



Supporting Information

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An Efficient Trapping of Oxonium Ylides with Imines:
A Highly Diastereoselective Three-Component Reaction for the
Synthesis of α -Hydroxyl- β -Amino Esters with Quaternary Stereocenters

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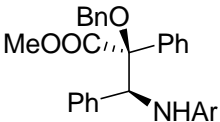
whu@chem.ecnu.edu

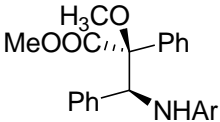
General methods: Melting points are uncorrected. NMR spectra were recorded on a Bruker-300 MHz spectrometer. HRMS (ESI) Mass spectra were recorded on BRUKER FT-MS. Dichloromethane was distilled over calcium hydride. Aldimines **3** were prepared from condensation of the corresponding aldehydes with 2-aminophenol according to the literature method.^[1] *N*-*tert*butylsulfonimines **6** were prepared accordingly.^[2] All reactions were carried out under argon atmosphere in a well-dried glassware.

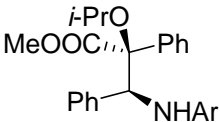
1. General procedure for the reaction of methyl phenyldiazoacetate with alcohols and imines 3b-f:

To a refluxing CH₂Cl₂ (4 mL) solution of Rh₂(OAc)₄ (2.38 mg, 1 mol%), benzyl alcohol **2a** (56.4 *ul*, 0.54 mmol) and imine **3a** (116.0 mg, 0.59 mmol) under argon atmosphere was added methyl

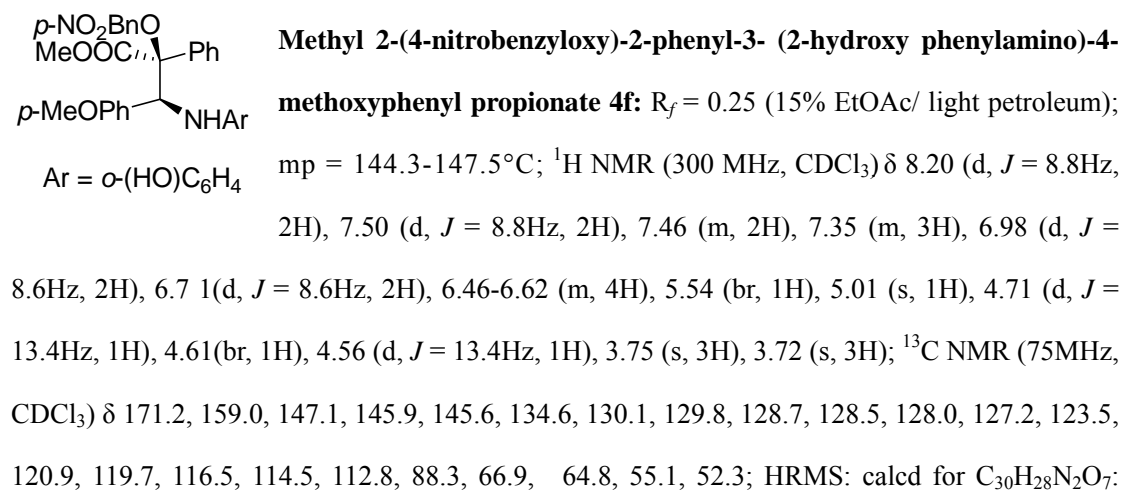
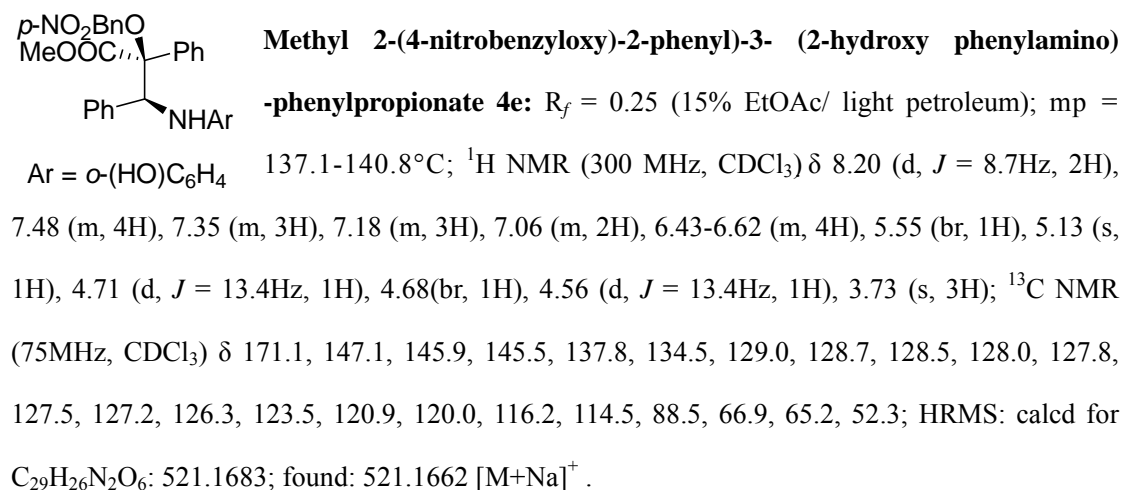
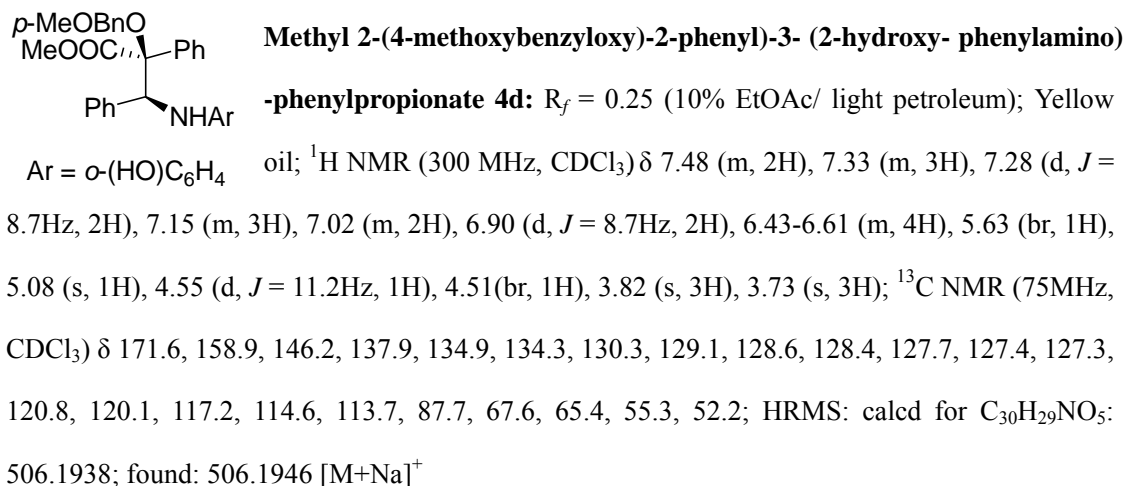
phenyldiazoacetate **1** (95 mg, 0.54 mmol) in 2 mL of CH₂Cl₂ over 1 h via a syringe pump. After completion of the addition, the reaction mixture was cooled to room temperature. Solvent was removed and the crude product was subject to ¹H-NMR analysis for the determination of chemo- and diastereoselectivity. The crude product was purified by flash chromatography on silica gel (eluent: EtOAc/light petroleum ether 1:9) to give **4a** (184mg, 75%). Products **4b~4i** were obtained by the same procedure.


Methyl 2-benzyloxy-2-phenyl-3-(2-hydroxyphenylamino) phenyl propionate 4a: *R_f* = 0.25 (10% EtOAc/ light petroleum); Yellow oil; ¹H NMR (300 MHz, CDCl₃) δ 7.54 (m, 2H), 7.27-7.53 (m, 8H), 7.18 (m, 3H), 7.08 (m, 2H), 6.63-6.81 (m, 4H), 5.93 (br, 1H), 5.20 (s, 1H), 4.71(br, 1H), 4.58 (d, *J* = 11.8Hz, 1H), 4.58 (d, *J* = 11.8Hz, 1H), 4.52 (d, *J* = 11.8Hz, 1H), 3.74 (s, 3H); ¹³C NMR (75MHz, CDCl₃) δ 171.6, 145.7, 138.1, 137.8, 134.7, 134.4, 129.1, 128.6, 128.4, 128.2, 127.7, 127.4, 127.3, 127.2, 127.0, 120.7, 119.6, 116.4, 114.5, 87.7, 67.8, 65.3, 52.2; HRMS: calcd for C₂₉H₂₇NO₄: 454.2013; found: 454.2013 [M+H]⁺.

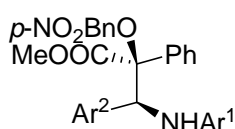

Methyl 2-methoxy-2-phenyl-3-(2-hydroxyphenylamino)-phenyl propionate 4b: *R_f* = 0.25 (10% EtOAc/ light petroleum); Yellow oil; ¹H NMR (300 MHz, CDCl₃) δ 7.46 (m, 2H), 7.33 (m, 3H), 7.15 (m, 3H), 6.99 (m, 2H), 6.43-6.61 (m, 4H), 5.89 (br, 1H), 4.98 (s, 1H), 4.51(br, 1H), 3.74 (s, 3H), 3.35 (s, 3H); ¹³C NMR (75MHz, CDCl₃) δ 171.6, 146.1, 137.8, 134.8, 134.3, 128.9, 128.6, 128.3, 127.7, 127.4, 127.3, 120.8, 120.1, 116.9, 114.6, 88.0, 65.4, 54.2, 52.2; HRMS: calcd for C₂₃H₂₃NO₄: 400.1519; found: 400.1530 [M+Na]⁺.


Methyl 2-isopropoxy-2-phenyl-3-(2-hydroxyphenyl amino)-phenyl propionate 4c: *R_f* = 0.25 (10% EtOAc/ light petroleum); Yellow oil; ¹H NMR (300 MHz, CDCl₃) δ 7.27-7.38 (m, 5H), 7.14 (m, 3H), 6.99 (m, 2H), 6.43-6.61 (m, 4H), 5.88 (br, 1H), 5.12 (s, 1H), 4.69(br, 1H), 3.93 (m, 1H), 3.71 (s, 3H), 1.22 (d, *J* = 6.1Hz, 3H), 1.02 (d, *J* = 6.1Hz, 3H); ¹³C NMR (75MHz, CDCl₃) δ 172.6,

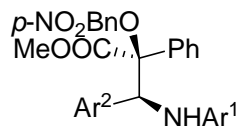
145.7, 138.1, 136.4, 134.5, 129.2, 128.9, 128.2, 127.4, 127.2, 127.1, 120.9, 119.4, 116.4, 114.6, 86.9, 68.8, 64.5, 51.9, 23.8, 23.1; HRMS: calcd for C₂₅H₂₇NO₄: 428.1832; found: 428.1821 [M+Na]⁺



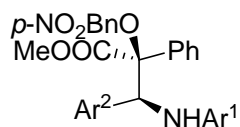
551.1789; found: 551.1779 [M+Na]⁺.



Methyl 2-(4-nitrobenzyloxy)-2-phenyl-3-(2-hydroxyphenyl amino)-4-cyanophenyl propionate 4g: $R_f = 0.25$ (15% EtOAc/ light petroleum); mp = 158.1-161.8°C; ¹H NMR (300 MHz, CDCl₃) δ 8.23 (d, $J = 8.6$ Hz, 2H), 7.50 (d, $J = 8.6$ Hz, 2H), 7.32-7.46 (m, 7H), 7.16 (d, $J = 8.2$ Hz, 2H), 6.46-6.62 (m, 4H), 5.54 (br, 1H), 5.24 (s, 1H), 4.91 (br, 1H), 4.67 (d, $J = 13.2$ Hz, 1H), 4.55 (d, $J = 13.2$ Hz, 1H), 3.74 (s, 3H); ¹³C NMR (75MHz, CDCl₃) δ 170.5, 147.3, 145.3, 144.7, 143.6, 134.0, 133.8, 131.2, 129.7, 129.1, 128.3, 127.3, 123.7, 121.2, 119.4, 118.7, 114.8, 114.6, 111.4, 87.9, 67.2, 64.9, 52.5; HRMS: calcd for C₃₀H₂₅N₃O₆: 546.1636; found: 546.1644 [M+Na]⁺.



Methyl 2-(4-nitrobenzyloxy)-2-phenyl-3-(2-hydroxy phenylamino)-3-(naphthalen-1-yl) propanoate 4h: $R_f = 0.25$ (15% EtOAc/ light petroleum); mp = 151.5-153.8°C; ¹H NMR (300 MHz, CDCl₃) δ 8.51 (m, 1H), 7.98 (d, $J = 8.2$ Hz, 2H), 7.87 (m, 1H), 7.72 (d, $J = 8.0$ Hz, 2H), 7.52 (m, 1H), 7.22-7.40 (m, 5H), 7.17 (m, 1H), 7.01 (d, $J = 8.2$ Hz, 2H), 6.46-6.67 (m, 6H), 5.34 (s, 1H), 4.72 (br, 1H), 4.55 (d, $J = 13.3$ Hz, 1H), 4.45 (d, $J = 13.2$ Hz, 1H), 3.72 (s, 3H); ¹³C NMR (75MHz, CDCl₃) δ 171.2, 146.9, 145.3, 144.9, 134.1, 133.3 (overlap), 129.1, 128.8, 128.6, 128.3, 127.8, 126.7, 125.8, 125.2, 124.5, 124.3, 123.3, 121.2 (overlap), 119.3, 115.4, 114.5 (overlap), 88.7, 66.5, 57.7, 52.6; HRMS: calcd for C₃₃H₂₈N₂O₆: 571.1840; found: 571.1814 [M+Na]⁺.

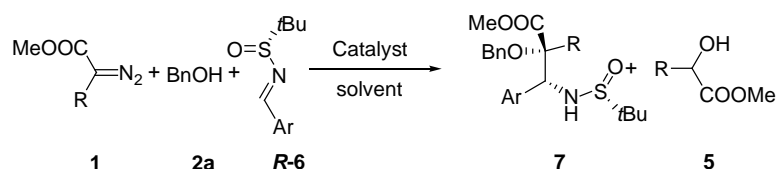


Methyl 2-(4-nitrobenzyloxy)-2-phenyl-3-(2-hydroxy phenylamino)-4-nitrophenyl propionate 4i: $R_f = 0.25$ (15% EtOAc/ light petroleum); mp = 146.1-148.6°C; ¹H NMR (300 MHz, CDCl₃) δ 8.23 (d, $J = 8.8$ Hz, 2H), 8.01 (d, $J = 8.7$ Hz, 2H), 7.50 (d, $J = 8.8$ Hz, 2H), 7.32-7.42 (m, 5H), 7.21 (d, $J = 8.7$ Hz, 2H), 6.46-6.68 (m, 4H), 5.29 (br, 2H), 4.88 (br, 1H), 4.67 (d, $J = 13.1$ Hz, 1H), 4.55 (d, $J = 13.1$ Hz, 1H), 3.75 (s, 3H); ¹³C NMR (75MHz, CDCl₃) δ 170.4, 147.4, 145.6, 145.2, 144.7, 133.9, 133.7, 129.9, 129.3, 128.3 (overlap), 127.4, 123.7, 122.6, 121.3, 119.7, 115.1, 114.6, 87.9, 67.2, 64.9, 52.5; HRMS: calcd for C₂₉H₂₅N₃O₈: 566.1534; found: 566.1532

[M+Na]⁺.

2. Optimizing the reaction condition for the reaction of methyl phenyl diazoacetate with alcohols and *N-tert*-butylsulfinimines **6**:

Table 1 Reaction of diazoacetate with benzyl alcohol and *N-tert*-butylsulfinimine

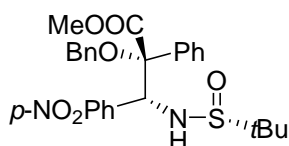


Entry	R	Ar	The ratio of 1 : 2 : 6	Catalyst	Solvent	Yield ^b of 7 (%)	dr of 7 ^[c]
1	C ₆ H ₅	<i>p</i> -O ₂ NC ₆ H ₄ (6a)	1 : 1 : 1	Rh ₂ (OAc) ₄	CH ₂ Cl ₂	25	>98 : 2
2	C ₆ H ₅	<i>p</i> -O ₂ NC ₆ H ₄ (6a)	1 : 1 : 1	Rh ₂ (OAc) ₄	THF	<5	>98 : 2
3	C ₆ H ₅	<i>p</i> -O ₂ NC ₆ H ₄ (6a)	1 : 1 : 1	Rh ₂ (OAc) ₄	Toluene	22	>98 : 2
4	C ₆ H ₅	<i>p</i> -O ₂ NC ₆ H ₄ (6a)	1 : 1 : 1	Rh ₂ (OAc) ₄	CH ₃ CN	14	>98 : 2
5	C ₆ H ₅	<i>p</i> -O ₂ NC ₆ H ₄ (6a)	1 : 1 : 1	Rh ₂ (OAc) ₄	ClCH ₂ CH ₂ Cl	10	>98 : 2
6	C ₆ H ₅	<i>p</i> -O ₂ NC ₆ H ₄ (6a)	2 : 2 : 1	Rh ₂ (OAc) ₄	CH ₂ Cl ₂	58	>98 : 2
7	C ₆ H ₅	<i>p</i> -O ₂ NC ₆ H ₄ (6a)	4 : 4 : 1	Rh ₂ (OAc) ₄	CH ₂ Cl ₂	55	>98 : 2
8	C ₆ H ₅	<i>p</i> -O ₂ NC ₆ H ₄ (6a)	2 : 2 : 1	Rh ₂ (tfa) ₄	CH ₂ Cl ₂	58	>98 : 2
9	C ₆ H ₅	<i>p</i> -O ₂ NC ₆ H ₄ (6a)	2 : 2 : 1	Rh ₂ (nttl) ₄	CH ₂ Cl ₂	58	>98 : 2
10	C ₆ H ₅	<i>p</i> -O ₂ NC ₆ H ₄ (6a)	2 : 2 : 1	Rh ₂ (cap) ₄	CH ₂ Cl ₂	20	>98 : 2
11	H	<i>p</i> -O ₂ NC ₆ H ₄ (6a)	2 : 2 : 1	Rh ₂ (OAc) ₄	CH ₂ Cl ₂	-- ^[d]	--
12	C ₆ H ₅	<i>p</i> -MeOC ₆ H ₄ (6c)	2 : 2 : 1	Rh ₂ (OAc) ₄	CH ₂ Cl ₂	-- ^[d]	--
13	C ₆ H ₅	C ₆ H ₅ (6d)	2 : 2 : 1	Rh ₂ (OAc) ₄	CH ₂ Cl ₂	8(7i)	>98 : 2
14	C ₆ H ₅	<i>p</i> -CNC ₆ H ₄ (6e)	2 : 2 : 1	Rh ₂ (OAc) ₄	CH ₂ Cl ₂	15(7j)	>98 : 2
15	C ₆ H ₅	<i>p</i> -CF ₃ C ₆ H ₄ (6f)	2 : 2 : 1	Rh ₂ (OAc) ₄	CH ₂ Cl ₂	15(7k)	>98 : 2

[a] All reaction were carried out in 40°C in the presence of Rhodium(II) catalyst (1 mol %). [b] Isolated yields after chromatography. [c] Determined by ¹H NMR of crude reaction mixtures. [d] Only O-H insertion product **5** was obtained.

3. General procedure for the reaction of methyl phenyldiazoacetate with alcohols and *N-tert*-butylsulfinimines **6**:

To a refluxing CH₂Cl₂ (8 mL) solution of Rh₂(OAc)₄ (3.6 mg, 2 mol%), benzyl alcohol **2a** (84.0 μ l, 0.8 mmol) and *N*-*tert*-butylsulfinimine **6a** (103.6 mg, 0.4 mmol) under argon atmosphere was added methyl phenyldiazoacetate **1** (141 mg, 0.8 mmol) in 4 mL of CH₂Cl₂ over 1 h via a syringe pump. After completion of the addition, the reaction mixture was cooled to room temperature. Solvent was removed and the crude product was subject to ¹H-NMR analysis for diastereoselectivity. The crude product was purified by flash chromatography on silica gel eluting with 30 % EtOAc-light petroleum to afford **7a**, yield 58%. Products **7a**–**7h** were obtained by the same procedure.



(R, R, R) Methyl 2-benzoxy-2-phenyl-3-*tert*-butylsulfinamino-4-

-nitrophenylpropionate 7a: $R_f = 0.23$ (30% EtOAc/ light petroleum);

Yellow oil; ¹H NMR (300 MHz, CDCl₃) δ 7.98 (d, $J = 8.9$ Hz, 2H),

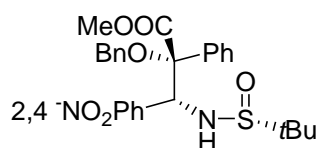
7.27-7.38 (m, 10H), 7.01 (d, $J = 8.9$ Hz, 2H), .5.21 (d, $J = 3.9$ Hz, 1H),

4.64 (d, $J = 3.9$ Hz, 1H), 4.55 (d, $J = 11.6$ Hz, 1H), 4.42 (d, $J = 11.6$ Hz, 1H), 3.84 (s, 3H), 1.18 (s,

9H); ¹³C NMR (75MHz, CDCl₃) δ 169.7, 147.5, 143.1, 137.5, 133.5, 130.7, 129.1, 128.3, 128.2,

127.6, 126.9, 122.1, 87.3, 68.2, 65.2, 56.1, 52.5, 22.4; HRMS: calcd for C₂₇H₃₀N₂O₆S₁; 533.1717;

found: 533.1732 [M+Na]⁺.



(R, R, R) Methyl 2-(4-benzoxy)-2-phenyl-3-*tert*-butyl

sulfinamino-2, 4-nitrophenyl propionate 7b: $R_f = 0.20$ (30%

EtOAc/ light petroleum); mp = 148.2-152.3°C; ¹H NMR (300

MHz, CDCl₃) δ 8.63 (d, $J = 2.3$ Hz, 1H), 8.13 (dd, $J_1 = 8.7$ Hz, $J_2 = 2.3$ Hz, 1H), 7.19-7.34

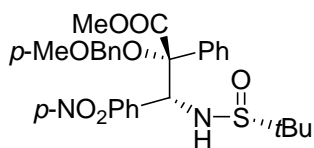
(m, 10H), 6.74 (d, $J = 8.7$ Hz, 1H), 6.68 (d, $J = 8.7$ Hz, 1H), 4.46 (d, $J = 11.8$ Hz, 1H), 4.35

(d, $J = 11.8$ Hz, 1H), 4.04 (d, $J = 8.7$ Hz, 1H), 3.78 (s, 3H), 1.17 (s, 9H); ¹³C NMR

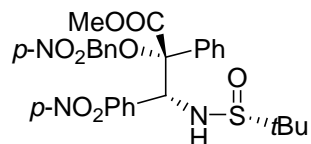
(75MHz, CDCl₃) δ 169.0, 150.5, 146.9, 138.9, 136.3, 132.0, 131.8, 129.3, 129.0, 128.3, 128.2,

127.9, 127.7, 124.7, 119.2, 87.4, 69.1, 58.4, 56.5, 53.1, 22.3; HRMS: calcd for C₂₇H₂₉N₃O₈S₁;

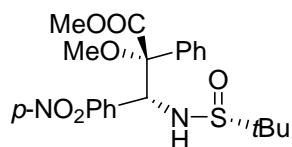
578.1568; found: 578.1588 [M+Na]⁺.



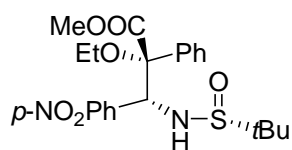
(R, R, R) Methyl 2-(4-methoxybenzoyl)-2-phenyl-3-tert-butylsulfenamino-4-nitrophenyl propionate 7c: $R_f = 0.23$ (30% EtOAc/light petroleum); mp = 146.3-149.5°C; ^1H NMR (300 MHz, CDCl_3) δ 7.96 (d, $J = 8.8\text{Hz}$, 2H), 7.33-7.39 (m, 5H), 7.23 (d, $J = 8.5\text{Hz}$, 2H), 7.01 (d, $J = 8.8\text{Hz}$, 2H), 6.87 (d, $J = 8.5\text{Hz}$, 2H), 5.19 (d, $J = 4.0\text{Hz}$, 1H), 4.64 (d, $J = 4.0\text{Hz}$, 1H), 4.46 (d, $J = 10.9\text{Hz}$, 1H), 4.33 (d, $J = 10.9\text{Hz}$, 1H), 3.85 (s, 3H), 3.82 (s, 3H), 1.17 (s, 9H); ^{13}C NMR (75MHz, CDCl_3) δ 169.9, 159.2, 147.5, 143.2, 133.7, 130.8, 129.6, 129.1, 128.6, 128.4, 128, 128.2, 122.1, 113.8, 87.3, 68.0, 65.2, 56.2, 55.3, 52.5, 22.5; HRMS : calcd for $\text{C}_{28}\text{H}_{32}\text{N}_2\text{O}_7\text{S}_1$; 563.1822; found: 563.1809 $[\text{M}+\text{Na}]^+$.



(R, R, R) Methyl 2-(4-nitrobenzoyl)-2-phenyl-3-tert-butylsulfenamino-4-nitrophenyl propionate 7d: $R_f = 0.23$ (30% EtOAc/light petroleum); mp = 130.2-134.5°C; ^1H NMR (300 MHz, CDCl_3) δ 8.21 (d, $J = 8.7\text{Hz}$, 2H), 7.99 (d, $J = 8.7\text{Hz}$, 2H), 7.50 (d, $J = 8.7\text{Hz}$, 2H), 7.34-7.40 (m, 5H), 7.04 (d, $J = 8.7\text{Hz}$, 2H), 5.21 (d, $J = 3.7\text{Hz}$, 1H), 4.67 (d, $J = 13.1\text{Hz}$, 1H), 4.61 (d, $J = 3.7\text{Hz}$, 1H), 4.51 (d, $J = 13.1\text{Hz}$, 1H), 3.84 (s, 3H), 1.17 (s, 9H); ^{13}C NMR (75MHz, CDCl_3) δ 169.3, 147.6, 147.3, 145.1, 142.9, 133.2, 130.7, 129.4, 128.5, 128.2, 127.2, 123.6, 122.2, 87.9, 67.3, 65.6, 56.2, 52.6, 22.4; HRMS: calcd for $\text{C}_{27}\text{H}_{29}\text{N}_3\text{O}_8\text{S}_1$; 578.1568; found: 578.1583 $[\text{M}+\text{Na}]^+$.

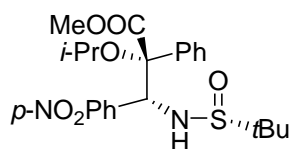


(R, R, R) Methyl 2-methoxyl-2-phenyl-3-tert-butylsulfenamino-4-nitrophenylpropionate 7e: $R_f = 0.23$ (30% EtOAc/light petroleum); mp = 113.0-116.6°C; ^1H NMR (300 MHz, CDCl_3) δ 7.97 (d, $J = 8.9\text{Hz}$, 2H), 7.33 (m, 5H), 6.98 (d, $J = 8.9\text{Hz}$, 2H), 5.08 (d, $J = 3.5\text{Hz}$, 1H), 4.61 (d, $J = 3.5\text{Hz}$, 1H), 3.86 (s, 3H), 3.31 (s, 3H); 1.19 (s, 9H); ^{13}C NMR (75MHz, CDCl_3) δ 169.9, 147.5, 143.2, 133.7, 130.7, 128.9, 128.3, 128.2, 122.1, 87.5, 65.2, 56.1, 54.6, 52.4, 22.5; HRMS: calcd for $\text{C}_{21}\text{H}_{26}\text{N}_2\text{O}_6\text{S}_1$; 457.1404; found: 457.1424 $[\text{M}+\text{Na}]^+$.



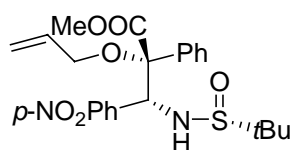
(R, R, R) Methyl 2-ethoxyl-2-phenyl-3-tert-butylsulfinamino-4-nitrophenylpropionate 7f: $R_f = 0.23$ (30% EtOAc/ light petroleum); mp = 102.2-104.5°C; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ

7.95 (d, $J = 8.8\text{Hz}$, 2H), 7.26-7.33 (m, 5H), 6.95 (d, $J = 8.8\text{Hz}$, 2H), 5.09 (d, $J = 3.8\text{Hz}$, 1H), 4.66 (d, $J = 3.8\text{Hz}$, 1H), 3.86 (s, 3H), 3.33-3.49 (m, 2H), 1.22 (m, 3H); 1.18 (s, 9H); $^{13}\text{C NMR}$ (75MHz, CDCl_3) δ 170.1, 147.5, 143.2, 134.1, 130.7, 128.8, 128.2, 128.0, 122.0, 87.0, 65.0, 62.1, 56.1, 52.4, 22.4, 15.2; HRMS: calcd for $\text{C}_{22}\text{H}_{28}\text{N}_2\text{O}_6\text{S}_1$; 471.1560; found: 471.1556 $[\text{M}+\text{Na}]^+$.



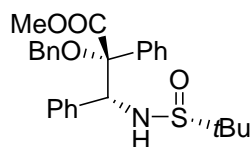
(R, R, R) Methyl 2-isopropoxyl-2-phenyl-3-tert-butylsulfinamino-4-nitrophenyl propionate 7g: $R_f = 0.23$ (30% EtOAc/ light petroleum); mp = 100.1-103.8°C; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ

7.98 (d, $J = 8.8\text{Hz}$, 2H), 7.22-7.34 (m, 5H), 6.98 (d, $J = 8.8\text{Hz}$, 2H), 5.25 (d, $J = 5.5\text{Hz}$, 1H), 4.44 (d, $J = 5.5\text{Hz}$, 1H), 3.84 (s, 3H), 3.80 (m, 1H), 1.20 (d, $J = 6.1\text{Hz}$, 3H), 1.18 (s, 9H), 0.92 (d, $J = 6.1\text{Hz}$, 3H); $^{13}\text{C NMR}$ (75MHz, CDCl_3) δ 170.4, 147.3, 143.9, 134.8, 130.6, 129.2, 128.8, 127.6, 121.9, 87.0, 70.1, 64.9, 56.2, 52.3, 23.8, 23.2, 22.4; HRMS: calcd for $\text{C}_{23}\text{H}_{30}\text{N}_2\text{O}_6\text{S}_1$; 485.1717; found: 485.1734 $[\text{M}+\text{Na}]^+$



(R, R, R) Methyl 2-alloxy-2-phenyl-3-tert-butylsulfinamino-4-nitrophenylpropionate 7h: $R_f = 0.23$ (30% EtOAc/ light petroleum); mp = 96.2-100.8°C; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ

7.97 (dd, $J_1 = 6.9\text{Hz}$, $J_2 = 1.9\text{Hz}$, 2H), 7.29-7.34 (m, 5H), 7.01 (dd, $J_1 = 6.9\text{Hz}$, $J_2 = 1.9\text{Hz}$, 2H), 5.25 (m, 1H), 5.26 (dd, $J_1 = 17.3\text{Hz}$, $J_2 = 1.7\text{Hz}$, 1H), 5.15 (m, 1H), 5.13 (d, $J = 4.0\text{Hz}$, 1H), 4.57 (d, $J = 4.0\text{Hz}$, 1H), 4.0 (m, 1H), 3.89 (m, 1H), 3.84 (s, 3H), 1.17 (s, 9H); $^{13}\text{C NMR}$ (75MHz, CDCl_3) δ 169.9, 147.5, 143.3, 133.8, 130.8, 128.9, 128.2(2C), 128.1, 122.1, 116.2, 87.2, 67.3, 65.1, 56.1, 52.5, 22.6; HRMS : calcd for $\text{C}_{23}\text{H}_{28}\text{N}_2\text{O}_6\text{S}_1$; 483.1560; found: 483.1581 $[\text{M}+\text{Na}]^+$.

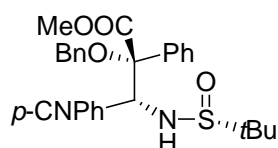


(R, R, R) Methyl 2,2-(4-benzoxy, phenyl)-3-tert-butylsulfinamino

-4-phenylpropionate 7i: $R_f = 0.23$ (30% EtOAc/ light petroleum);

Yellow oil; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.12-7.43 (m, 13H),

6.86 (d, $J = 7.6\text{Hz}$, 2H), 5.10 (d, $J = 4.2\text{Hz}$, 1H), 4.62 (d, $J = 4.2\text{Hz}$, 1H), 4.55 (d, $J = 11.7\text{Hz}$, 1H), 4.42 (d, $J = 11.7\text{Hz}$, 1H), 3.81 (s, 3H), 1.16 (s, 9H); $^{13}\text{C NMR}$ (75MHz, CDCl_3) δ 170.3, 138.0, 135.6, 134.4, 129.8, 128.7, 128.5, 128.3, 127.9, 127.8, 127.4, 127.1, 126.9, 87.7, 67.9, 65.9, 55.9, 52.2, 22.5; HRMS : calcd for $\text{C}_{27}\text{H}_{31}\text{NO}_4\text{S}$; 488.1866; found: 488.1872 $[\text{M}+\text{Na}]^+$.

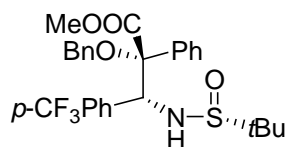


(R, R, R) Methyl 2,2-(4-benzoxy, phenyl)-3-tert-butylsulfinamino-

4-cyanophenylpropionate 7j: $R_f = 0.23$ (30% EtOAc/ light

petroleum); Yellow oil; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.40 (d, J

= 8.3Hz, 2H), 7.25-7.38 (m, 10H), 6.95 (d, $J = 8.3\text{Hz}$, 2H), .515 (d, $J = 4.0\text{Hz}$, 1H), 4.62 (d, $J = 4.0\text{Hz}$, 1H), 4.54 (d, $J = 11.6\text{Hz}$, 1H), 4.40 (d, $J = 11.6\text{Hz}$, 1H), 3.83 (s, 3H), 1.17 (s, 9H); $^{13}\text{C NMR}$ (75MHz, CDCl_3) δ 169.9, 141.1, 137.6, 133.7, 130.8, 130.6, 129.0, 128.4, 128.2, 127.6, 126.9, 118.7, 111.8, 87.4, 68.2, 65.4, 56.1, 52.5, 22.5; HRMS : calcd for $\text{C}_{28}\text{H}_{30}\text{N}_2\text{O}_4\text{S}_1$; 513.1818; found: 513.1828 $[\text{M}+\text{Na}]^+$



(R, R, R) Methyl 2,2-(4-benzoxy, phenyl)-3-tert-butylsulfinamino-

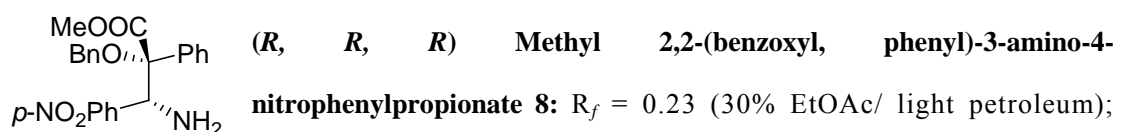
4-trifluorophenylpropionate 7k: $R_f = 0.23$ (30% EtOAc/ light

petroleum); mp = 89.1-90.5°C; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ

7.38 (d, $J = 8.1\text{Hz}$, 2H), 7.25-7.36 (m, 10H), 6.96 (d, $J = 8.1\text{Hz}$, 2H), .516 (d, $J = 4.0\text{Hz}$, 1H), 4.63 (d, $J = 4.0\text{Hz}$, 1H), 4.55 (d, $J = 11.7\text{Hz}$, 1H), 4.42 (d, $J = 11.7\text{Hz}$, 1H), 3.83 (s, 3H), 1.17 (s, 9H); $^{13}\text{C NMR}$ (75MHz, CDCl_3) δ 170.0, 139.8, 137.8, 133.9, 130.2, 128.9, 128.5, 128.3, 128.1, 127.5, 126.9, 14.0, 123.9, 87.5, 68.2, 65.4, 56.0, 52.4, 22.5; HRMS : calcd for $\text{C}_{28}\text{H}_{30}\text{F}_3\text{N}_1\text{O}_4\text{S}_1$; 556.1740; found: 556.1756 $[\text{M}+\text{Na}]^+$

4. Conversion of the three-component product 7a to 8.

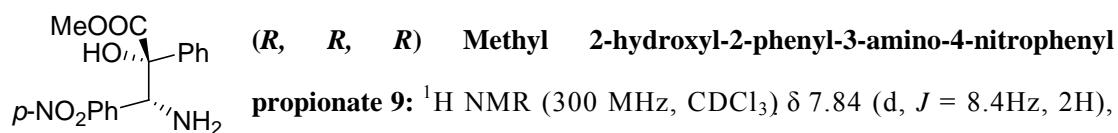
Compound **7a** (0.2 mmol) was dissolved in 2.0 mL of methanol. To the solution was added 0.6 mL of 3 N HCl in 1,4-dioxane (2.0 mmol). The mixture was stirred at room temperature for 2h, and was then concentrated. The resulting residue was added 10 mL saturated aqueous NaHCO₃. The aqueous phase was extracted with EtOAc (3×10ml), and the combined organic phase was dried over anhydrous MgSO₄. After filtration, the solvent was removed to give amine **8** as colorless oil.



Colorless oil; 98% *ee* was determined by HPLC with a chiral Diacel Chiralcel column; (30% *i*PrOH in hexane at 1.0 mL/min); Retention time: $t_{\text{major}} = 8.4$ min, and $t_{\text{minor}} = 10.4$ min. $[\alpha]_D^{12} = +26.3$ ($c = 1.0\text{g/ml}$, EtOAc). ¹H NMR (300 MHz, CDCl₃) δ 7.95 (d, $J = 7.5\text{Hz}$, 2H), 7.18-7.35 (m, 10H), 7.13 (d, $J = 7.5\text{Hz}$, 2H), 4.71(s, 1H), 4.57 (d, $J = 11.6\text{Hz}$, 1H), 4.46 (d, $J = 11.6\text{Hz}$, 1H), 3.80 (s, 3H), 3.66 (br, 2H); ¹³C NMR (75MHz, CDCl₃) δ 170.4, 147.4, 145.5, 137.7, 133.9, 129.8, 128.8, 128.4, 128.3, 128.0, 127.7, 127.3, 122.4, 87.9, 68.3, 63.2, 52.4; HRMS: calcd for C₂₃H₂₂N₂O₅; 407.1601; found: 407.1609 [M+Na]⁺.

5. Procedure for the preparation of 9:

To a solution of **8** (0.14 g, 0.34mmol) in EtOH (4 ml) was added 10% Pd on carbon (0.22 g) and conc. HCl (0.8 ml). The reaction mixture was charged with an atmosphere of H₂ and stirred at r.t. for 14 h. The catalyst was filtered off, and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel eluting with CHCl₃ : CH₃OH = 50:1 to give α -hydroxyl- β -amino **9**.

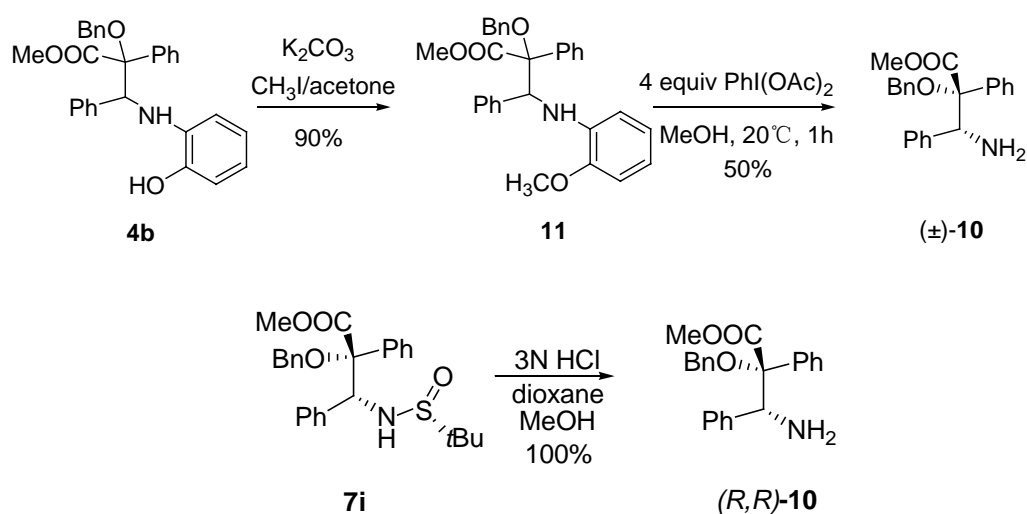


7.20-7.41 (m, 5H), 6.62 (d, $J = 8.4\text{Hz}$, 2H), 4.63(s, 1H), 3.62 (br, 2H), 3.58(s, 3H), 2.02 (br, 1H); ¹³C NMR (75MHz, CDCl₃) δ 173.7, 146.4, 139.6, 129.8, 128.9, 128.4, 127.9, 126.4, 114.8, 81.4, 61.7, 52.8; HRMS: calcd for C₁₆H₁₆N₂O₅; 339.0951;

found: 339.0968 [M+Na]⁺.

6. Determination of relative configuration of adducts **4**:

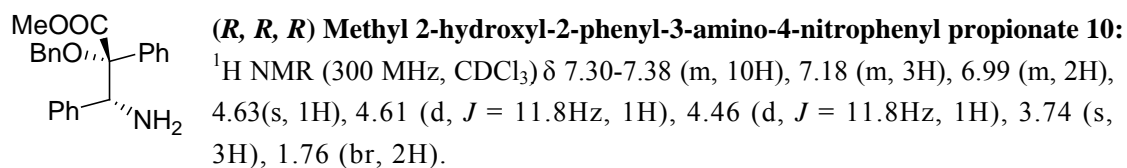
Adduct **4b** was deprotected to give racemic **10**. ¹H NMR of compound **10** was identical to that of (*R,R*)-**10** indicating that both **4** and **7** were erythro isomers.



Compound **11**: A mixture of compound **4b** (0.47 g, 1 mmol), CH_3I (5.7g, 40mmol) and K_2CO_3 (0.8 g) in acetone (12.5 ml) was stirred at room temperature for 8 h. Aqueous NH_4Cl was added to quench the reaction. After a usual work up, **11** was obtained 90% yield.^[3]

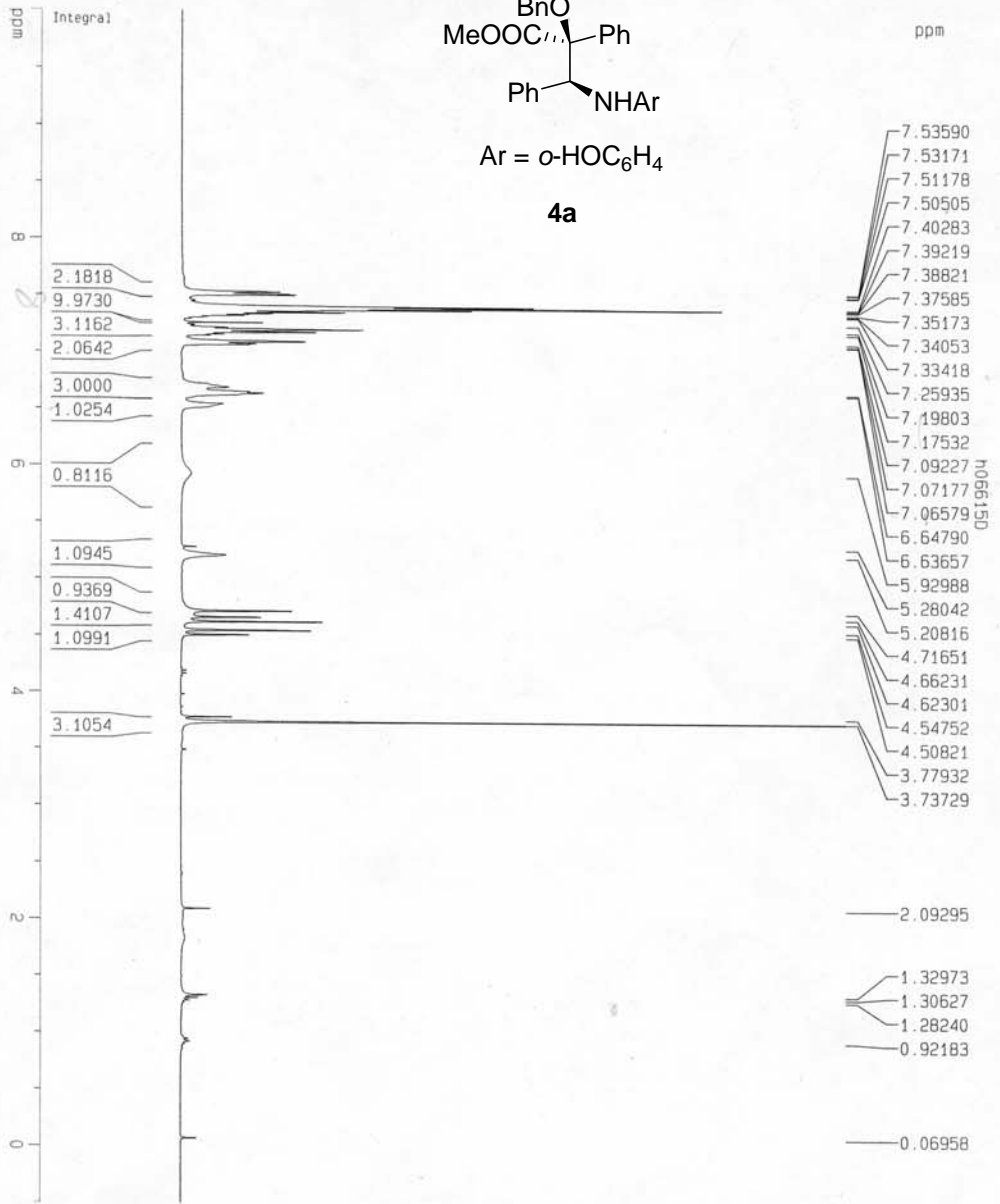
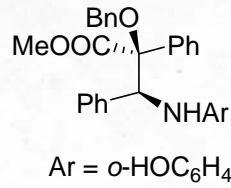
Preparation of (\pm)-**10**: A methanol (1 ml) solution of **11** (0.11 g, 0.22 mmol) was added to a solution of iodobenzene diacetate (0.3 g, 0.9 mmol) in methanol (3 ml) at 20°C over 30 min. via a syringe pump. The reaction was allowed to stir at the temperature for additional 30min. 1N HCl (5 ml) was added and the resulting mixture was stirred for 1.5 h. The reaction mixture was washed with saturated NaHCO_3 (10 ml), extracted with EtOAc (2 x 5 ml), and the combined organic layer were washed with saturated NaCl (5 ml). The organic layer was dried over anhydrous MgSO_4 . After filtrated and concentrated, the crude product was purified by flash chromatography on silica gel eluting with 25 % EtOAc -light petroleum to give (\pm)-**10** in 50% yield.^[4]

Preparation of (*R, R*)-**10**: The procedure was the same as for the preparation of **8**.



References and Notes

- [1] H. E. A. Astudillo, N. C. J. Chokotho, T. C. Jarvis, C. D. Johnson, C. C. Lewis, P. D. McDonnell, *Tetrahedron* **1985**, *41*, 5919.
- [2] G. V. Liu, D. A. Cogan, T. D. Owens, T. P. Tang, J. A. Ellman, *J. Org. Chem.* **1999**, *64*, 1278.
- [3] S. Kobayashi, H. Ishitani, M. Ueno, *J. Am. Chem. Soc.* **1998**, *120*, 431.
- [4] J. R. Porter, J. F. Traverse, A. H. Hoveyda, M. L. Snapper, *J. Am. Chem. Soc.* **2001**, *123*, 10409.



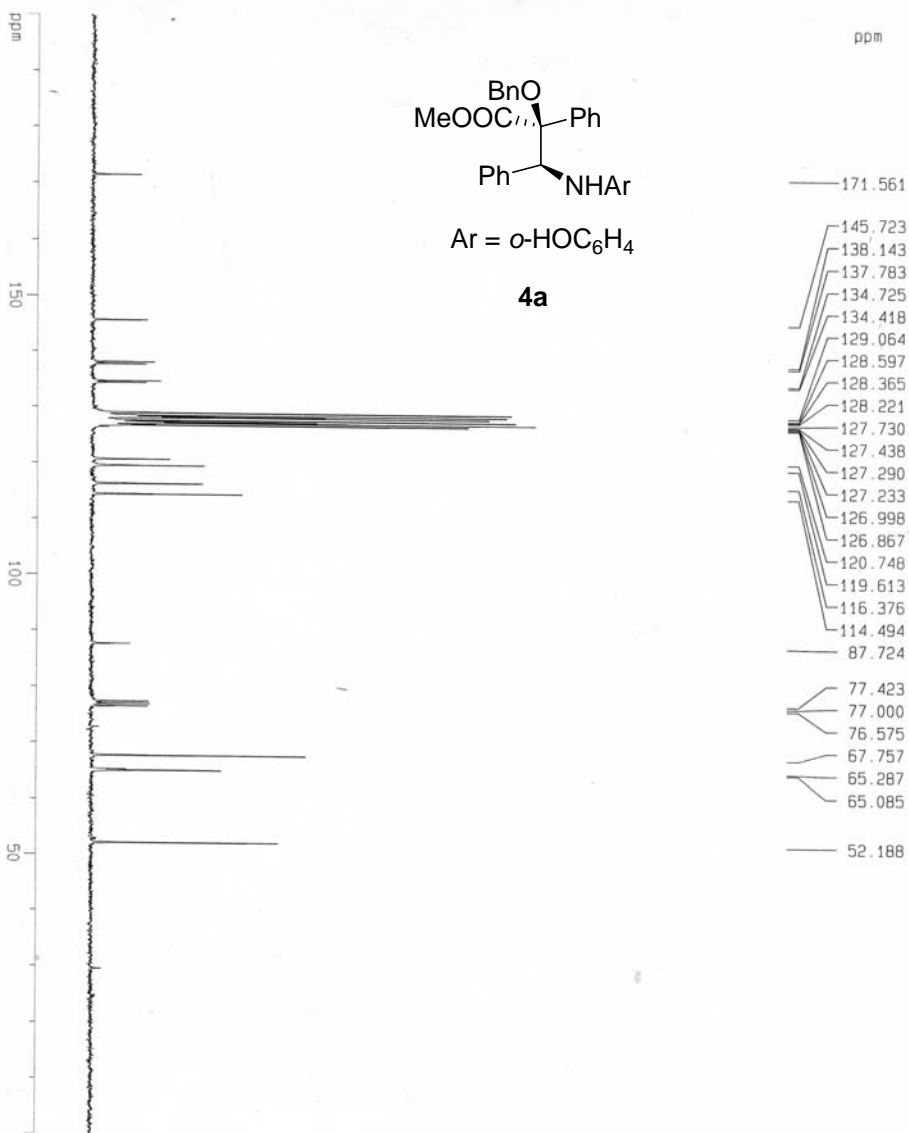
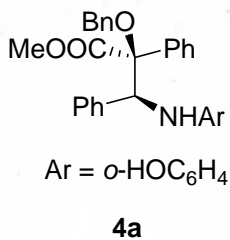
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H066150

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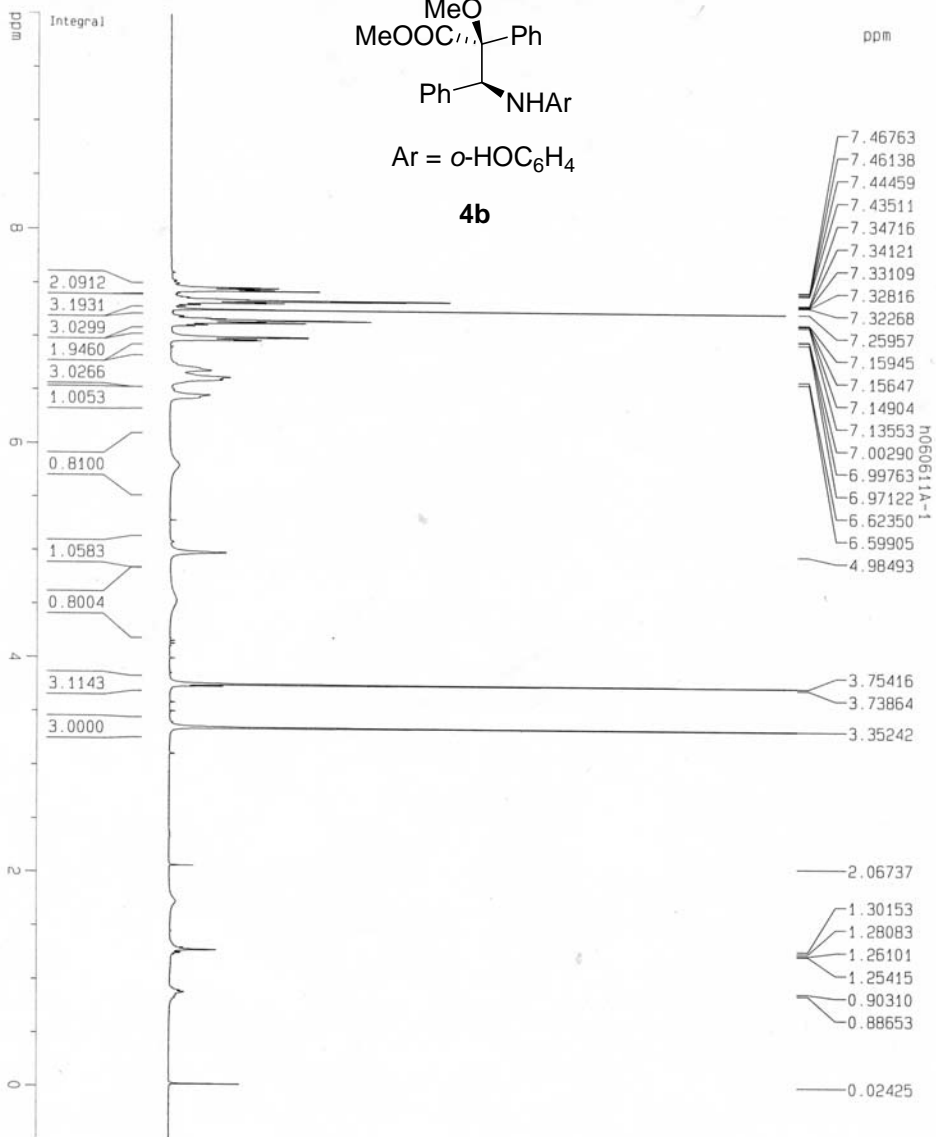
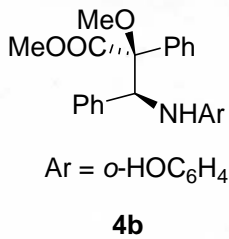
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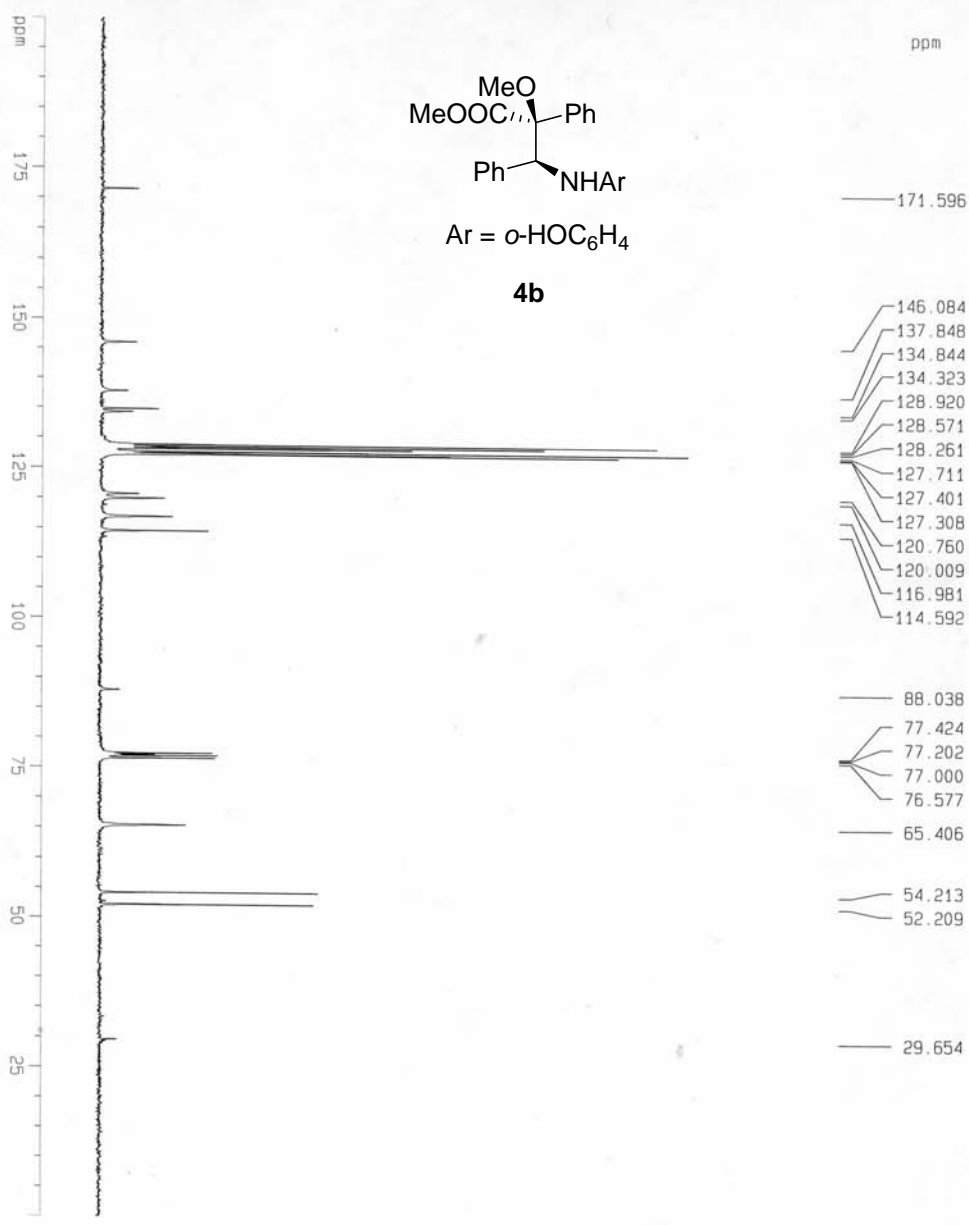
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 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 296
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h060611A-1

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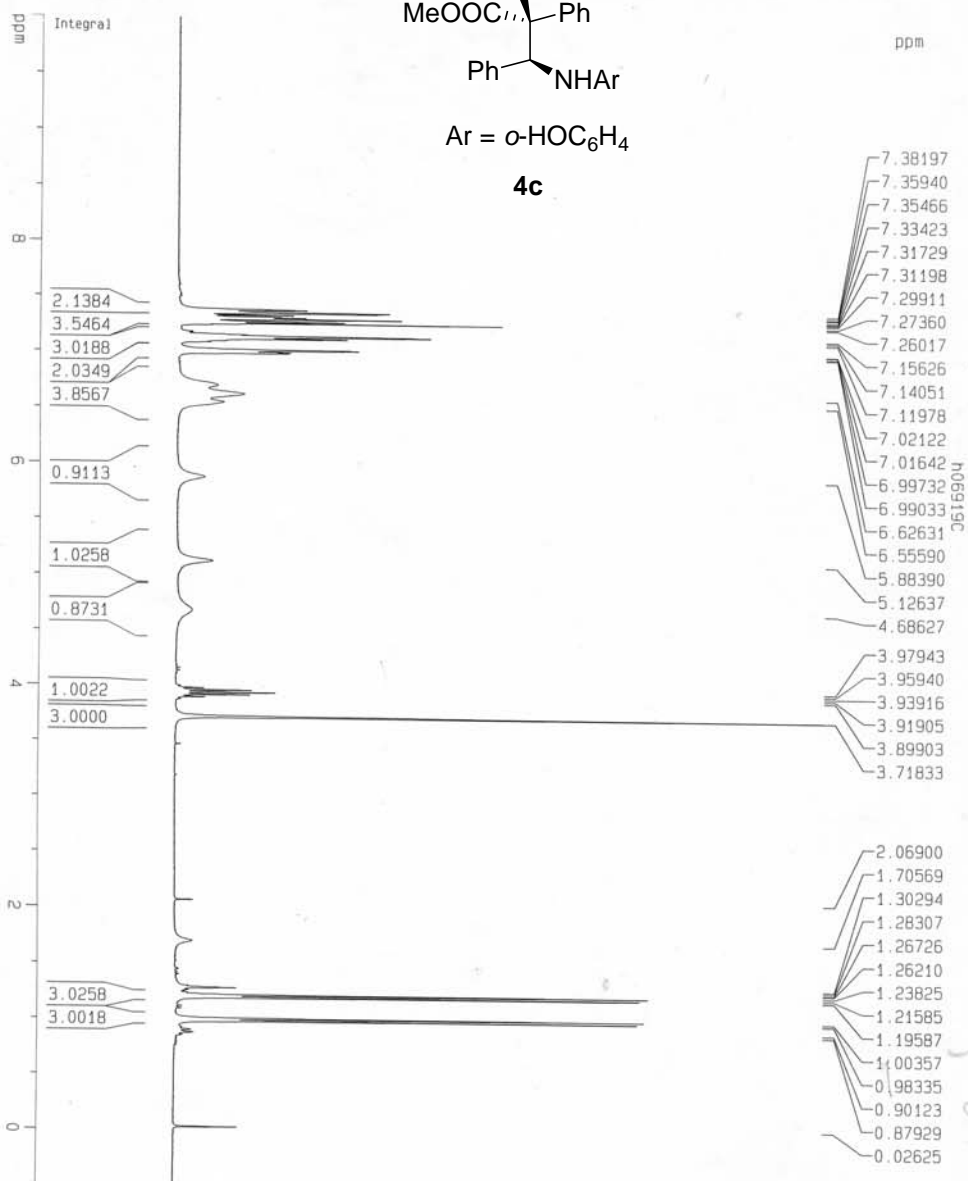
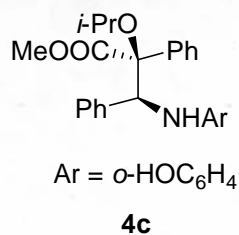
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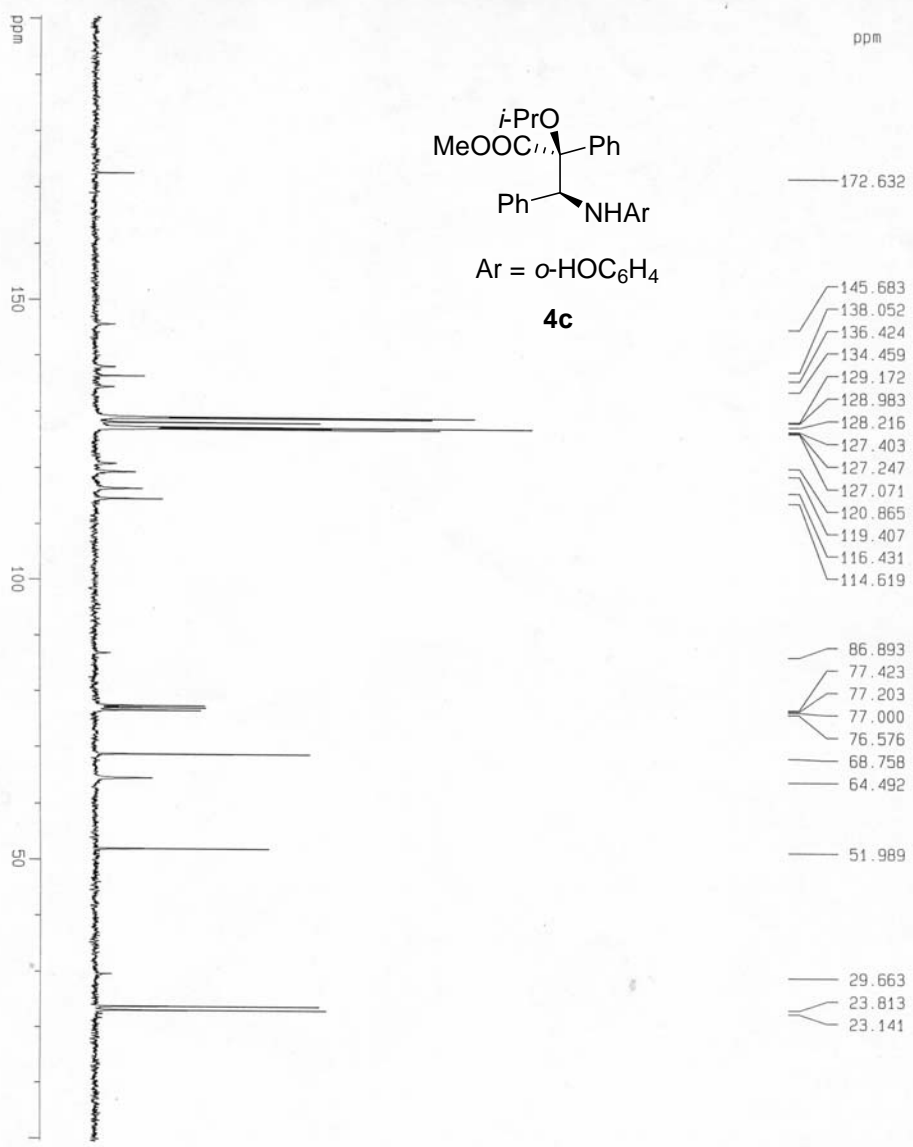
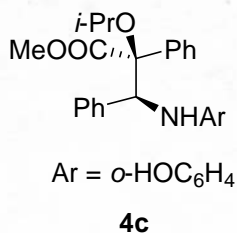
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h06919c

Current Data Parameters

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PROCNO 1

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TE 300.0 K

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PL1 -6.00 dB

SFO1 75.475598 MHz

***** CHANNEL f2 *****

COPPRG2 waltz16

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PCPD2 80.00 usec

PL2 -2.00 dB

PL12 17.70 dB

PL13 17.71 dB

SFO2 300.1312005 MHz

F2 - Processing parameters

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1D NMR plot parameters

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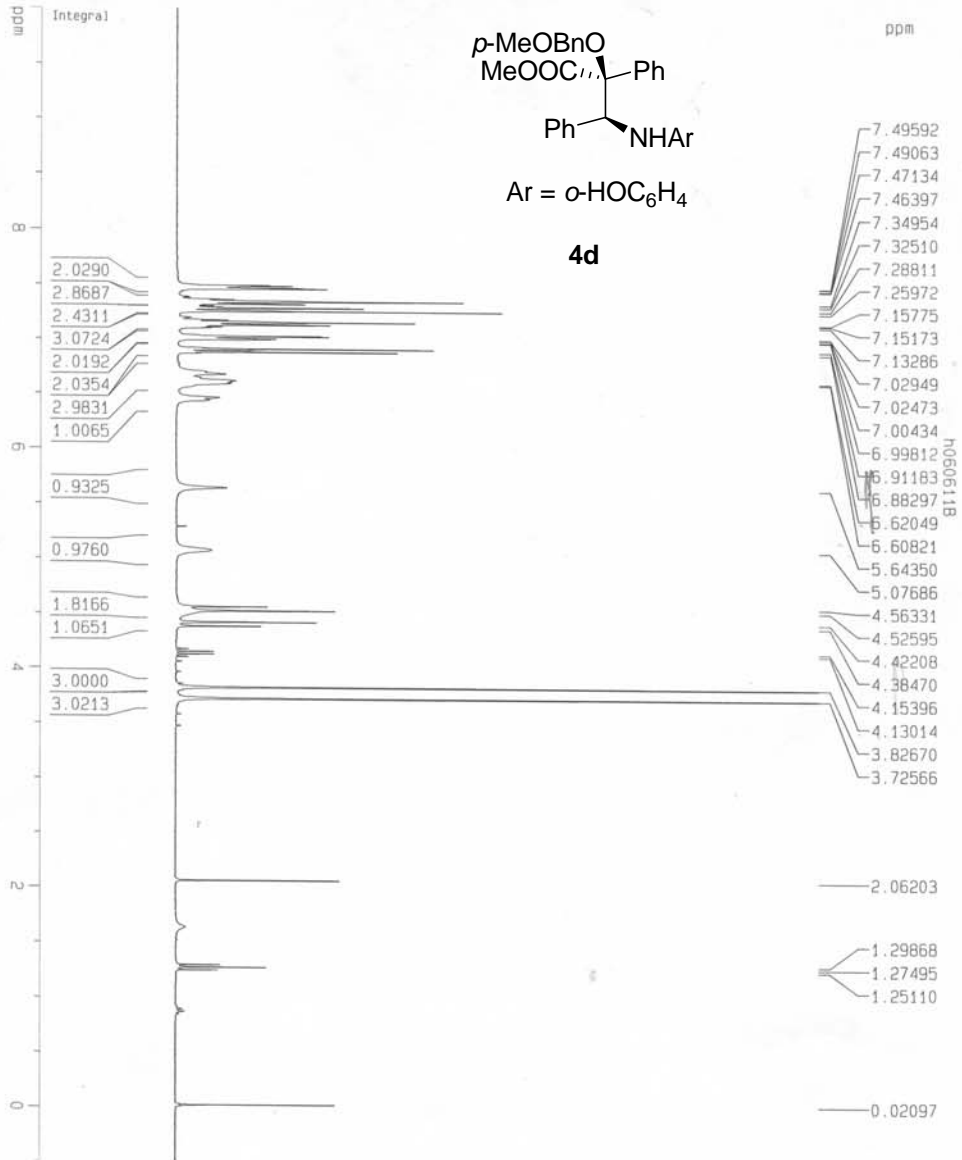
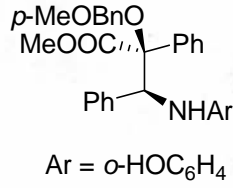
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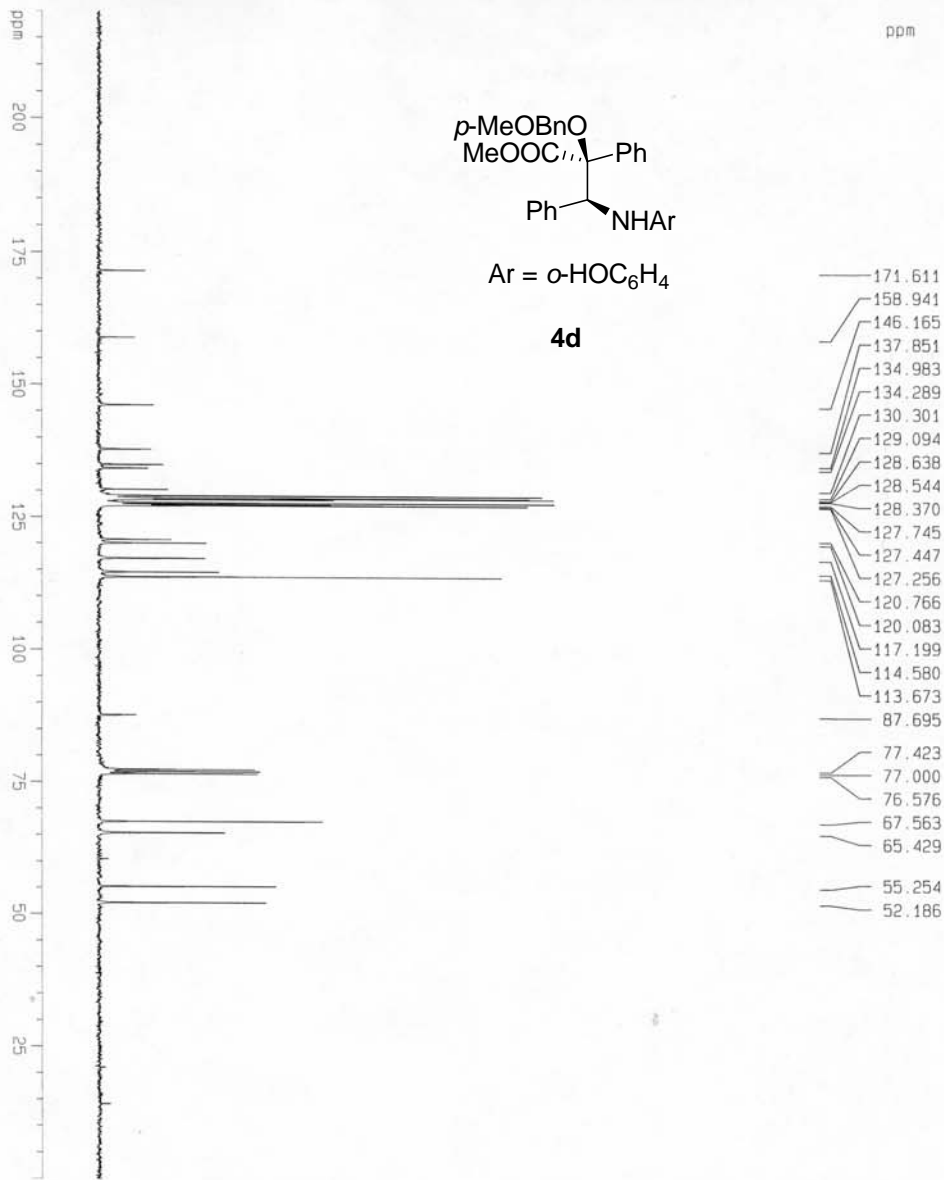
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 D1 1.00000000 sec

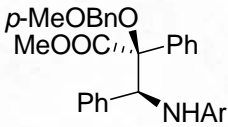
***** CHANNEL f1 *****
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SFO1 300.1320882 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300124 MHz
 KW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 20.00 cm
 FIP 10.000 ppm
 FL 3001.30 Hz
 FSP -0.500 ppm
 F2 -150.06 Hz
 SPHCK 0.32500 ppm/cm
 HZCM 157.56825 Hz/cm



h050611B



Ar = o-HOC₆H₄

4d

Current Data Parameters
NAME 1399-nix-720
EXPNO 2
PROCNO 1

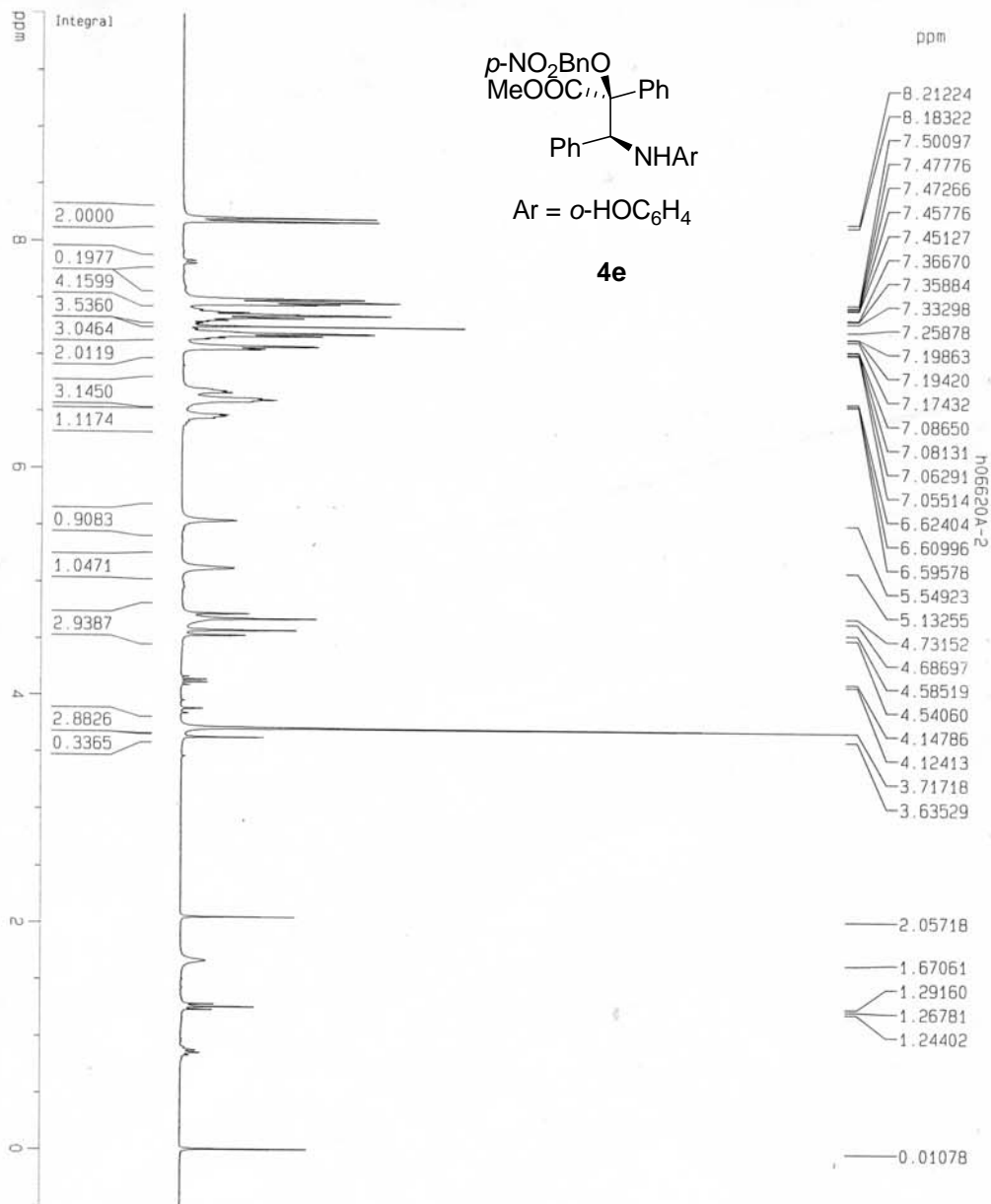
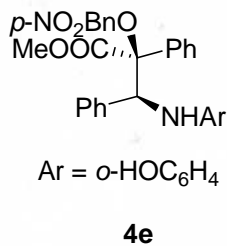
F2 - Acquisition Parameters
Date_ 20060614
Time 12.29
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TO 65936
SOLVENT CDCl3
NS 1980
DS 4
SMH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.445188 sec
RG 8192
DE 22.050 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SF01 75.4775598 MHz

***** CHANNEL f2 *****
CPOPH2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.467548 MHz
MDM EN
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR Plot Parameters
CX 20.00 cm
CY 8.00 cm
F1P 220.005 ppm
F1 16603.29 Hz
F2P -0.005 ppm
F2 -0.38 Hz
SFOCK 11.00050 ppm/cm
HZCM 830.18311 Hz/cm



Current Data Parameters
 NAME 1399-hhx-744
 EXPNO 1
 PROCNO 1

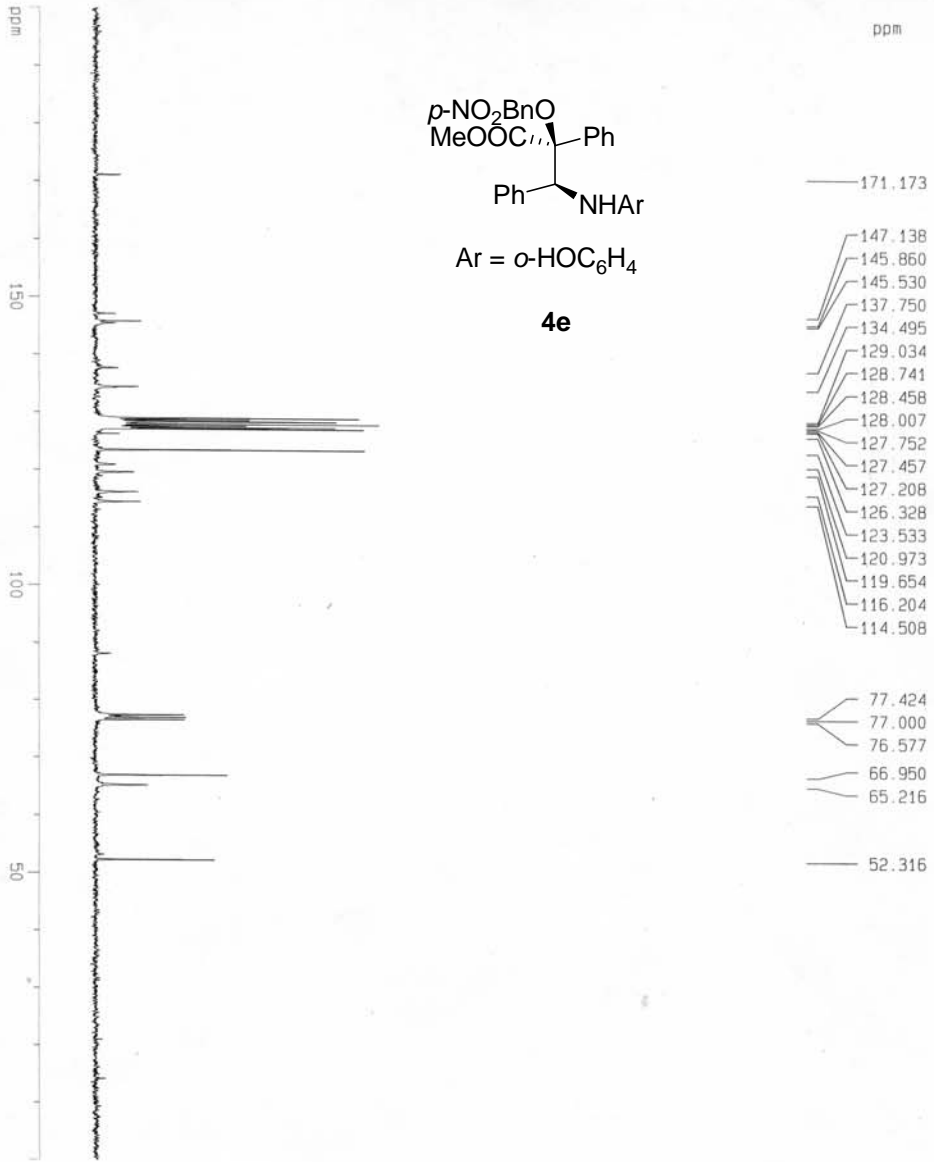
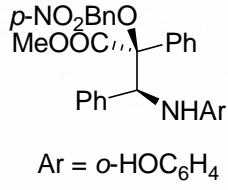
F2 - Acquisition Parameters
 Date_ 20060623
 Time 15.54
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 2930
 FIDRES 32768
 SOLVENT CDCl3
 NS 32
 DS 0
 SMH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 512
 DW 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SFO1 300.1320982 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300124 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 14.00 cm
 FIP 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm

106620A-2



Current Data Parameters
 NAME 1399-nhx-743
 EXPNO 2
 PROCNO 1

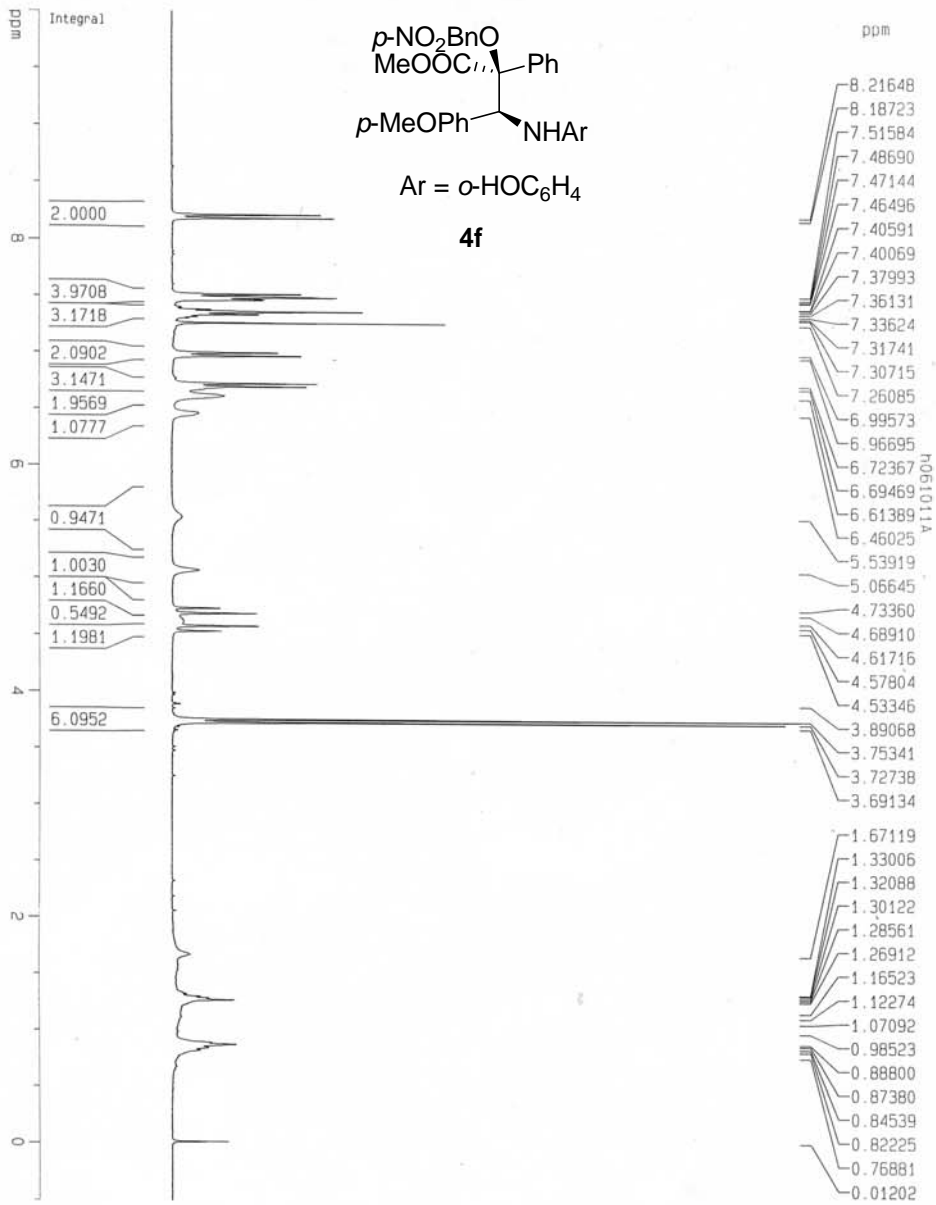
F2 - Acquisition Parameters
 Date_ 20080626
 Time 9:00
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 640
 DS 4
 SMH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451188 sec
 RG 8192
 DM 22.050 usec
 DE 5.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.50 usec
 PL1 -8.00 dB
 SFO1 75.4775998 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.467547 MHz
 KW 598
 EN 0
 LB 3.00 Hz
 GB 0
 PC 1.40

10 NMR plot parameters
 CX 20.00 cm
 CY 5.00 cm
 FIP 200.000 ppm
 F1 15093.55 Hz
 F2 -0.005 ppm
 F2 -0.38 Hz
 PPKICK 10.00025 ppm/cm
 HZCK 754.69647 Hz/cm



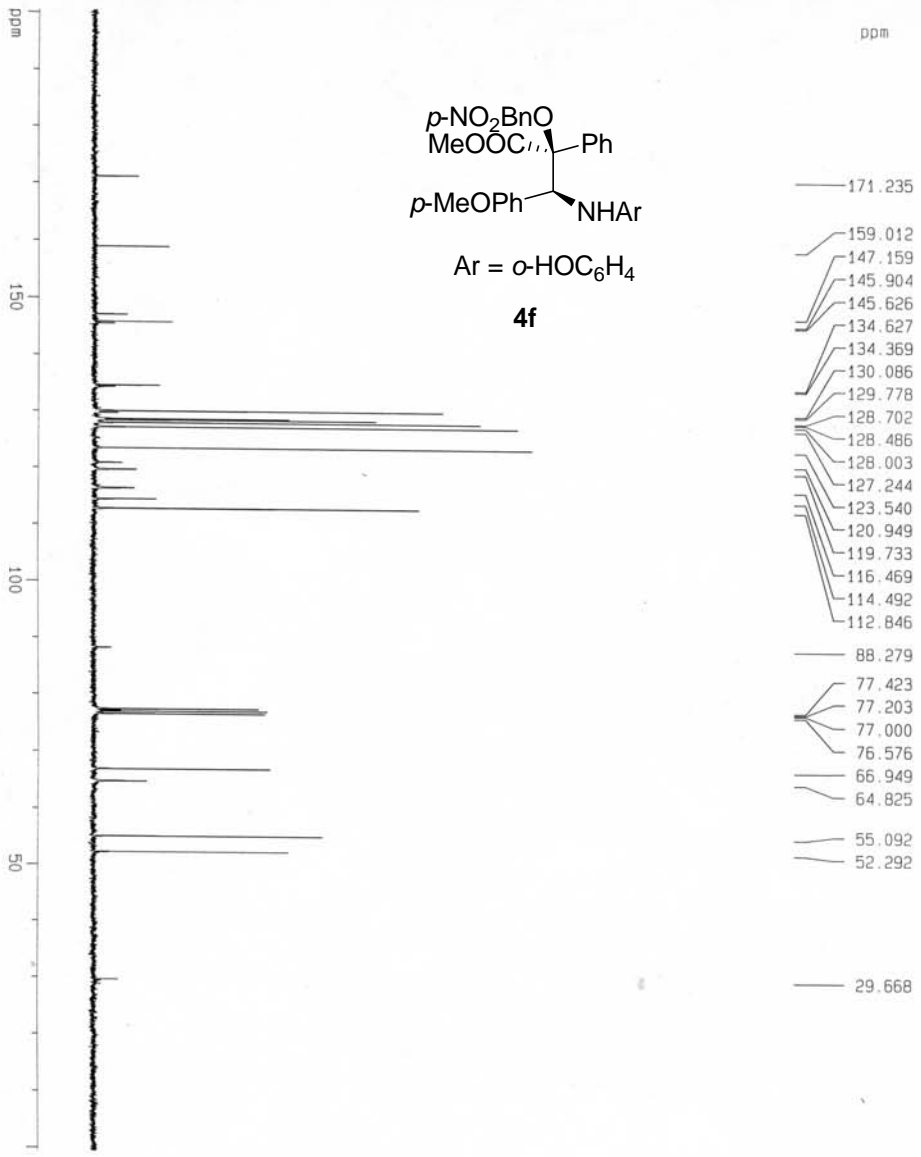
Current Data Parameters
 NAME 1399-hiv-903
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20061011
 Time 17.57
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 512
 DM 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.000000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SFO1 300.132082 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300124 MHz
 KW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

10 NMR plot parameters
 CX 20.00 cm
 CY 12.00 cm
 FIP 10.000 ppm
 F1 3001.30 Hz
 F2P -0.300 ppm
 F2 -150.06 Hz
 PPM1CM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm

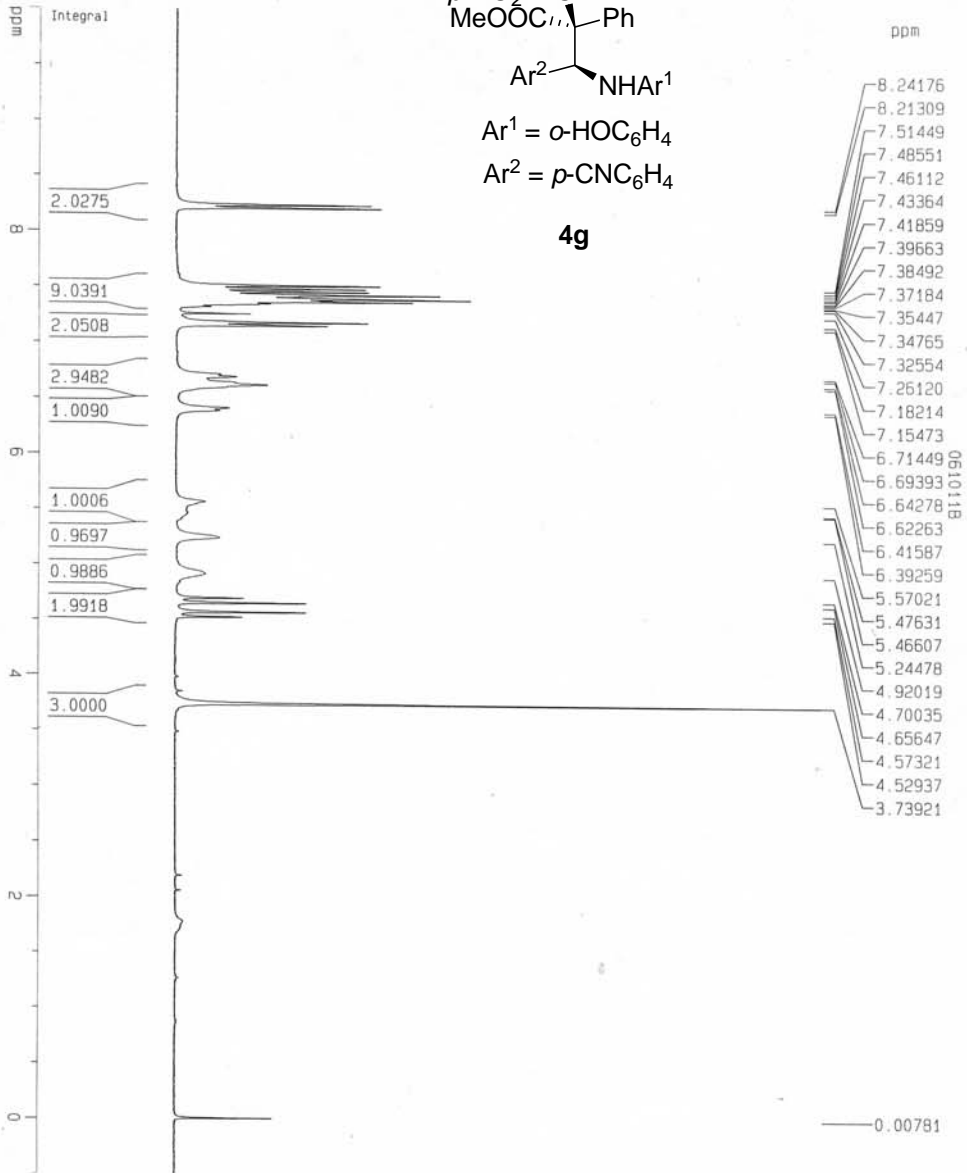
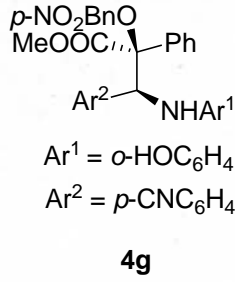


- 171.235
- 159.012
- 147.159
- 145.904
- 145.626
- 134.627
- 134.369
- 130.086
- 129.778
- 128.702
- 128.486
- 128.003
- 127.244
- 123.540
- 120.949
- 119.733
- 116.469
- 114.492
- 112.846
- 88.279
- 77.423
- 77.203
- 77.000
- 76.576
- 66.949
- 64.825
- 55.092
- 52.292
- 29.668

h061011A

```

Current Data Parameters
NAME      1399-nh-903
EXPNO    2
PROCNO   1
-----
F2 - Acquisition Parameters
Date_    20061011
Time     20:28
INSTRUM  av300
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg
TD        65536
SOLVENT  CDCl3
NS        1286
DS        4
SMH       22675.736 HZ
FIDRES    0.346004 HZ
AQ         1.4451189 sec
RG         8192
DM         22.050 usec
DE         6.00 usec
TE         300.0 K
D1         2.00000000 sec
d11        0.03000000 sec
d12        0.00002000 sec
-----
***** CHANNEL f1 *****
NUC1       13C
P1         5.50 usec
PL1        -6.00 dB
SFO1       75.473598 MHz
-----
***** CHANNEL f2 *****
CPDPRG2   MAlt216
NUC2       1H
PCPD2      80.00 usec
PL2        -2.00 dB
PL12       17.70 dB
PL13       17.71 dB
SFO2       300.1312005 MHz
-----
F2 - Processing parameters
SI         65536
SF         75.467350 MHz
WDW        EM
SSB        0
LB         1.00 HZ
GB         0
PC         1.40
-----
1D NMR plot parameters
CX         20.00 cm
CY         8.00 cm
F1P        200.500 DDM
F1         13131.28 HZ
F2P        -0.500 DDM
F2         -31.73 HZ
PPMCK     10.05000 ppm/cm
HZCM      738.45087 HZ/cm
  
```



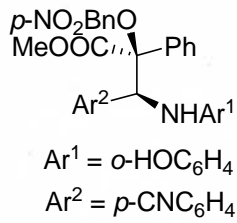
Current Data Parameters
 NAME 1399-hix-907
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20061012
 Time 10.10
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 512
 DM 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

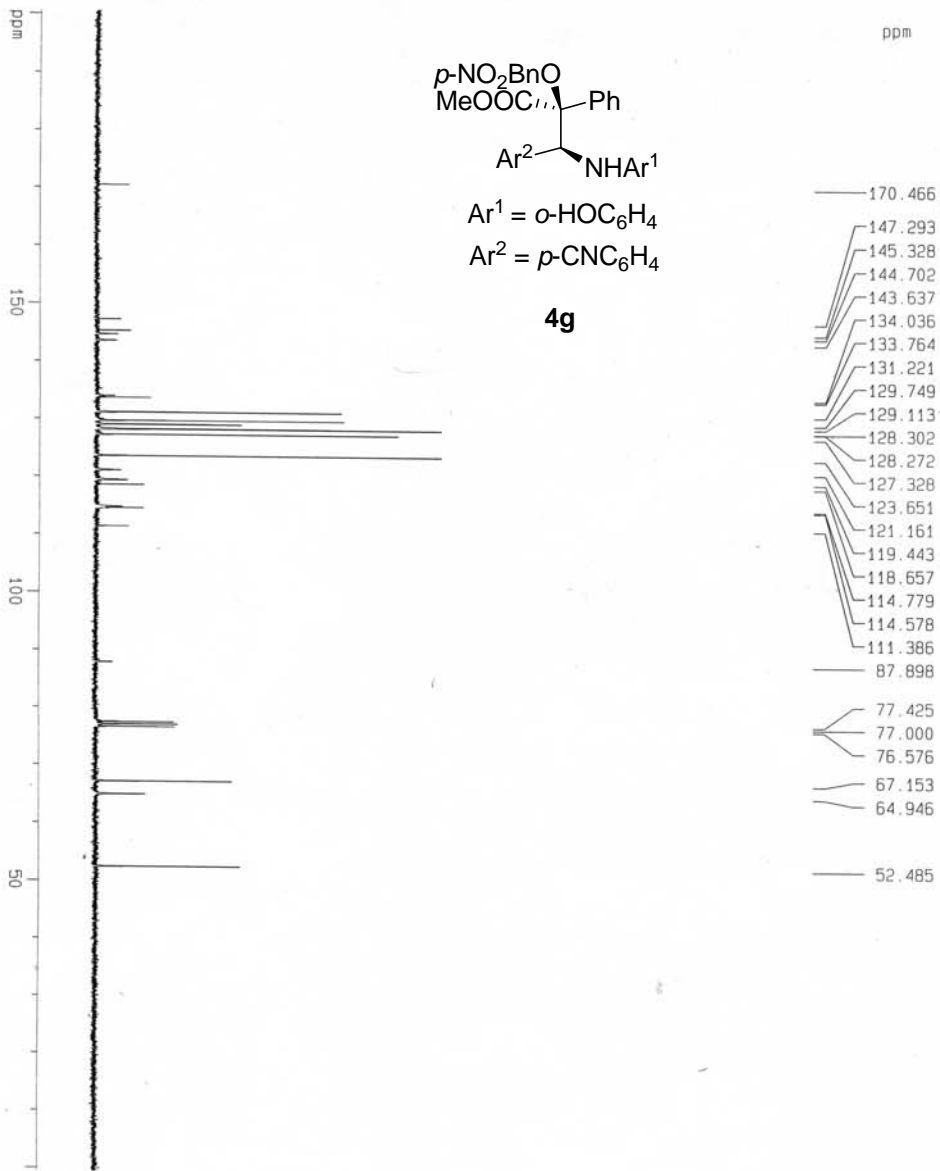
***** CHANNEL f1 *****
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SFO1 300.1320882 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300124 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 14.00 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPMCKM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm



4g



061011B

Current Data Parameters
 NAME 1399-nlx-907
 EXPNO 2
 PROCNO 1

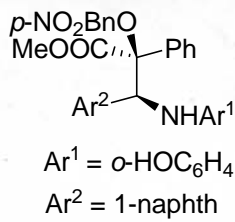
F2 - Acquisition Parameters
 Date_ 20061012
 Time 10.13
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 2999
 SOLVENT CDCl3
 NS 558
 DS 4
 SMH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451189 sec
 RG 8192
 OW 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SF01 75.475598 MHz

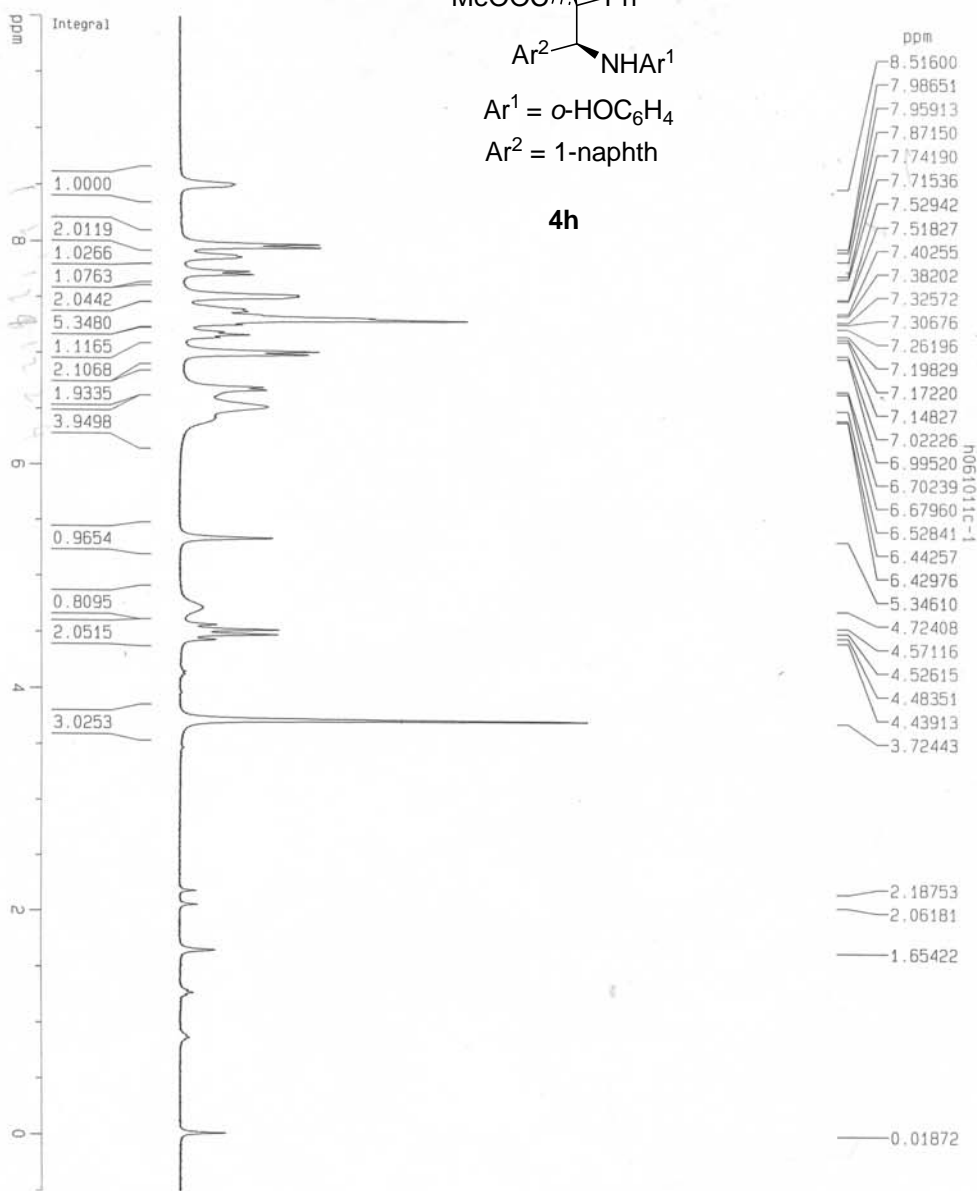
***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SF02 300.1312005 MHz

F2 - Processing parameters
 SI 65536
 SF 75.467752 MHz
 MVM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 6.00 cm
 F1P 200.500 ppm
 F1 15131.29 Hz
 F2P -37.73 Hz
 F2 10.05000 ppm/cm
 HZCM 758.45099 Hz/cm



4h



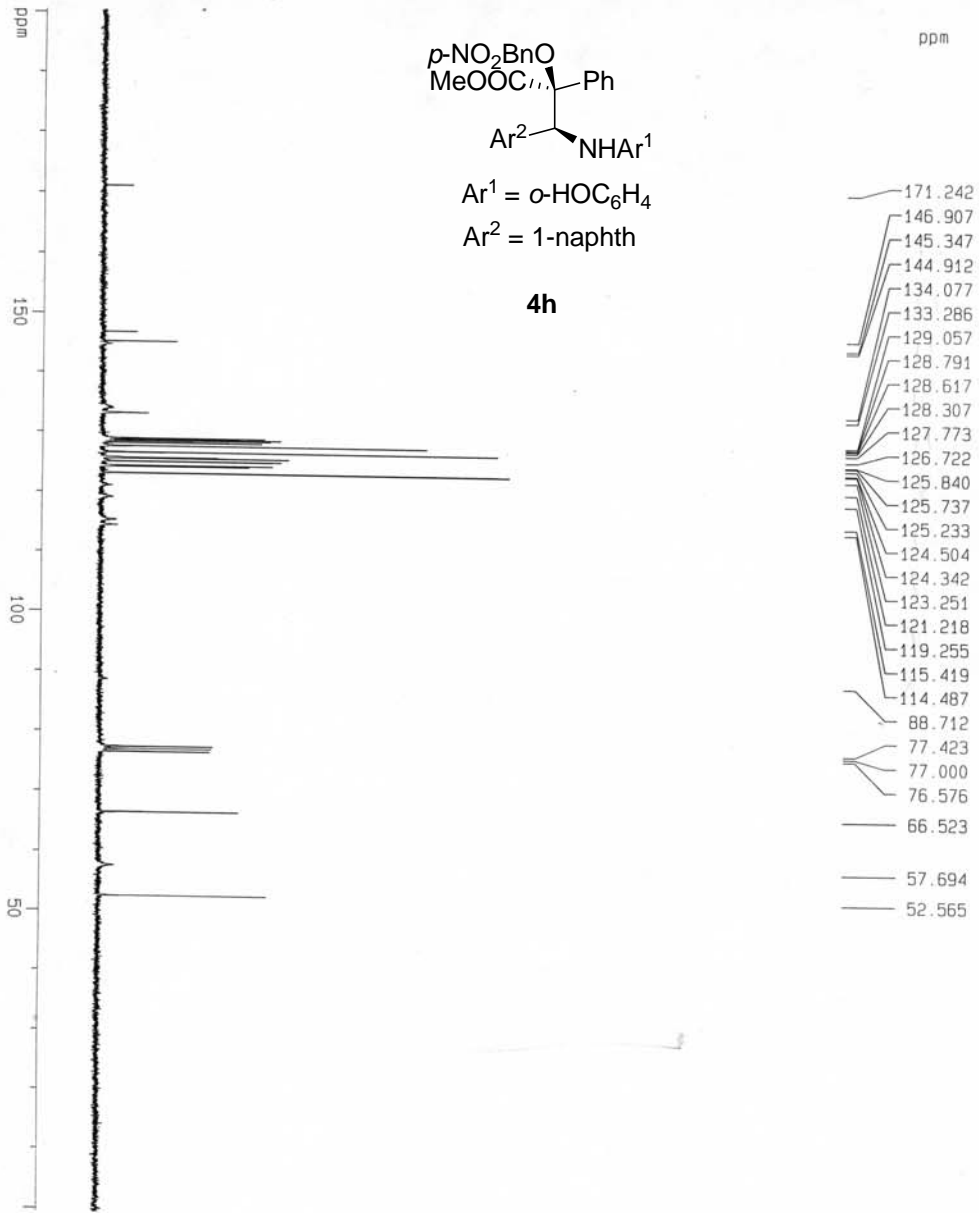
Current Data Parameters
 NAME 1399-nhx-912
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20061012
 Time 18.28
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 256
 DM 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.000000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SF01 300.1320882 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300124 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CV 7.00 cm
 F1p 10.000 ppm
 F1 3001.30 Hz
 F2p -0.500 ppm
 F2 -150.06 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm



h061011c-1

Current Data Parameters
 NAME 1399-nhx-912
 EXPNO 2
 PROCNO 1

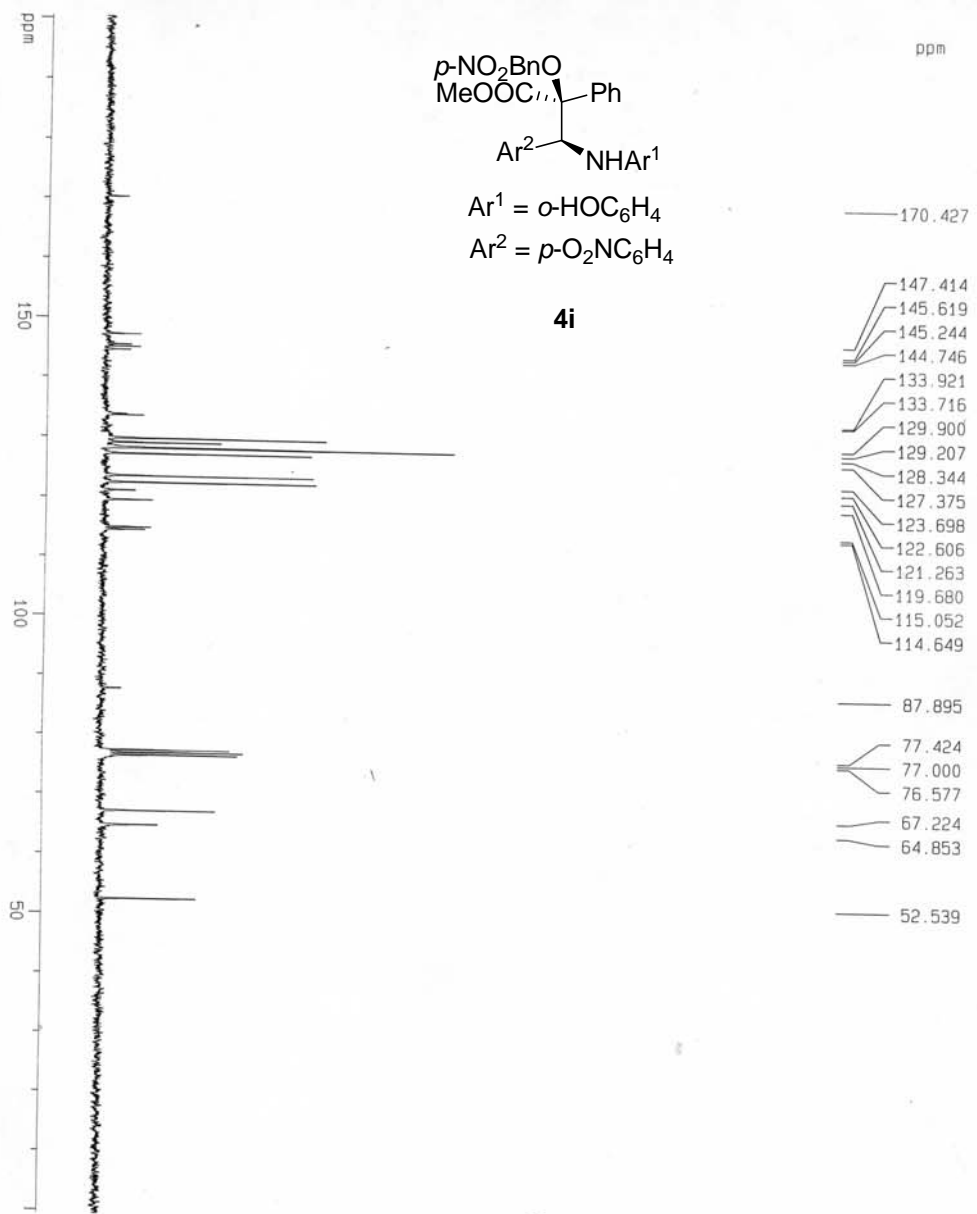
F2 - Acquisition Parameters
 Date_ 20061012
 Time 20.36
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 1006
 DS 4
 SMH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.443188 sec
 RG 6192
 DM 22.050 usec
 DE 6.00 usec
 IE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 D12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SFO1 75.477599 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 65536
 SF 75.467540 MHz
 MDN EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 7.00 cm
 F1P 200.500 ppm
 F1 15131.28 Hz
 F2P -0.500 ppm
 F2 -37.73 Hz
 PRICK 10.05000 ppm/cm
 HZCM 758.45087 Hz/cm



h0610128

Current Data Parameters
 NAME 1399-nhx-915
 EXPNO 2
 PROCNO 1

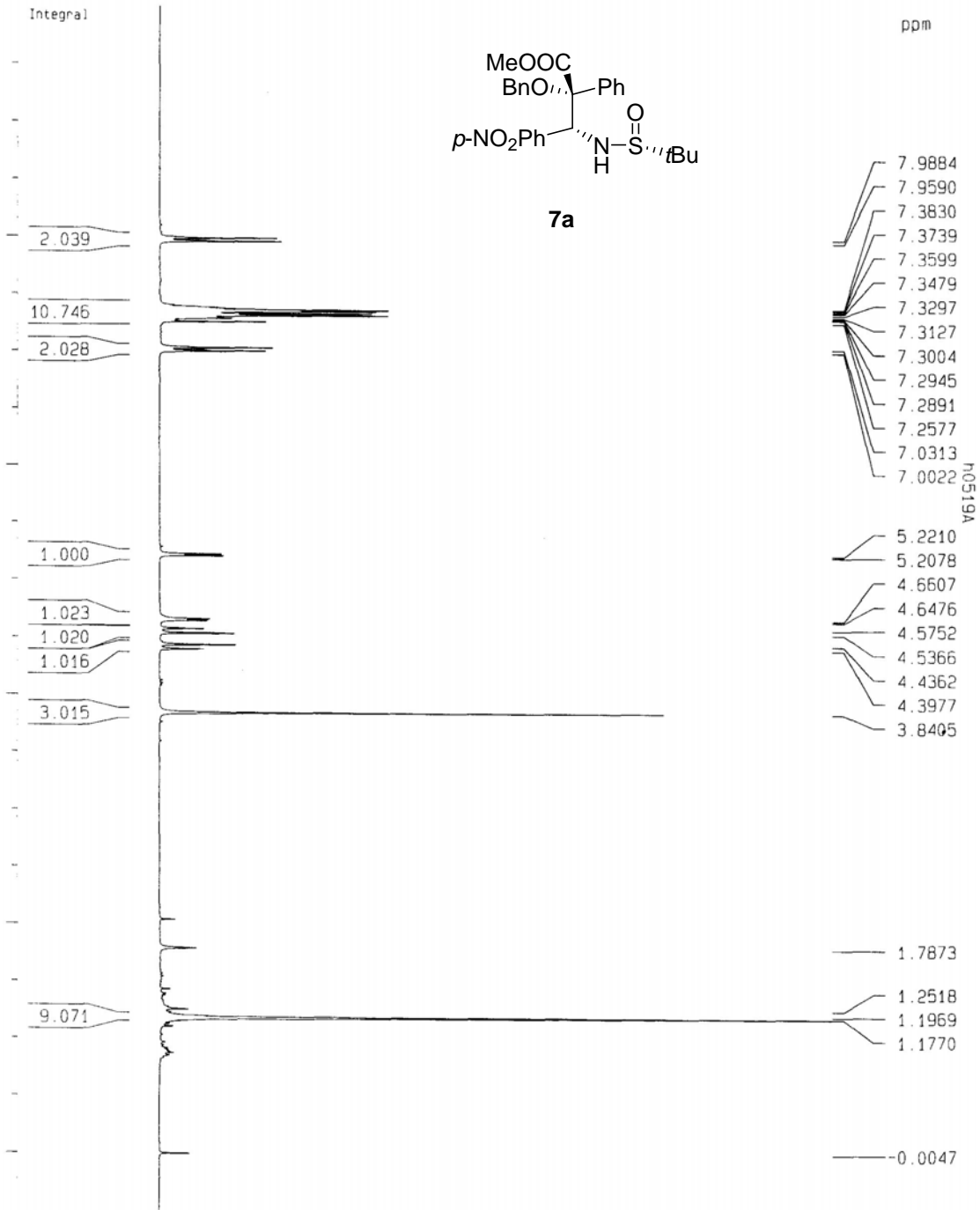
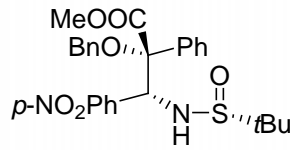
F2 - Acquisition Parameters
 Date_ 20051014
 Time 16.30
 INSTRUM sv300
 PROBNM 5 mm QNP 1H/13
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 621
 DS 4
 SMI 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451188 sec
 RG 8192
 DE 22.050 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SF01 75.4775998 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 P2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SF02 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677532 MHz
 MDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CI 5.00 cm
 F1P 200.500 ppm
 F1 15131.28 Hz
 F2P -0.500 ppm
 F2 -37.73 Hz
 PPMCK 10.05000 ppm/cm
 HZCK 758.45087 Hz/cm



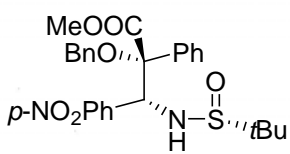
Current Data Parameters
 NAME 1399-hix-859
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060904
 Time 12.05
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 9
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 512
 JM 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

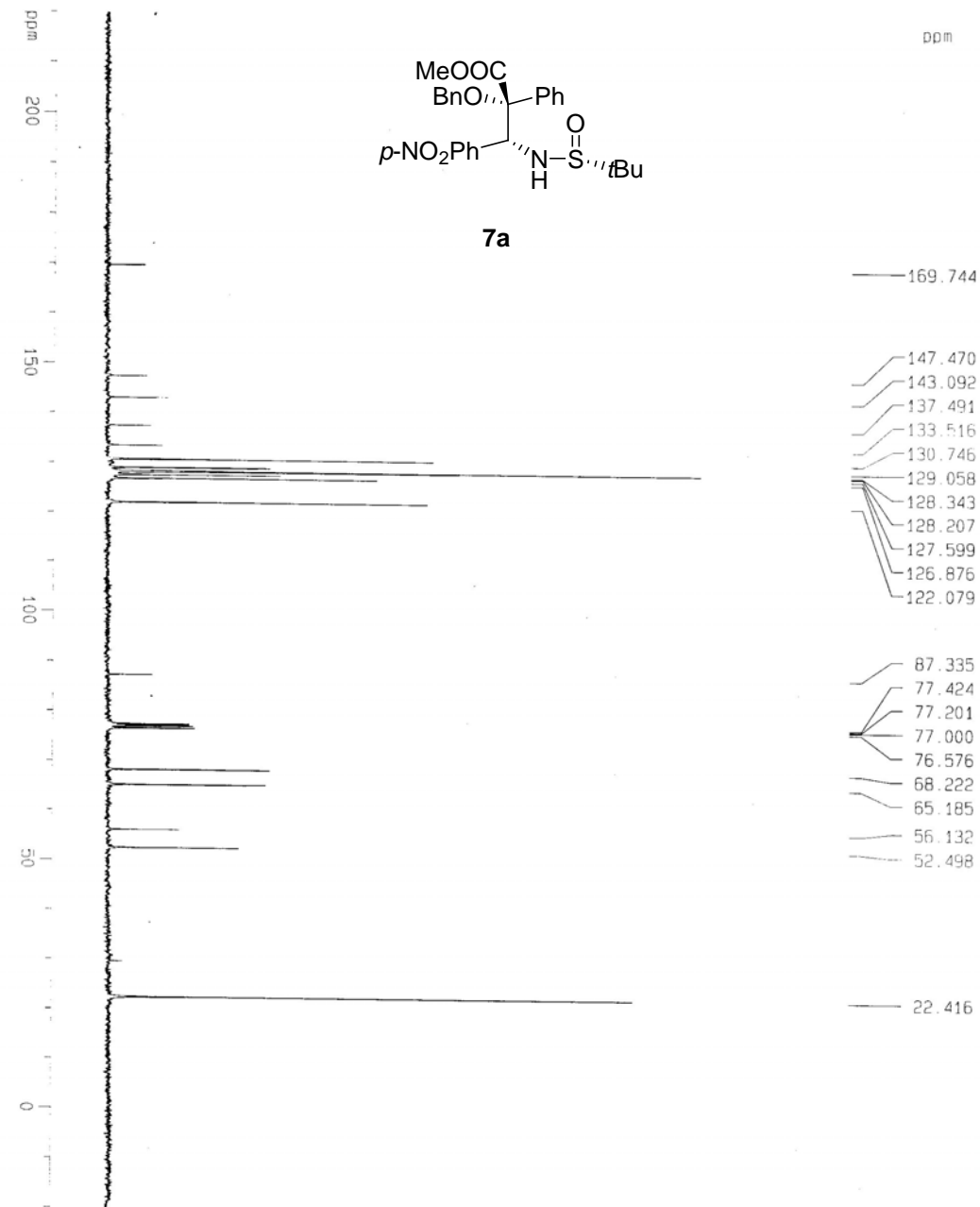
==== CHANNEL f1 =====
 NUC1 1H
 p1 3.00 usec
 PL1 -2.00 dB
 SFO1 300.1320882 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300130 MHz
 MDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 DC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 20.00 cm
 F1P 10.000 ppm
 F2P 3001.30 Hz
 F2 -0.500 ppm
 -150.06 Hz
 FPMCM 0.52500 ppm/Hz
 FZCM 157.56825 Hz/Hz



7a



- 169.744
- 147.470
- 143.092
- 137.491
- 133.516
- 130.746
- 129.058
- 128.343
- 128.207
- 127.599
- 126.876
- 122.079
- 87.335
- 77.424
- 77.201
- 77.000
- 76.576
- 68.222
- 65.185
- 56.132
- 52.498
- 22.416

Current Data Parameters
 NAME 1399-nhx-582
 EXPNO 2
 PROCNO 1

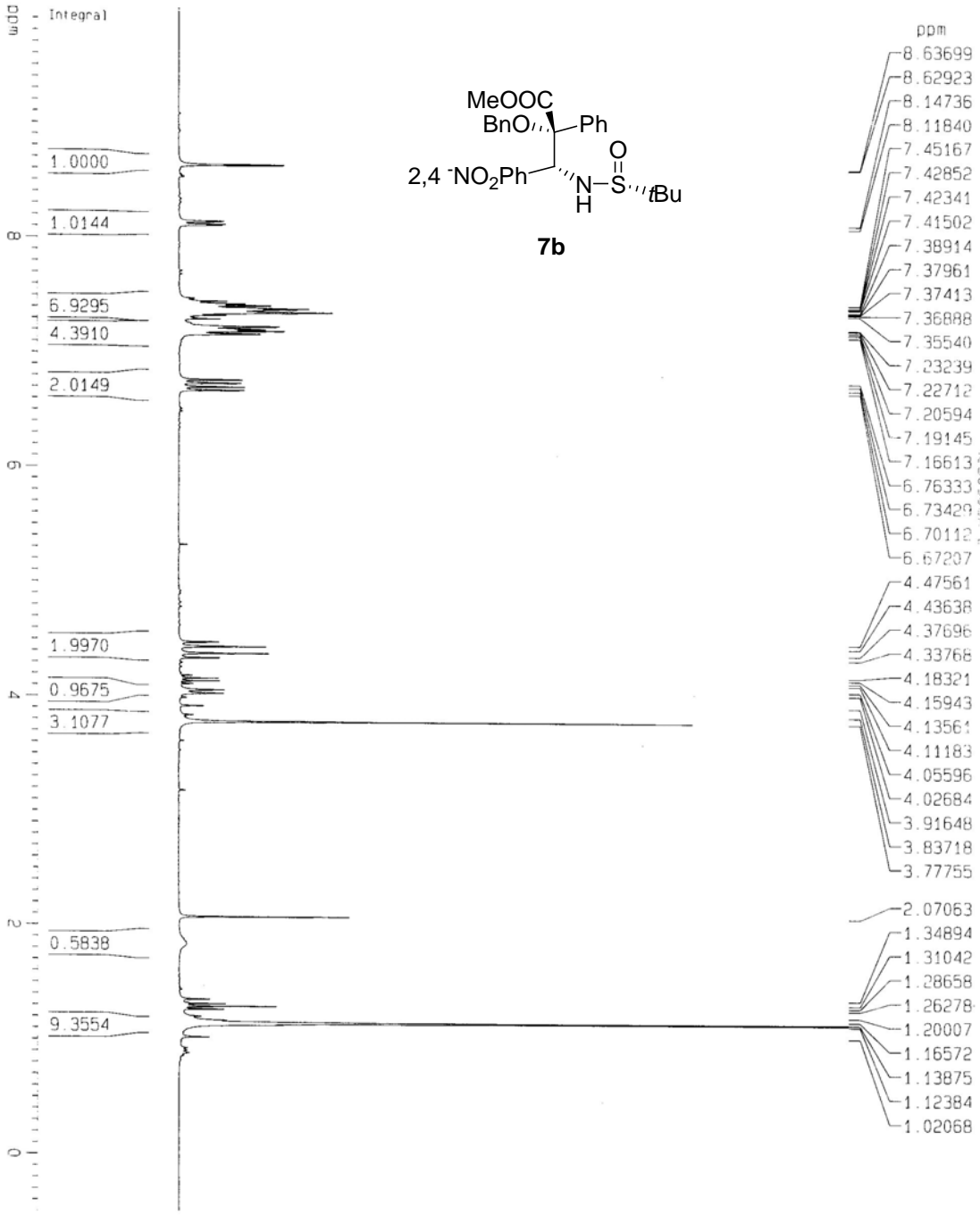
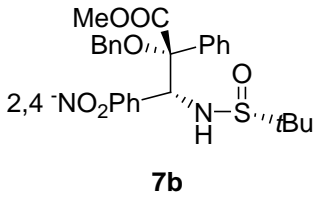
F2 - Acquisition Parameters
 Date_ 20060111
 Time 17:56
 INSTRUM av300
 PROBNM 5 mm QNP 1H/13
 PULPROG zgpg
 TD 65536
 SOLVENT DMSO
 NS 275
 DS 4
 SWH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451188 sec
 RG 8192
 DW 22.050 usec
 DE 6.00 usec
 TE 300.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 D12 0.00020000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SFO1 75.4775598 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 P2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677577 MHz
 KW 8
 EN
 SSB 0
 LB 3.00-Hz
 GB 0
 CB 0
 PC 1.40

13C NMR D102 Parameters
 CV 20.00 cm
 F1 10.00 cm
 F1P 220.005 ppm
 F1 16603.29 Hz
 F2P -20.289 ppm
 F2 -1531.19 Hz
 PPM0M 12.01472 ppm/cm
 AZCM 906.72386 Hz/cm



- ppm
- 8.63699
 - 8.62923
 - 8.14736
 - 8.11840
 - 7.45167
 - 7.42852
 - 7.42341
 - 7.41502
 - 7.38914
 - 7.37961
 - 7.37413
 - 7.36888
 - 7.35540
 - 7.23239
 - 7.22712
 - 7.20594
 - 7.19145
 - 7.16613
 - 6.76333
 - 6.73429
 - 6.70112
 - 6.67207
 - 4.47561
 - 4.43638
 - 4.37696
 - 4.33768
 - 4.18321
 - 4.15943
 - 4.13561
 - 4.11183
 - 4.05596
 - 4.02684
 - 3.91648
 - 3.83718
 - 3.77755
 - 2.07063
 - 1.34894
 - 1.31042
 - 1.28658
 - 1.26278
 - 1.20007
 - 1.16572
 - 1.13875
 - 1.12384
 - 1.02068

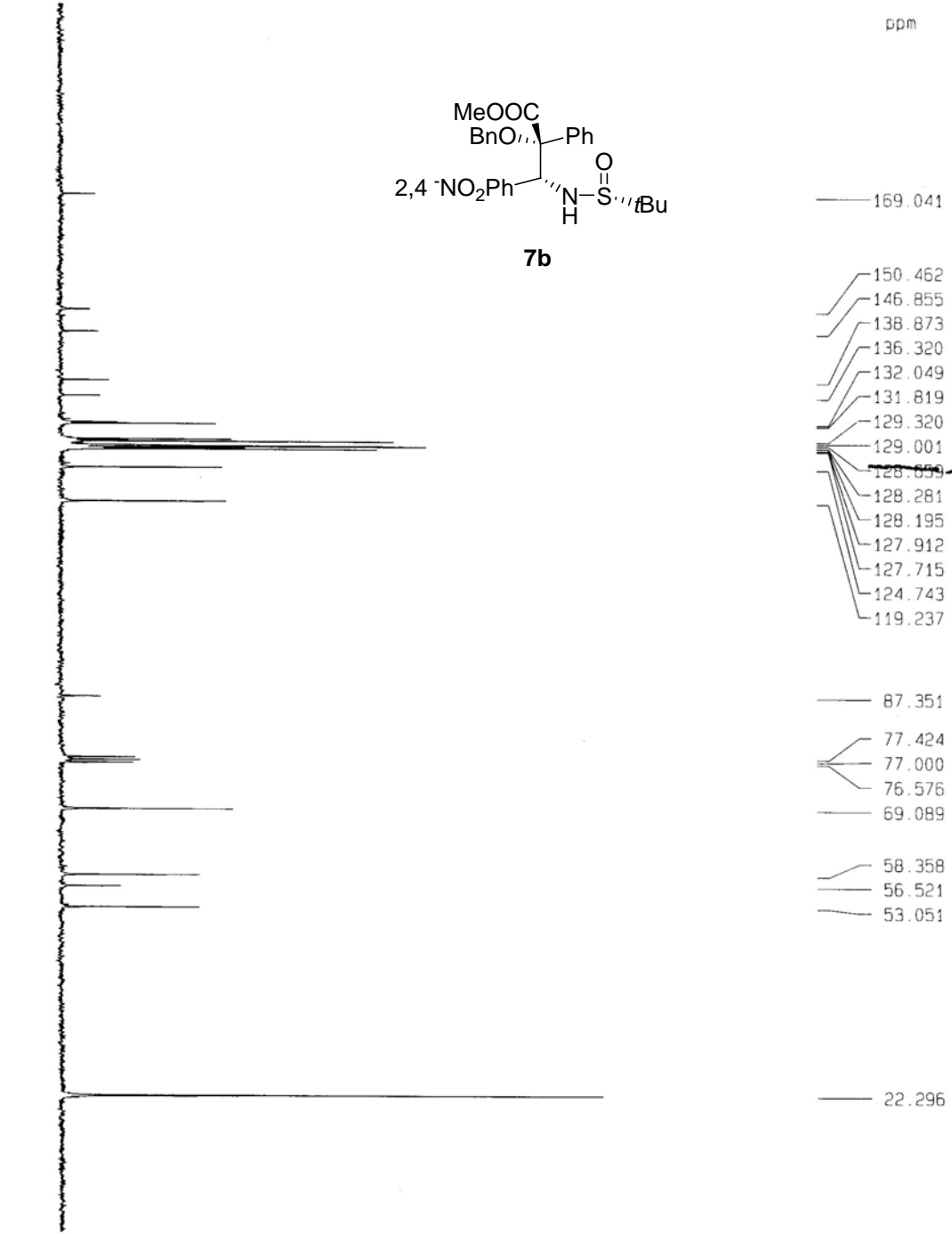
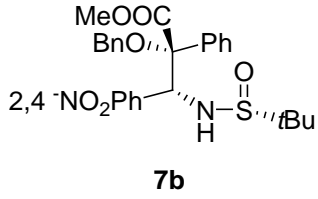
Current Data Parameters
 NAME 1399-hhx-410
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050906
 Time 11.21
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 512
 DW 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

CHANNEL f1
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SF01 300.1320882 MHz

F2 - Processing parameters
 S1 32768
 SF 300.1300013 MHz
 KDM EM
 SSB 0
 -B 0.10 Hz
 SB 0
 PC 1.00

1D NMR Plot Parameters
 CX 20.00 cm
 CY 20.00 cm
 F1P 10.000 DPM
 F1 3001.30 Hz
 F2P -0.500 DPM
 F2 -150.06 Hz
 CPD/CM 0.52500 CPM/cm
 ZCM 157.56825 Hz/cm



- 169.041
- 150.462
- 146.855
- 138.873
- 136.320
- 132.049
- 131.819
- 129.320
- 129.001
- 128.659
- 128.281
- 128.195
- 127.912
- 127.715
- 124.743
- 119.237
- 87.351
- 77.424
- 77.000
- 76.576
- 69.089
- 58.358
- 56.521
- 53.051
- 22.296

Current Data Parameters
 NAME 1399-nrx-410
 EXPNO 2
 PROCNO 1

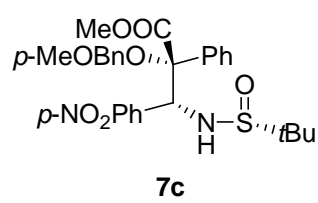
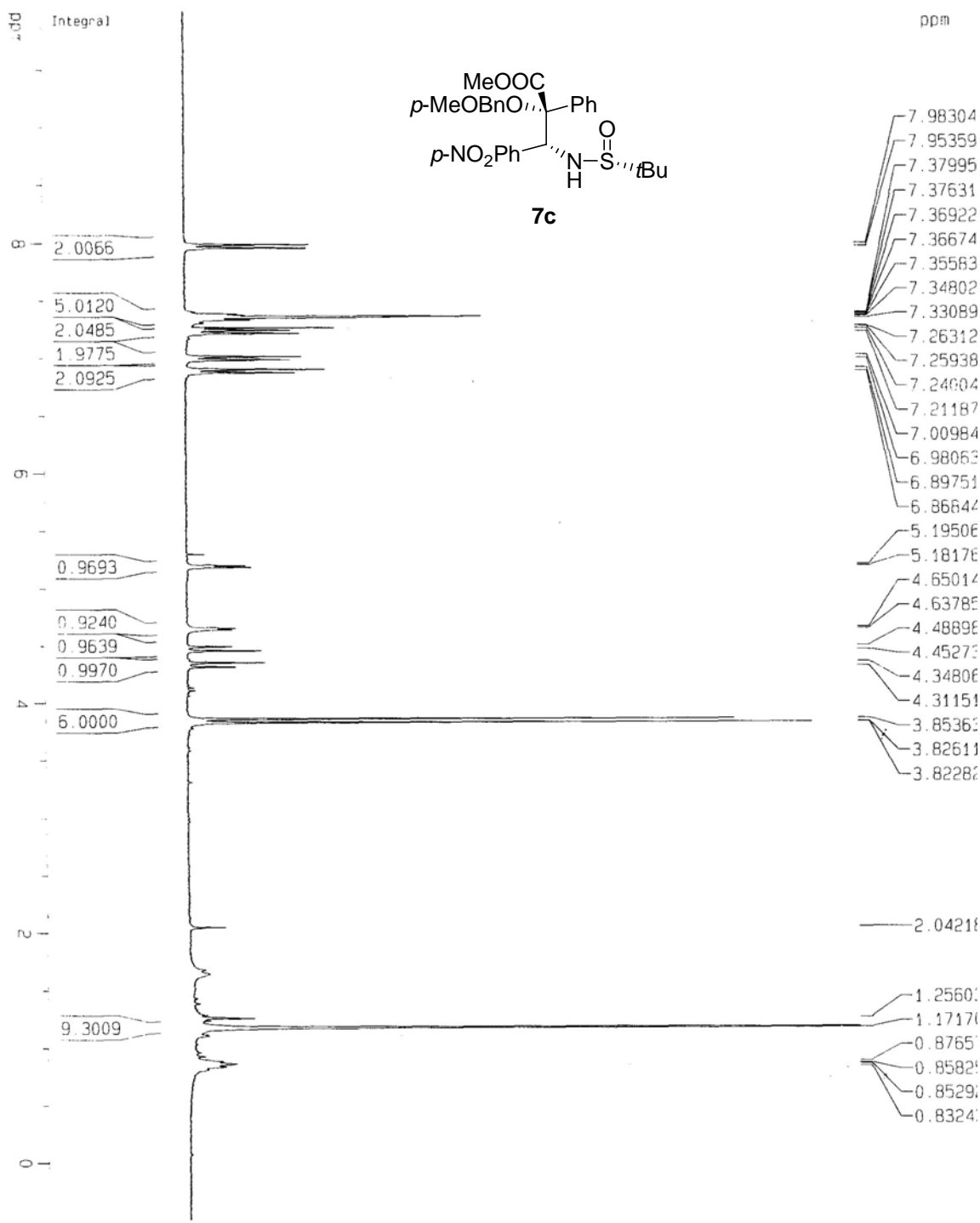
F2 - Acquisition Parameters
 Date_ 20050909
 Time 11.50
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 2909
 FIDRES 65536
 SOLVENT CDCl3
 NS 360
 DS 4
 SFO1 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451188 sec
 RG 8192
 DM 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 p1 5.50 usec
 pl1 -6.00 dB
 SF01 75.477598 MHz

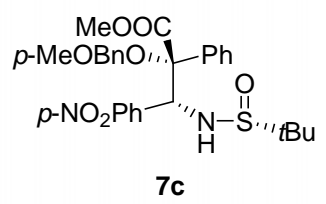
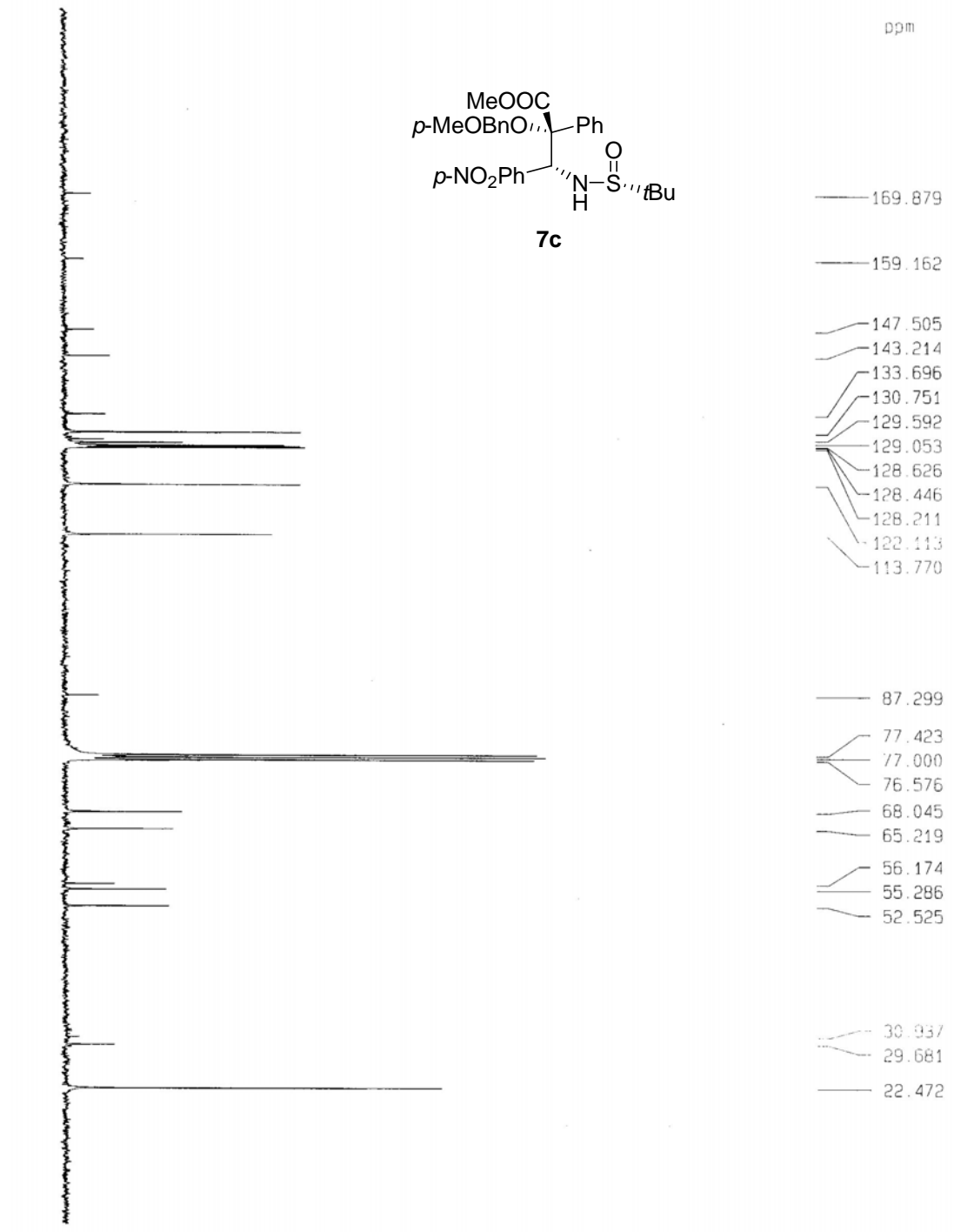
***** CHANNEL f2 *****
 CPOPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SF02 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677552 MHz
 MDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 5.00 cm
 F1P 200.005 ppm
 F1 15093.93 Hz
 F2P -0.005 ppm



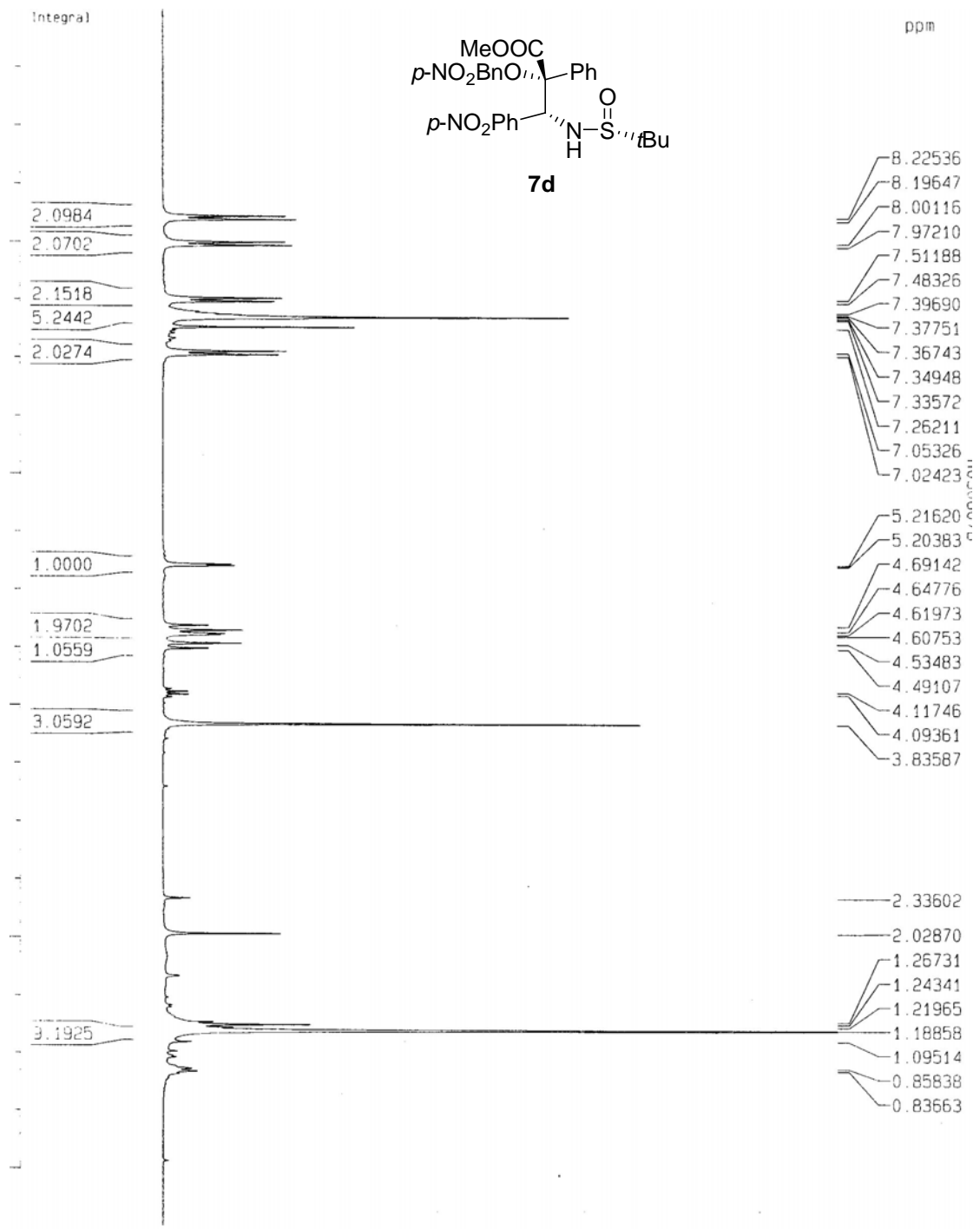
Current Data Parameters
 NAME 1399-hhx-273
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameter
 Date_ 20050531
 Time 9.31
 INSTRUM av300
 PROBRD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 s
 RG 128
 DW 83.400 us
 DE 6.00 us
 TE 300.0 K
 D1 1.00000000 s
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 3.00 us
 PL1 -2.00 dB
 SF01 300.1320882 MHz
 F2 - Processing parameter
 SI 32768
 SF 300.1300124 MHz
 KW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00
 1D NMR plot parameters
 CX 20.00 C
 CY 20.00 C
 F1P 10.000 P
 F1 3001.30 Hz
 F2P -0.500 P
 F2 -150.05 Hz
 dPRGM 0.52500 P
 -ZCM 157.56825 Hz



- 169.879
- 159.162
- 147.505
- 143.214
- 133.696
- 130.751
- 129.592
- 129.053
- 128.626
- 128.446
- 128.211
- 122.113
- 113.770
- 87.299
- 77.423
- 77.000
- 76.576
- 68.045
- 65.219
- 56.174
- 55.286
- 52.525
- 30.937
- 29.681
- 22.472

```

NAME          1399-251-583
EXPNO         2
PROCNO       1
-----
F2 - Acquisition Parameters
Date_         20060111
Time          19.03
INSTRUM      ac300
PROBHD       5 mm QNP-1H/13
PULPROG      zgpg3
TD           65536
SOLVENT      DMSO
NS           4491
DS           4
SMH          22675.736 HZ
FIDRES       0.346004 HZ
AQ           1.441189 sec
RG           8192
DM           22.050 usec
DE           6.00 usec
TE           300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec
-----
***** CHANNEL f1 *****
NUC1         13C
P1           5.50 usec
PL1         -6.00 dB
SFO1        75.475598 MHz
-----
***** CHANNEL f2 *****
CPDPRG2     waltz16
NUC2         1H
PCPD2       80.00 usec
PL2         -2.00 dB
PL12        1.70 dB
PL13        17.71 dB
SFO2        300.1312005 MHz
-----
F2 - Processing Parameters
SI           32768
SF           75.481532 MHz
WDW          EM
SSB          0
-B          3.00 Hz
GB           0
PC           1.40
-----
1D NMR plot parameters
CX           20.00 cm
CY           5.00 cm
F1P          200.630 ppm
F1           15093.55 Hz
F2P          -0.000 ppm
  
```



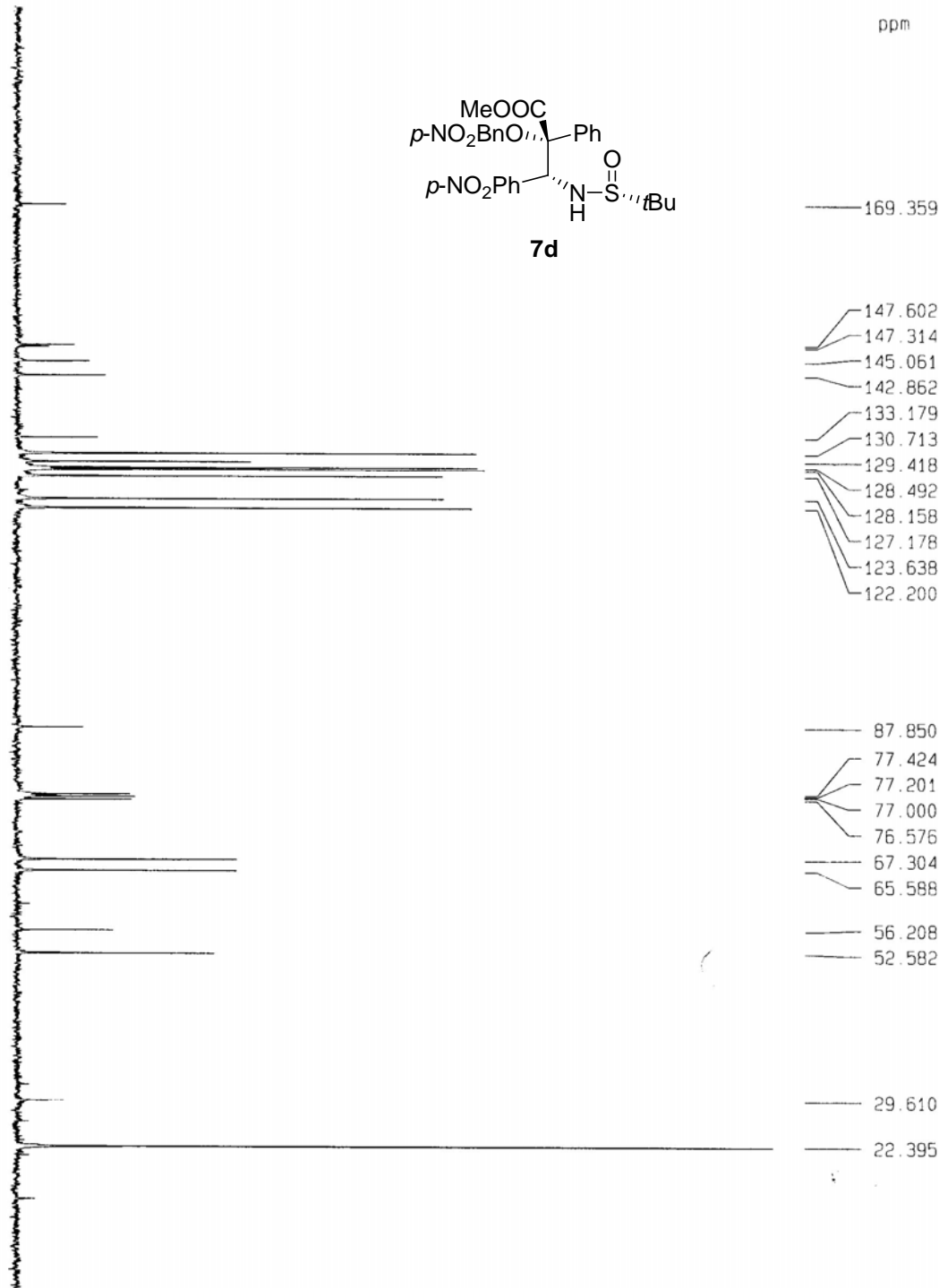
Current Data Parameters
 NAME 1399-nhx-289
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050608
 Time 19.09
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 se
 RG 128
 DM 83.400 us
 DE 6.00 us
 TE 300.0 K
 O1 1.00000000 se

===== CHANNEL f1 =====
 NUC1 1H
 P1 3.00 us
 PL1 -2.00 dB
 SFO1 300.1320882 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300124 MHz
 WDW EM
 SSB 0
 -B 0.10 Hz
 GB 0
 -C 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 20.00 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 DPMCM 1.52500 ppm
 -ZCM 15.56825 Hz



Current Data Parameters
 NAME 1399-nh-x-289
 EXPNO 2
 PROCNO 1

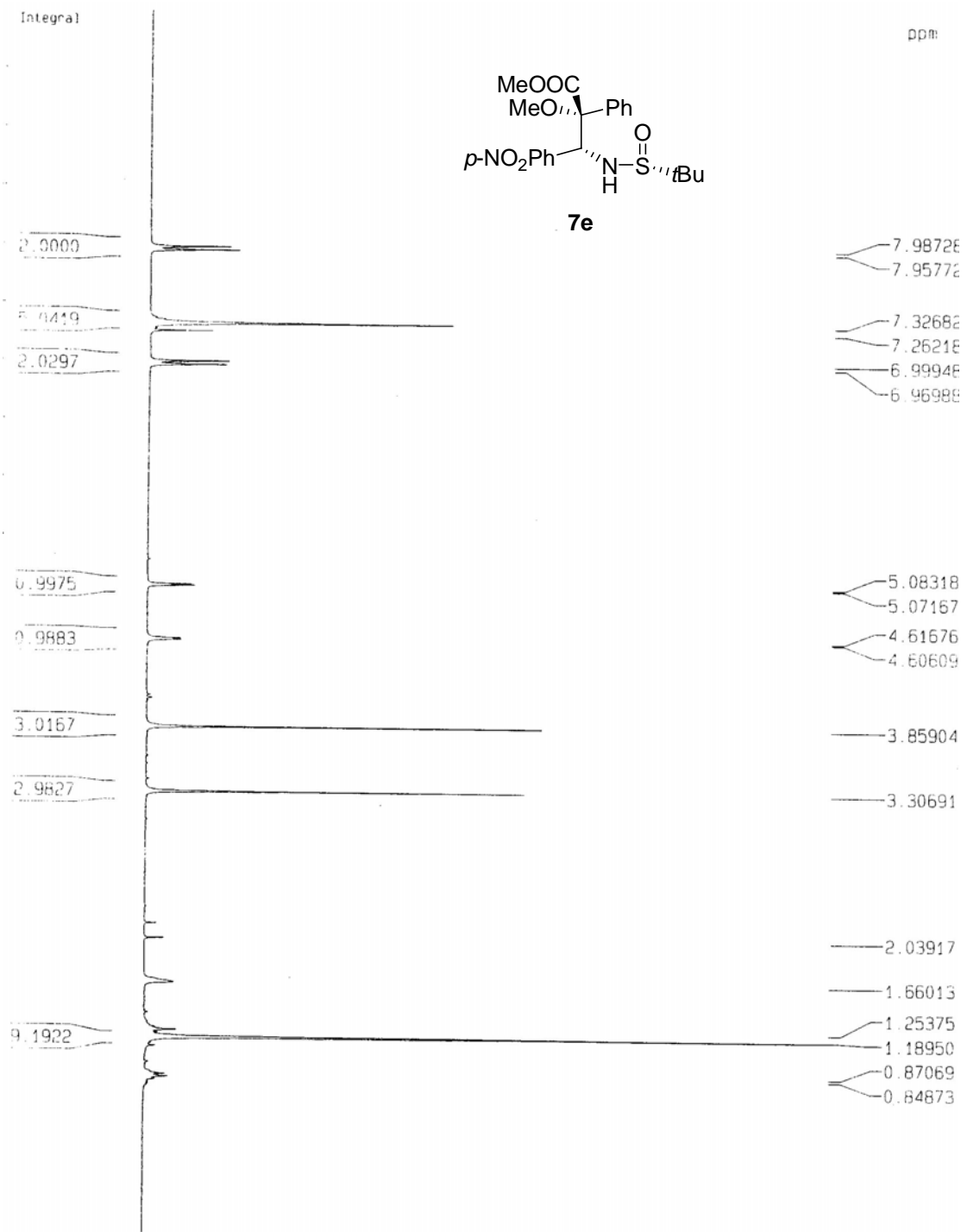
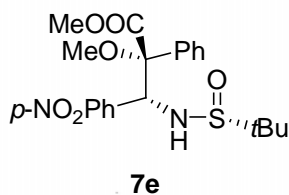
F2 - Acquisition Parameters
 Date_ 20050608
 Time 19.15
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg
 TD 65536
 SOLVENT COCl3
 NS 368
 DS 4
 SMH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451188 sec
 RG 8192
 DM 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SFO1 75.4775598 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677559 MHz
 MDM EM
 SSB 0
 AB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.00 cm
 F1P 200.005 ppm



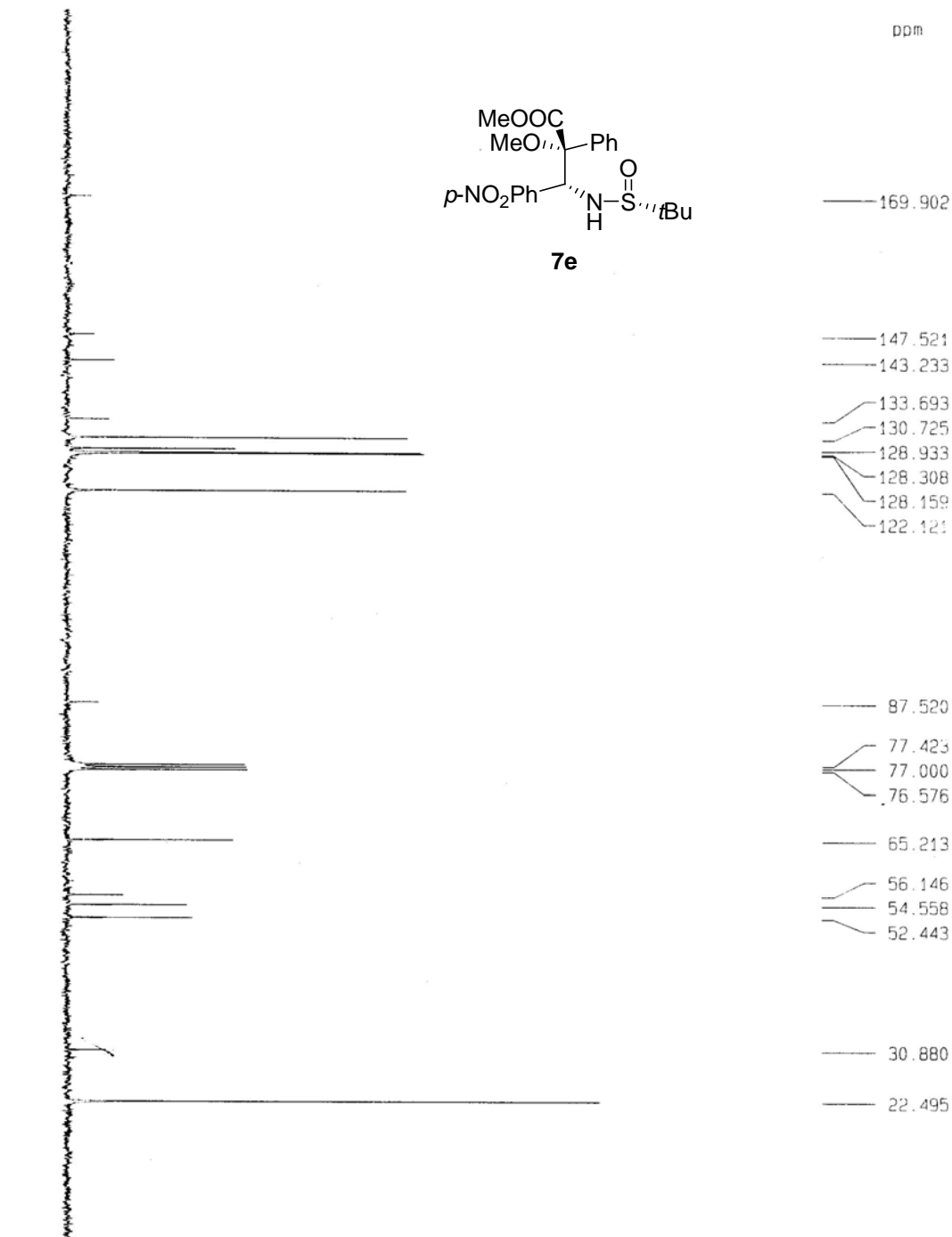
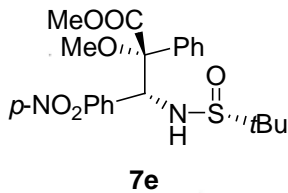
Current Data Parameters
 NAME 1399-thx-314
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050629
 Time 17.05
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5995.2041
 FIDRES 0.1829591
 AQ 2.7329011
 RG 296
 JM 83.400
 DE 6.00
 TE 300.0
 D1 1.00000000

===== CHANNEL f1 =====
 NUC1 1H
 P1 3.00
 PL1 -2.00
 SF01 300.1320882

F2 - Processing parameters
 SI 32768
 SF 300.1300124
 MDW EM
 SSB 0
 GB 0.10
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00
 CY 15.00
 F1 10.00
 F2 3001.30
 F3 -0.500
 F4 -150.06
 PRGM 0.52500



ppm

169.902

147.521

143.233

133.693

130.725

128.933

128.308

128.159

122.121

87.520

77.423

77.000

76.576

65.213

56.146

54.558

52.443

30.880

22.495

Current Data Parameters

NAME 1399-hix-314

EXPNO 2

PROCNO 1

F2 - Acquisition Parameters

Date_ 20050630

Time 12.21

INSTRUM AV300

PROBHD 5 mm QNP 1H/13

PULPROG zgpg

TD 65536

SOLVENT CDCl3

NS 900

DS 4

SWH 22675.736 Hz

FIDRES 0.346004 Hz

AQ 1.4451189 sec

RG 8192

DM 22.050 usec

DE 6.00 usec

TE 300.0 K

D1 2.00000000 sec

D11 0.03000000 sec

D12 0.00002000 sec

***** CHANNEL f1 *****

NUC1 13C

P1 5.50 usec

P11 -6.00 dB

SFO1 75.4775998 MHz

***** CHANNEL f2 *****

CPDPRG2 waltz16

NUC2 1H

PCPD2 60.00 usec

PL2 -2.00 dB

PL12 17.70 dB

PL13 17.71 dB

SFO2 300.1312005 MHz

F2 - Processing parameters

SI 32768

SF 75.4677598 MHz

WDW EM

SSB 0

LB 3.00 Hz

GB 0

PC 1.40

F0 NMR FID: Parameters

SI 32768

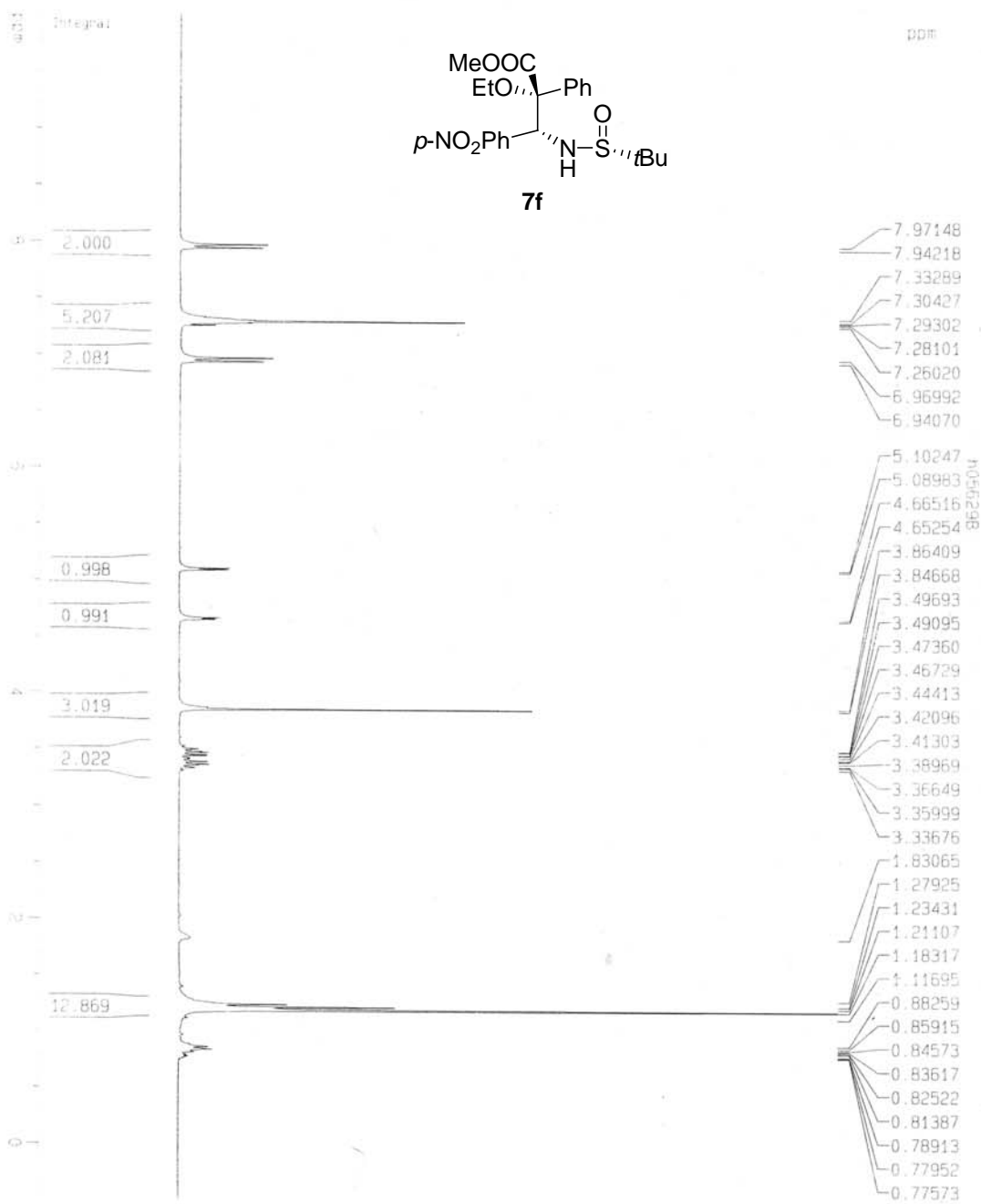
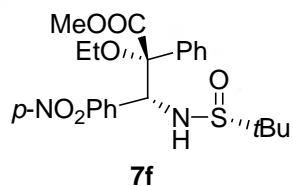
CF 20.00 cm

CP 9.00 cm

SP 200.305 ppm

F1 14052.53 Hz

F20 -0.025 ppm



Current Data Parameters:

NAME 1399-nhx-317

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20050630

Time 11.53

INSTRUM s3300

PROBHD 5 mm QNP 1H/13

PULPROG zg30

TD 32768

SOLVENT CDCl3

NS 8

DS 0

SWH 5995.204 Hz

FIDRES 0.182959 Hz

AQ 2.7329011 sec

RG 256

DM 83.400 usec

DE 5.00 usec

TE 300.0 K

D1 1.00000000 sec

***** CHANNEL f1 *****

NUC1 1H

P1 3.00 usec

PL1 -2.00 dB

SFO1 300.1320882 MHz

F2 - Processing parameters

SI 32768

SF 300.1320122 MHz

WDW EM

SSB 0

LB 0.10 Hz

GB 0

PC 1.00

10 NMR plot parameters

CX 20.00 cm

CY 15.00 cm

F1P 10.000 ppm

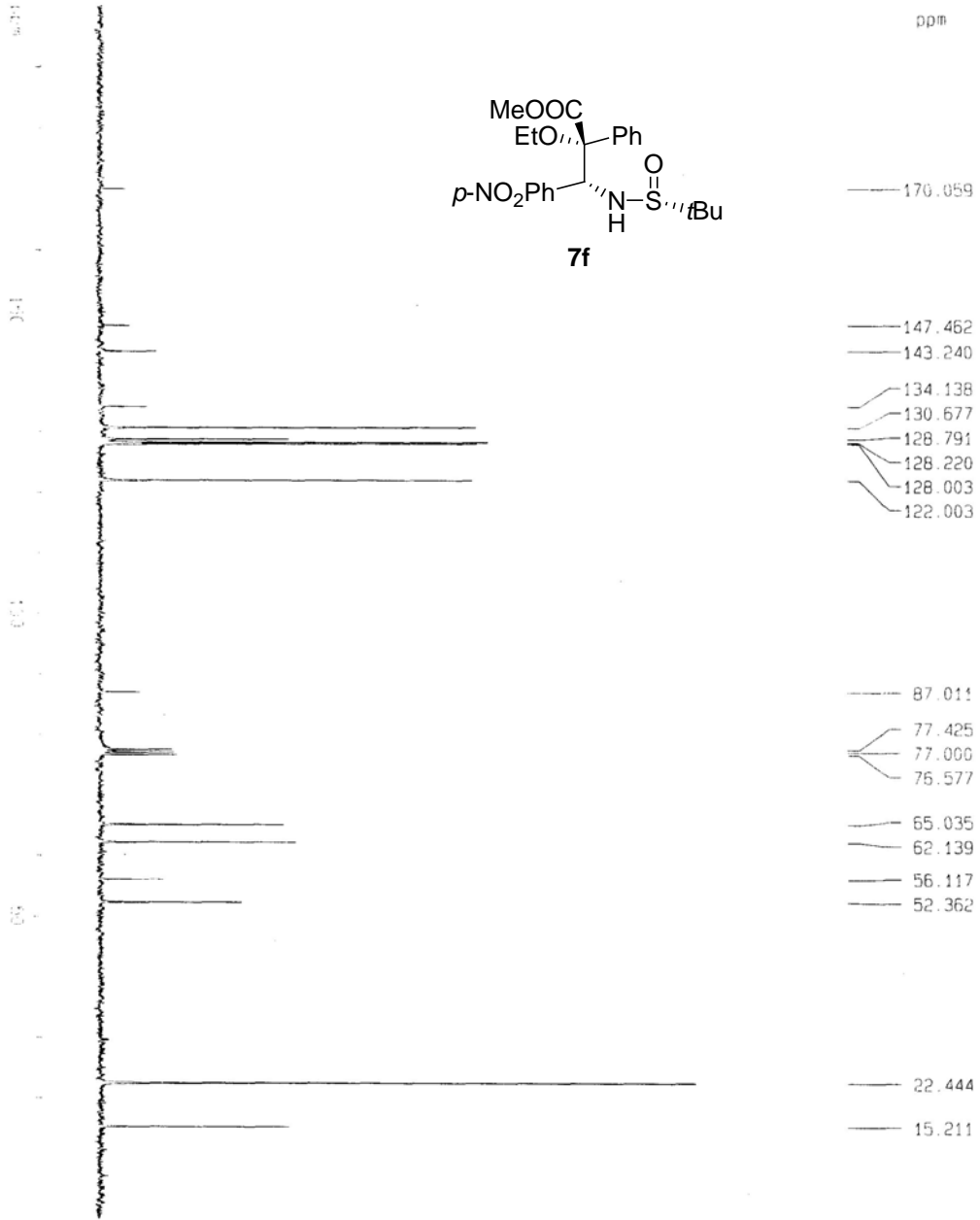
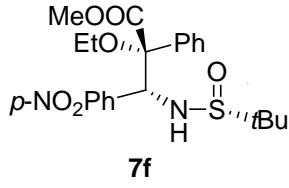
F1 300.130 Hz

F2P -0.500 ppm

F2 -15.05 Hz

APMCM 0 Hz, 500 ppm/cm

AZCM 15.754585 Hz/cm



Current Data Parameters

NAME	1398-nhx-317
EXPNO	2
PROCNO	1

2 - Acquisition Parameters

Date_	20050530
Time	17.28
INSTRUM	AV300
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg3
TD	65536
SOLVENT	CDCl3
VS	256
DS	4
SM	22875.736 HZ
TDRES	0.346004 HZ
AQ	1.4451188 sec
TG	8192
GM	22.050 usec
DE	6.00 usec
TE	300.0 K
TI	2.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

***** CHANNEL f1 *****

NUC1	13C
P1	5.50 usec
PL1	-6.00 dB
SFO1	75.475598 MHz

***** CHANNEL f2 *****

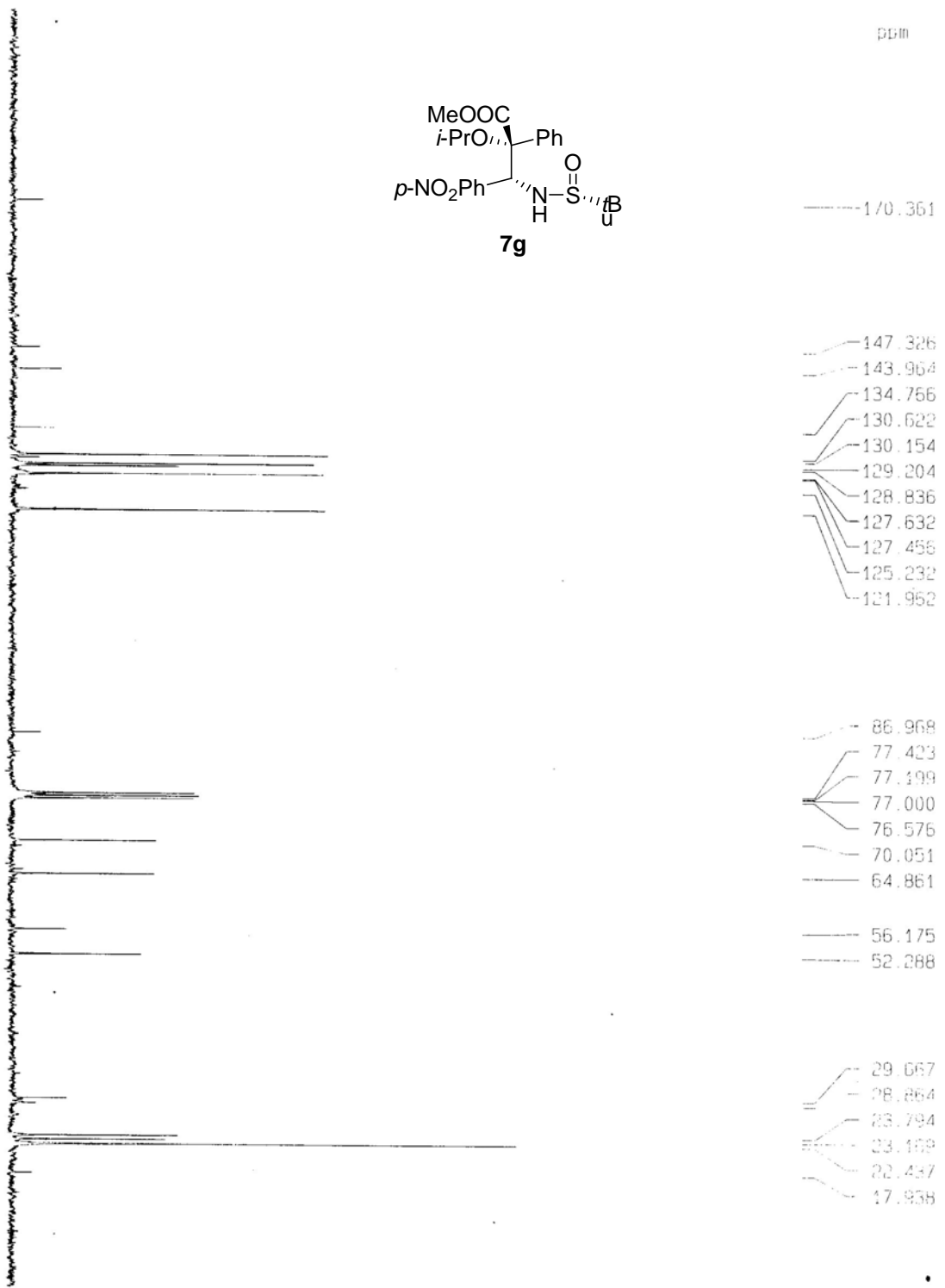
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	17.70 dB
PL13	17.71 dB
SFO2	300.1312005 MHz

2 - Processing parameters

SI	32768
SF	75.4677548 MHz
MDM	EM
SSB	0
B	3.00 Hz
GB	0
PC	1.40

10 - NMR 101: parameters

SI	20.00 sec
SI	10.00 sec
SI	200.005 sec
SI	15093.93 Hz
SI	-0.005 sec
SI	-0.35 Hz
SI	10.0050 sec
SI	54.1503 sec



```

NAME          13C-NMR-7g-1
EXPNO         2
PROCNO        1
F2 - Acquisition Parameters
Date_         20060306
Time         12.25
INSTRUM      AV300
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg
TD           65536
SOLVENT      CDCl3
VS           849
DS           4
SMH          22675.736
FIDRES       0.346004
AQ           1.4451166
RG           8152
CM           22.050
DE           5.00
TE           300.0
D1           2.00000000
d11          0.03000000
d12          0.00020000

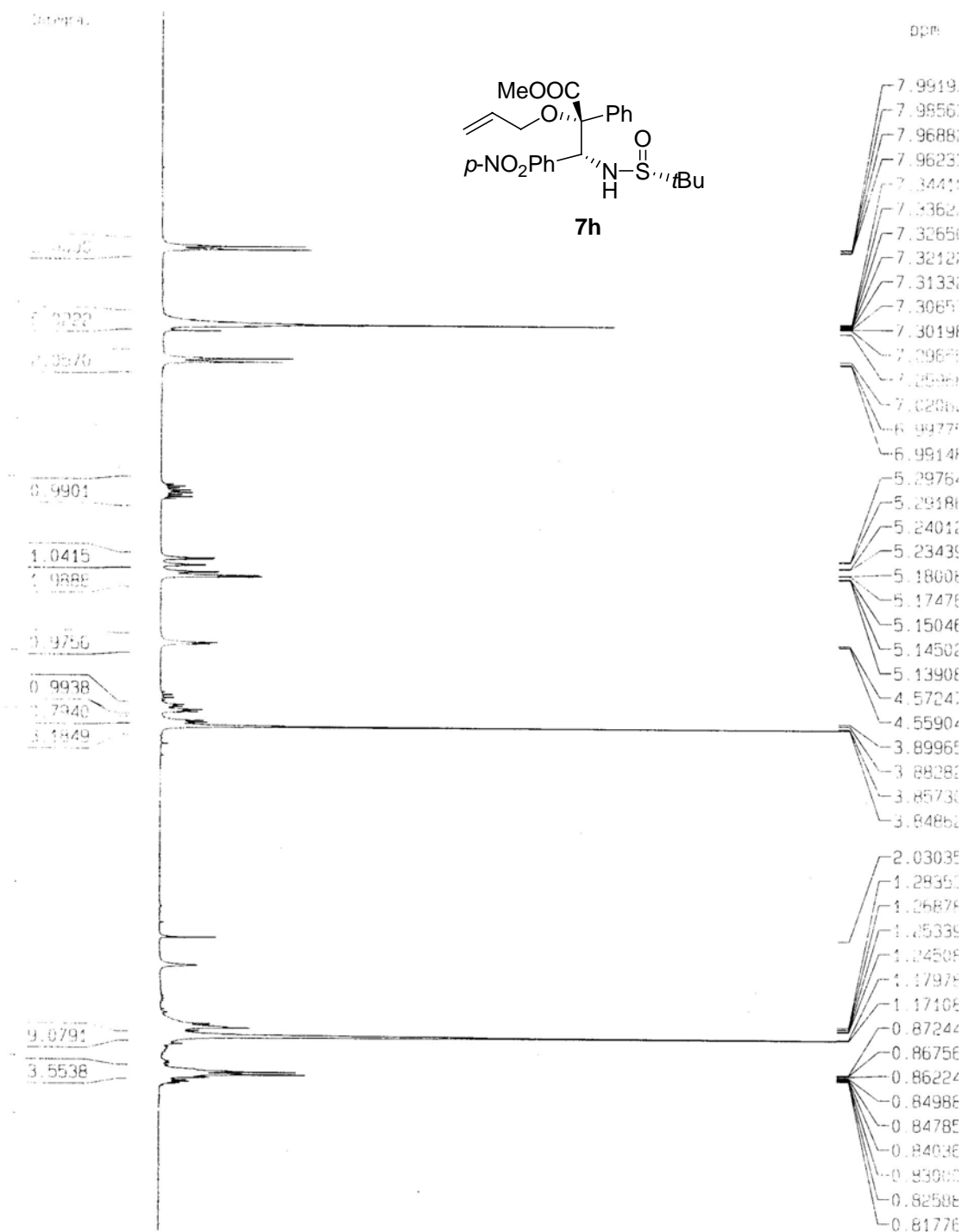
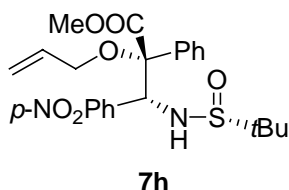
***** CHANNEL f1 *****
NUC1          13C
P1           5.50
PL1          -6.00
SFO1         75.4775986

***** CHANNEL f2 *****
CPDPRG2      waltz16
NUC2          1H
P2           80.00
PL2          -2.00
PL12         17.10
PL13         17.10
SFO2         300.1312005

F2 - Processing parameters
SI           32768
SF           75.4677532
AQ           1.4451166
RG           8152
CM           22.050
DE           5.00
TE           300.0
D1           2.00000000
d11          0.03000000
d12          0.00020000

1D 13C NMR Plot Parameters
SI           32768
SF           75.4677532
AQ           1.4451166
RG           8152
CM           22.050
DE           5.00
TE           300.0

```



Current Data Parameters

NAME 139d-nhx-319

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20050701

Time 11.35

INSTRUM av300

PROBHD 5 mm QNP 1H/13

PULPROG zg30

ID 32768

SOLVENT CDCl3

NS 8

DS 0

SWH 5995.204 Hz

FIDRES 0.182959 Hz

AQ 2.7329011 sec

RG 1024

DM 83.400 us

DE 6.00 us

TE 300.0 K

D1 1.00000000 sec

***** CHANNEL f1 *****

NUC1 1H

Q1 3.00 us

PL1 -2.00 dB

SFO1 300.1320882 MHz

F2 - Processing parameters

SF 300.1300124 MHz

WDW EM

SSB 0

LB 0.10 Hz

GB 0

PC 1.00

ID NMR plot parameters

CX 20.00 cm

CA 25.00 cm

FLP 10.000 dB

F1 3001.30 Hz

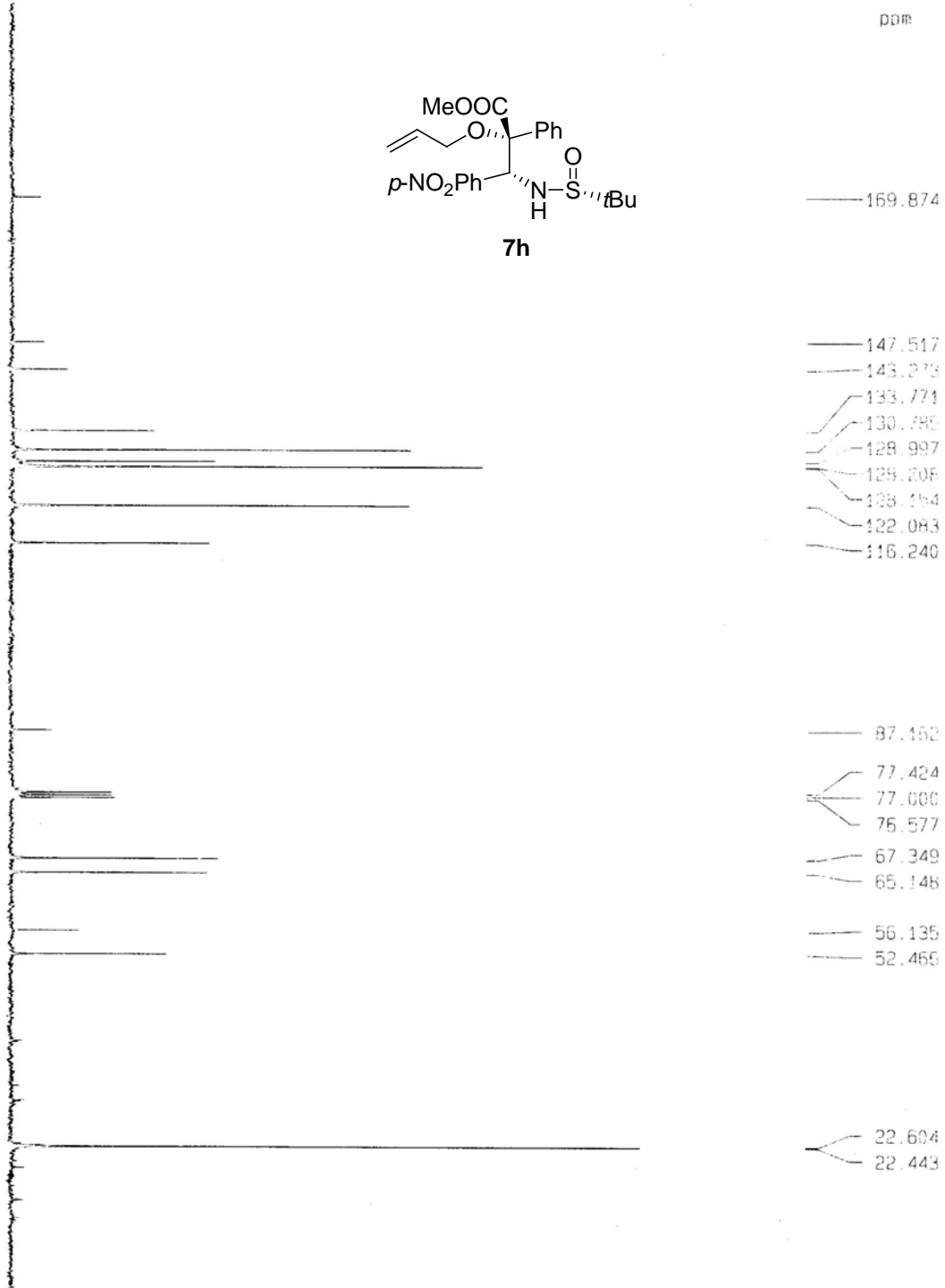
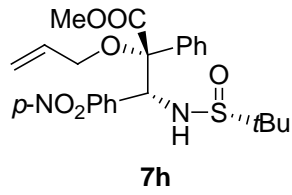
F2P -0.500 dB

NUC 15N

NUC2 13C

NUC3 56825 Hz

DDM



```

Current Data Parameters
NAME      1399-rh-319
EXPNO    2
PROCNO   1

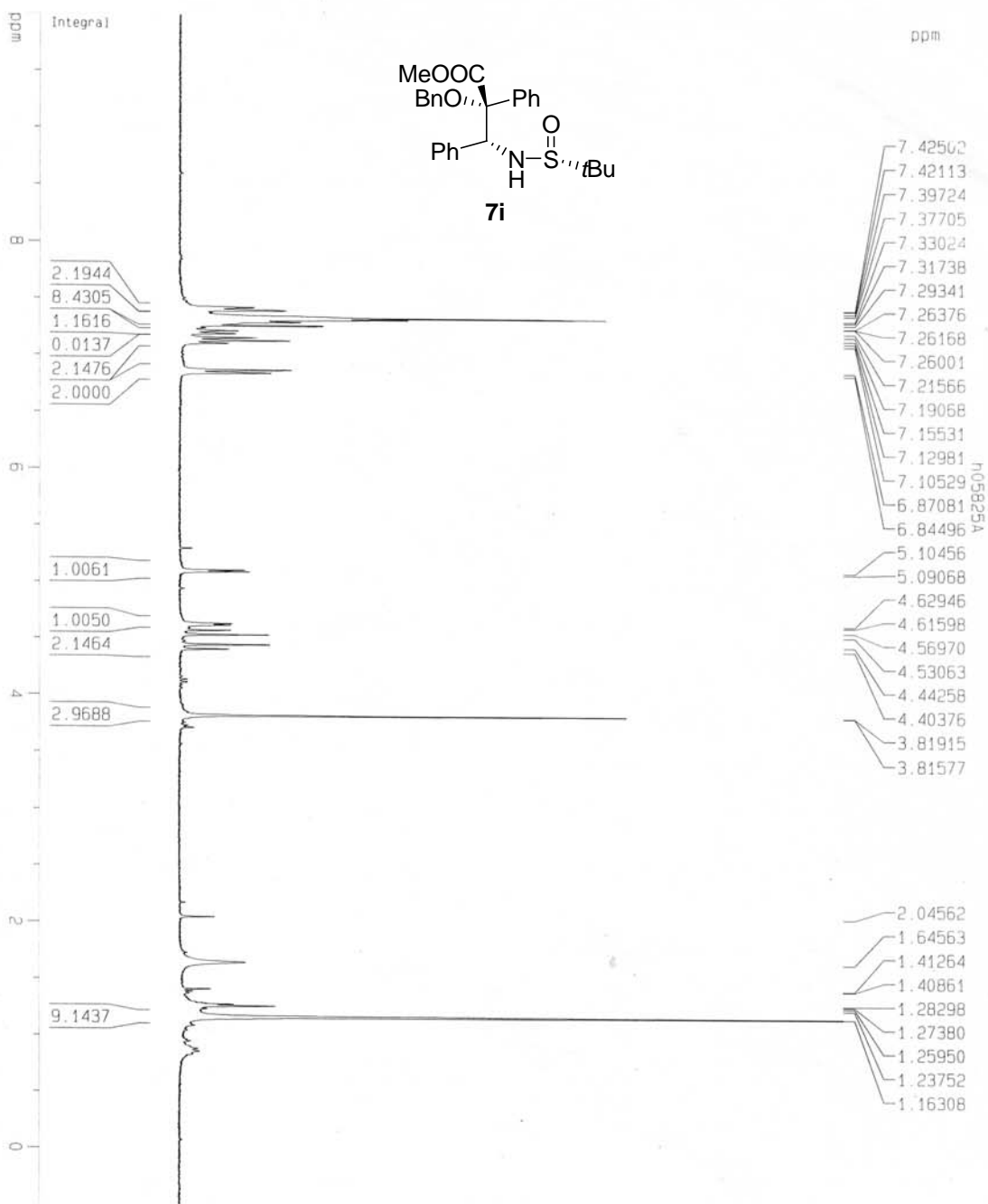
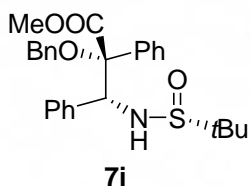
F2 - Acquisition Parameters
Date_    20050701
Time     14.41
INSTRUM  AV300
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD       32768
FIDRES   0.345004 Hz
AQ       1.4451188 sec
RG       8192
DM       22.050 usec
DE       6.00 usec
TE       300.0 K
D1       2.00000000 sec
d11      0.03000000 sec
d12      0.00022000 sec

***** CHANNEL f1 *****
NUC1     13C
P1       5.50 usec
PL1      -6.00 dB
SFO1     75.475598 MHz

***** CHANNEL f2 *****
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PL2      -2.00 dB
PL12     17.70 dB
PL13     17.71 dB
SFO2     300.1312005 MHz

F2 - Processing parameters
SI       32768
SF       75.4817642 MHz
WDW      EM
SSB      0
GB       0
AR       3.30 Hz
SR       0
CR       0
CF       1.46

F2 - 13C NMR Parameters
SI       32768
SF       75.4817642 MHz
WDW      EM
SSB      0
GB       0
AR       3.30 Hz
SR       0
CR       0
CF       1.46
  
```



Current Data Parameters
 NAME 1399-nhx-347
 EXPNO 1
 PROCNO 1

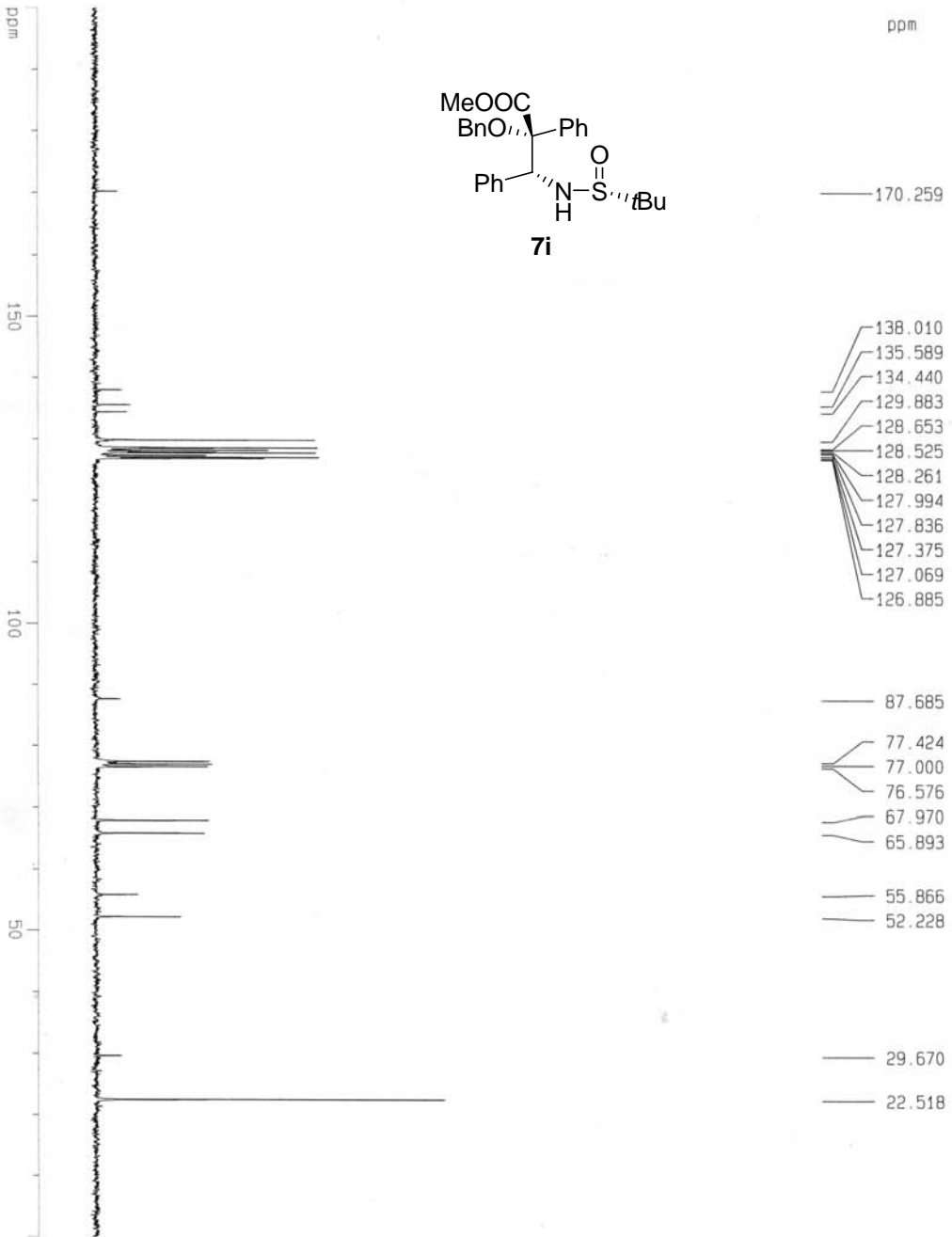
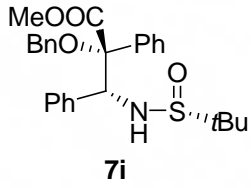
F2 - Acquisition Parameters
 Date_ 20050826
 Time 15.53
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 256
 DW 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SF01 300.1320882 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300122 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 20.00 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPKMCH 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm

h06804A



- 170.259
- 138.010
- 135.589
- 134.440
- 129.883
- 128.653
- 128.525
- 128.261
- 127.994
- 127.836
- 127.375
- 127.069
- 126.885
- 87.685
- 77.424
- 77.000
- 76.576
- 67.970
- 65.893
- 55.866
- 52.228
- 29.670
- 22.518

Current Data Parameters
 NAME 1399-rhx-831
 EXPNO 2
 PROCNO 1

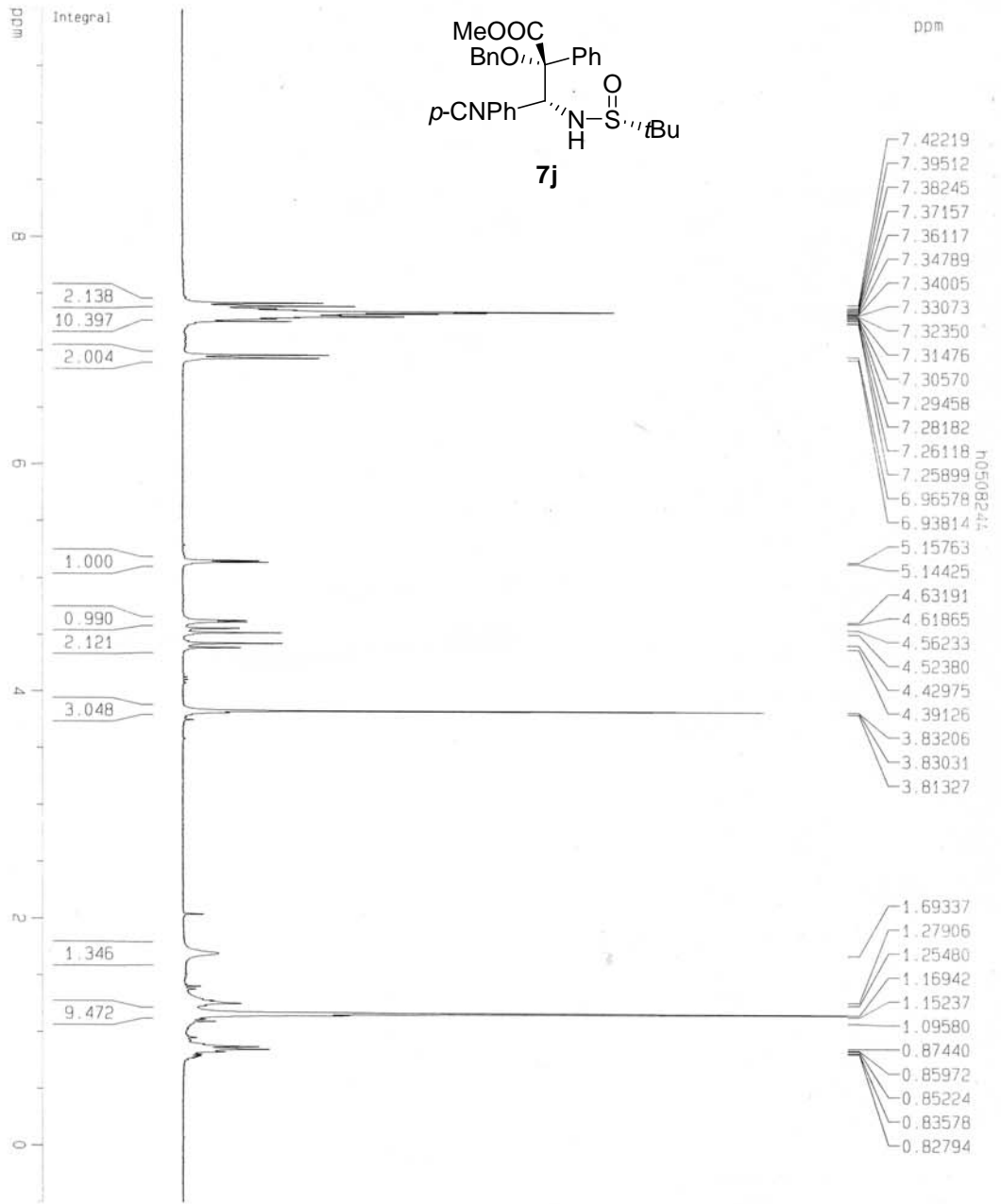
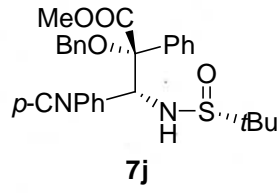
F2 - Acquisition Parameters
 Date_ 20060804
 Time 17.25
 INSTNM av300
 PROGNO 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 336
 DS 4
 SMH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451188 sec
 RG 8192
 DM 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SF01 75.4775598 MHz

***** CHANNEL f2 *****
 CPDPRG2 Waltz16
 NUC2 1H
 PQR2 80.00 usec
 PL2 -2.00 dB
 PLL2 17.70 dB
 PLL3 17.71 dB
 SF02 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677598 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

10 NMR plot parameters
 CX 20.00 cm
 CY 6.00 cm
 F1p 200.005 ppm
 F1 15093.93 Hz
 F2p -0.005 ppm
 F2 -0.38 Hz
 pPNUC 10.00050 ppm/cm
 rZCM 754.71527 Hz/cm



Current Data Parameters

NAME 1399-nhx-345

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20050825

Time 11:56

INSTRUM av300

PROBHD 5 mm QNP 1H/13

PULPROG zg30

TD 32768

SOLVENT CDCl3

NS 16

DS 0

SWH 5995.204 Hz

FIDRES 0.182959 Hz

AQ 2.7329011 sec

RG 1024

DM 83.400 usec

DE 6.00 usec

TE 300.0 K

D1 1.000000000 sec

***** CHANNEL f1 *****

NUC1 1H

pl 3.00 usec

PL1 -2.00 dB

SFO1 300.1320882 MHz

F2 - Processing parameters

SI 32768

SF 300.1300124 MHz

WDW EM

SSB 0

LB 0.10 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 20.00 cm

CY 25.00 cm

F1P 10.000 ppm

F1 3001.30 Hz

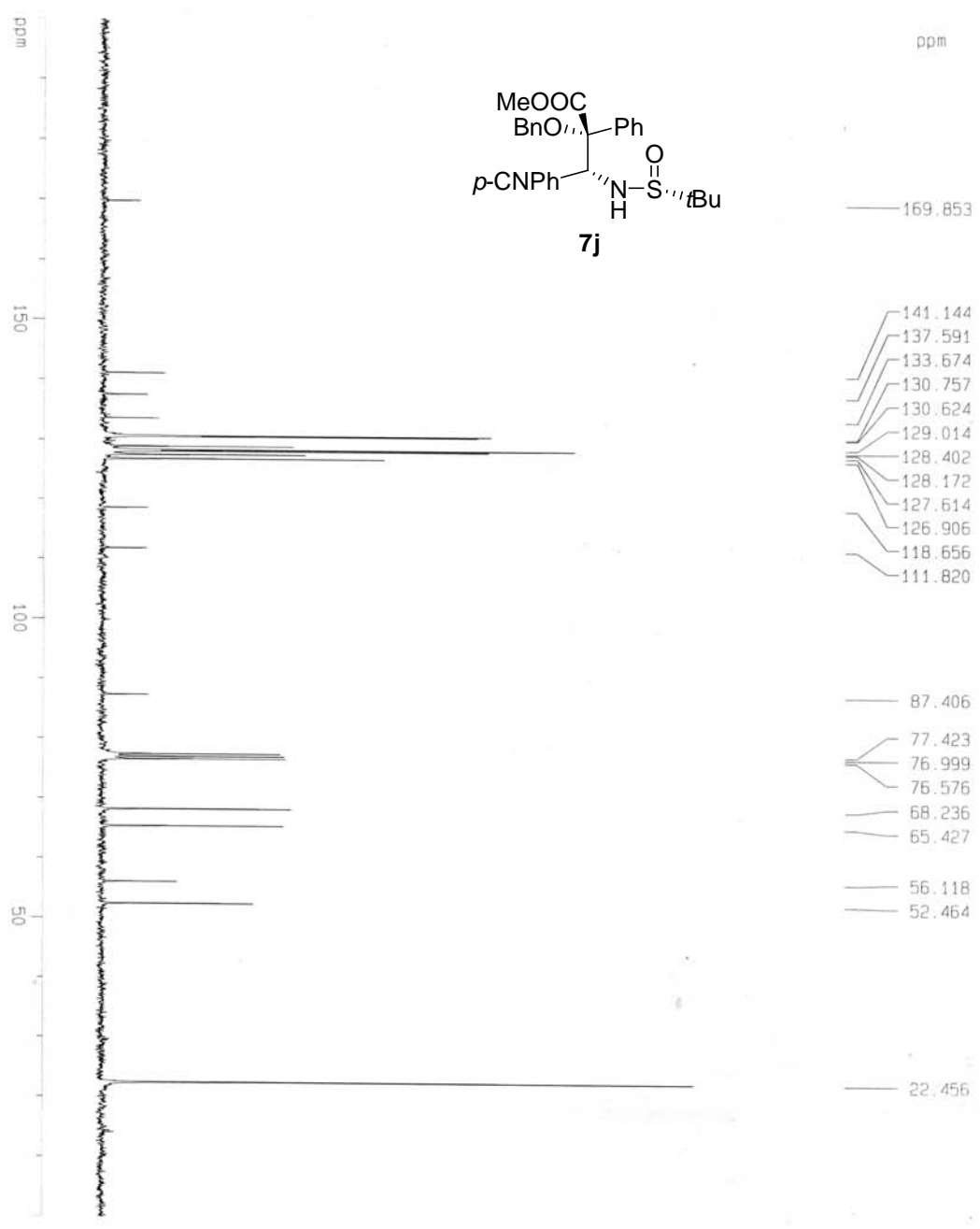
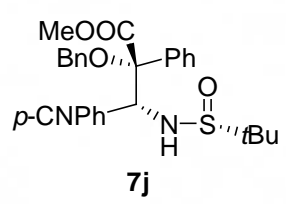
F2P -0.500 ppm

F2 -150.06 Hz

PPMCM 0.52500 ppm/cm

HZCM 157.56829 Hz/cm

h050824A



- 169.853
- 141.144
- 137.591
- 133.674
- 130.757
- 130.624
- 129.014
- 128.402
- 128.172
- 127.614
- 126.906
- 118.656
- 111.820
- 87.406
- 77.423
- 76.999
- 76.576
- 68.236
- 65.427
- 56.118
- 52.464
- 22.456

Current Data Parameters
 NAME 1399-nhx-345
 EXPNO 2
 PROCNO 1

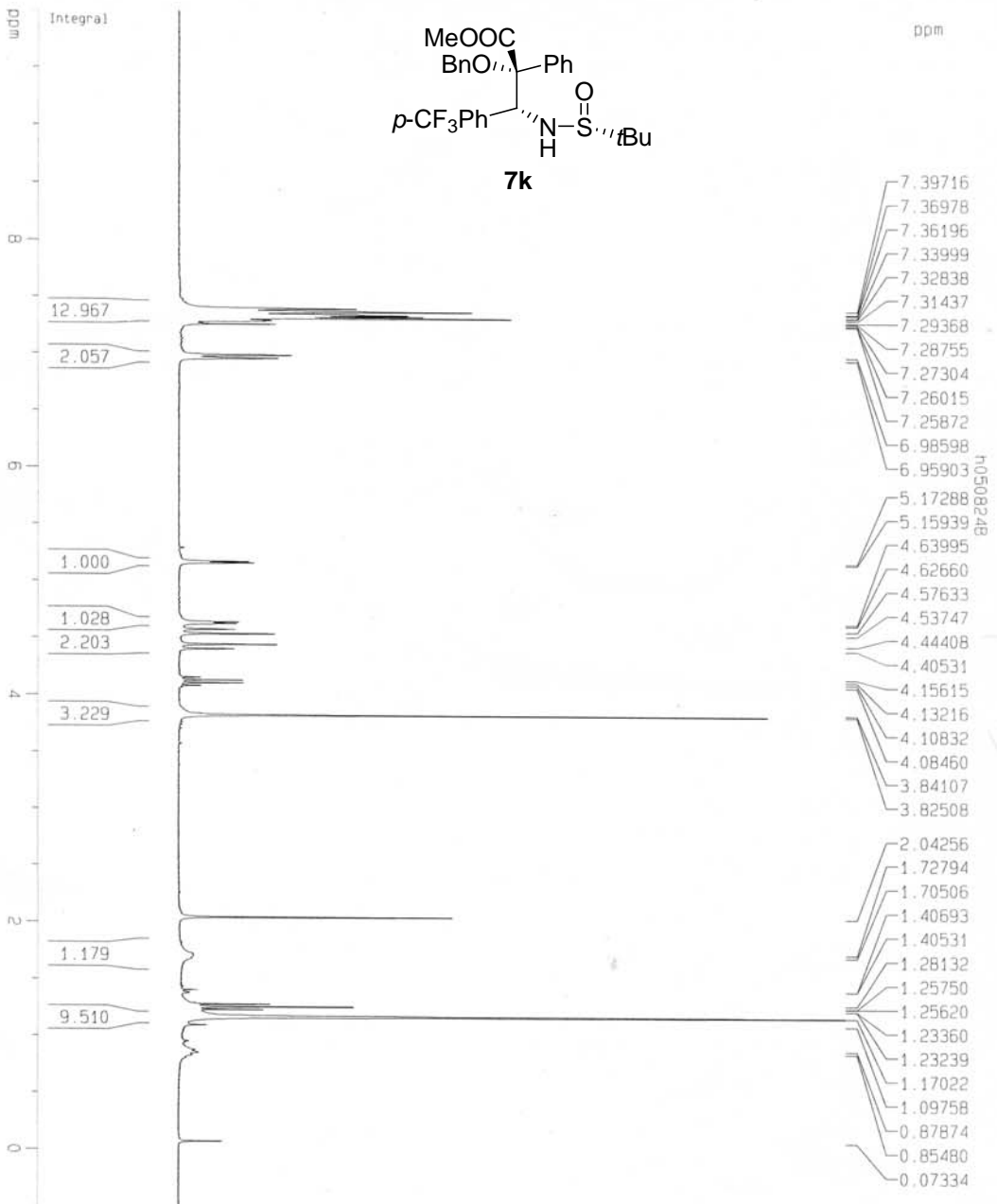
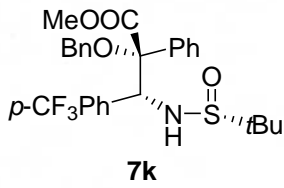
F2 - Acquisition Parameters
 Date_ 20050926
 Time 11:28
 INSTRUM av300
 PROCNO 5
 PULPROG zgpg30
 TD 2999
 ID 65536
 SOLVENT CDCl3
 NS 860
 DS 4
 SWH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451188 sec
 RG 8192
 DM 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SFO1 75.4775598 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677534 MHz
 MDW EM
 SSB 0
 AB 3.00 Hz
 GB 0
 PC 1.40

10 NMR Plot Parameters
 CX 20.00 cm
 CY 10.00 cm
 F1P 200.005 ppm
 F1 15093.93 Hz
 F2P -0.005 ppm
 F2 -0.38 Hz
 PRGCM 10.00050 ppm/cm
 FZCM 754.71927 Hz/cm



h050824B
p-cf3

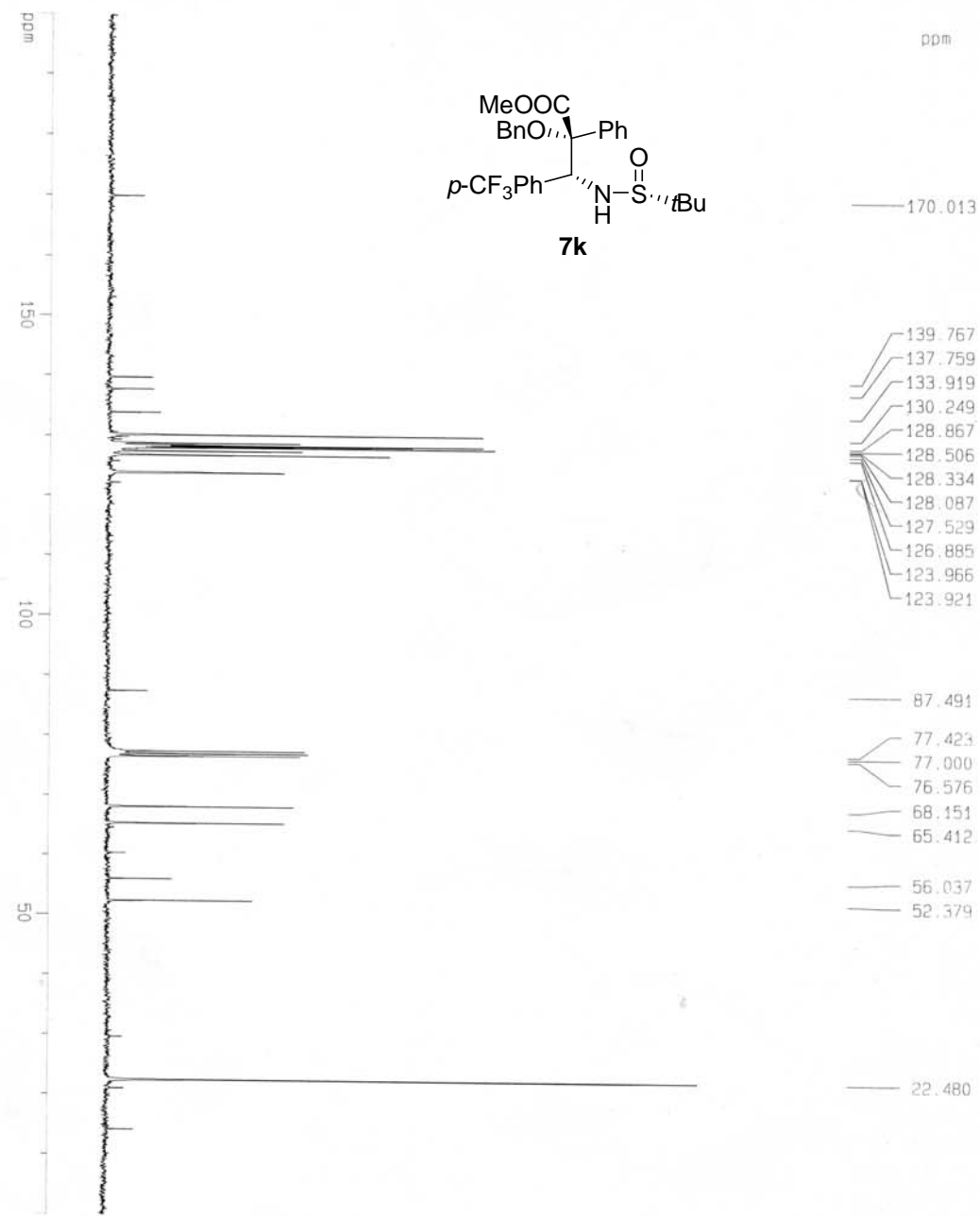
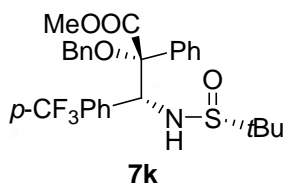
Current Data Parameters
 NAME 1399-nhx-346
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050825
 Time 12.02
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 1024
 DW 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SFO1 300.1320882 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300124 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

10 NMR plot parameters
 CX 20.00 cm
 CY 25.00 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPMCKM 0.52500 ppm/cm
 HZCM 157.35625 Hz/cm



10508248

- 170.013
- 139.767
- 137.759
- 133.919
- 130.249
- 128.867
- 128.506
- 128.334
- 128.087
- 127.529
- 126.885
- 123.966
- 123.921
- 87.491
- 77.423
- 77.000
- 76.576
- 68.151
- 65.412
- 56.037
- 52.379
- 22.480

Current Data Parameters
 NAME 1399-hny-346
 EXPNO 2
 PROCNO 1

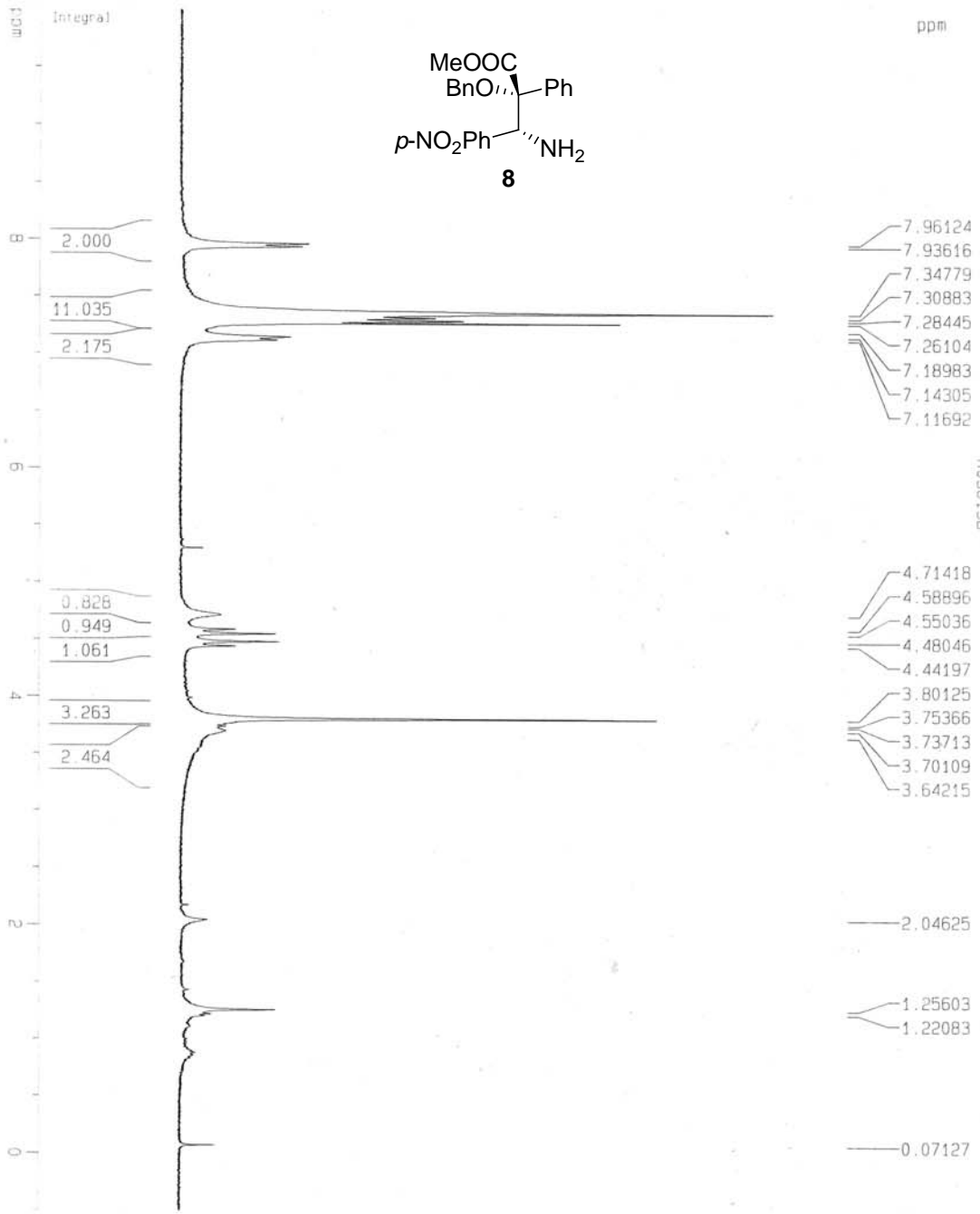
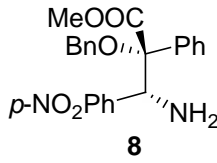
F2 - Acquisition Parameters
 Date_ 20050829
 Time 13:41
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1412
 DS 4
 SMH 22875.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451188 sec
 RG 8192
 DE 22.050 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SFO1 75.4775598 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677529 MHz
 KDM EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

10 NMR Plot parameters
 CX 20.00 cm
 CV 10.00 cm
 FIP 200.005 ppm
 F1 15093.93 Hz
 F2P -0.005 ppm
 F2 -0.38 Hz
 PPMQX 10.00050 ppm/cm
 HZCM 754.71527 Hz/cm



h056138

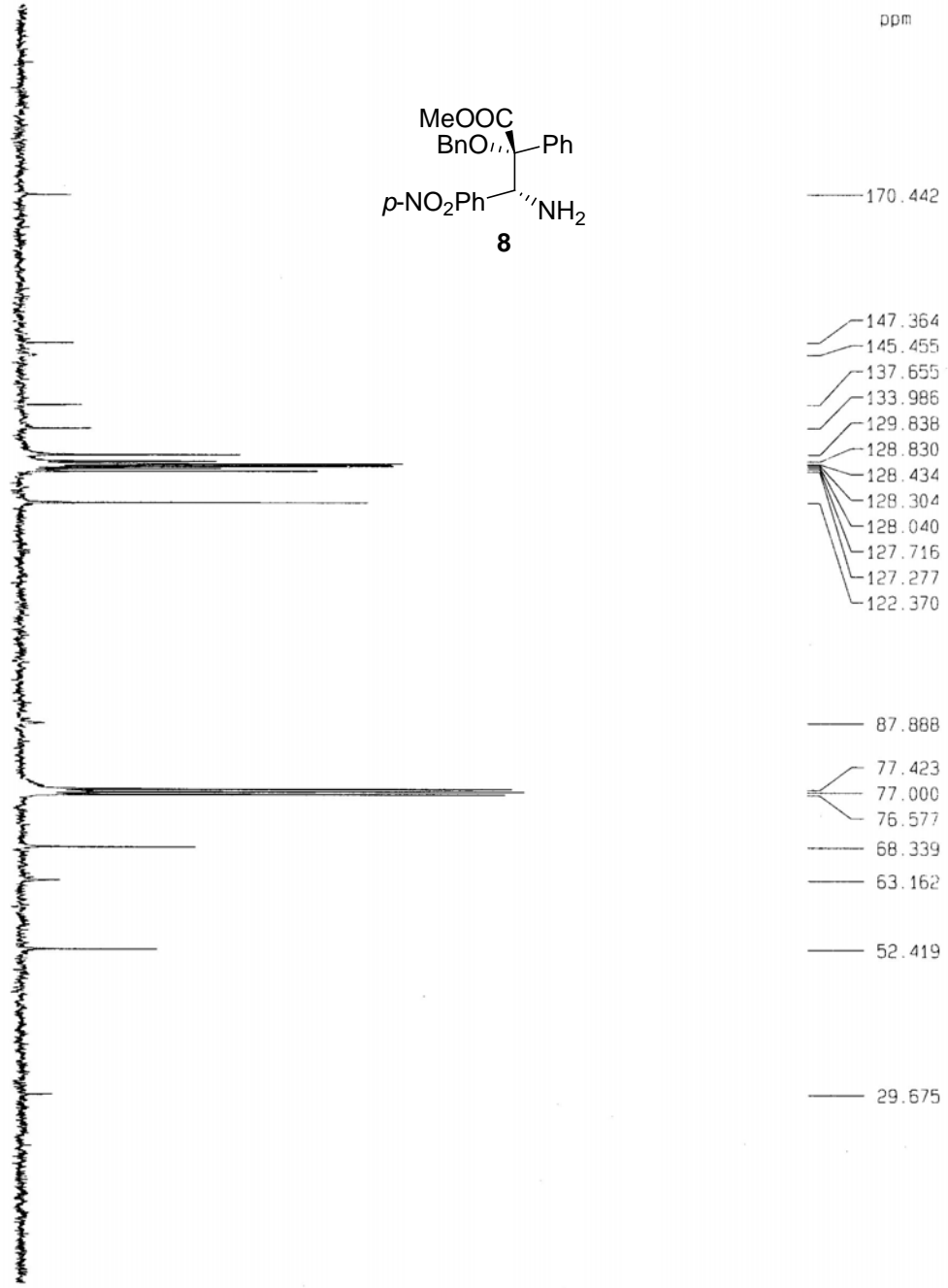
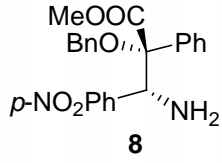
Current Data Parameters
 NAME 1399-hhx-298
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050617
 Time 16.52
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 512
 DW 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SF01 300.1320882 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300122 MHz
 MDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 SFO1 0.52500 ppm/cm
 SFO2 157.56825 Hz/cm



ppm

170.442

147.364

145.455

137.655

133.986

129.838

128.830

128.434

128.304

128.040

127.716

127.277

122.370

87.868

77.423

77.000

76.577

68.339

63.162

52.419

29.675

Current Data Parameters
 NAME 1399-nix-298
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050621
 Time 12.18
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg
 ID 55536
 SOLVENT CDCl3
 NS 2145

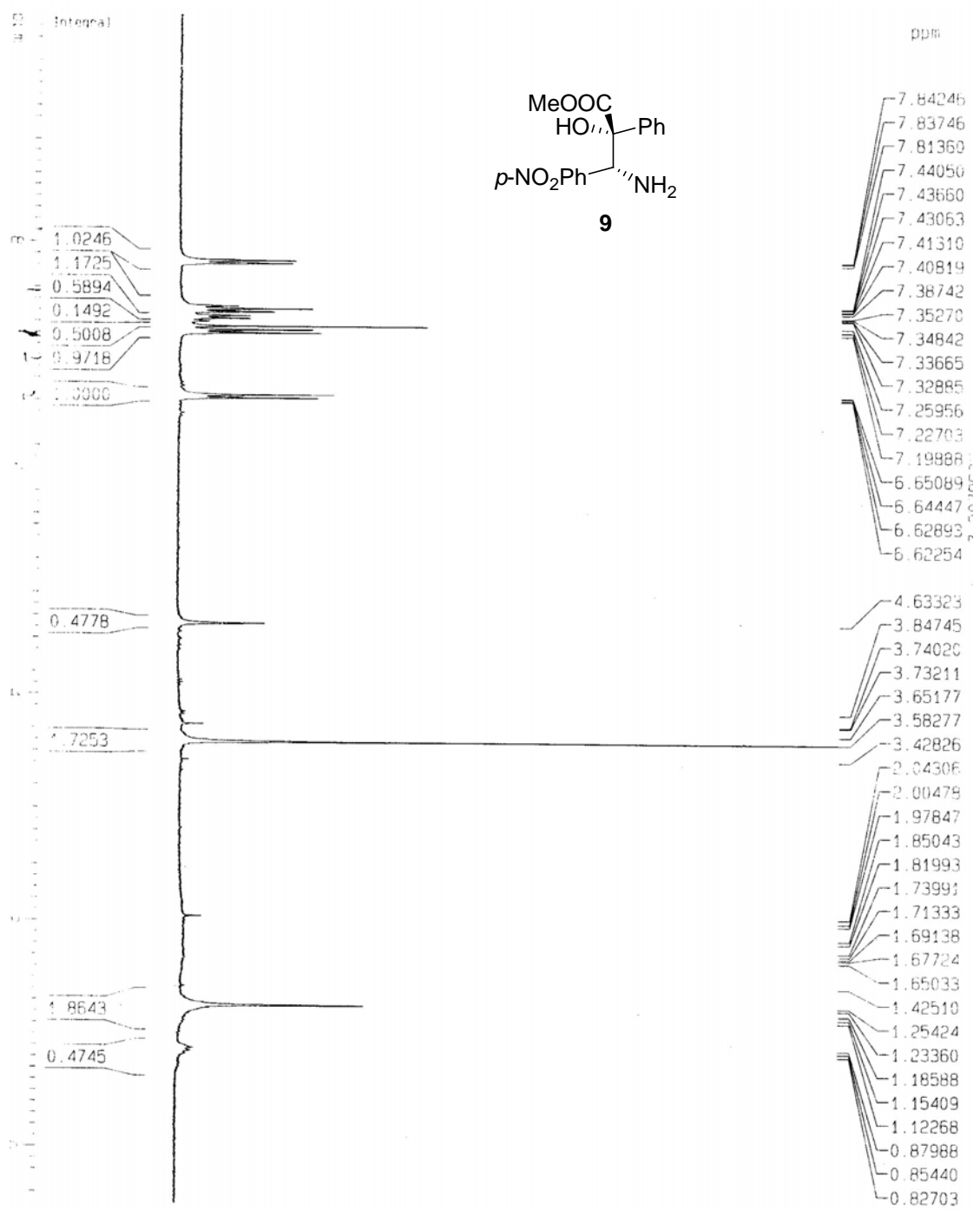
DS 4
 SMH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.445188 sec
 RG 8192
 DM 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 O1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00020000 sec

CHANNEL f1
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SF01 75.477598 MHz

CHANNEL f2
 CPDPRG2 waltz16
 NUC2 1H
 PCPRG2
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SF02 300.1312005 MHz

F2 - Processing parameters
 S1 32768
 SF 75.4677524 MHz
 MDW EN
 SSB 0
 BI 3.00 MHz
 GB 0
 PC 1.45

F0 NMR plot parameters
 CX 20.00 cm
 CY 5.00 cm
 F1P 200.005 ppm



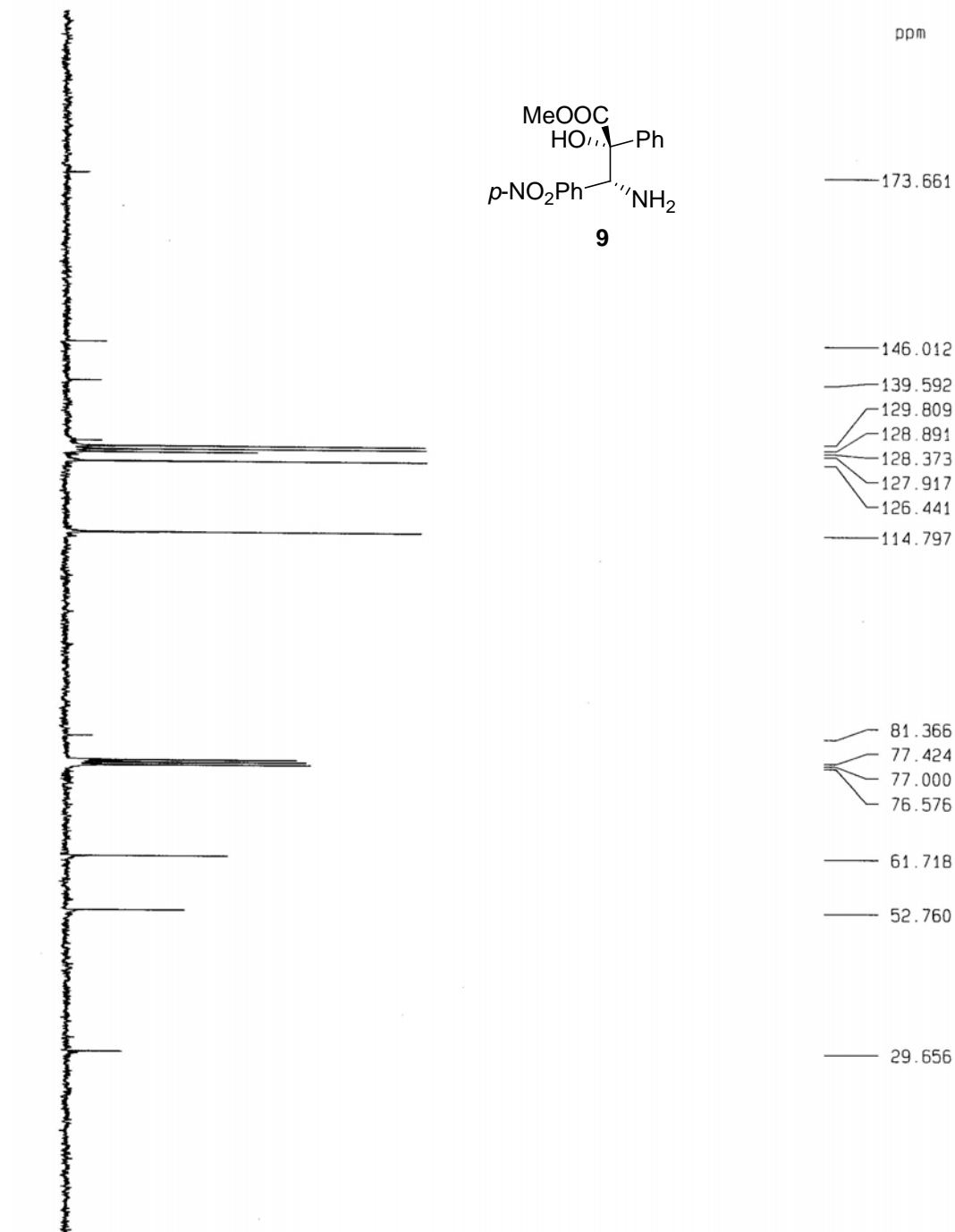
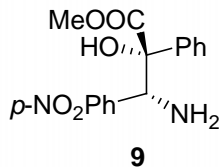
Current Data Parameters
 NAME 1395-nfx-309
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050628
 Time 14.46
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 256
 CW 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SF01 300.1320882 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300124 MHz
 WDW EM
 SSB 0
 -B 0.10 Hz
 -C 0
 -PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.00 cm
 ZP 10.000 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 150.06 Hz
 SFO1 51503 ppm/cm
 SFO2 1518925 Hz/cm



- 173.661
- 146.012
- 139.592
- 129.809
- 128.891
- 128.373
- 127.917
- 126.441
- 114.797
- 81.366
- 77.424
- 77.000
- 76.576
- 61.718
- 52.760
- 29.656

```

Current Data Parameters
NAME      1399-hnx-855
EXPNO    2
PROCNO   1

F2 - Acquisition Parameters
Date_    20060929
Time     16:27
INSTRUM  av300
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg
TD        65536
SOLVENT  DMSO
NS        766
DS        4
SMH       22675.736 HZ
FIDRES   0.346004 HZ
AQ        1.4451188 sec
RG         8192
DM         22.050 usec
DE         5.00 usec
TE        300.0 K
D1        2.00000000 sec
d11       0.03000000 sec
d12       0.00002000 sec

***** CHANNEL f1 *****
NUC1      13C
P1        5.50 usec
PL1       -6.00 dB
SFO1      75.4775598 MHz

***** CHANNEL f2 *****
CPOPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       -2.00 dB
PL12      17.70 dB
PL13      17.71 dB
SFO2      300.1312005 MHz

F2 - Processing parameters
SI         32768
SF         75.4677534 MHz
WDW        EM
SSB        0
LB         3.00 HZ
GB         0
PC         1.40

1D NMR plot parameters
CX         20.00 cm
CY         6.00 cm
CZ         200.005 ppm
F1         15093.93 HZ
F2P       -0.500 ppm
  
```