184, 1096, 1036; MS (70 eV, EI) *m/z* (%): 326 ($M^+(^{81}Br)$, 4.52), 324 ($M^+(^{79}Br)$, 4.86), 160 (100); HRMS calcd for $C_{16}H_{21}O_2{}^{79}Br$ (M^+): 324.0725.

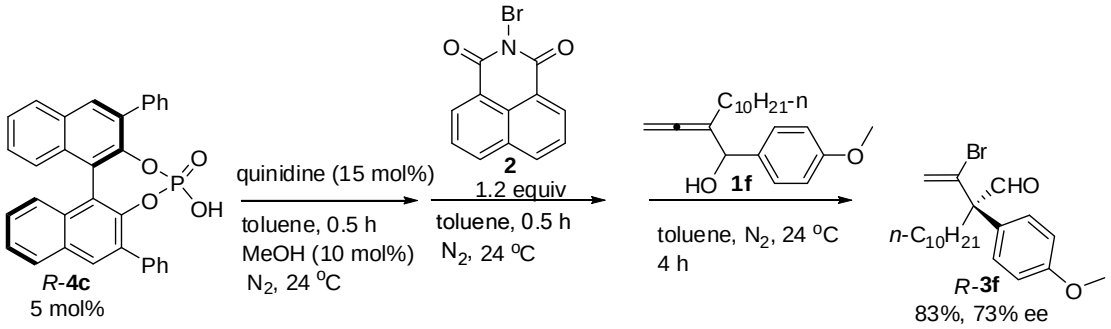
Found: 324.0732.

5. Synthesis of (+)-3-bromo-2-hexyl-2-(4-methoxyphenyl)-3-butenal *R*-**3e** (gbj-8-185)



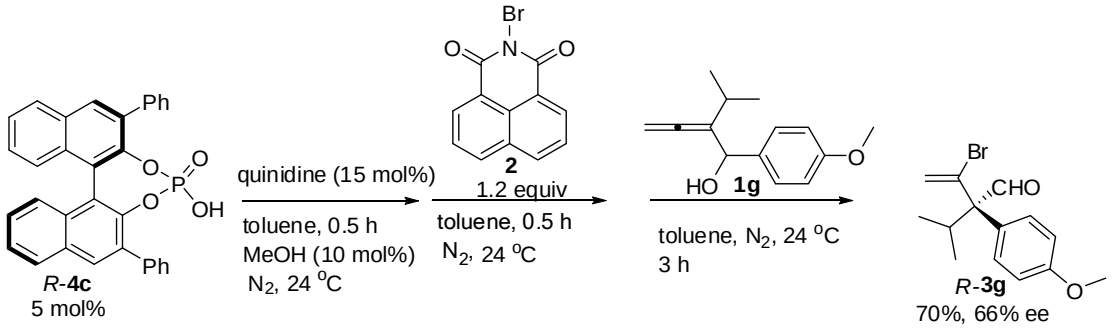
The reaction of quinidine (24.4 mg, 0.075 mmol), *R*-**4c** (12.5 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (165.8 mg, 0.6 mmol), and allenol **1e** (130.8 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 2.5 h afforded *R*-**3e** (145.4 mg, 85%, 72% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ = 230 nm, t_R (major) = 6.4 min, t_R (minor) = 8.6 min) as a liquid: $[\alpha]^{20}_D$ = +69.6 ° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.59 (s, 1H, CHO), 7.24 (d, *J* = 9.0 Hz, 2H, ArH), 6.92 (d, *J* = 9.0 Hz, 2H, ArH), 6.02 (d, *J* = 2.4 Hz, 1H, one proton of $=CH_2$), 5.93 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 3.79 (s, 3H, OCH_3), 2.31-2.14 (m, 1H, one proton of CH_2), 2.13-1.96 (m, 1H, one proton of CH_2), 1.48-1.10 (m, 8H, 4× CH_2), 0.89 (t, *J* = 6.5 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.7, 159.1, 134.3, 129.3, 128.5, 121.0, 114.2, 65.0, 55.1, 31.6, 31.5, 29.6, 24.7, 22.6, 14.0; IR (neat) *v* (cm^{-1}) 3000, 2954, 2930, 2856, 2719, 1727, 1607, 1580, 1511, 1464, 1442, 1417, 1378, 1299, 1255, 1184, 1144, 1097, 1035; MS (70 eV, EI) *m/z* (%): 340 (M^+(^{81}Br), 4.15), 338 (M^+(^{79}Br), 4.55), 160 (100); HRMS calcd for $C_{17}H_{23}O_2{}^{79}Br$ (M^+): 338.0881. Found: 338.0877.

6. Synthesis of (+)-3-bromo-2-decyl-2-(4-methoxyphenyl)-3-butenal *R*-**3f** (Gbj-8-175)



The reaction of quinidine (24.5 mg, 0.075 mmol), *R*-**4c** (12.4 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (165.7 mg, 0.6 mmol), and allenol **1f** (158.1 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 4 h afforded *R*-**3f** (163.4 mg, 83%, 73% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ = 230 nm, t_R (major) = 5.2 min, t_R (minor) = 8.7 min) as a liquid: $[\alpha]^{20}_D$ = +62.8 ° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H, CHO), 7.24 (d, *J* = 8.7 Hz, 2H, ArH), 6.92 (d, *J* = 9.0 Hz, 2H, ArH), 6.02 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 5.93 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 3.80 (s, 3H, OCH_3), 2.28-2.13 (m, 1H, one proton of CH_2), 2.13-1.96 (m, 1H, one proton of CH_2), 1.46-1.06 (m, 16H, 8× CH_2), 0.88 (t, *J* = 6.8 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.8, 159.2, 134.3, 129.3, 128.6, 121.1, 114.2, 65.1, 55.2, 31.9, 31.7, 30.0, 29.6, 29.4, 29.3, 24.8, 22.7, 14.1; IR (neat) *v* (cm^{-1}) 2997, 2953, 2925, 2853, 2718, 1728, 1608, 1580, 1511, 1464, 1441, 1378, 1299, 1255, 1184, 1142, 1101, 1037; MS (70 eV, EI) *m/z* (%): 396 (M^+(^{81}Br), 2.53), 394 (M^+(^{79}Br), 2.92), 160 (100); HRMS calcd for $C_{21}H_{31}O_2{}^{79}Br$ (M^+): 394.1507. Found: 394.1516.

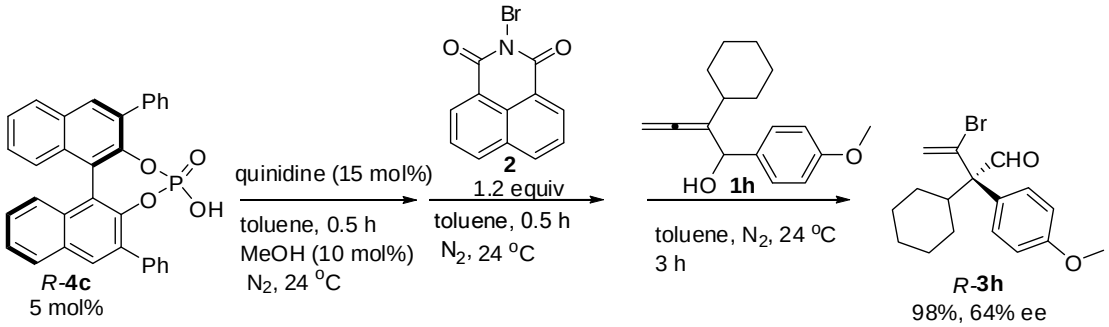
7. Synthesis of (+)-3-bromo-2-isopropyl-2-(4-methoxyphenyl)-3-butenal *R*-**3g** (gbj-9-17)



The reaction of quinidine (24.3 mg, 0.075 mmol), *R*-**4c** (12.6 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (165.9 mg, 0.6 mmol), and allenol **1g** (109.3 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 3 h afforded *R*-**3g** (104.1 mg, 70%, 66% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ = 230 nm, t_R (major) = 25.0 min, t_R (minor) = 17.9 min) as a liquid: $[\alpha]^{20}_D$ = +18.1 ° (*c* = 1.03, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H, CHO), 7.17 (d, *J* = 9.0 Hz, 2H, ArH), 6.93 (d, *J* = 9.0 Hz, 2H, ArH), 6.01 (d, *J* = 2.4 Hz, 1H, one proton of $=CH_2$), 5.98 (d, *J* = 2.4 Hz, 1H, one proton of $=CH_2$), 3.81 (s, 3H, OCH_3), 2.91 (heptet, *J* = 6.8 Hz, 1H, CH), 0.97 (d, *J* = 6.9 Hz, 3H, CH_3), 0.92 (d, *J* = 6.6 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.7, 158.8, 133.0, 130.7, 127.2, 123.4, 113.6, 69.2, 55.1, 30.4, 18.4, 18.2; IR (neat) *v* (cm^{-1}) 2967, 2936, 2877, 2837, 2720, 1727, 1609, 1579, 1512, 1464, 1442, 1417, 1389, 1370, 1296, 1255, 1186, 1124, 1097, 1074, 1036; MS (70 eV, EI) *m/z* (%): 298 (M^+(^{81}Br), 15.75), 296 (M^+(^{79}Br), 14.24), 188 (100); HRMS calcd for $C_{14}H_{17}O_2{}^{79}Br$ (M^+): 296.0412. Found: 296.0413.

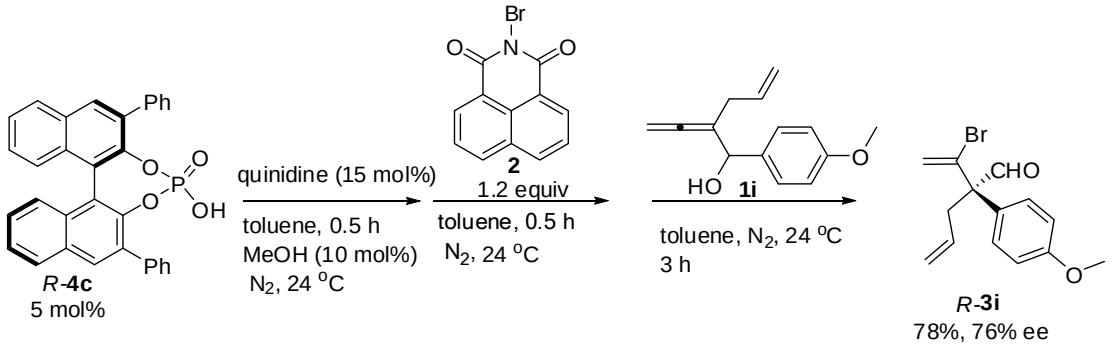
8. Synthesis of (+)-3-bromo-2-cyclohexyl-2-(4-methoxyphenyl)-3-butenal *R*-**3h**

(gbj-9-15)



The reaction of quinidine (24.5 mg, 0.075 mmol), *R*-**4c** (12.6 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (166.1 mg, 0.6 mmol), and allenol **1h** (129.5 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 3 h afforded *R*-**3h** (166.0 mg, 98%, 64% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 95/5, 0.6 mL/min, λ = 230 nm, t_R (major) = 18.6 min, t_R (minor) = 21.7 min) as a liquid: $[\alpha]^{20}_D$ = +12.2 ° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.57 (s, 1H, CHO), 7.14 (d, *J* = 9.3 Hz, 2H, ArH), 6.92 (d, *J* = 8.7 Hz, 2H, ArH), 6.00 (d, *J* = 2.1 Hz, 1H, one proton of $=CH_2$), 5.96 (d, *J* = 2.4 Hz, 1H, one proton of $=CH_2$), 3.81 (s, 3H, OCH_3), 2.51 (t, *J* = 11.9 Hz, 1H, CH), 1.92-1.52 (m, 5H, 2×CH_2 and one proton of CH_2), 1.48-1.22 (m, 2H, CH_2), 1.14-0.74 (m, 3H, CH_2 and one proton of CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.5, 158.7, 133.0, 130.6, 127.4, 123.3, 113.5, 69.2, 55.1, 41.2, 28.8, 28.6, 26.8, 26.7, 26.3; IR (neat) *v* (cm^{-1}) 3000, 2932, 2853, 2714, 1727, 1609, 1579, 1512, 1462, 1453, 1416, 1296, 1255, 1185, 1154, 1123, 1037; MS (70 eV, EI) *m/z* (%): 338 (M^+(^{81}Br), 11.50), 336 (M^+(^{79}Br), 12.17), 307 (100); HRMS calcd for $C_{17}H_{21}O_2{}^{79}Br$ (M^+): 336.0725. Found: 336.0725.

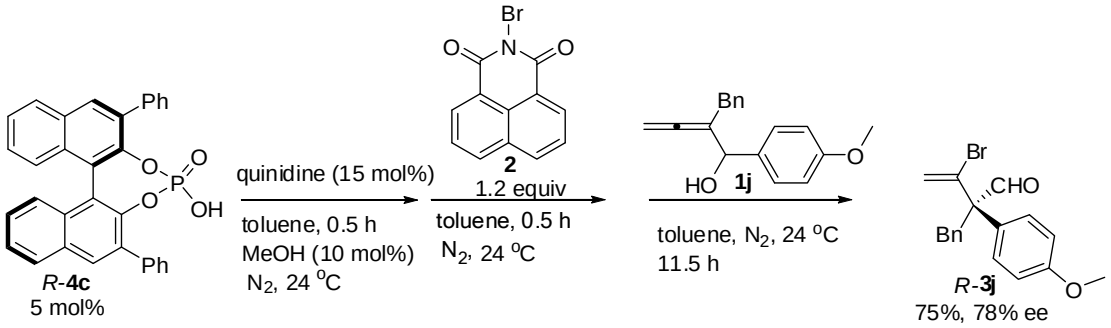
9. Synthesis of (+)-2-allyl-3-bromo-2-(4-methoxyphenyl)-3-butenal *R*-**3i** (gbj-8-199)



The reaction of quinidine (24.4 mg, 0.075 mmol), *R*-**4c** (12.5 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (165.1 mg, 0.6 mmol), and allenol **1i** (107.8 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 3 h afforded *R*-**3i** (114.3 mg, 78%, 76% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 98/2, 1.2 mL/min, λ = 230 nm, t_R (major) = 25.0 min, t_R (minor) = 22.9 min) as a liquid: $[\alpha]^{20}_D$ = +88.5 ° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.64 (s, 1H, CHO), 7.24 (d, *J* = 9.0 Hz, 2H, ArH), 6.93 (d, *J* = 9.0 Hz, 2H, ArH), 5.94 (d, *J* = 2.7 Hz, one proton of $=CH_2$), 5.92 (d, *J* = 3.0 Hz, one proton of $=CH_2$), 5.82-5.60 (m, 1H, =CH), 5.19 (dd, *J* = 17.0 Hz and 1.1 Hz, 1H, one proton of $=CH_2$), 5.11 (d, *J* = 10.2 Hz, 1H, one proton of $=CH_2$), 3.80 (s, 3H, OCH_3), 3.05 (dd, *J* = 14.1 and 6.6 Hz, 1H, one proton of CH_2), 2.86 (dd, *J* = 14.1 and 7.5 Hz, 1H, one proton of CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.7, 159.2, 133.6, 132.7, 129.4, 127.9, 121.5, 118.8, 114.2, 64.7, 55.2, 36.4; IR (neat) *v* (cm^{-1}) 3078, 3036, 3005, 2957, 2935, 2911, 2837, 2720, 1731, 1640, 1608, 1580, 1511, 1463, 1442, 1417, 1299, 1255, 1185, 1098, 1033; MS (70 eV, EI) *m/z* (%): 296 (M^+(^{81}Br), 14.66), 294 (M^+(^{79}Br), 16.82), 145 (100); HRMS calcd for $C_{14}H_{15}O_2{}^{79}Br$ (M^+): 294.0255. Found: 294.0251.

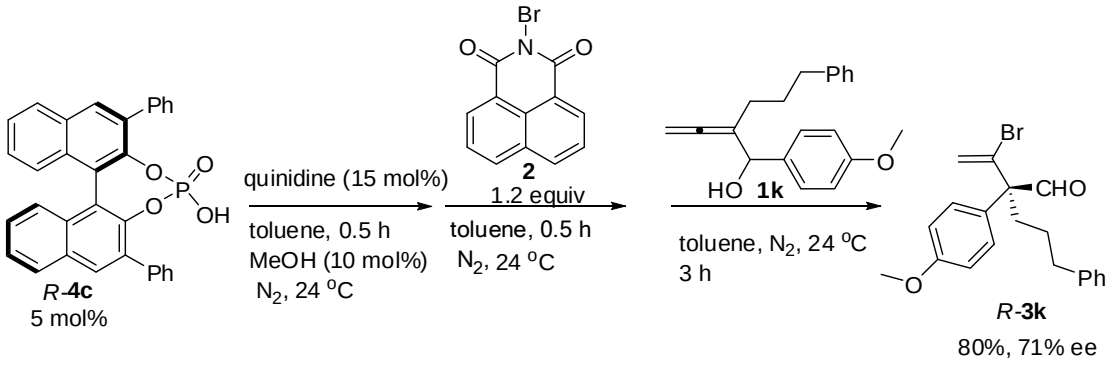
10. Synthesis of (+)-2-benzyl-3-bromo-2-(4-methoxyphenyl)-3-butenal *R*-**3j**

(gbj-10-77)



The reaction of quinidine (24.4 mg, 0.075 mmol), *R*-**4c** (12.5 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (165.4 mg, 0.6 mmol), and allenol **1j** (133.6 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 11.5 h afforded *R*-**3j** (129.1 mg, 75%, 78% ee, HPLC conditions: AD-H column, *n*-hexane/*i*-PrOH = 80/20, 0.5 mL/min, λ = 230 nm, t_R (major) = 12.2 min, t_R (minor) = 10.2 min) as a liquid: $[\alpha]^{20}_D$ = +68.6 ° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.66 (s, 1H, CHO), 7.28-7.04 (m, 7H, ArH), 6.91 (d, *J* = 9.0 Hz, 2H, ArH), 5.82 (s, 2H, $=CH_2$), 3.80 (s, 3H, OCH_3), 3.64 (d, *J* = 13.2 Hz, 1H, one proton of $ArCH_2$), 3.42 (d, *J* = 13.2 Hz, 1H, one proton of $ArCH_2$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.4, 159.3, 136.2, 133.3, 130.7, 129.6, 128.5, 127.9, 126.7, 122.2, 114.2, 66.7, 55.2, 38.3; IR (neat) *v* (cm^{-1}) 3061, 3032, 3003, 2954, 2931, 2838, 2726, 1723, 1620, 1605, 1581, 1510, 1455, 1443, 1417, 1314, 1293, 1255, 1207, 1184, 1123, 1078, 1032; MS (70 eV, EI) *m/z* (%): 346 (M^+(^{81}Br), 12.25), 344 (M^+(^{79}Br), 14.04), 174 (100); HRMS calcd for $C_{18}H_{17}O_2{}^{79}Br$ (M^+): 344.0412. Found: 344.0413.

11. Synthesis of (+)-3-bromo-2-(3-phenylpropyl)-2-(4-methoxyphenyl)-3-butenal *R*-**3k** (gbj-9-6)



The reaction of quinidine (24.3 mg, 0.075 mmol), *R*-**4c** (12.6 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (166.1 mg, 0.6 mmol), and allenol **1k** (147.8 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 3 h afforded *R*-**3k** (150.1 mg, 80%, 72% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ = 230 nm, t_R (major) = 17.4 min, t_R (minor) = 23.2 min; $[\alpha]^{20}_D$ = +77.5 ° (*c* = 1.00, $CHCl_3$)). After recrystlization from the solution of CH_2Cl_2 and *n*-hexane for three times, *R*-**3k** was obtained in 99% ee (31.2 mg, 17%), HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ = 230 nm, t_R (major) = 12.1 min, t_R (minor) = 15.9 min; $[\alpha]^{20}_D$ = +110.2 ° (*c* = 0.83, $CHCl_3$); solid; mp. 84.0~85.1 °C (CH_2Cl_2/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.57 (s, 1H, CHO), 7.38-7.07 (m, 7H, ArH), 6.90 (d, *J* = 8.7 Hz, 2H, ArH), 5.94 (d, *J* = 2.4 Hz, 1H, one proton of =CH_2), 5.90 (d, *J* = 2.4 Hz, 1H, one proton of =CH_2), 3.77 (s, 3H, OCH_3), 2.83-2.57 (m, 2H, CH_2), 2.37-2.18 (m, 1H, one proton of CH_2), 2.17-1.98 (m, 1H, one proton of CH_2), 1.76-1.44 (m, 2H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.5, 159.1, 141.7, 134.0, 129.2, 128.3, 128.23, 128.19, 125.8, 121.2, 114.2, 65.0, 55.1, 36.0, 31.0, 26.5; IR (KBr) *v* (cm^{-1}) 3081, 3061, 3026, 3002, 2953, 2935, 2837, 2721, 1727, 1606, 1581, 1511, 1454, 1417, 1299, 1256, 1185, 1122, 1082, 1034; MS (70 eV, EI) *m/z* (%): 374 (M^+(^{81}Br), 2.37), 372 (M^+(^{79}Br), 2.31), 160 (100); Anal. calcd. for $C_{20}H_{21}O_2Br$: C

64.35, H 5.67; found: C 64.62, H 5.71. The absolute configuration of **3k** was determined by the X-ray diffraction study (Figure 1).

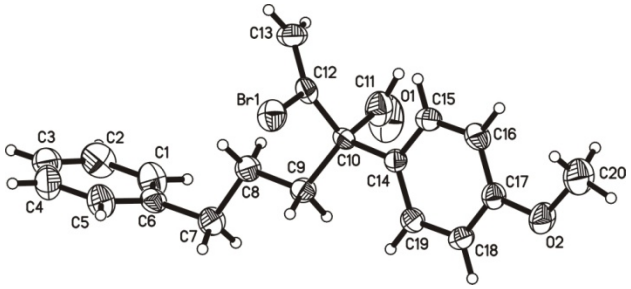
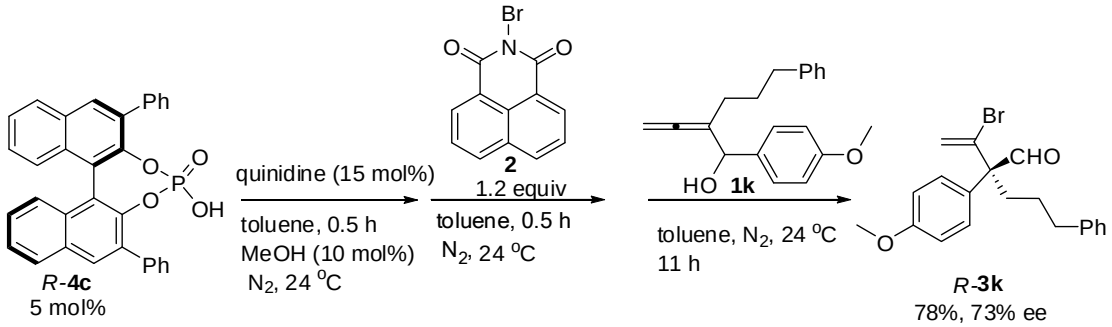


Figure 1. ORTEP Representation of *R*-**3k**

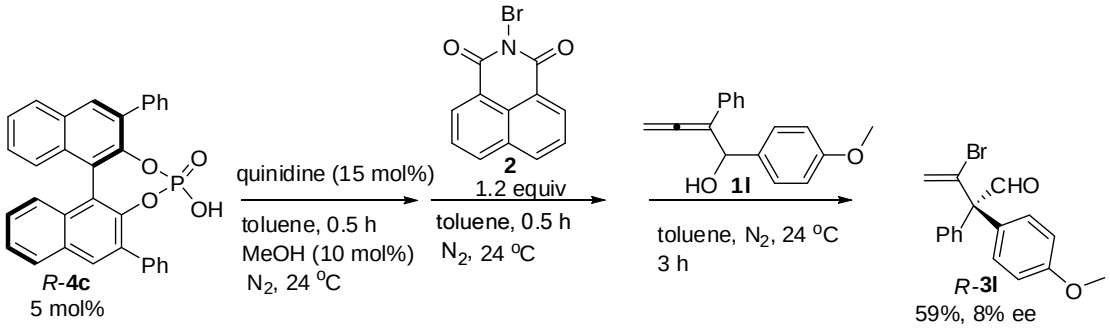
Synthesis of *R*-**3k** on a 3 mmol scale (gbj-11-4)



The reaction of quinidine (145.5 mg, 0.45 mmol), *R*-**4c** (75.1 mg, 0.15 mmol), MeOH (12.5 μL, 0.3 mmol), **2** (993.2 mg, 3.6 mmol), and allenol **1k** (882.5 mg, 3.0 mmol) in 60 mL of toluene at 24 °C for 11 h afforded *R*-**3k** (872.2 mg, 78%, 73% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ= 230 nm, t_R (major) = 13.0 min, t_R (minor) = 16.4 min). Recrystlization from the solution of CH_2Cl_2 and *n*-hexane for twice afforded *R*-**3k** (451.3 mg, 40% in total yield) with 94% ee (HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ = 230 nm, t_R (major) = 13.6 min, t_R (minor) = 17.6 min)); $[\alpha]^{20}_D$ = +105.1 ° (*c* = 1.00, $CHCl_3$); solid; mp. 82.7~83.9 °C (CH_2Cl_2/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.58 (s, 1H, CHO), 7.38-7.23 (m, 2H, ArH), 7.23-7.08 (m, 5H, ArH), 6.89 (d, *J* = 8.7 Hz, 2H, ArH), 5.95 (d, *J* = 2.7 Hz, 1H, one proton of =CH_2), 5.90 (d, *J* = 2.7 Hz, 1H,

one proton of =CH_2), 3.78 (s, 3H, OCH_3), 2.82-2.58 (m, 2H, CH_2), 2.34-2.18 (m, 1H, one proton of CH_2), 2.17-2.02 (m, 1H, one proton of CH_2), 1.74-1.46 (m, 2H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.6, 159.2, 141.7, 134.1, 129.3, 128.41, 128.36, 128.29, 125.8, 121.2, 114.3, 65.1, 55.2, 36.1, 31.3, 26.5.

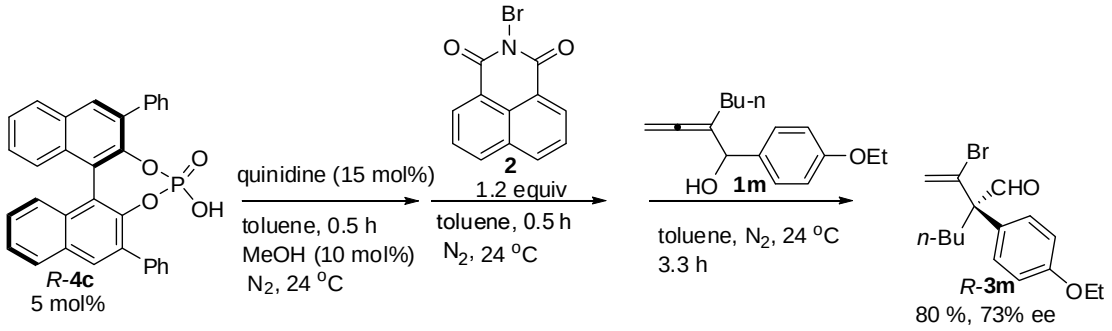
12. Synthesis of (+)-3-bromo-2-phenyl-2-(4-ethoxyphenyl)-3-butenal *R*-**3l** (gbj-11-192)



The reaction of quinidine (9.8 mg, 0.03 mmol), *R*-**4c** (5.0 mg, 0.01 mmol), MeOH (0.8 μL, 0.02 mmol), **2** (66.0 mg, 0.24 mmol), and allenol **1l** (50.0 mg, 0.2 mmol) in 4 mL of toluene at 24 °C for 3 h afforded *R*-**3l** (38.7 mg, 59%, 8% ee, HPLC conditions: AS-H column, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 230 nm, t_R (major) = 6.1 min, t_R (minor) = 5.5 min) as a liquid; $[\alpha]^{20}_D$ = +0.2 ° (*c* = 0.86, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.12 (s, 1H, CHO), 7.45-7.32 (m, 3H, ArH), 7.27-7.19 (m, 2H, ArH), 7.16-7.08 (m, 2H, ArH), 6.95-6.87 (m, 2H, ArH), 6.00 (d, *J* = 2.4 Hz, 1H, one proton of =CH_2), 5.69 (d, *J* = 2.4 Hz, 1H, one proton of =CH_2), 3.82 (s, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.3, 159.1, 137.2, 133.3, 131.4, 130.1, 128.6, 128.5, 128.0, 124.4, 113.9, 70.9, 55.3; IR (neat) *v* (cm^{-1}) 3059, 3033, 3002, 2959, 2930, 2838, 2729, 1732, 1608, 1580, 1505, 1462, 1446, 1417, 1299, 1256, 1185, 1116, 1088, 1035;

MS (70 eV, EI) *m/z* (%): 332 (M^+(^{81}Br), 1.90), 330 (M^+(^{79}Br), 1.89), 303 (M^+(^{81}Br)-CHO, 37.15), 301 (M^+(^{79}Br)-CHO, 36.80), 222 (100); HRMS calcd for $C_{17}H_{15}O_2{}^{79}Br$ (M^+): 330.0255. Found: 330.0261.

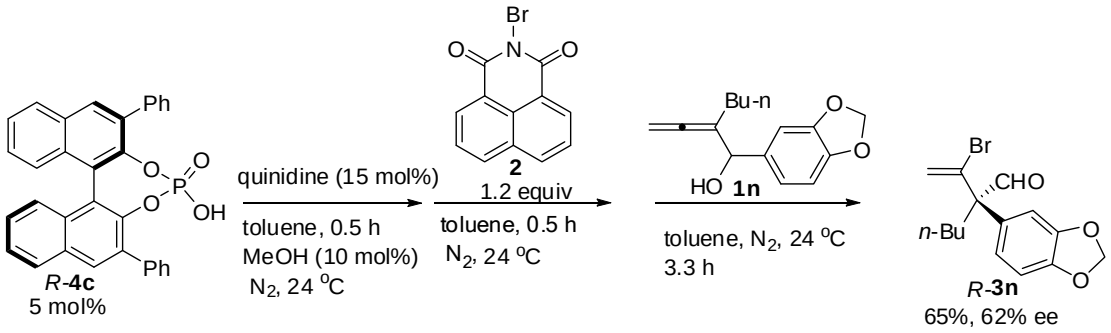
13. Synthesis of (+)-3-bromo-2-butyl-2-(4-ethoxyphenyl)-3-butenal *R*-**3m** (gbj-9-30)



The reaction of quinidine (24.5 mg, 0.075 mmol), *R*-**4c** (12.6 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (166.1 mg, 0.6 mmol), and allenol **1m** (122.3 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 3.3 h afforded *R*-**3m** (129.9 mg, 80%, 73% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ = 230 nm, t_R (major) = 5.2 min, t_R (minor) = 6.0 min) as a liquid; $[\alpha]^{20}_D$ = +79.3 ° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H, CHO), 7.23 (d, *J* = 9.0 Hz, 2H, ArH), 6.91 (d, *J* = 9.0 Hz, 2H, ArH), 6.02 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 5.93 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 4.02 (q, *J* = 7.0 Hz, 2H, CH_2), 2.30-2.13 (m, 1H, one proton of CH_2), 2.13-1.94 (m, 1H, one proton of CH_2), 1.52-1.08 (m, 7H, CH_3 and 2×CH_2), 0.93 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.7, 158.6, 134.3, 129.3, 128.3, 121.0, 114.7, 65.0, 63.4, 31.4, 26.9, 23.1, 14.8, 13.9; IR (neat) *v* (cm^{-1}) 2957, 2931, 2872, 2822, 2719, 1727, 1608, 1580, 1511, 1477, 1393, 1296, 1253, 1185, 1117, 1093, 1047, 1011; MS (70 eV, EI) *m/z* (%): 326 (M^+(^{81}Br),

4.80), 324 (M^+(^{79}Br), 2.90), 174 (100); HRMS calcd for $C_{16}H_{21}O_2{}^{79}Br$ (M^+): 324.0725. Found: 324.0728.

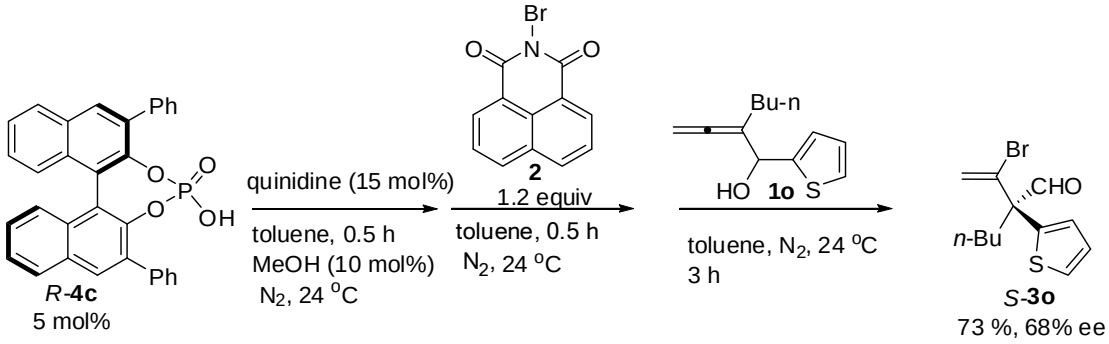
14. Synthesis of (+)-3-bromo-2-butyl-2-(3,4-methylenedioxyphenyl)-3-butenal *R*-**3n** (gbj-8-191)



The reaction of quinidine (24.6 mg, 0.075 mmol), *R*-**4c** (12.6 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (165.8 mg, 0.6 mmol), and allenol **1n** (123.8 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 3.3 h afforded *R*-**3n** (105.6 mg, 65%, 62% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ = 230 nm, t_R (major) = 6.0 min, t_R (minor) = 8.3 min) as a liquid: $[\alpha]^{20}_D$ = +61.6 ° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.59 (s, 1H, CHO), 6.88-6.74 (m, 3H, ArH), 6.04 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 5.99 (s, 2H, OCH_2O), 5.94 (d, *J* = 3.0 Hz, 1H, one proton of $=CH_2$), 2.24-2.10 (m, 1H, one proton of CH_2), 2.10-1.96 (m, 1H one proton of CH_2), 1.48-1.07 (m, 4H, 2×CH_2), 0.93 (t, *J* = 7.4 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.4, 148.2, 147.3, 134.1, 130.4, 121.8, 121.1, 108.42, 108.36, 101.3, 65.1, 31.6, 26.9, 23.0, 13.9; IR (neat) *v* (cm^{-1}) 2957, 2931, 2872, 2774, 2716, 1728, 1618, 1505, 1487, 1438, 1380, 1351, 1243, 1166, 1112, 1094, 1040; MS (70 eV, EI) *m/z* (%): 326 (M^+(^{81}Br), 22.72), 324 (M^+(^{79}Br), 25.66), 115 (100); HRMS

calcd for $C_{15}H_{17}O_3{}^{79}Br$ (M^+): 324.0361. Found: 324.0368.

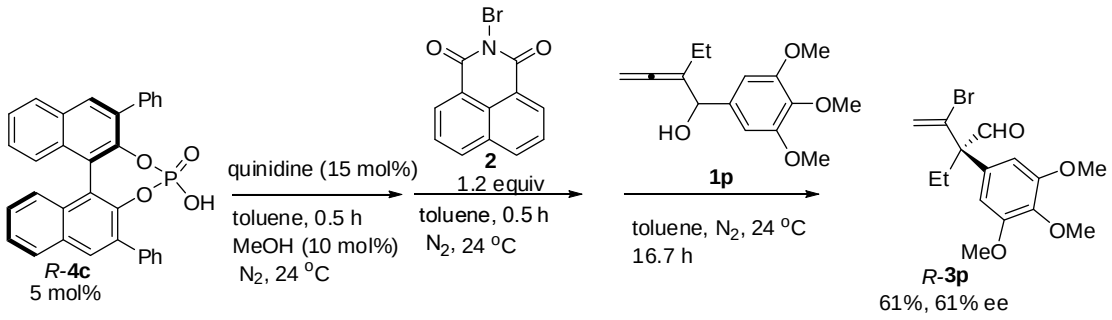
15. Synthesis of (+)-3-bromo-2-butyl-2-thienyl-3-butenal *S*-**3o** (gbj-9-44)



The reaction of quinidine (24.3 mg, 0.075 mmol), *R*-**4c** (12.6 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (166.2 mg, 0.6 mmol), and allenol **1o** (103.4 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 3 h afforded *S*-**3o** (104.1 mg, 73%, 68% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ = 230 nm, t_R (major) = 5.1 min, t_R (minor) = 8.3 min) as a liquid: $[\alpha]^{20}_D$ = +56.6 ° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.58 (s, 1H, CHO), 7.35 (dd, *J* = 4.8 Hz and 1.5 Hz, 1H, ArH), 7.12-6.94 (m, 2H, ArH), 6.03 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 5.93 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 2.32-2.07 (m, 2H, CH_2), 1.50-1.17 (m, 4H, 2×CH_2), 0.93 (t, *J* = 7.1 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 193.7, 140.5, 133.1, 127.2, 127.1, 126.4, 121.1, 63.6, 33.4, 26.6, 22.9, 13.9; IR (neat) *v* (cm^{-1}) 3108, 3069, 2957, 2932, 2871, 2816, 2714, 1732, 1619, 1466, 1429, 1380, 1238, 1156, 1095, 1047; MS (70 eV, EI) *m/z* (%): 288 (M^+(^{81}Br), 1.27), 286 (M^+(^{79}Br), 1.37), 259 (100); HRMS calcd for $C_{12}H_{15}OS^{79}Br$ (M^+): 286.0027. Found: 286.0030.

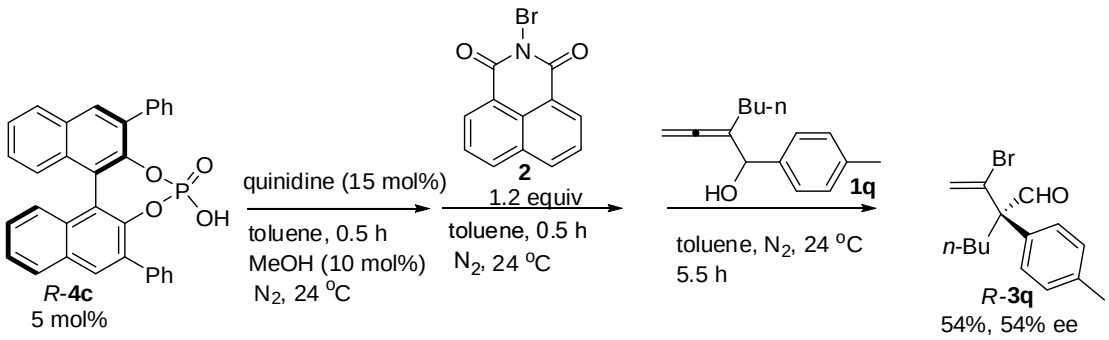
16. Synthesis of (+)-3-bromo-2-butyl-2-(3,4,5-trimethoxylphenyl)-3-butenal *R*-**3p**

(gbj-11-85)



The reaction of quinidine (24.5 mg, 0.075 mmol), *R*-**4c** (12.4 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (165.5mg, 0.6 mmol), and allenol **1p** (132.2 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 16.7 h afforded *R*-**3p** (104.1 mg, 61%, 61% ee, HPLC conditions: OD-H column, *n*-hexane/*i*-PrOH = 95/5, 0.8 mL/min, λ = 230 nm, t_R (major) = 12.2 min, t_R (minor) = 14.9 min) as a liquid: $[\alpha]^{20}_D$ = +42.5 ° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.62 (s, 1 H, CHO), 6.53 (s, 2H, ArH), 6.06 (d, *J* = 2.4 Hz, 1H, one proton of $=CH_2$), 6.01 (d, *J* = 2.7 Hz, 1H, one proton of $=CH_2$), 3.87 (s, 9H, 3×OCH_3), 2.40-2.22 (m, 1H, one proton of CH_2), 2.20-2.01 (m, 1H, one proton of CH_2), 0.94 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.3, 153.3, 137.7, 133.3, 131.9, 121.7, 105.4, 66.0, 60.7, 56.1, 24.6, 9.2; IR (neat) *v* (cm^{-1}) 2974, 2937, 2881, 2835, 2724, 1726, 1619, 1588, 1510, 1461, 1415, 1321, 1247, 1187, 1128, 1007; MS (70 eV, EI) *m/z* (%): 344 (M^+(^{81}Br), 44.67), 342 (M^+(^{79}Br), 48.93), 234 (100); HRMS calcd for $C_{15}H_{19}O_4{}^{79}Br$ (M^+): 342.0467. Found: 324.0473.

17. Synthesis of (+)-3-bromo-2-butyl-2-(4-methylphenyl)-3-butenal *R*-**3q** (gbj-11-199)

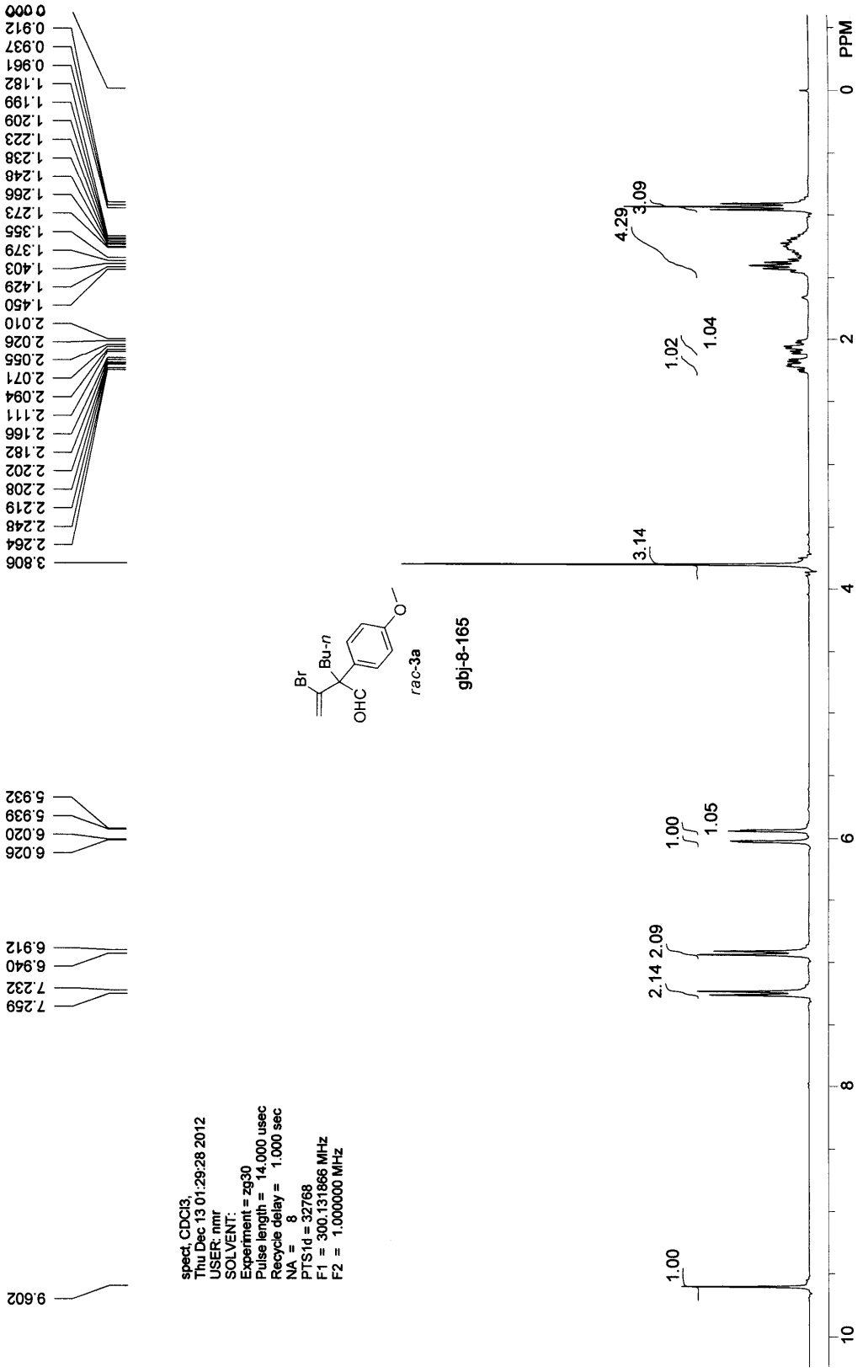


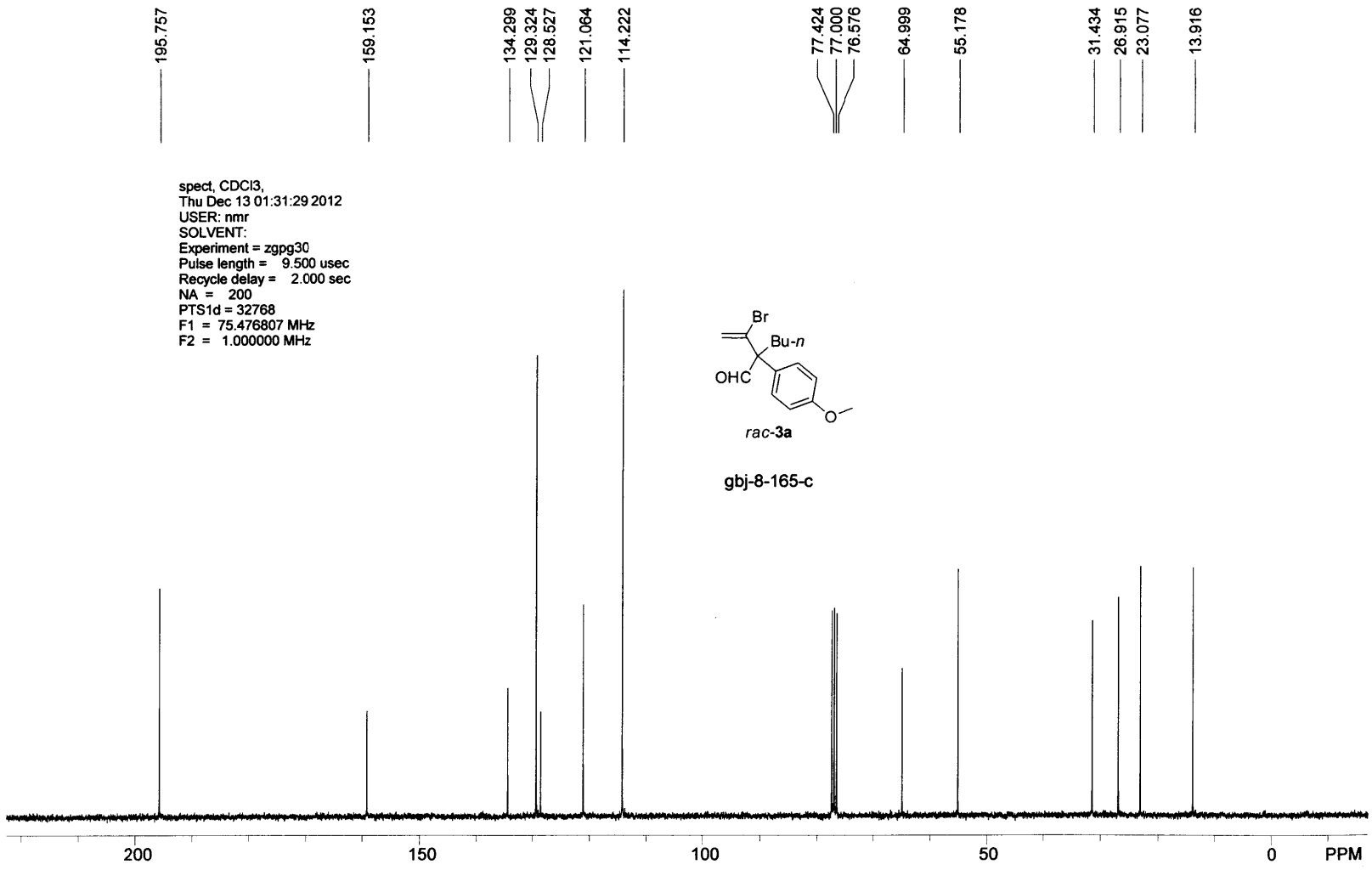
The reaction of quinidine (24.3 mg, 0.075 mmol), *R*-**4c** (12.5 mg, 0.025 mmol), MeOH (2.0 μL, 0.05 mmol), **2** (165.5 mg, 0.6 mmol), and allenol **1q** (108.0 mg, 0.5 mmol) in 10 mL of toluene at 24 °C for 5.5 h afforded *R*-**3q** (79.7 mg, 54%, 54% ee, HPLC conditions: OJ-H column, *n*-hexane/*i*-PrOH = 80/20, 1.2 mL/min, λ = 230 nm, t_R (major) = 4.5 min, t_R (minor) = 5.8 min) as a liquid: $[\alpha]^{20}_D$ = +50.3 ° (*c* = 0.99, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.63 (s, 1H, CHO), 7.21 (s, 4H, ArH), 6.02 (d, *J* = 3.0 Hz, 1H, one proton of $=CH_2$), 5.94 (d, *J* = 2.4 Hz, 1H, one proton of $=CH_2$), 2.35 (s, 3H, CH_3), 2.28-2.15 (m, 1H, one proton of CH_2), 2.14-2.00 (m, 1H one proton of CH_2), 1.49-1.08 (m, 4H, 2×CH_2), 0.93 (t, *J* = 7.2 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.0, 137.8, 134.1, 133.7, 129.6, 128.0, 121.2, 65.4, 31.4, 26.9, 23.1, 21.0, 13.9; IR (neat) *v* (cm^{-1}) 3026, 2957, 2930, 2871, 2718, 1729, 1619, 1511, 1466, 1409, 1380, 1195, 1160, 1093, 1020; MS (70 eV, EI) *m/z* (%): 296 (M^+(^{81}Br), 1.42), 294 (M^+(^{79}Br), 1.65), 143 (100). HRMS calcd for $C_{15}H_{19}O^{79}Br$ (M^+): 294.0619. Found: 294.0622.

Reference:

1) C. Fu, J. Li, S. Ma, *Chem. Commun*. 2005, 4119-4121.

^{1}H and ^{13}C NMR spectra of all these compounds

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129.324
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55.178
31.434
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Thu Dec 13 01:31:29 2012
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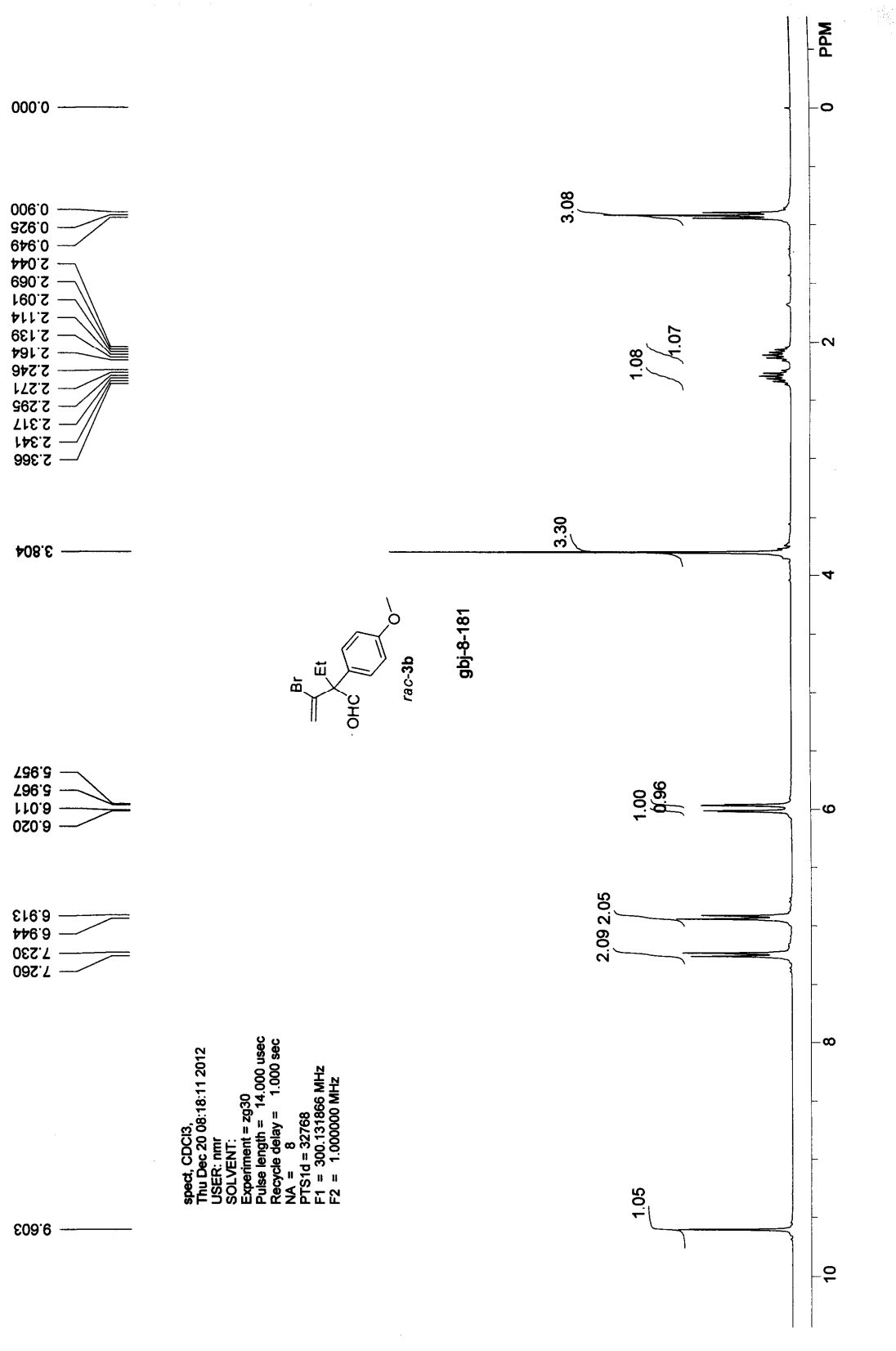
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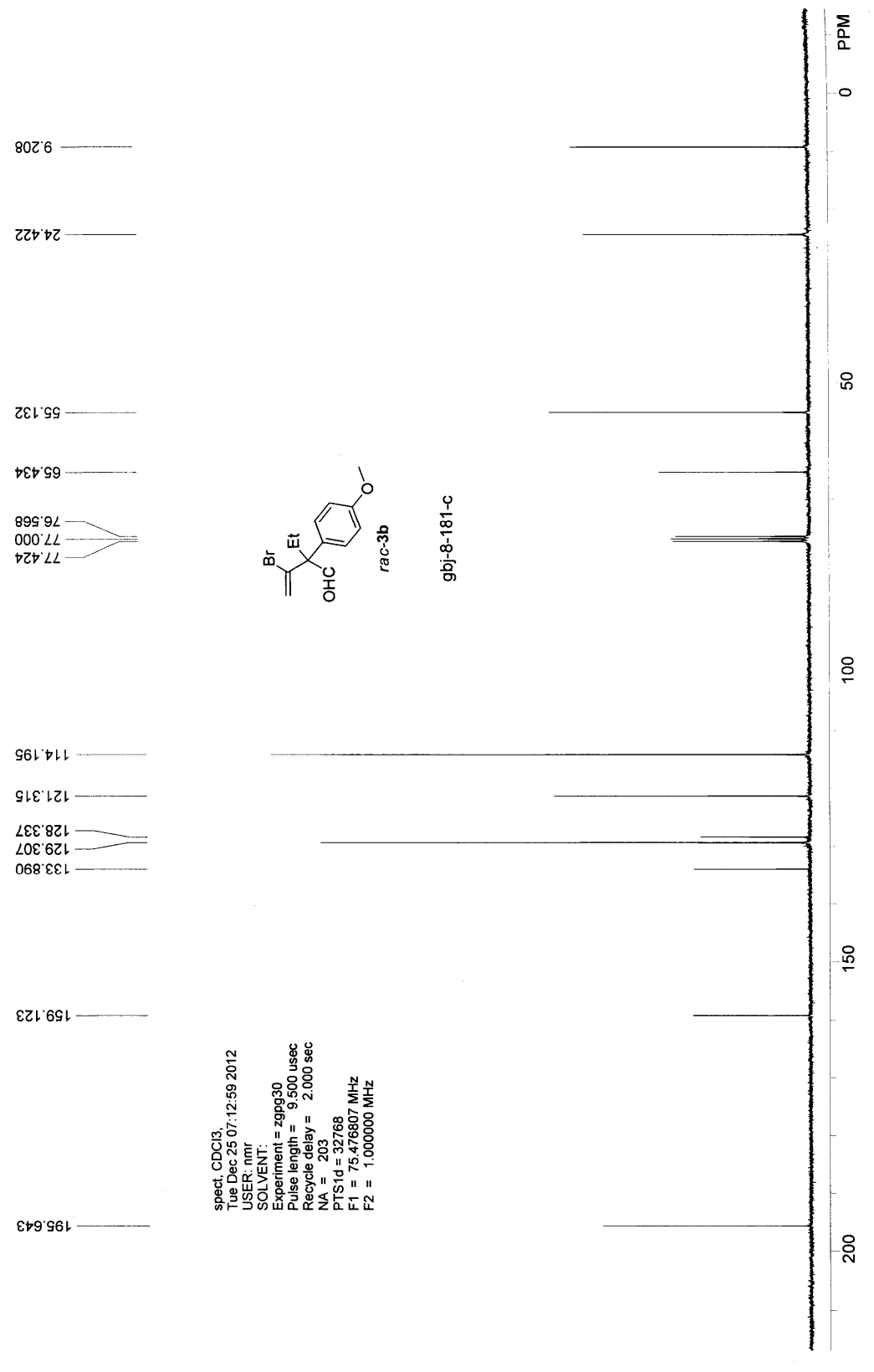
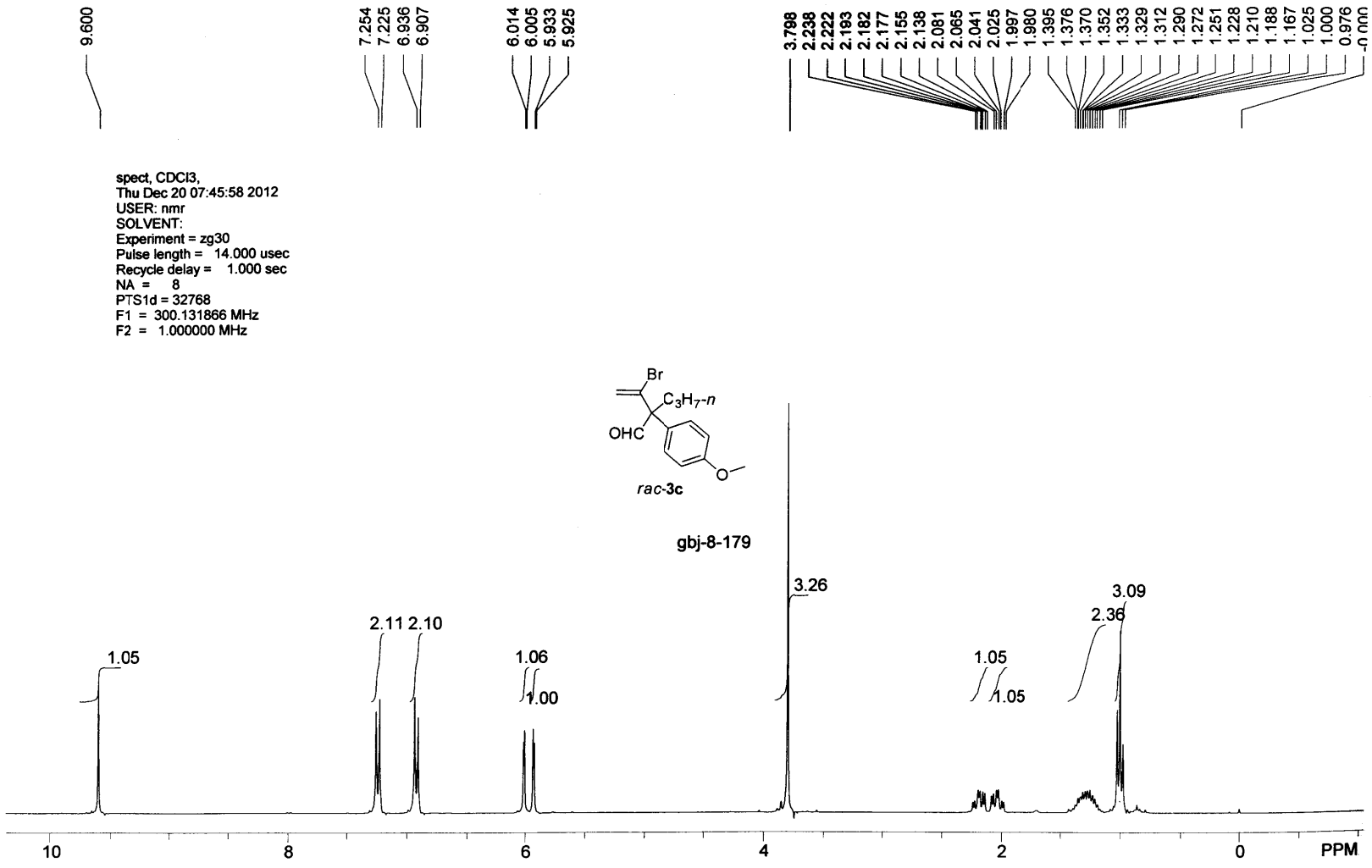
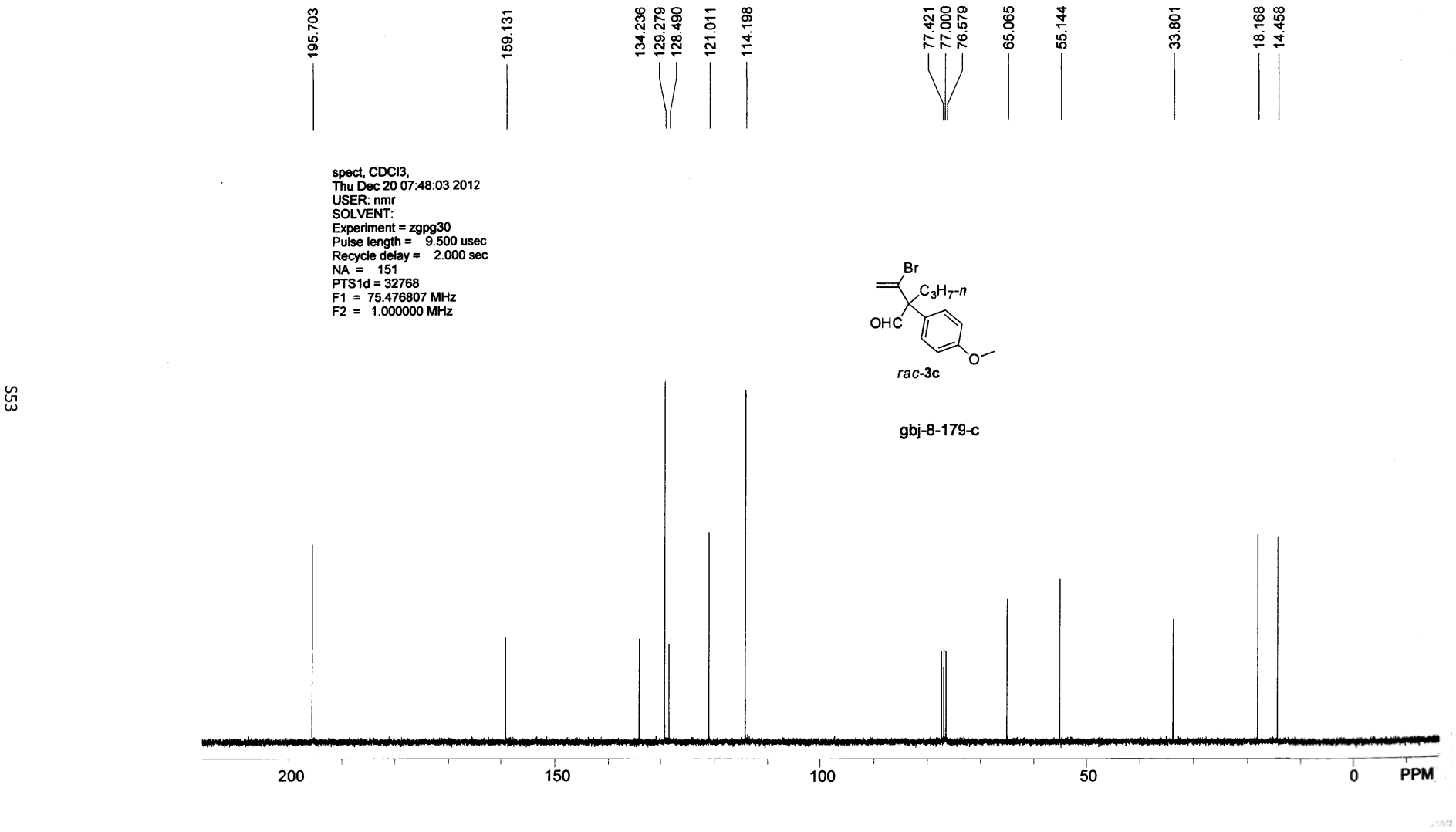
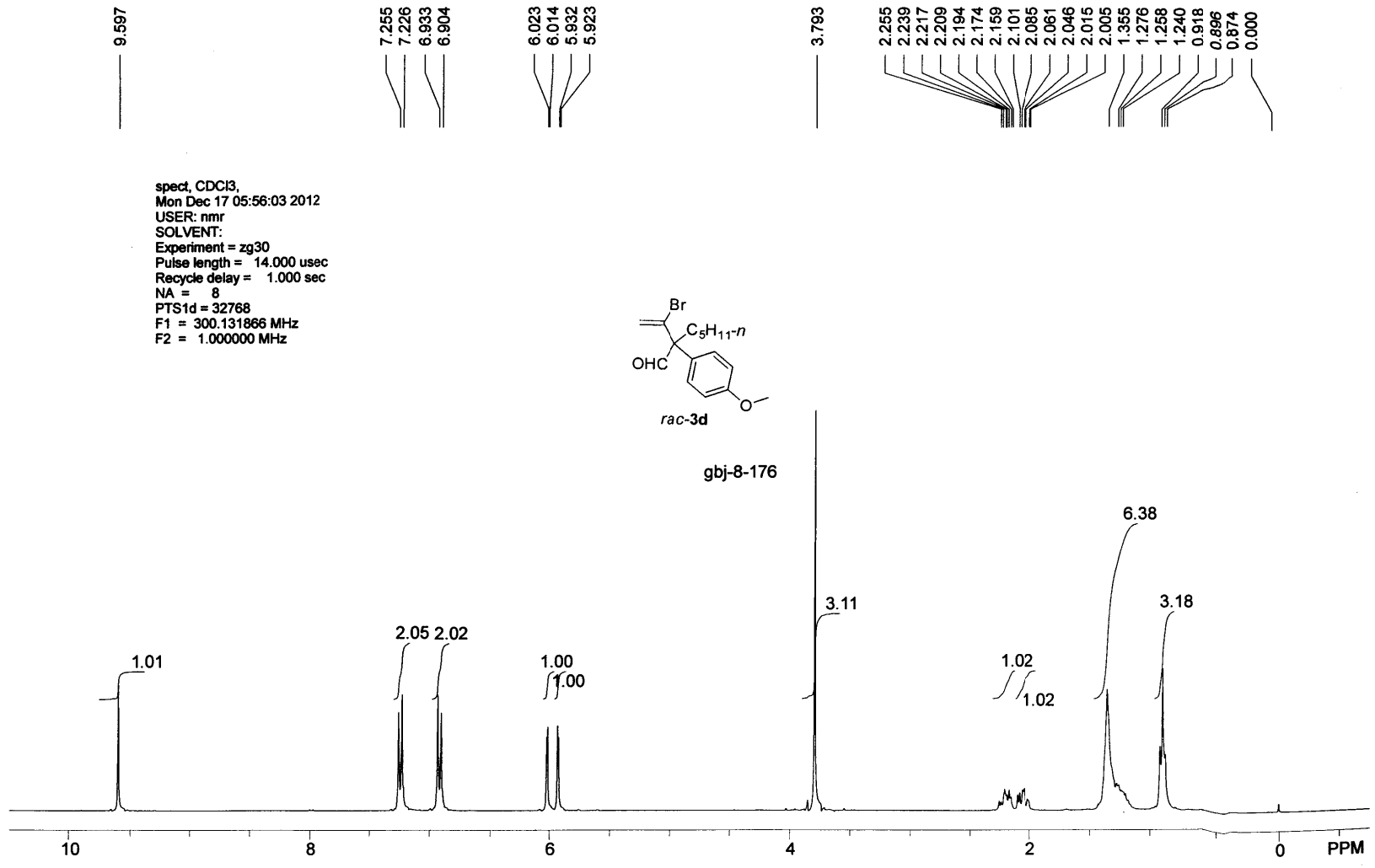
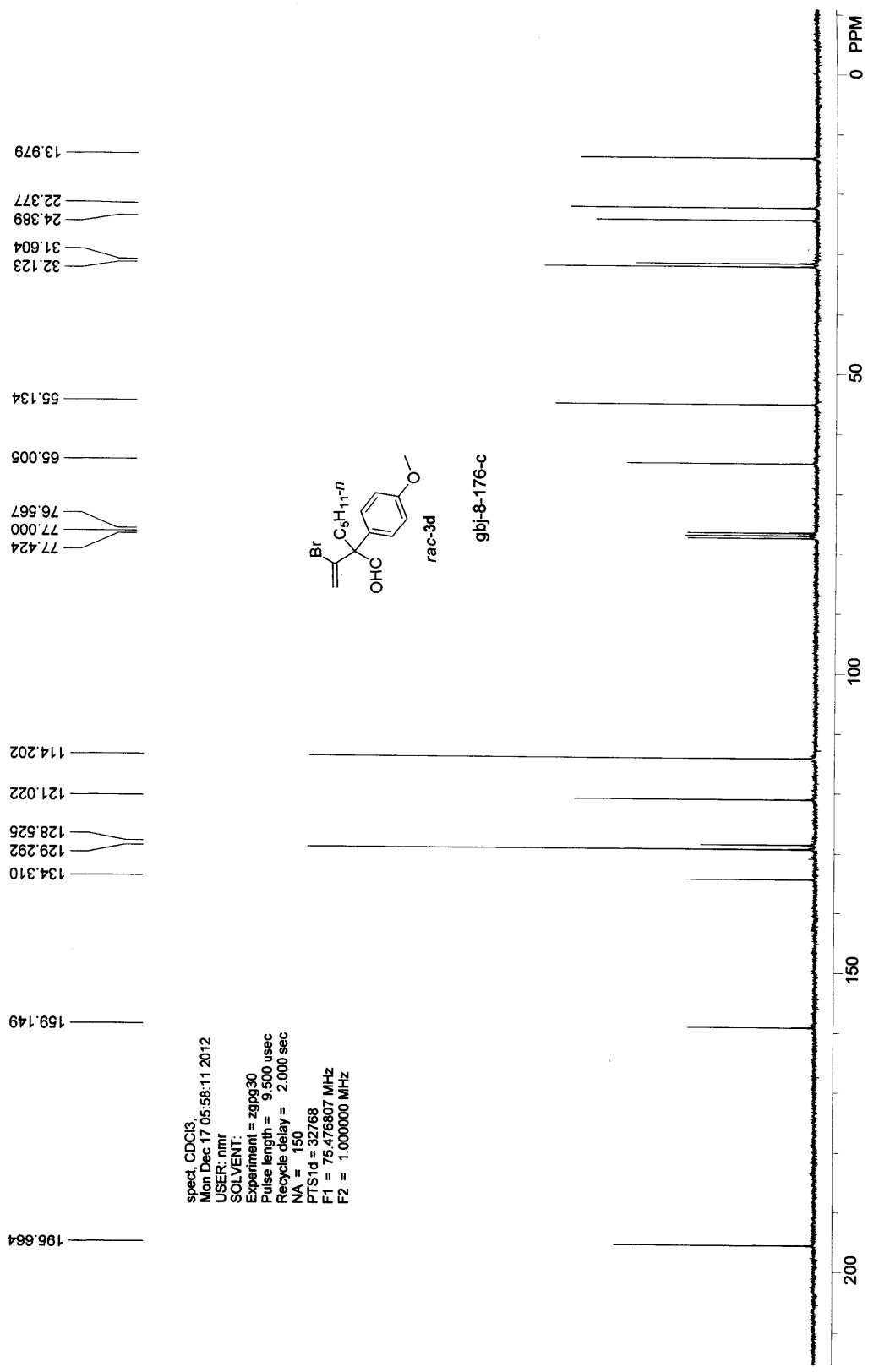
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-8-181
rac-3b
Br
Et
OHC
O
PPM

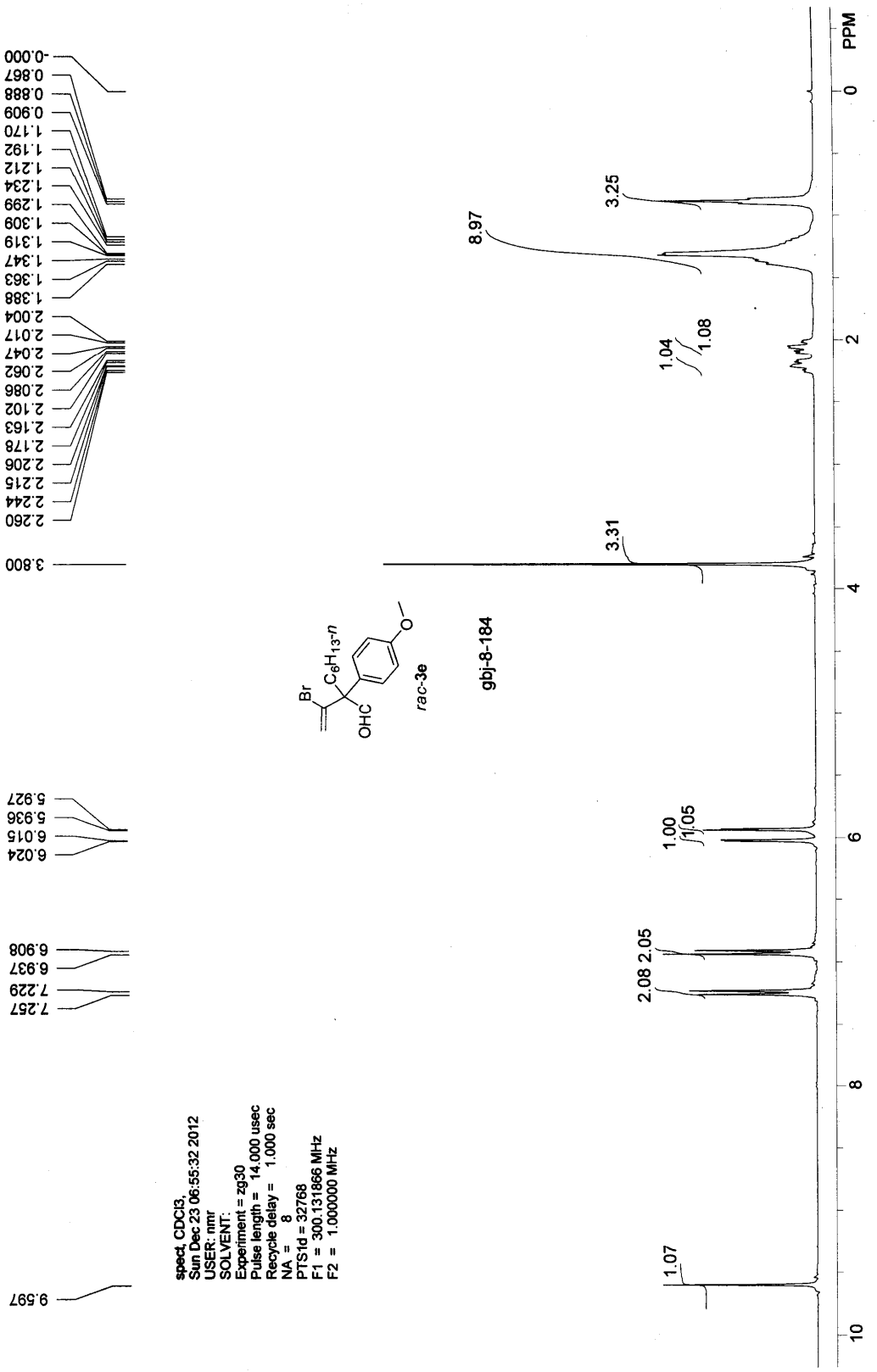

9.208
24.422
55.132
65.434
76.568
77.000
77.424
114.195
121.315
128.337
129.307
133.890
159.123
195.643
spect, CDCl3,
Tue Dec 25 07:12:59 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 203
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
Et
OHC
O
rac-3b
gbj-8-181-c
PPM
0
50
100
150
200

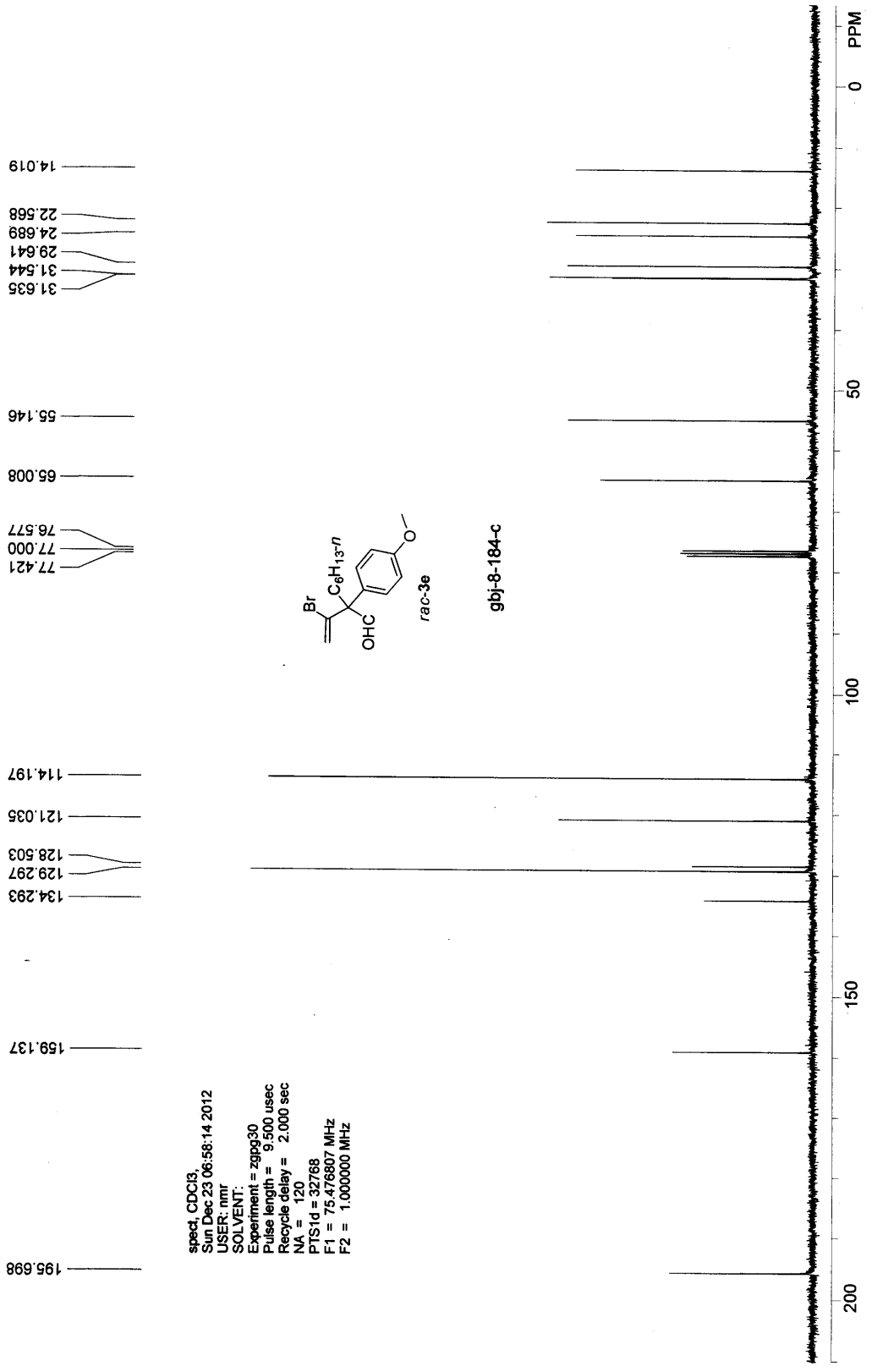

9.600
7.254
7.225
6.936
6.907
6.014
6.005
5.933
5.925
3.798
2.238
2.222
2.193
2.182
2.177
2.155
2.138
2.081
2.065
2.041
2.025
1.997
1.980
1.395
1.376
1.370
1.352
1.333
1.329
1.312
1.290
1.272
1.251
1.228
1.210
1.188
1.167
1.025
1.000
0.976
spect, CDCl3,
Thu Dec 20 07:45:58 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
C3H7-n
OHC
O
rac-3c
gbj-8-179
1.05
2.11
2.10
1.06
1.00
3.26
1.05
1.05
2.36
3.09
10
8
6
4
2
0
PPM

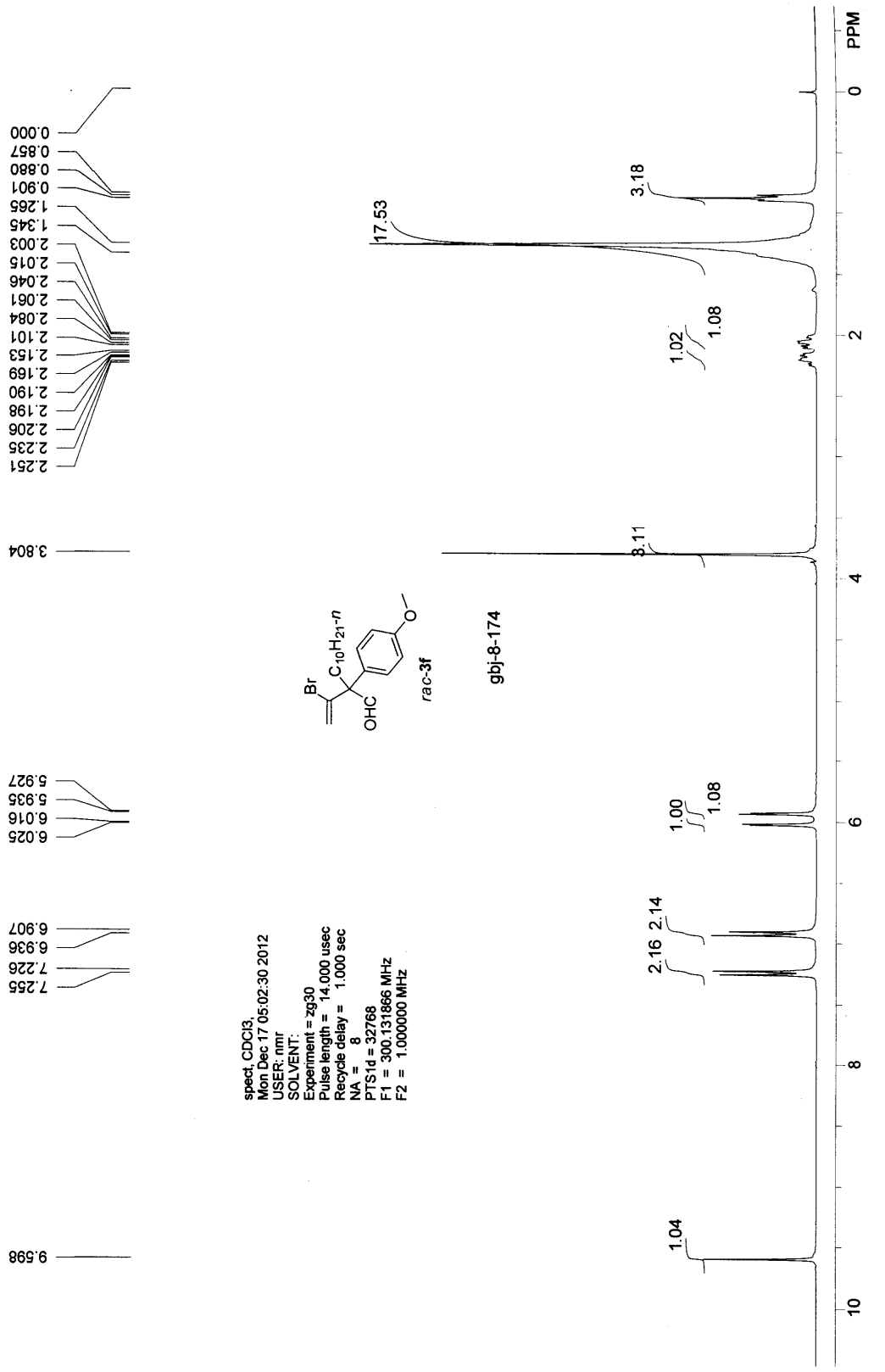

195.703
159.131
134.236
129.279
128.490
121.011
114.198
77.421
77.000
76.579
65.065
55.144
33.801
18.168
14.458
spect, CDCl3,
Thu Dec 20 07:48:03 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 151
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
C3H7-n
OHC
O
rac-3c
gbj-8-179-c
200
150
100
50
0
PPM

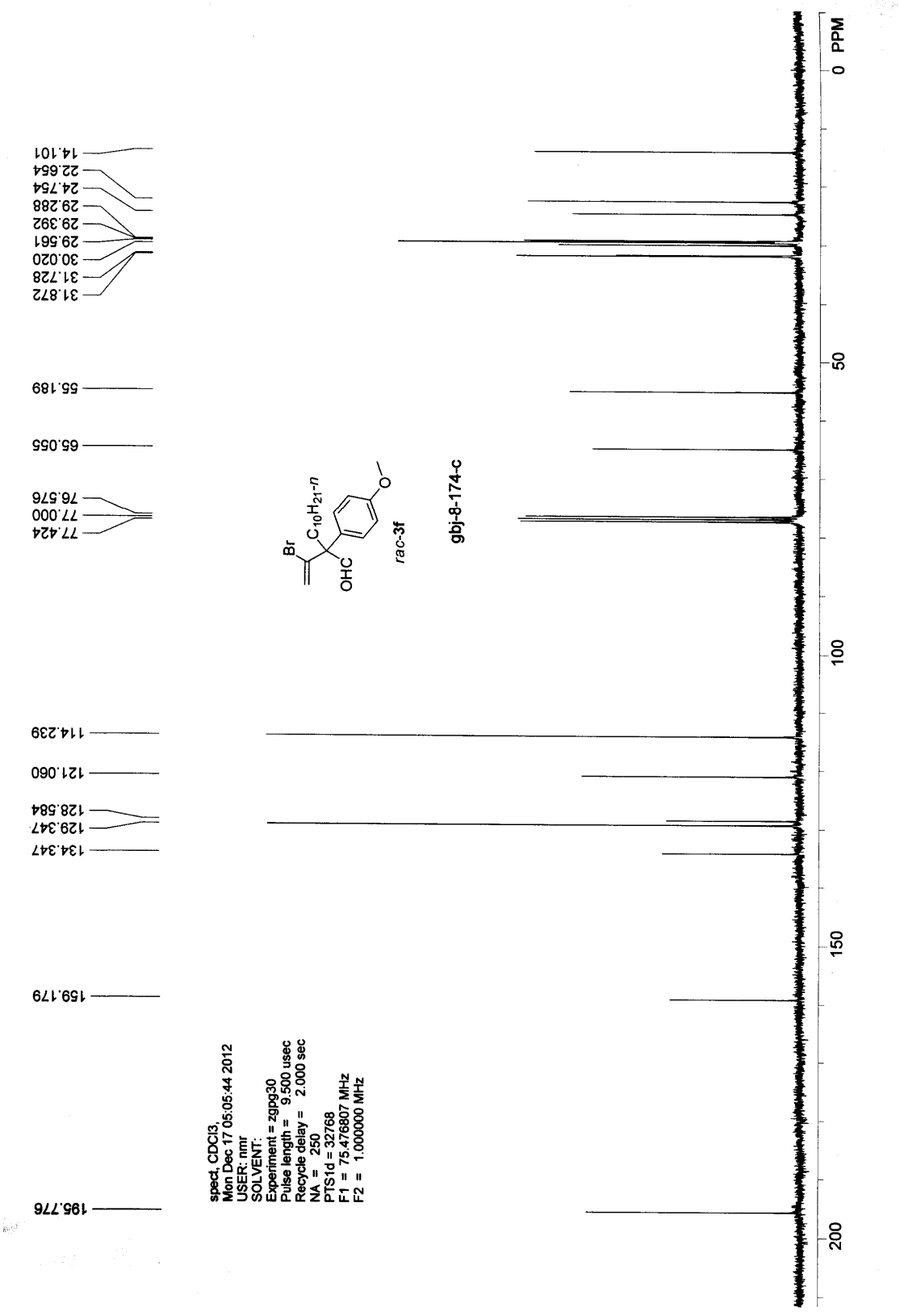

9.597
7.255
7.226
6.933
6.904
6.023
6.014
5.932
5.923
3.793
2.255
2.239
2.217
2.209
2.194
2.174
2.159
2.101
2.085
2.061
2.046
2.015
2.005
1.355
1.276
1.258
1.240
0.918
0.896
0.874
0.000
spect, CDCl3,
Mon Dec 17 05:56:03 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
C5H11-n
OHC
O
rac-3d
gbj-8-176
1.01
2.05
2.02
1.00
1.00
3.11
1.02
1.02
6.38
3.18
10
8
6
4
2
0
PPM

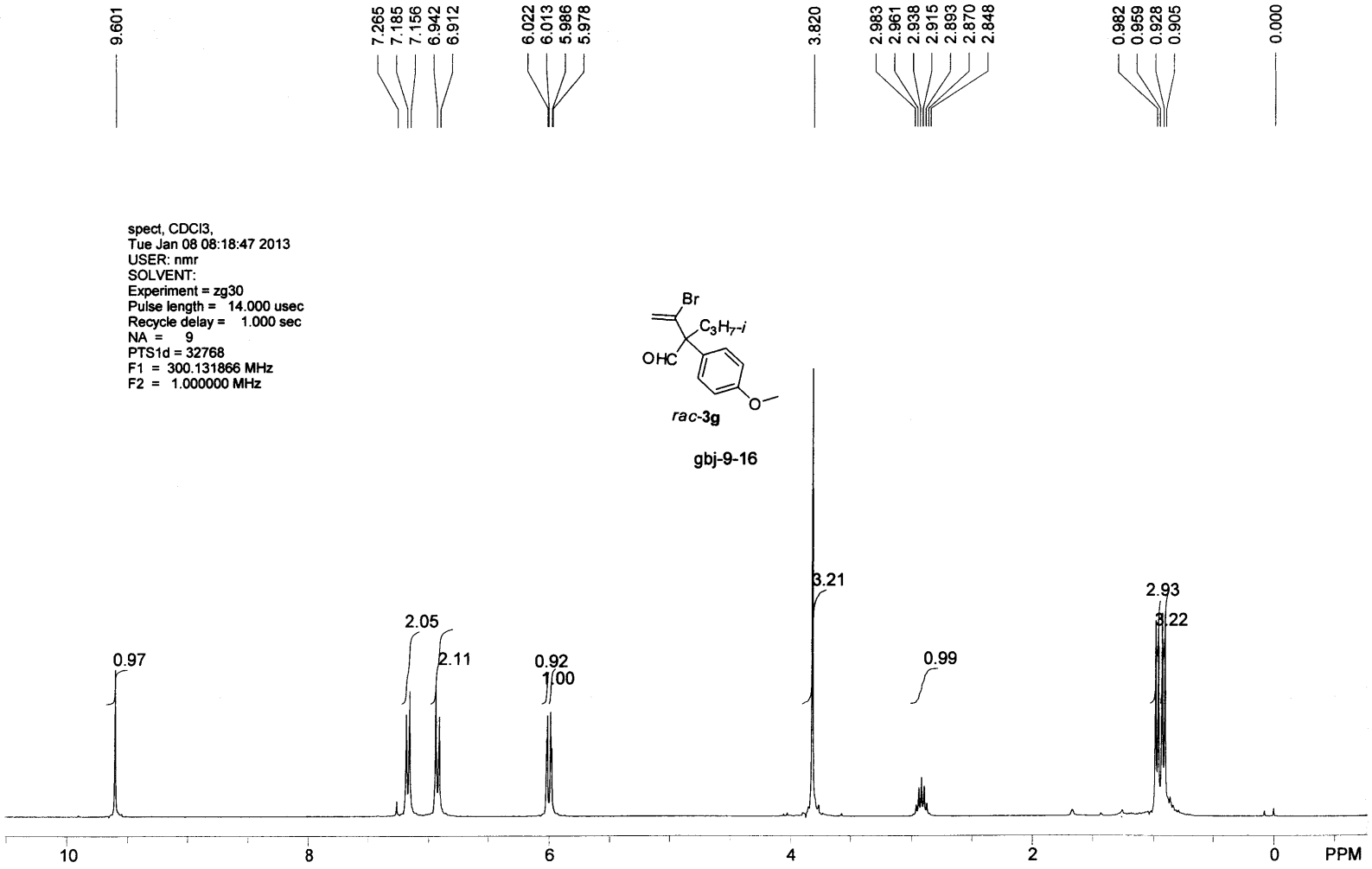

13.979
22.377
24.389
31.604
32.123
55.134
65.005
76.567
77.000
77.424
114.202
121.022
128.525
129.292
134.310
159.149
195.664
spect, CDCl3,
Mon Dec 17 05:58:11 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 150
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
C5H11-n
OHC
O
rac-3d
gbj-8-176-c
0 PPM
50
100
150
200

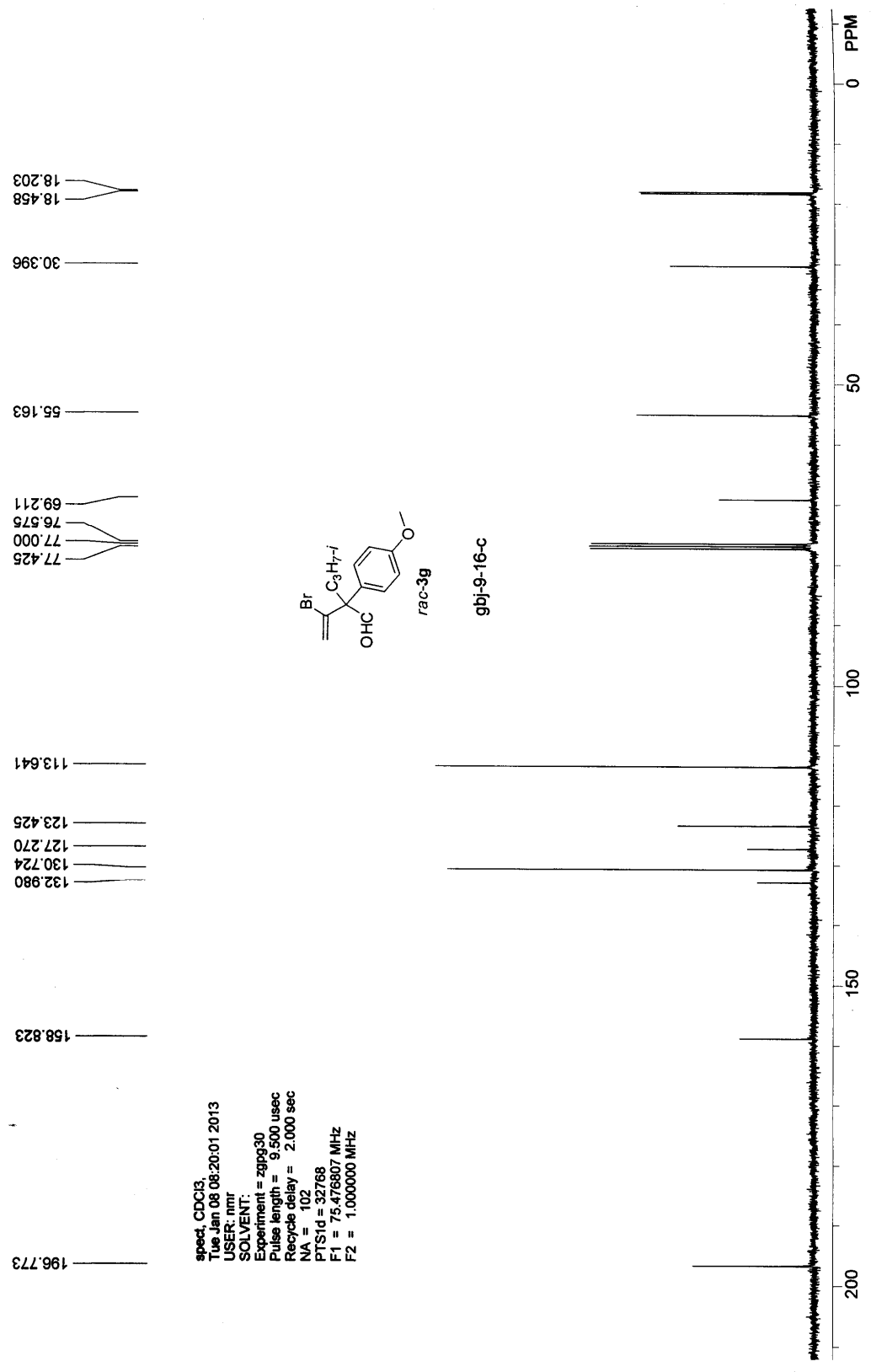

spect, CDCl3,
Sun Dec 23 06:55:32 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-8-184
rac-3e
Br
C6H13-n
OHC
O
PPM

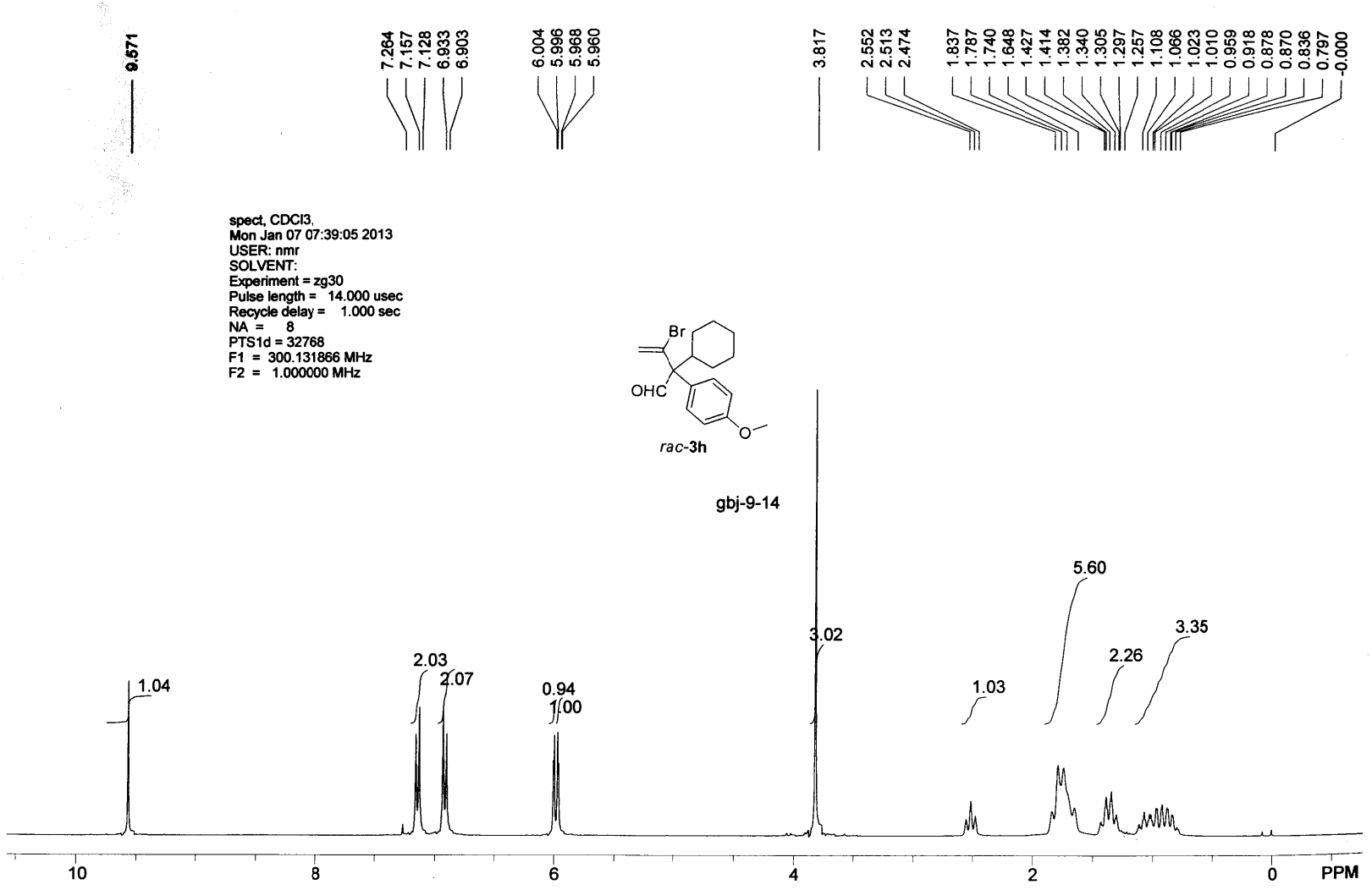

14.019
22.568
24.689
29.641
31.544
31.635
55.146
65.008
76.577
77.000
77.421
114.197
121.035
128.503
129.297
134.293
159.137
195.698
Br
C6H13-n
OHC
O
rac-3e
gbj-8-184-c
spect, CDCl3,
Sun Dec 23 06:58:14 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 120
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
PPM
0
50
100
150
200

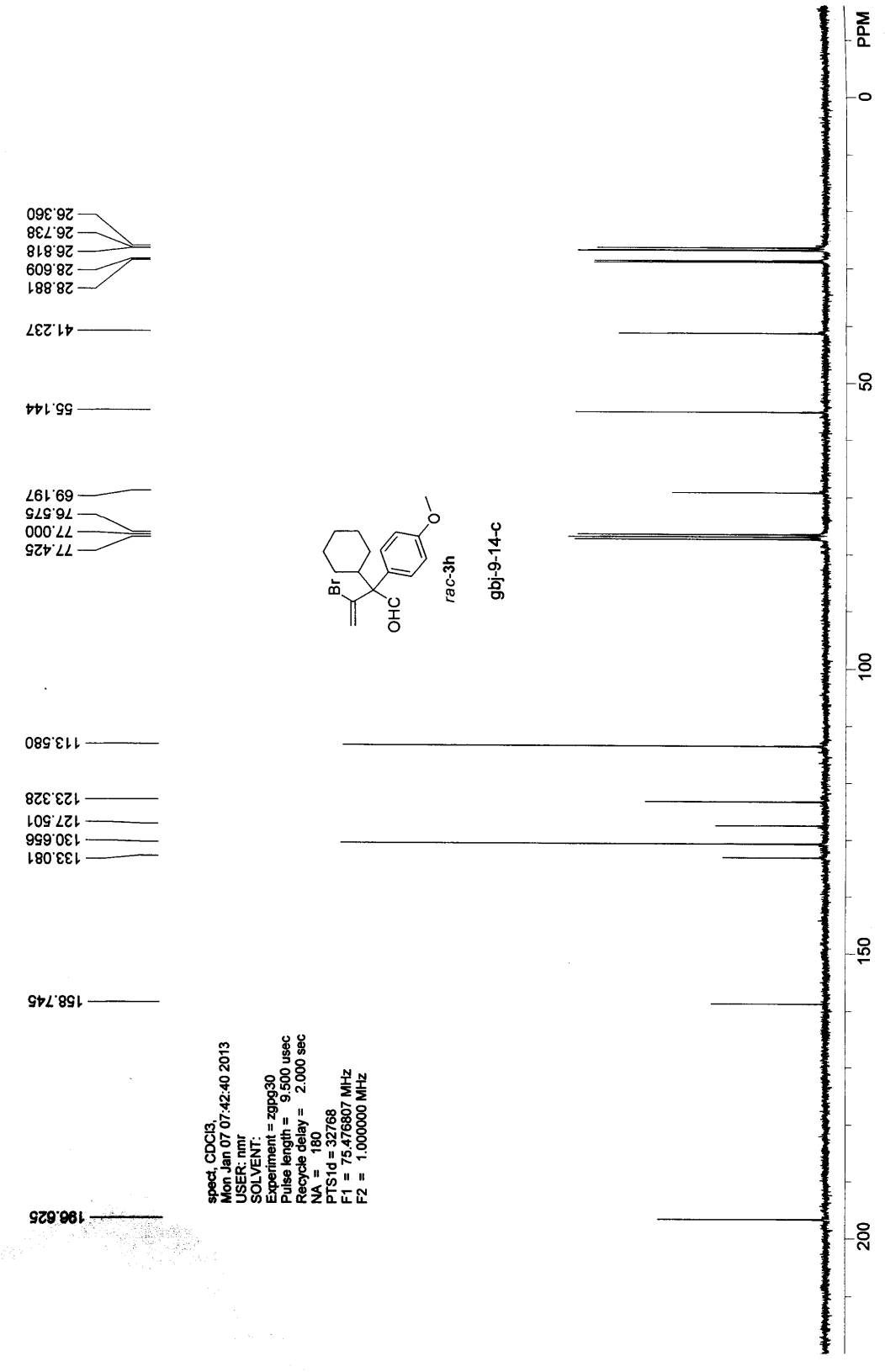

spect, CDCl3,
Mon Dec 17 05:02:30 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-8-174
rac-3f
Br
OHC
C10H21-n
O
9.598
7.255
7.226
6.936
6.907
6.025
6.016
5.935
5.927
3.804
2.251
2.235
2.206
2.198
2.190
2.169
2.153
2.101
2.084
2.061
2.046
2.015
2.003
1.345
1.265
0.901
0.880
0.857
0.000
1.04
2.16
2.14
1.00
1.08
3.11
17.53
3.18
1.02
1.08
10
8
6
4
2
0
PPM

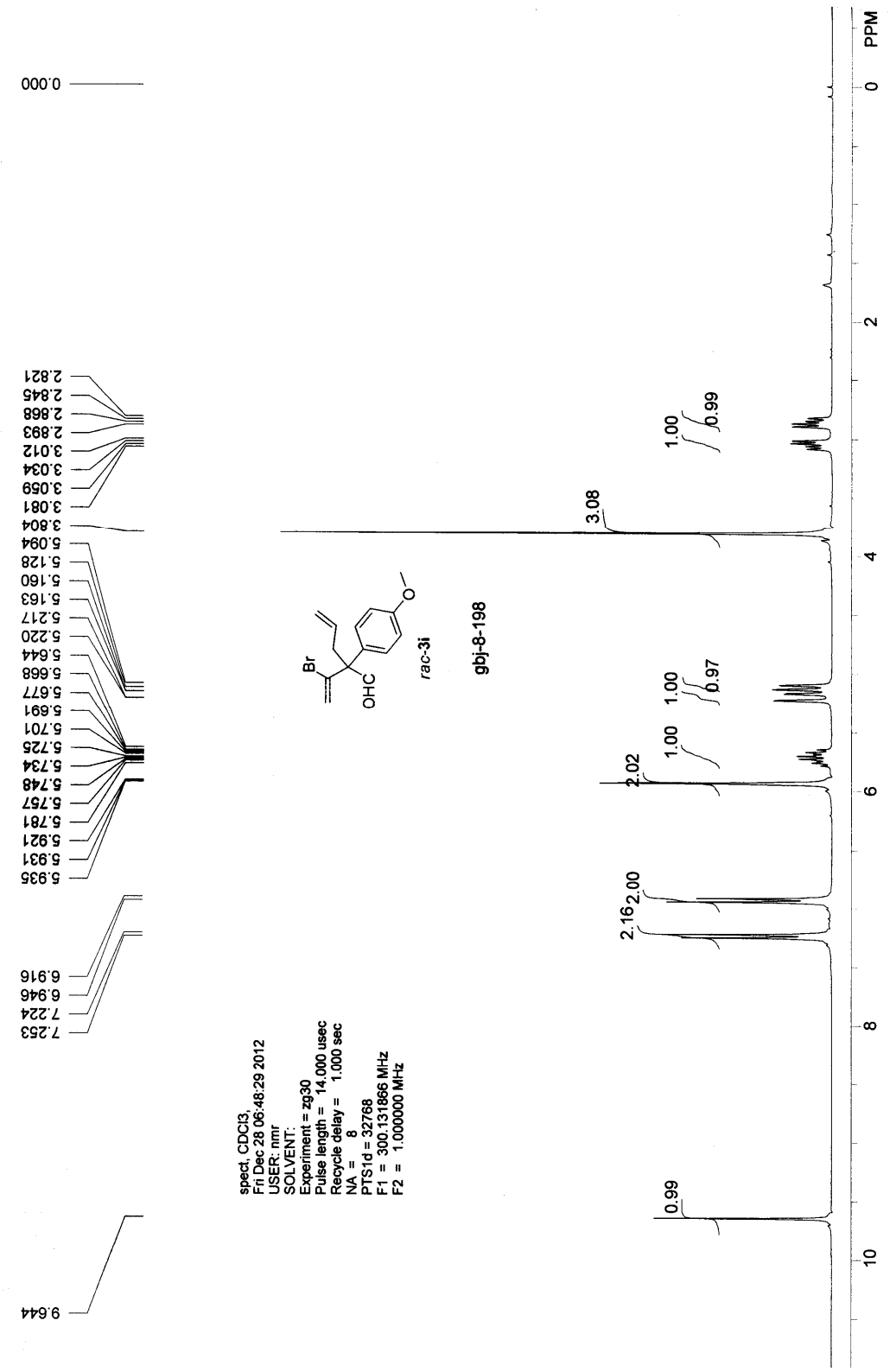

14.101
22.654
24.754
29.288
29.392
29.561
30.020
31.728
31.872
55.189
65.055
76.576
77.000
77.424
114.239
121.060
128.584
129.347
134.347
159.179
185.776
spect, CDCl3,
Mon Dec 17 05:05:44 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 250
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
$C_{10}H_{21}$-n
OHC
O
rac-3f
gbj-8-174-c
0 PPM
50
100
150
200

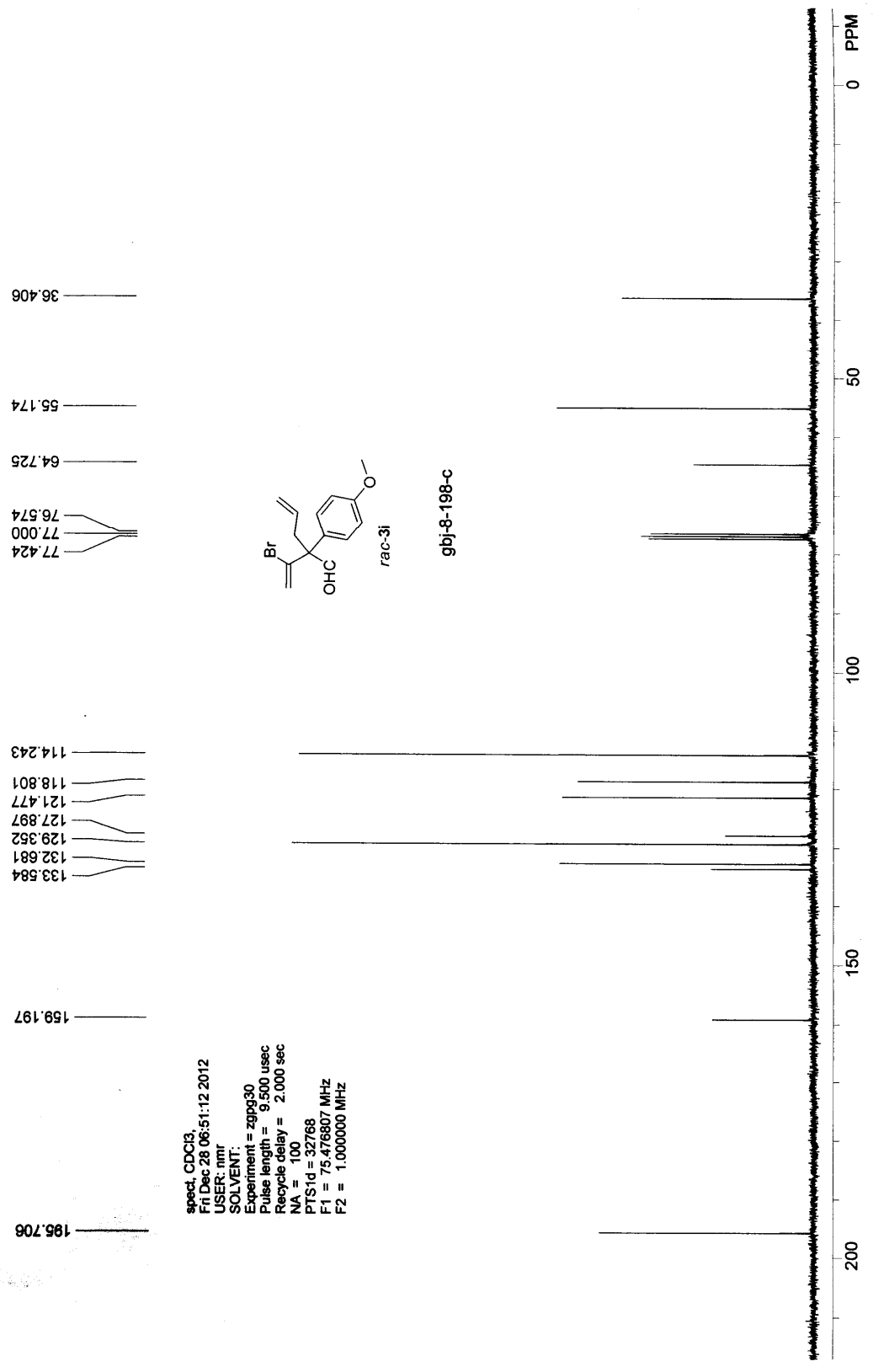

9.601
7.265
7.185
7.156
6.942
6.912
6.022
6.013
5.986
5.978
3.820
2.983
2.961
2.938
2.915
2.893
2.870
2.848
0.982
0.959
0.928
0.905
0.000
spect, CDCl3,
Tue Jan 08 08:18:47 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 9
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
C3H7-i
OHC
O
rac-3g
gbj-9-16
0.97
2.05
2.11
0.92
1.00
3.21
0.99
2.93
3.22
10
8
6
4
2
0
PPM

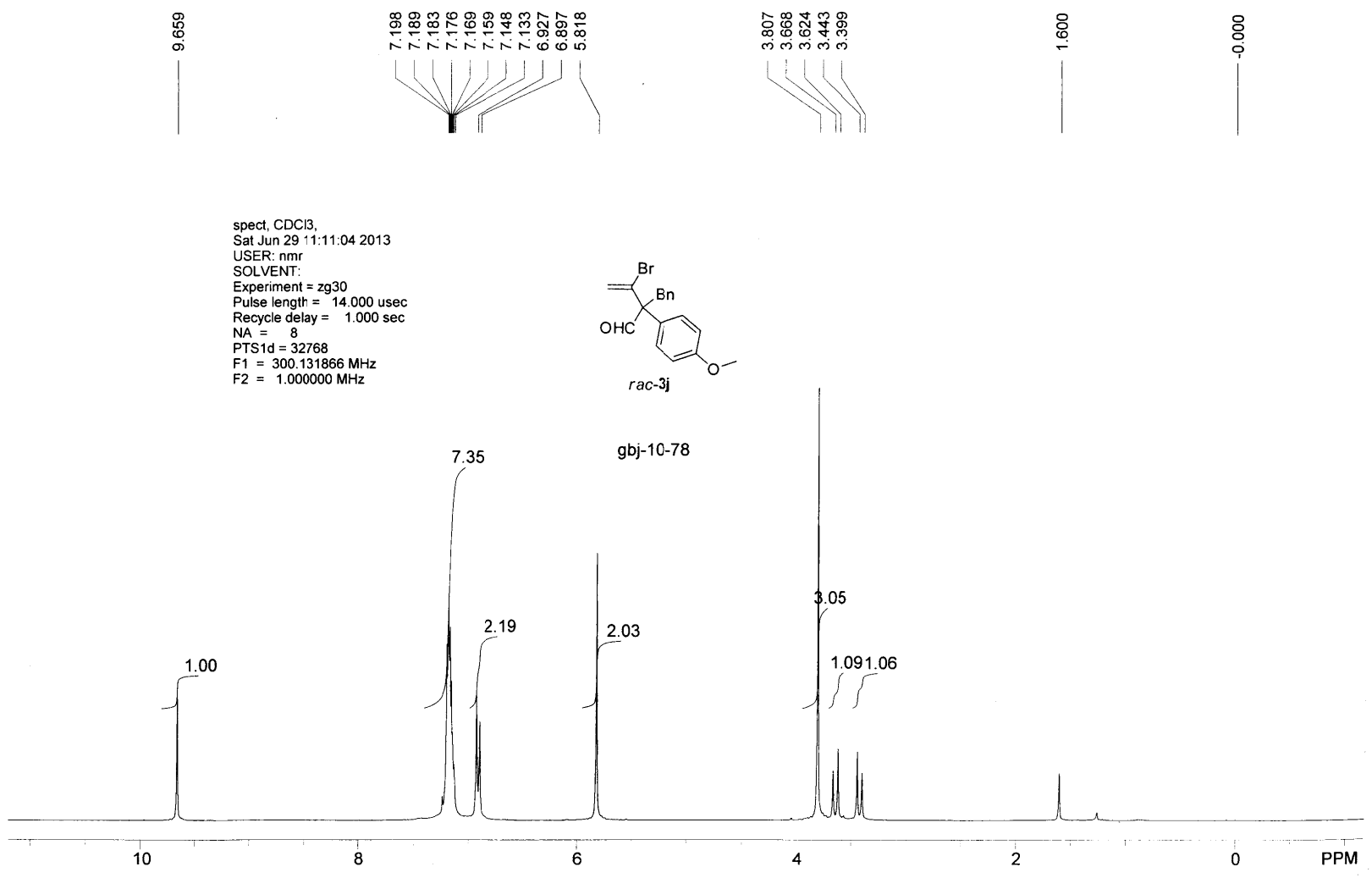

18.203
18.458
30.396
55.163
69.211
76.575
77.000
77.425
113.641
123.425
127.270
130.724
132.980
158.823
196.773
spect, CDCl3,
Tue Jan 08 08:20:01 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 102
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
C3H7-i
OHC
O
rac-3g
gbj-9-16-c
200
150
100
50
0
PPM

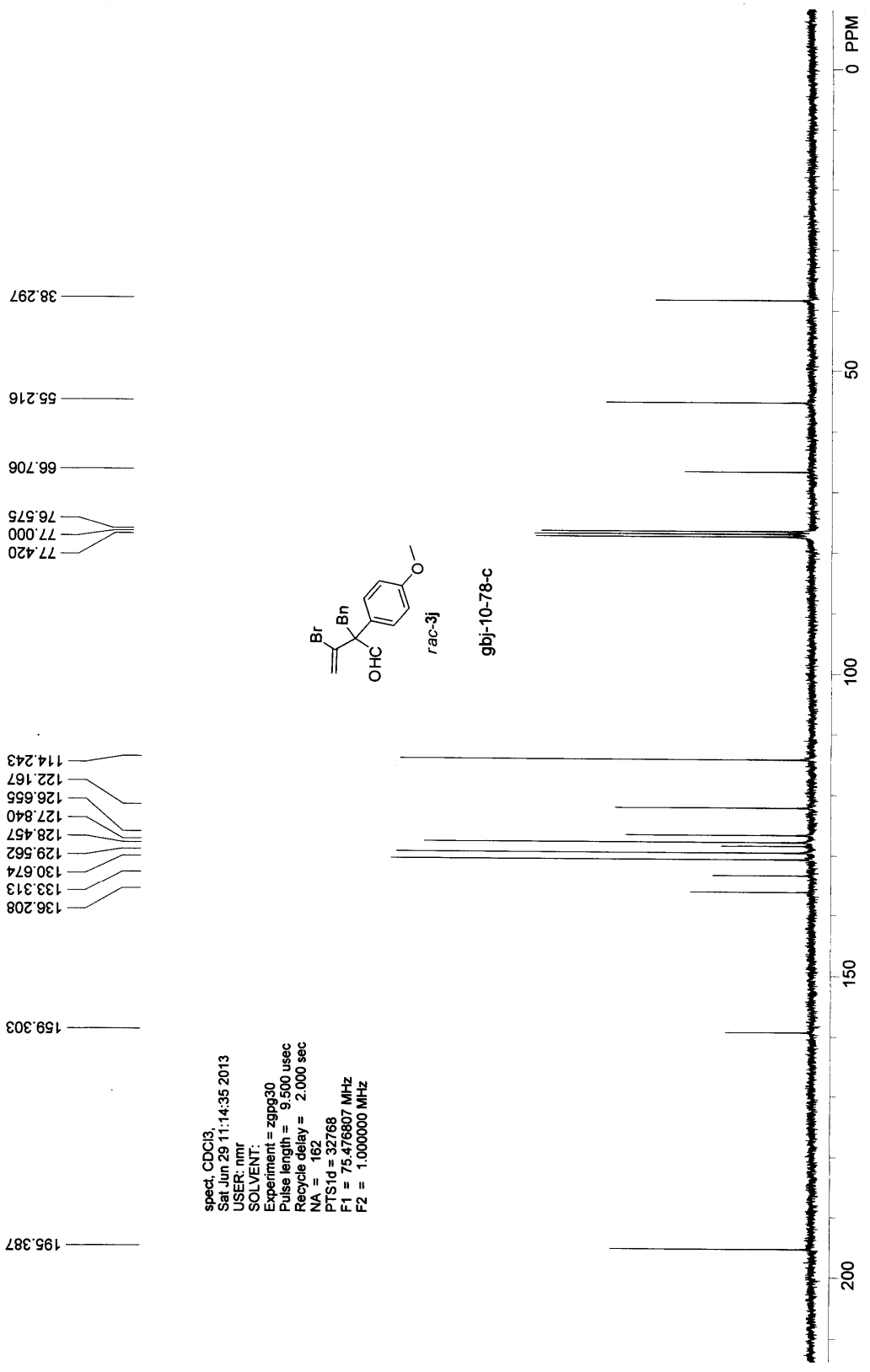

9.571
7.264
7.157
7.128
6.933
6.903
6.004
5.996
5.968
5.960
3.817
2.552
2.513
2.474
1.837
1.787
1.740
1.648
1.427
1.414
1.382
1.340
1.305
1.297
1.257
1.108
1.066
1.023
1.010
0.959
0.918
0.878
0.870
0.836
0.797
-0.000
spect, CDCl3,
Mon Jan 07 07:39:05 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
OHC
O
rac-3h
gbj-9-14
1.04
2.03
2.07
0.94
1.00
3.02
1.03
5.60
2.26
3.35
10
8
6
4
2
0
PPM

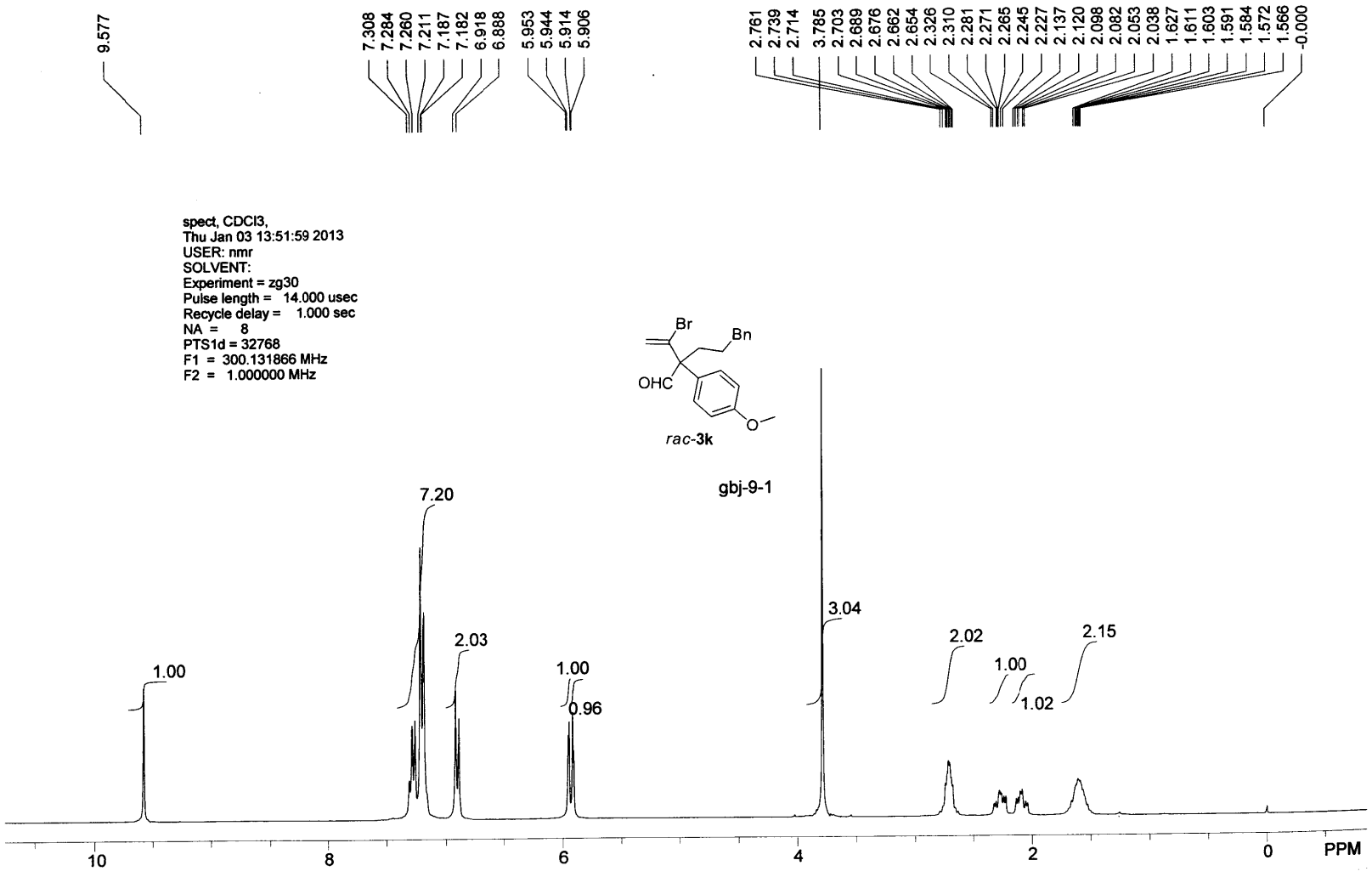

spect, CDCl3,
Mon Jan 07 07:42:40 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 180
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
gbj-9-14-c
rac-3h
Br
OHC
O
PPM
0
50
100
150
200
26.360
26.738
26.818
28.609
28.881
41.237
55.144
69.197
76.575
77.000
77.425
113.580
123.328
127.501
130.656
133.081
158.745
196.625

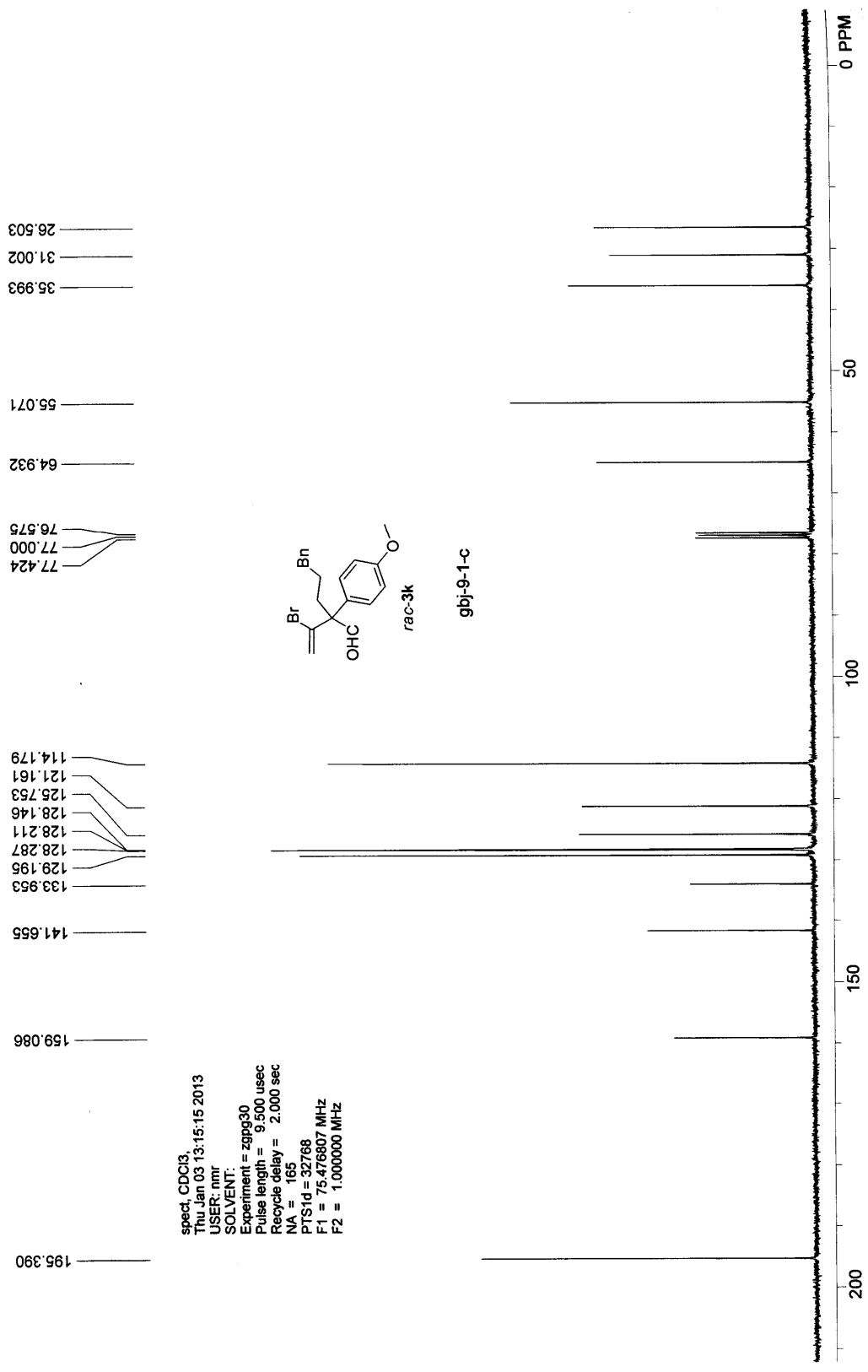

spect, CDCl3,
Fri Dec 28 06:48:29 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-8-198
rac-3i
Br
OHC
O
3.08
2.16
2.00
2.02
1.00
1.00
1.00
0.99
0.97
0.99
PPM
0
2
4
6
8
10
0.000
9.644
7.253
6.946
6.916
5.935
5.931
5.921
5.781
5.757
5.748
5.734
5.725
5.701
5.691
5.677
5.668
5.644
5.220
5.217
5.163
5.160
5.128
5.094
3.804
3.081
3.059
3.034
3.012
2.893
2.868
2.845
2.821

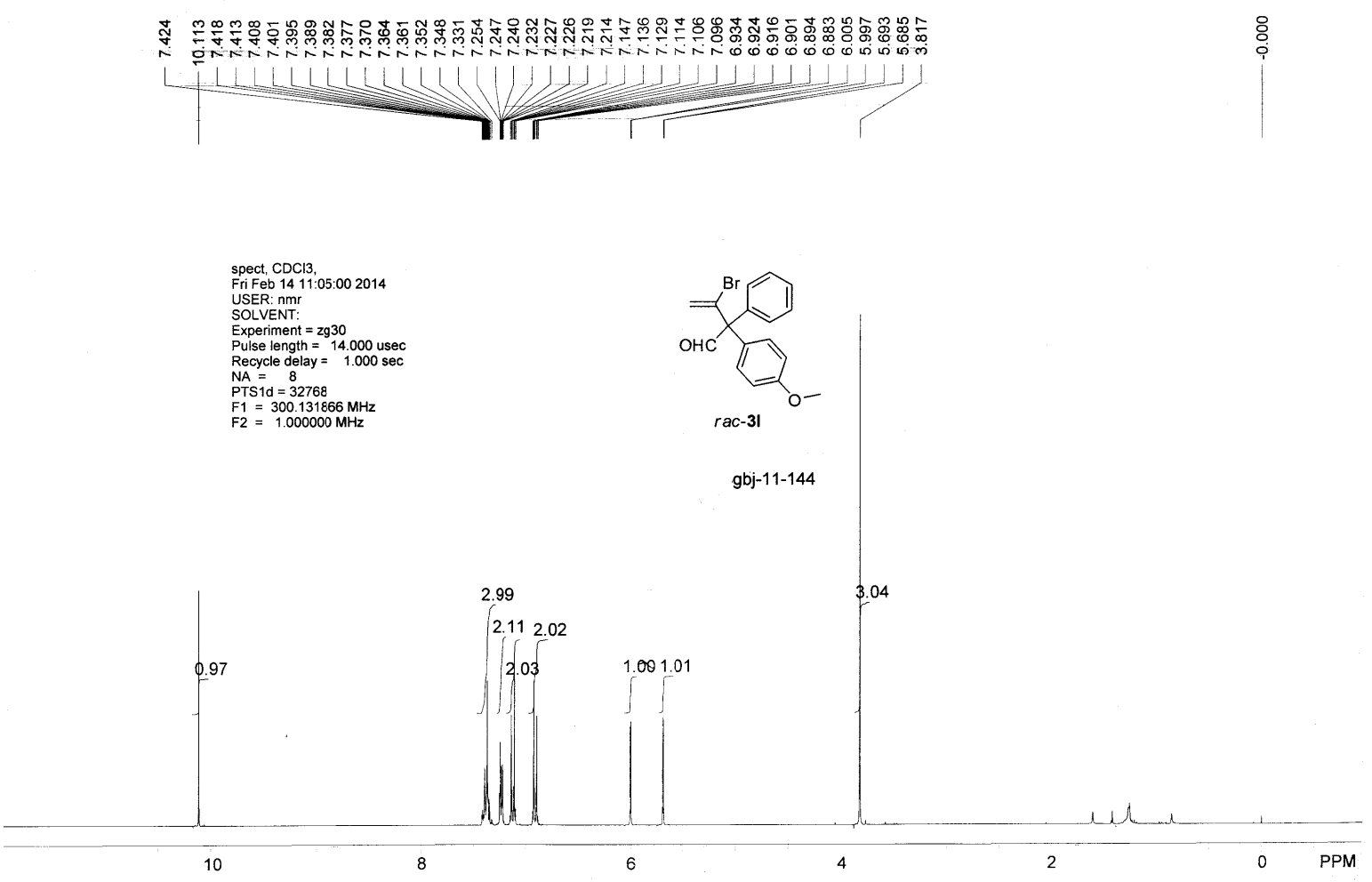

spect, CDCl3,
Fri Dec 28 06:51:12 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 100
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
186.706
159.197
133.584
132.681
129.352
127.897
121.477
118.801
114.243
77.424
77.000
76.574
64.725
55.174
36.406
Br
OHC
O
rac-3i
gbj-8-198-c
PPM
0
50
100
150
200

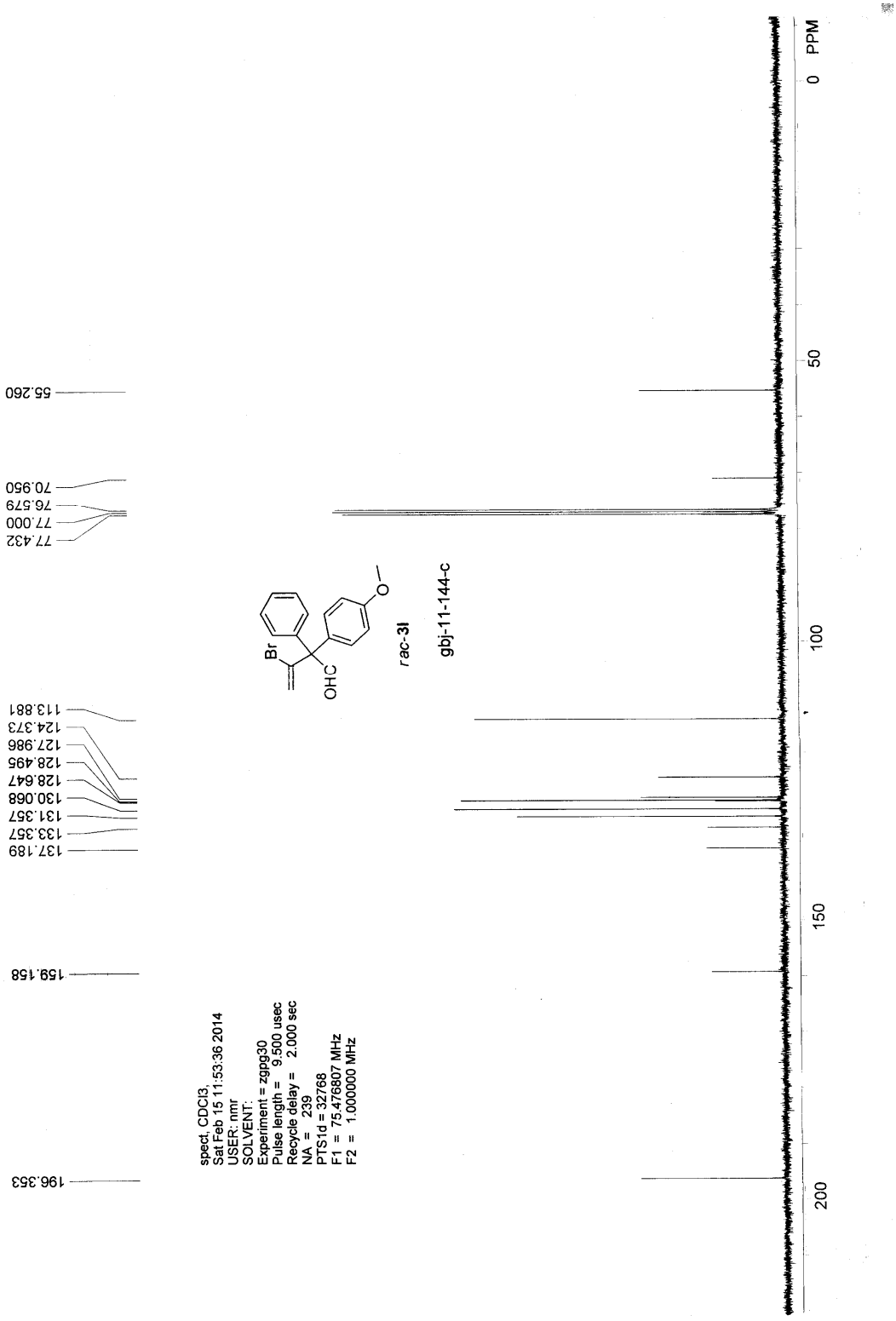

9.659
7.198
7.189
7.183
7.176
7.169
7.159
7.148
7.133
6.927
6.897
5.818
3.807
3.668
3.624
3.443
3.399
1.600
-0.000
spect, CDCl3,
Sat Jun 29 11:11:04 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
Bn
OHC
O
rac-3j
gbj-10-78
7.35
2.19
2.03
3.05
1.09
1.06
1.00
10
8
6
4
2
0
PPM

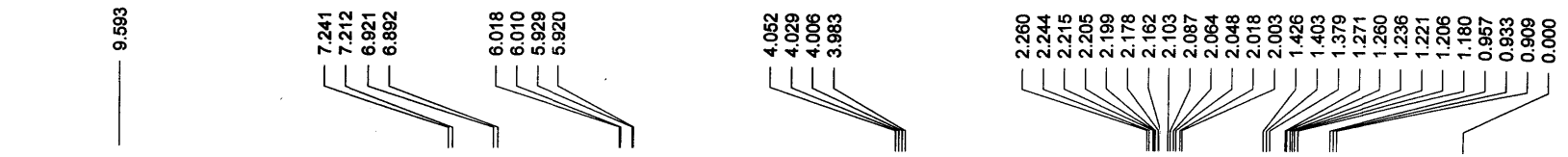

195.387
159.303
136.208
133.313
130.674
129.562
128.457
127.840
126.655
122.167
114.243
77.420
77.000
76.575
66.706
55.216
38.297
spect, CDCl3,
Sat Jun 29 11:14:35 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 162
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
Bn
OHC
O
rac-3j
gbj-10-78-c
200
150
100
50
0 PPM


9.577
7.308
7.284
7.260
7.211
7.187
7.182
6.918
6.888
5.953
5.944
5.914
5.906
2.761
2.739
2.714
3.785
2.703
2.689
2.676
2.662
2.654
2.326
2.310
2.281
2.271
2.265
2.245
2.227
2.137
2.120
2.098
2.082
2.053
2.038
1.627
1.611
1.603
1.591
1.584
1.572
1.566
-0.000
spect, CDCl3,
Thu Jan 03 13:51:59 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
Bn
OHC
O
rac-3k
gbj-9-1
7.20
2.03
1.00
1.00
0.96
3.04
2.02
1.00
1.02
2.15
10
8
6
4
2
0
PPM

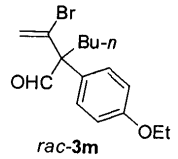

spect, CDCl3,
Thu Jan 03 13:15:15 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 165
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
gbj-9-1-c
rac-3k
Br
Bn
OHC
O
195.390
159.086
141.655
133.953
129.195
128.287
128.211
128.146
125.753
121.161
114.179
77.424
77.000
76.575
64.932
55.071
35.993
31.002
26.503
200
150
100
50
0 PPM


7.424
10.113
7.418
7.413
7.408
7.401
7.395
7.389
7.382
7.377
7.370
7.364
7.361
7.352
7.348
7.331
7.254
7.247
7.240
7.232
7.227
7.226
7.219
7.214
7.147
7.136
7.129
7.114
7.106
7.096
6.934
6.924
6.916
6.901
6.894
6.883
6.005
5.997
5.693
5.685
3.817
-0.000
spect, CDCl3,
Fri Feb 14 11:05:00 2014
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
OHC
O
rac-3l
gbj-11-144
0.97
2.99
2.11
2.02
2.03
1.09
1.01
3.04
10
8
6
4
2
0
PPM

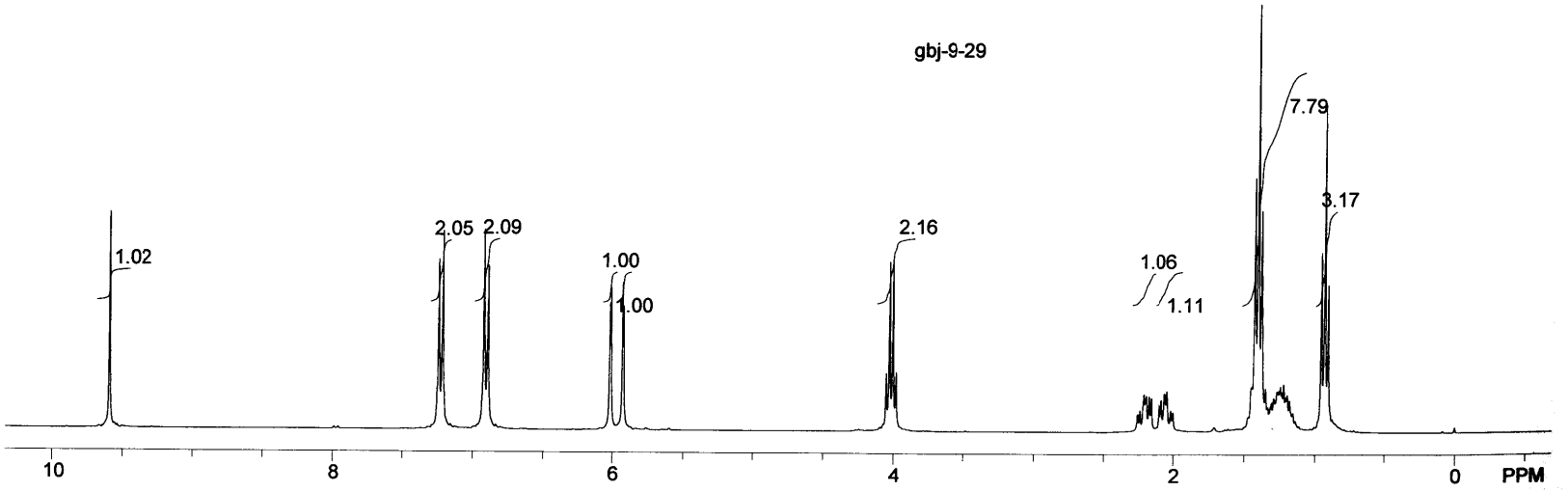

spect, CDCl3,
Sat Feb 15 11:53:36 2014
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 239
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
196.353
158.158
137.189
133.357
131.357
130.068
128.647
128.495
127.986
124.373
113.881
77.432
77.000
76.579
70.950
55.260
Br
OHC
O
rac-3l
gbj-11-144-c
0
50
100
150
200
PPM

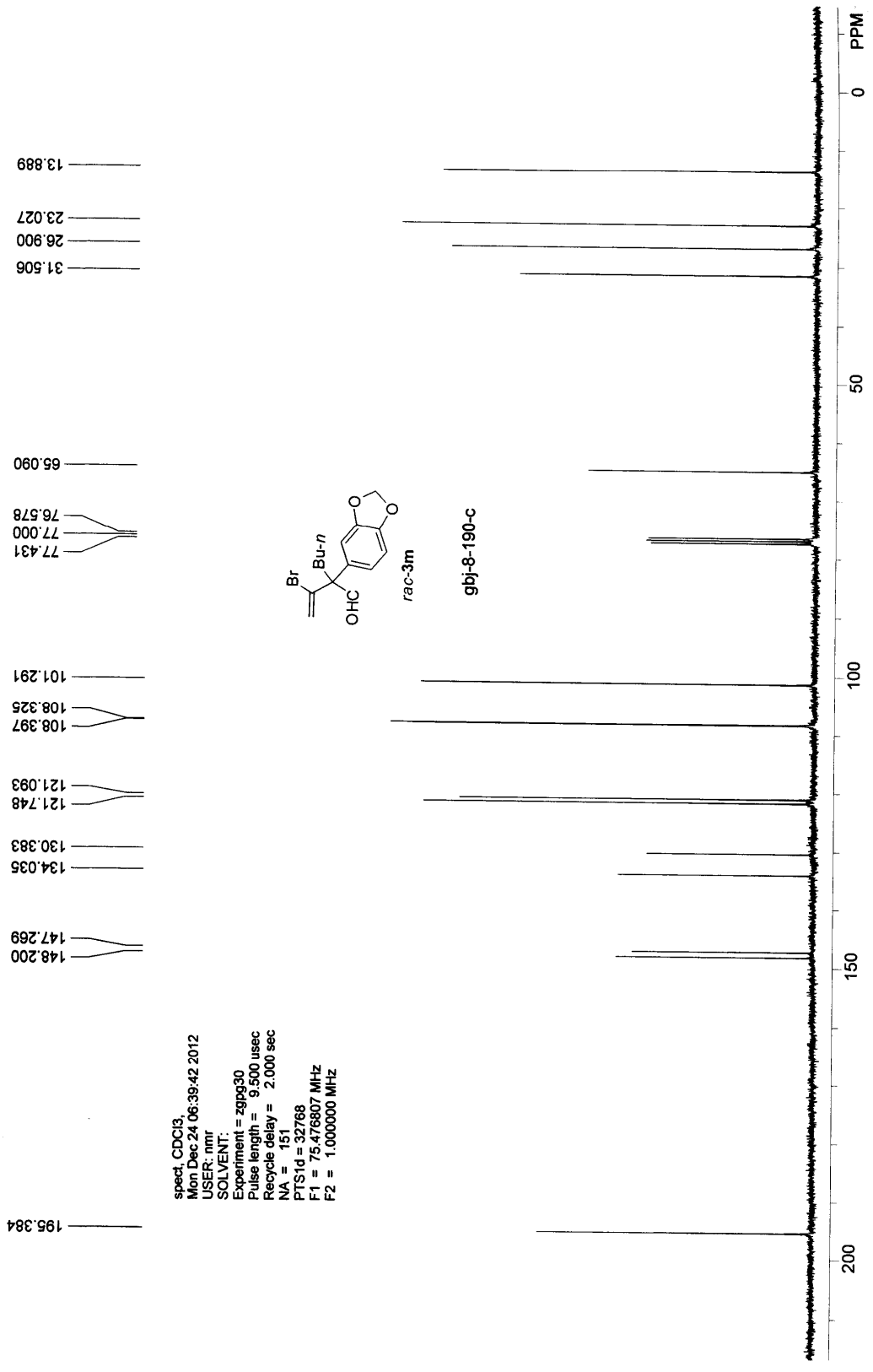


spect, CDCl3,
Thu Jan 17 07:34:49 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz

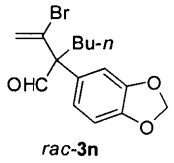


rac-**3m**



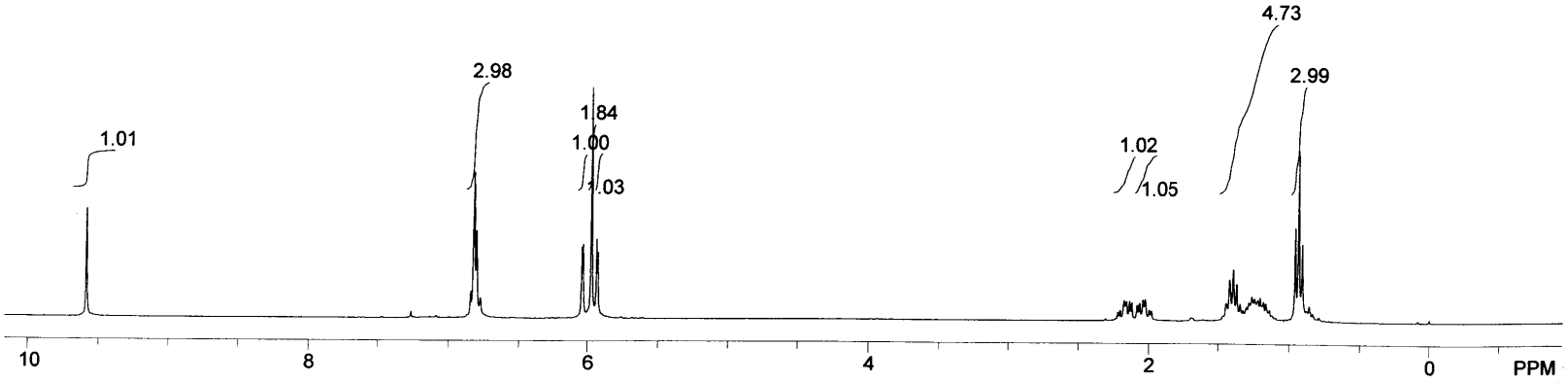

spect, CDCl3,
Mon Dec 24 06:39:42 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 151
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
195.384
148.200
147.269
134.035
130.383
121.748
121.093
108.397
108.325
101.291
77.431
77.000
76.578
65.090
31.506
26.900
23.027
13.889
Br
Bu-n
OHC
rac-3m
gbj-8-190-c
PPM
0
50
100
150
200

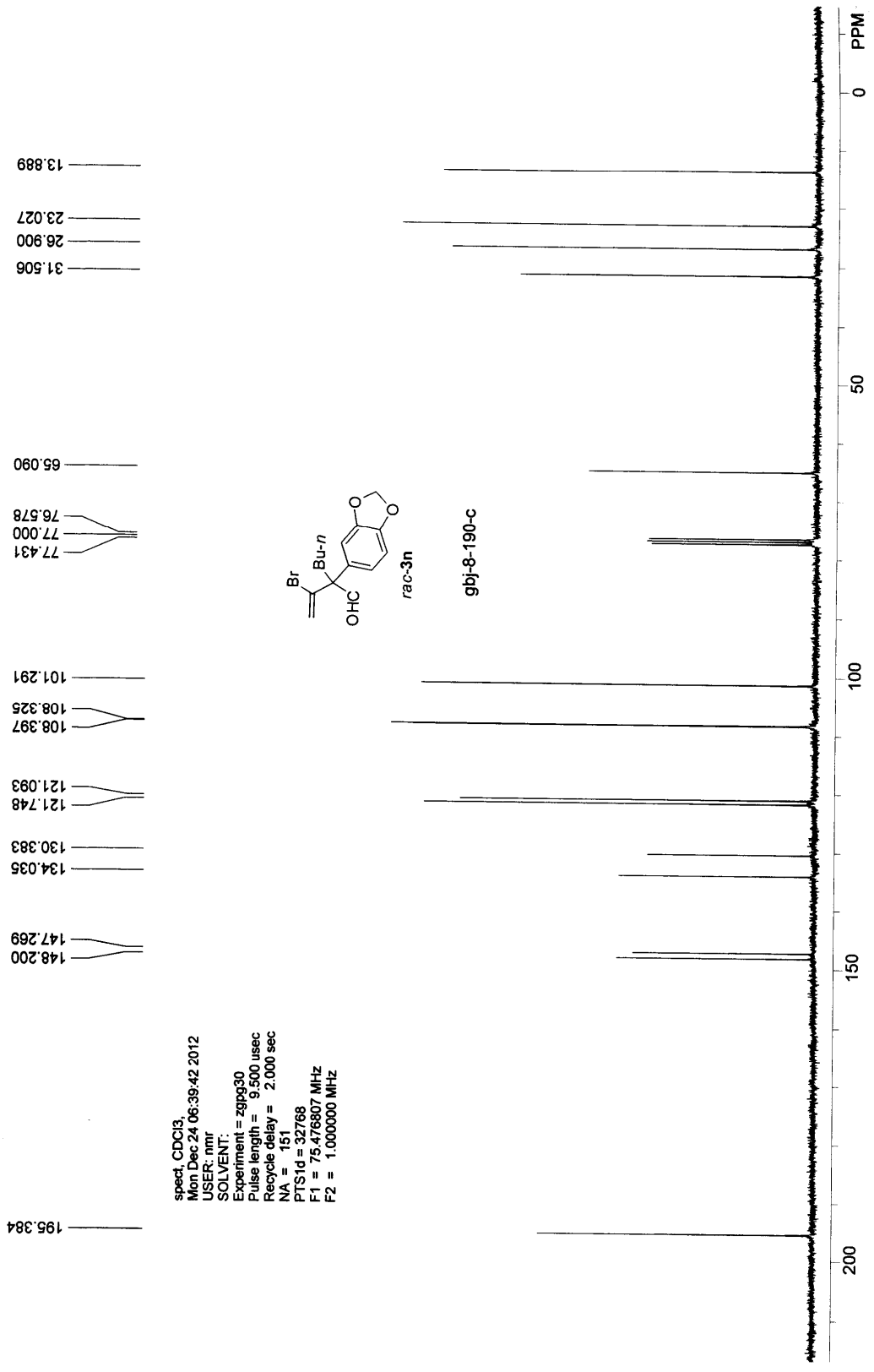
spect, CDCl3,
Tue Dec 25 12:37:11 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz

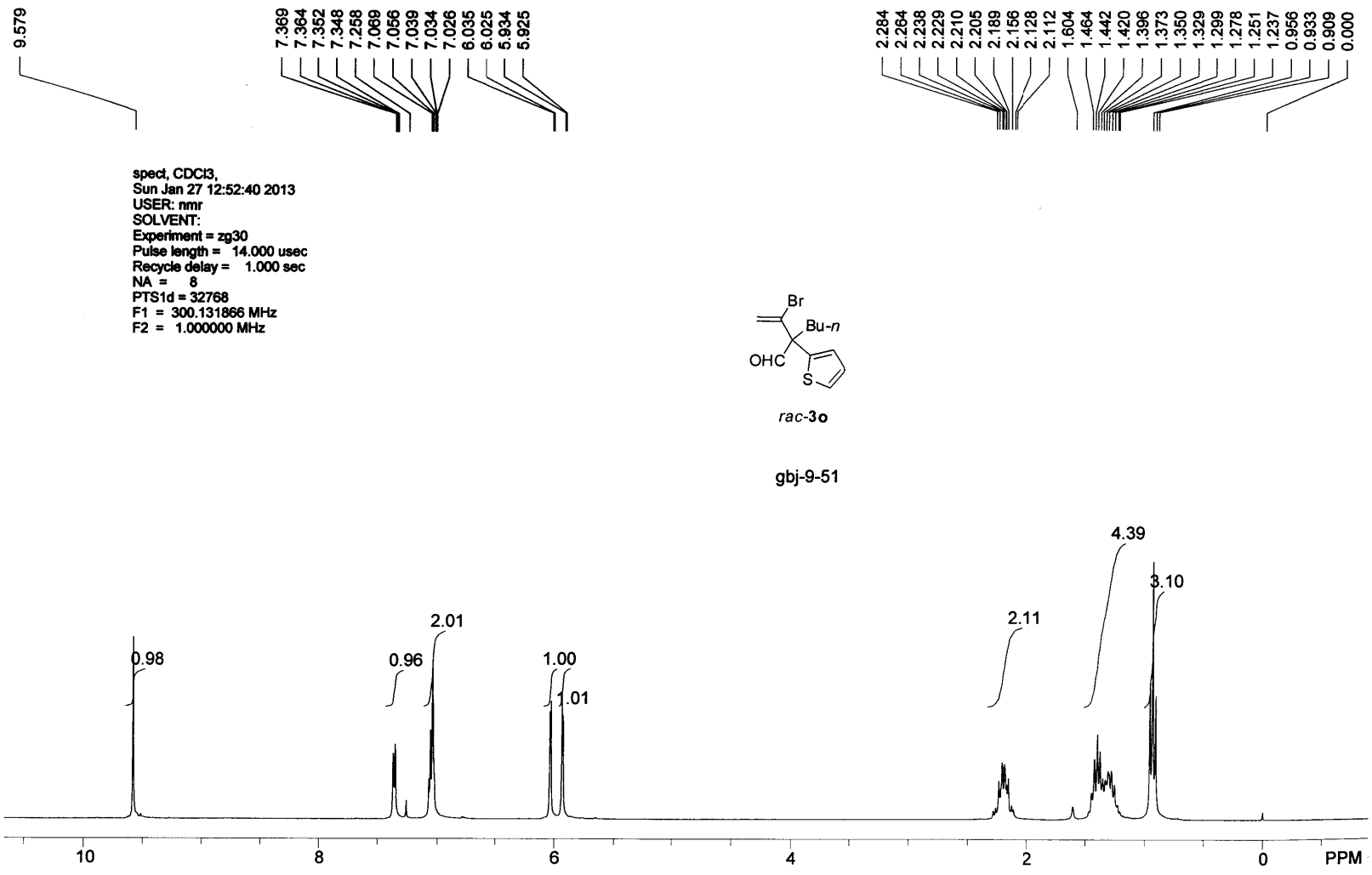


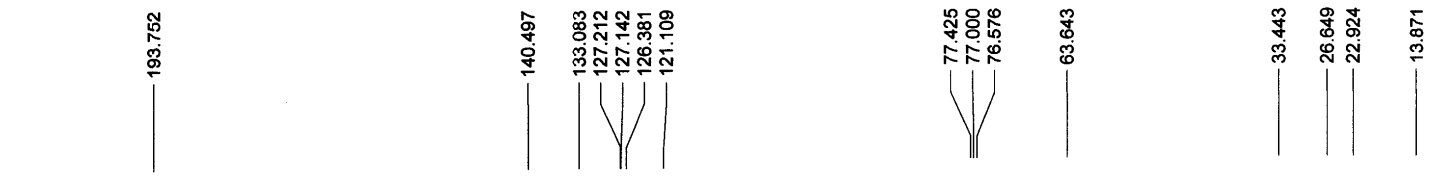
rac-**3n**

gbj-8-190

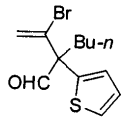



spect, CDCl3,
Mon Dec 24 06:39:42 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 151
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
195.384
148.200
147.269
134.035
130.383
121.748
121.093
108.397
108.325
101.291
77.431
77.000
76.578
65.090
31.506
26.900
23.027
13.889
Br
Bu-n
OHC
rac-3n
gbj-8-190-c
PPM
0
50
100
150
200


9.579
7.369
7.364
7.352
7.348
7.258
7.069
7.056
7.039
7.034
7.026
6.035
6.025
5.934
5.925
2.284
2.264
2.238
2.229
2.210
2.205
2.189
2.156
2.128
2.112
1.604
1.464
1.442
1.420
1.396
1.373
1.350
1.329
1.299
1.278
1.251
1.237
0.956
0.933
0.909
0.000
spect, CDCl3,
Sun Jan 27 12:52:40 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
Bu-n
OHC
S
rac-3o
gbj-9-51
0.98
0.96
2.01
1.00
1.01
2.11
4.39
3.10
10
8
6
4
2
0
PPM

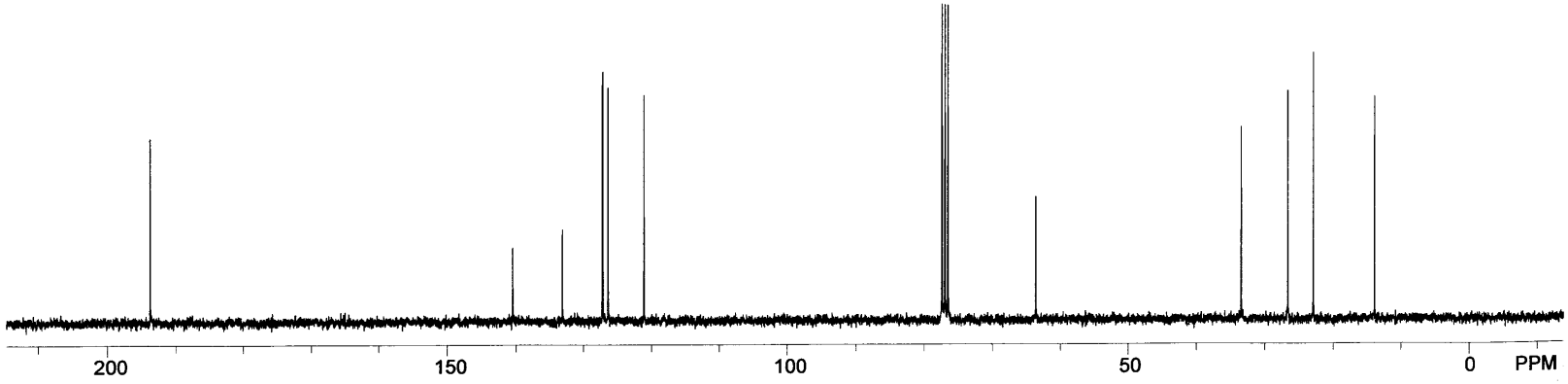


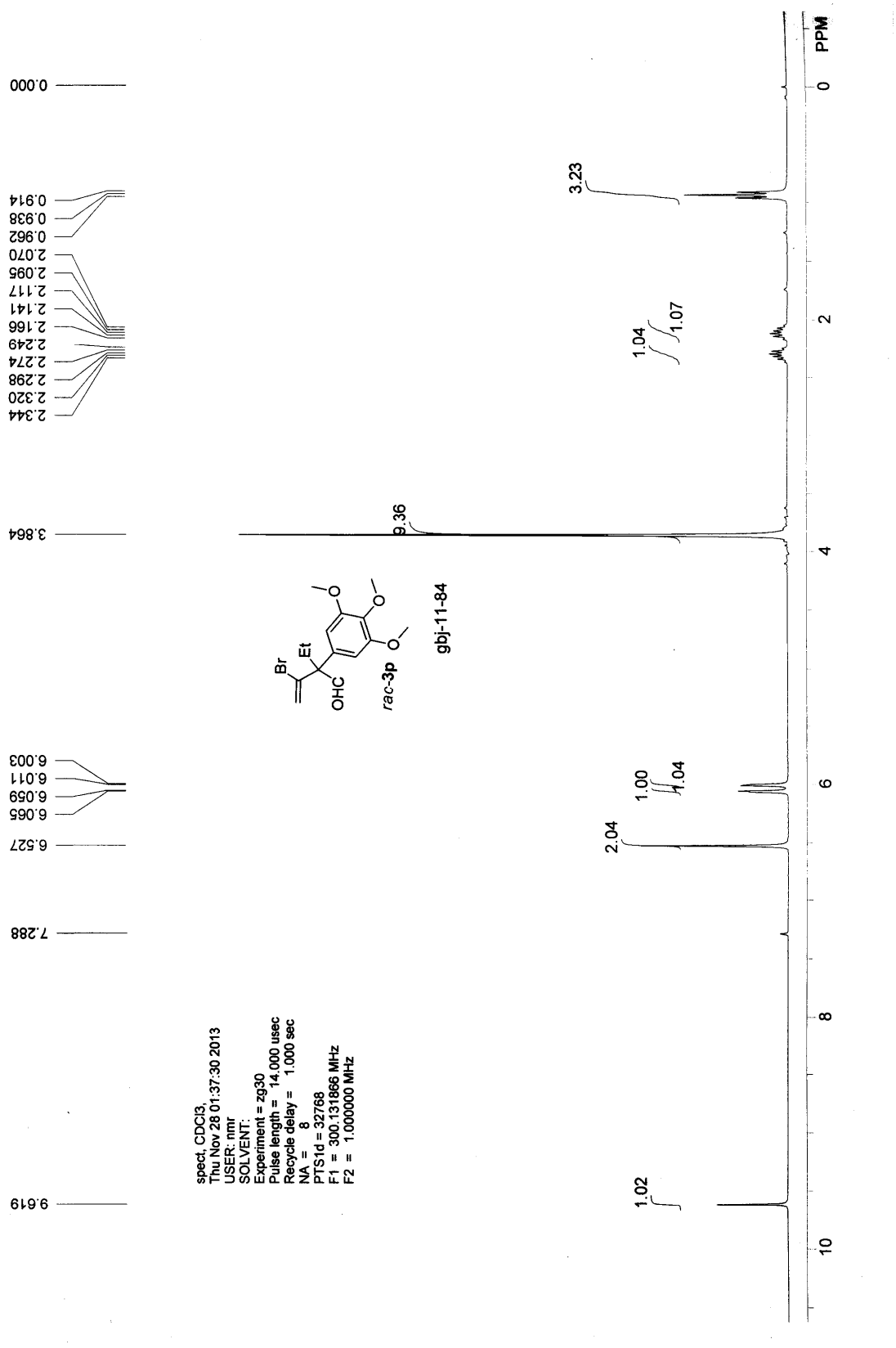
spect, CDCl3,
Sun Jan 27 12:56:18 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 152
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz

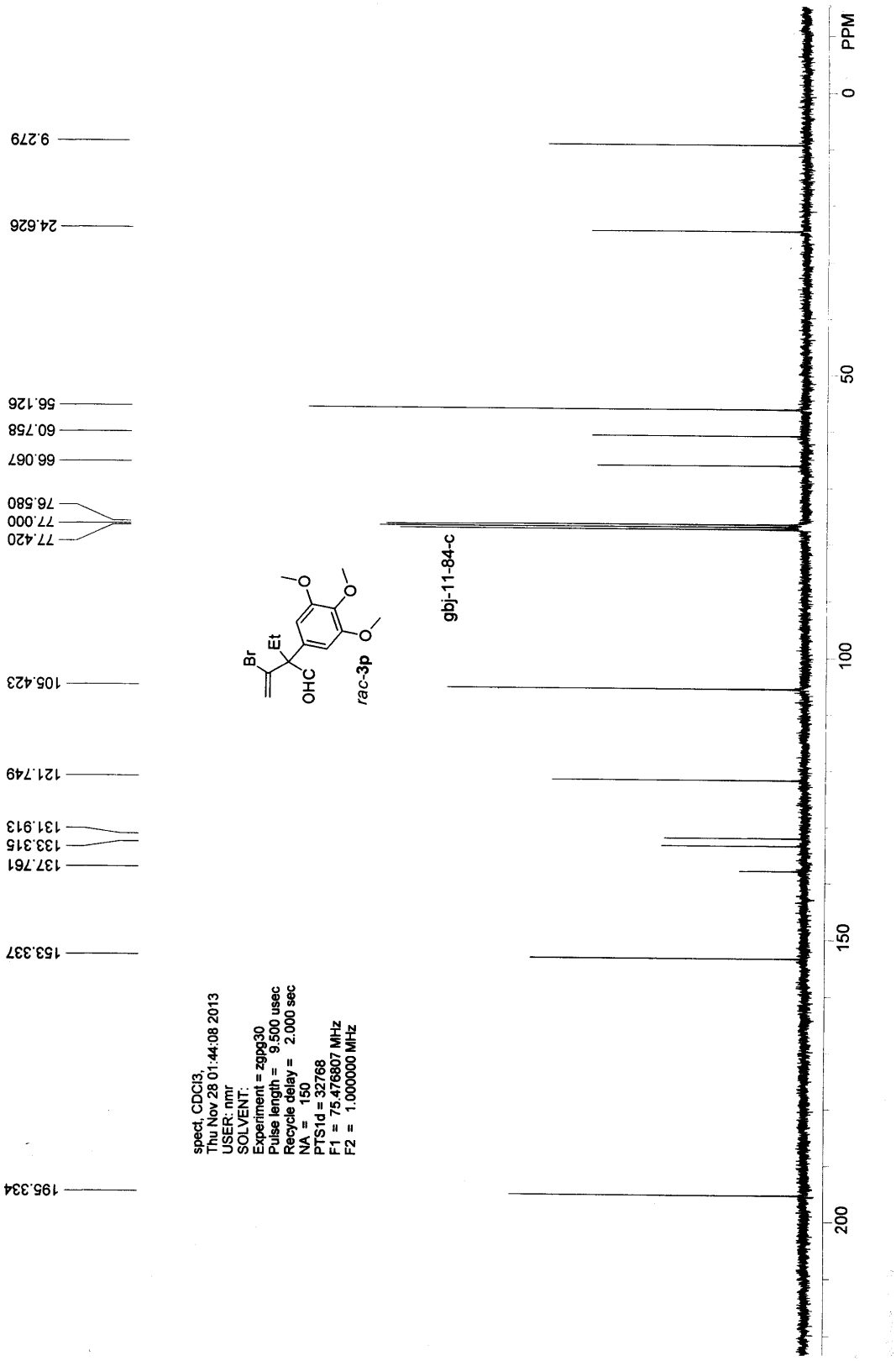


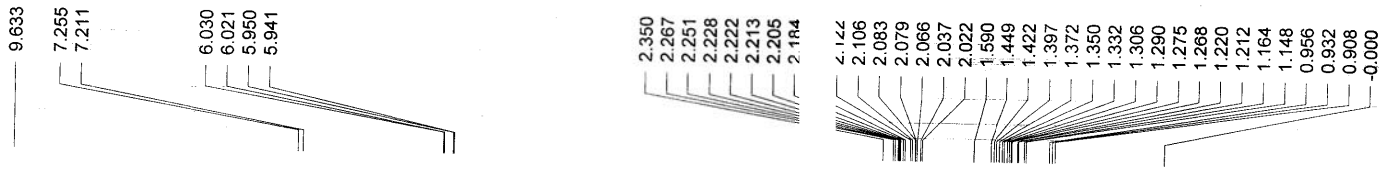
rac-**3o**

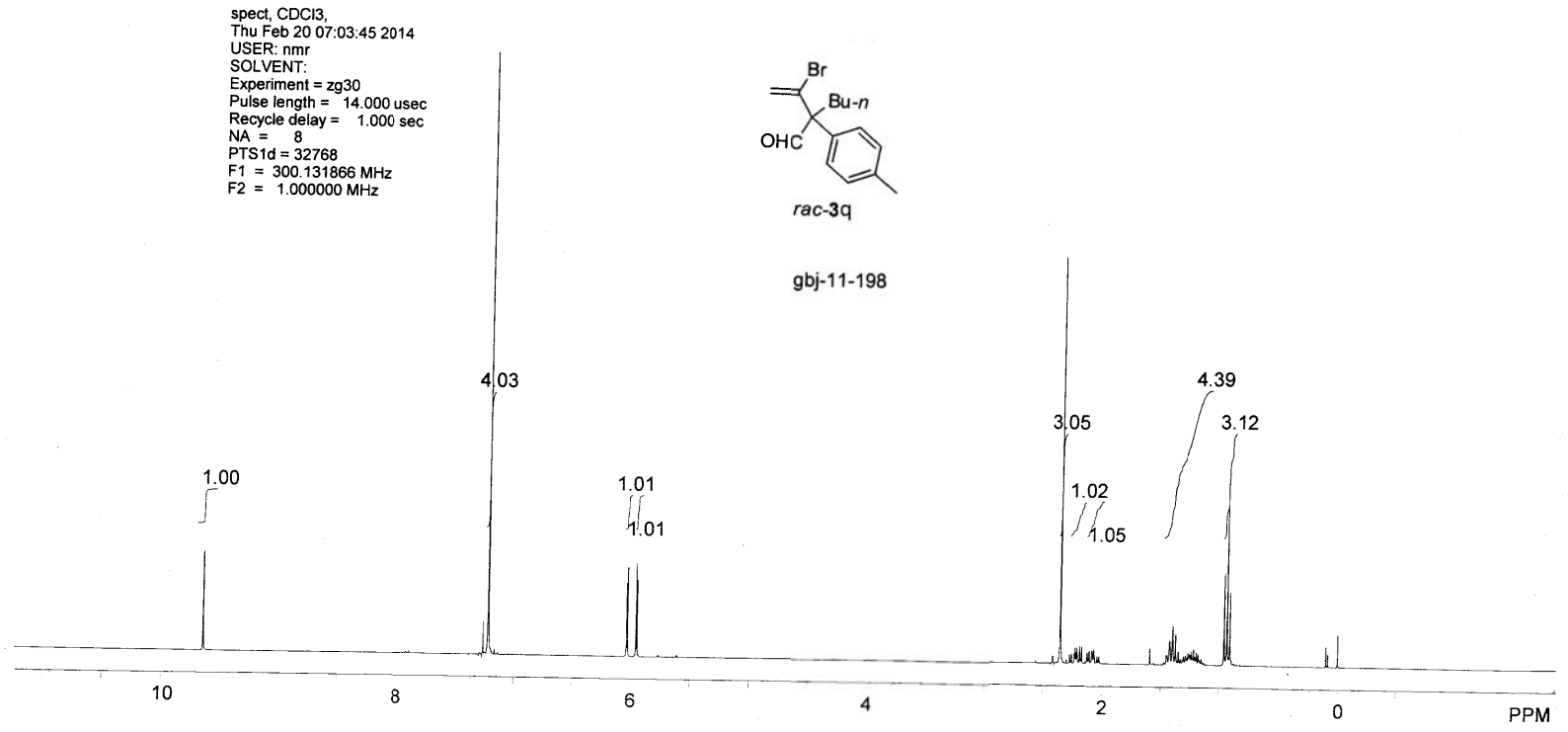
gbj-9-51-c

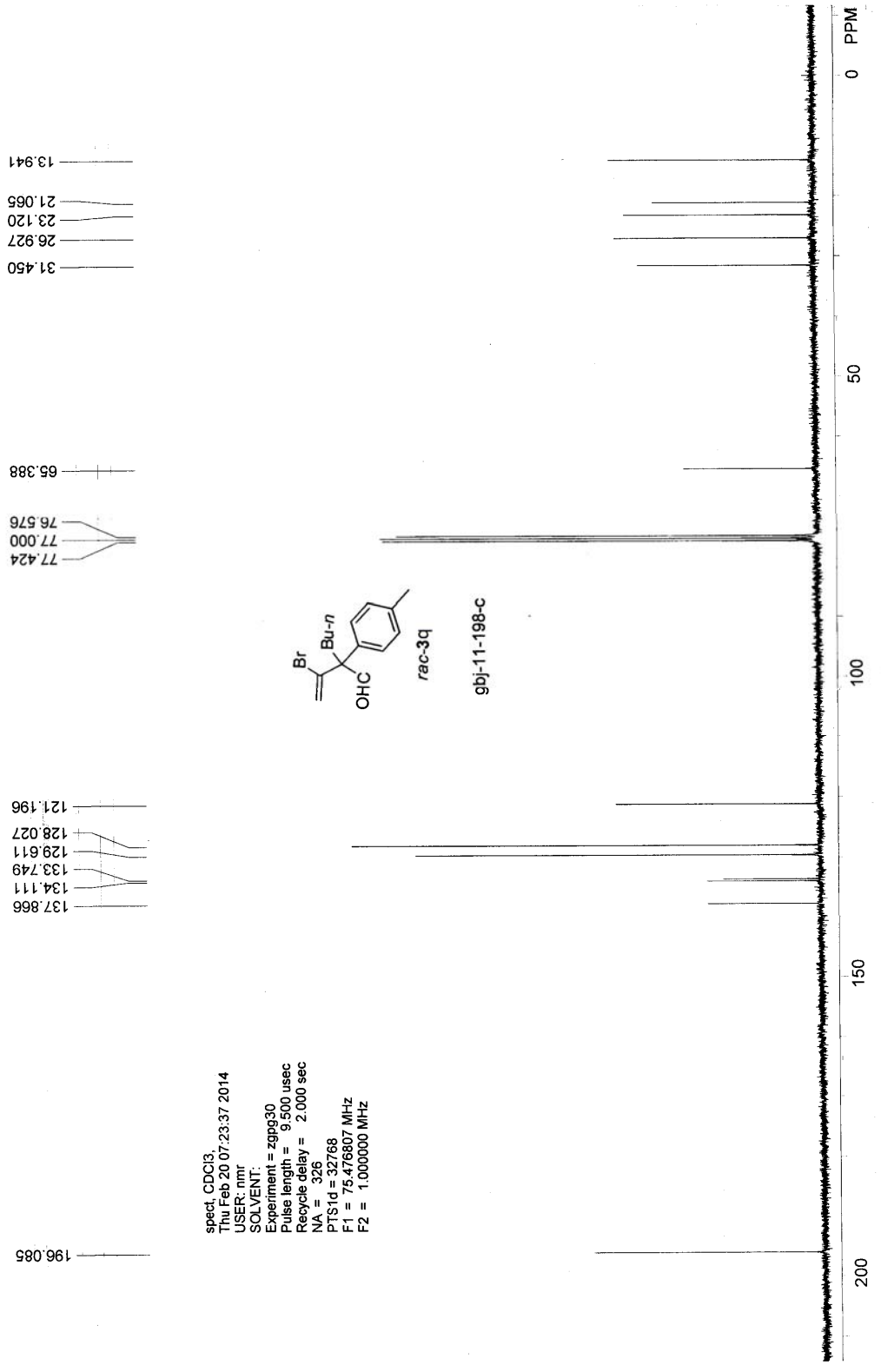


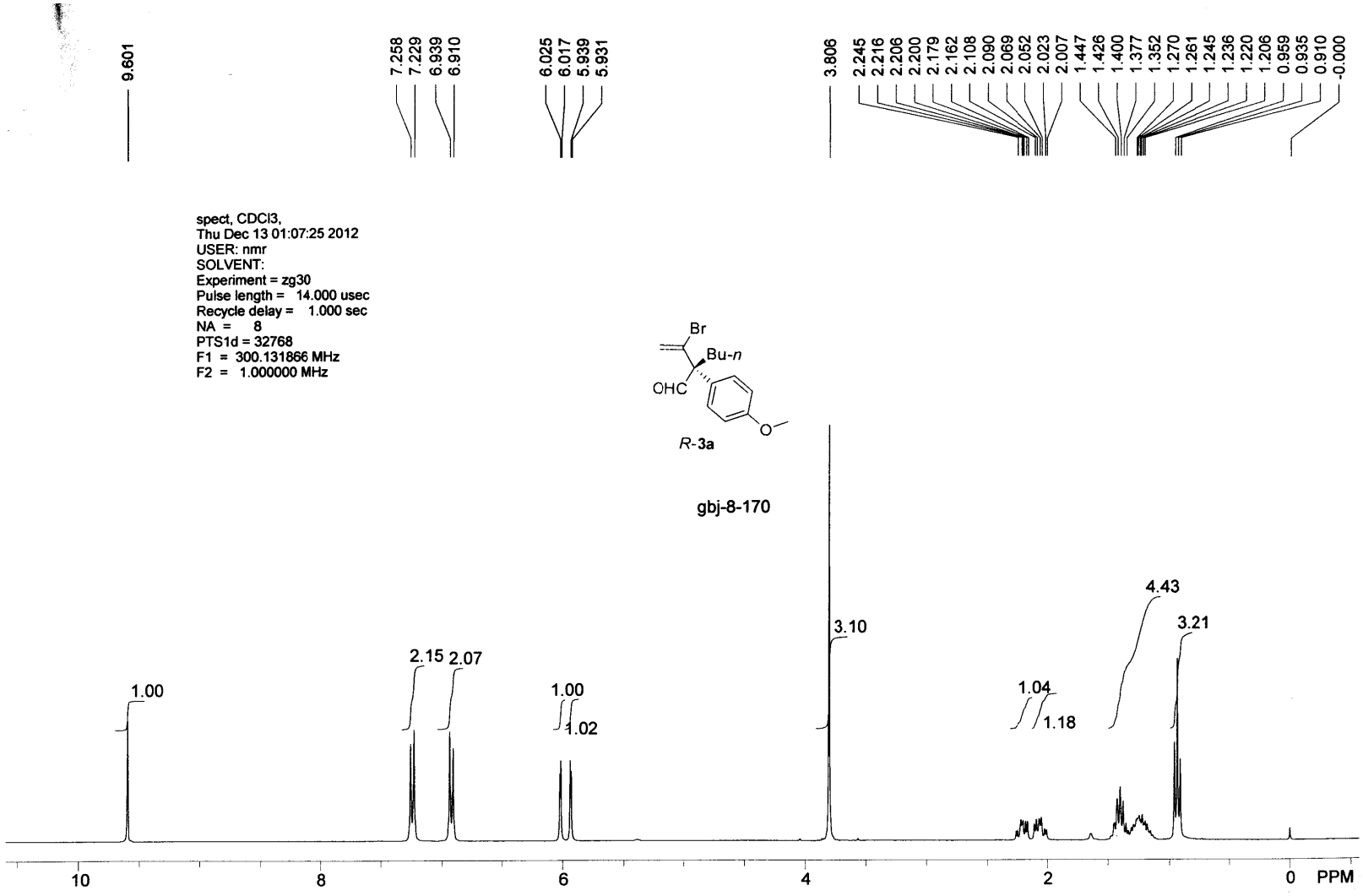

spect, CDCl3,
Thu Nov 28 01:37:30 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-11-84
rac-3p
Br
Et
OHC
O
O
O
9.619
7.288
6.627
6.065
6.059
6.011
6.003
3.864
2.344
2.320
2.298
2.274
2.249
2.166
2.141
2.117
2.095
2.070
0.962
0.938
0.914
0.000
9.36
3.23
1.07
1.04
2.04
1.00
1.04
1.02
PPM
0
2
4
6
8
10

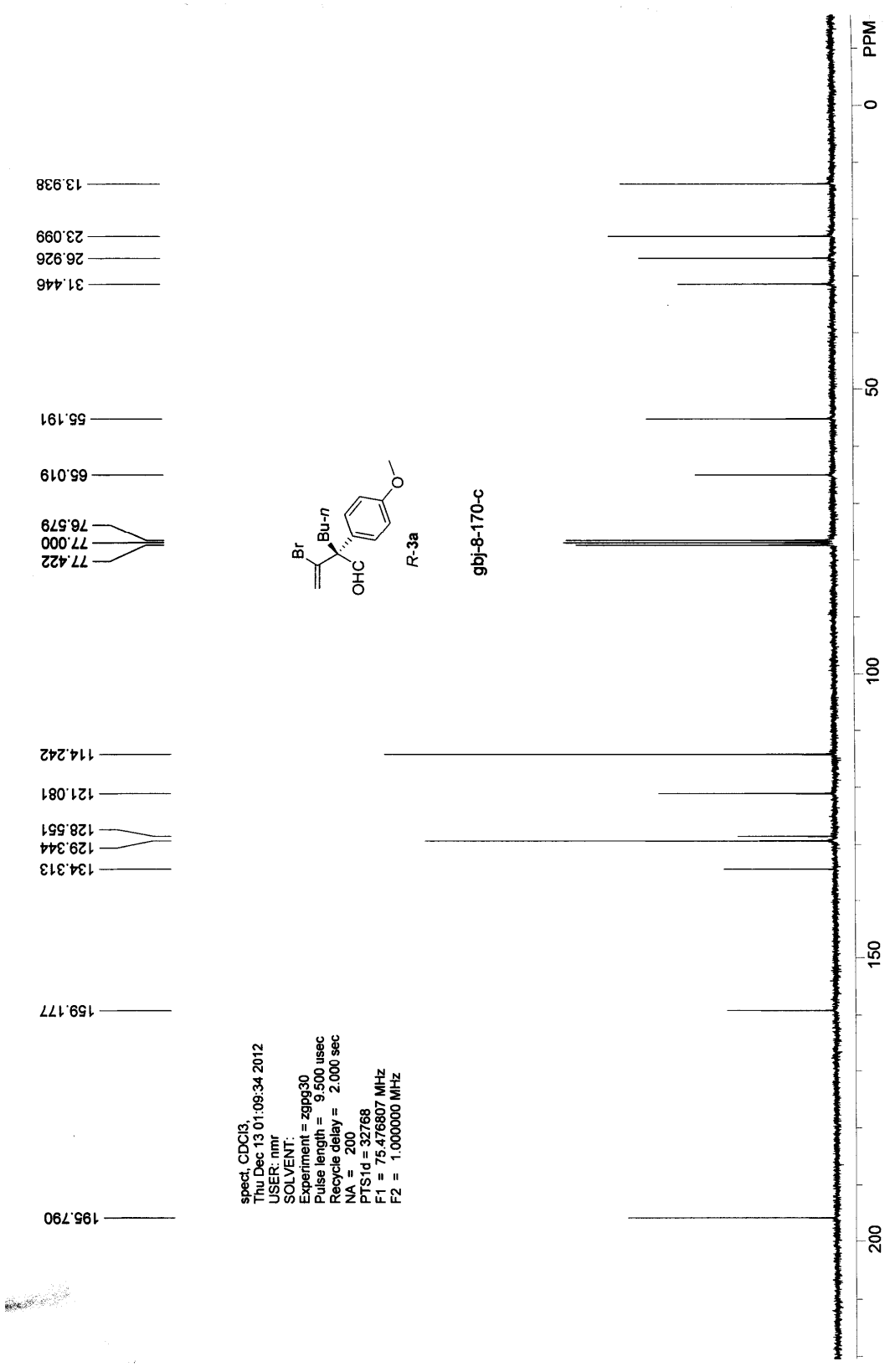

9.279
24.626
56.126
60.758
66.067
76.580
77.000
77.420
105.423
121.749
131.913
133.315
137.761
153.337
195.334
spect, CDCl3,
Thu Nov 28 01:44:08 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 150
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
gbj-11-84-c
Br
Et
OHC
rac-3p
0
50
100
150
200
PPM


9.633
7.255
7.211
6.030
6.021
5.950
5.941
2.350
2.267
2.251
2.228
2.222
2.213
2.205
2.106
2.083
2.079
2.066
2.037
2.022
1.590
1.449
1.422
1.397
1.372
1.350
1.332
1.306
1.290
1.275
1.268
1.220
1.212
1.164
1.148
0.956
0.932
0.908
-0.000


spect, CDCl3,
Thu Feb 20 07:03:45 2014
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
Bu-n
OHC
rac-3q
gbj-11-198
1.00
4.03
1.01
1.01
3.05
1.02
1.05
4.39
3.12
10
8
6
4
2
0
PPM


13.941
21.065
23.120
26.927
31.450
65.388
76.576
77.000
77.424
121.196
128.027
129.611
133.749
134.111
137.866
196.085
spect, CDCl3,
Thu Feb 20 07:23:37 2014
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 326
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
Bu-n
OHC
rac-3q
gbj-11-198-c
0
50
100
150
200
PPM

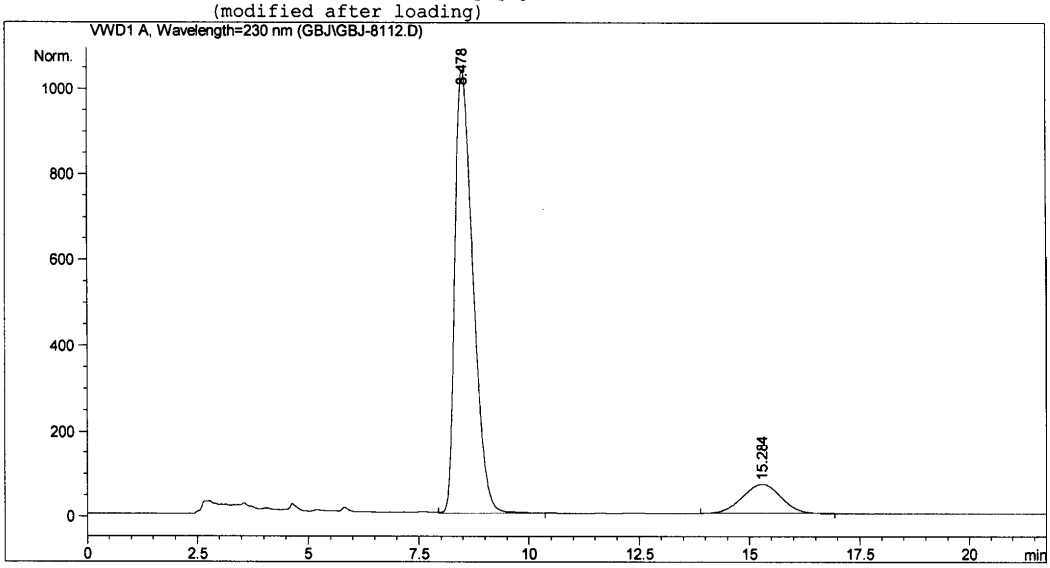

9.601
7.258
7.229
6.939
6.910
6.025
6.017
5.939
5.931
3.806
2.245
2.216
2.206
2.200
2.179
2.162
2.108
2.090
2.069
2.052
2.023
2.007
1.447
1.426
1.400
1.377
1.352
1.270
1.261
1.245
1.236
1.220
1.206
0.959
0.935
0.910
-0.000
spect, CDCl3,
Thu Dec 13 01:07:25 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
Bu-n
OHC
O
R-3a
gbj-8-170
1.00
2.15
2.07
1.00
1.02
3.10
1.04
1.18
4.43
3.21
10
8
6
4
2
0
PPM


13.938
23.099
26.926
31.446
55.191
65.019
76.579
77.000
77.422
114.242
121.081
128.551
129.344
134.313
159.177
195.790
spect, CDCl3,
Thu Dec 13 01:09:34 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 200
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
Bu-n
OHC
O
R-3a
gbj-8-170-c
PPM
0
50
100
150
200

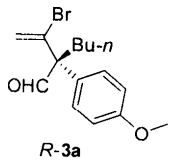
Data File D:\HPCHEM\1\DATA\GBJ\GBJ-8112.D Sample Name: gbj-8-17(

OJ-H, n-Hexane:i-PrOH =80/20, 1.20 mL/min, 230 nm

```
==========================================================================
Injection Date  : 12/14/2012 1:14:31 AM
Sample Name     : gbj-8-170                    Location :   -
Acq. Operator   : gbj
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/14/2012 1:13:20 AM by gbj
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/14/2012 1:38:16 AM by gbj
                  (modified after loading)
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```



R-**3a**

Signal 1: VWD1 A, Wavelength=230 nm

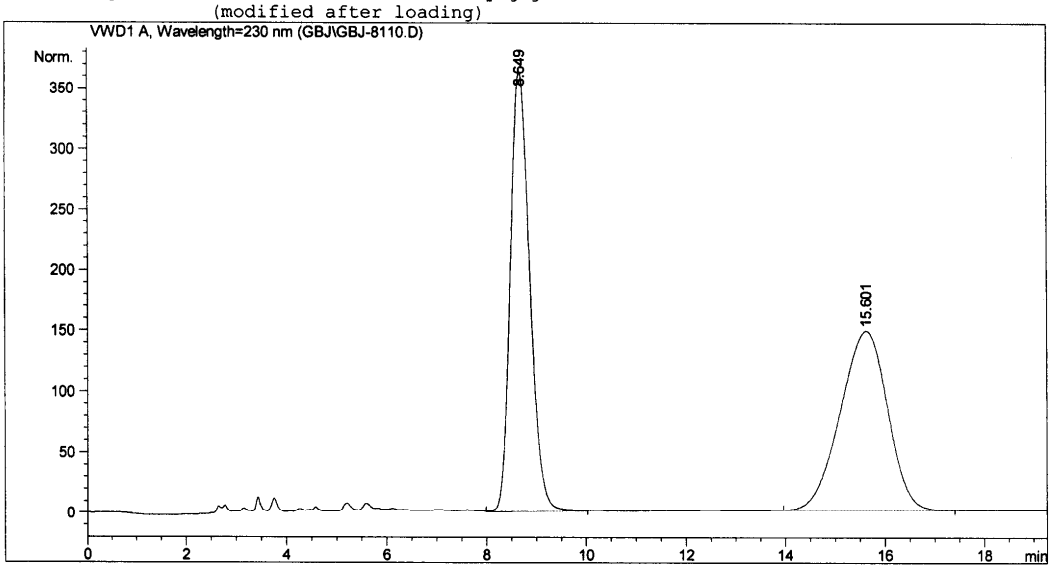
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.478	VB	0.4440	2.95991e4	1036.00488	87.4298
2	15.284	BB	0.9656	4255.60498	68.85917	12.5702
Totals :				3.38547e4	1104.86405	

```
 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

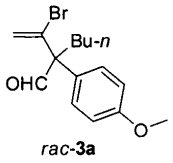
Data File D:\HPCHEM\1\DATA\GBJ\GBJ-8110.D Sample Name: gbj-8-165

OJ-H, n-Hexane:i-PrOH =80/20, 1.20 mL/min, 230 nm

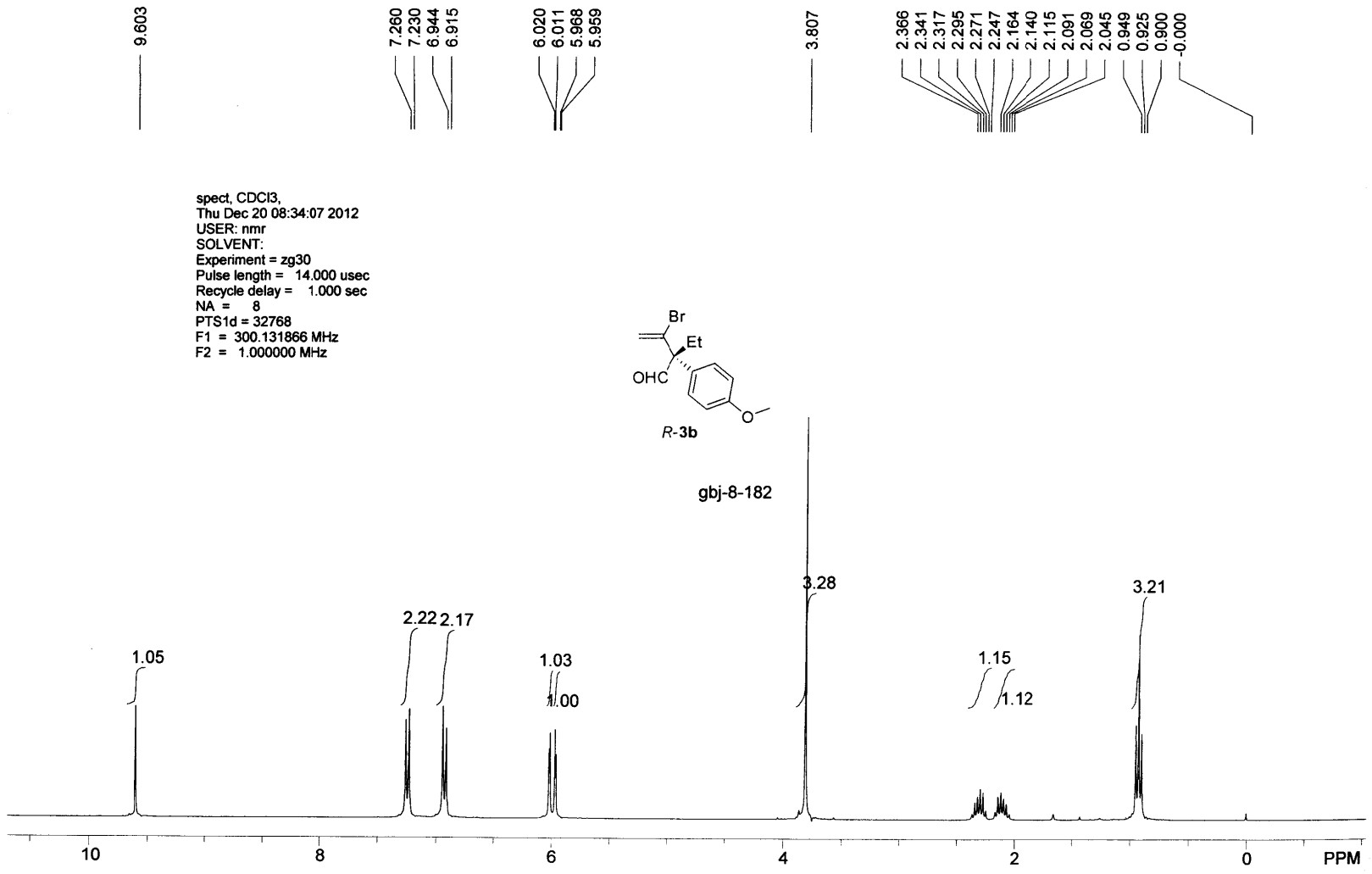
```
==========================================================================
Injection Date  : 12/14/2012 12:17:21 AM
Sample Name     : gbj-8-165                    Location :   -
Acq. Operator   : gbj
Method          : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/14/2012 12:14:19 AM by gbj
                  (modified after loading)
```

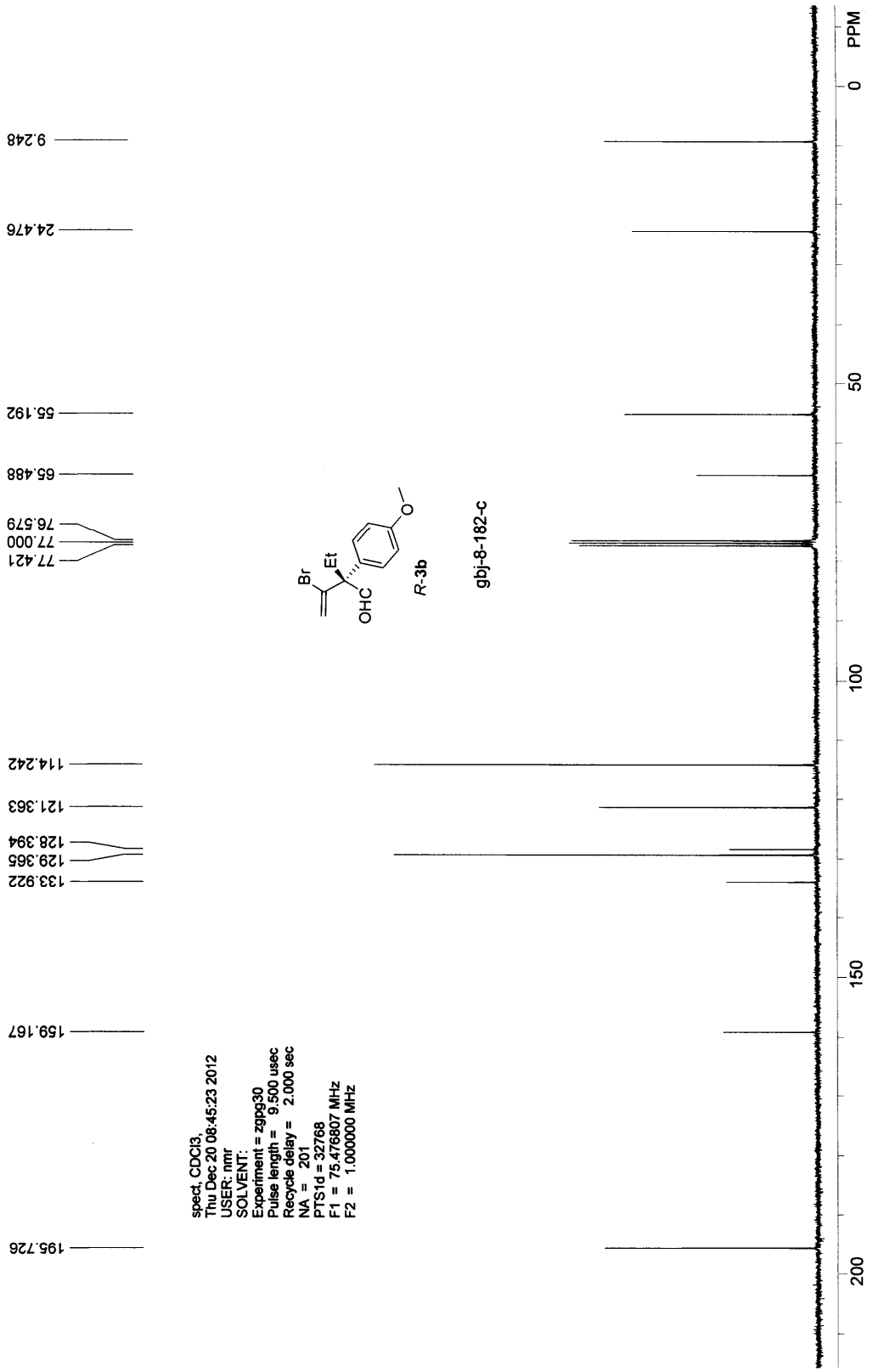



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   8.649 VB    0.4209 9830.75879  362.88113  50.1633
   2  15.601 BB    1.0416 9766.77148  147.28003  49.8367

Totals :                  1.95975e4   510.16116

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```



rac-**3a**


9.603
7.260
7.230
6.944
6.915
6.020
6.011
5.968
5.959
3.807
2.366
2.341
2.317
2.295
2.271
2.247
2.164
2.140
2.115
2.091
2.069
2.045
0.949
0.925
0.900
-0.000
spect, CDCl3,
Thu Dec 20 08:34:07 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
Et
OHC
O
R-3b
gbj-8-182
1.05
2.22
2.17
1.03
1.00
3.28
1.15
1.12
3.21
10
8
6
4
2
0
PPM

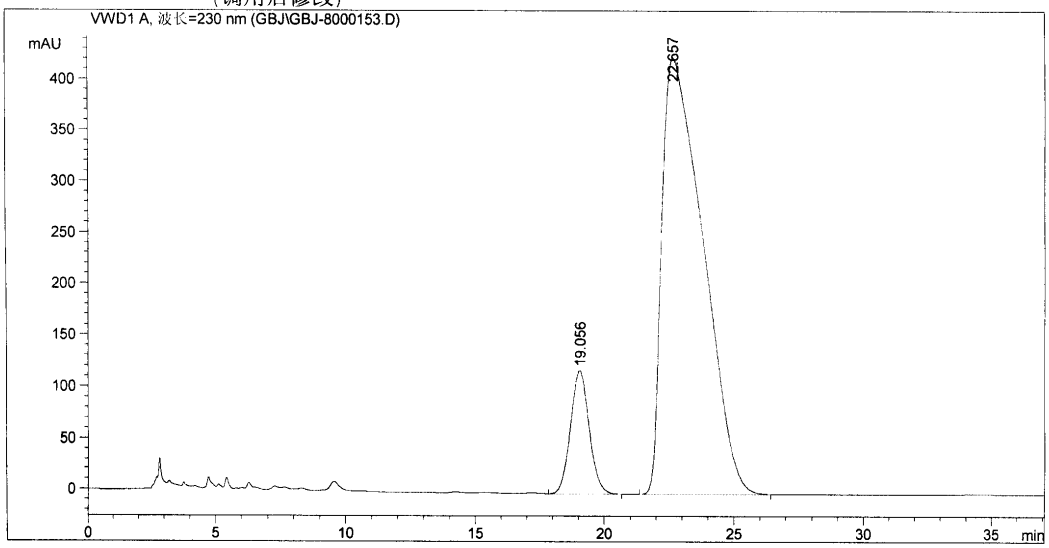

9.248
24.476
55.192
65.488
76.579
77.000
77.421
114.242
121.363
128.394
129.365
133.922
159.167
195.726
spect, CDCl3,
Thu Dec 20 08:45:23 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 201
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
Et
OHC
O
R-3b
gbj-8-182-c
PPM
0
50
100
150
200

数据文件 D:\Chem32\1\DATA\GBJ\GBJ-8000153.D
样品名: gbj-8-182

OJ-H, n-hexane/i-PrOH =80/20, 1.2 ml/min; 230 nm

==========

进样日期 : 2004-1-1 0:49:47
样品名称 : gbj-8-182 位置 : -
操作者 : gbj
仪器 : 仪器 1
方法 : D:\CHEM32\1\METHODS\JJS.M
最后修改 : 2004-1-1 0:03:08 : gbj-8-182
(调用后修改)

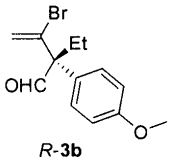


==========

面积百分比报告

==========

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标使用乘积因子和稀释因子



R-**3b**

信号 1: VWD1 A, 波长=230 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	19.056	VV	0.7636	6094.59521	120.15231	11.5248
2	22.657	BV	1.5022	4.67878e4	425.68427	88.4752

总量 : 5.28824e4 545.83657

==========

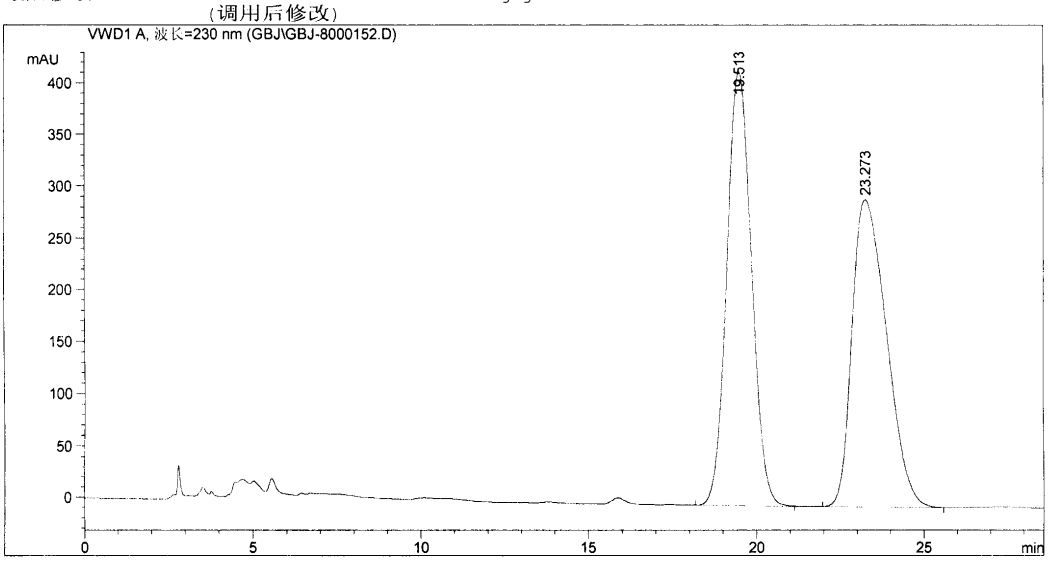
*** 报告结束 ***

数据文件　　D:\Chem32\1\DATA\GBJ\GBJ-8000152.D
样品名: gbj-8-181

OJ-H, n-hexane/i-PrOH =80/20, 1.2 ml/min; 230 nm

==========

进样日期　　: 2004-1-1 0:19:43
样品名称　　: gbj-8-181　　位置　: -
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\JJS.M
最后修改　　: 2004-1-1 0:03:08　:　gbj-8-182
(调用后修改)

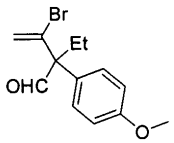


==========

面积百分比报告

==========

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子



rac-**3b**

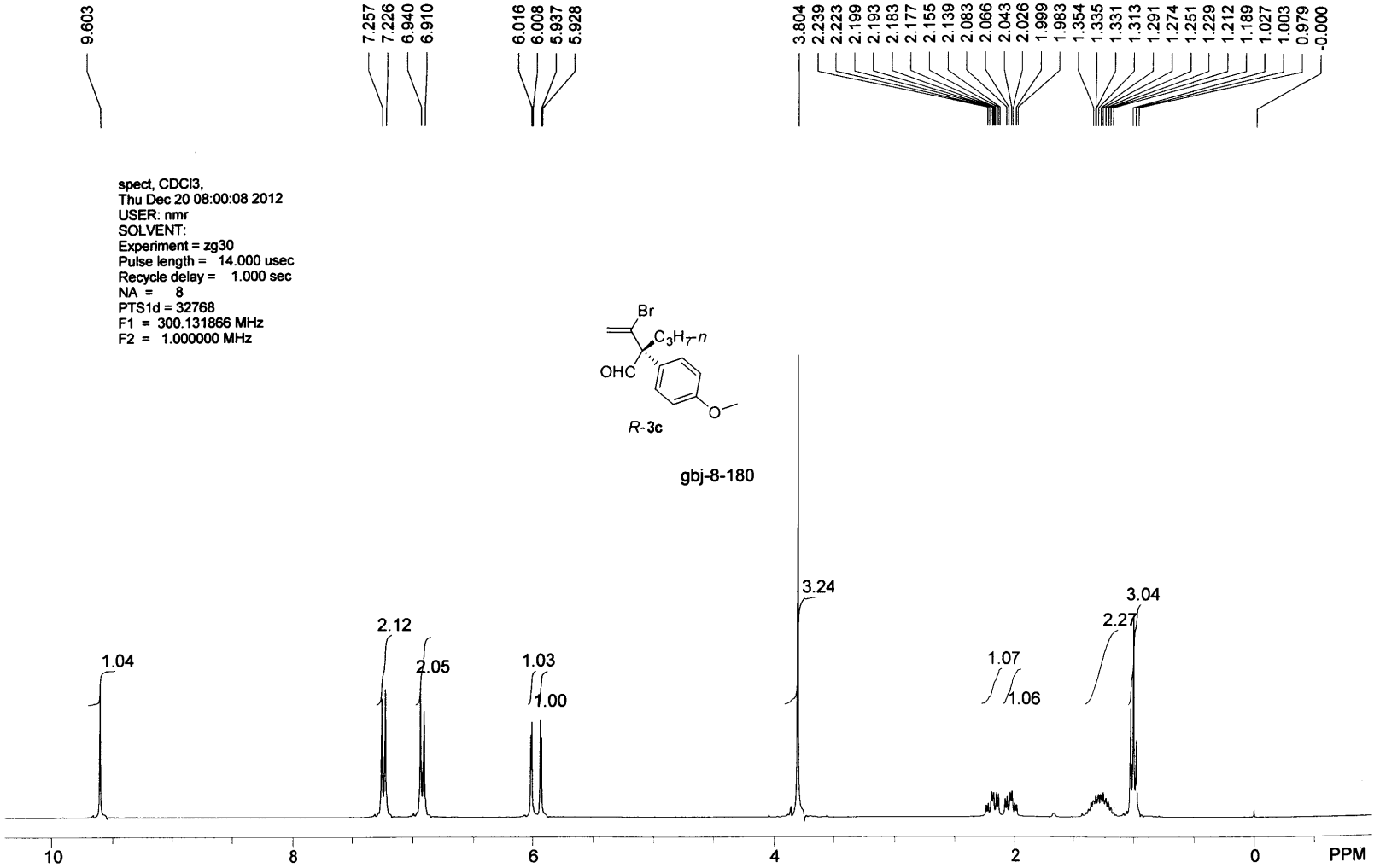
信号 1: VWD1 A, 波长=230 nm

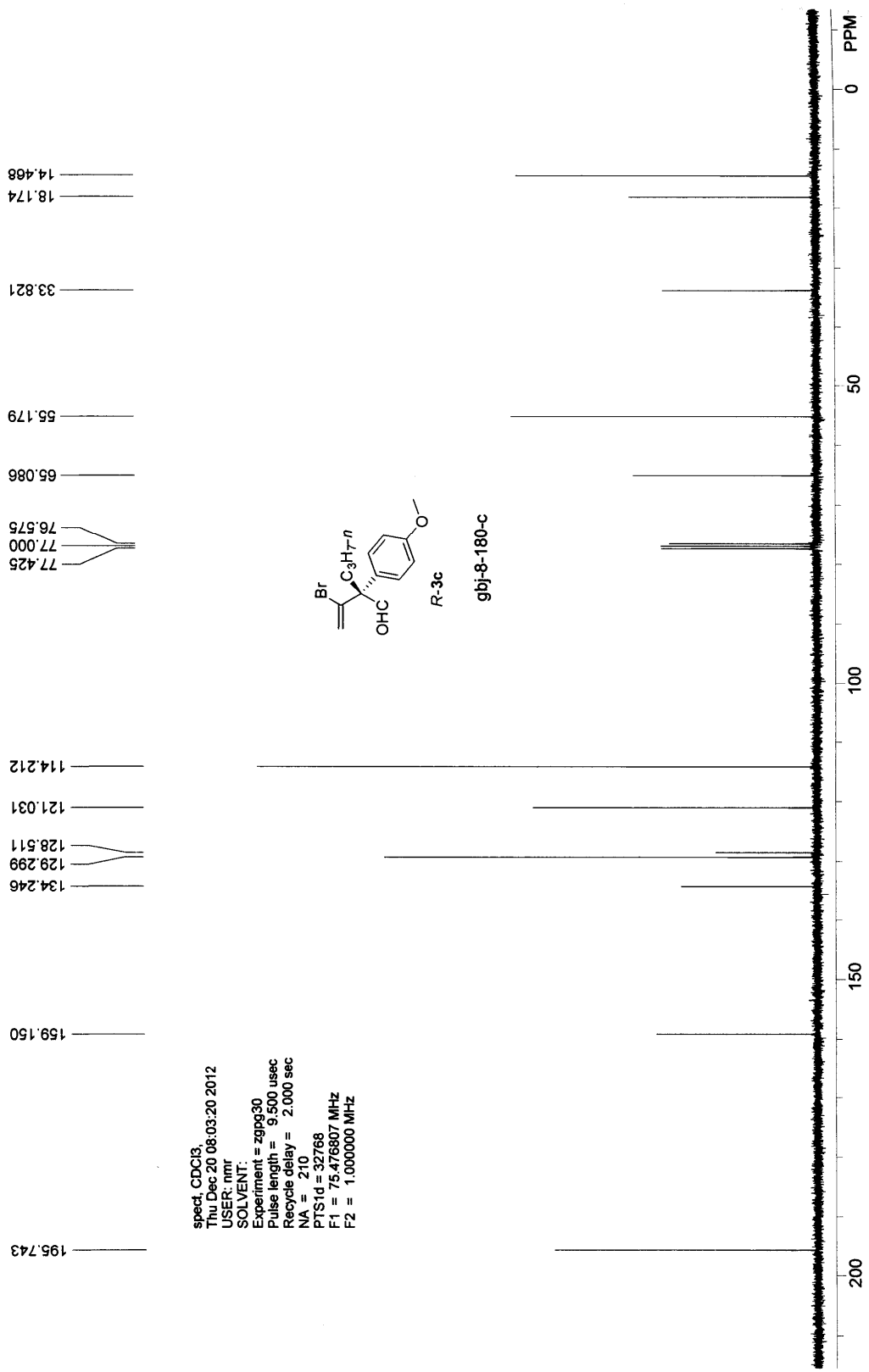
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	19.513	BV	0.7972	2.13492e4	418.72253	50.0655
2	23.273	BB	1.1222	2.12933e4	296.34747	49.9345

总量 :　　4.26425e4　　715.07001

==========

*** 报告结束 ***

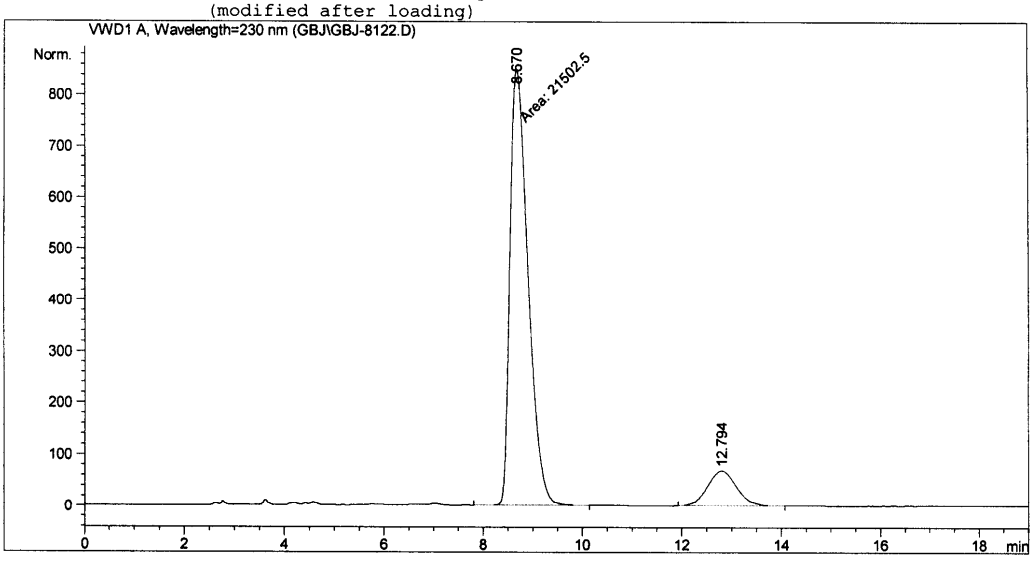

9.603
7.257
7.226
6.940
6.910
6.016
6.008
5.937
5.928
3.804
2.239
2.223
2.199
2.193
2.183
2.177
2.155
2.139
2.083
2.066
2.043
2.026
1.999
1.983
1.354
1.335
1.331
1.313
1.291
1.274
1.251
1.229
1.212
1.189
1.027
1.003
0.979
-0.000
spect, CDCl3,
Thu Dec 20 08:00:08 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
C3H7n
OHC
O
R-3c
gbj-8-180
1.04
2.12
2.05
1.03
1.00
3.24
1.07
1.06
2.27
3.04
10
8
6
4
2
0
PPM


spect, CDCl3,
Thu Dec 20 08:03:20 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 210
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
195.743
159.150
134.246
129.299
128.511
121.031
114.212
77.425
77.000
76.575
65.086
55.179
33.821
18.174
14.468
Br
C3H7-n
OHC
O
R-3c
gbj-8-180-c
PPM
0
50
100
150
200

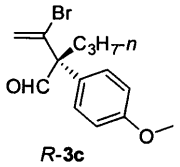
Data File D:\HPCHEM\1\DATA\GBJ\GBJ-8122.D Sample Name: gbj-8-180

OJ-H, n-Hexane:i-PrOH = 80/20, 1.2 mL/min, 230 nm

```
==========================================================================
Injection Date  : 12/19/2012 12:34:46 AM
Sample Name     : gbj-8-180                    Location :   -
Acq. Operator   : gbj
Method          : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/19/2012 12:03:18 AM by WG
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   8.670 MM    0.4215 2.15025e4   850.23608  88.3018
   2  12.794 BB    0.6468 2848.64990   68.71005  11.6982

Totals :                  2.43512e4   918.94614

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```



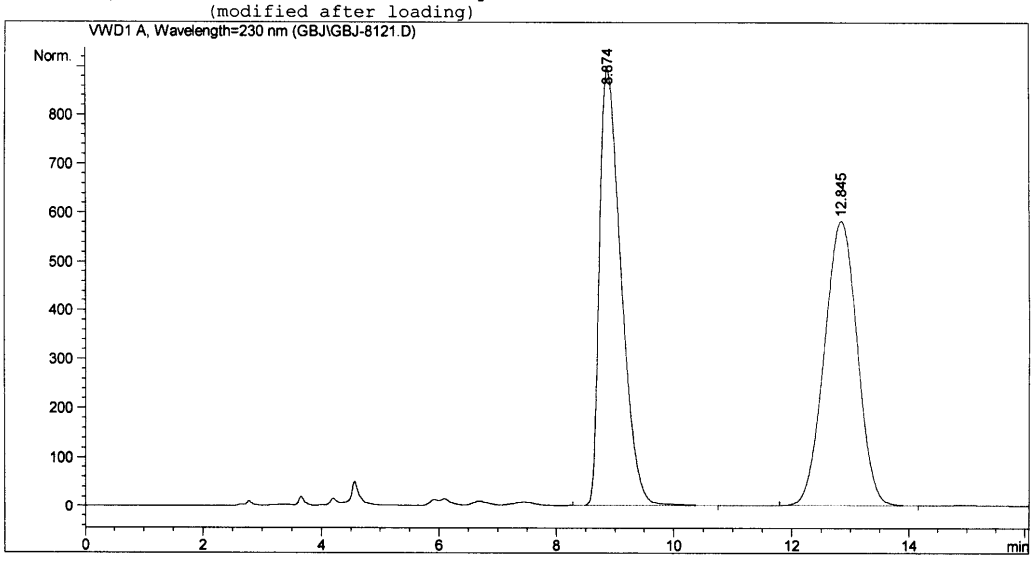
R-**3c**

Instrument 1 12/19/2012 12:55:18 AM gbj Page 1 of 1

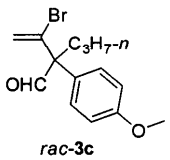
Data File D:\HPCHEM\1\DATA\GBJ\GBJ-8121.D Sample Name: gbj-8-179

OJ-H, n-Hexane:i-PrOH = 80/20, 1.2 mL/min, 230 nm

```
==========================================================================
Injection Date  : 12/19/2012 12:17:40 AM
Sample Name     : gbj-8-179                    Location :   -
Acq. Operator   : gbj
Method          : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/19/2012 12:03:18 AM by WG
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

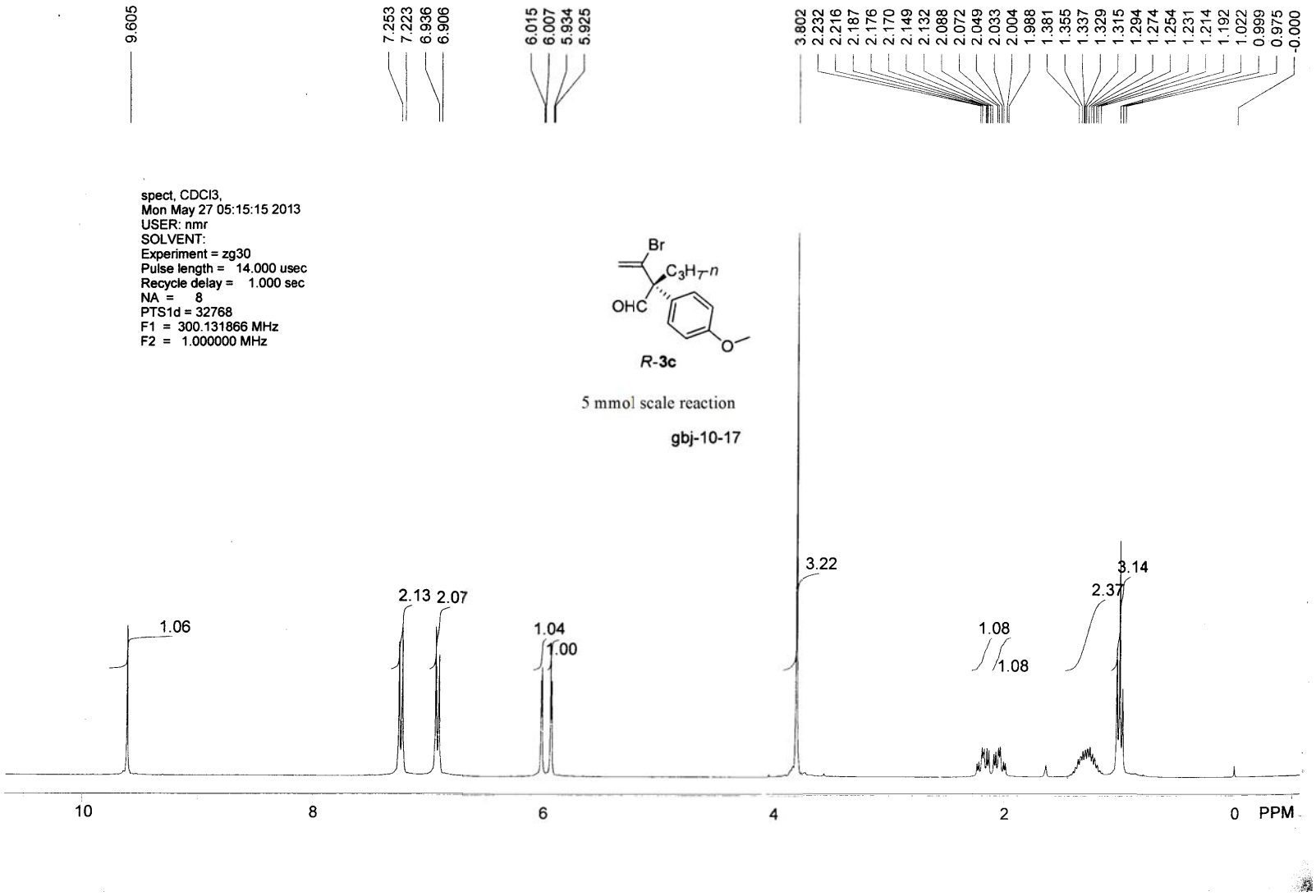


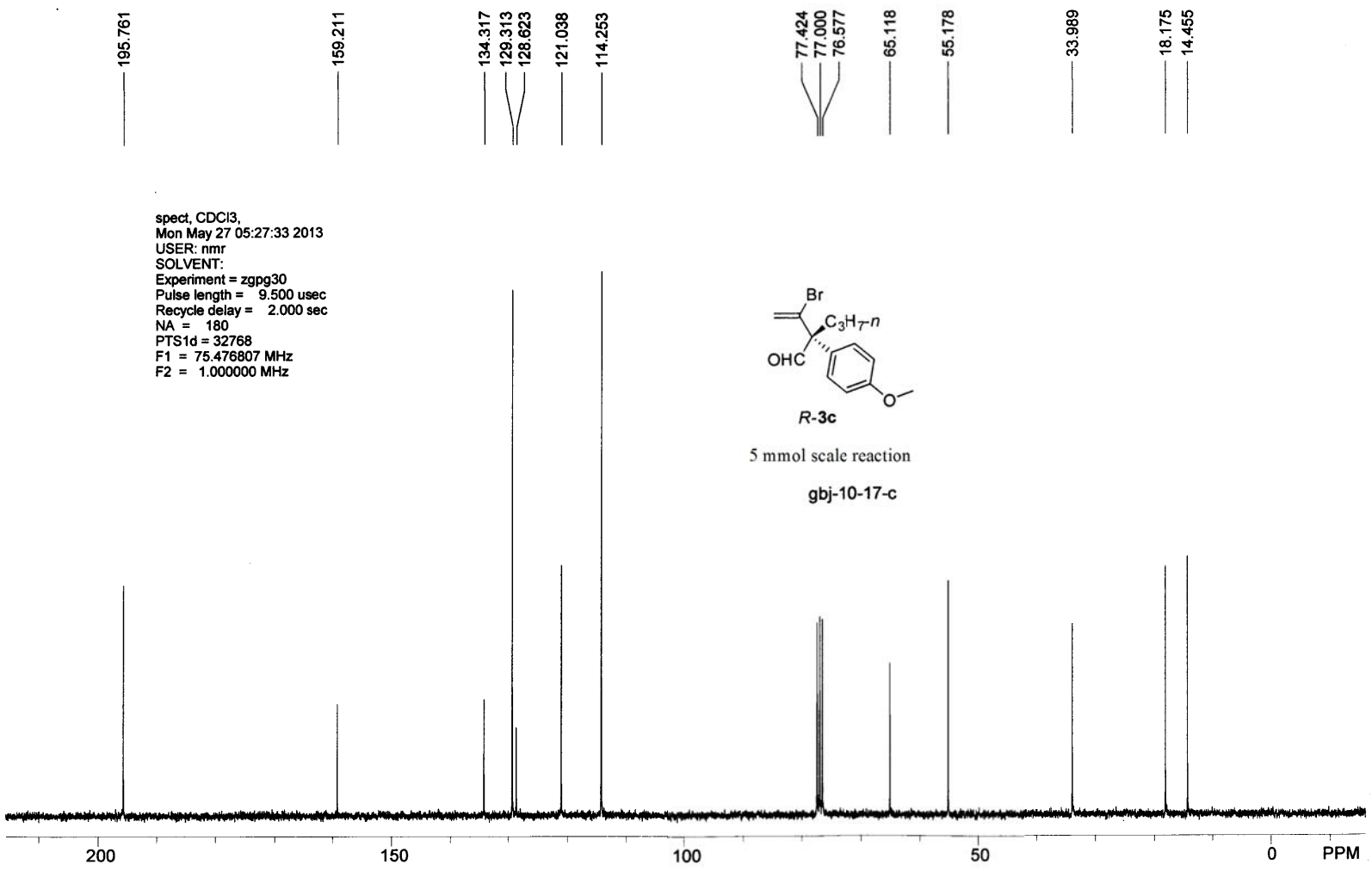
rac-**3c**

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.874	BP	0.3980	2.28660e4	892.22607	50.0165
2	12.845	BB	0.6121	2.28509e4	582.39941	49.9835
Totals :				4.57169e4	1474.62549	

```
 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```


9.605
7.253
7.223
6.936
6.906
6.015
6.007
5.934
5.925
3.802
2.232
2.216
2.187
2.176
2.170
2.149
2.132
2.088
2.072
2.049
2.033
2.004
1.988
1.381
1.355
1.337
1.329
1.315
1.294
1.274
1.254
1.231
1.214
1.192
1.022
0.999
0.975
-0.000
spect, CDCl3,
Mon May 27 05:15:15 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
C3H7-n
OHC
O
R-3c
5 mmol scale reaction
gbj-10-17
1.06
2.13
2.07
1.04
1.00
3.22
1.08
1.08
2.37
3.14
10
8
6
4
2
0
PPM

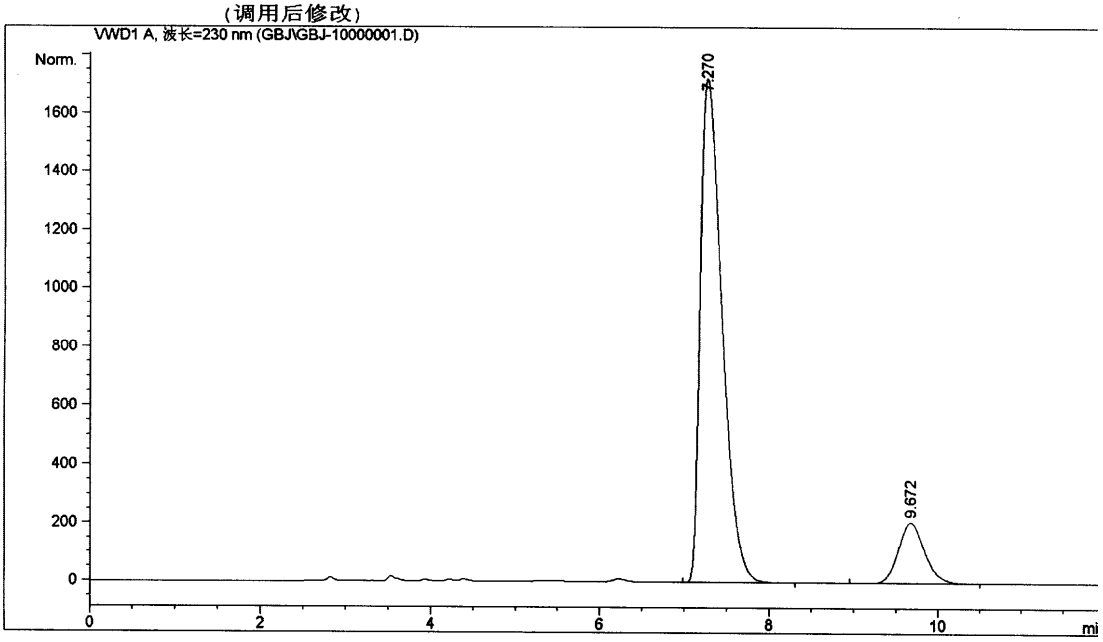

195.761
159.211
134.317
129.313
128.623
121.038
114.253
77.424
77.000
76.577
65.118
55.178
33.989
18.175
14.455
spect, CDCl3,
Mon May 27 05:27:33 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 180
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
C3H7-n
OHC
O
R-3c
5 mmol scale reaction
gbj-10-17-c
200
150
100
50
0
PPM

据文件　　D:\Chem32\1\DATA\GBJ\GBJ-10000001.D
品名: gbj-10-17

OJ-H; n-Hexane/i-PrOH =80/20; 1.2 ml/min; 230nm

==========

进样日期　　: 2013-5-27 10:53:17
样品名称　　: gbj-10-17　　位置　:　-
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2013-5-27 10:38:38　:　cd
(调用后修改)



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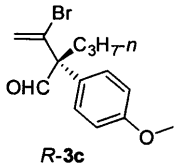
面积百分比报告

==========

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子



R-3c
5 mmol scale reaction

信号 1: VWD1 A, 波长=230 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	7.270	VB	0.2735	3.04862e4	1722.31641	86.9986
2	9.672	BB	0.3318	4555.99414	209.91898	13.0014

总量 :　　3.50422e4　1932.23538

==========

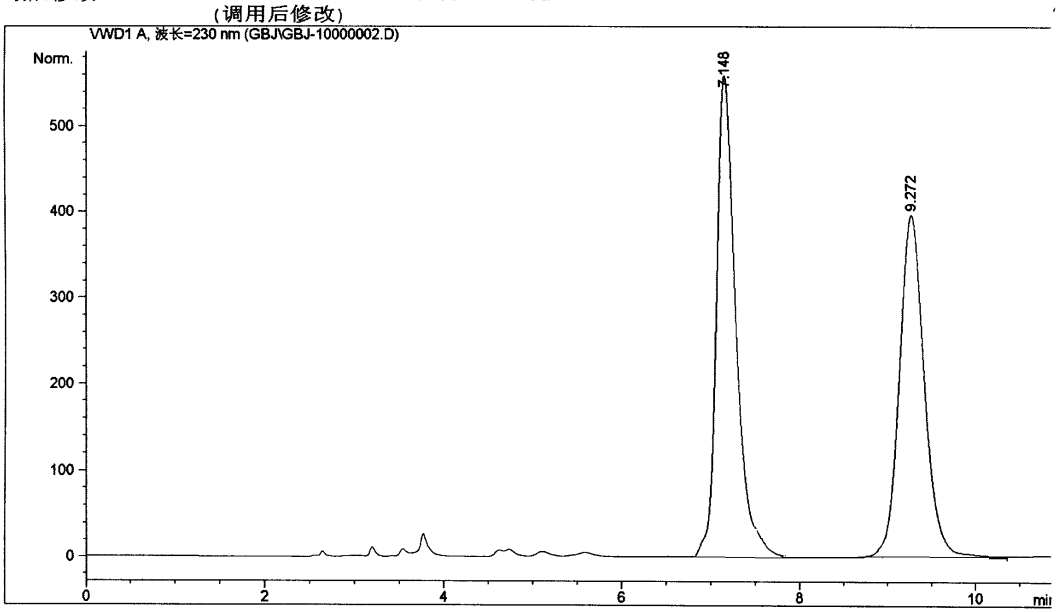
*** 报告结束 ***

数据文件　　D:\Chem32\1\DATA\GBJ\GBJ-10000002.D
羊品名: gbj-10-17-rac

OJ-H; n-Hexane/i-PrOH =80/20; 1.2 ml/min; 230nm

==========

进样日期	: 2013-5-27 11:07:53		
样品名称	: gbj-10-17-rac	位置	: -
操作者	: gbj		
仪器	: 仪器 1		
方法	: D:\CHEM32\1\METHODS\ZYY_LC.M		
最后修改	: 2013-5-27 10:38:38 : cd		
	(调用后修改)		



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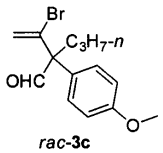
面积百分比报告

==========

排序	:	信号
乘积因子	:	1.0000
稀释因子	:	1.0000

内标使用乘积因子和稀释因子



rac-3c

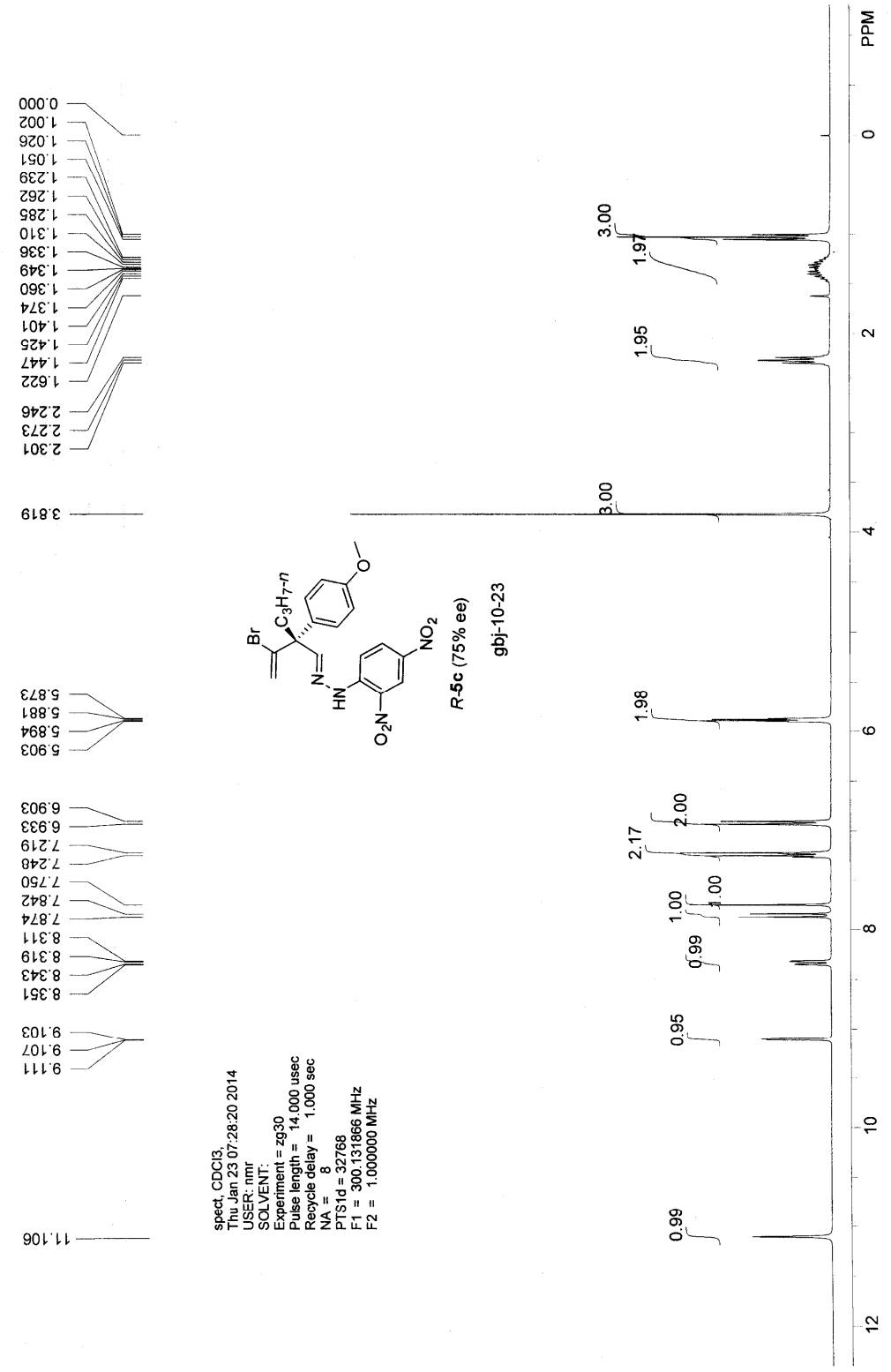
信号 1: VWD1 A, 波长=230 nm

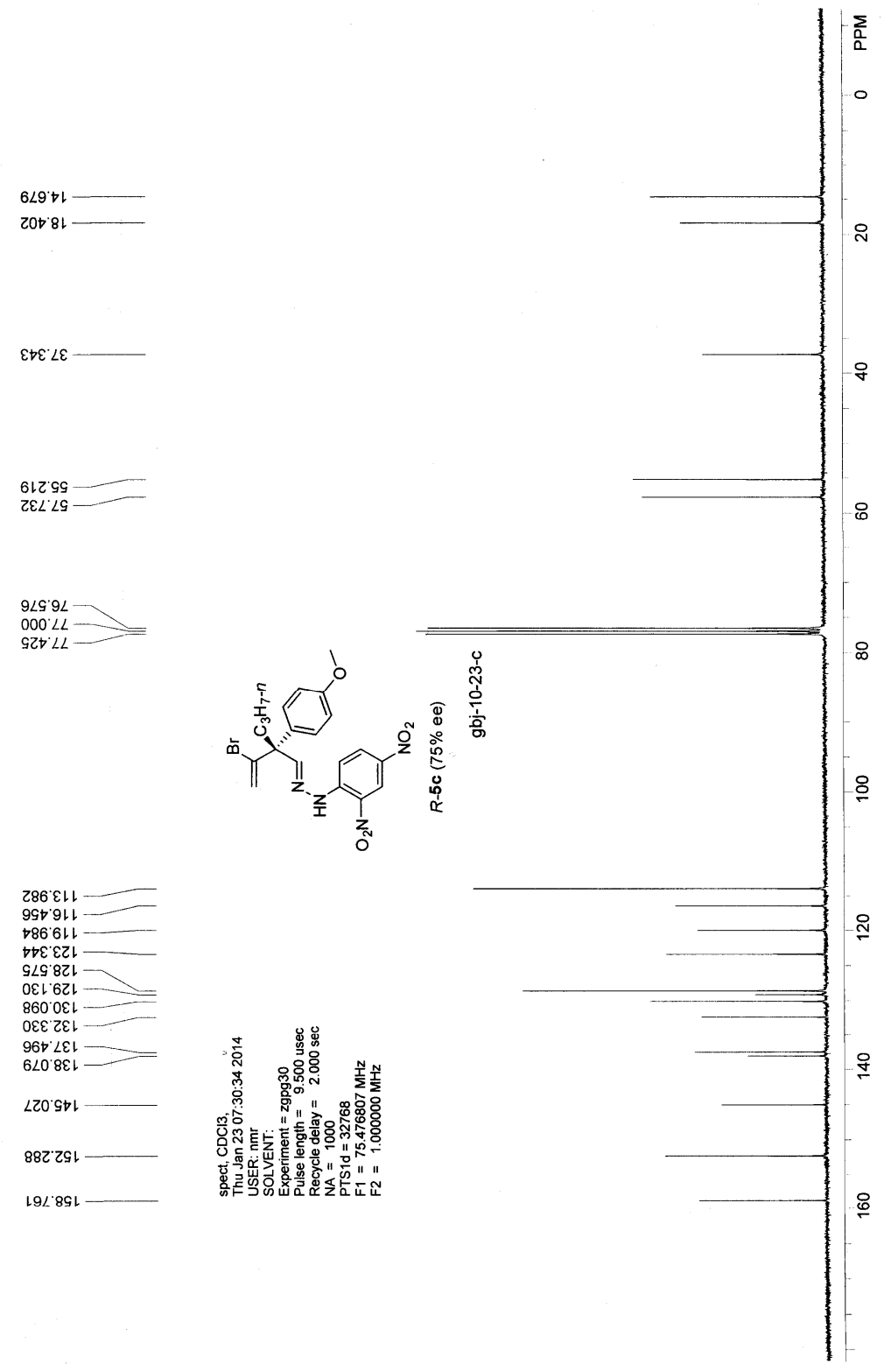
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	7.148	VV	0.2222	8204.66016	559.11871	50.3720
2	9.272	VV	0.3088	8083.48535	397.21329	49.6280

总量 :　　1.62881e4　　956.33200

==========

*** 报告结束 ***


spect, CDCl3,
Thu Jan 23 07:28:20 2014
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-10-23
R-5c (75% ee)
PPM

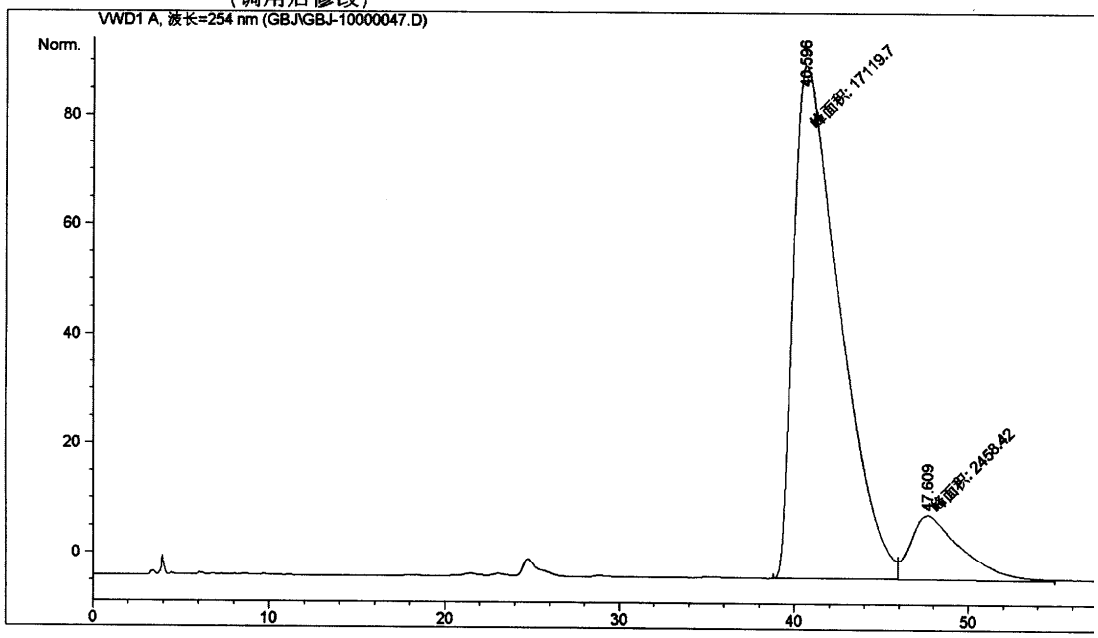

14.679
18.402
37.343
55.219
57.732
76.576
77.000
77.425
113.982
116.456
119.984
123.344
128.575
129.130
130.098
132.330
137.496
138.079
145.027
152.288
158.761
spect, CDCl3,
Thu Jan 23 07:30:34 2014
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 1000
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
gbj-10-23-c
R-5c (75% ee)
Br
C3H7-n
O
N
HN
NO2
O2N
0
20
40
60
80
100
120
140
160
PPM

据文件　　D:\Chem32\1\DATA\GBJ\GBJ-10000047.D
品名: gbj-10-23

OD-H, n-Hexane/i-PrOH =95/5, 1.0 ml/min; 254 nm

==========

进样日期	: 2013-6-11 20:03:23		
样品名称	: gbj-10-23	位置	: -
操作者	: gbj		
仪器	: 仪器 1		
方法	: D:\CHEM32\1\METHODS\ZYY_LC.M		
最后修改	: 2013-6-11 19:05:21 : gbj		
	(调用后修改)		



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面积百分比报告

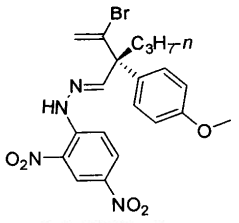
==========

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子

信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	40.596	MF	3.0456	1.71197e4	93.68555	87.4430
2	47.609	MM	3.4650	2458.42285	11.82493	12.5570

总量 :　　1.95781e4　　105.51049



R-5c (75% ee)

==========

*** 报告结束 ***

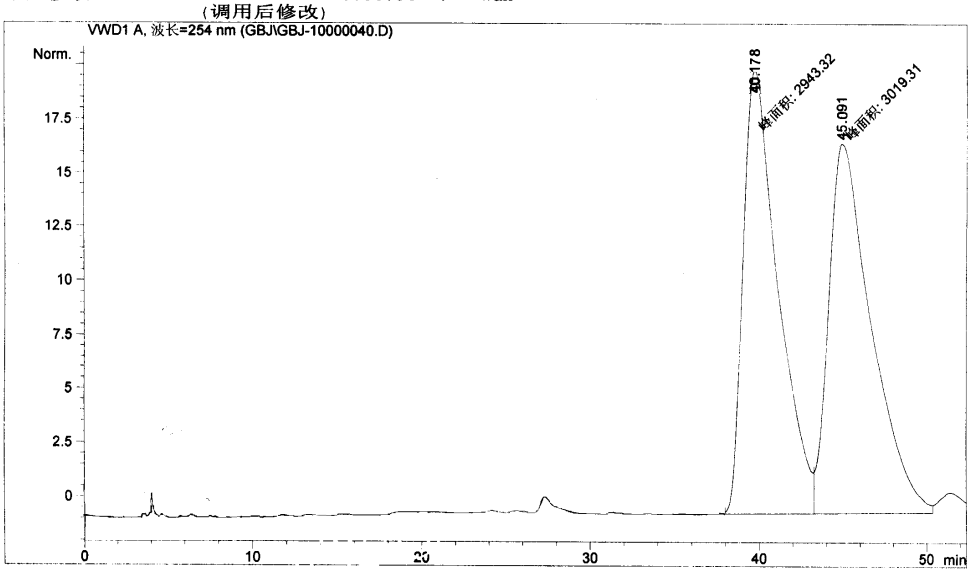
1 2013-6-11 21:03:38 gbj　　页 1 / 1

数据文件　　D:\CHEM32\1\DATA\GBJ\GBJ-10000040.D
样品名: gbj-10-23-rac

OD-H,n-Hexane/i-PrOH =70/30, 1.0 ml/min; 254 nm

==========

进样日期　　: 2013-6-11 13:56:47
样品名称　　: gbj-10-23-rac　　位置　:　-
操作者　　: gbj
仪器　　: 仪器 1
采集方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2013-6-11 13:50:34　:　gbj
(调用后修改)
分析方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2004-1-1 0:06:54　:　wdx
(调用后修改)



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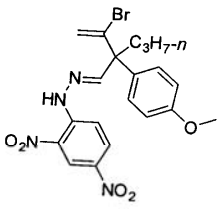
面积百分比报告

==========

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子

信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	40.178	MF	2.3927	2943.32153	20.50200	49.3628
2	45.091	FM	2.9376	3019.30957	17.13020	50.6372
总量 :				5962.63110	37.63220	

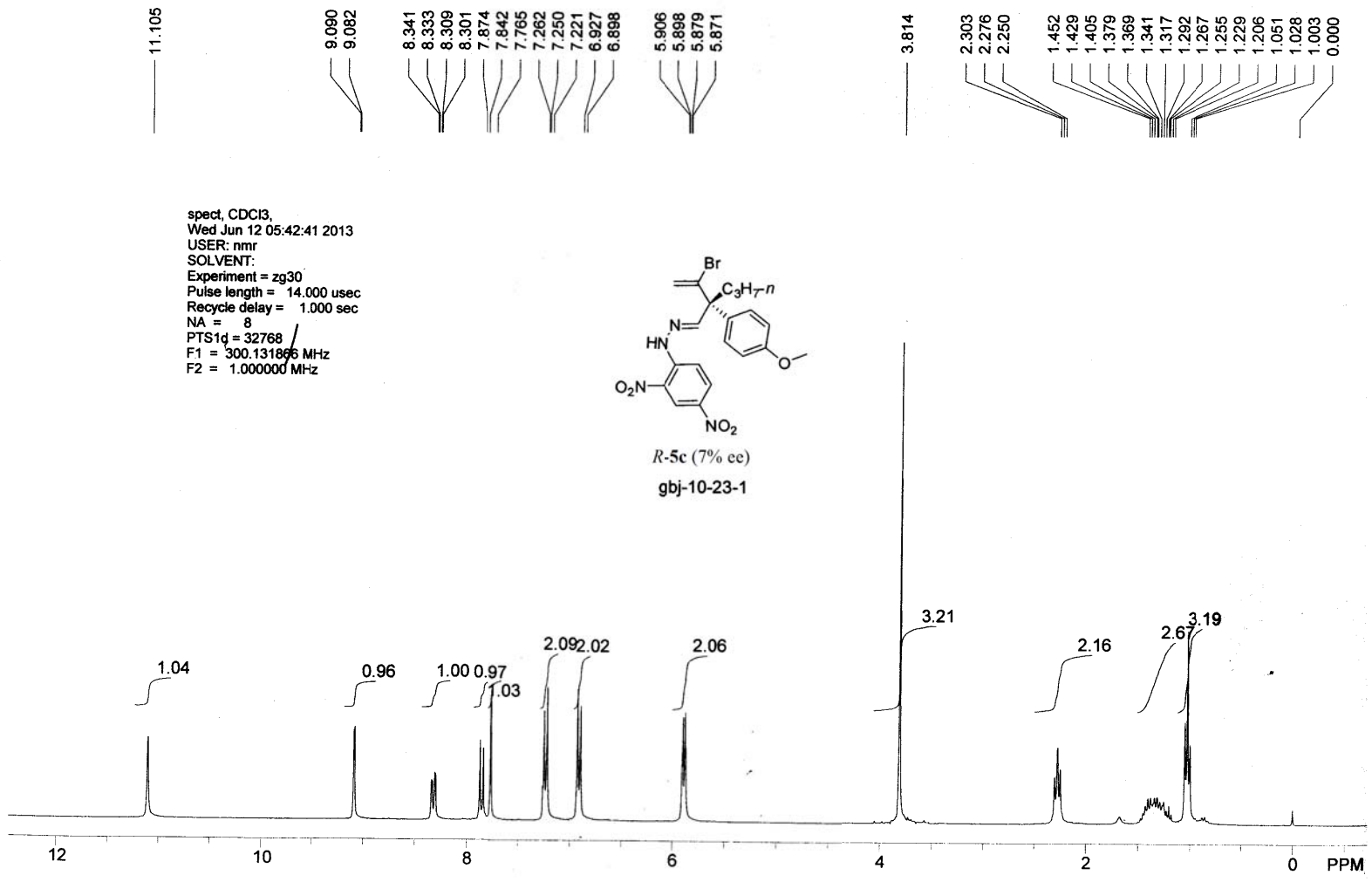


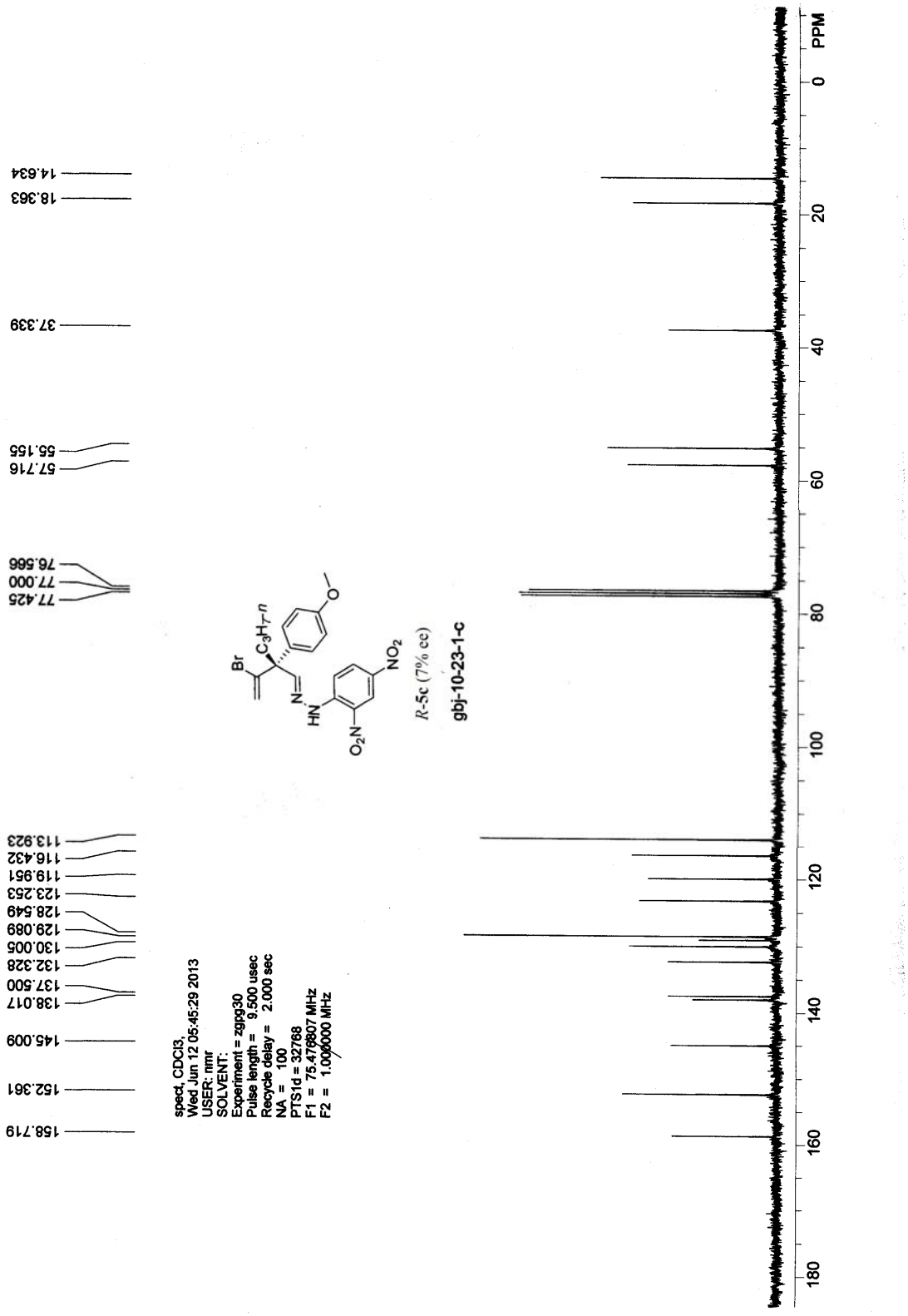
rac-5c

==========

*** 报告结束 ***

仪器 1 2013-6-11 14:48:57 gbj　　页 1 / 1


11.105
9.090
9.082
8.341
8.333
8.309
8.301
7.874
7.842
7.765
7.262
7.250
7.221
6.927
6.898
5.906
5.898
5.879
5.871
3.814
2.303
2.276
2.250
1.452
1.429
1.405
1.379
1.369
1.341
1.317
1.292
1.267
1.255
1.229
1.206
1.051
1.028
1.003
0.000
spect, CDCl3,
Wed Jun 12 05:42:41 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
C3H7-n
N
HN
O2N
NO2
O
R-5c (7% ee)
gbj-10-23-1
1.04
0.96
1.00
0.97
1.03
2.09
2.02
2.06
3.21
2.16
2.67
3.19
12
10
8
6
4
2
0
PPM

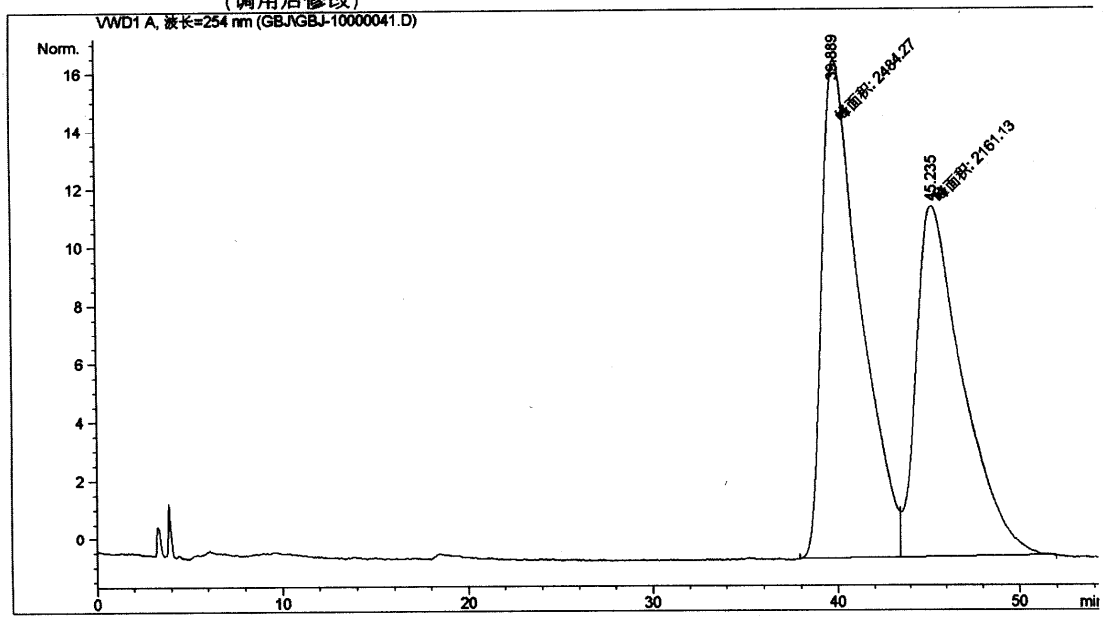

14.634
18.363
37.339
55.155
57.716
76.566
77.000
77.425
113.923
116.432
119.961
123.253
128.549
129.089
130.005
132.328
137.500
138.017
145.009
152.361
158.719
spect, CDCl3,
Wed Jun 12 05:45:29 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 100
PTS1d = 32768
F1 = 75.478807 MHz
F2 = 1.000000 MHz
Br
C_3H_{7-n}
O
N
HN
NO_2
O_2N
R-5c (7% ee)
gbj-10-23-1-c
PPM
0
20
40
60
80
100
120
140
160
180

文件　　D:\Chem32\1\DATA\GBJ\GBJ-10000041.D
名：gbj-10-23 jiejin

OD-H,n-Hexane/i-PrOH =95/5, 1.0 ml/min; 254 nm

==========

进样日期　　: 2013-6-10 15:46:06
样品名称　　: gbj-10-23 jiejin　　[illegible] :
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2013-6-10 14:50:34 : gbj
(调用后修改)

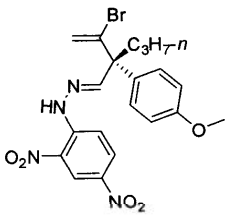


==========

面积百分比报告

==========

排序　　: 信号
乘积因子　　: 1.0000
稀释因子　　: 1.0000
内标使用乘积因子和稀释因子



R-5c (7% ee)

信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	39.889	MF	2.4175	2484.27271	17.12708	53.4781
2	45.235	FM	2.9932	2161.13184	12.03362	46.5219

总量 :　　4645.40454　　29.16069

==========

*** 报告结束 ***

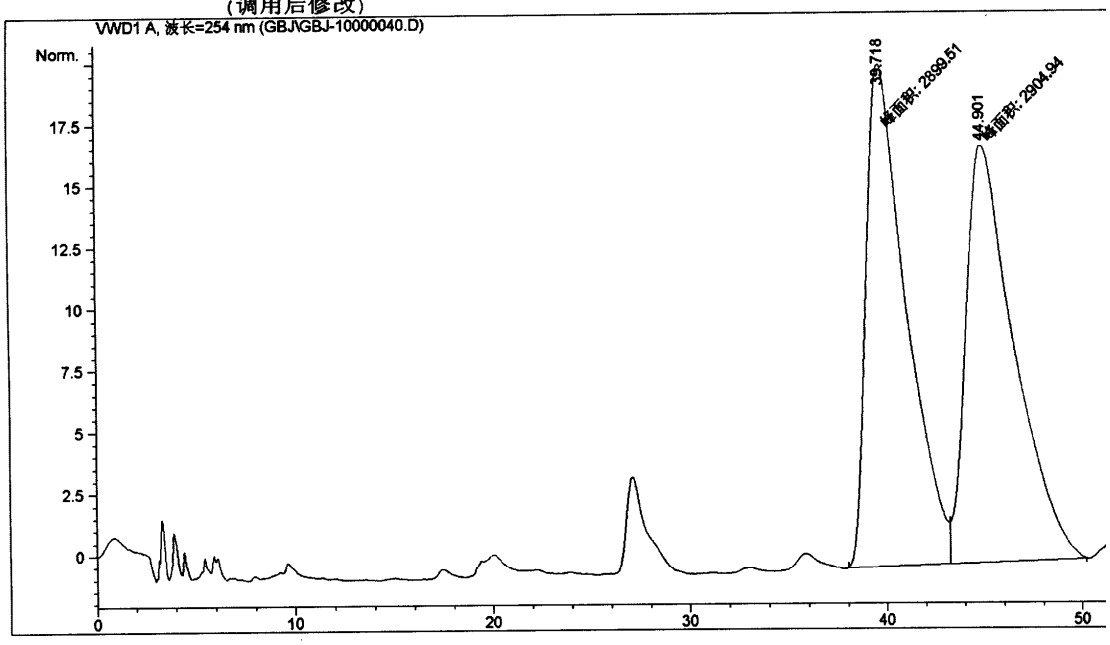
器 1 2013-6-10 16:41:50 gbj　　页 1 / 1

文件　　D:\Chem32\1\DATA\GBJ\GBJ-10000040.D
名：gbj-10-23-rac

OD-H,n-Hexane/i-PrOH =70/30, 1.0 ml/min; 254 nm

==========

进样日期　　: 2013-6-10 14:51:47
样品名称　　: gbj-10-23-rac　　位置　:　-
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2013-6-10 14:50:34　:　gbj
(调用后修改)

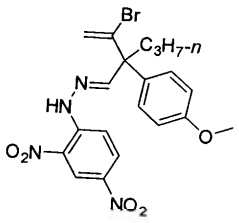


==========

面积百分比报告

==========

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子



rac-5c

信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	39.718	MF	2.3709	2899.51367	20.38296	49.9532
2	44.901	FM	2.8643	2904.94263	16.90300	50.0468
总量 :				5804.45630	37.28596	

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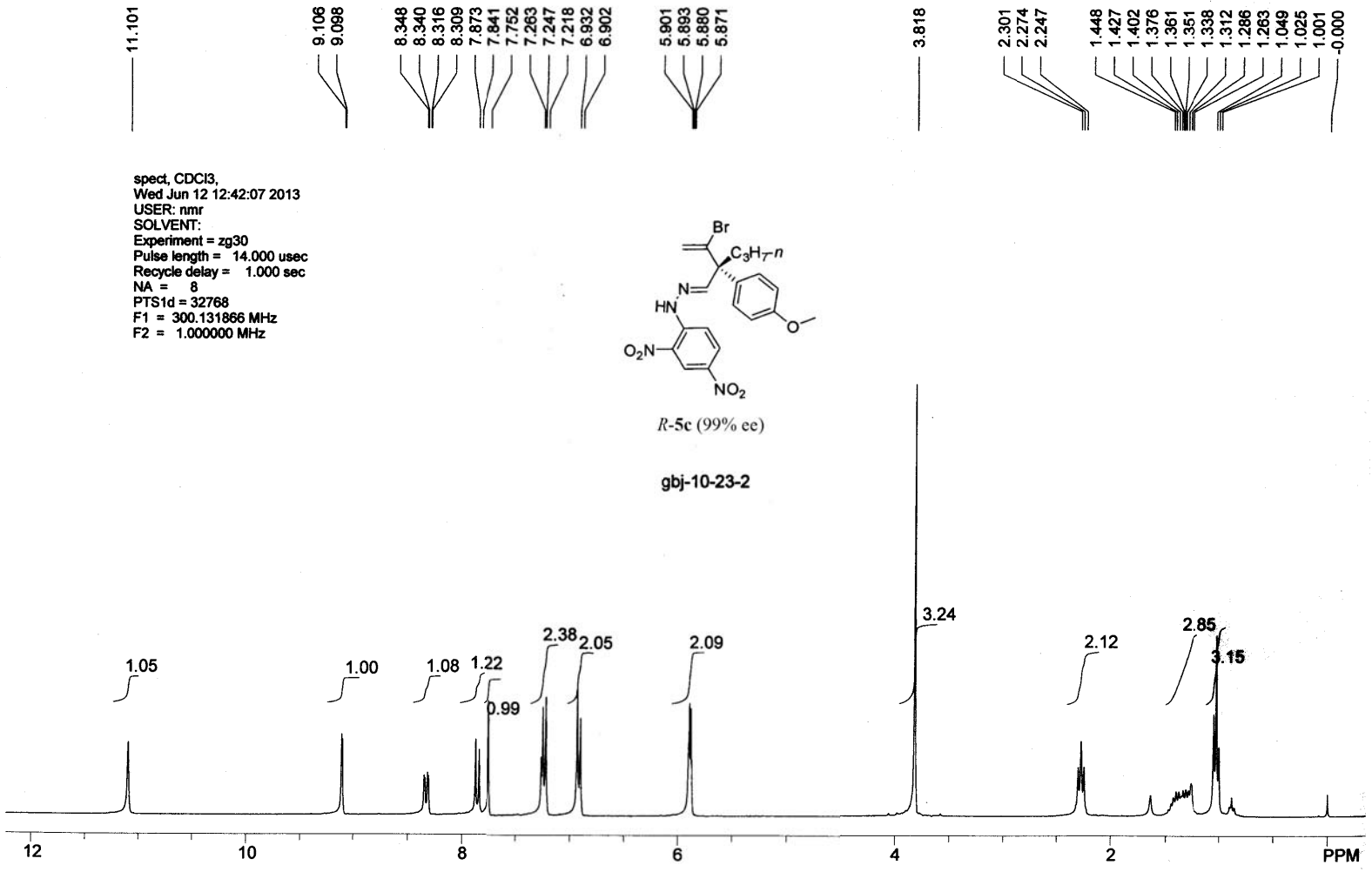
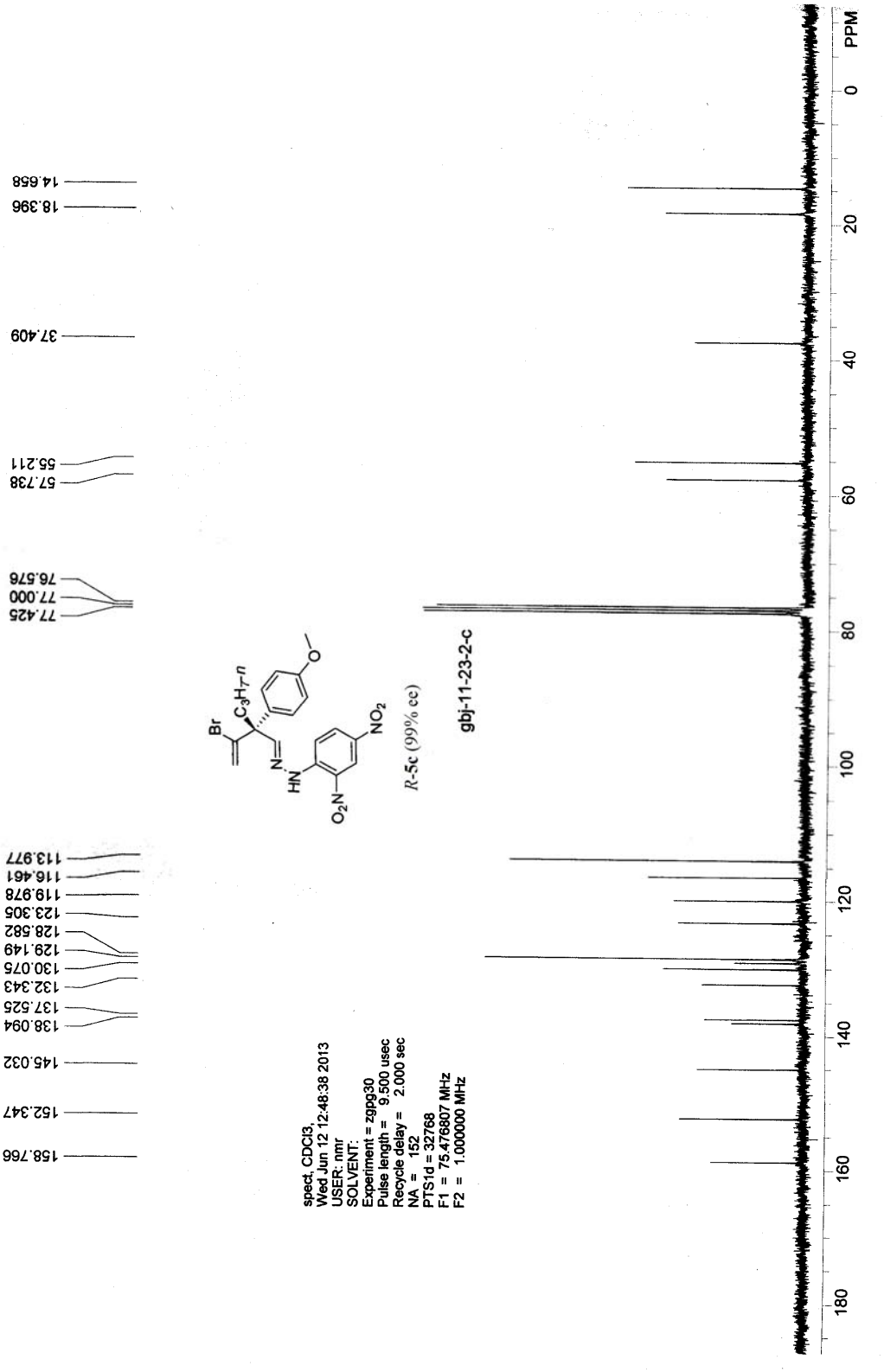
*** 报告结束 ***


11.101
9.106
9.098
8.348
8.340
8.316
8.309
7.873
7.841
7.752
7.263
7.247
7.218
6.932
6.902
5.901
5.893
5.880
5.871
3.818
2.301
2.274
2.247
1.448
1.427
1.402
1.376
1.361
1.351
1.338
1.312
1.286
1.263
1.049
1.025
1.001
-0.000
spect, CDCl3,
Wed Jun 12 12:42:07 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
C3H7-n
N
HN
O
O2N
NO2
R-5c (99% ee)
gbj-10-23-2
1.05
1.00
1.08
1.22
0.99
2.38
2.05
2.09
3.24
2.12
2.85
3.15
12
10
8
6
4
2
PPM

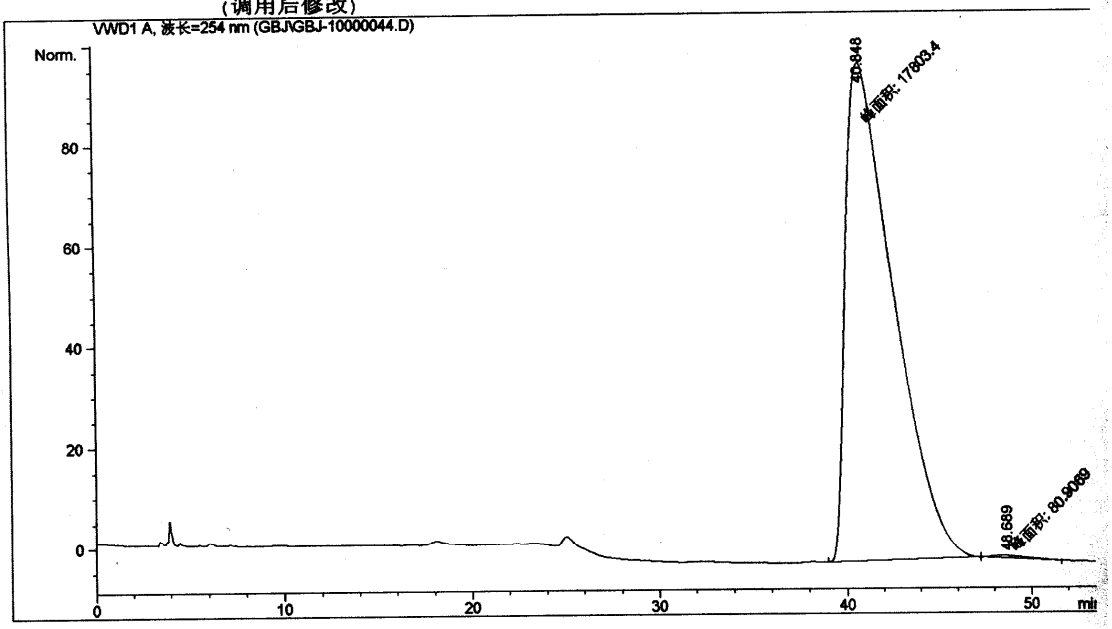

158.766
152.347
145.032
138.094
137.525
132.343
130.075
129.149
128.582
123.305
119.978
116.461
113.977
77.425
77.000
76.576
57.738
55.211
37.409
18.396
14.658
PPM
0
20
40
60
80
100
120
140
160
180
gbj-11-23-2-c
R-5c (99% ee)
spect, CDCl3,
Wed Jun 12 12:48:38 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 152
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz

文件　　D:\Chem32\1\DATA\GBJ\GBJ-10000044.D
名：gbj-10-23muye

OD-H, n-Hexane/i-PrOH =95/5, 1.0 ml/min; 254 nm

==========

进样日期　　: 2013-6-11 15:02:24
样品名称　　: gbj-10-23muye　　位置　:　-
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2013-6-11 14:01:37　:　gbj
(调用后修改)



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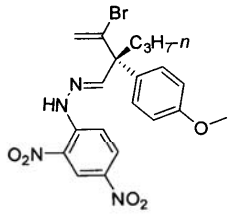
面积百分比报告

==========

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子

信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	40.848	MM	3.0004	1.78034e4	98.89624	99.5476
2	48.689	MM	2.3683	80.90693	5.69382e-1	0.4524
总量 :				1.78843e4		99.46562



R-5c (99% ee)

==========

*** 报告结束 ***

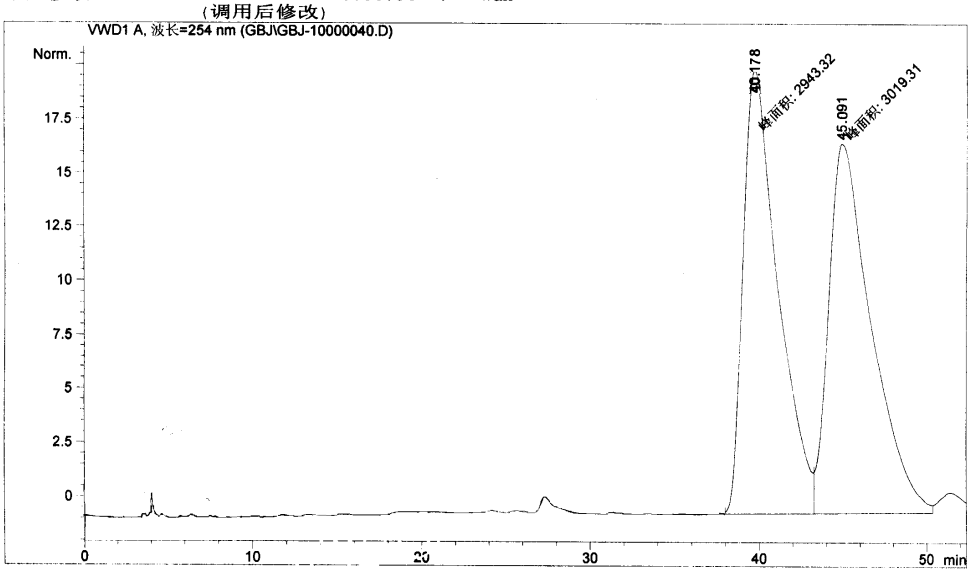
器 1 2013-6-11 15:58:23　gbj　　页　1 / 1

数据文件　　D:\CHEM32\1\DATA\GBJ\GBJ-10000040.D
样品名: gbj-10-23-rac

OD-H,n-Hexane/i-PrOH =70/30, 1.0 ml/min; 254 nm

==========

进样日期　　: 2013-6-11 13:56:47
样品名称　　: gbj-10-23-rac　　位置　:　-
操作者　　: gbj
仪器　　: 仪器 1
采集方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2013-6-11 13:50:34　:　gbj
(调用后修改)
分析方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2004-1-1 0:06:54　:　wdx
(调用后修改)



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面积百分比报告

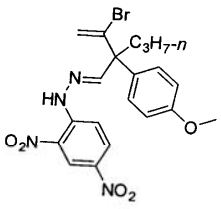
==========

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子

信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	40.178	MF	2.3927	2943.32153	20.50200	49.3628
2	45.091	FM	2.9376	3019.30957	17.13020	50.6372

总量 :　　5962.63110　　37.63220



rac-5c

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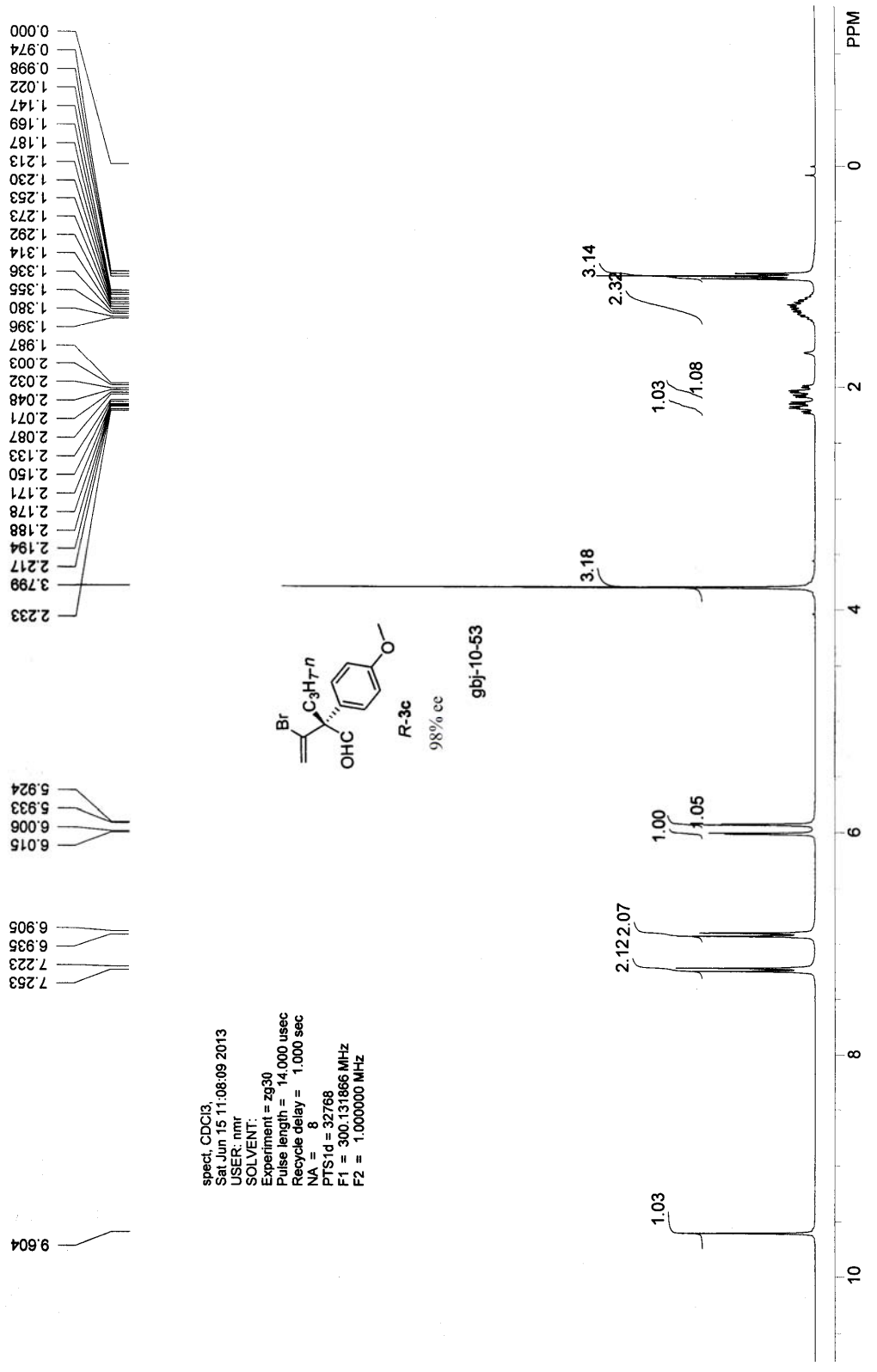
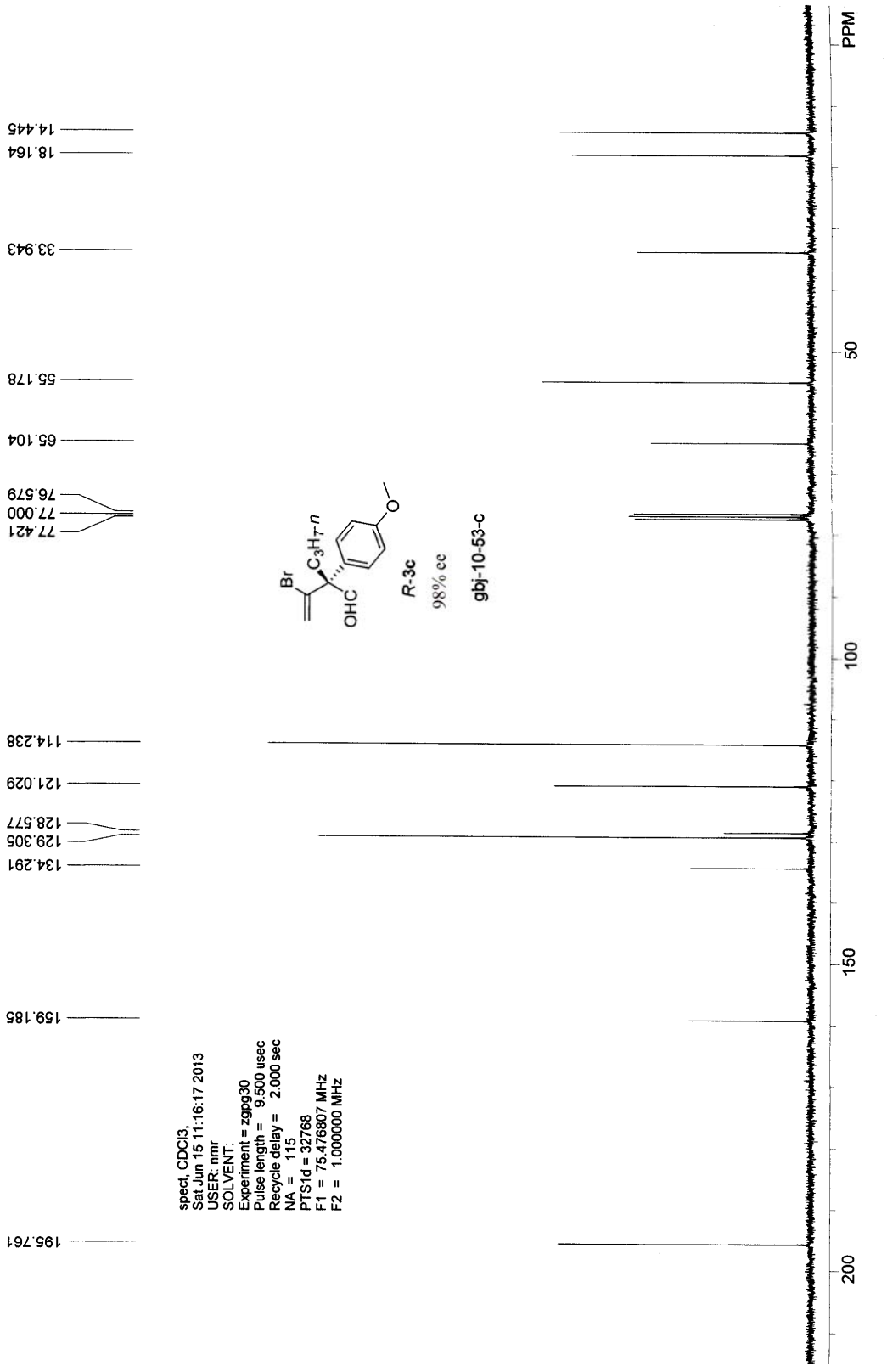
*** 报告结束 ***

仪器 1 2013-6-11 14:48:57 gbj　　页 1 / 1

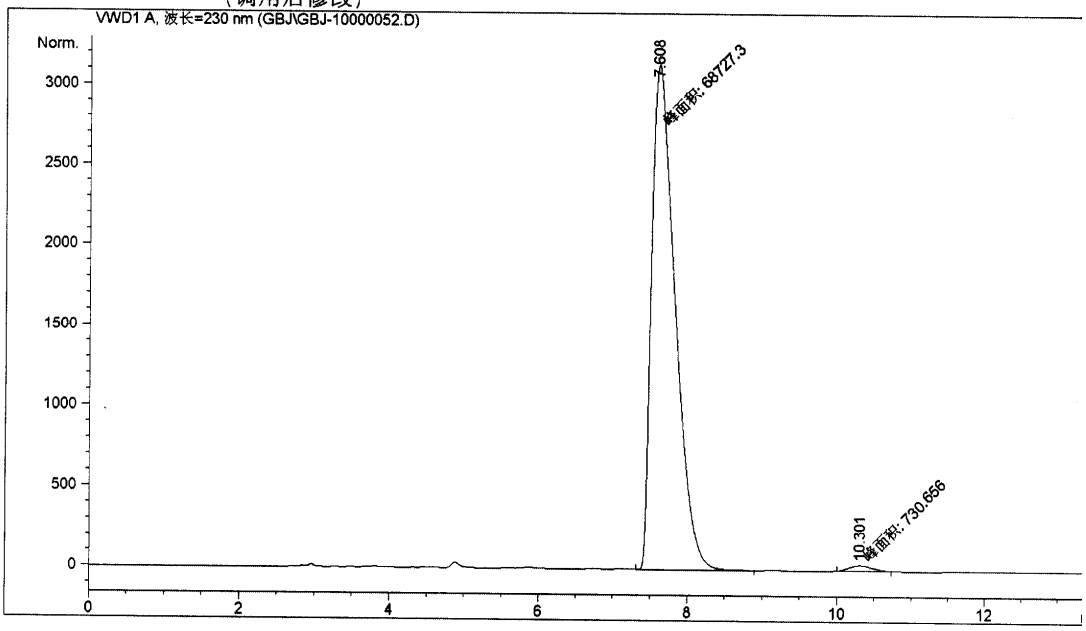

spect, CDCl3,
Sat Jun 15 11:08:09 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-10-53
R-3c
98% ee
Br
OHC
C3H7-n
O
9.604
7.253
7.223
6.935
6.905
6.015
6.006
5.933
5.924
3.799
3.180
2.233
2.217
2.194
2.188
2.178
2.171
2.150
2.133
2.087
2.071
2.048
2.032
2.003
1.987
1.396
1.380
1.355
1.336
1.314
1.292
1.273
1.253
1.230
1.213
1.187
1.169
1.147
1.022
0.998
0.974
0.000
1.03
2.12
2.07
1.00
1.05
1.03
1.08
3.14
2.32
PPM
10
8
6
4
2
0


spect, CDCl3,
Sat Jun 15 11:16:17 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 115
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
195.761
159.185
134.291
129.305
128.577
121.029
114.238
77.421
77.000
76.579
65.104
55.178
33.943
18.164
14.445
Br
C3H7-n
OHC
O
R-3c
98% ee
gbj-10-53-c
PPM
200
150
100
50

数据文件　　D:\Chem32\1\DATA\GBJ\GBJ-10000052.D
样品名: gbj-10-53

OJ-H, n-Hexane/i-PrOH =80/20, 1.20 ml/min; 230 nm

进样日期　　: 2013-6-15 15:43:59
样品名称　　: gbj-10-53　　位置　:　-
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2013-6-15 15:33:23　:　lxj
(调用后修改)



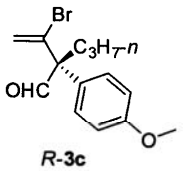
面积百分比报告

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子

信号 1: VWD1 A, 波长=230 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	7.608	MM	0.3646	6.87273e4	3141.51123	98.9481
2	10.301	MM	0.3568	730.65613	34.12573	1.0519

总量 :　　6.94580e4　3175.63696



R-3c
98% ee

*** 报告结束 ***

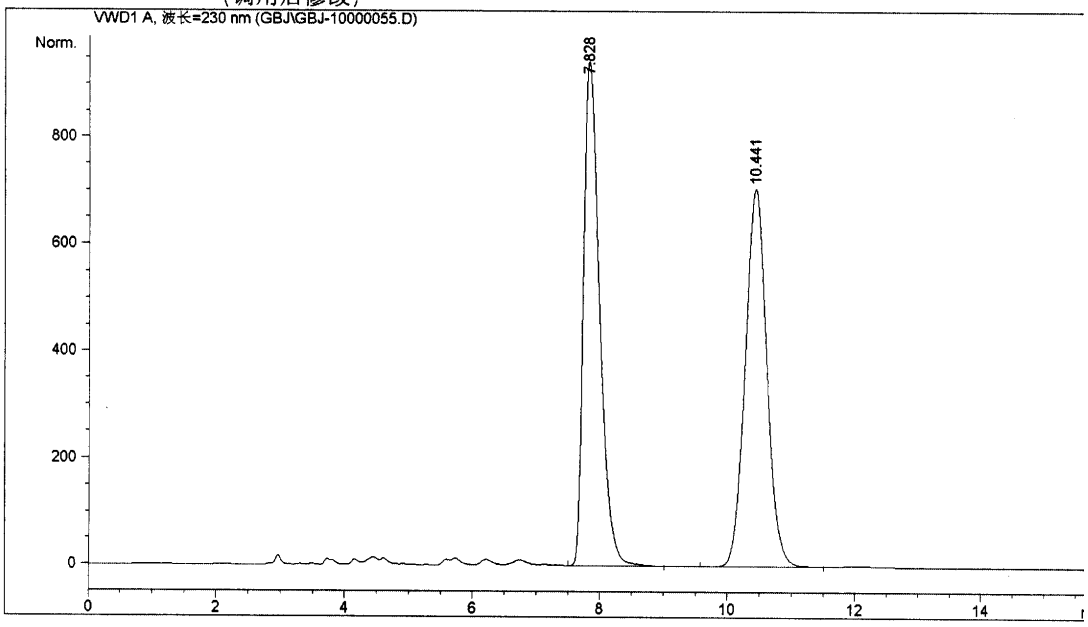
仪器 1 2013-6-15 16:01:19　gbj　　页 1 / 1

数据文件　　D:\Chem32\1\DATA\GBJ\GBJ-10000055.D
样品名: gbj-10-53-rac

OJ-H, n-Hexane/i-PrOH =80/20, 1.20 ml/min; 230 nm

==========

进样日期	: 2013-6-15 16:31:13		
样品名称	: gbj-10-53-rac	位置	: -
操作者	: gbj		
仪器	: 仪器 1		
方法	: D:\CHEM32\1\METHODS\ZYY_LC.M		
最后修改	: 2013-6-15 15:33:23 : lxj (调用后修改)		



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面积百分比报告

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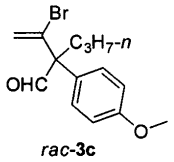
排序	:	信号
乘积因子	:	1.0000
稀释因子	:	1.0000

内标使用乘积因子和稀释因子



rac-3c

信号 1: VWD1 A, 波长=230 nm

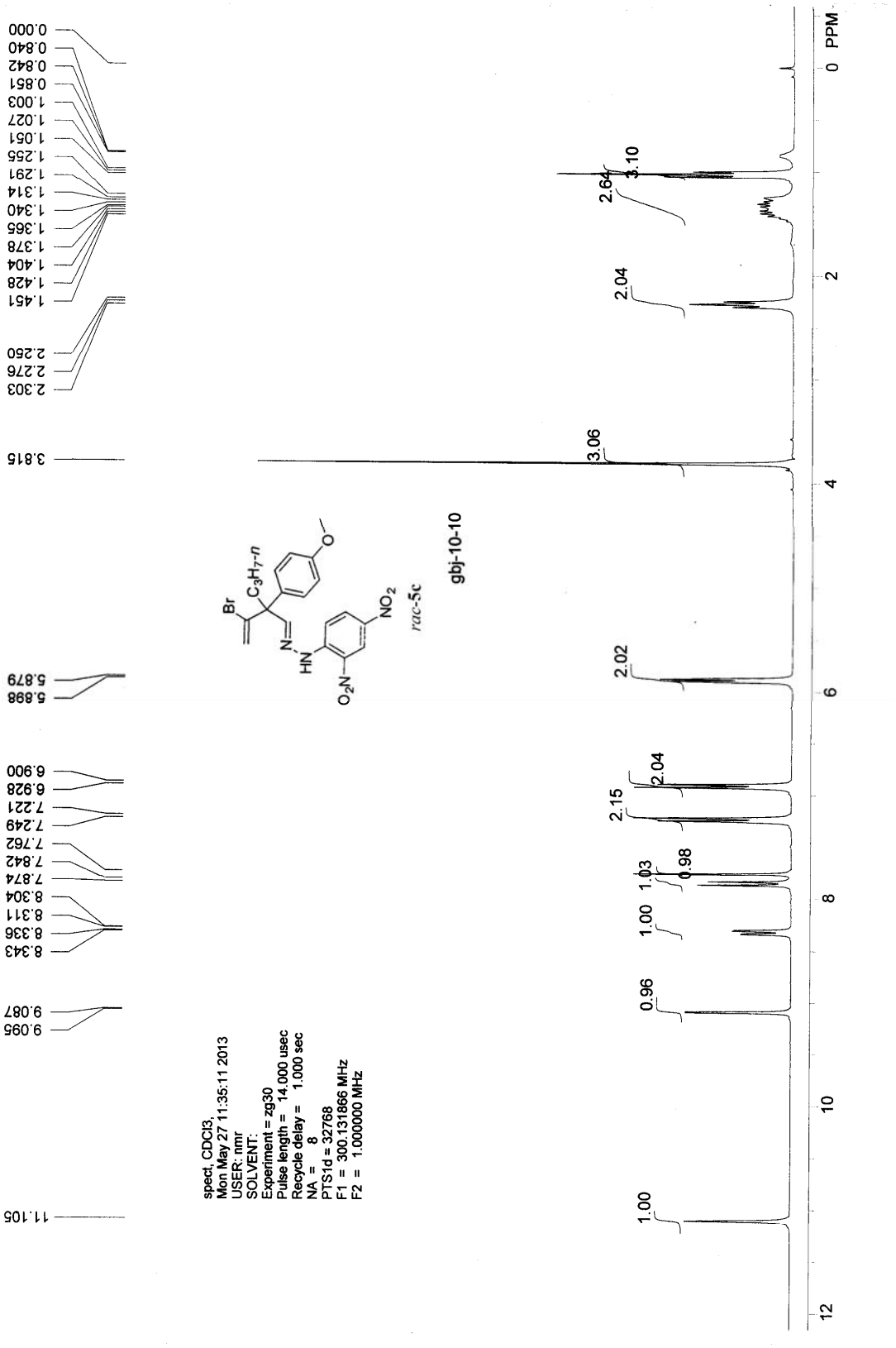
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	7.828	VB	0.2708	1.66321e4	945.34326	49.9513
2	10.441	VB	0.3613	1.66645e4	705.97638	50.0487

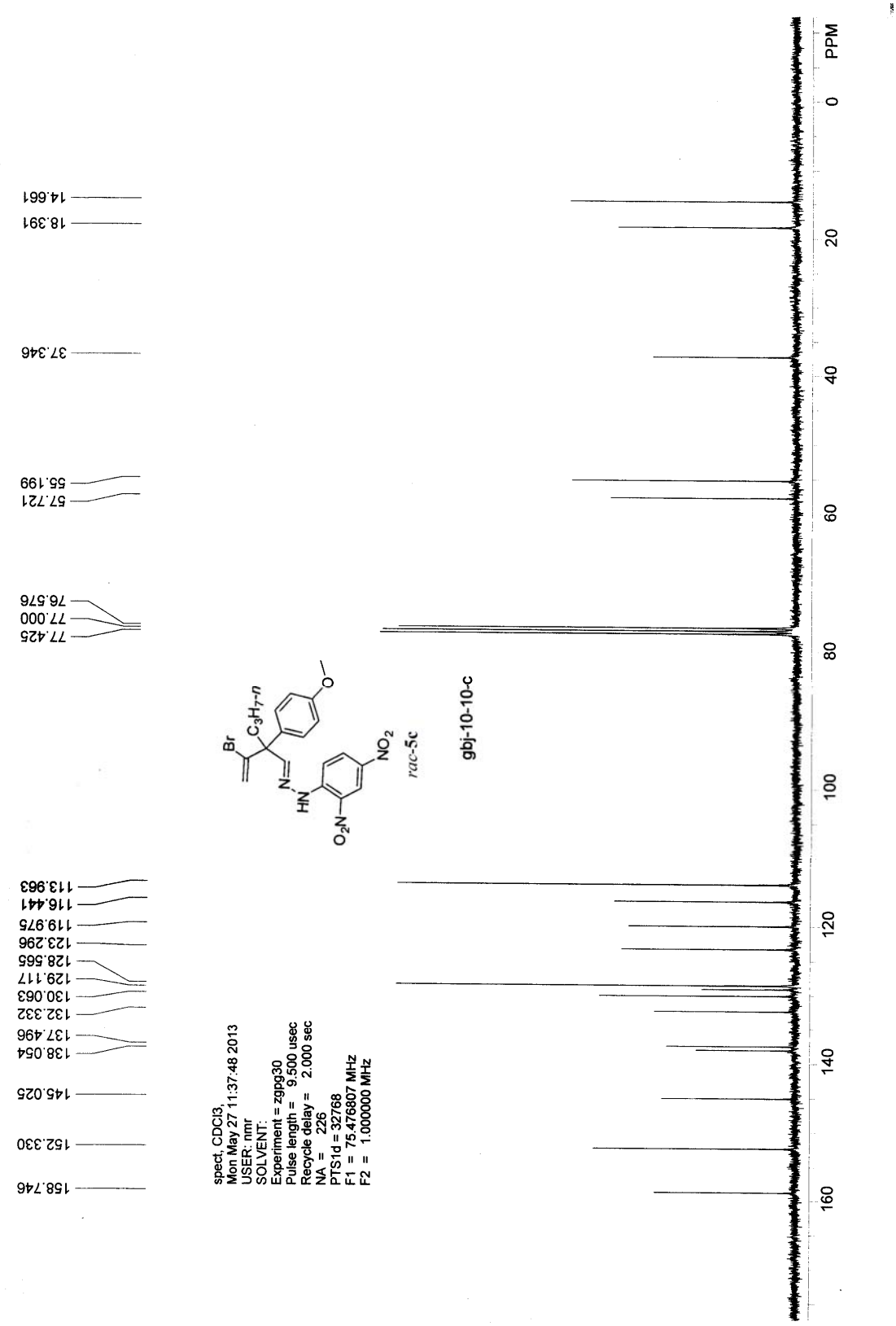
总量 :　　3.32966e4　1651.31964

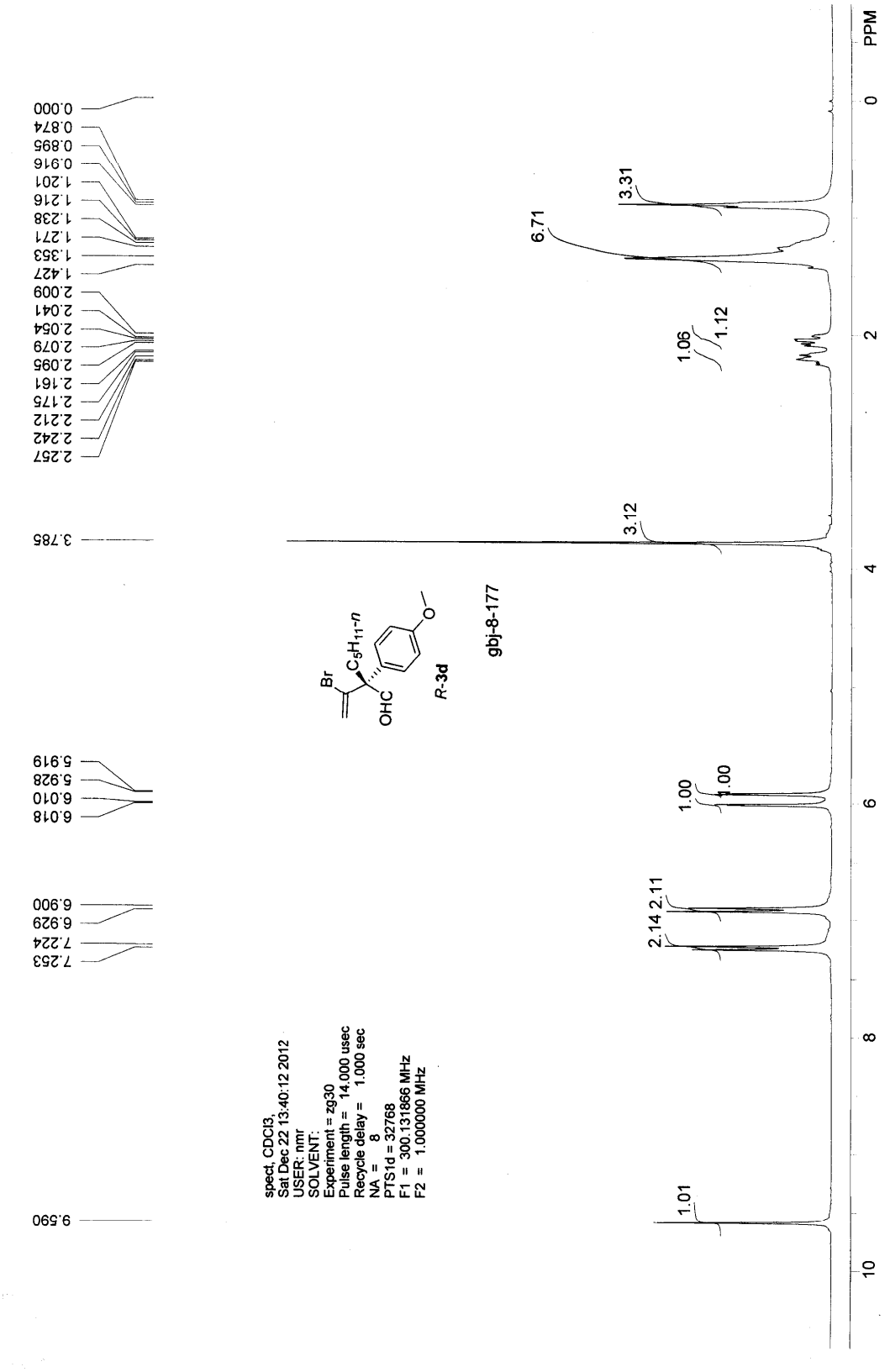
==========

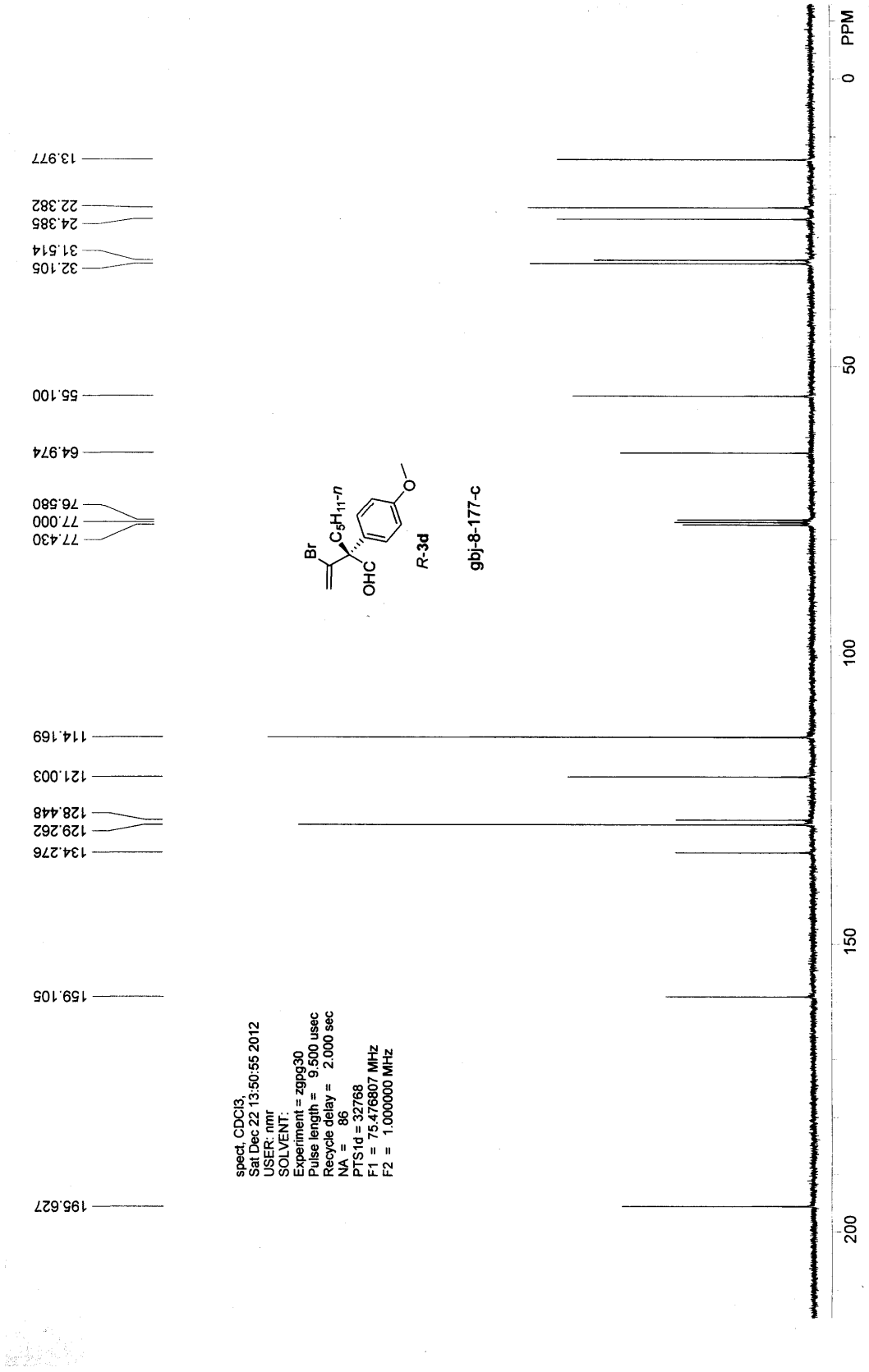
*** 报告结束 ***

仪器 1 2013-6-15 16:47:34 gbj　　页 1 / 1


spect, CDCl3,
Mon May 27 11:35:11 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-10-10
rac-5c
Br
C3H7-n
O
N
HN
NO2
O2N
PPM
0
2
4
6
8
10
12
1.00
0.96
1.00
1.03
0.98
2.15
2.04
2.02
3.06
2.04
2.64
3.10
11.105
9.095
9.087
8.343
8.336
8.311
8.304
7.874
7.842
7.762
7.249
7.221
6.928
6.900
5.898
5.879
3.815
2.303
2.276
2.260
1.451
1.428
1.404
1.378
1.365
1.340
1.314
1.291
1.255
1.051
1.027
1.003
0.851
0.842
0.840
0.000


14.661
18.391
37.346
55.199
57.721
76.576
77.000
77.425
113.963
116.441
119.975
123.296
128.565
129.117
130.063
132.332
137.496
138.054
145.025
152.330
158.746
spect, CDCl3,
Mon May 27 11:37:48 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 226
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
C3H7-n
O
N
HN
NO2
O2N
rac-5c
gbj-10-10-c
0
20
40
60
80
100
120
140
160
PPM

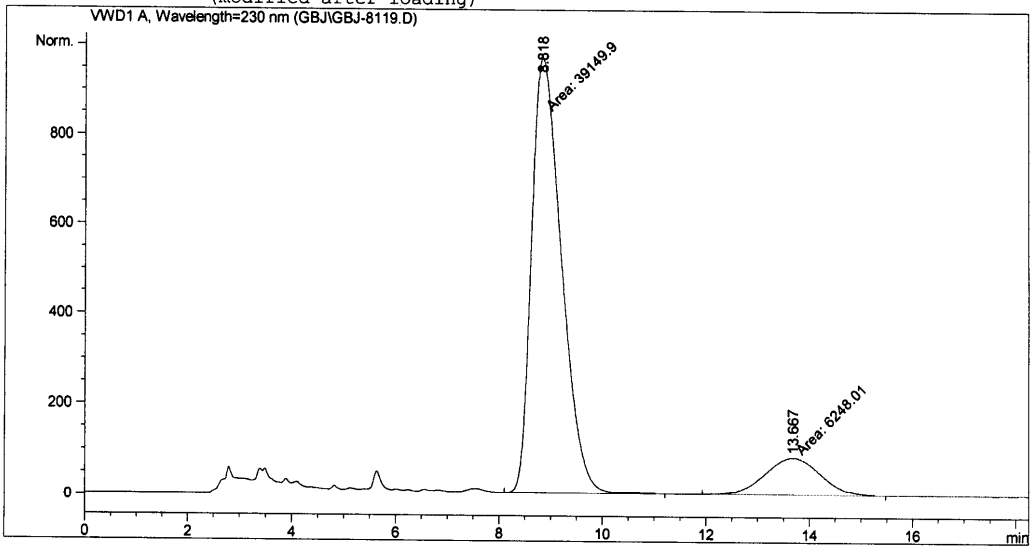

spect, CDCl3,
Sat Dec 22 13:40:12 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-8-177
R-3d
Br
OHC
C5H11-n
O
9.590
7.253
7.224
6.929
6.900
6.018
6.010
5.928
5.919
3.785
2.257
2.242
2.212
2.175
2.161
2.095
2.079
2.054
2.041
2.009
1.427
1.353
1.271
1.238
1.216
1.201
0.916
0.895
0.874
0.000
1.01
2.14
2.11
1.00
1.00
3.12
6.71
3.31
1.06
1.12
10
8
6
4
2
0
PPM


13.977
22.382
24.385
31.514
32.105
55.100
64.974
76.580
77.000
77.430
114.169
121.003
128.448
129.262
134.276
159.105
195.627
spect, CDCl3,
Sat Dec 22 13:50:55 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 86
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
OHC
C_5H_{11}-*n*
R-**3d**
gbj-8-177-c
200
150
100
50
0
PPM

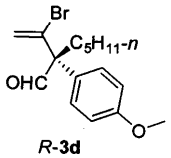
Data File D:\HPCHEM\1\DATA\GBJ\GBJ-8119.D Sample Name: gbj-8-177

OJ-H, n-Hexane:i-PrOH = 80/20, 1.2 mL/min, 230 nm

```
==========================================================================
Injection Date  : 12/17/2012 12:52:55 AM
Sample Name     : gbj-8-177                    Location :   -
Acq. Operator   : gbj
Method          : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/17/2012 12:02:59 AM by zyy
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]     %
----|-------|----|-------|----------|----------|--------|
   1   8.818 MM    0.6727 3.91499e4   970.00580  86.2372
   2  13.667 MM    1.2618 6248.00537   82.53072  13.7628

Totals :                  4.53979e4  1052.53651

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```



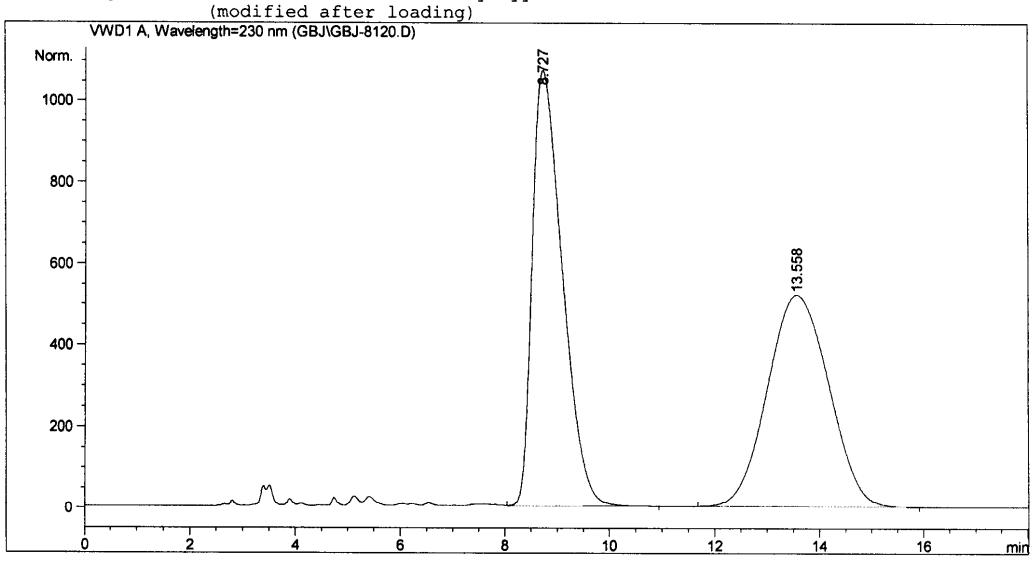
R-**3d**

Instrument 1 12/17/2012 1:15:01 AM gbj

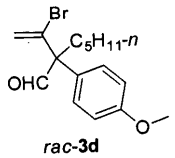
Data File D:\HPCHEM\1\DATA\GBJ\GBJ-8120.D Sample Name: gbj-8-176

OJ-H, n-Hexane:i-PrOH = 80/20, 1.2 mL/min, 230 nm

```
==========================================================================
Injection Date  : 12/17/2012 1:12:09 AM
Sample Name     : gbj-8-176                    Location :   -
Acq. Operator   : gbj
Method          : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/17/2012 12:02:59 AM by zyy
                  (modified after loading)
```

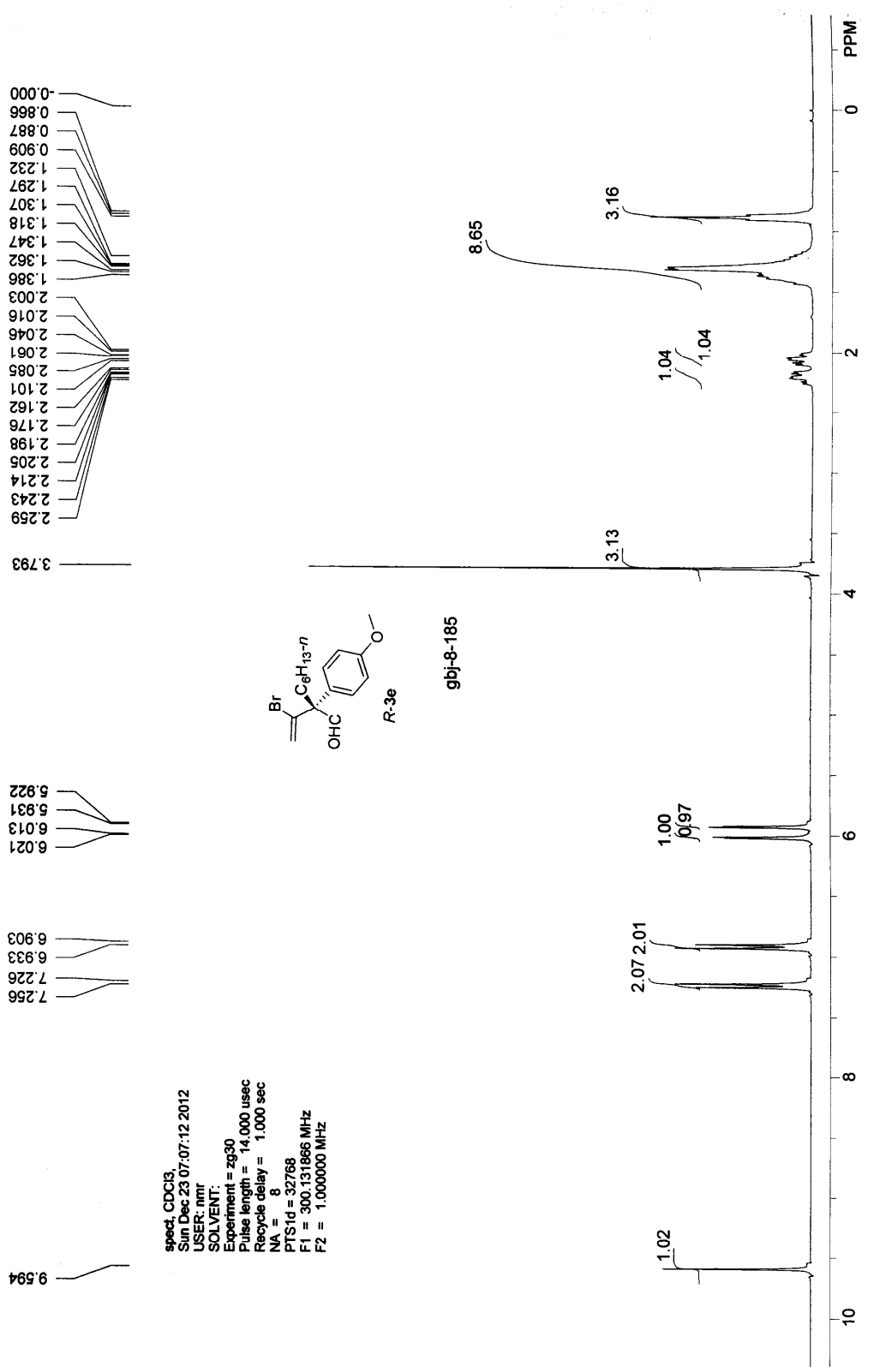


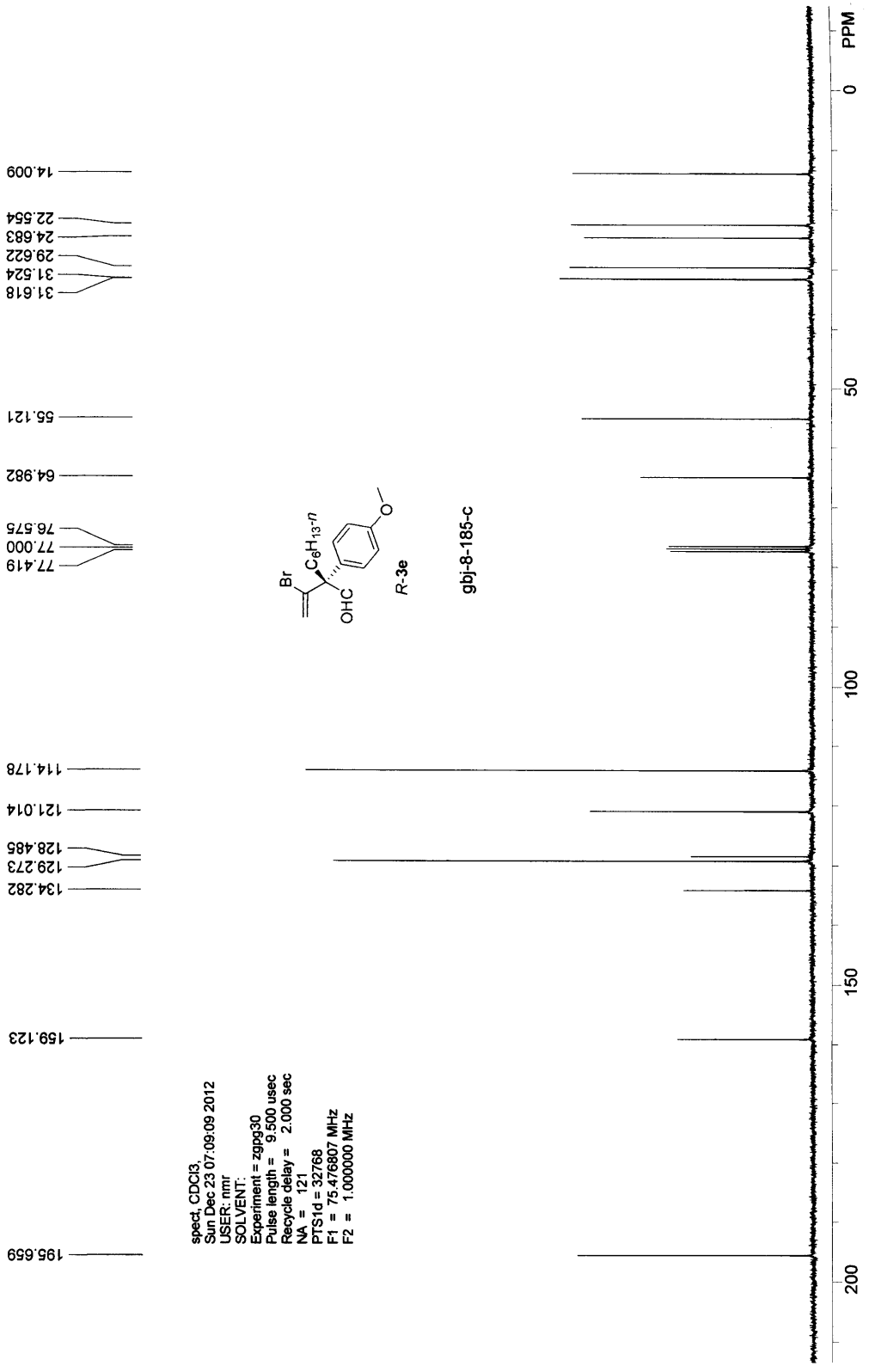

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   8.727 VB    0.6163 4.29738e4  1071.87573  50.2843
   2  13.558 BB    1.3078 4.24879e4   518.81262  49.7157

Totals :                  8.54618e4  1590.68835

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```



rac-**3d**

Instrument 1 12/17/2012 1:30:32 AM gbj Page 1 of 1

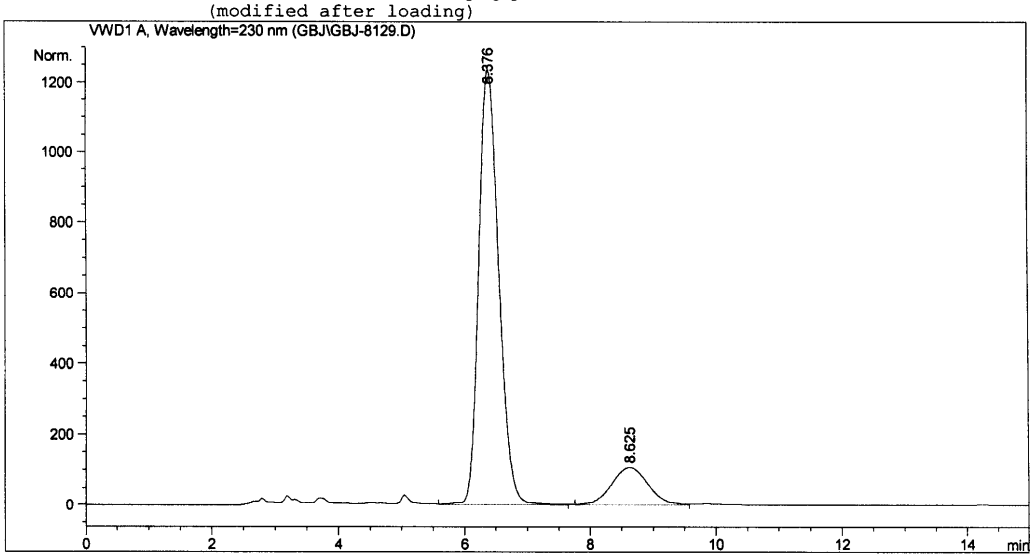

spect, CDCl3,
Sun Dec 23 07:07:12 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-8-185
R-3e
Br
OHC
C6H13-n
O
PPM
9.594
7.256
7.226
6.933
6.903
6.021
6.013
5.931
5.922
3.793
2.259
2.243
2.214
2.205
2.198
2.176
2.162
2.101
2.085
2.061
2.046
2.016
2.003
1.386
1.362
1.347
1.318
1.307
1.297
1.232
0.909
0.887
0.866
-0.000
1.02
2.07
2.01
1.00
0.97
3.13
8.65
1.04
1.04
3.16


14.009
22.554
24.683
29.622
31.524
31.618
55.121
64.982
76.575
77.000
77.419
114.178
121.014
128.485
129.273
134.282
159.123
195.659
spect, CDCl3,
Sun Dec 23 07:09:09 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 121
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
C_6H_{13}-n
OHC
O
R-3e
gbj-8-185-c
PPM
0
50
100
150
200

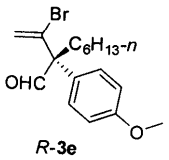
Data File D:\HPCHEM\1\DATA\GBJ\GBJ-8129.D Sample Name: gbj-8-185

OJ-H, n-Hexane:i-PrOH= 80/20, 1.2 mL/min, 230 nm

```
==========================================================================
Injection Date  : 12/25/2012 9:47:08 AM
Sample Name     : gbj-8-185                    Location :   -
Acq. Operator   : gbj
Method          : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/25/2012 9:09:59 AM by qhj
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   6.376 VB    0.3437 2.75027e4  1237.72107  85.9990
   2   8.625 BV    0.6495 4477.56201  107.39820  14.0010

Totals :                  3.19803e4  1345.11927

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

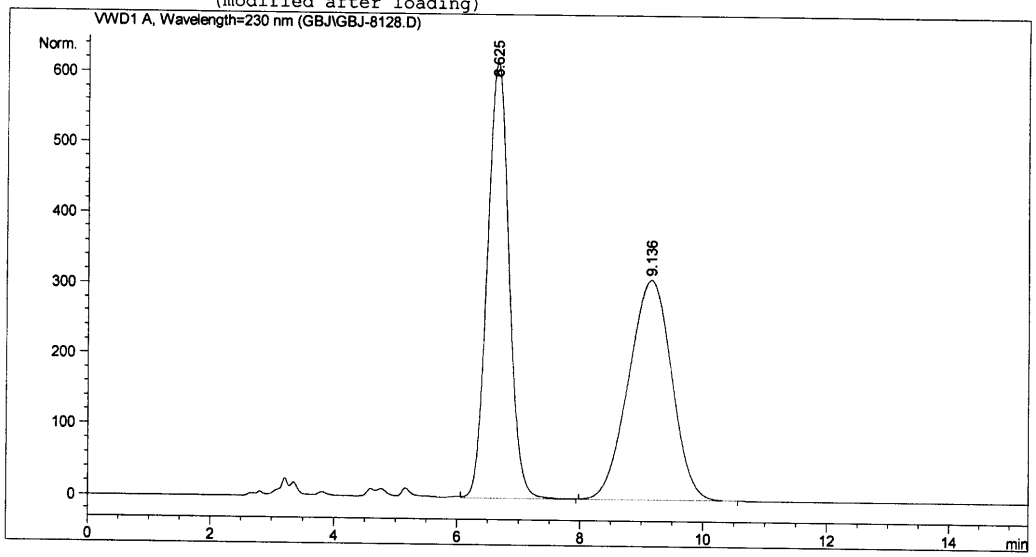


R-**3e**

Data File D:\HPCHEM\1\DATA\GBJ\GBJ-8128.D Sample Name: gbj-8-184

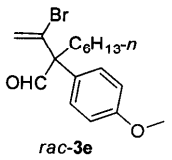
OJ-H, n-Hexane:i-PrOH= 80/20, 1.2 mL/min, 230 nm

```
Injection Date  : 12/25/2012 9:30:27 AM
Sample Name     : gbj-8-184                    Location :  -
Acq. Operator   : gbj
Method          : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/25/2012 9:09:59 AM by qhj
                  (modified after loading)
```



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs



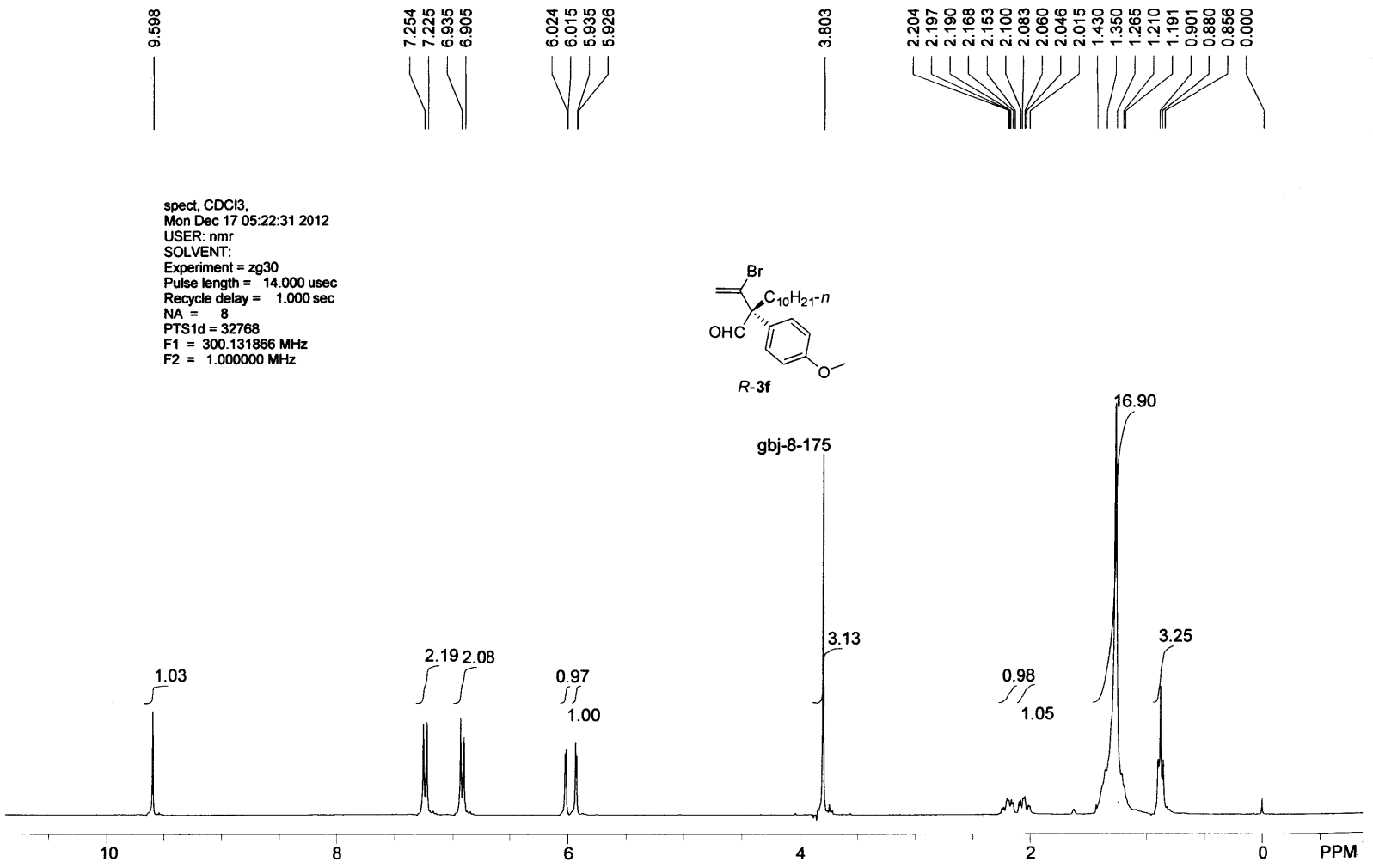
rac-**3e**

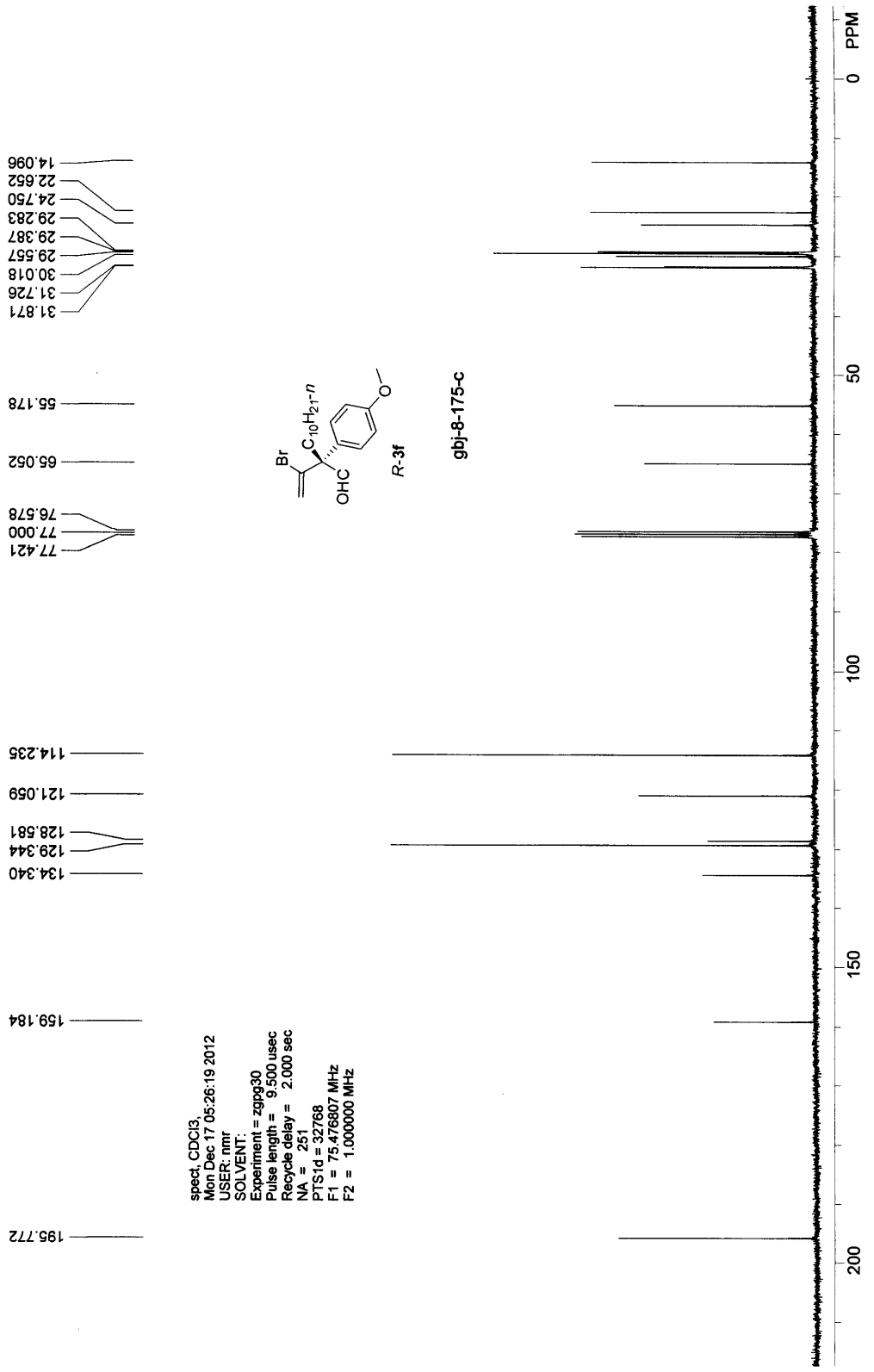
Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.625	VB	0.3825	1.51787e4	618.35791	50.1408
2	9.136	BB	0.7606	1.50935e4	311.39893	49.8592
Totals :				3.02723e4	929.75684	

Results obtained with enhanced integrator!

*** End of Report ***

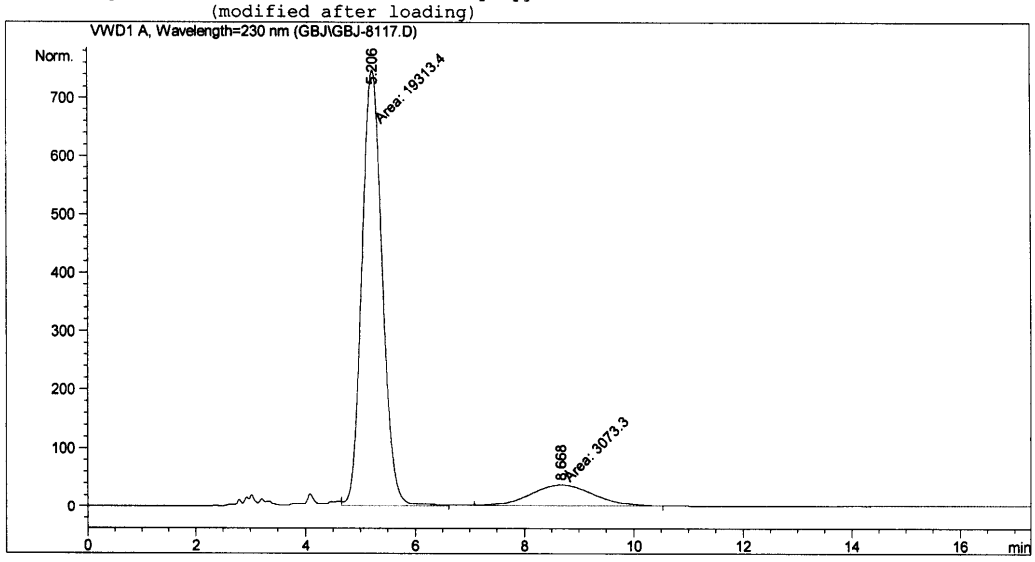

9.598
7.254
7.225
6.935
6.905
6.024
6.015
5.935
5.926
3.803
2.204
2.197
2.190
2.168
2.153
2.100
2.083
2.060
2.046
2.015
1.430
1.350
1.265
1.210
1.191
0.901
0.880
0.856
0.000
spect, CDCl3,
Mon Dec 17 05:22:31 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
C10H21-n
OHC
O
R-3f
gbj-8-175
16.90
3.13
3.25
2.19
2.08
1.03
0.97
1.00
0.98
1.05
10
8
6
4
2
0
PPM


14.096
22.652
24.750
29.283
29.387
29.557
30.018
31.726
31.871
55.178
65.052
76.578
77.000
77.421
114.235
121.059
128.581
129.344
134.340
159.184
195.772
spect, CDCl3,
Mon Dec 17 05:26:19 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 251
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
$C_{10}H_{21}$-n
OHC
O
R-3f
gbj-8-175-c
0
50
100
150
200
PPM

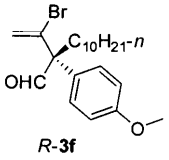
Data File D:\HPCHEM\1\DATA\GBJ\GBJ-8117.D Sample Name: gbj-8-175

OJ-H, n-Hexane:i-PrOH = 80/20, 1.2 mL/min, 230 nm

```
==========================================================================
Injection Date  : 12/17/2012 12:18:18 AM
Sample Name     : gbj-8-175                    Location :   -
Acq. Operator   : gbj
Method          : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/17/2012 12:02:59 AM by zyy
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   5.206 MM    0.4307 1.93134e4   747.31372  86.2718
   2   8.668 MM    1.3875 3073.29663   36.91563  13.7282

Totals :                  2.23867e4   784.22935

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```



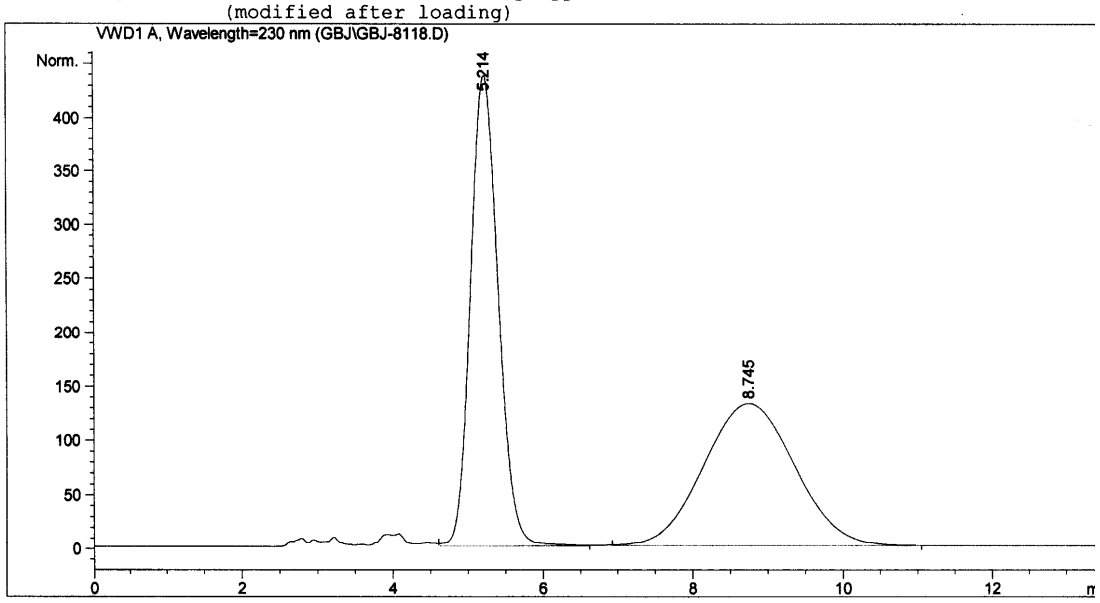
R-**3f**

Instrument 1 12/17/2012 12:51:00 AM gbj Page 1 of 1

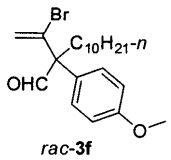
:a File D:\HPCHEM\1\DATA\GBJ\GBJ-8118.D Sample Name: gbj-8-1

OJ-H, n-Hexane:i-PrOH = 80/20, 1.2 mL/min, 230 nm

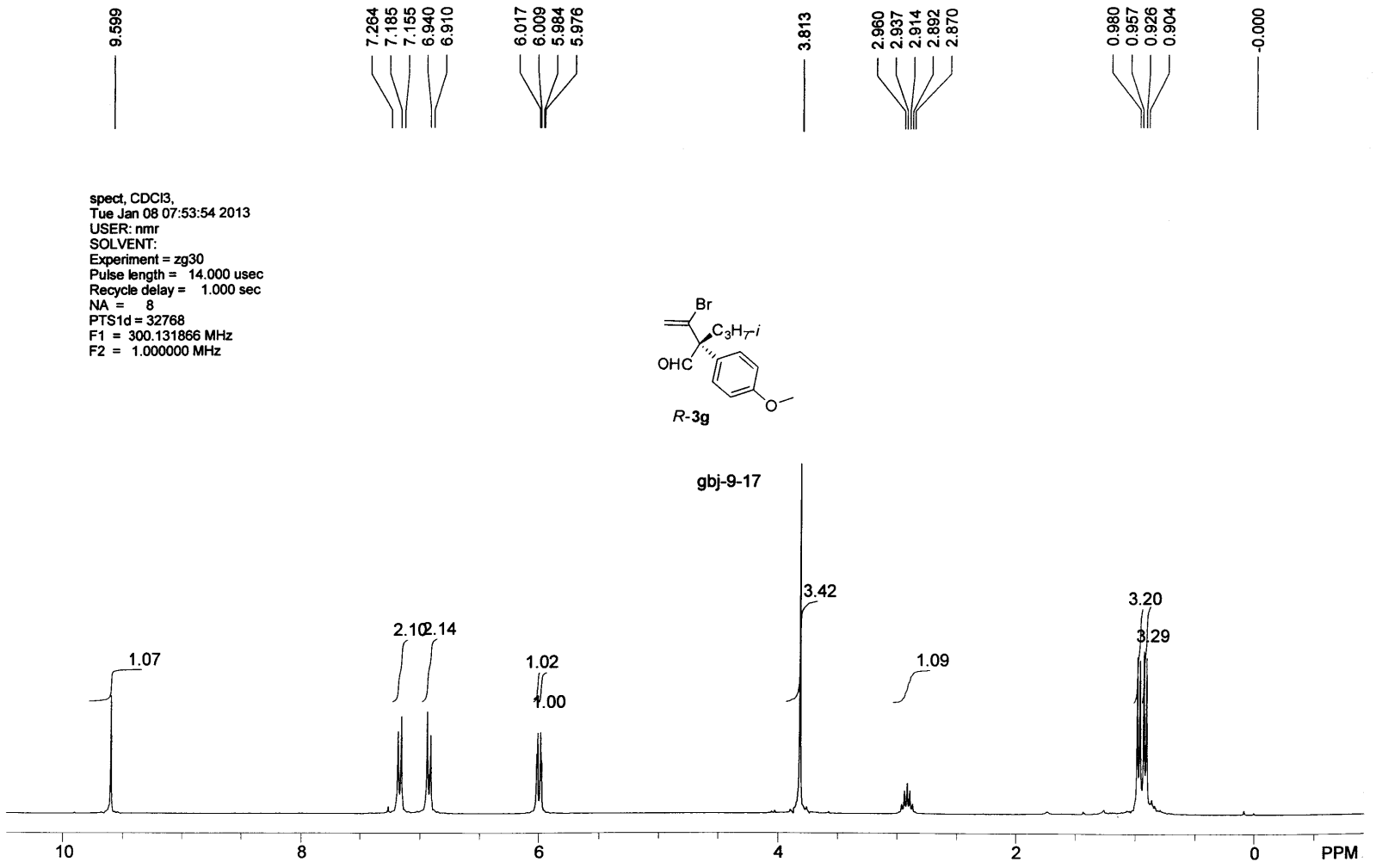
```
==========================================================================
Injection Date  : 12/17/2012 12:38:18 AM
Sample Name     : gbj-8-174                        Location :   -
Acq. Operator   : gbj
Method          : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/17/2012 12:02:59 AM by zyy
                  (modified after loading)
```

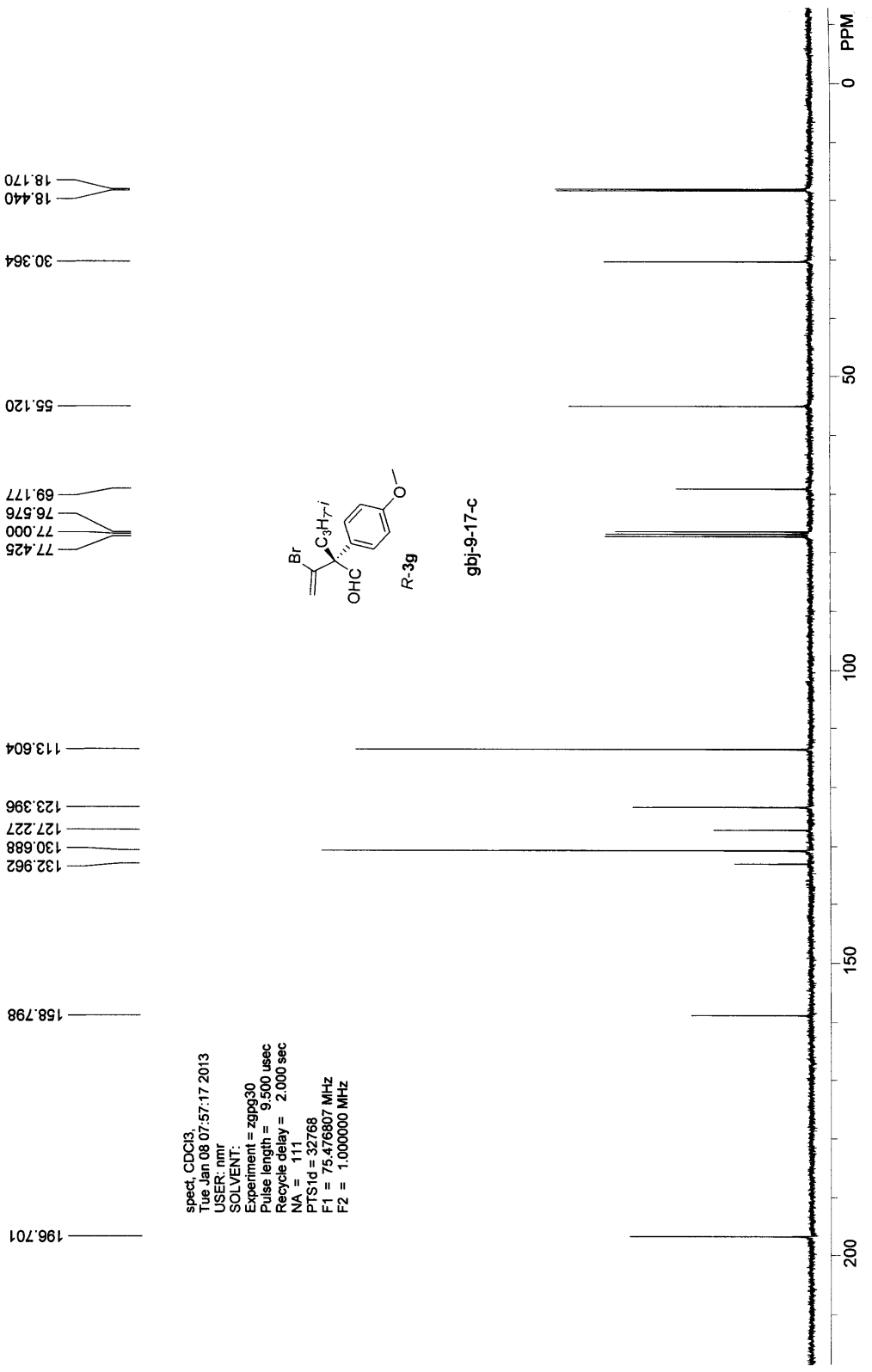



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]     %
----|-------|----|-------|----------|----------|--------|
   1   5.214 VB    0.4028 1.12661e4   436.91611  50.1087
   2   8.745 BB    1.3375 1.12173e4   131.67439  49.8913

Totals :                  2.24834e4   568.59050

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```



rac-**3f**


9.599
7.264
7.185
7.155
6.940
6.910
6.017
6.009
5.984
5.976
3.813
2.960
2.937
2.914
2.892
2.870
0.980
0.957
0.926
0.904
-0.000
spect, CDCl3,
Tue Jan 08 07:53:54 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
C3H7-i
OHC
O
R-3g
gbj-9-17
1.07
2.10
2.14
1.02
1.00
3.42
1.09
3.20
3.29
10
8
6
4
2
0
PPM

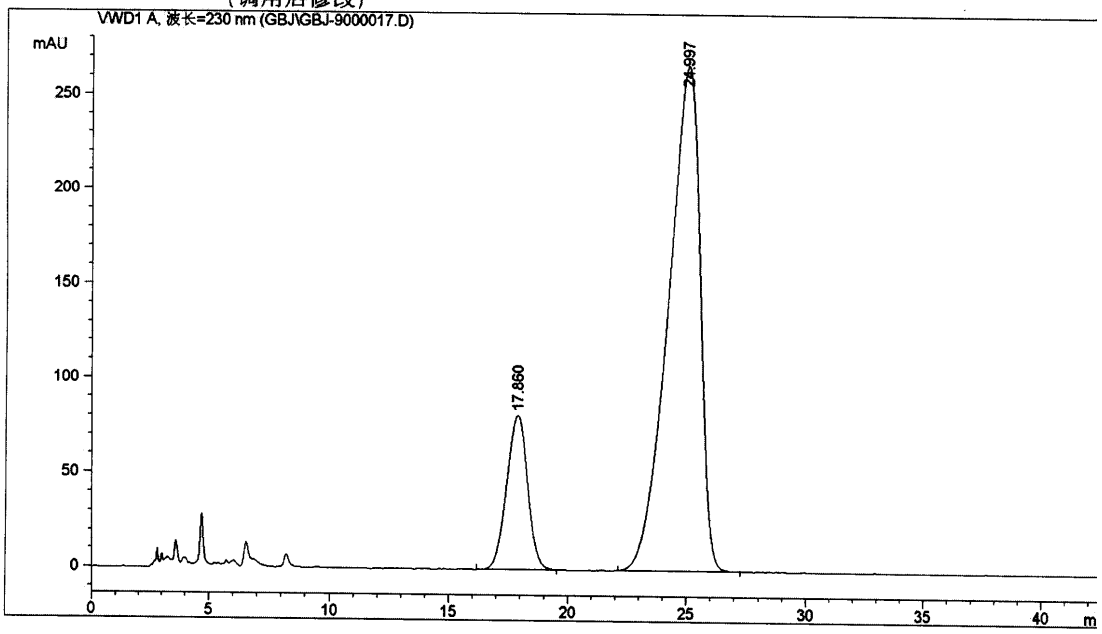

18.170
18.440
30.364
55.120
69.177
76.576
77.000
77.425
113.604
123.396
127.227
130.688
132.962
158.798
196.701
spect, CDCl3,
Tue Jan 08 07:57:17 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 111
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
C3H7-i
OHC
O
R-3g
gbj-9-17-c
0
50
100
150
200
PPM

据文件　　D:\Chem32\1\DATA\GBJ\GBJ-9000017.D
品名: gbj-9-17

OJ-H; Hexane/iPrOH=80/20; 1.2 ml/min, 230nm

==========

进样日期　　: 2013-1-9 16:46:47
样品名称　　: gbj-9-17　　位置　: 样品瓶1
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2013-1-9 15:54:18　:　gbj
(调用后修改)

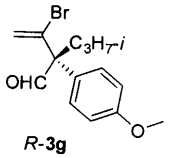


==========

面积百分比报告

==========

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子



R-3g

信号 1: VWD1 A, 波长=230 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	17.860	BB	0.9649	4987.16650	81.11022	16.8176
2	24.997	BV	1.3828	2.46672e4	266.83478	83.1824

总量 :　　2.96544e4　　347.94500

==========

*** 报告结束 ***

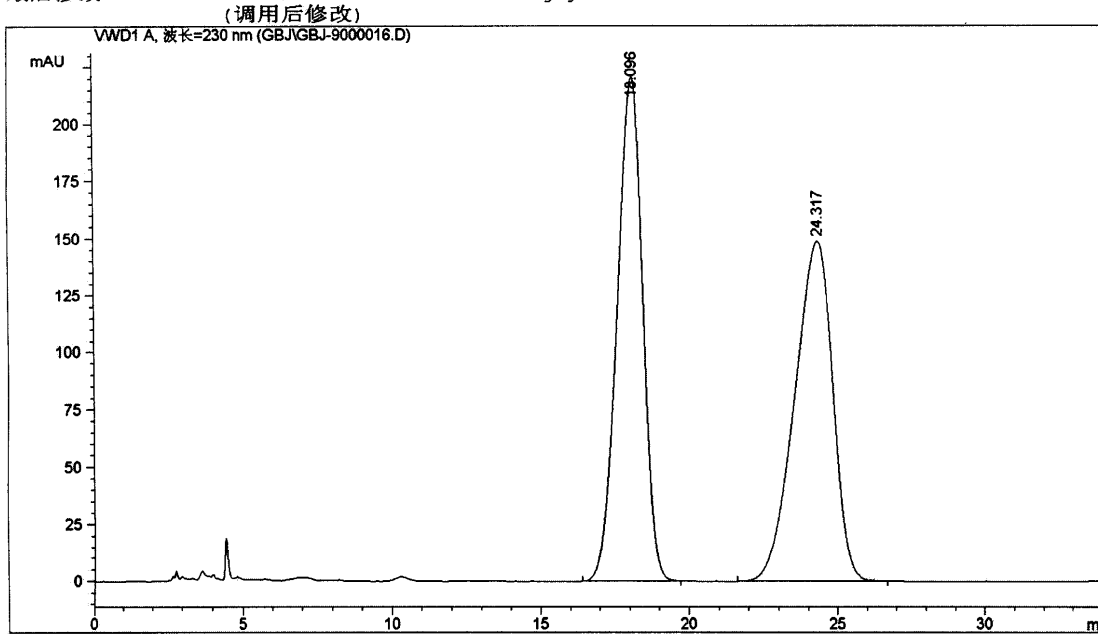
器 1 2013-1-9 17:30:06　gbj　　页 1 / 1

据文件　　D:\Chem32\1\DATA\GBJ\GBJ-9000016.D
品名：gbj-9-16

OJ-H; Hexane/iPrOH=80/20; 1.2 ml/min, 230nm

==========

进样日期　　：2013-1-9 16:10:46
样品名称　　：gbj-9-16　　位置　：样品瓶1
操作者　　：gbj
仪器　　：仪器 1
方法　　：D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　：2013-1-9 15:54:18　：　gbj
(调用后修改)



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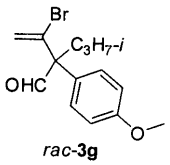
面积百分比报告

==========

排序　　：　信号
乘积因子　　：　1.0000
稀释因子　　：　1.0000
内标使用乘积因子和稀释因子



rac-**3g**

信号 1: VWD1 A, 波长=230 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	18.096	BB	0.9174	1.31869e4	220.54422	49.8509
2	24.317	BB	1.3449	1.32658e4	148.73380	50.1491

总量 :　　2.64526e4　　369.27802

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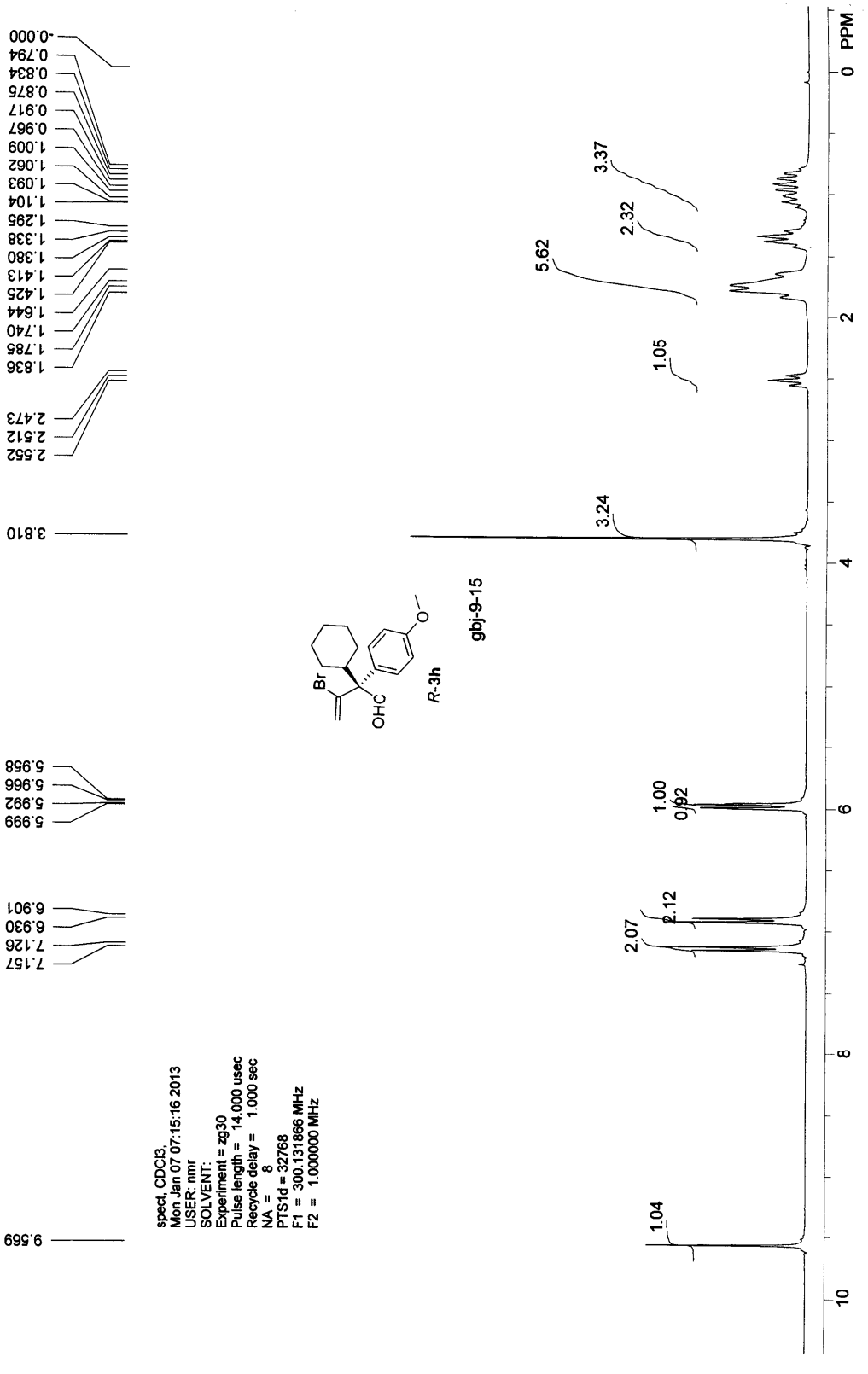
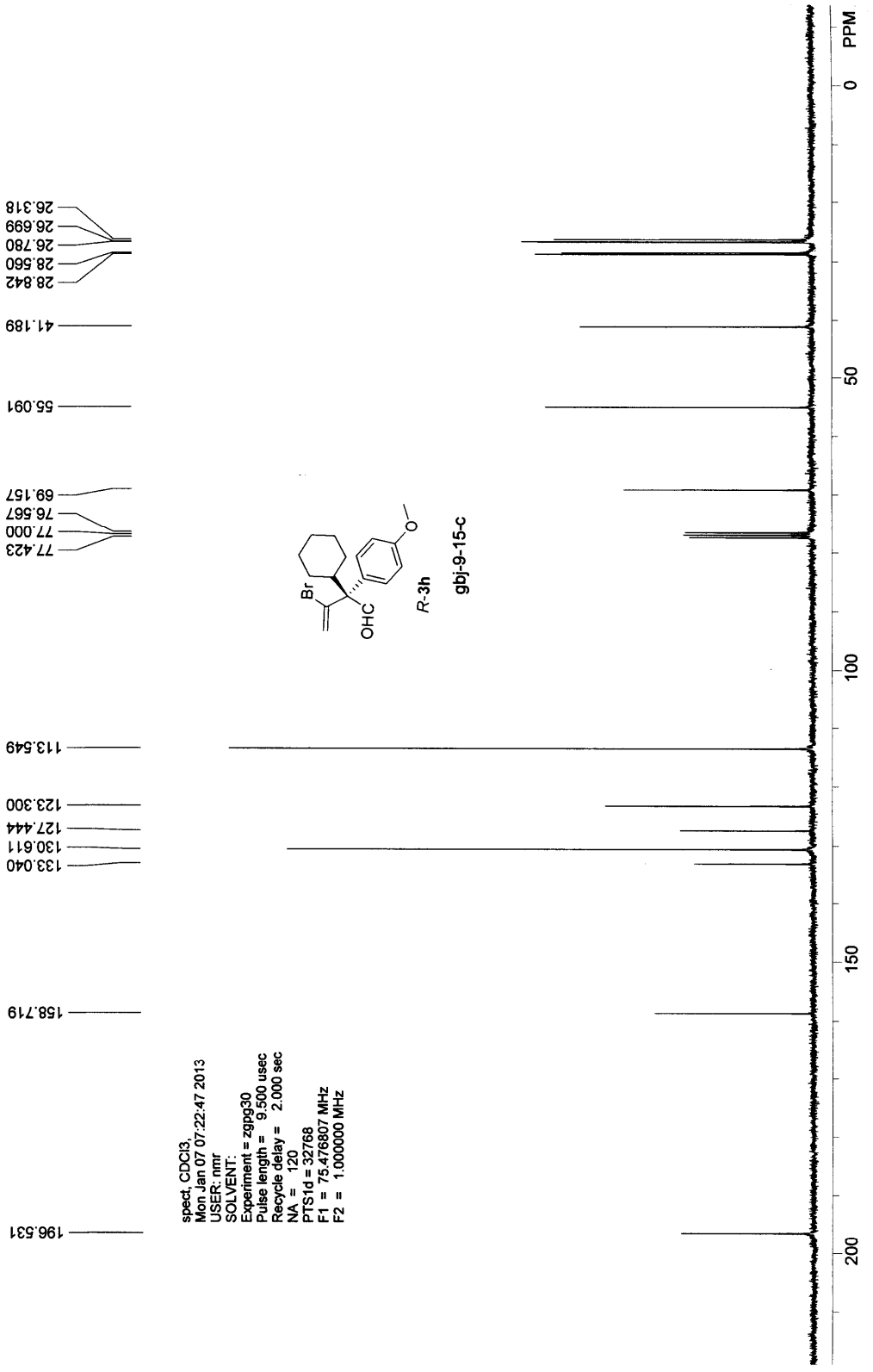
*** 报告结束 ***

器 1 2013-1-9 16:45:37　gbj　　页　1 / 1


spect, CDCl3,
Mon Jan 07 07:15:16 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-9-15
R-3h
Br
OHC
O
9.569
7.157
7.126
6.930
6.901
5.999
5.992
5.966
5.958
3.810
2.562
2.512
2.473
1.836
1.785
1.740
1.644
1.425
1.413
1.380
1.338
1.295
1.104
1.093
1.062
1.009
0.967
0.917
0.875
0.834
0.794
-0.000
1.04
2.07
2.12
1.00
0.92
3.24
1.05
5.62
2.32
3.37
10
8
6
4
2
0 PPM

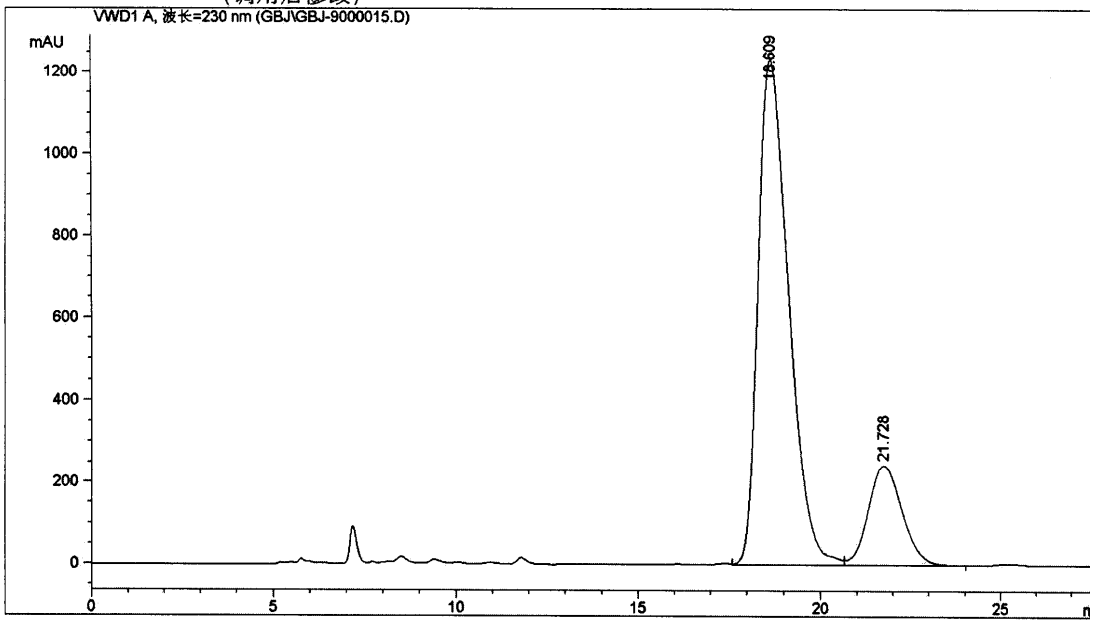

196.531
158.719
133.040
130.611
127.444
123.300
113.549
77.423
77.000
76.567
69.157
55.091
41.189
28.842
28.560
26.780
26.699
26.318
PPM
0
50
100
150
200
spect, CDCl3,
Mon Jan 07 07:22:47 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 120
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
OHC
O
R-3h
gbj-9-15-c

据文件　　D:\Chem32\1\DATA\GBJ\GBJ-9000015.D
品名: gbj-9-15

OJ-H; Hexane/iPrOH=95/5; 0.6 ml/min, 230nm

==========

进样日期	: 2013-1-9 15:25:52		
样品名称	: gbj-9-15	位置	: 样品瓶1
操作者	: gbj		
仪器	: 仪器 1		
采集方法	: D:\CHEM32\1\METHODS\ZYY_LC.M		
最后修改	: 2013-1-9 14:55:27 : gbj (调用后修改)		
分析方法	: D:\CHEM32\1\METHODS\ZYY_LC.M		
最后修改	: 2013-1-9 15:54:18 : gbj (调用后修改)		



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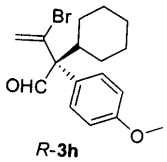
面积百分比报告

==========

排序	:	信号
乘积因子	:	1.0000
稀释因子	:	1.0000

内标使用乘积因子和稀释因子



R-3h

信号 1: VWD1 A, 波长=230 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	18.609	VV	0.9005	7.16974e4	1231.50586	81.8310
2	21.728	VB	1.0061	1.59190e4	244.34642	18.1690
总量 :				8.76164e4	1475.85228	

==========

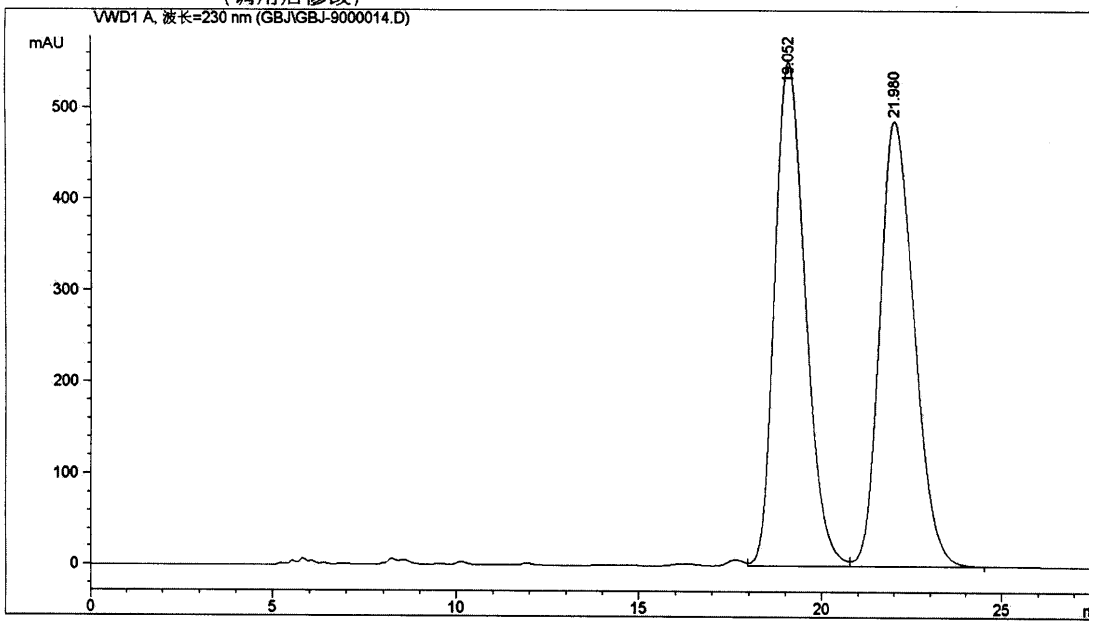
*** 报告结束 ***

据文件　　D:\Chem32\1\DATA\GBJ\GBJ-9000014.D
品名: gbj-9-14

OJ-H; Hexane/iPrOH=95/5; 0.6 ml/min, 230nm

==========

进样日期　　: 2013-1-9 14:56:12
样品名称　　: gbj-9-14　　位置　: 样品瓶1
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2013-1-9 14:55:27　:　gbj
(调用后修改)



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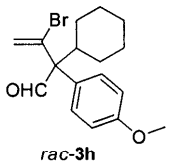
面积百分比报告

==========

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子



rac-3h

信号 1: VWD1 A, 波长=230 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	19.052	VV	0.8955	3.20997e4	551.88623	49.9449
2	21.980	VB	1.0276	3.21706e4	486.57935	50.0551

总量 :　　6.42703e4　1038.46558

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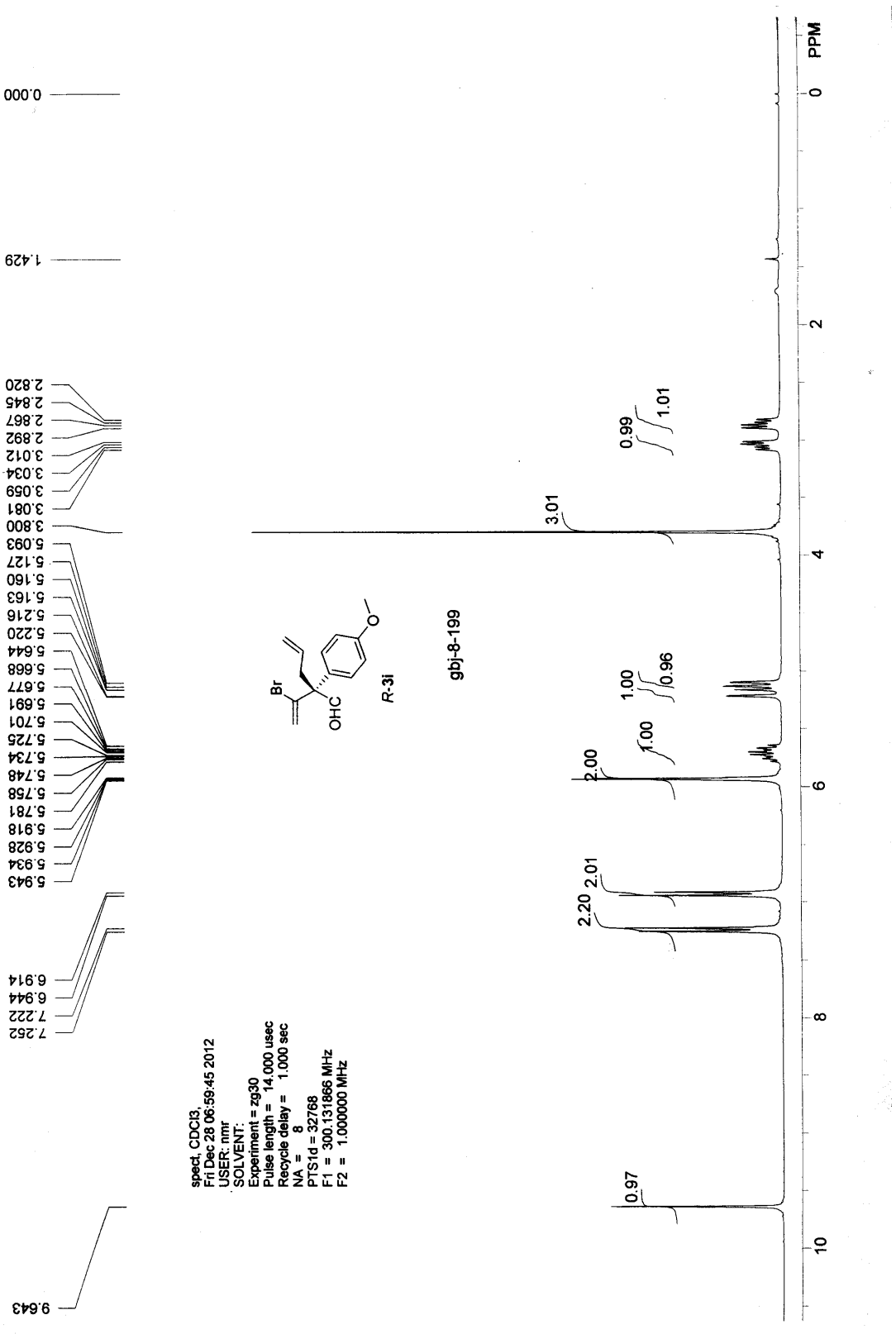
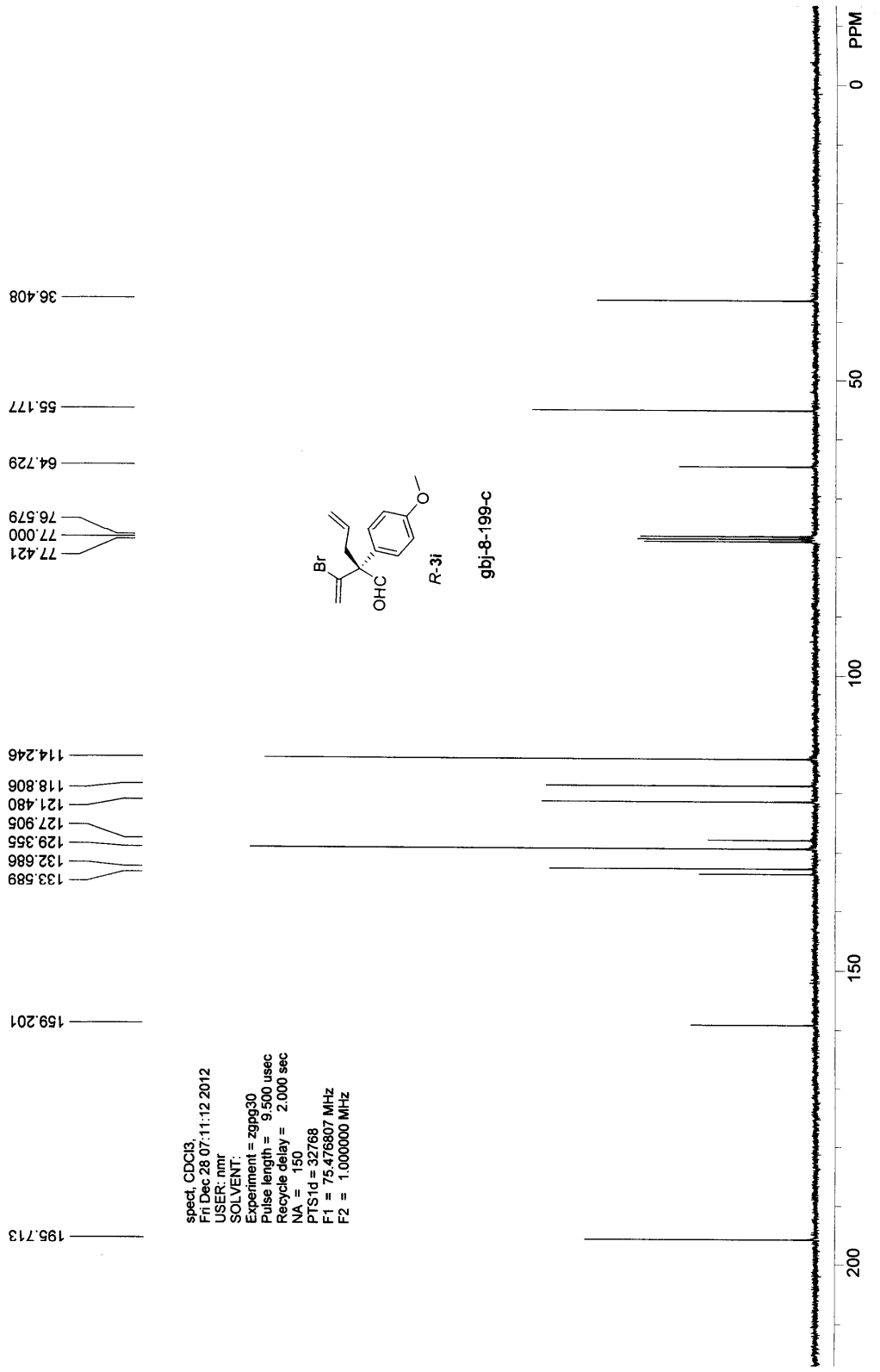
*** 报告结束 ***

器 1 2013-1-9 15:24:33　gbj　　页 1 / 1

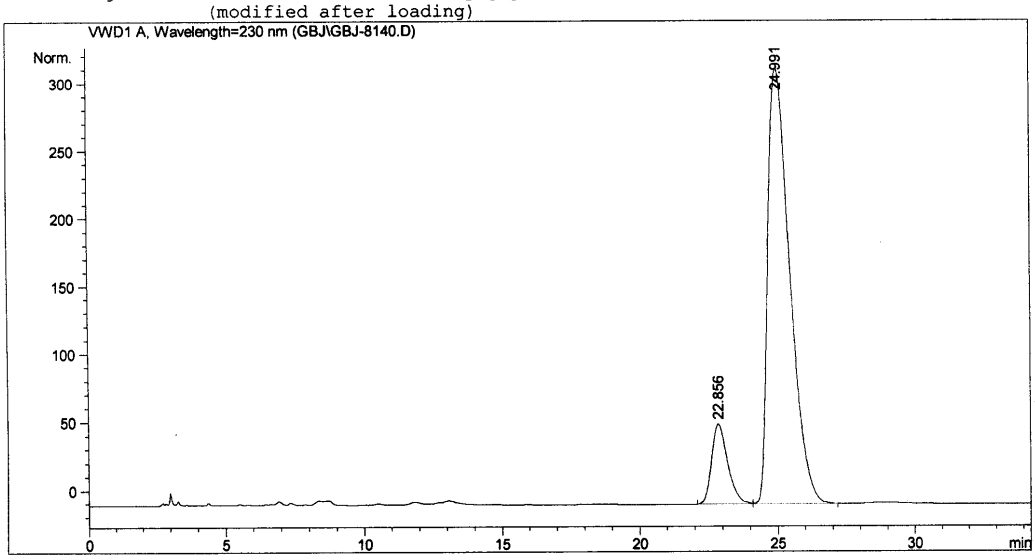

spect, CDCl3,
Fri Dec 28 06:59:45 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-8-199
R-3i
Br
OHC
O
PPM
0.000
1.429
2.820
2.845
2.867
2.892
3.012
3.034
3.059
3.081
3.800
5.093
5.127
5.160
5.163
5.216
5.220
5.644
5.668
5.677
5.691
5.701
5.725
5.734
5.748
5.758
5.781
5.918
5.928
5.934
5.943
6.914
6.944
7.222
7.252
9.643
0.97
2.20
2.01
2.00
1.00
1.00
0.96
3.01
0.99
1.01


spect, CDCl3,
Fri Dec 28 07:11:12 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 150
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
195.713
159.201
133.589
132.686
129.355
127.905
121.480
118.806
114.246
77.421
77.000
76.579
64.729
55.177
36.408
Br
OHC
O
R-3i
gbj-8-199-c
PPM
0
50
100
150
200

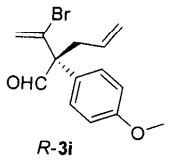
Data File D:\HPCHEM\1\DATA\GBJ\GBJ-8140.D Sample Name: gbj-8-199

OJ-H, n-Hexane:i-PrOH=98/2, 1.2 mL/min, 230 nm

```
==========================================================================
Injection Date  : 12/28/2012 9:10:16 PM
Sample Name     : gbj-8-199                    Location :  -
Acq. Operator   : gbj
Method          : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/28/2012 8:00:12 PM by gbj
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  22.856 BV    0.6228 2431.37769   59.08436  12.0708
   2  24.991 VB    0.8526 1.77112e4   321.40039  87.9292

Totals :                  2.01426e4   380.48475

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

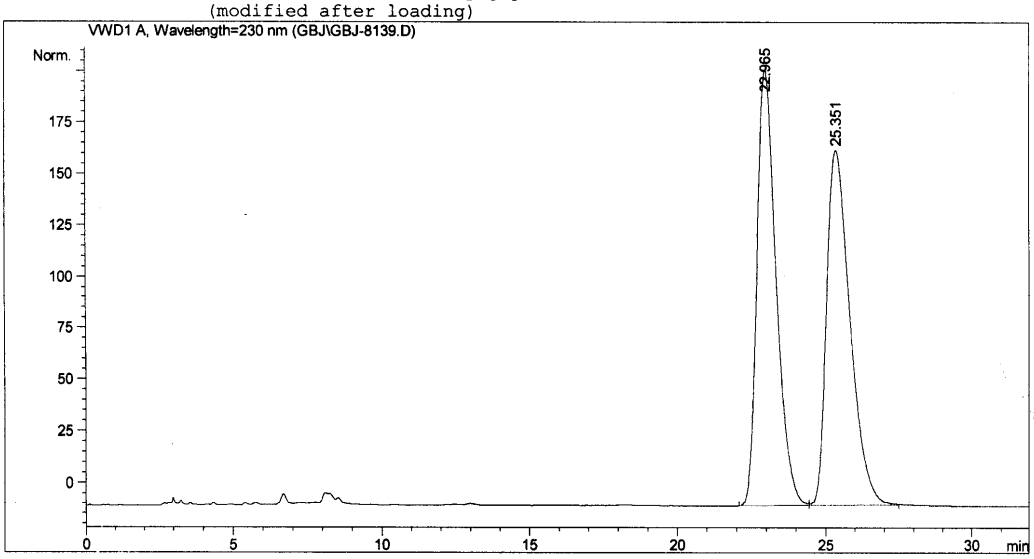


R-**3i**

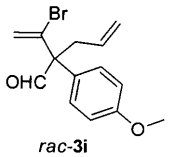
Data File D:\HPCHEM\1\DATA\GBJ\GBJ-8139.D Sample Name: gbj-8-198

OJ-H, n-Hexane:i-PrOH=98/2, 1.2 mL/min, 230 nm

```
==========================================================================
Injection Date  : 12/28/2012 8:36:39 PM
Sample Name     : gbj-8-198                    Location :   -
Acq. Operator   : gbj
Method          : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/28/2012 8:00:12 PM by gbj
                  (modified after loading)
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

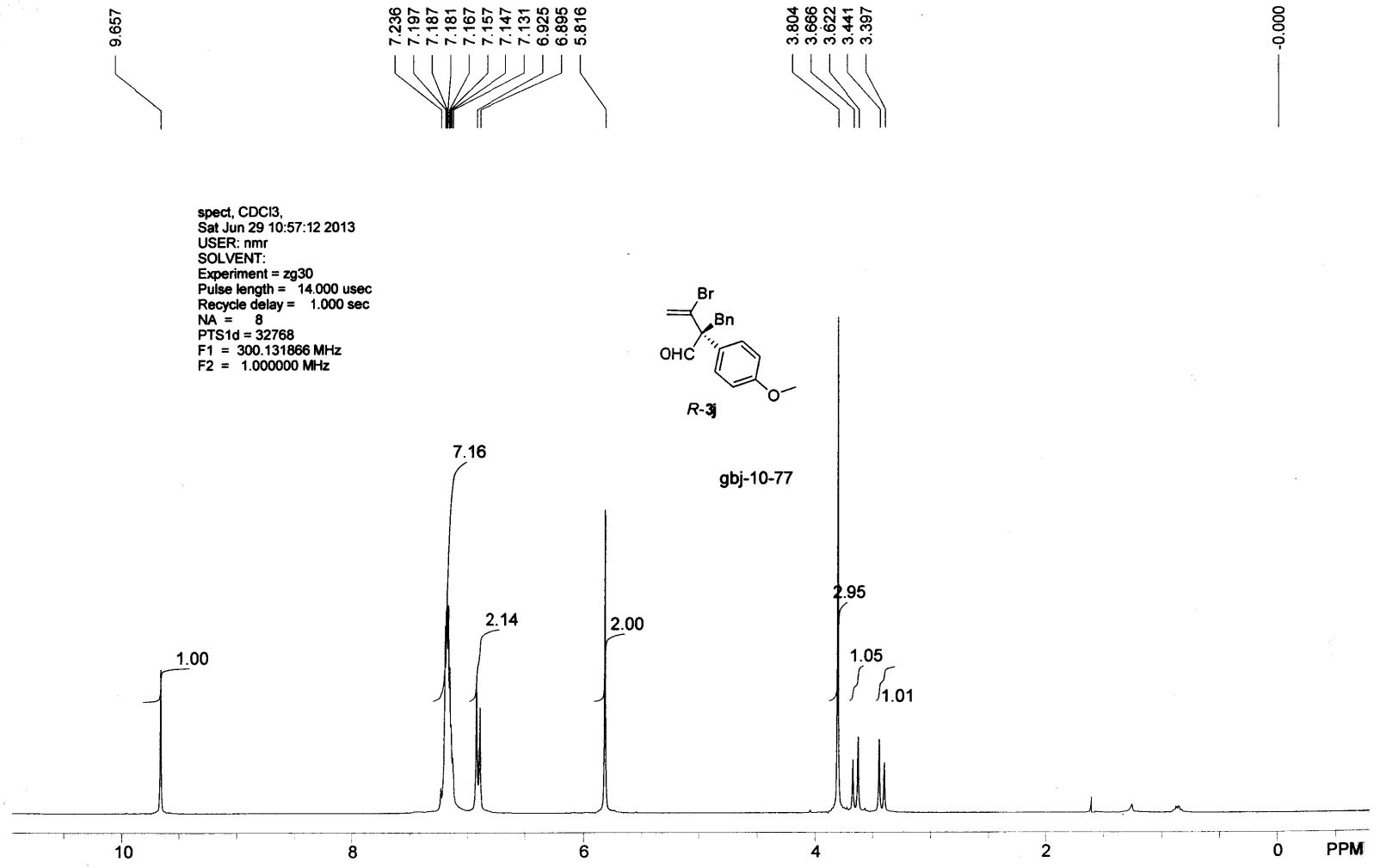


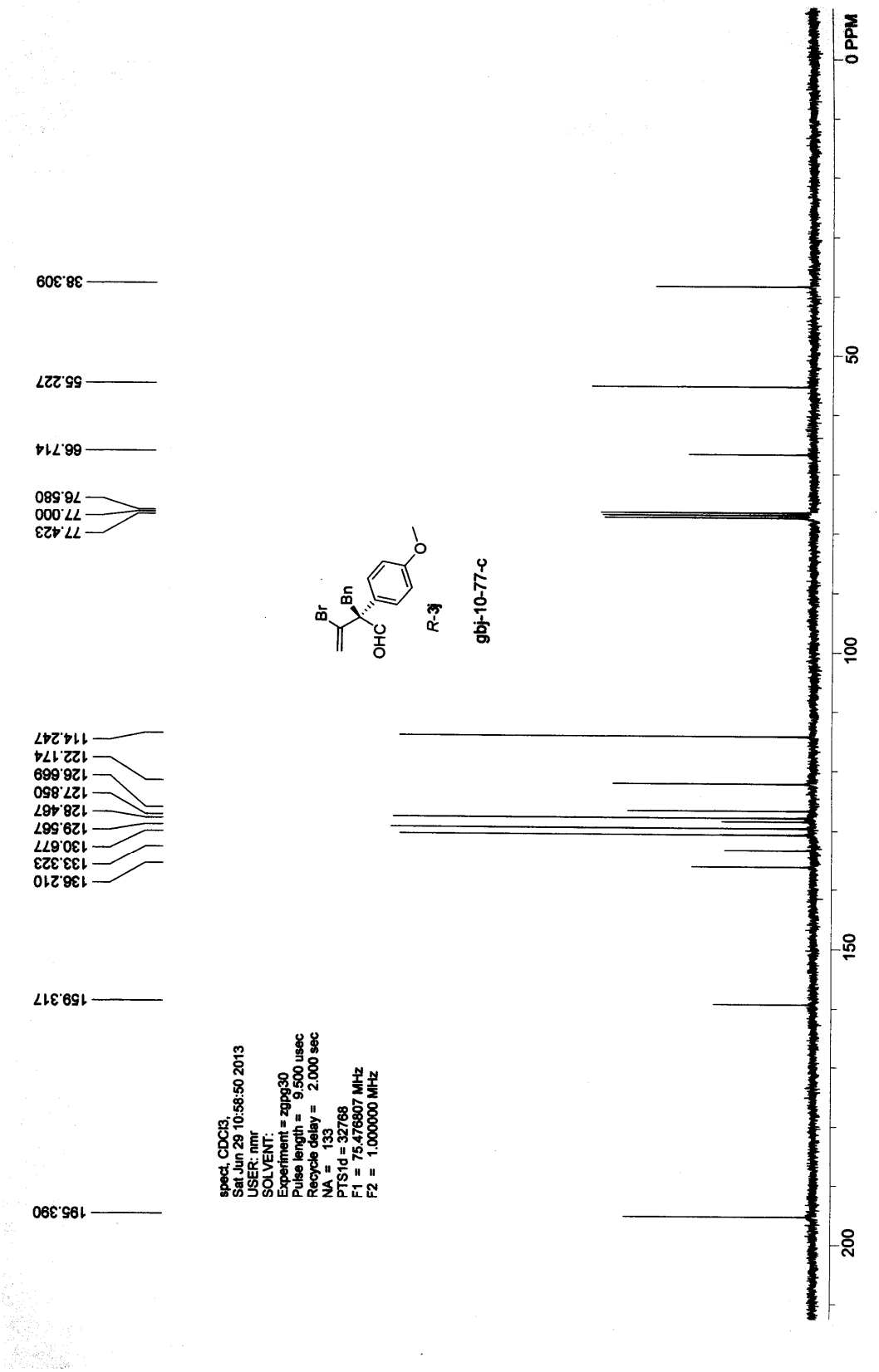
rac-**3i**

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	22.965	BV	0.6883	9544.05078	212.00467	50.0101
2	25.351	VB	0.8402	9540.18652	172.53101	49.9899
Totals :				1.90842e4	384.53568	

```
 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```


9.657
7.236
7.197
7.187
7.181
7.167
7.157
7.147
7.131
6.925
6.895
5.816
3.804
3.666
3.622
3.441
3.397
-0.000
spect, CDCl3,
Sat Jun 29 10:57:12 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
Bn
OHC
O
R-3j
gbj-10-77
7.16
2.14
2.00
2.95
1.05
1.01
1.00
10
8
6
4
2
0
PPM

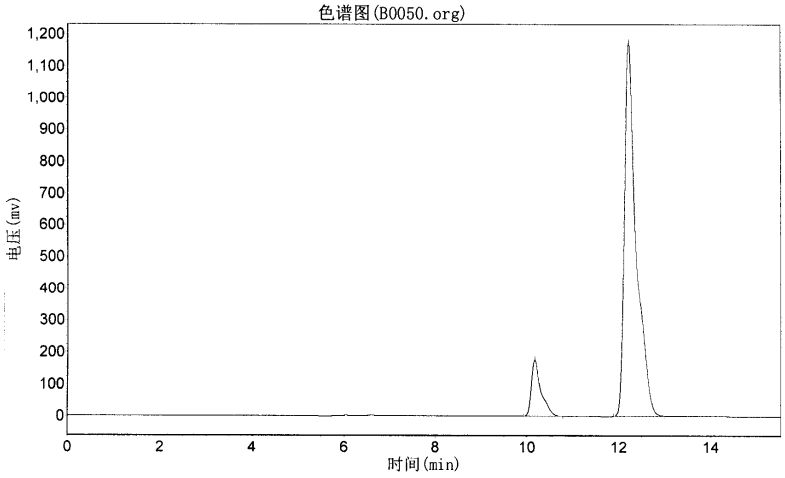

195.390
159.317
136.210
133.323
130.677
129.567
128.467
127.850
126.669
122.174
114.247
77.423
77.000
76.580
66.714
55.227
38.309
spect, CDCl3,
Sat Jun 29 10:58:50 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 133
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
Bn
OHC
O
R-3j
gbj-10-77-c
200
150
100
50
0 PPM

gbj-10-77

实验时间：2014-01-23,19:24:56
谱图文件:D:\浙大智达\N2000\样品\B0050.org

实验者：gbj
报告时间：2014-01-23,19:42:41
积分方法:面积归一法

实验内容简介：
AD-H, n-hexane/iPrOH = 80/20, 230 nm, 0.50 ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		10.180	175589.984	2535402.500	10.9289
2		12.223	1172402.375	20663656.000	89.0711
总计			1347992.359	23199058.500	100.0000

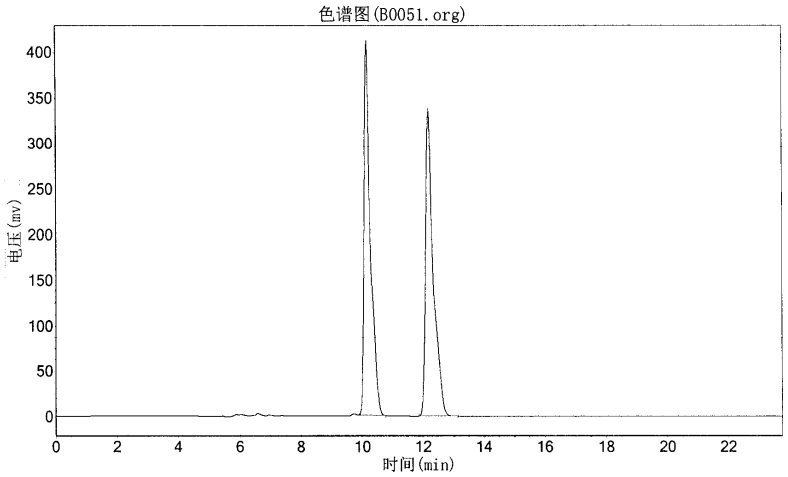
R-3j

2014-01-23

gbj-10-78

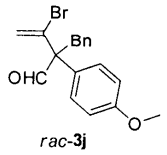
实验时间：2014-01-23,19:44:14
谱图文件:D:\浙大智达\N2000\样品\B0051.org

实验者：gbj
报告时间：2014-01-23,20:09:02
积分方法:面积归一法



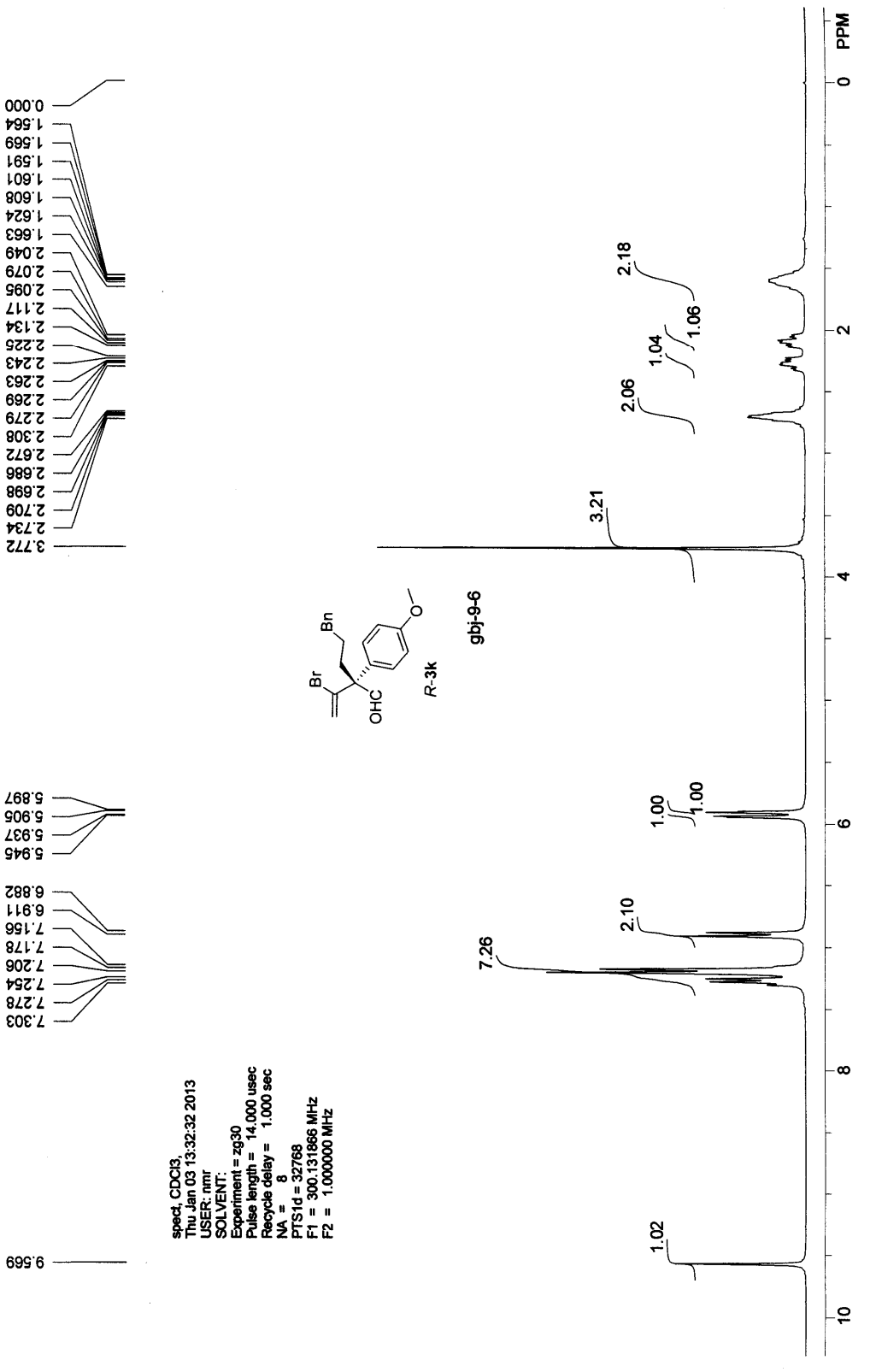
分析结果表

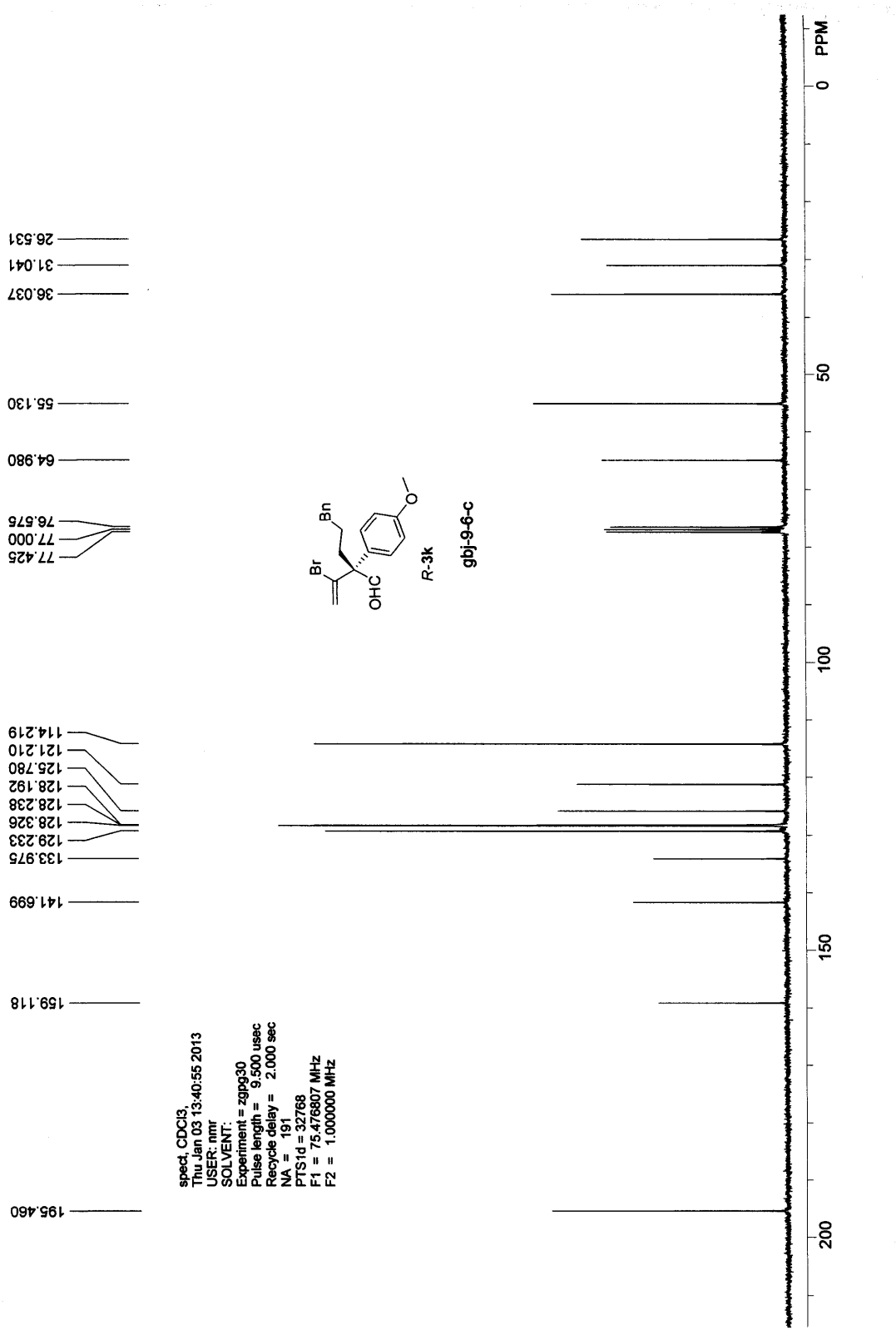
峰号	峰名	保留时间	峰高	峰面积	含量
1		10.155	407719.344	5895146.000	50.0400
2		12.188	335074.781	5885728.500	49.9600
总计			742794.125	11780874.500	100.0000



rac-**3j**

2014-01-23


spect, CDCl3,
Thu Jan 03 13:32:32 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131886 MHz
F2 = 1.000000 MHz
gbj-9-6
R-3k
Br
Bn
OHC
O
9.569
7.303
7.278
7.254
7.206
7.178
7.156
6.911
6.882
5.945
5.937
5.905
5.897
3.772
2.734
2.709
2.698
2.686
2.672
2.308
2.279
2.269
2.263
2.243
2.225
2.134
2.117
2.095
2.079
2.049
1.663
1.624
1.608
1.601
1.591
1.569
1.564
0.000
7.26
3.21
2.18
2.10
2.06
1.06
1.04
1.00
1.00
1.02
PPM

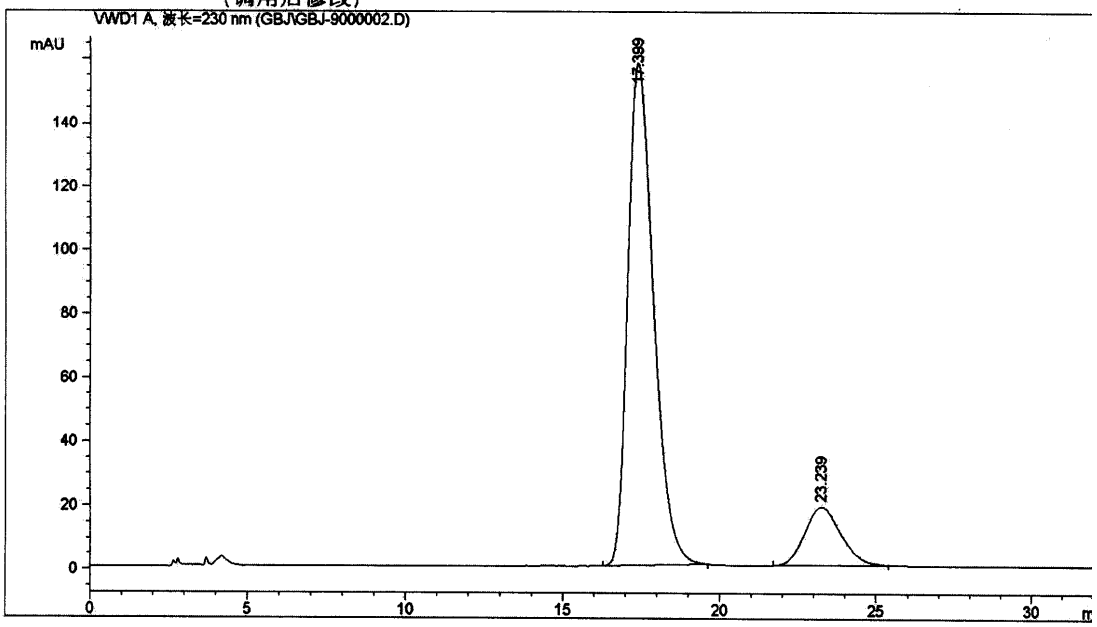

195.460
159.118
141.699
133.975
129.233
128.326
128.238
128.192
125.780
121.210
114.219
77.425
77.000
76.575
64.980
55.130
36.037
31.041
26.531
Br
Bn
OHC
O
R-3k
gbj-9-6-c
sped, CDCl3,
Thu Jan 03 13:40:55 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 191
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
PPM
0
50
100
150
200

据文件　　D:\Chem32\1\DATA\GBJ\GBJ-9000002.D
品名: gbj-9-6

OJ-H; Hexane/iPrOH=80/20; 1.2 ml/min, 230nm

==========

进样日期　　: 2013-1-8 10:49:37
样品名称　　: gbj-9-6　　瓶置　　: 样品瓶1
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\Chem32\1\METHODS\DEF_LC.M
最后修改　　: 2013-1-8 10:56:20　:　gbj
(调用后修改)



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面积百分比报告

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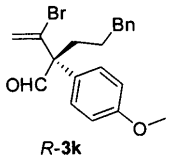
排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子



R-3k

信号 1: VWD1 A, 波长=230 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	17.399	BB	0.8770	8966.60059	157.45175	85.7440
2	23.239	BB	1.0974	1490.81042	18.35784	14.2560

总量 :　　1.04574e4　　175.80960

==========

*** 报告结束 ***

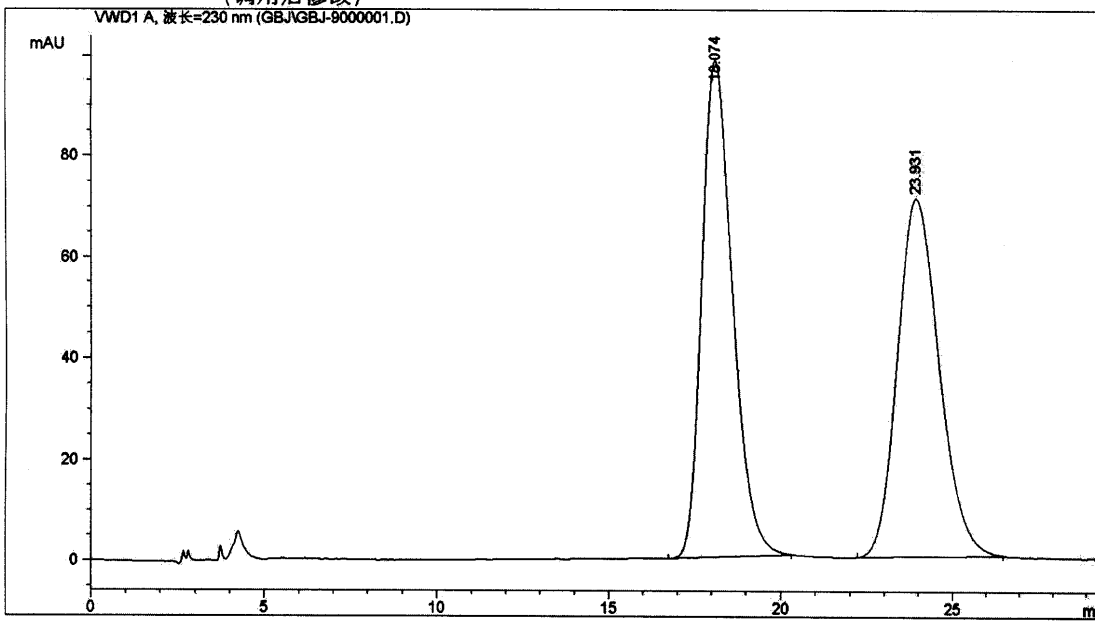
器 1 2013-1-8 11:22:51　gbj　　页 1 / 1

据文件　　D:\CHEM32\1\DATA\GBJ\GBJ-9000001.D
品名: gbj-9-1

OJ-H; Hexane/iPrOH=80/15; 1.2 ml/min, 230nm

==========

进样日期	: 2013-1-8 10:17:12	
样品名称	: gbj-9-1	位置　: 样品瓶1
操作者	: gbj	
仪器	: 仪器 1	
采集方法	: D:\Chem32\1\METHODS\DEF_LC.M	
最后修改	: 2013-1-8 10:09:09 : lqk	
	(调用后修改)	
分析方法	: D:\Chem32\1\METHODS\DEF_LC.M	
最后修改	: 2013-1-8 10:56:20 : gbj	
	(调用后修改)	



==========

面积百分比报告

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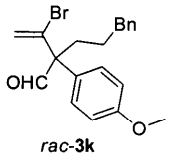
排序　　: 信号
乘积因子　　: 1.0000
稀释因子　　: 1.0000
内标使用乘积因子和稀释因子



rac-3k

信号 1: VWD1 A, 波长=230 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	18.074	BB	0.9349	6076.61914	98.14552	50.0421
2	23.931	BB	1.3125	6066.39941	70.71217	49.9579
总量 :				1.21430e4	168.85770	

==========

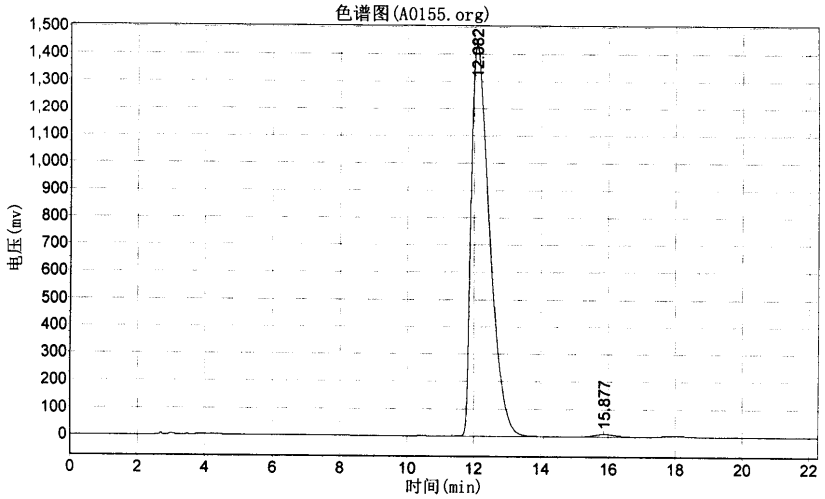
*** 报告结束 ***

gbj-9-6-re''

实验时间：2013-09-29, 15:16:24
谱图文件:D:\浙大智达\N2000\样品\A0155.org

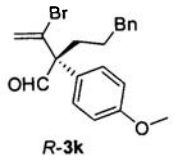
实验者：gbj
报告时间：2013-09-29, 15:40:08
积分方法:面积归一法

实验内容简介：
OJ-H, n-Hexane:i-PrOH=80/20, 1.2 mL/min, 230 nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.082	1433178.500	54565620.000	99.3426
2		15.877	8745.838	361062.563	0.6574
总计			1441924.338	54926682.563	100.0000



R-3k

Recrystallization from CH_2Cl_2/n-hexane for three times

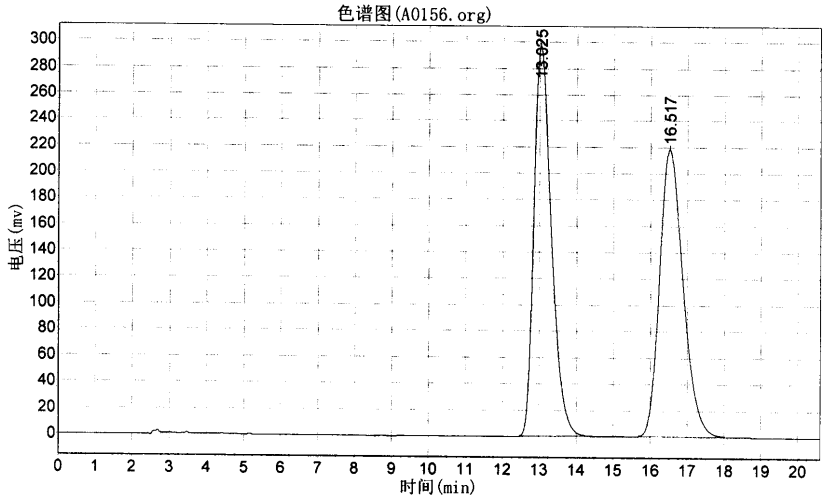
2013-09-29

gbj-9-6-re''-rac

实验时间：2013-09-29,15:41:32
谱图文件:D:\浙大智达\N2000\样品\A0156.org

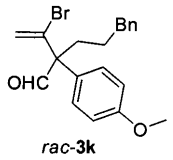
实验者：gbj
报告时间：2013-09-29,16:02:54
积分方法:面积归一法

实验内容简介：
OJ-H, n-Hexane:i-PrOH=80/20, 1.2 mL/min, 230 nm



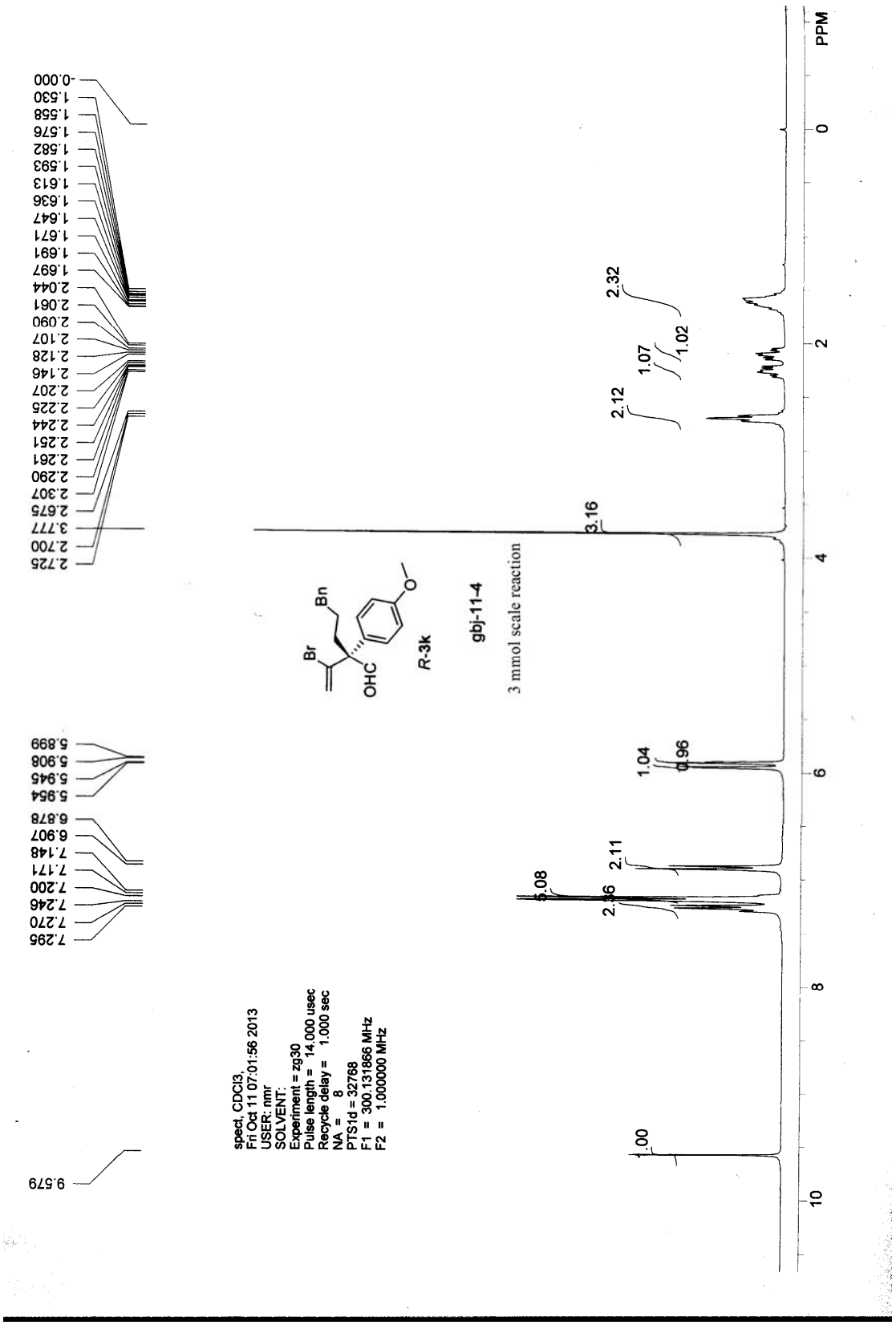
分析结果表

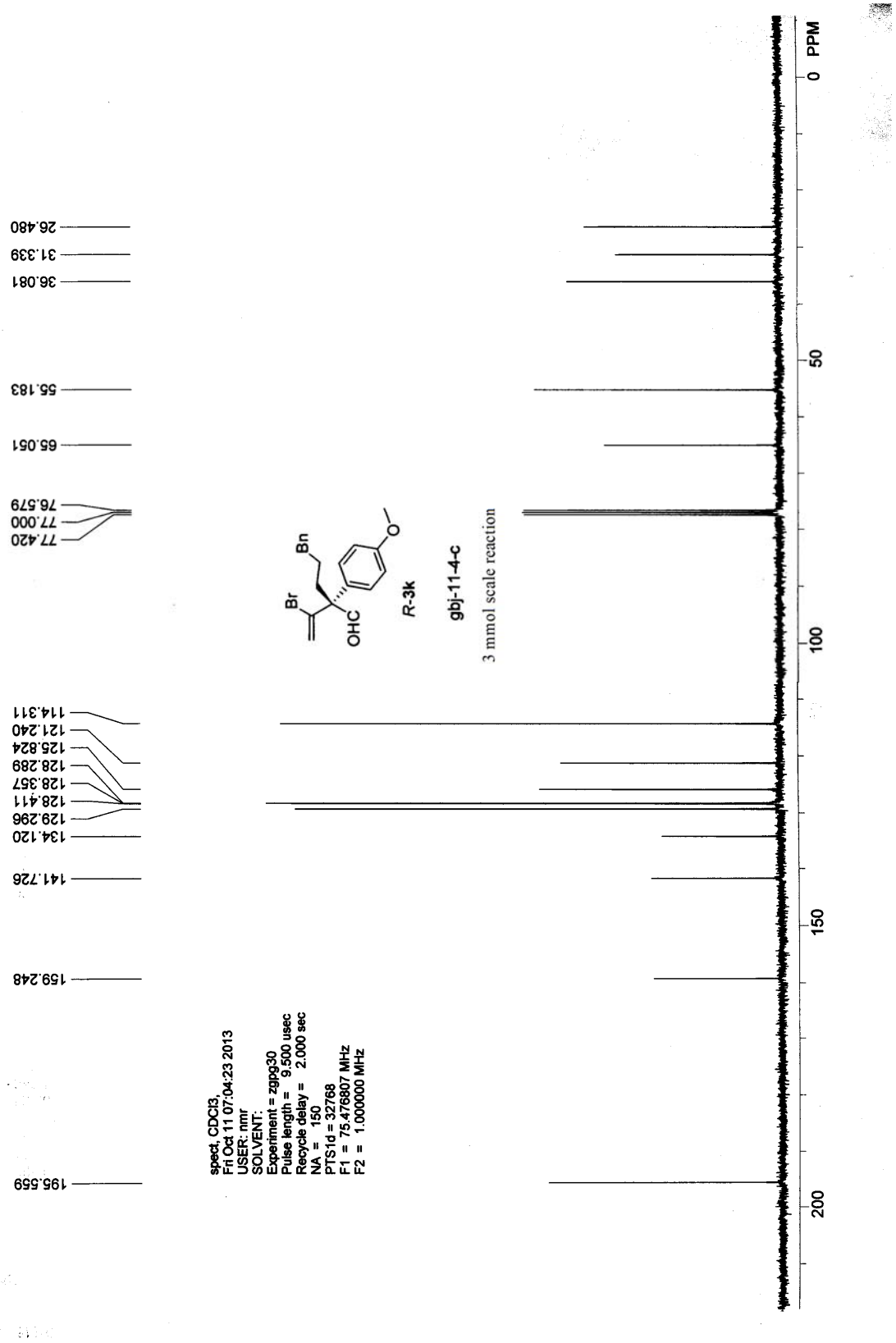
峰号	峰名	保留时间	峰高	峰面积	含量
1		13.025	296638.094	9749461.000	50.4013
2		16.517	217890.938	9594220.000	49.5987
总计			514529.031	19343681.000	100.0000



rac-**3k**

2013-09-29


spect, CDCl3,
Fri Oct 11 07:01:56 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-11-4
3 mmol scale reaction
R-3k
Br
Bn
OHC
O
PPM

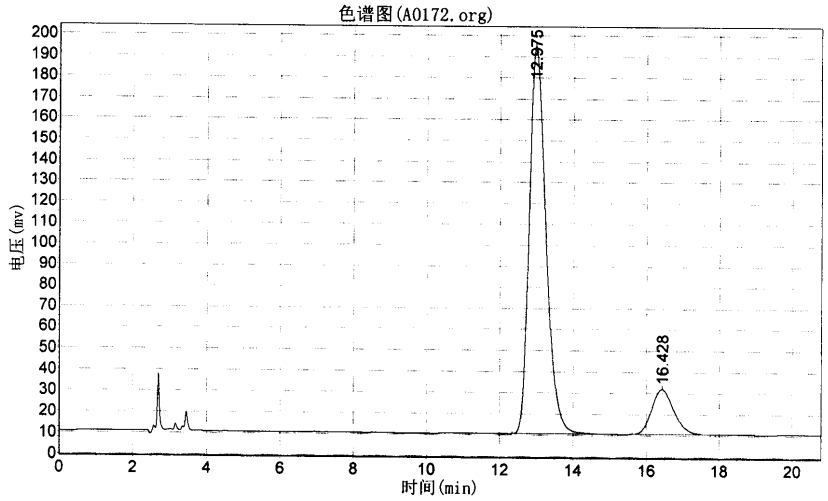

26.480
31.339
36.081
55.183
65.051
76.579
77.000
77.420
114.311
121.240
125.824
128.289
128.357
128.411
129.296
134.120
141.726
159.248
195.559
spect, CDCl3,
Fri Oct 11 07:04:23 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 150
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
Bn
OHC
O
R-3k
gbj-11-4-c
3 mmol scale reaction
0 PPM
50
100
150
200

gbj-11-4

实验时间：2013-10-12,9:46:12
谱图文件:D:\浙大智达\N2000\样品\A0172.org

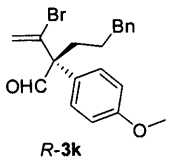
实验者：gbj
报告时间：2013-10-12,10:07:48
积分方法:面积归一法

实验内容简介：
OJ-H, n-Hexane:i-PrOH=80/20, 1.2 mL/min, 230 nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.975	184242.047	5814136.000	86.5628
2		16.428	21283.324	902530.063	13.4372
总计			205525.371	6716666.063	100.0000



R-3k

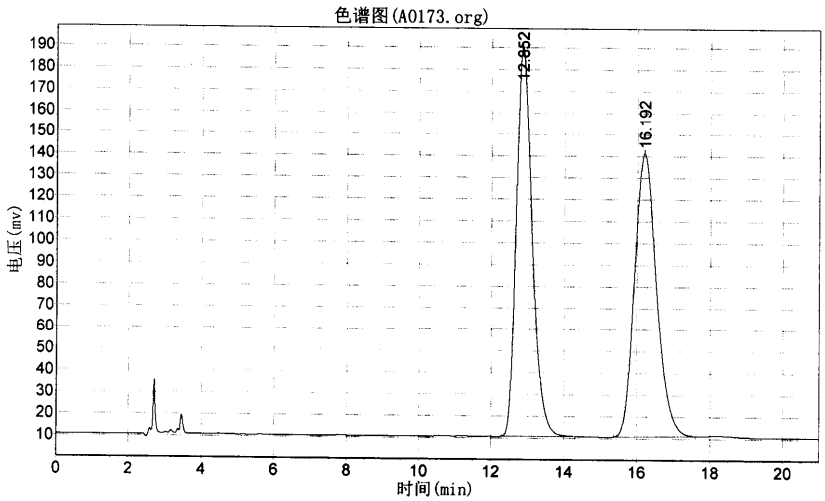
2013-10-12

gbj-11-4-rac

实验时间：2013-10-12,10:08:53
谱图文件:D:\浙大智达\N2000\样品\A0173.org

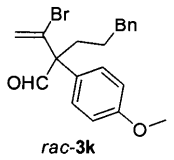
实验者：gbj
报告时间：2013-10-12,10:30:40
积分方法:面积归一法

实验内容简介：
OJ-H, n-Hexane:i-PrOH=80/20, 1.2 mL/min, 230 nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.852	178652.641	5448835.000	50.1216
2		16.192	130059.906	5422397.500	49.8784
总计			308712.547	10871232.500	100.0000



rac-**3k**

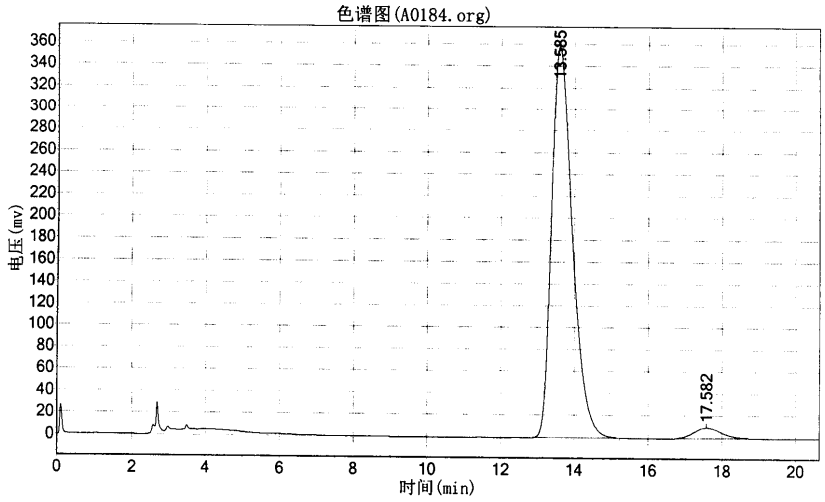
2013-10-12

gbj-11-4jiejing2

实验时间：2013-11-06,9:40:05
谱图文件:D:\浙大智达\N2000\样品\A0184.org

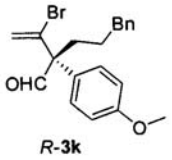
实验者：gbj
报告时间：2013-11-06,10:01:42
积分方法:面积归一法

实验内容简介：
OJ-H, n-Hexane:i-PrOH=80/20, 1.2 mL/min, 230 nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		13.585	357801.625	14014994.000	96.7883
2		17.582	9261.667	465058.344	3.2117
总计			367063.292	14480052.344	100.0000



R-3k

Recrystallization from CH_2Cl_2/n-hexane for twice

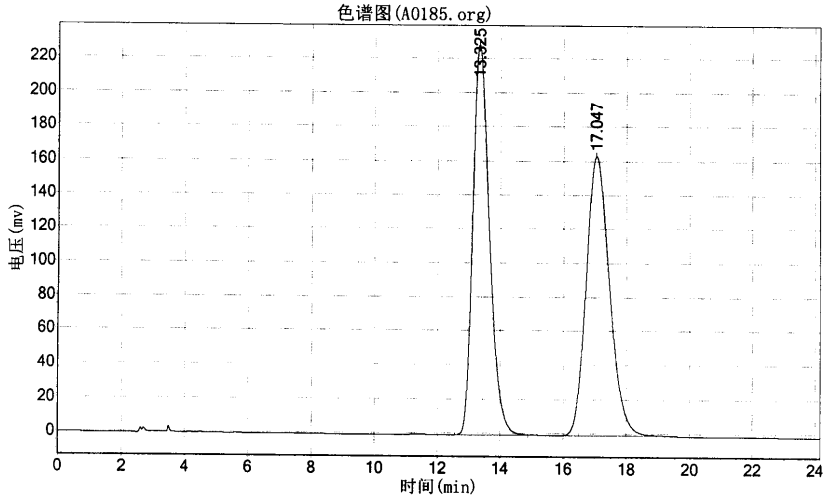
2013-11-06

gbj-11-4-rac

实验时间：2013-11-06,10:03:01
谱图文件:D:\浙大智达\N2000\样品\A0185.org

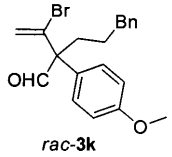
实验者：gbj
报告时间：2013-11-06,10:28:20
积分方法:面积归一法

实验内容简介：
OJ-H, n-Hexane:i-PrOH=80/20, 1.2 mL/min, 230 nm



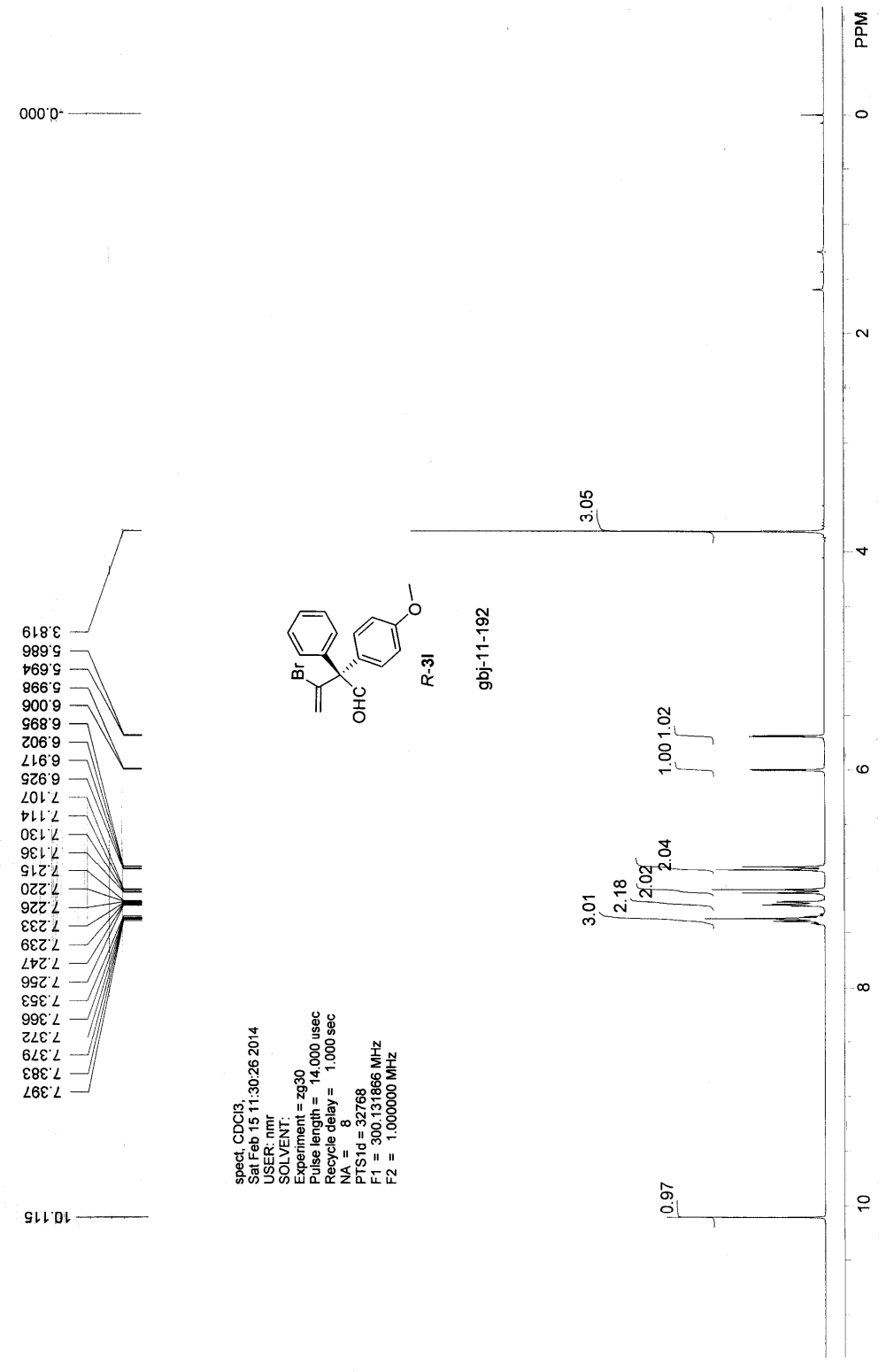
分析结果表

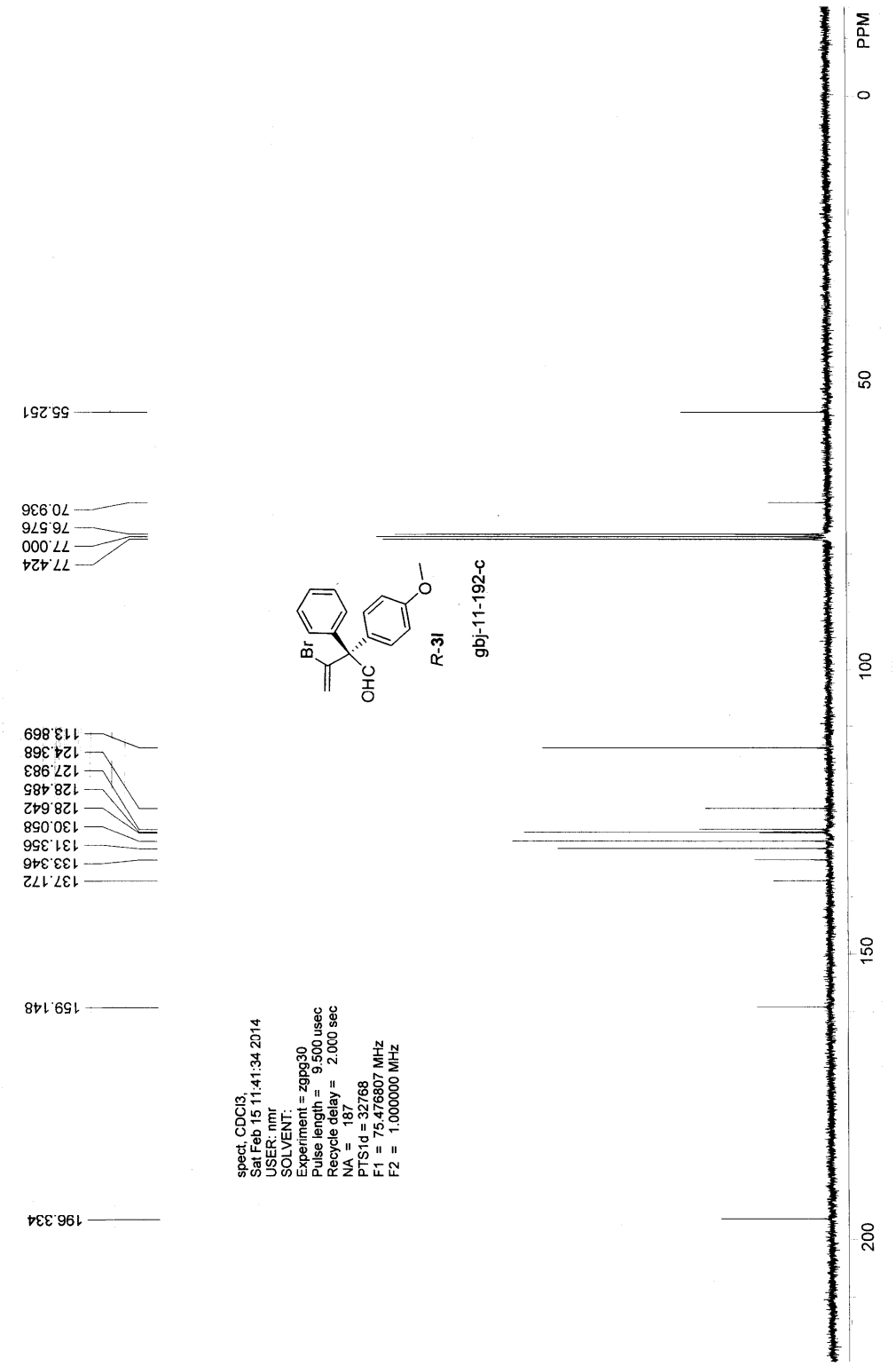
峰号	峰名	保留时间	峰高	峰面积	含量
1		13.325	228785.078	8396188.000	50.0590
2		17.047	163688.422	8376411.000	49.9410
总计			392473.500	16772599.000	100.0000



rac-**3k**

2013-11-06


spect. CDCl3
Sat Feb 15 11:30:26 2014
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-11-192
R-3l
Br
OHC
O
3.05
3.01
2.18
2.02
2.04
1.00
1.02
0.97
PPM

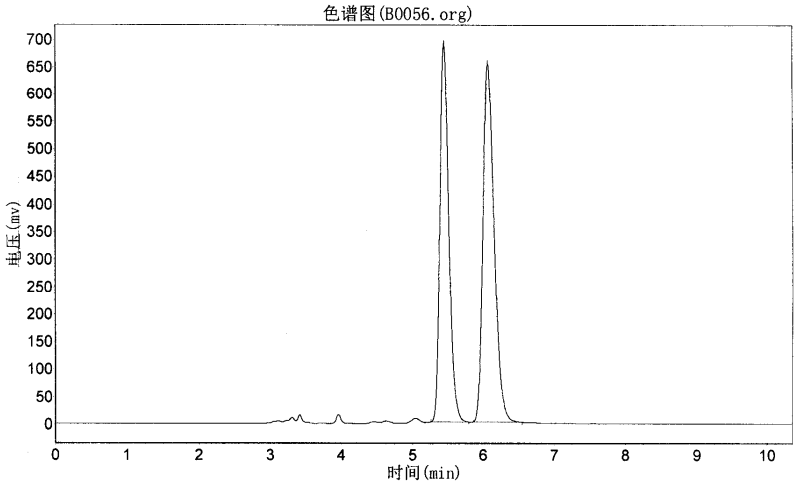

55.251
70.936
76.576
77.000
77.424
113.869
124.368
127.983
128.485
128.642
130.058
131.356
133.346
137.172
159.148
196.334
spect, CDCl3,
Sat Feb 15 11:41:34 2014
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 187
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
OHC
O
R-3l
gbj-11-192-c
0
50
100
150
200
PPM

gbj-11-192

实验时间：2014-02-15,19:09:08
谱图文件:D:\浙大智达\N2000\样品\B0056.org

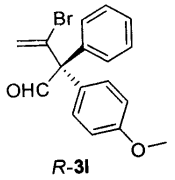
实验者：gbj
报告时间：2014-02-23,13:15:43
积分方法:面积归一法

实验内容简介：
AS-H, n-hexane/iPrOH = 80/20, 230 nm, 1.0 ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		5.452	687774.875	6036592.000	45.8403
2		6.072	650860.063	7132147.000	54.1597
总计			1338634.938	13168739.000	100.0000



R-3l

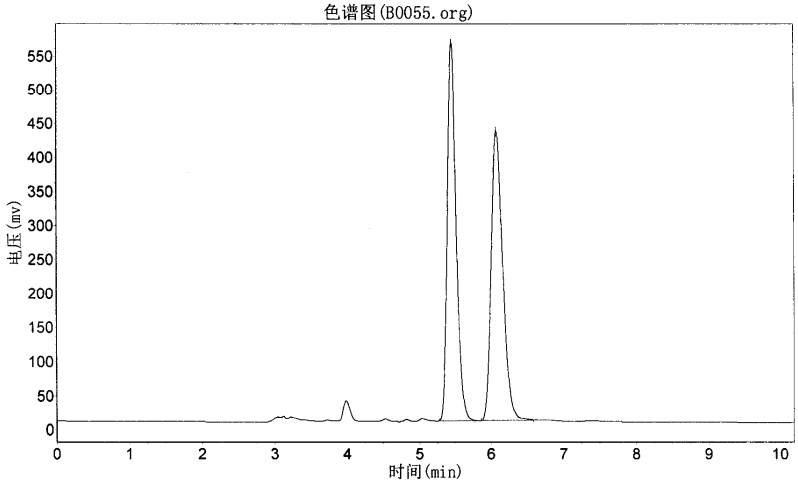
2014-02-23

gbj-11-144

实验时间：2014-02-15,18:56:20
谱图文件:D:\浙大智达\N2000\样品\B0055.org

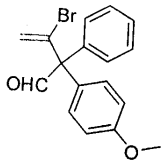
实验者：gbj
报告时间：2014-02-23,13:17:14
积分方法:面积归一法

实验内容简介:
AS-H, n-hexane/iPrOH = 80/20, 230 nm, 1.0 ml/min



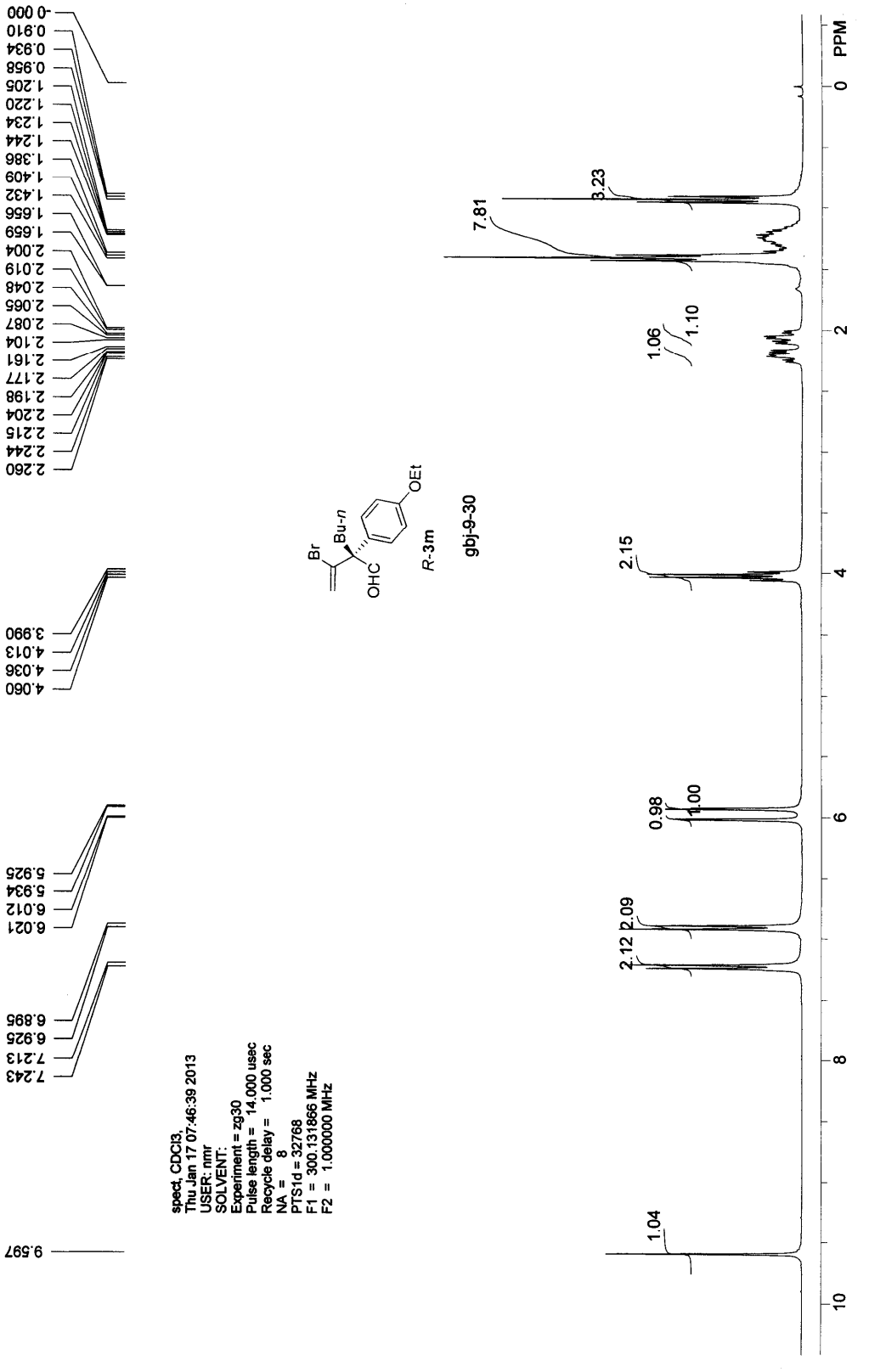
分析结果表

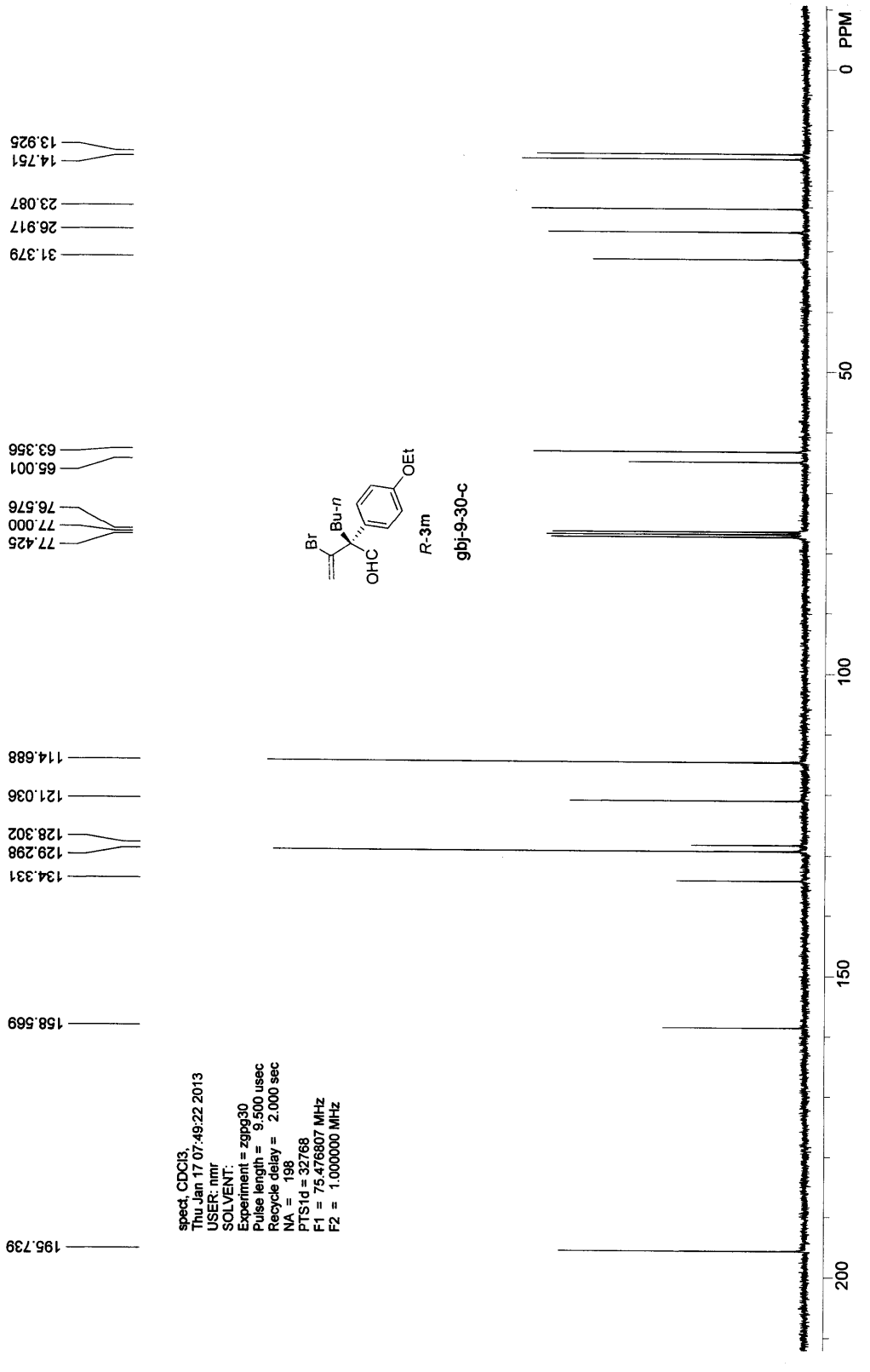
峰号	峰名	保留时间	峰高	峰面积	含量
1		5.453	556580.688	4727065.500	50.0557
2		6.075	425159.719	4716546.000	49.9443
总计			981740.406	9443611.500	100.0000



rac-**3l**

2014-02-23

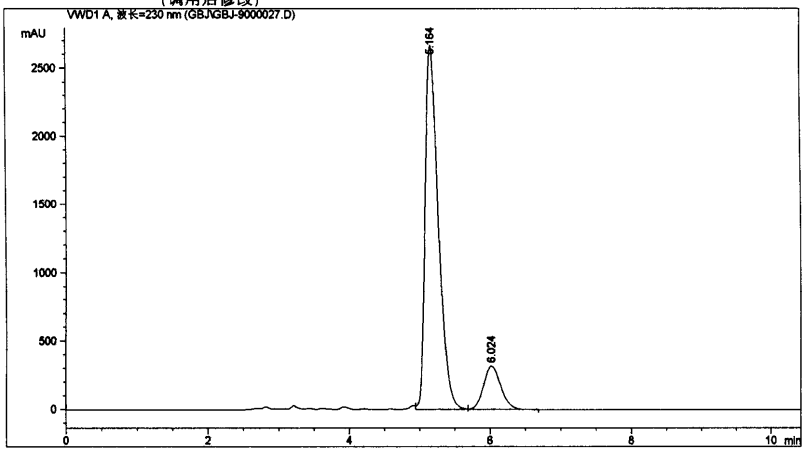

spect, CDCl3,
Thu Jan 17 07:46:39 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
Bu-n
OHC
OEt
R-3m
gbj-9-30
7.81
3.23
2.15
1.06
1.10
0.98
1.00
2.12
2.09
1.04
PPM


13.925
14.751
23.087
26.917
31.379
63.356
65.001
76.576
77.000
77.425
114.688
121.036
128.302
129.298
134.331
158.569
195.739
spect, CDCl3,
Thu Jan 17 07:49:22 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 198
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
Bu-n
OHC
OEt
R-3m
gbj-9-30-c
0 PPM
50
100
150
200

数据文件　　D:\Chem32\1\DATA\GBJ\GBJ-9000027.D
样品名: gbj-9-30

OJ-H; Hexane/iPrOH=80/20; 1.2 ml/min, 230 nm

进样日期　　: 2013-1-17 19:50:24
样品名称　　: gbj-9-30　　位置　: -
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2013-1-17 19:25:35 : lxj
(调用后修改)



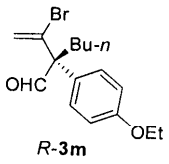
面积百分比报告

排序　　: 信号
乘积因子　　: 1.0000
稀释因子　　: 1.0000
内标使用乘积因子和稀释因子

信号 1: VWD1 A, 波长=230 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	5.164	VV	0.1978	3.45495e4	2665.43726	86.3428
2	6.024	VB	0.2672	5464.83545	316.11801	13.6572

总量 :　　4.00144e4　2981.55527



R-**3m**

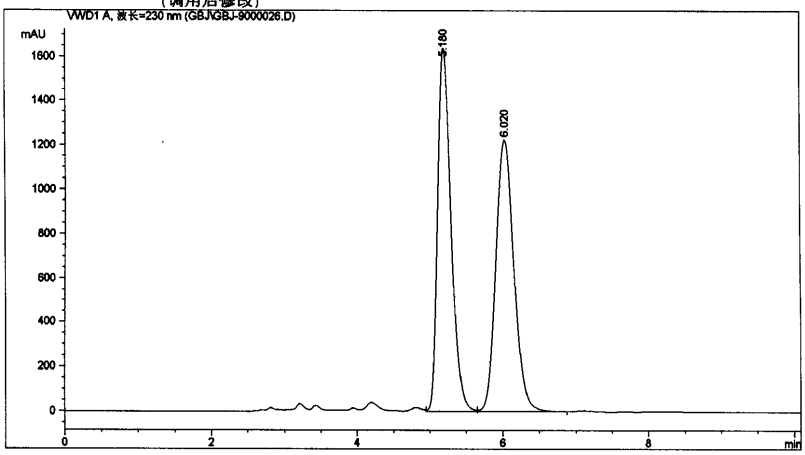
*** 报告结束 ***

仪器 1 2013-1-17 20:01:23 gbj　　页 1 / 1

数据文件　　D:\Chem32\1\DATA\GBJ\GBJ-9000026.D
样品名: gbj-9-29

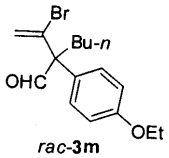
OJ-H; Hexane/iPrOH=80/20; 1.2 ml/min, 230 nm

进样日期　　: 2013-1-17 19:37:40
样品名称　　: gbj-9-29　　位置　:　-
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2013-1-17 19:25:35　:　lxj
(调用后修改)



面积百分比报告

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子



rac-3m

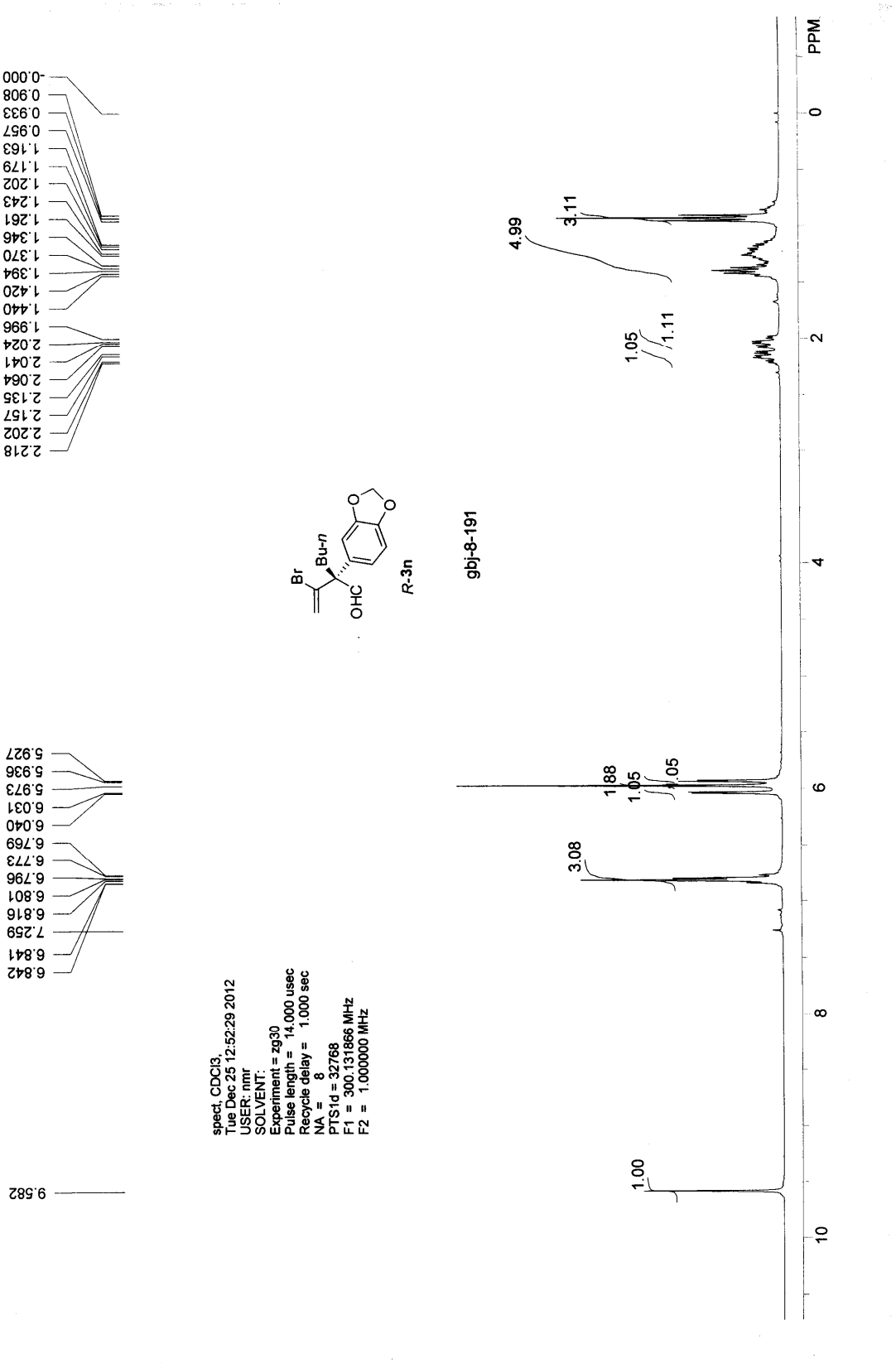
信号 1: VWD1 A, 波长=230 nm

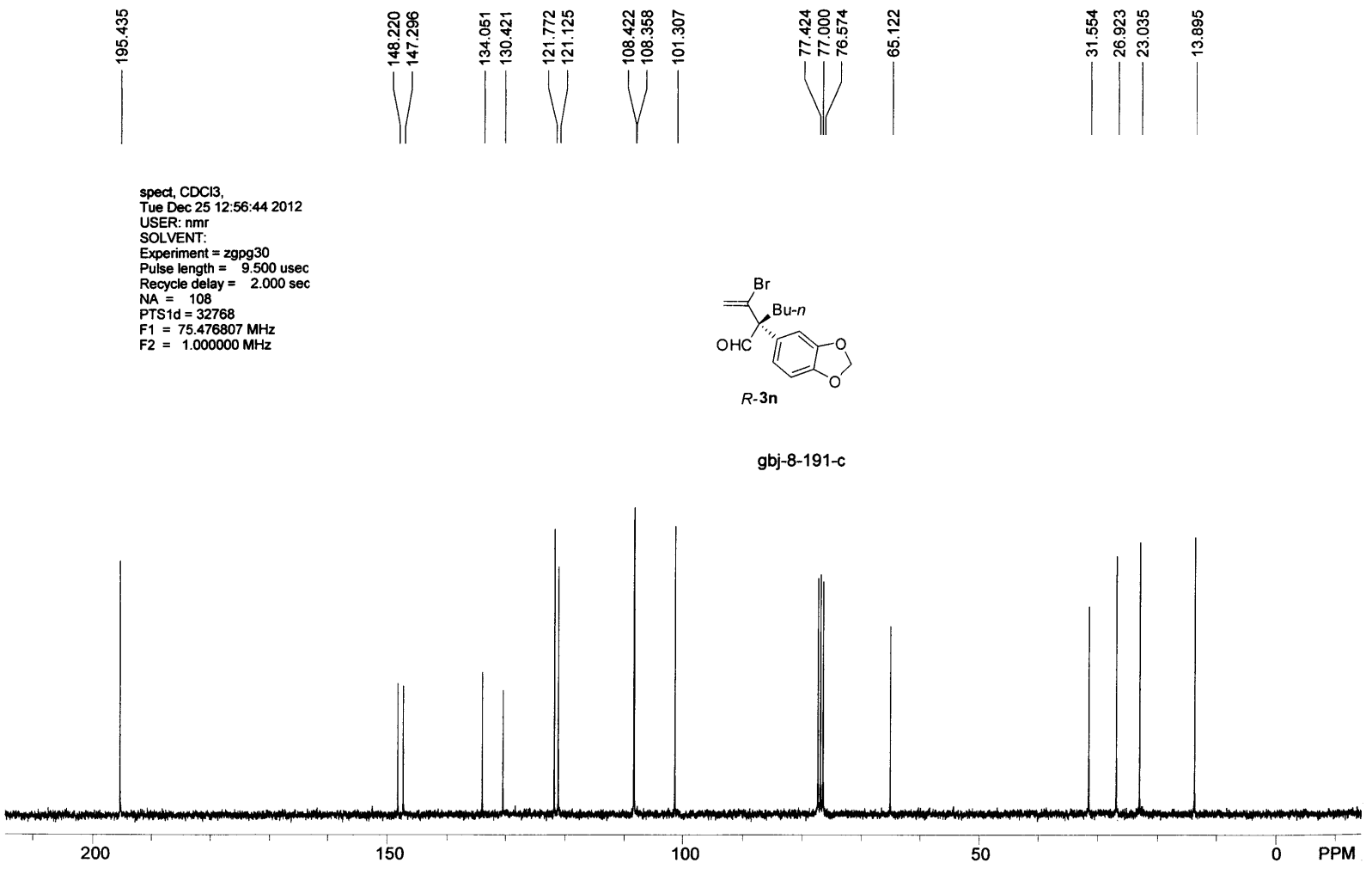
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	5.180	VV	0.1954	2.09622e4	1643.35706	49.7885
2	6.020	VV	0.2671	2.11403e4	1223.74927	50.2115

总量 :　　4.21024e4　2867.10632

*** 报告结束 ***

仪器 1 2013-1-17 19:48:12　gbj　　页　1 / 1


spect, CDCl3,
Tue Dec 25 12:52:29 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-8-191
R-3n
Br
Bu-n
OHC
O
O
PPM
0
2
4
6
8
10
4.99
3.11
1.05
1.11
1.88
1.05
1.05
3.08
1.00
9.582
7.259
6.842
6.841
6.816
6.801
6.796
6.773
6.769
6.040
6.031
5.973
5.936
5.927
2.218
2.202
2.157
2.135
2.064
2.041
2.024
1.996
1.440
1.420
1.394
1.370
1.346
1.261
1.243
1.202
1.179
1.163
0.957
0.933
0.908
-0.000

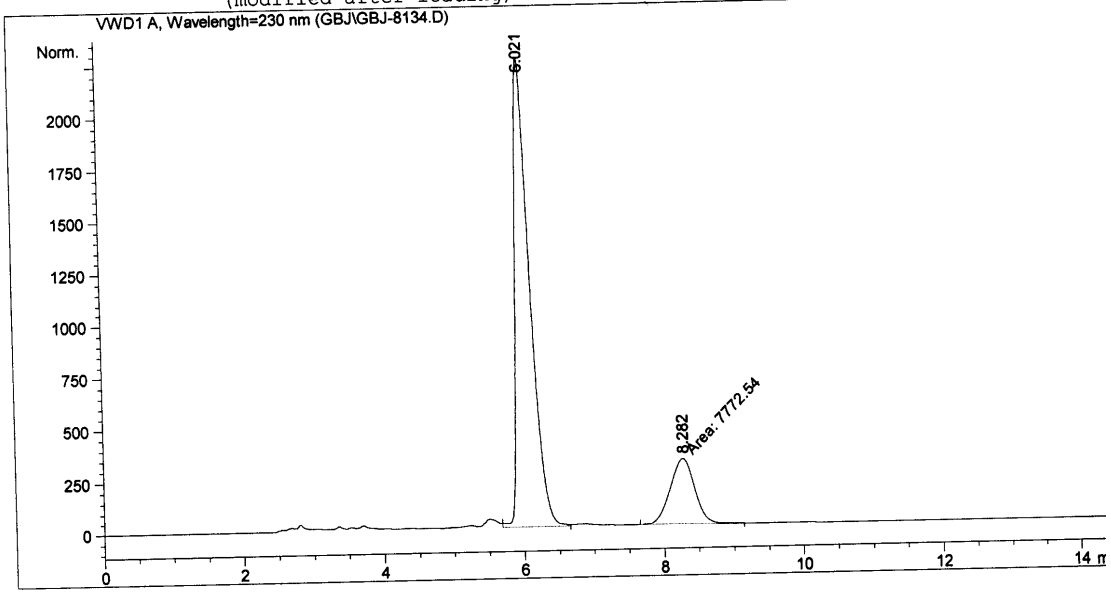

195.435
148.220
147.296
134.051
130.421
121.772
121.125
108.422
108.358
101.307
77.424
77.000
76.574
65.122
31.554
26.923
23.035
13.895
spect, CDCl3,
Tue Dec 25 12:56:44 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 108
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
Bu-n
OHC
O
O
R-3n
gbj-8-191-c
200
150
100
50
0
PPM

File D:\HPCHEM\1\DATA\GBJ\GBJ-8134.D

Sample Name: gbj-8-19

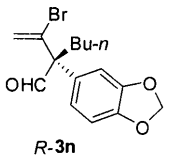
OJ-H, n-Hexane:i-PrOH= 80/20, 1.2 mL/min, 230 nm

```
Injection Date  : 12/25/2012 1:19:20 PM
Sample Name     : gbj-8-191                    Location :  -
Acq. Operator   : gbj
Method          : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/25/2012 12:47:49 PM by gbj
                  (modified after loading)
```



Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```



R-**3n**

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.021	VV	0.2237	3.32731e4	2266.35791	81.0637
2	8.282	MM	0.4118	7772.53711	314.56506	18.9363
Totals :				4.10456e4	2580.92297	

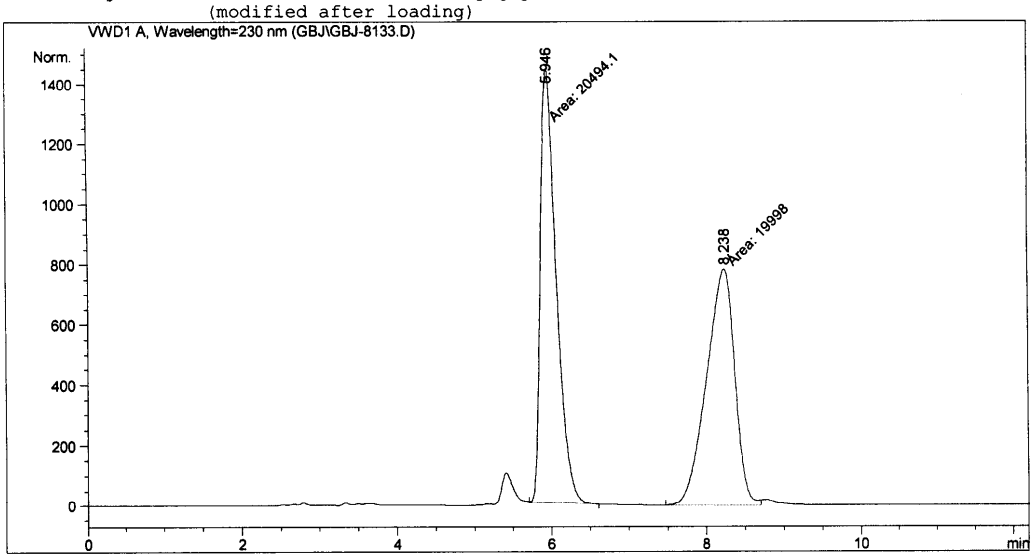
Results obtained with enhanced integrator!

*** End of Report ***

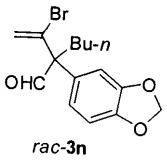
Data File D:\HPCHEM\1\DATA\GBJ\GBJ-8133.D Sample Name: gbj-8-190

OJ-H, n-Hexane:i-PrOH= 80/20, 1.2 mL/min, 230 nm

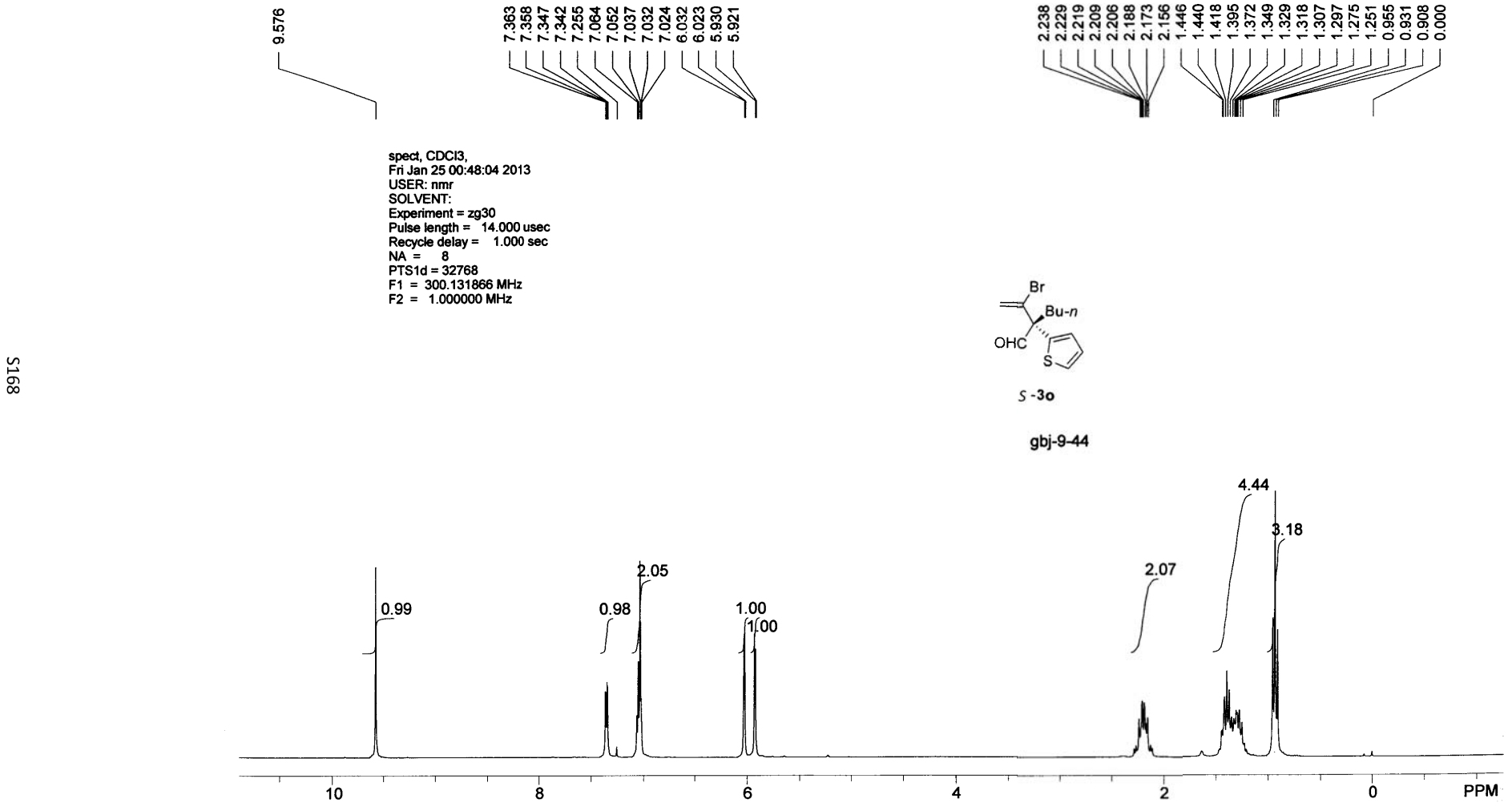
```
==========================================================================
Injection Date  : 12/25/2012 1:04:30 PM
Sample Name     : gbj-8-190                    Location :   -
Acq. Operator   : gbj
Method          : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 12/25/2012 12:47:49 PM by gbj
                  (modified after loading)
```

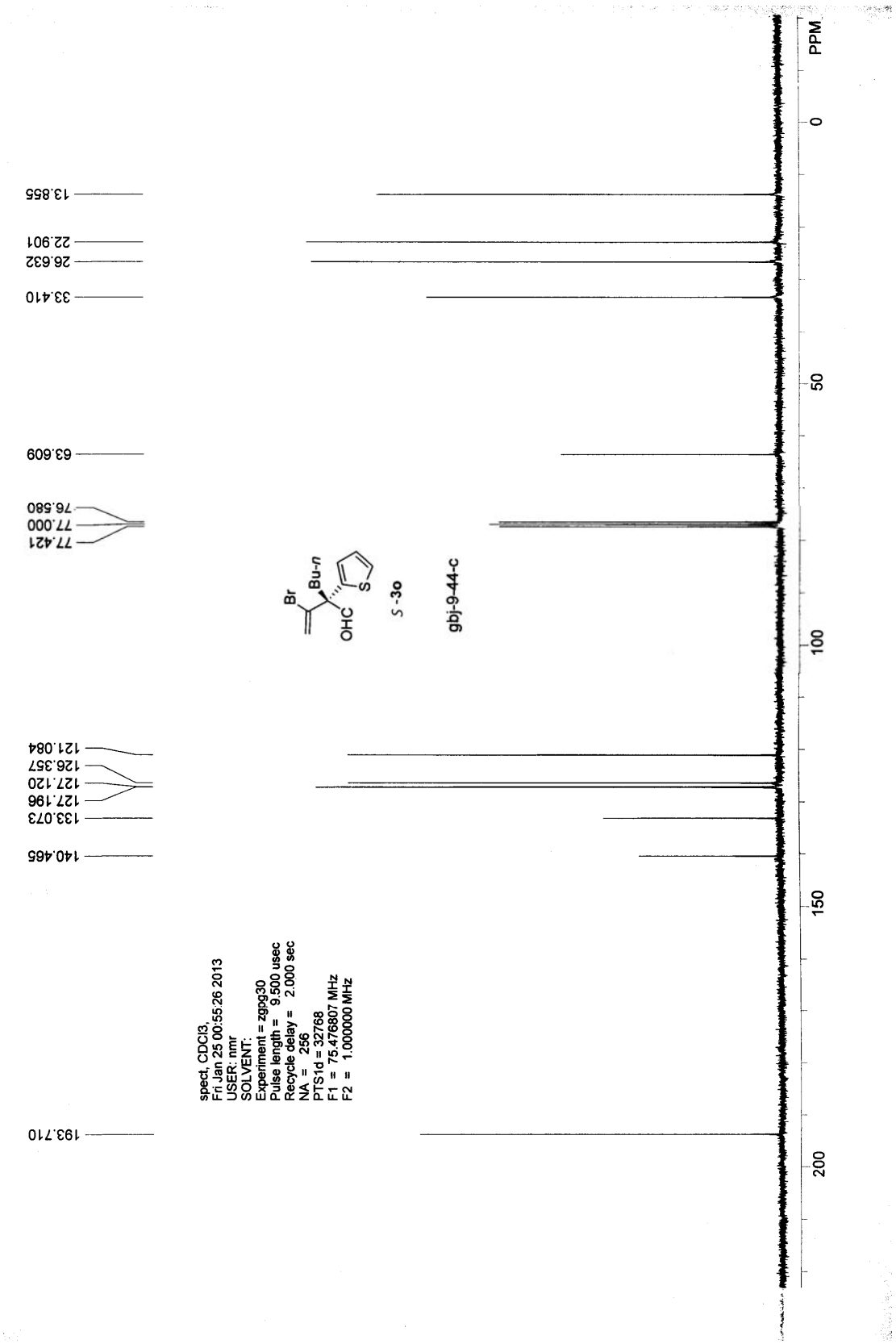



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   5.946 MM    0.2371 2.04941e4  1440.64673  50.6126
   2   8.238 MM    0.4241 1.99980e4   785.84131  49.3874

Totals :                  4.04921e4  2226.48804

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```



rac-**3n**

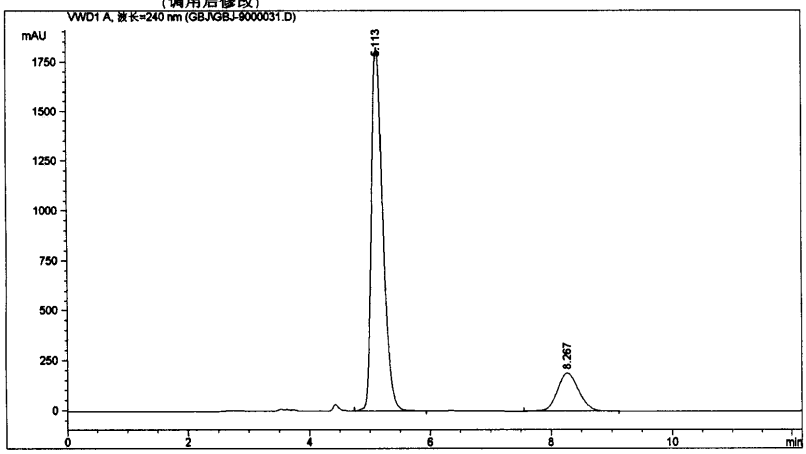

9.576
7.363
7.358
7.347
7.342
7.255
7.064
7.052
7.037
7.032
7.024
6.032
6.023
5.930
5.921
2.238
2.229
2.219
2.209
2.206
2.188
2.173
2.156
1.446
1.440
1.418
1.395
1.372
1.349
1.329
1.318
1.307
1.297
1.275
1.251
0.955
0.931
0.908
0.000
spect, CDCl3,
Fri Jan 25 00:48:04 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
Br
Bu-n
OHC
S
S-3o
gbj-9-44
0.99
0.98
2.05
1.00
1.00
2.07
4.44
3.18
10
8
6
4
2
0
PPM


13.855
22.901
26.632
33.410
63.609
76.580
77.000
77.421
121.084
126.357
127.120
127.196
133.073
140.465
193.710
Br
Bu-n
OHC
S
S-3o
gbj-9-44-c
spect, CDCl3,
Fri Jan 25 00:55:26 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 256
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
PPM
0
50
100
150
200

数据文件　　D:\Chem32\1\DATA\GBJ\GBJ-9000031.D
样品名：gbj-9-44

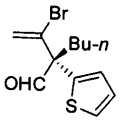
OJ-H; Hexane/iPrOH =80/20; 1.2 ml/min, 230 nm

进样日期　　: 2013-1-28 9:33:42
样品名称　　: gbj-9-44　　位置　:　-
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2013-1-28 9:06:37　:　lqk
(调用后修改)



面积百分比报告

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子



S-**3o**

信号 1: VWD1 A, 波长=240 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	5.113	VV	0.2029	2.39923e4	1824.84106	83.7636
2	8.267	BB	0.3799	4650.55811	189.21135	16.2364

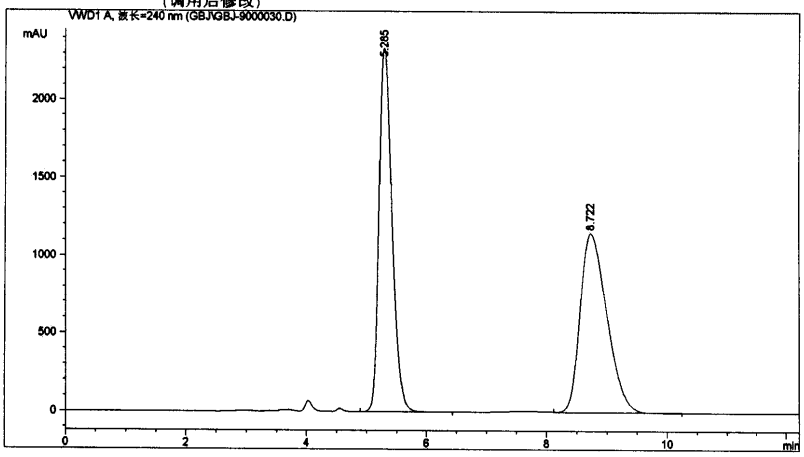
总量 :　　2.86429e4　2014.05241

*** 报告结束 ***

数据文件　　D:\Chem32\1\DATA\GBJ\GBJ-9000030.D
样品名: gbj-9-51

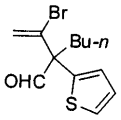
OJ-H; Hexane/iPrOH =80/20; 1.2 ml/min, 230 nm

进样日期　　: 2013-1-28 9:19:43
样品名称　　: gbj-9-51　　位置　:　-
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2013-1-28 9:06:37　:　lqk
(调用后修改)



面积百分比报告

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子



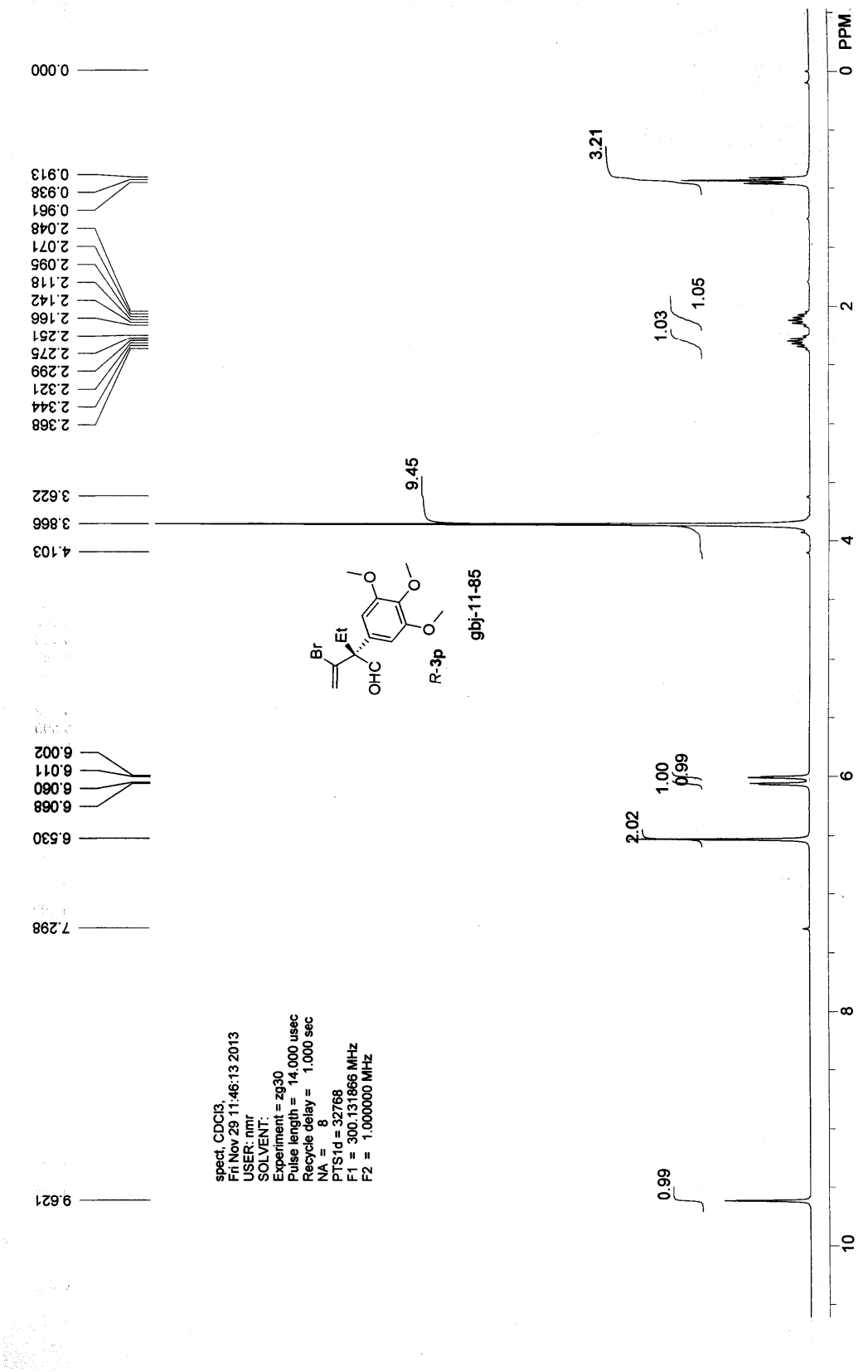
rac-**3o**

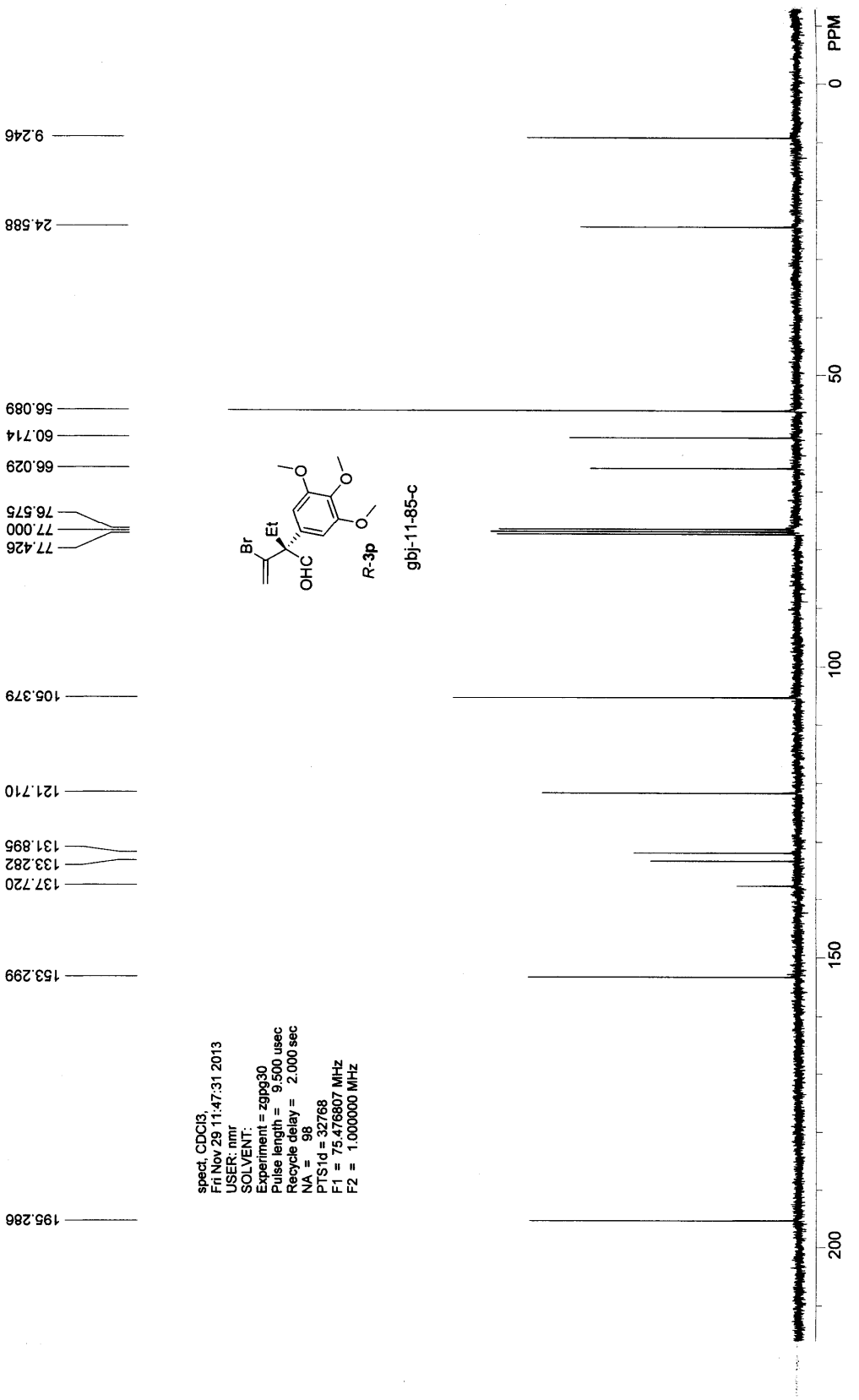
信号 1: VWD1 A, 波长=240 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	5.285	VB	0.2356	3.56210e4	2342.70557	50.0802
2	8.722	VB	0.4808	3.55069e4	1150.91724	49.9198

总量 :　　7.11279e4　3493.62280

*** 报告结束 ***


spect, CDCl3,
Fri Nov 29 11:46:13 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-11-85
R-3p
Br
Et
OHC
9.621
7.298
6.530
6.068
6.060
6.011
6.002
4.103
3.866
3.622
2.368
2.344
2.321
2.299
2.275
2.251
2.166
2.142
2.118
2.095
2.071
2.048
0.961
0.938
0.913
0.000
9.45
3.21
2.02
1.00
0.99
1.03
1.05
0.99
PPM

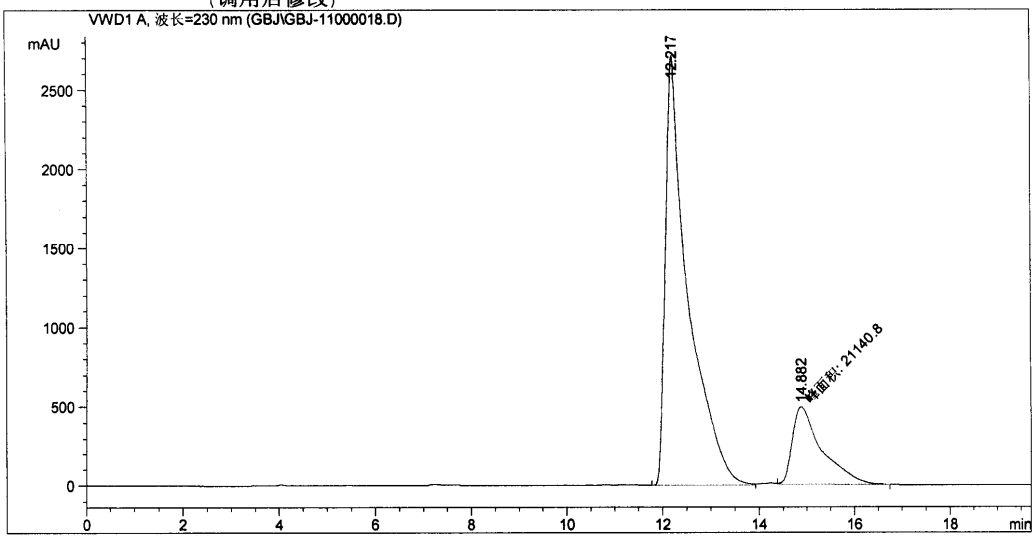

9.246
24.588
56.089
60.714
66.029
76.575
77.000
77.426
105.379
121.710
131.895
133.282
137.720
153.299
195.286
spect, CDCl3,
Fri Nov 29 11:47:31 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 98
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
Br
Et
OHC
R-3p
gbj-11-85-c
PPM
0
50
100
150
200

数据文件　　D:\Chem32\1\DATA\GBJ\GBJ-11000018.D
样品名: gbj-11-85

OD-H, n-hexane/i-PrOH =95/5, 0.8 ml/min; 230nm

==========

进样日期	: 2013-11-29 15:08:35	
样品名称	: gbj-11-85	位置 : -
操作者	: gbj	
仪器	: 仪器 1	
方法	: D:\CHEM32\1\METHODS\JJS.M	
最后修改	: 2013-11-29 14:30:25 : gbj	
	(调用后修改)	



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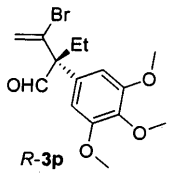
面积百分比报告

==========

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标使用乘积因子和稀释因子



R-3p

信号 1: VWD1 A, 波长=230 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	12.217	VV	0.4437	8.85860e4	2712.18286	80.7332
2	14.882	MM	0.7156	2.11408e4	492.38663	19.2668

总量 : 1.09727e5 3204.56949

==========

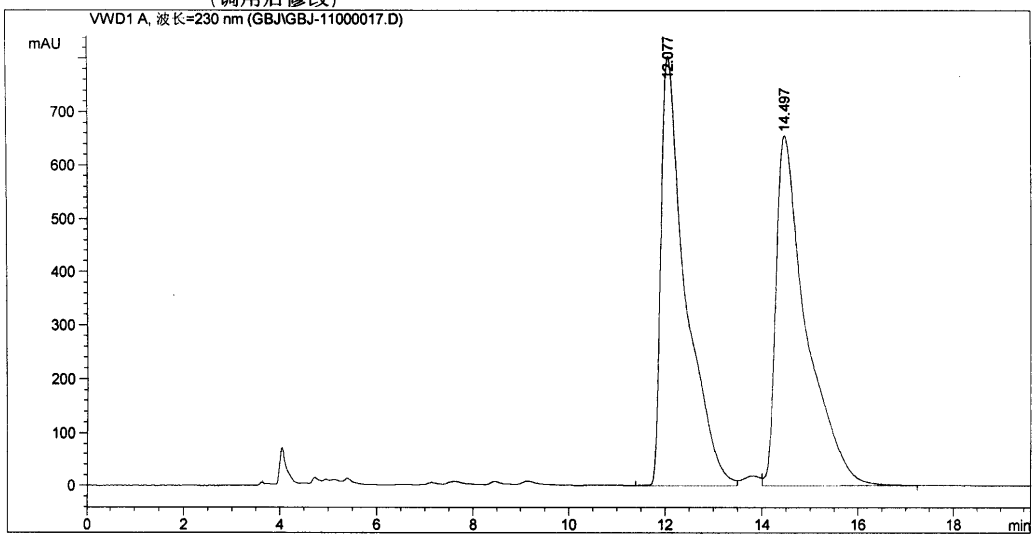
*** 报告结束 ***

数据文件　　D:\Chem32\1\DATA\GBJ\GBJ-11000017.D
样品名: gbj-11-84

OD-H, n-hexane/i-PrOH =95/5, 0.8 ml/min; 230nm

==========

进样日期　　: 2013-11-29 14:47:38
样品名称　　: gbj-11-84　　位置　:　-
操作者　　: gbj
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\JJS.M
最后修改　　: 2013-11-29 14:30:25　:　gbj
(调用后修改)

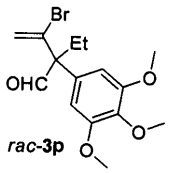


==========

面积百分比报告

==========

排序　　:　信号
乘积因子　　:　1.0000
稀释因子　　:　1.0000
内标使用乘积因子和稀释因子



rac-3p

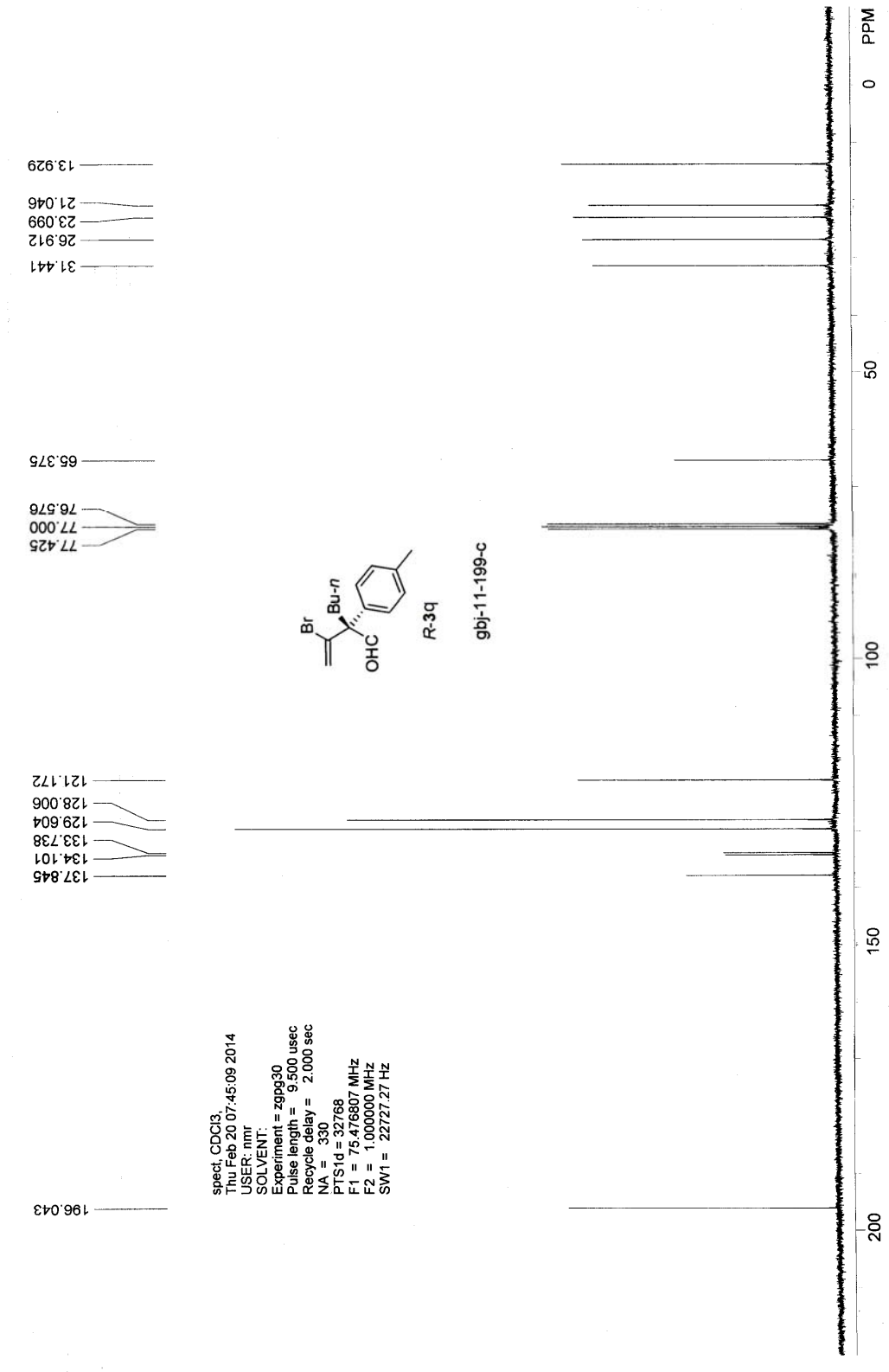
信号 1: VWD1 A, 波长=230 nm

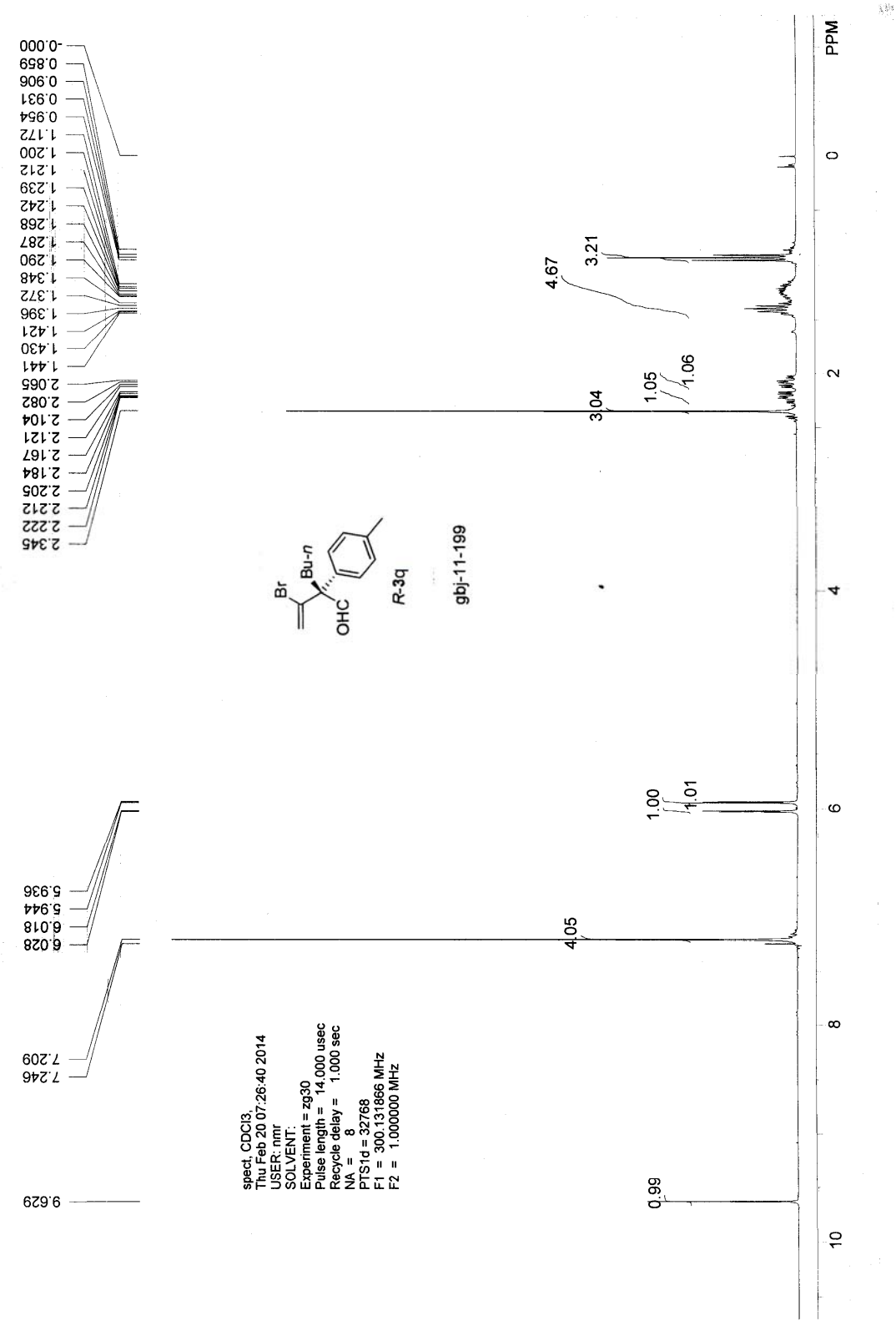
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	12.077	VV	0.5046	2.83377e4	803.48798	49.8169
2	14.497	VB	0.6269	2.85459e4	654.33990	50.1831

总量 :　　5.68836e4　1457.82788

==========

*** 报告结束 ***


13.929
21.046
23.099
26.912
31.441
65.375
76.576
77.000
77.425
121.172
128.006
129.604
133.738
134.101
137.845
196.043
PPM
0
50
100
150
200
Br
Bu-n
OHC
R-3q
gbj-11-199-c
spect, CDCl3,
Thu Feb 20 07:45:09 2014
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 330
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz


spect, CDCl3,
Thu Feb 20 07:26:40 2014
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
gbj-11-199
R-3q
Br
Bu-n
OHC
9.629
7.246
7.209
6.028
6.018
5.944
5.936
2.345
2.222
2.212
2.205
2.184
2.167
2.121
2.104
2.082
2.065
1.441
1.430
1.421
1.396
1.372
1.348
1.290
1.287
1.268
1.242
1.239
1.212
1.200
1.172
0.954
0.931
0.906
0.859
-0.000
0.99
1.00
1.01
4.05
3.04
1.05
1.06
4.67
3.21
PPM
10
8
6
4
2
0

gbj-11-199

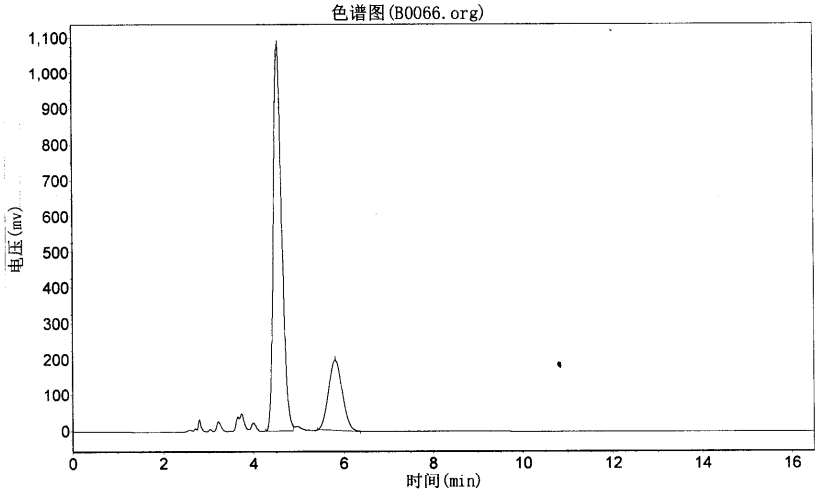
实验时间：2014-02-20,16:12:22
谱图文件:D:\浙大智达\N2000\样品\B0066.org

实验者：gbj
报告时间：2014-02-20,16:32:18
积分方法:面积归一法

使用仪器类型:气相色谱　　检测器:FID　　进样器:分流

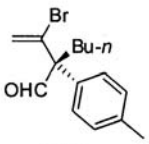
柱温:程序升温

色谱图(B0066.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		4.547	1080967.375	13827850.000	76.8174
2		5.812	196018.641	4173086.500	23.1826
总计			1276986.016	18000936.500	100.0000



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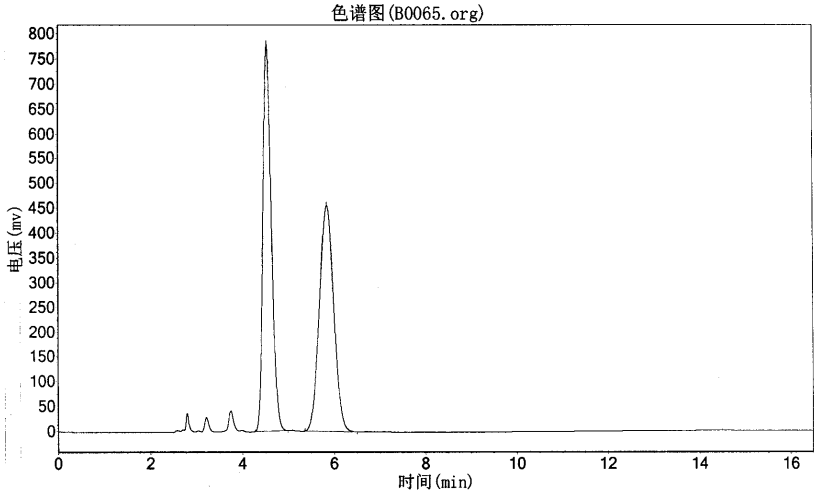
gbj-11-198

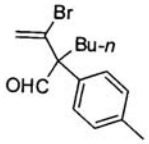
实验时间：2014-02-20,15:54:12
谱图文件:D:\浙大智达\N2000\样品\B0065.org

实验者：gbj
报告时间：2014-02-20,16:31:14
积分方法:面积归一法



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		4.548	778064.750	9939070.000	49.9030
2		5.847	456001.250	9977722.000	50.0970
总计			1234066.000	19916792.000	100.0000



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