Angewandte Chemie
Eine Zeitschrift der Gesellschaft Deutscher Chemiker

Supporting Information

69451 Weinheim, Germany

Highly Enantioselective Direct Michael Addition of Nitroalkanes to Nitroolefins Catalyzed by La(OTf)$_3$/*N*,*N*′-Dioxide Complexes

Xu Yang,[a] *Xin Zhou,*[a] *Lili Lin,*[a] *Lu Chang,*[a] *Xiaohua Liu,*[a] *Xiaoming Feng* [a,b,]*

[a] Key Laboratory of Green Chemistry & Technology, Ministry of Education, College of Chemistry, Sichuan University, Chengdu 610064, China
[b] State Key Laboratory of Biotherapy, Sichuan University, Chengdu 610041, China

xmfeng@scu.edu.cn

Supporting Information

Contents

1. General……S1
2. Characterization of *N*,*N*′-Dioxide Ligands……S2
3. Typical Procedure for the synthesis of 1,3-dinitroalkanes……S2
4. Characterization of the Conjugate Addition Products……S2
5. References……S13
6. Copy of NMR Spectra for *N*,*N*′-Dioxide Ligand……S14
7. Copy of NMR Spectra for the products……S20

1. General

^{1}H NMR spectra were recorded on commercial instruments (300 or 400 MHz). Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$, δ = 7.26). Spectra are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration, and assignment. ^{13}C NMR spectra were collected on commericial instruments (100 or 75 MHz) with complete proton decoupling. Chemical shifts are reported in ppm from the tetramethylsilane with the solvent resonance as internal standard ($CDCl_3$, δ = 77.0). The enantiomeric excesses were determined by HPLC analysis on chiral DAICEL CHIRALCEL AS-H, OD-H or CHIRALPAK AD-H column at 210 nm. Optical rotations are measured on a commerical polarimeter and are reported as follows: $[\alpha]_D^T$ (c = g/100 mL, solvent).

Solvents were dried according to standard procedures. Nitroalkanes were obtained from commercial sources and used without further purification. Nitroalkenes were prepared according to the literature procedures.[1] The *N*,*N*′-dioxide ligands were prepared according to the methods reported in the literature.[2] Racemic samples of **3a-3n** were prepared with 10 mol% Et_3N as the catalyst and the nitroalkane as solvent.

2. Characterization of *N,N'*-Dioxide Ligands

For the characterization of the ligands **L1**,[2b] **L4**,[2b] **L6**,[2c,2d] **L7**,[2b] and **L8**,[2e] see literature.

L2: white solid; m.p. 176-179 ^{o}C; $[\alpha]^{25}_{D}$ = -110.0 (c = 0.196, in $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 13.12 (s, 2H), 7.50-7.52 (d, *J* = 8.0 Hz, 4H), 7.27-7.31 (m, 4H), 7.08-7.12 (t, *J* = 7.6 Hz, 2H), 4.26-4.31 (m, 2H), 3.75-3.88 (m, 4H), 3.57-3.62 (m, 2H), 3.37-3.45 (dd, *J* = 20.0, 9.6 Hz, 2H), 2.38-2.56 (m, 6H), 2.02-2.07 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 164.8, 137.4, 128.9, 124.4, 120.2, 76.1, 70.1, 61.0, 27.6, 20.4 ppm; ESI-HRMS: calcd for $C_{25}H_{33}N_4O_4$ [M+H$^+$] 439.2340, found 439.2345.

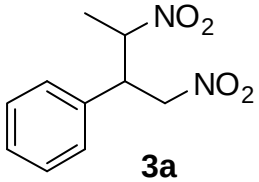
L3: white solid; m.p. 166-170 ^{o}C; $[\alpha]^{25}_{D}$ = -89.4 (c = 0.174, in $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 13.60 (s, 2H), 7.59-7.61 (d, *J* = 7.6 Hz, 4H), 7.29-7.33 (t, *J* = 7.6 Hz, 4H), 7.07-7.10 (t, *J* = 7.6 Hz, 2H), 3.60-3.65 (dd, *J* = 10.8, 8.4 Hz, 2H), 3.49-3.54 (t, *J* = 8.8 Hz, 2H), 3.33-3.41 (m, 2H), 3.16-3.25 (m, 4H), 2.39-2.59 (m, 6H), 2.11-2.20 (m, 2H), 1.97-2.06 (m, 2H), 1.78-1.89 (m, 2H), 1.36-1.44 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 165.8, 138.1, 129.1, 124.2, 120.3, 75.9, 68.7, 67.1, 27.9, 24.3, 23.9, 20.5 ppm; ESI-HRMS: calcd for $C_{25}H_{33}N_4O_4$ [M+H$^+$] 481.2809, found 481.2816.

L5: white powder; m.p. 95-99 ^{o}C; $[\alpha]^{25}_{D}$ = -125.0 (c = 0.136, in $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 13.49 (s, 2H), 7.49-7.51 (d, *J* = 8.8 Hz, 4H), 7.22-7.24 (d, *J* = 8.8 Hz, 4H), 3.61-3.71 (m, 4H), 3.45-3.59 (m, 4H), 3.29-3.36 (dd, *J* = 9.6, 20.0 Hz, 2H), 2.68-2.76 (m, 2H), 2.49-2.57 (m, 4H), 2.40-2.47 (m, 2H), 2.04-2.12 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 165.3, 136.4, 129.2, 129.1, 121.4, 76.7, 68.2, 65.1, 27.7, 20.5, 20.0 ppm; ESI-HRMS: calcd for $C_{25}H_{33}N_4O_4$ [M+H$^+$] 521.1717, found 521.1721.

3. Typical Procedure for the Synthesis of 1,3-Dinitroalkanes

The mixture of ligand **L1** (10.9 mg, 0.024 mmol), $La(OTf)_3$ (11.7 mg, 0.02 mmol) was stirred in CH_2Cl_2 (0.4 mL) at 30 ^{o}C under N_2 atmosphere for 30 min to form the complex catalyst. Then nitroethane (29 μL, 0.4 mmol), imidazole (20 μL, c = 1 mol/L in CH_2Cl_2, 10 mol%) and the β-nitrostyrene (0.2 mmol) in CH_2Cl_2 (0.6 mL) were sequentially added to the mixture. After stirring at 30 ^{o}C for 2.5 d, the reaction mixtures were directly purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1:4 to 1:9) to afford the *syn/anti* mixtures of chiral 1,3-dinitro compounds. After the mixtures of products were analyzed by HPLC analysis, the pure *syn* diastereoisomers were afforded through another column chromatography separation on silica gel (ethyl acetate/petroleum ether = 1:4 to 1:9) and then the pure *syn* products were used to ^{1}H NMR, ^{13}C NMR and the determination of optical rotation and melting points.

4. Characterization of the Conjugate Addition Products



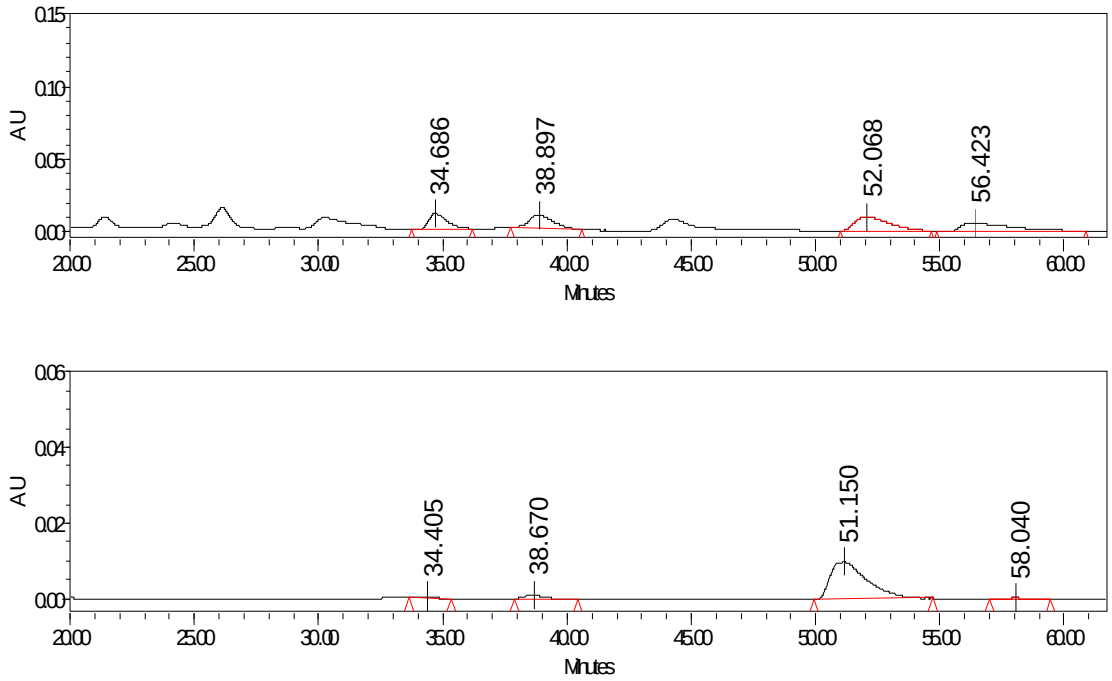
3a

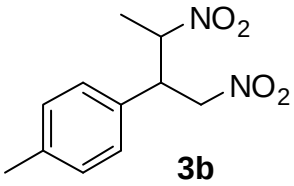
2-Phenyl-1,3-dinitrobutane (3a).

The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (96% *ee*) of the *syn* diastereoisomer and diastereoselectivity (93:7 d.r.) of the reaction were determined by HPLC analysis using a chiral AS-H column (hexane/2-propanol

90:10, 0.5 mL/min, UV 210 nm, t_r (*syn*. major) = 51.150 min, t_r (*syn*. minor) = 58.040 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:5, R_f 0.4) to afford a white solid. m.p. 80-81°C. $[\alpha]^{20}_D$ (*syn*) = +7.3 (c = 0.11, in CH_2Cl_2). ^1H NMR (400 MHz, $CDCl_3$): δ 7.38-7.33 (m, 3H), 7.17-7.15 (m, 2H), 5.00-4.92 (m, 2H), 4.83 (dd, *J* = 13.6, 8.4 Hz, 1H), 4.05-4.00 (m, 1H), 1.59 (d, *J* = 6.8 Hz, 3H).

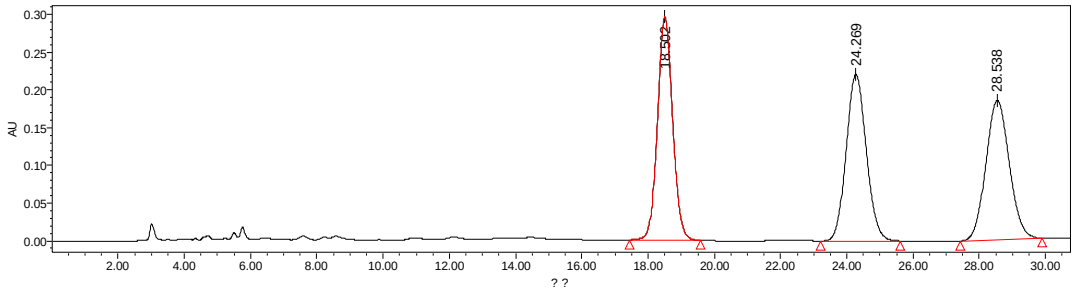


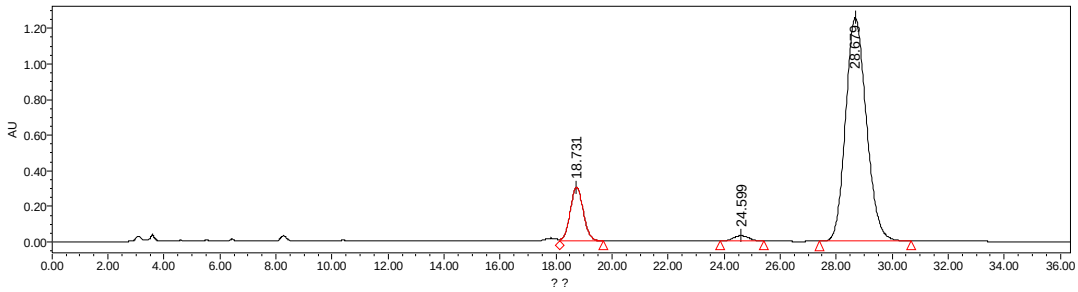


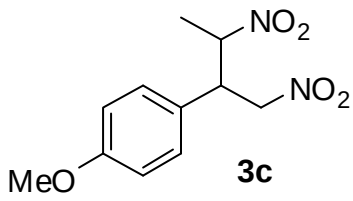
3b

2-(4-Methylphenyl)-1,3-dinitrobutane (3b).
The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (96% *ee*) of the *syn* diastereoisomer and diastereoselectivity (88:12 d.r.) of the reaction were determined by HPLC analysis using a chiral OD-H column (hexane/2-propanol 90:10, 1.0 mL/min, UV 210 nm, t_r (*syn*. minor) = 24.599 min, t_r (*syn*. major) = 28.679 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:6, R_f 0.3) to afford a white solid. m.p. 52-53°C. $[\alpha]^{20}_D$ (*syn*) = +14.7 (c = 0.15, in CH_2Cl_2). ^1H NMR (400 MHz, $CDCl_3$): δ 7.15 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 8.0 Hz, 2H), 4.97-4.88 (m, 2H), 4.85 (dd, *J* = 13.6, 8.4 Hz, 1H), 4.00-3.95 (m, 1H), 2.32 (s, 3H), 1.57 (d, *J* = 6.8 Hz, 3H).



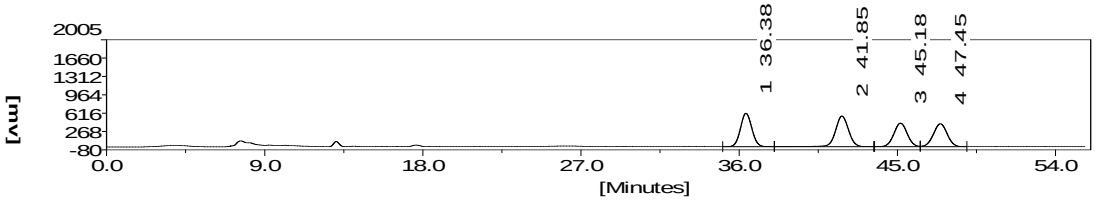


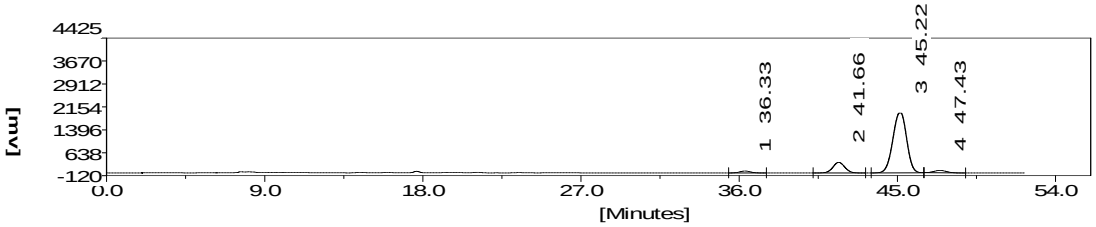


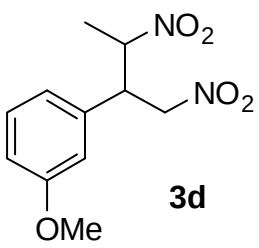
3c

2-(4-Methoxyphenyl)-1,3-dinitrobutane (3c). The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (93% *ee*) of the *syn* diastereoisomer and diastereoselectivity (86:14 d.r.) of the reaction were determined by HPLC analysis using a chiral AD-H column (hexane/2-propanol 90:10, 0.4 mL/min, UV 210 nm, t_r (*syn*. major) = 45.22 min, t_r (*syn*. minor) = 47.43 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:6, R_f 0.2) to afford a white solid. m.p. 75-77°C. $[\alpha]^{20}_D$ (*syn*) = +11.7 (c = 0.06, in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.02-6.98 (m, 2H), 6.82-6.79 (m, 2H), 4.87-4.82 (m, 2H), 4.72 (dd, *J* = 13.6, 8.4 Hz, 1H), 3.91-3.86 (m, 1H), 3.72 (s, 3H), 1.51 (d, *J* = 6.8 Hz, 3H).

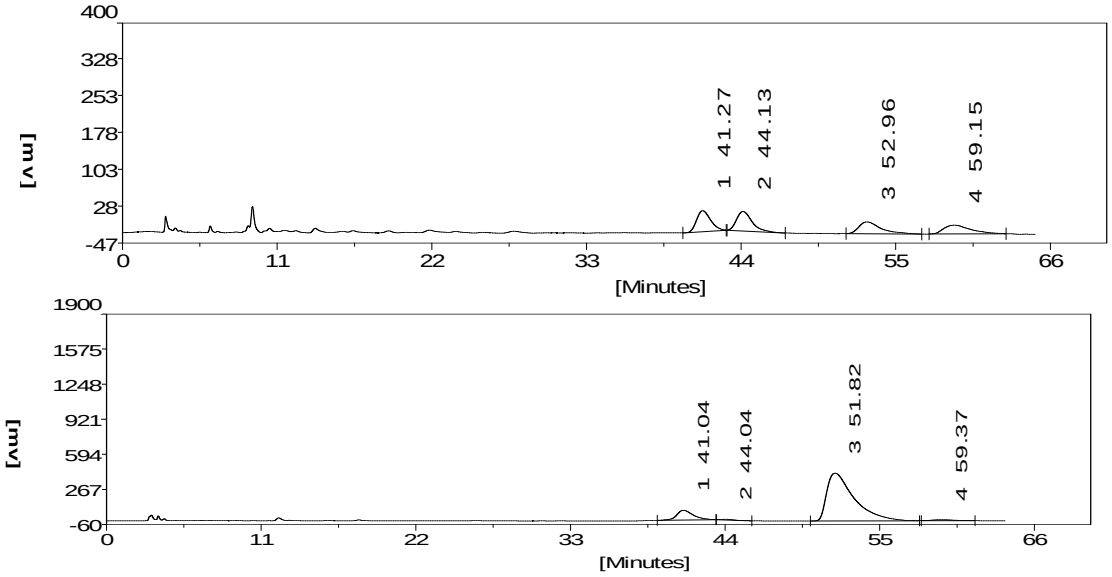


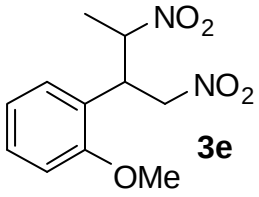




2-(3-Methoxyphenyl)-1,3-dinitrobutane (3d).
The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (97% *ee*) of the *syn* diastereoisomer and diastereoselectivity (84:16 d.r.) of the reaction were determined by HPLC analysis using a chiral AS-H column (hexane/2-propanol 90:10, 1.0 mL/min, UV 210 nm, t_r (*syn*. major) = 51.82 min, t_r (*syn*. minor) = 59.37 min).

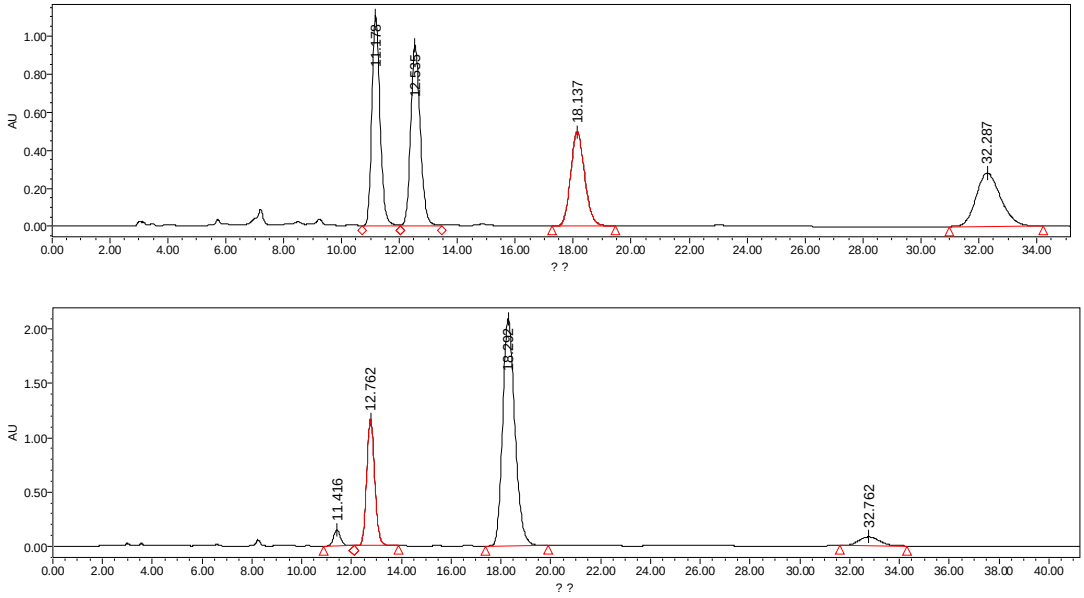
syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:6, R_f 0.2) to afford a gel product. $[\alpha]^{20}_D$ (*syn*) = +6.3 (c = 0.49, in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29-7.25 (m, 1H), 6.87 (dd, *J* = 8.0, 2.0 Hz, 1H), 6.73 (d, *J* = 7.6 Hz, 1H), 6.68 (t, *J* = 2.0 Hz, 1H), 4.98-4.89 (m, 2H), 4.81 (dd, *J* = 13.6, 8.0 Hz, 1H), 4.03-3.99 (m, 1H), 3.79 (s, 3H), 1.60 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 160.1, 135.0, 130.4, 119.9, 114.3, 114.0, 84.0, 76.0, 55.3, 47.3, 16.7. ESI-HRMS: calcd for $C_{11}H_{14}N_2O_5$ [M+Na$^+$] 277.0795, found 277.0802.

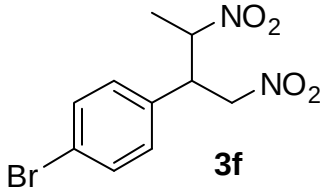




2-(2-Methoxyphenyl)-1,3-dinitrobutane (3e).
The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (90% *ee*) of the *syn* diastereoisomer and diastereoselectivity (71:29 d.r.) of the reaction were determined by HPLC analysis using a chiral OD-H column (hexane/2-propanol 90:10, 1.0 mL/min, UV 210 nm, t_r (*syn*. major) = 18.29 min, t_r (*syn*. minor) = 32.76 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:6, R_f 0.25) to afford a white solid. m.p. 59-61^{o}C. $[\alpha]^{20}_D$ (*syn*) = -10.0 (c = 0.18, in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32-7.27 (m, 1H), 7.05 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.92-6.88 (m, 2H), 5.21-5.14 (m, 1H), 4.92-4.83 (m, 2H), 4.30 (dd, *J* = 14.2, 7.6 Hz, 1H), 3.85 (s, 3H), 1.58 (d, *J* = 6.8 Hz, 3H).

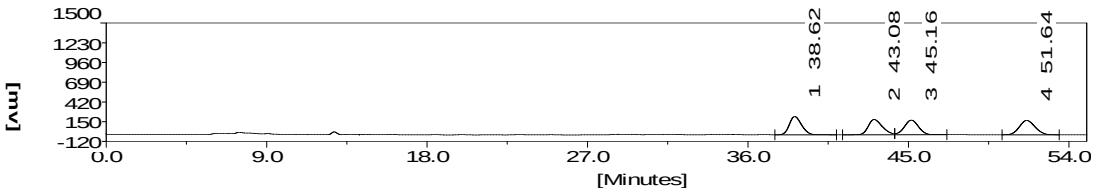


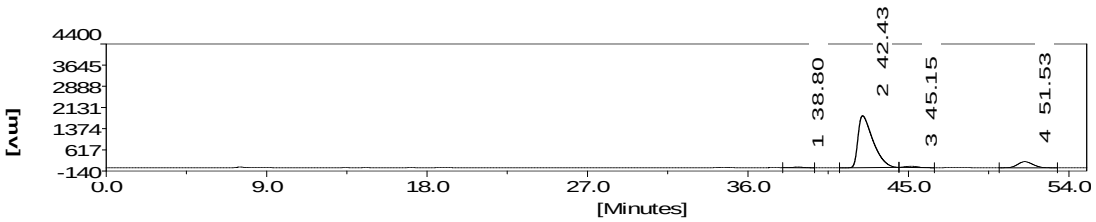


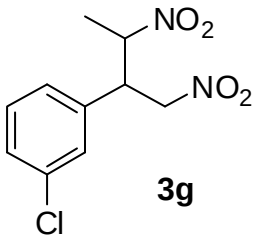
3f

2-(4-Bromophenyl)-1,3-dinitrobutane (3f).
The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (96% *ee*) of the *syn* diastereoisomer and diastereoselectivity (89:11 d.r.) of the reaction were determined by HPLC analysis using a chiral AD-H column (hexane/2-propanol 90:10, 0.4 mL/min, UV 210 nm, t_r (*syn*. major) = 42.43 min, t_r (*syn*. minor) = 45.15 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:6, R_f 0.2) to afford a white solid. m.p. 76-77°C. $[\alpha]^{20}_D$ (*syn*) = +13.6 (c = 0.11, in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51-7.48 (m, 2H), 7.04 (d, *J* = 8.4 Hz, 2H), 4.96-4.88 (m, 2H), 4.79 (dd, *J* = 13.6, 8.4 Hz, 1H), 4.02-3.96 (m, 1H), 1.60 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 131.5, 131.4, 128.6, 122.4, 82.8, 74.9, 45.9, 15.8. ESI-HRMS: calcd for $C_{10}H_{11}BrN_2O_4$ [M+H$^-$] 302.9986, found 302.9852.



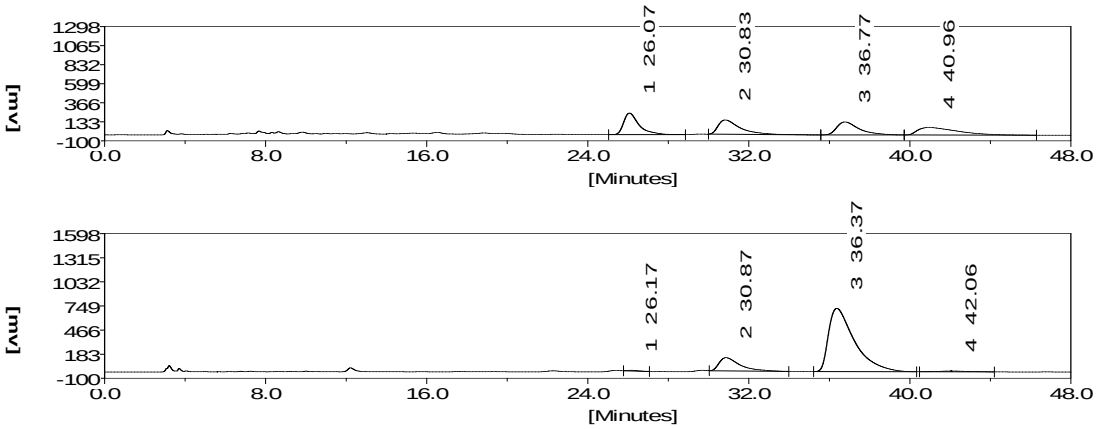


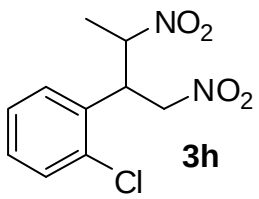


3g

2-(3-Chlorophenyl)-1,3-dinitrobutane (3g).
The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (97% *ee*) of the *syn* diastereoisomer and diastereoselectivity (86:14 d.r.) of the reaction were determined by HPLC analysis using a chiral AS-H column (hexane/2-propanol 90:10, 1.0 mL/min, UV 210 nm, t_r (*syn*. major) = 36.37 min, t_r (*syn*. minor) = 42.06 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:6, R_f 0.25) to afford a white solid. m.p. 82-84°C. $[\alpha]^{20}_D$ (*syn*) = +8.3 (c = 0.12, in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35-7.28 (m, 2H), 7.17 (d, *J* = 1.6 Hz, 1H), 7.07-7.04 (m, 1H), 4.98-4.89 (m, 2H), 4.80 (dd, *J* = 13.6, 8.4 Hz, 1H), 4.04-3.99 (m, 1H), 1.62-1.60 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 134.5, 134.2, 129.6, 128.4, 127.4, 124.9, 82.8, 74.8, 45.9, 15.8. ESI-HRMS: calcd for $C_{10}H_{11}ClN_2O_4$ [M+Na$^+$] 281.0300, found 281.0310.



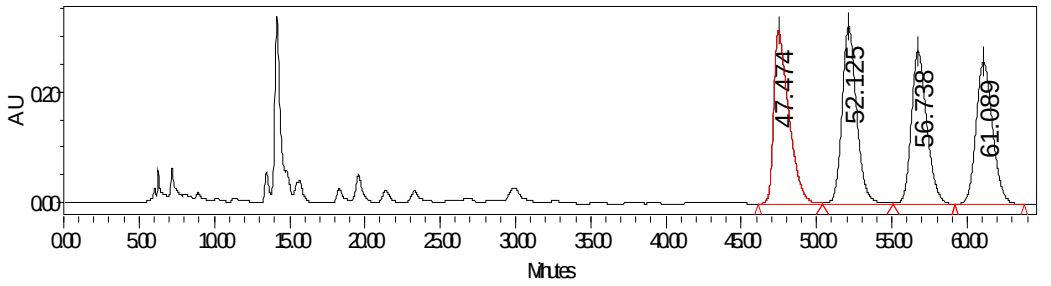


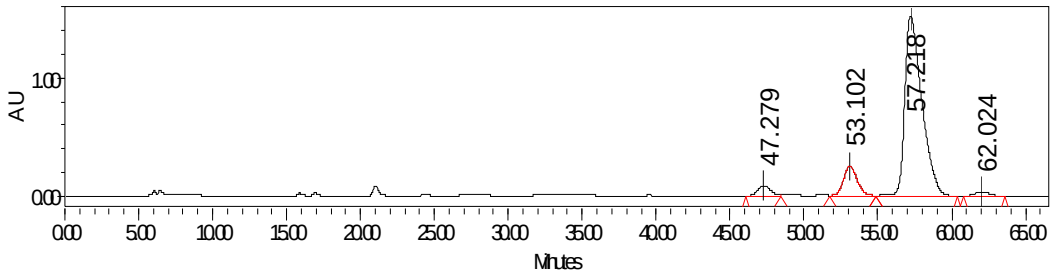
3h

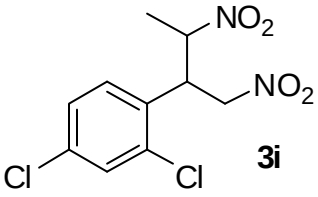
2-(2-Chlorophenyl)-1,3-dinitrobutane (3h).
The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (96% *ee*) of the *syn* diastereoisomer and diastereoselectivity (85:15 d.r.) of the reaction were determined by HPLC analysis using a chiral AD-H column (hexane/2-propanol 98:2, 0.5 mL/min, UV 210 nm, t_r (*syn*. major) = 57.22 min, t_r (*syn*. minor) = 62.02 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:6, R_f 0.3) to afford a yellow oil. $[\alpha]^{20}_D$ (*syn*) = -11.1 (c = 0.45, in CH_2Cl_2). ^{1}H NMR (400 MHz,

$CDCl_3$): δ 7.47-7.44 (m, 1H), 7.32-7.27 (m, 1H), 7.14 (dd, *J* = 7.2, 2.0 Hz, 1H), 5.20-5.13 (m, 1H), 4.97-4.85 (m, 2H), 4.73-4.68 (dd, *J* = 13.6, 6.8 Hz, 1H), 1.64-1.63 (d, *J* = 6.4 Hz, 3H).

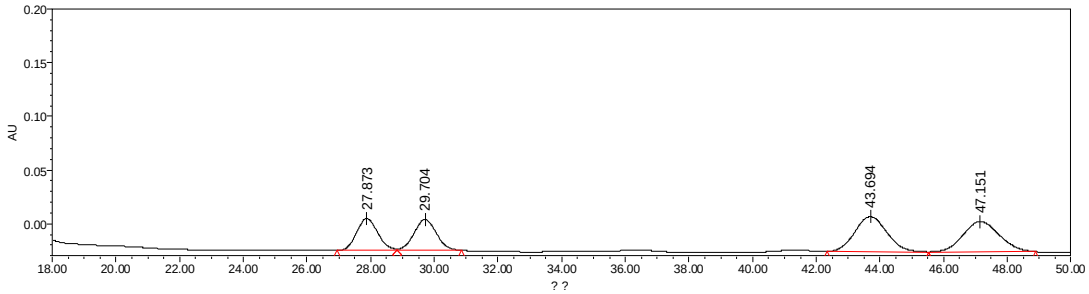


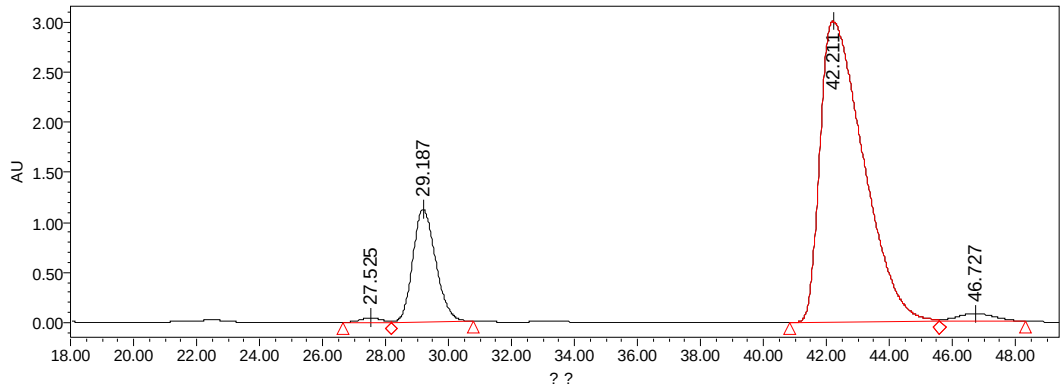


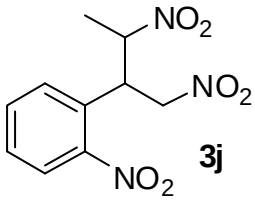


2-(2,4-Dichlorophenyl)-1,3-dinitrobutane (3i). The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (95% *ee*) of the *syn* diastereoisomer and diastereoselectivity (83:17 d.r.) of the reaction were determined by HPLC analysis using a chiral OD-H column (hexane/2-propanol 90:10, 0.7 mL/min, UV 210 nm, t_r (*syn*. major) = 42.21 min, t_r (*syn*. minor) = 46.73 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:4, R_f 0.45) to afford a yellow oil. $[\alpha]^{20}_D$ (*syn*) = -6.9 (c = 0.48, in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48 (t, *J* = 2.0 Hz, 1H), 7.28-7.25 (m, 1H), 7.08 (d, *J* = 8.4 Hz, 1H), 5.17-5.10 (m, 1H), 4.94-4.82 (m, 2H), 4.64 (dd, *J* = 13.6, 6.8 Hz, 1H), 1.65-1.63 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 135.7, 135.3, 130.6, 130.0, 129.1, 128.1, 82.8, 74.9, 42.7, 16.6. ESI-HRMS: calcd for $C_{10}H_{10}Cl_2N_2O_4$ [M+H$^-$] 293.0101, found 292.9900.

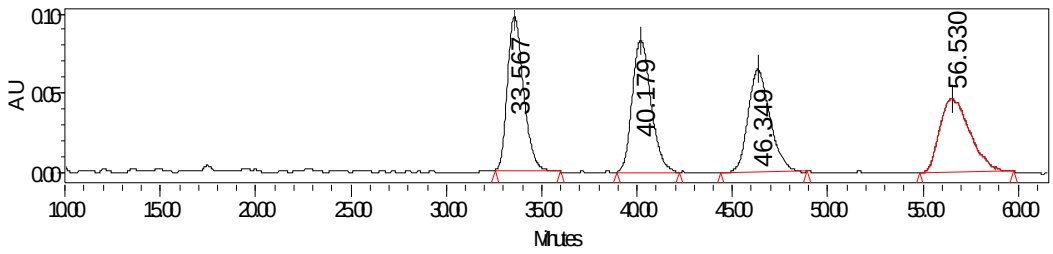


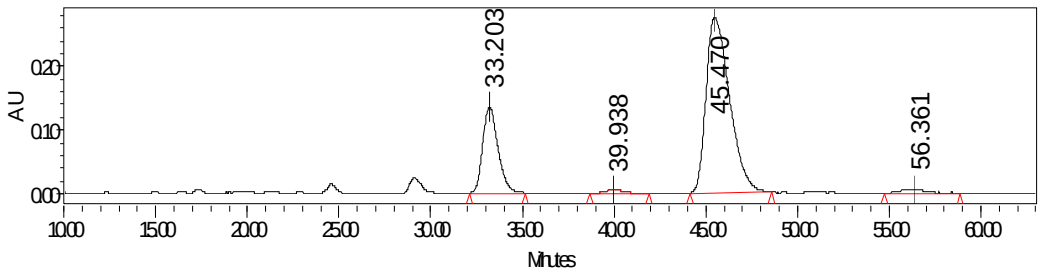


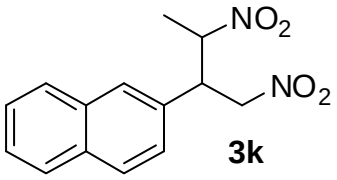


2-(2-Nitrophenyl)-1,3-dinitrobutane (3j).
The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (96% *ee*) of the *syn* diastereoisomer and diastereoselectivity (76:24 d.r.) of the reaction were determined by HPLC analysis using a chiral AS-H column (hexane/2-propanol 90:10, 1.0 mL/min, UV 210 nm, t_r (*syn*. major) = 45.47 min, t_r (*syn*. minor) = 56.36 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:6, R_f 0.2) to afford a white solid. m.p. 99-101°C. $[\alpha]^{20}_D$ (*syn*) = +19.2 (c = 0.13, in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (dd, *J* = 8.4, 0.8 Hz, 1H), 7.65-7.51 (m, 2H), 7.34 (dd, *J* = 8.0, 0.8 Hz, 1H), 5.30 (dt, *J* = 14.8, 6.8 Hz, 1H), 5.03 (dd, *J* = 13.6, 5.2 Hz, 1H), 4.93 (dd, *J* = 13.6, 6.8 Hz, 1H), 4.72 (dd, *J* = 12.8, 6.4 Hz, 1H), 1.73 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 150.0, 134.0, 130.2, 129.2, 126.1, 83.5, 75.8, 41.6, 17.5. ESI-HRMS: calcd for $C_{10}H_{11}N_3O_6$ [M+Na^+] 292.0540, found 292.0547.



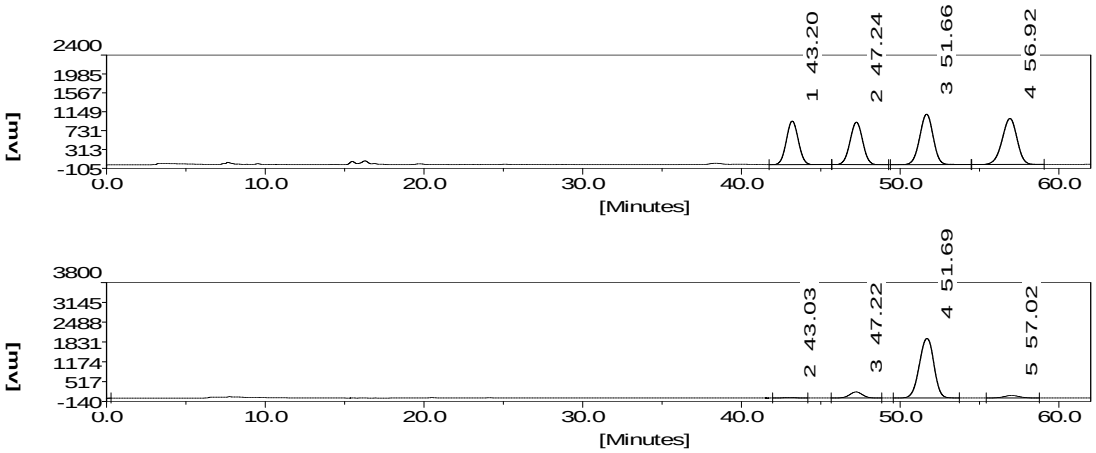


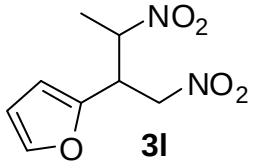


3k

2-(2-Naphthyl)-1,3-dinitrobutane (3k).
The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (92% *ee*) of the *syn* diastereoisomer and diastereoselectivity (92:8 d.r.) of the reaction were determined by HPLC analysis using a chiral AD-H column (hexane/2-propanol 90:10, 0.5 mL/min, UV 210 nm, t_r (*syn*. major) = 51.69 min, t_r (*syn*. minor) = 57.02 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:6, R_f 0.35) to afford a yellow oil. $[\alpha]^{20}_D$ (*syn*) = -38.8 (c = 0.50, in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.19 (d, *J* = 8.4 Hz, 1H), 7.92-7.85 (dd, *J* = 21.2, 8.4 Hz, 2H), 7.69-7.64 (t, *J* = 8.4 Hz, 1H), 7.59-7.55 (t, *J* = 8.0 Hz, 1H), 7.46 (t, *J* = 8.0 Hz, 1H), 7.33 (dd, *J* = 7.2, 0.8 Hz, 1H), 5.22-5.15 (m, 2H), 5.01 (dd, *J* = 6.4, 2.0 Hz, 2H), 1.60 (d, *J* = 6.4 Hz, 3H).

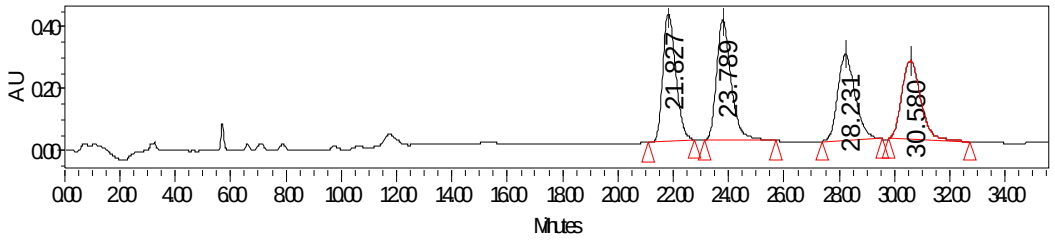


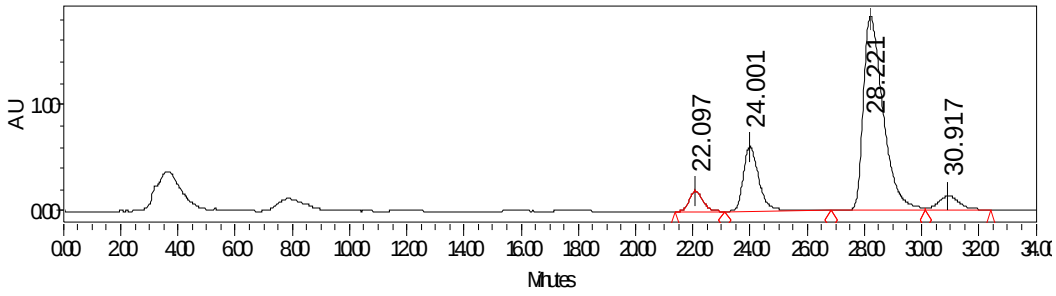


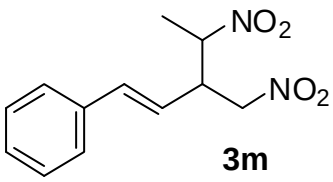
3l

2-(2-Furyl)-1,3-dinitrobutane (3l).
The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (86% *ee*) of the *syn* diastereoisomer and diastereoselectivity (81:19 d.r.) of the reaction were determined by HPLC analysis using a chiral OD-H column (hexane/2-propanol 90:10, 1.0 mL/min, UV 210 nm, t_r (*syn*. major) = 28.22 min, t_r (*syn*. minor) = 30.92 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:6, R_f 0.35) to afford a yellow oil. $[\alpha]^{20}_D$ (*syn*) = +24.3 (c = 0.28, in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 (d, *J* = 1.6 Hz, 1H), 6.34 (dd, *J* = 3.2, 2.0 Hz, 1H), 6.27 (d, *J* = 3.6 Hz, 1H), 4.97-4.81 (m, 3H), 4.25-4.21 (m, 1H), 1.61 (d, *J* = 6.8 Hz, 3H).



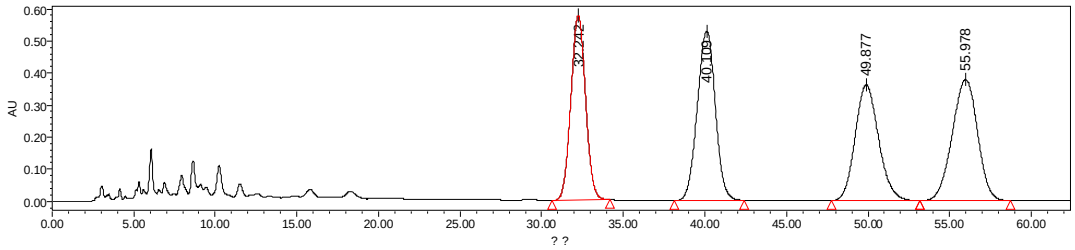


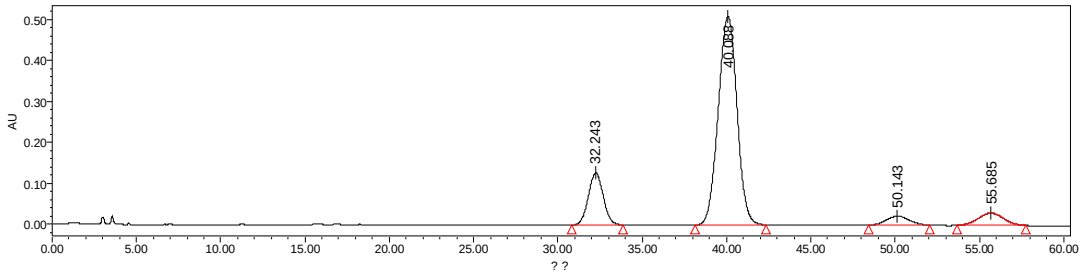


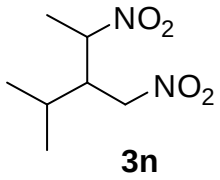
3m

2-styryl-1,3-dinitrobutane (3m).
The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (85% *ee*) of the *syn* diastereoisomer and diastereoselectivity (81:19 d.r.) of the reaction were determined by HPLC analysis using a chiral OD-H column (hexane/2-propanol 90:10, 1.0 mL/min, UV 210 nm, t_r (*syn*. major) = 40.088 min, t_r (*syn*. minor) = 55.685 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:5, R_f 0.3) to afford a white solid. m.p. 55-57°C. $[\alpha]^{20}_D$ (*syn*) = +52.7 (c = 0.11, in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36-7.27 (m, 5H), 6.64-6.60 (d, *J* = 15.6 Hz, 1H), 5.91 (dd, *J* = 15.6, 9.6 Hz, 1H), 4.85-4.79 (m, 1H), 4.77-4.72 (m, 1H), 4.59 (dd, *J* = 13.2, 8.0 Hz, 1H), 1.63 (dd, *J* = 6.8, 2.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 136.8, 134.3, 127.7, 125.7, 118.7, 82.1, 75.3, 44.6, 15.7. ESI-HRMS: calcd for $C_{12}H_{14}N_2O_4$ [M+Na^+] 273.0846, found 273.0852.



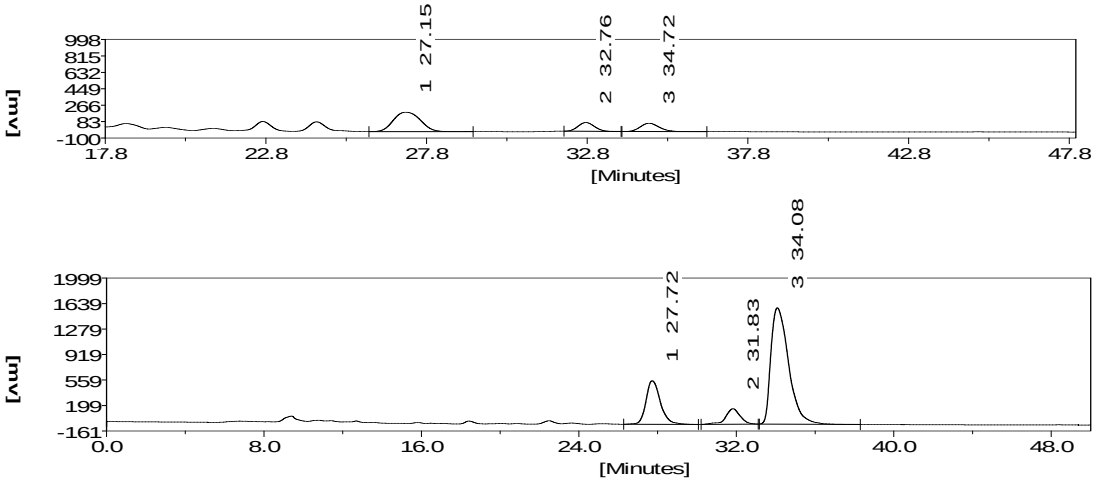


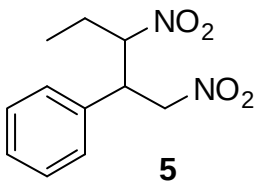


3n

2-isopropyl-1,3-dinitrobutane (3n).
The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (83% *ee*) of the *syn* diastereoisomer and diastereoselectivity (78:22 d.r.) of the reaction were determined by HPLC analysis using a chiral OD-H column (hexane/2-propanol 90:10, 0.4 mL/min, UV 210 nm, t_r (*syn*. major) = 31.83 min, t_r (*syn*. minor) = 34.08 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:9, R_f 0.4) to afford a yellow oil. $[\alpha]^{20}_D$ (*syn*) = -3.6 (c = 0.59, in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.78 (t, *J* = 6.8 Hz, 1H), 4.52-4.41 (m, 2H), 2.89-2.84 (m, 1H), 1.89-1.82 (m, 1H), 1.06 (d, *J* = 6.8 Hz, 5H), 0.92 (d, *J* = 6.8 Hz, 4H).



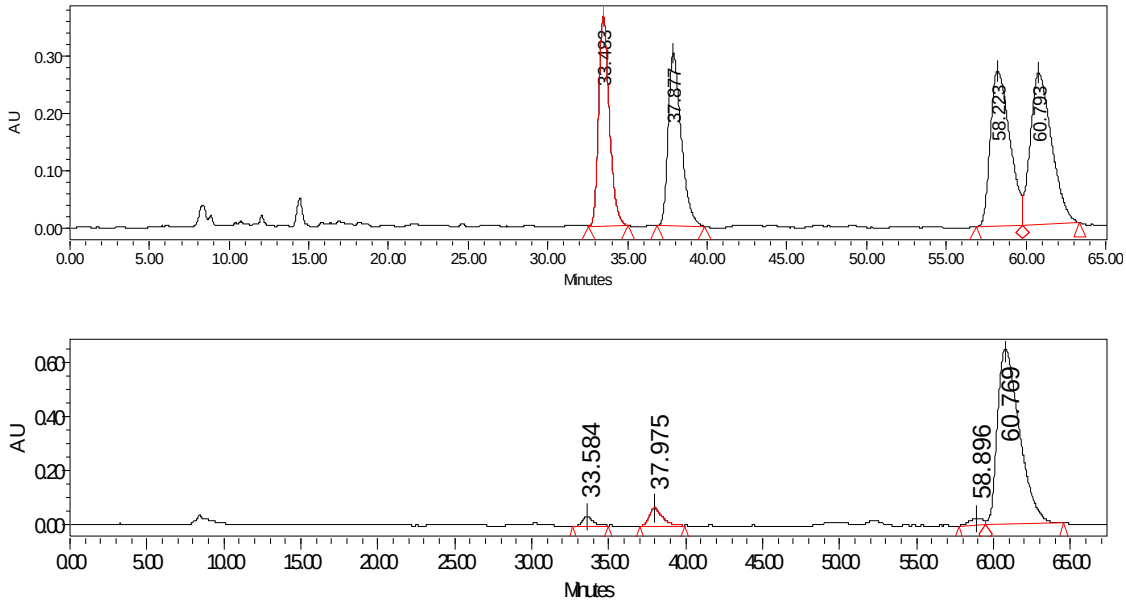


5

2-Phenyl-1,3-dinitropentane (5).
The *syn/anti* mixtures of products were analyzed to determine the diastereoselectivity of the reaction and enantioselectivity of the *syn* diastereoisomer. The ee value (95% *ee*) of the *syn* diastereoisomer and diastereoselectivity (92:8 d.r.) of the reaction were determined by HPLC analysis using a chiral AS-H column (hexane/2-propanol 90:10, 0.4 mL/min, UV 210 nm, t_r (*syn*. minor) = 58.90 min, t_r (*syn*. major) = 60.77 min).

syn Isomer, purified by flash chromatography (ethyl acetate : petroleum ether = 1:6, R_f 0.3) to afford a white solid. m.p. 54-56^{o}C. $[\alpha]^{20}_D$ (*syn*) = +10.0 (c = 0.11, in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30-7.26 (m, 3H), 7.10-7.07 (m, 2H), 4.85-4.79 (ddd, *J* =

13.4, 6.4, 2.8 Hz, 1H), 4.74-4.68 (m, 2H), 3.97 (dd, *J* = 14.4, 6.8 Hz, 1H), 1.99-1.91 (m, 1H), 1.83-1.75 (m, 1H), 0.95 (dt, *J* = 7.2, 2.4 Hz, 3H) .



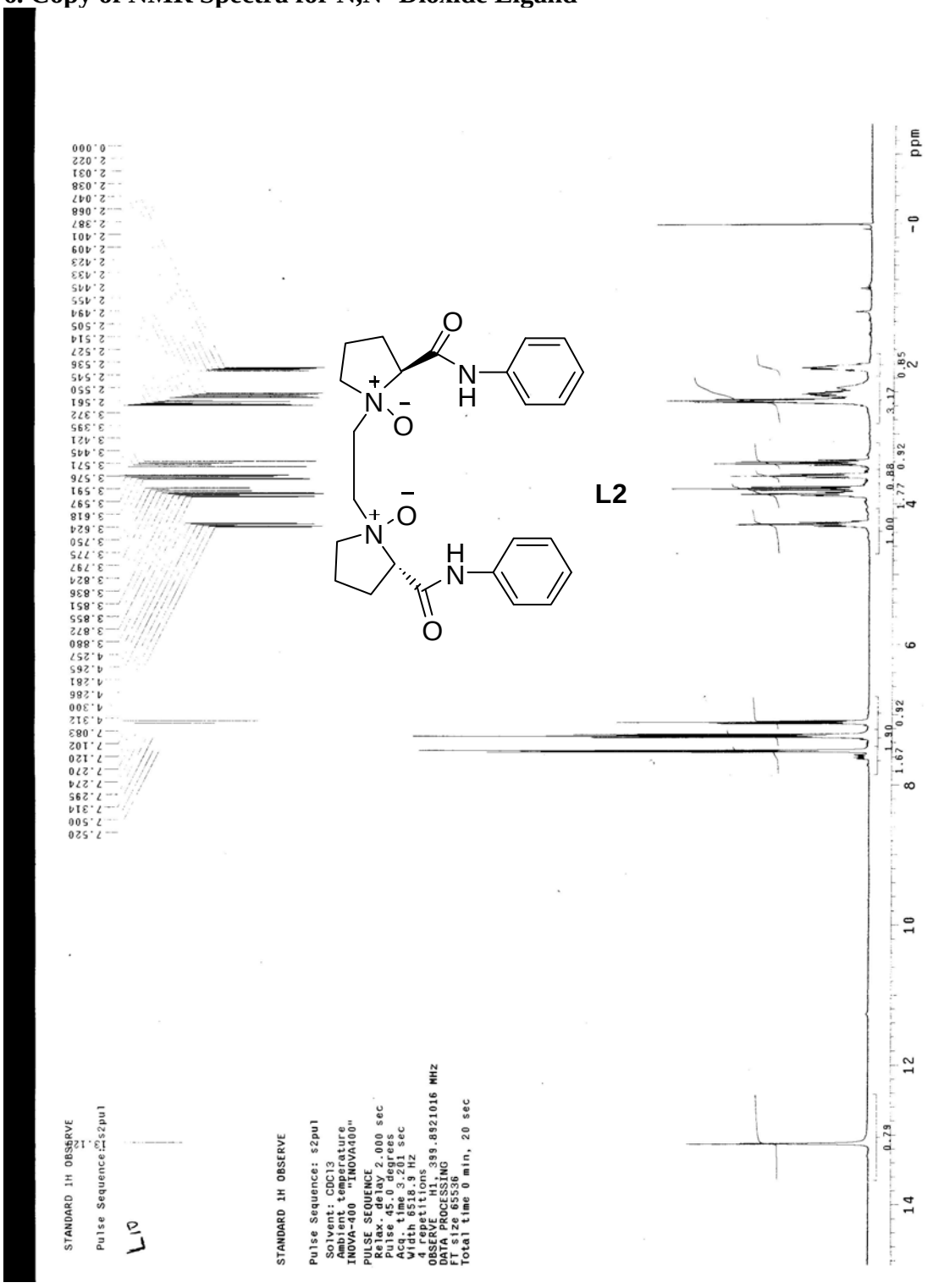
5. References

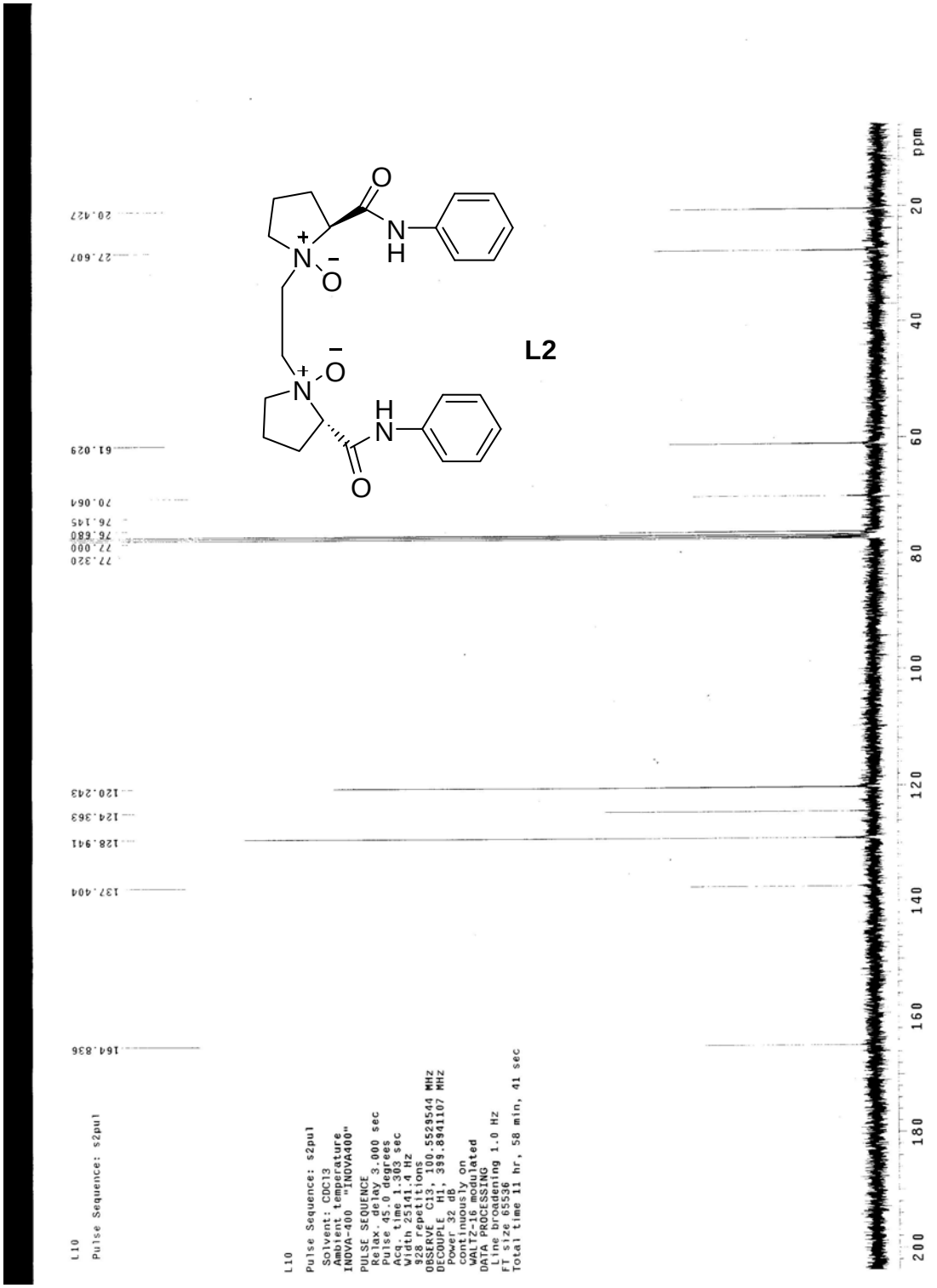
[1] a) D. M. Mampreian, A. H. Hoveyda, *Org. Lett*. **2004**, *6*, 2829; b) C. Dockendorff, S. Sahli, M. Olsen, L. Milhau, M. Lautens, *J. Am. Chem. Soc*. **2005**, *127*, 15028; c) A. Côté, V. N. G. Lindsay, A. B. Charette, *Org. Lett*. **2007**, *9*, 85.

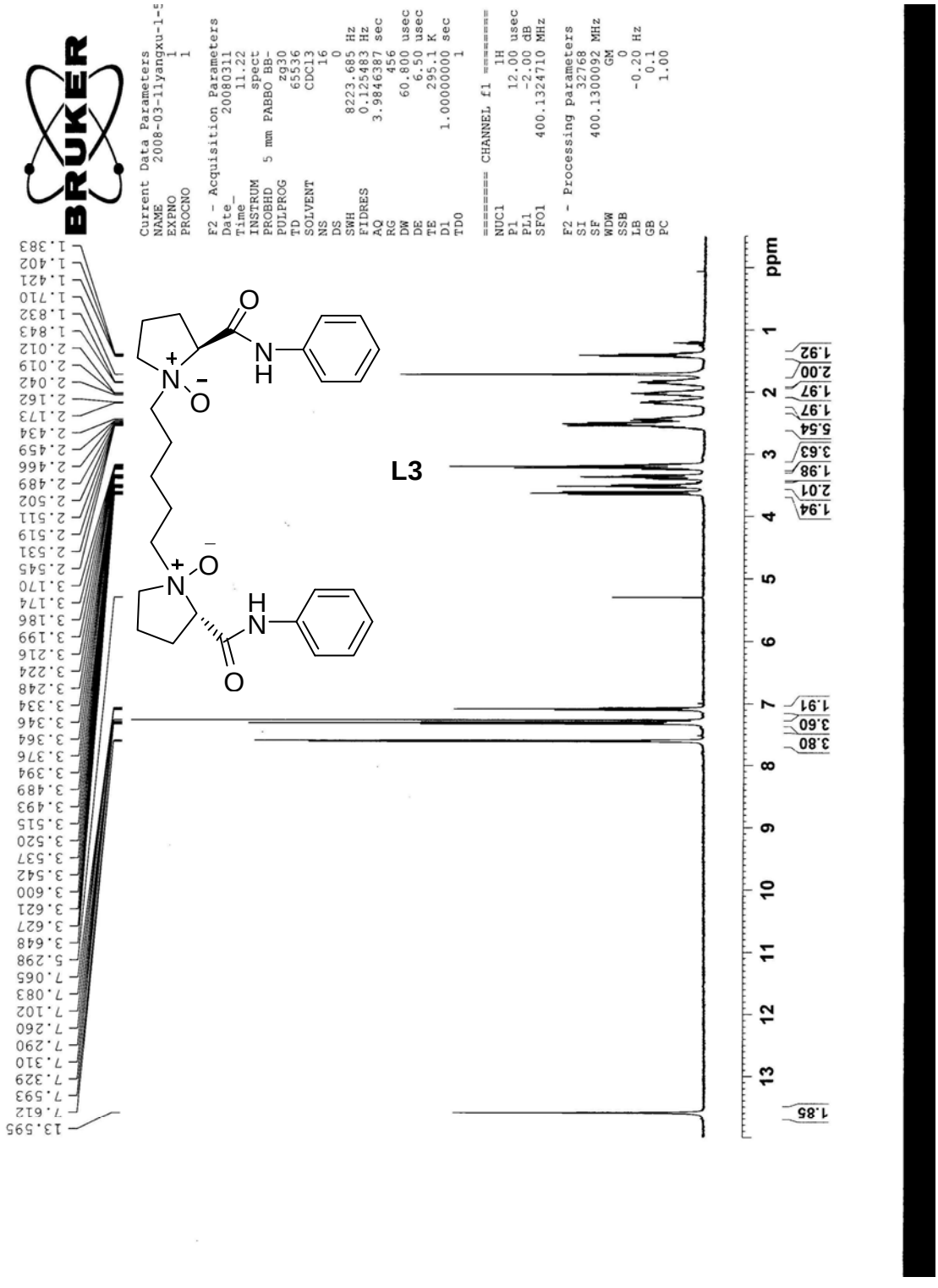
[2] a) Y. H. Wen, X. Huang, J. L. Huang, Y. Xiong, B. Qin, X. M. Feng, *Synlett*. 2005, 2445; b) X. Zhang, D. H. Chen, X. H. Liu, X. M. Feng, *J. Org. Chem.* 2007, *72*, 5227; c) D. J. Shang, J. G. Xin, Y. L.Liu, X. Zhou, X. H. Liu, X. M. Feng, *J. Org. Chem.* 2008, *73*, 630; d) Z. P. Yu, X. H. Liu, Z. H. Dong, M. S. Xie, X. M. Feng, *Angew. Chem*. 2008, *120*, 1328; *Angew. Chem. Int. Ed*. 2008, *47*, 1308; e) J. L. Huang, J. Wang, X. H. Chen, Y. H. Wen, X. H. Liu, X. M. Feng, *Adv. Synth. Catal.* 2008, *350*, 287.

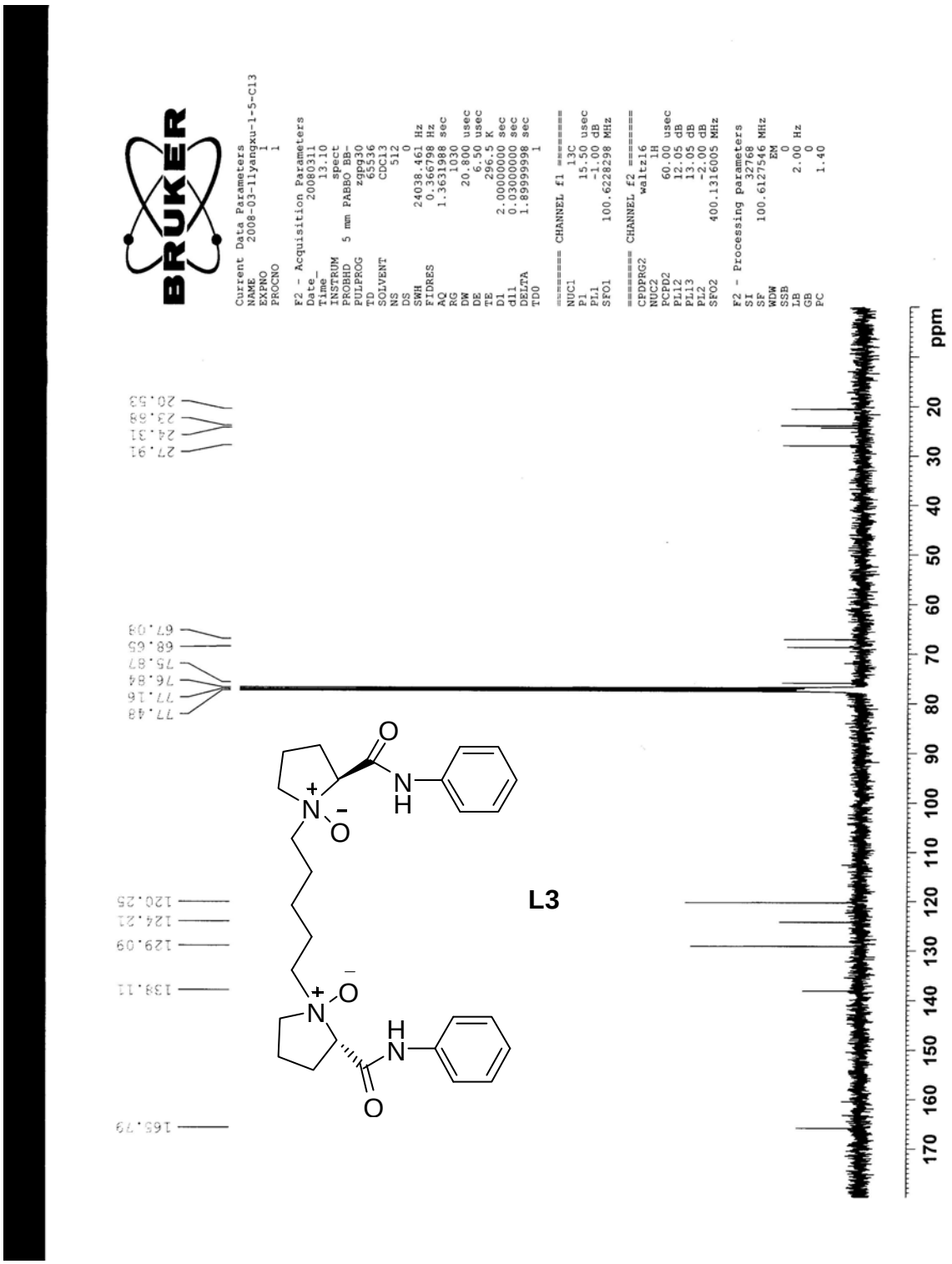
[3] S. F. Lu, D. M. Du, J. X. Xu, S. W. Zhang, *J. Am. Chem. Soc.* **2006**, *128*, 7418.

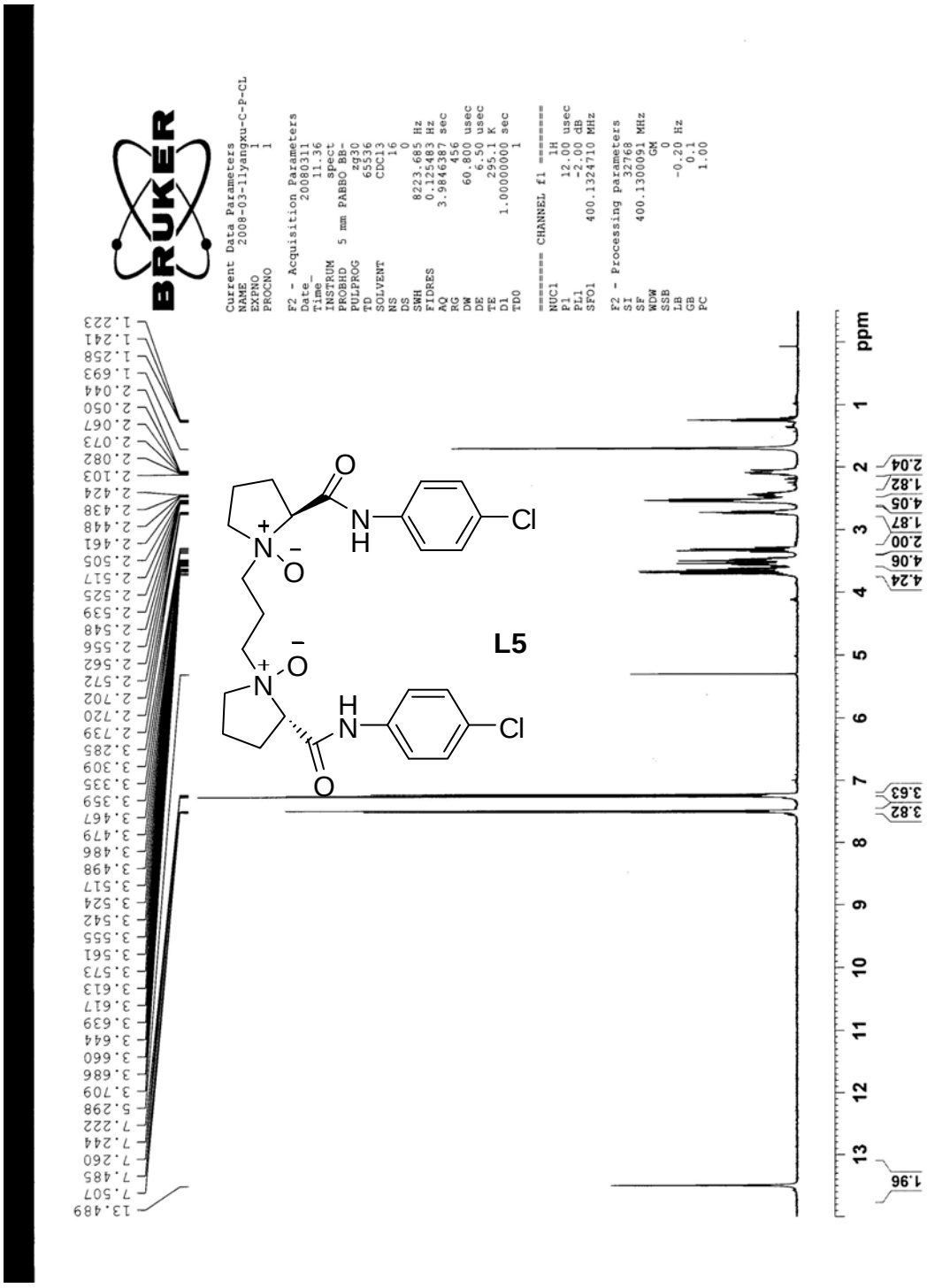
6. Copy of NMR Spectra for N,N'-Dioxide Ligand

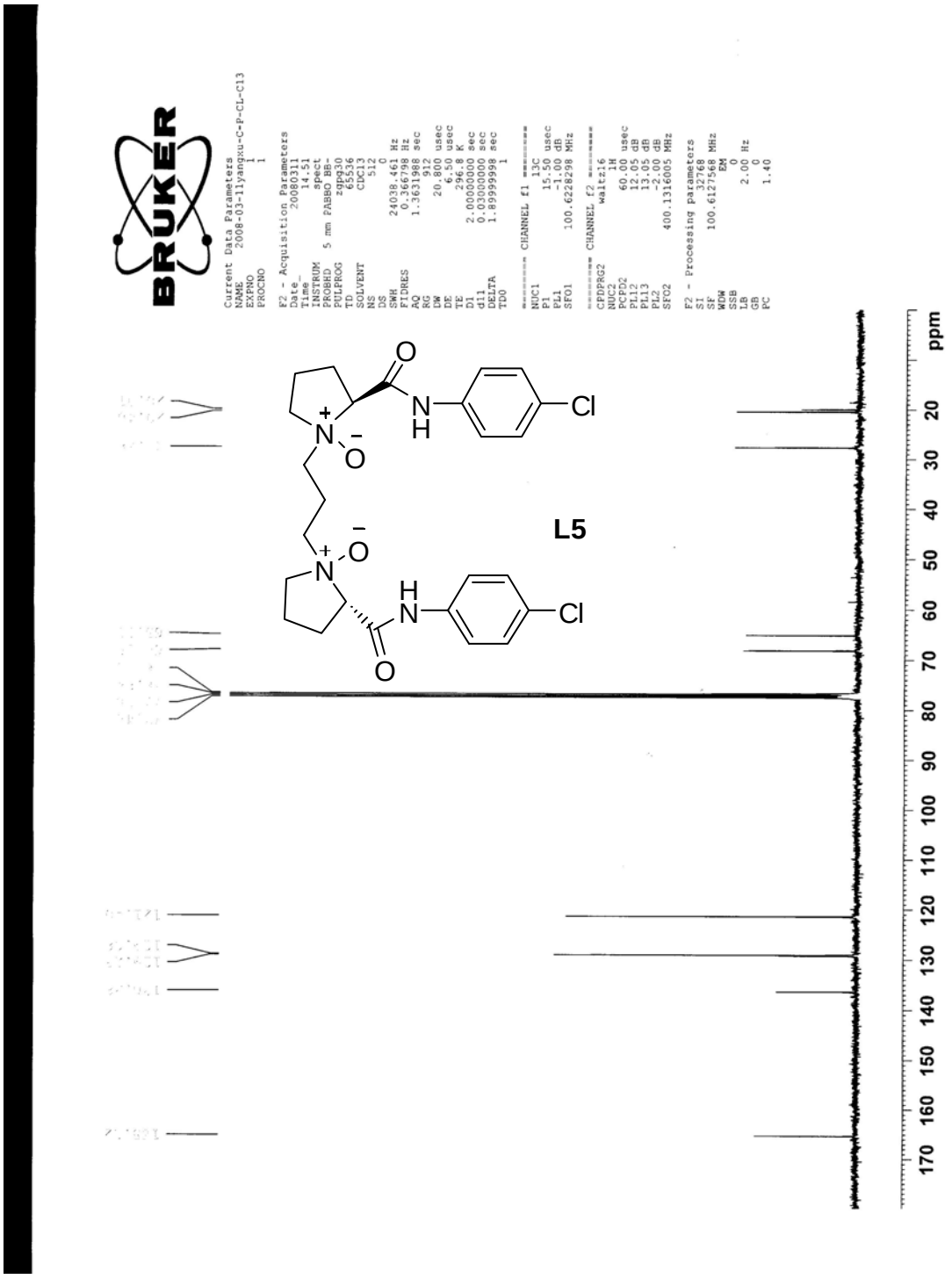



L2
20.427
27.607
61.029
70.064
76.145
76.680
77.000
77.320
120.243
124.363
128.941
137.404
164.836
ppm
20
40
60
80
100
120
140
160
180
200
L10
Pulse Sequence: s2pul
L10
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-400 "INOVA400"
PULSE SEQUENCE
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 1.303 sec
Width 25141.4 Hz
8838 repetitions
OBSERVE C13, 100.5529544 MHz
DECOUPLE H1, 399.8941107 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 11 hr, 58 min, 41 sec

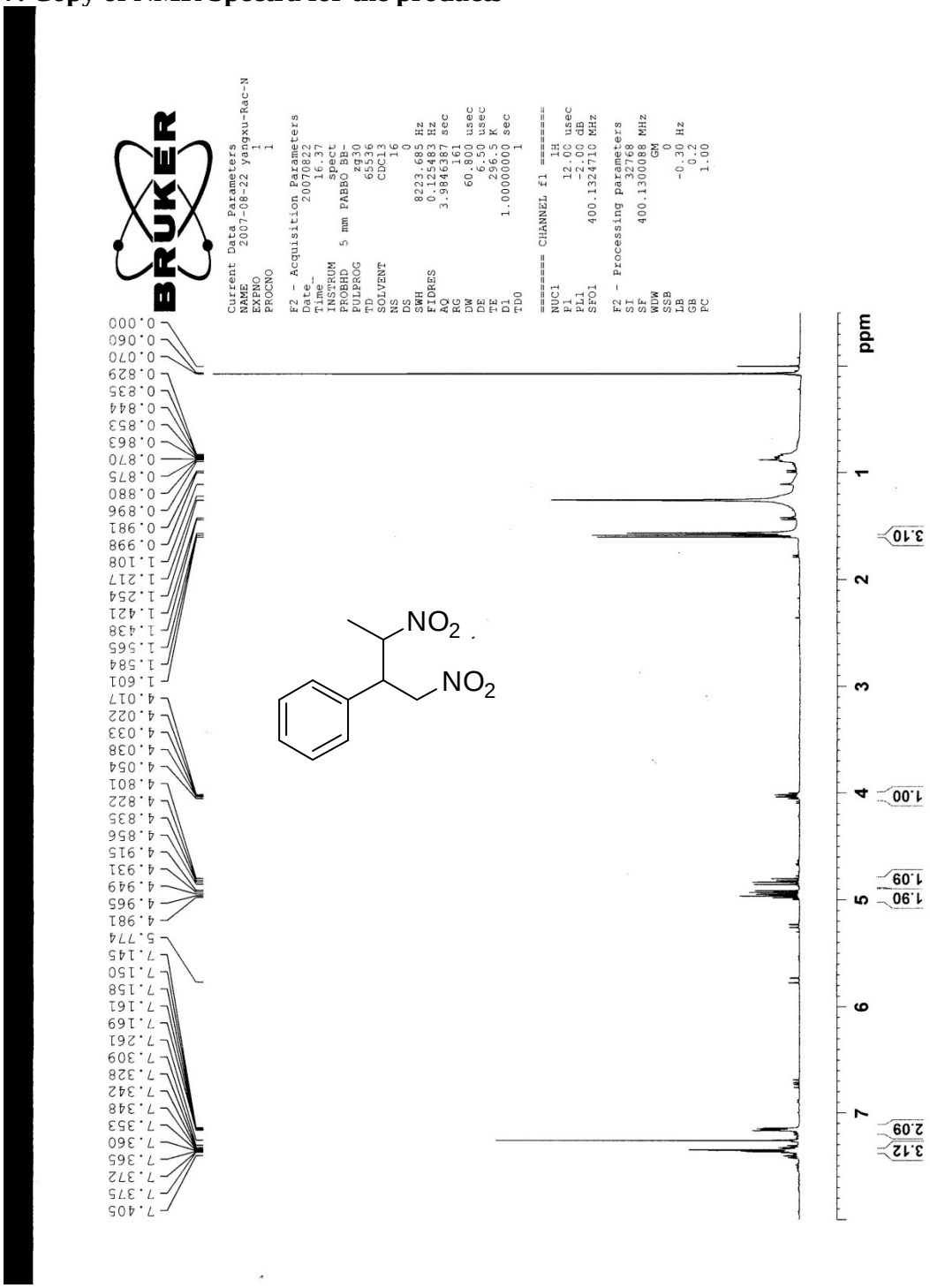

BRUKER
L3
ppm

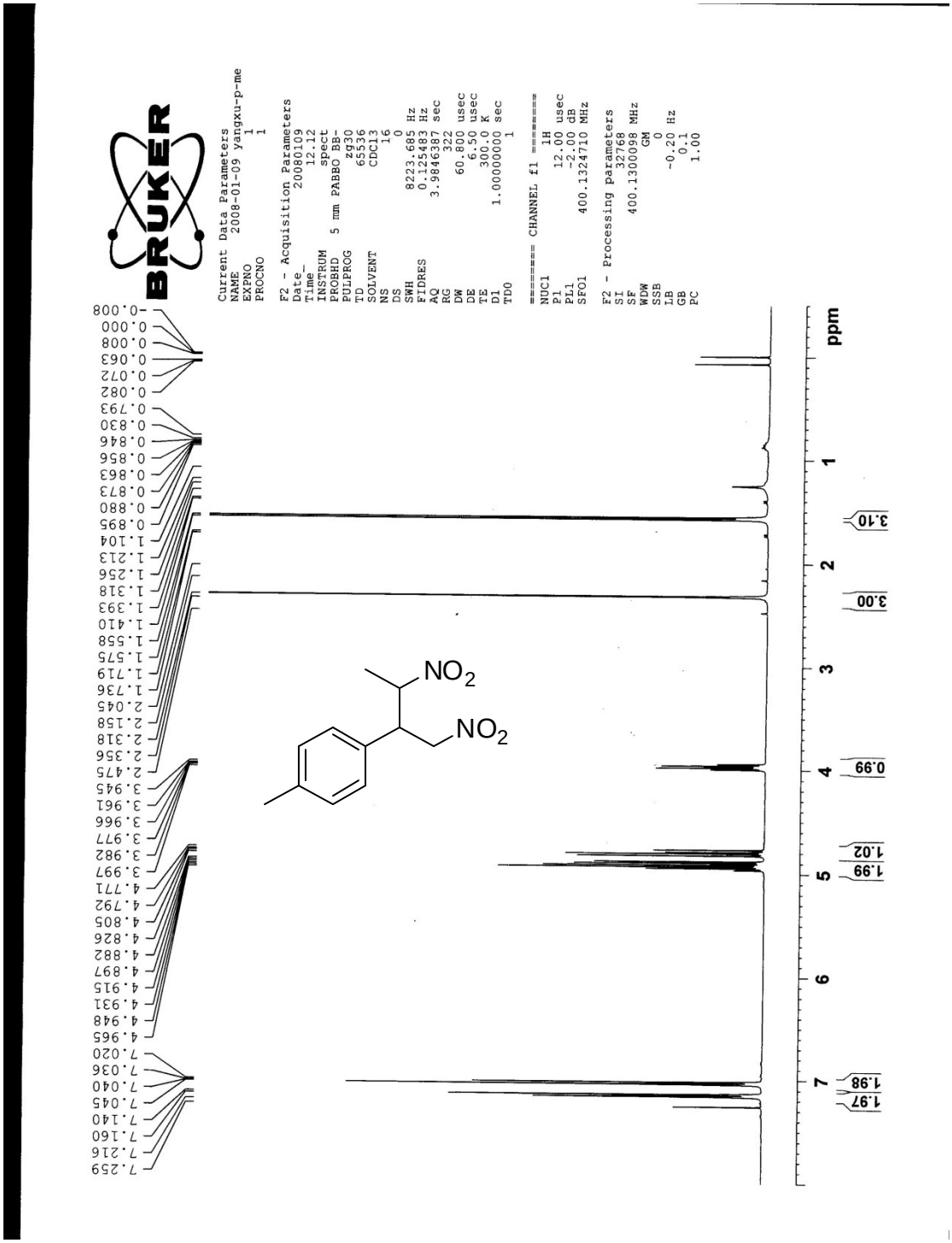

BRUKER
Current Data Parameters
NAME 2008-03-liyangxu-1-5-C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080311
Time_ 13.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.36319888 sec
RG 1030
DW 20.800 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 12.05 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127546 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
20.53
23.58
24.31
27.91
67.08
68.65
75.87
76.84
77.16
77.48
120.25
124.21
129.09
138.11
165.79
L3
ppm

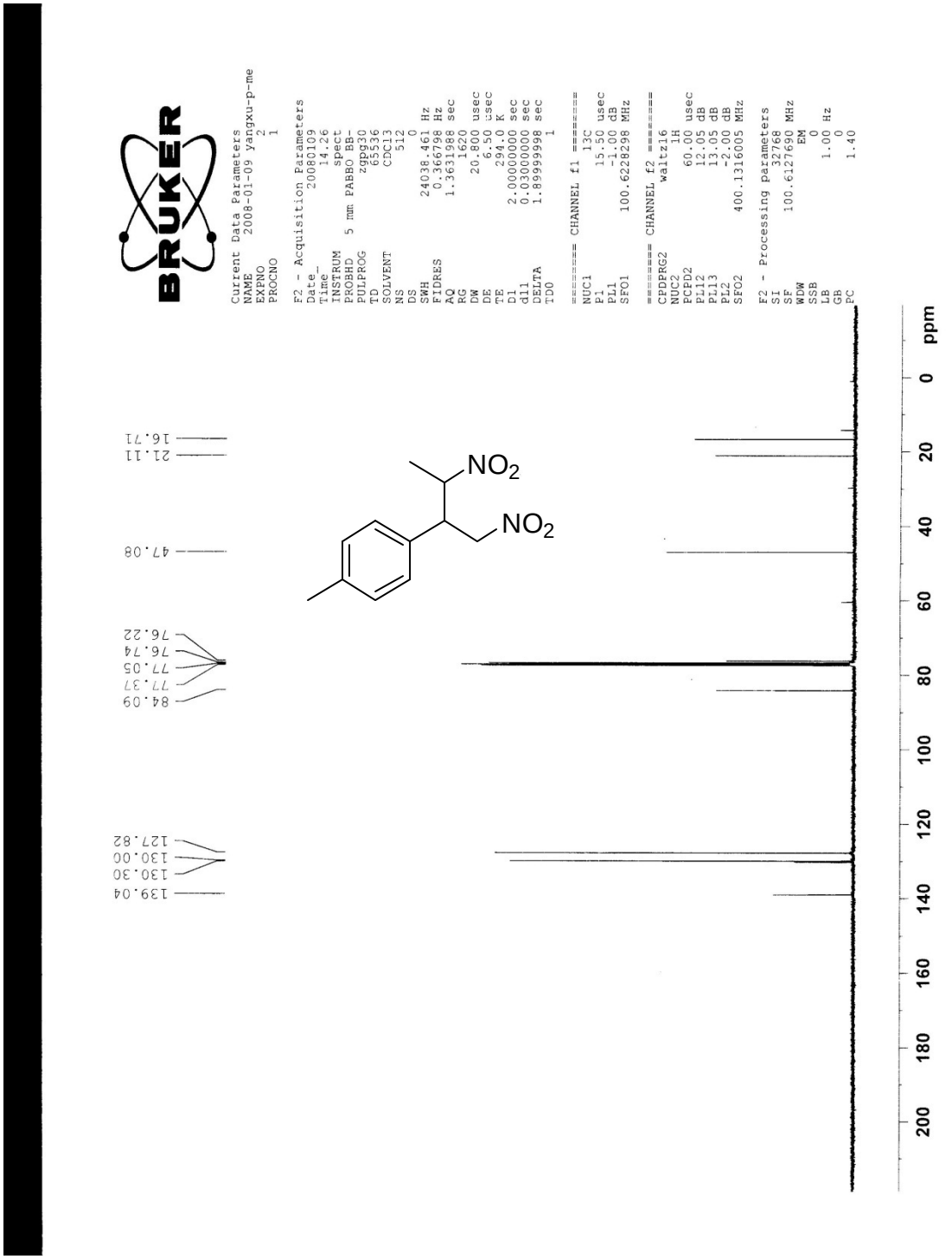

BRUKER
Current Data Parameters
NAME 2008-03-lilyangxu-C-P-CL
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080311
Time_ 11.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 456
DW 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300091 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
L5
ppm
1.223
1.241
1.258
1.693
2.044
2.050
2.067
2.073
2.082
2.103
2.424
2.438
2.448
2.461
2.505
2.517
2.525
2.539
2.548
2.556
2.562
2.572
2.702
2.720
2.739
3.285
3.309
3.335
3.359
3.467
3.479
3.486
3.498
3.517
3.524
3.542
3.555
3.561
3.573
3.613
3.617
3.639
3.644
3.660
3.686
3.709
5.298
7.222
7.244
7.260
7.485
7.507
13.489
2.04
1.82
4.05
1.87
2.00
4.06
4.24
3.82
3.63
1.96

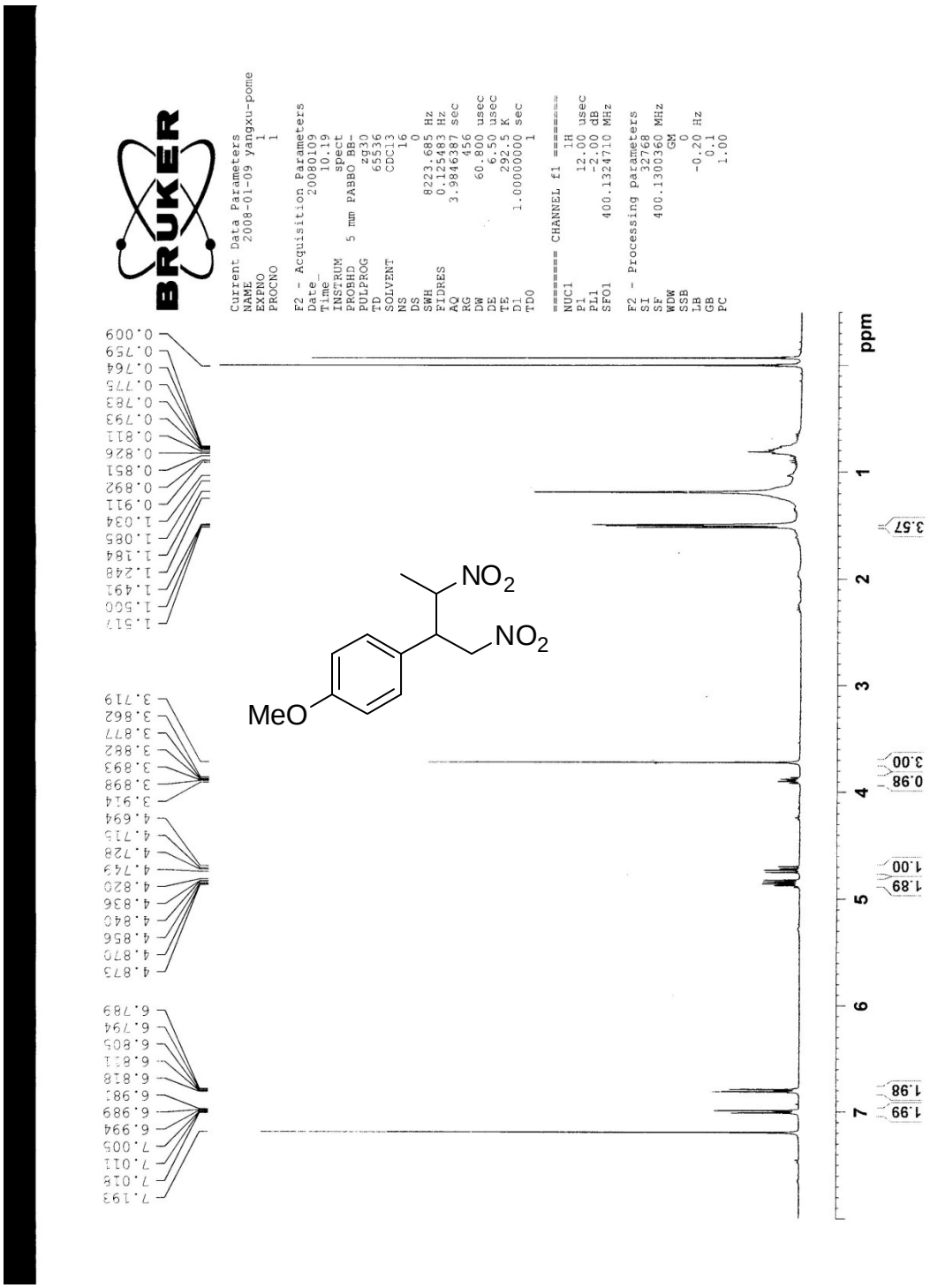

BRUKER
Current Data Parameters
NAME 2008-03-11yangxu-C-P-CL-C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080311
Time_ 14.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 912
DW 20.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 12.05 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127568 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
L5
ppm

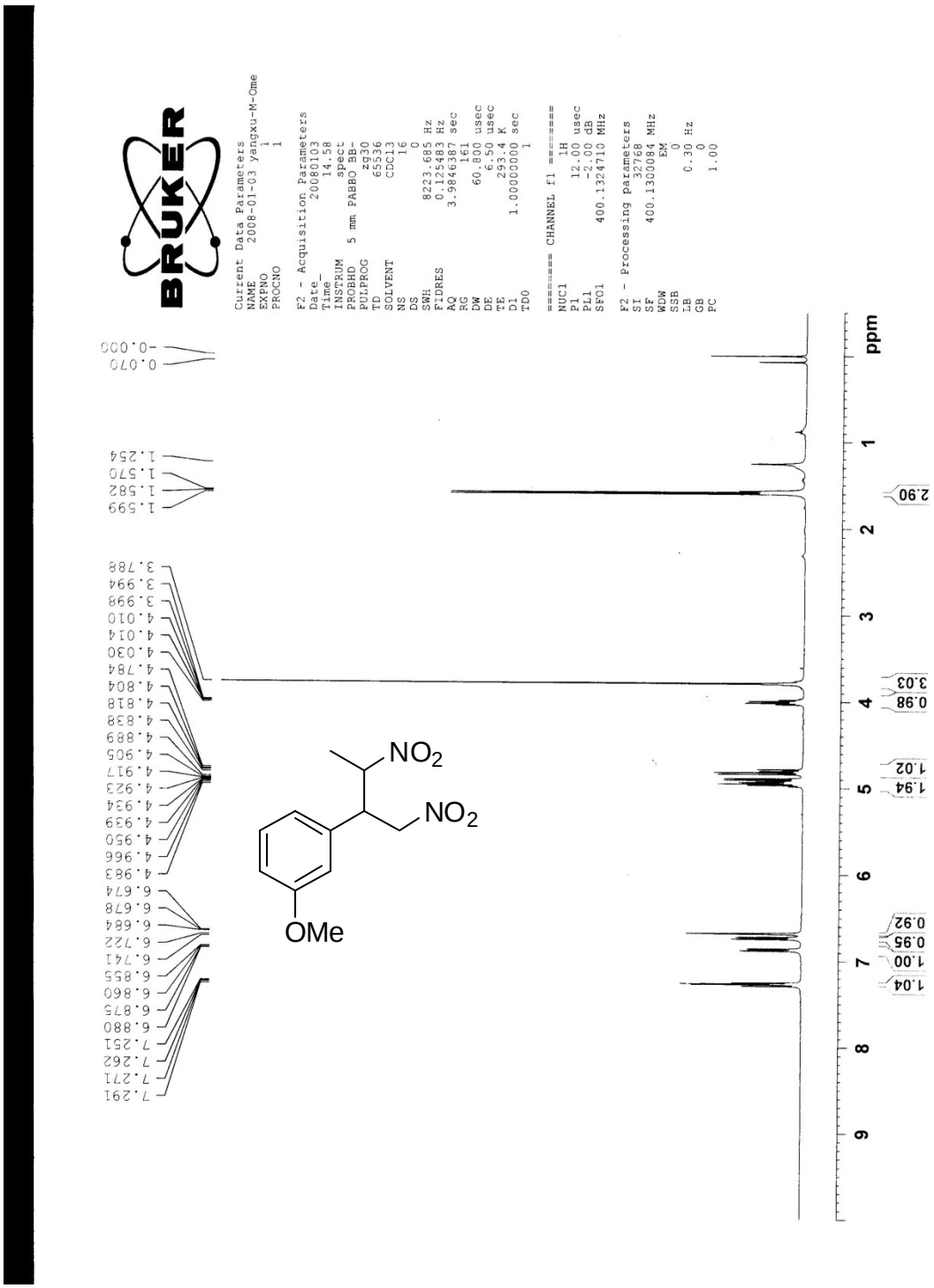
7. Copy of NMR Spectra for the products

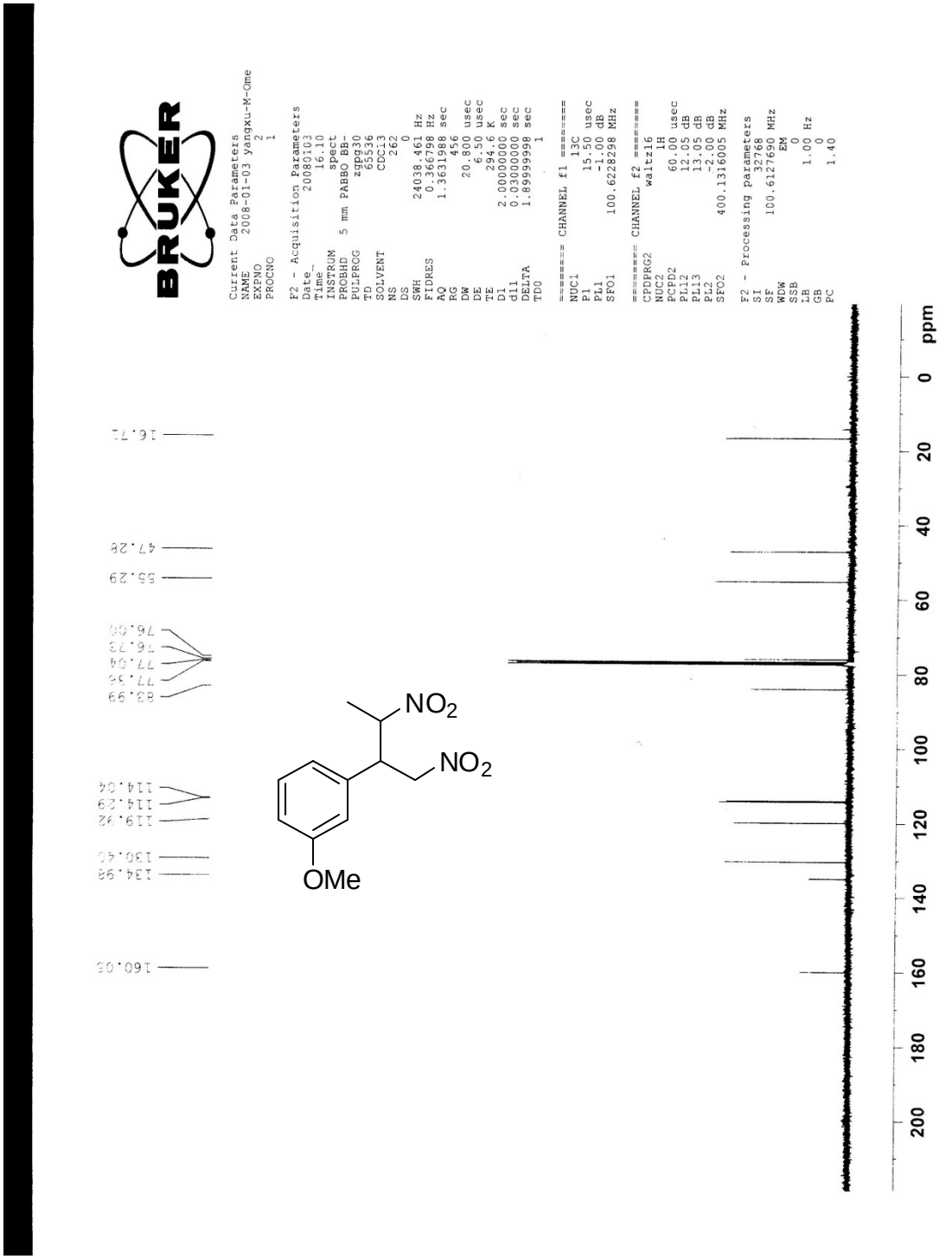


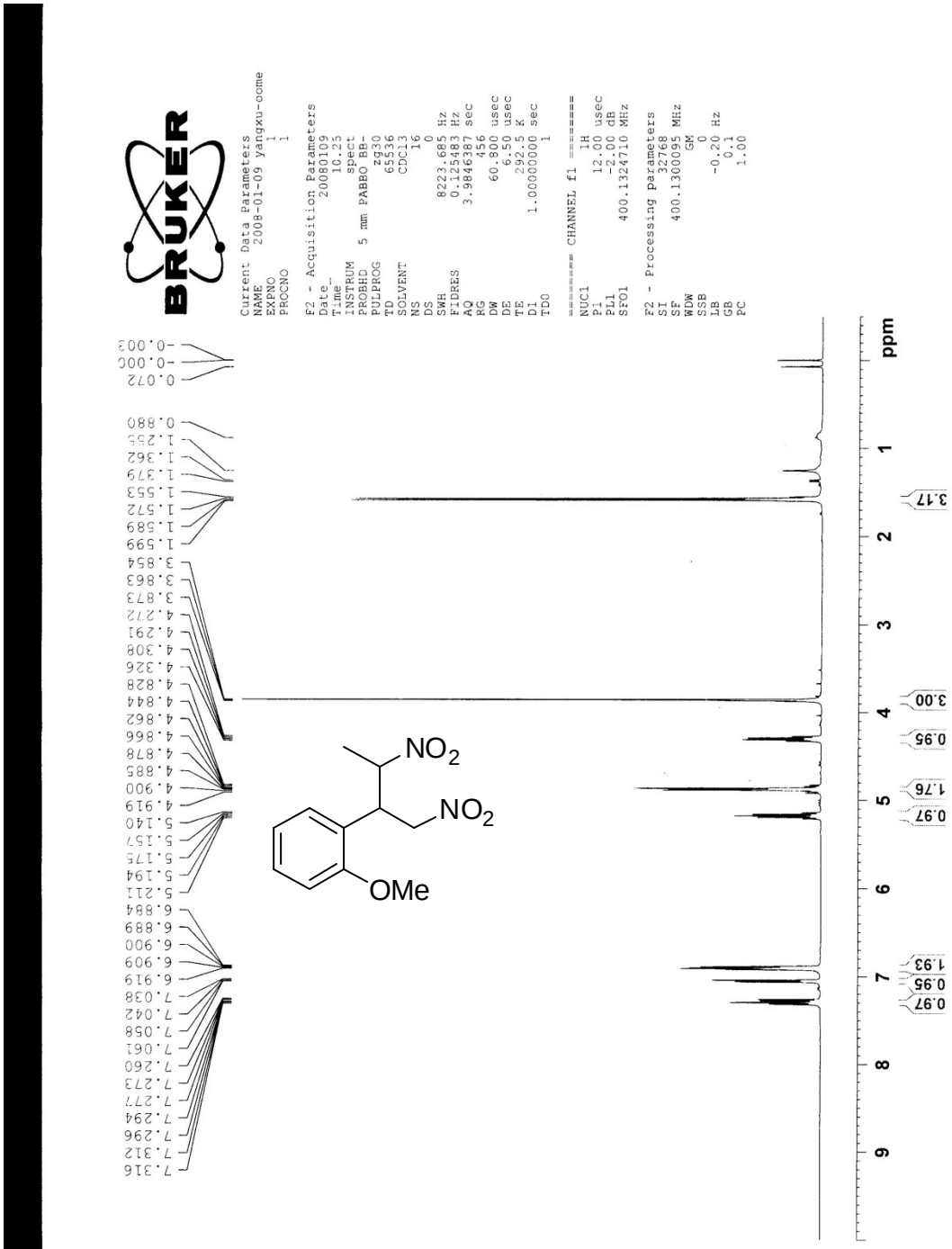

Current Data Parameters
NAME 2008-01-09 yangxu-p-me
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080109
Time_ 12.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 322
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300098 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
NO2
NO2
ppm
3.10
3.00
0.99
1.02
1.99
1.98
1.97

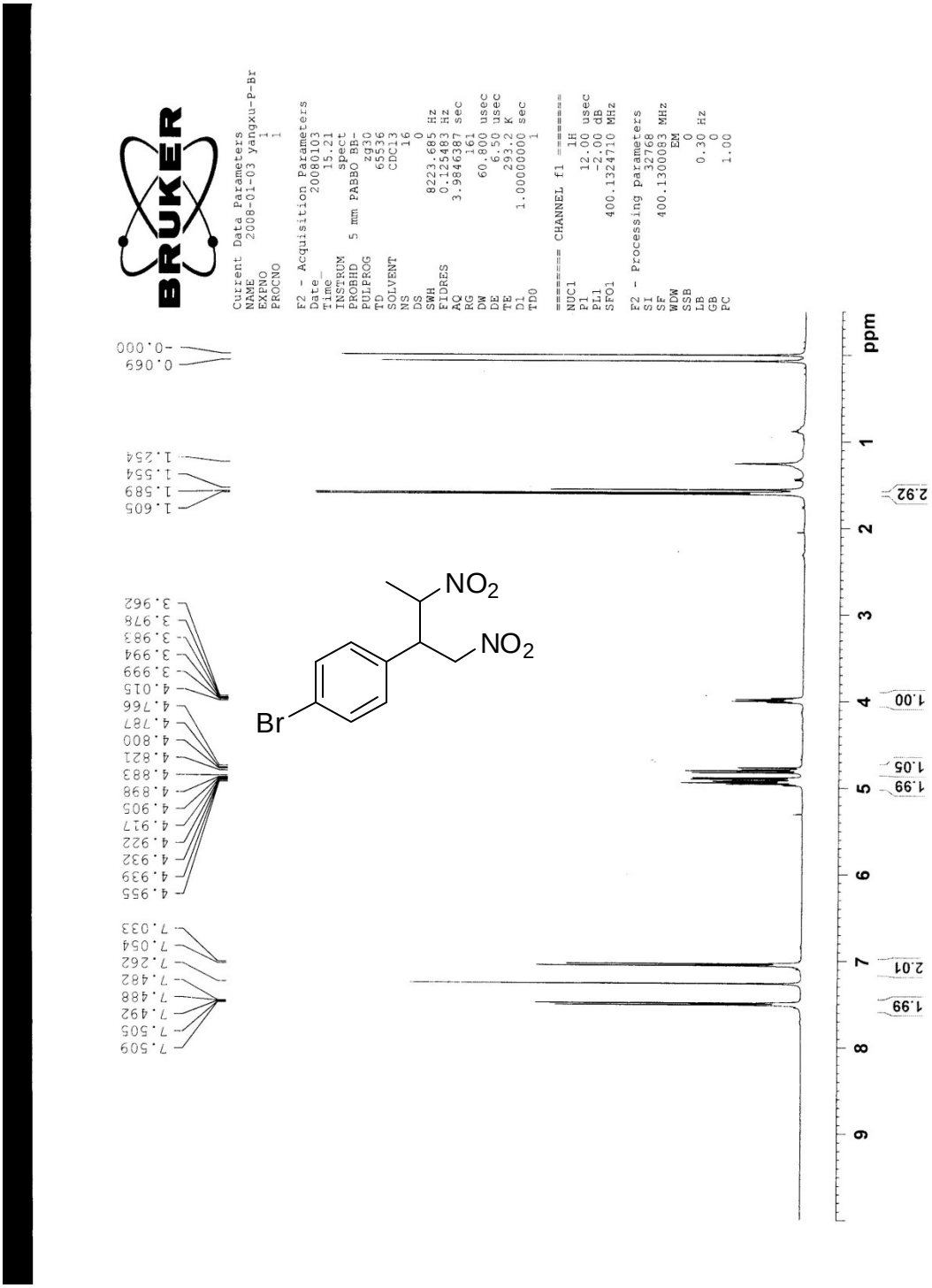

Current Data Parameters
NAME 2008-01-09 yangxu-p-me
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080109
Time_ 14.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 1620
DW 20.800 usec
DE 6.50 usec
TE 294.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 12.05 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
NO2
NO2
16.71
21.11
47.08
76.22
76.74
77.05
77.37
84.09
127.82
130.00
130.30
139.04
ppm
0
20
40
60
80
100
120
140
160
180
200

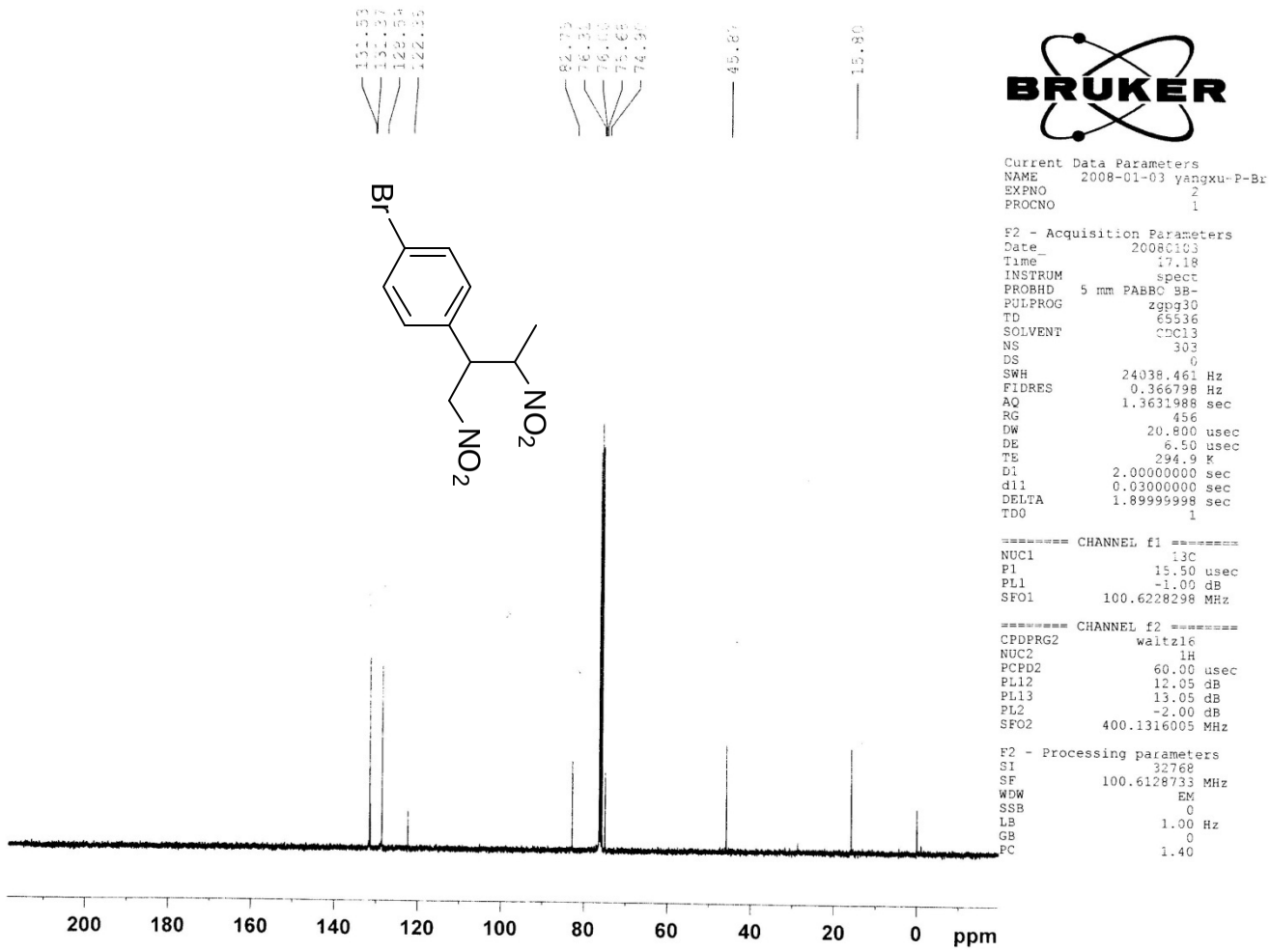

Current Data Parameters
NAME 2008-01-09 yangxu-pome
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080109
Time_ 10.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 456
DW 60.800 usec
DE 6.50 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300360 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
NO2
NO2
MeO
ppm
3.57
3.00
0.98
1.00
1.89
1.98
1.99

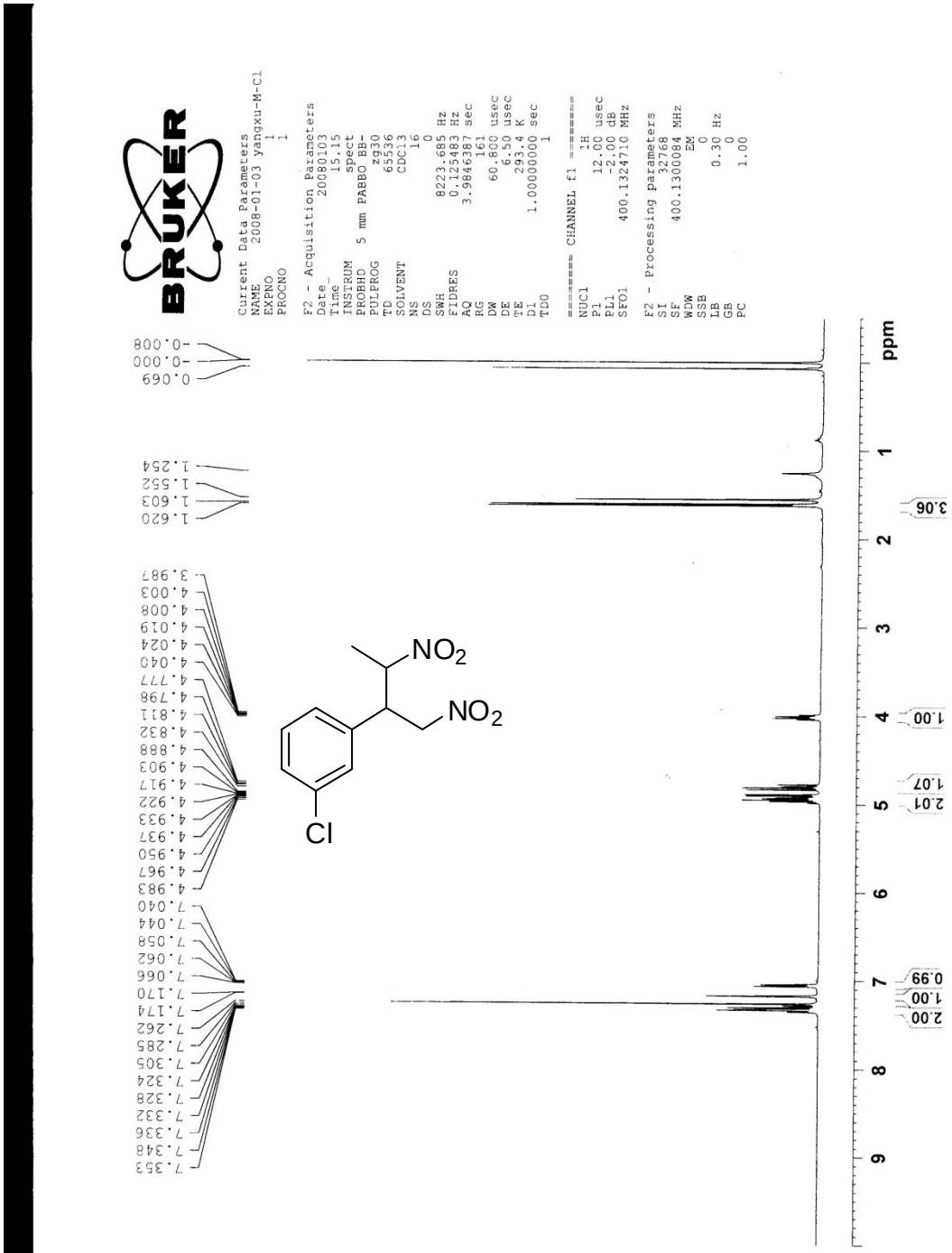

Current Data Parameters
NAME 2008-01-03 yangxu-M-Ome
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080103
Time_ 14.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300084 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NO2
NO2
OMe
ppm

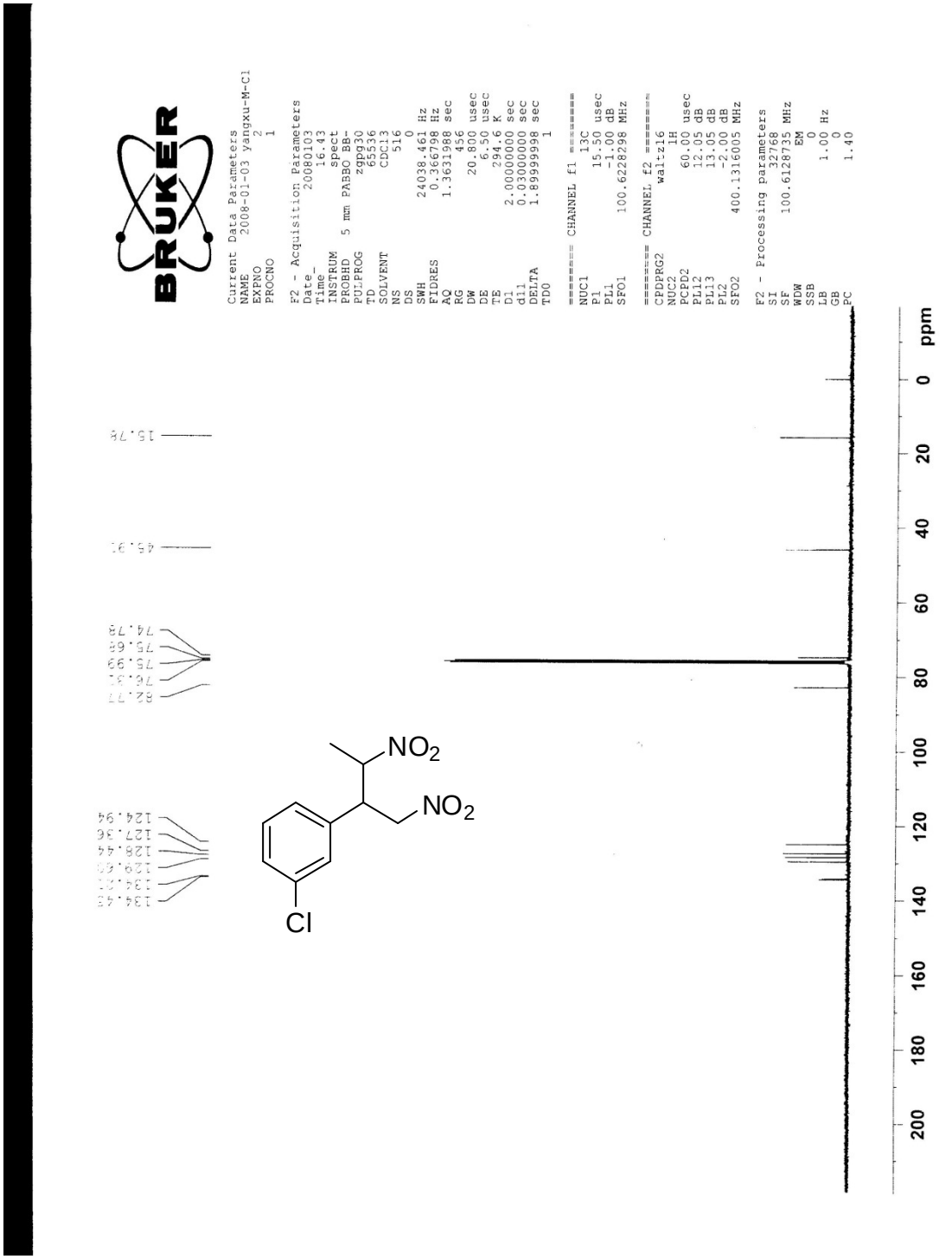

BRUKER
Current Data Parameters
NAME 2008-01-03 yangxu-M-Ome
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080103
Time 16.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 262
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 456
DW 20.800 usec
DE 6.50 usec
TE 294.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 12.05 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
16.71
47.28
55.29
76.00
76.32
77.04
77.36
83.99
114.04
114.29
119.92
130.40
134.98
160.05
NO2
NO2
OMe
200
180
160
140
120
100
80
60
40
20
0
ppm

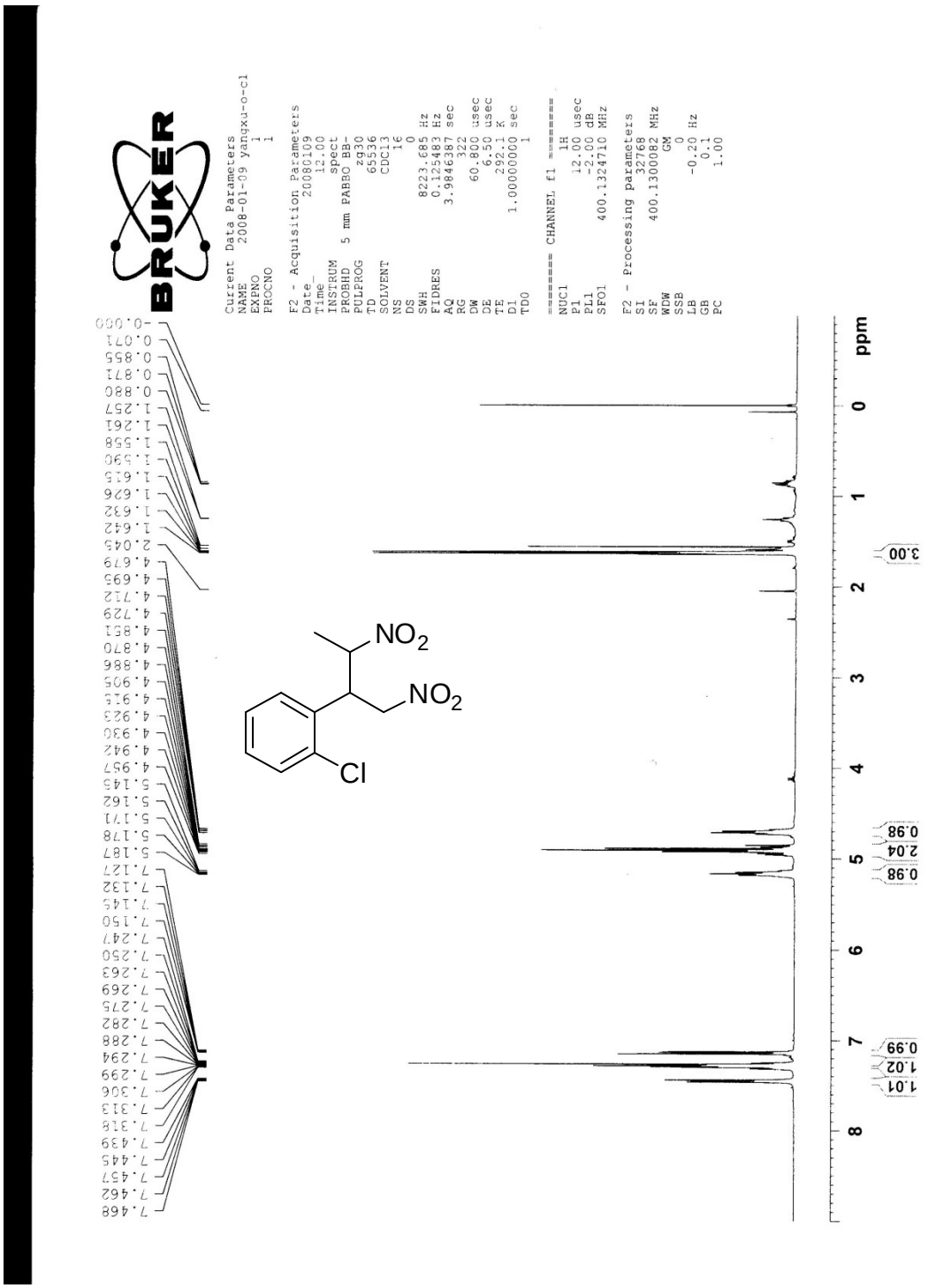

Current Data Parameters
NAME 2008-01-09 yangxu-ome
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080109
Time_ 10.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 456
DW 60.900 usec
DE 6.50 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300095 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
NO2
NO2
OMe
ppm
3.17
3.00
0.95
1.76
0.97
1.93
0.95
0.97

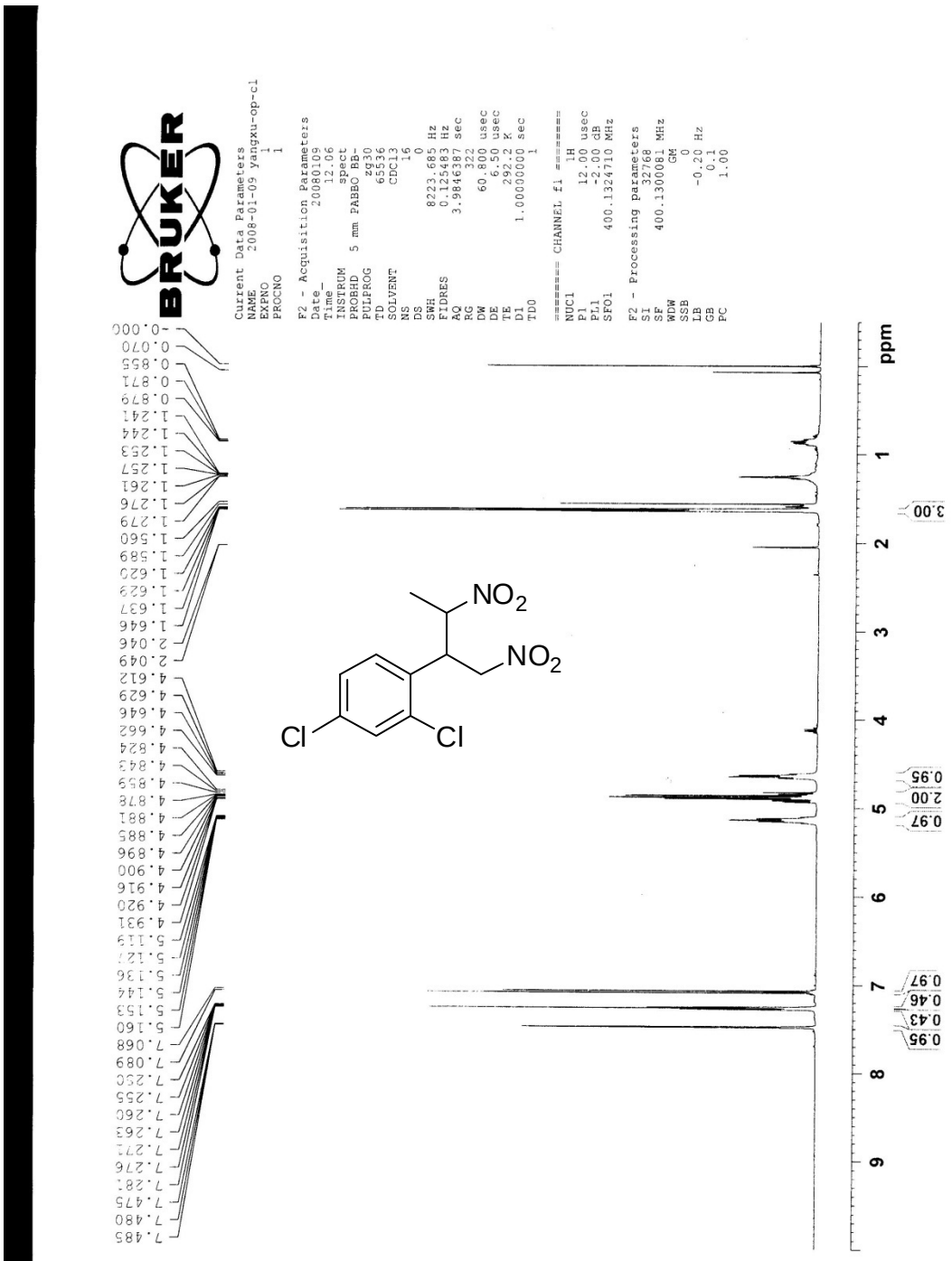

Current Data Parameters
NAME 2008-01-03 yangxu-P-Br
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080103
Time 15.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 293.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NO2
NO2
Br
ppm

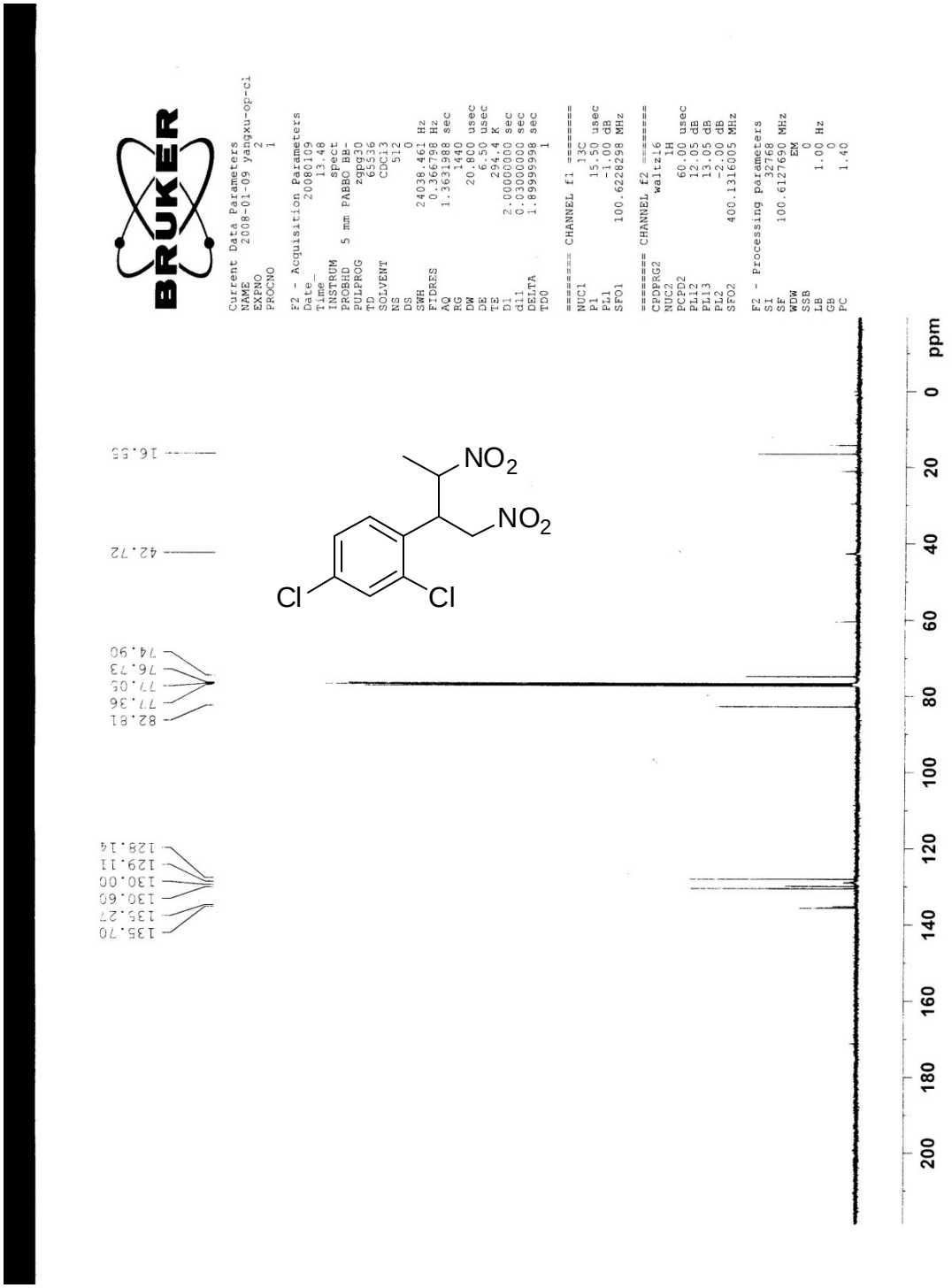

131.53
132.37
128.54
122.36
82.75
76.54
76.03
75.65
74.36
45.84
15.80
Br
NO2
NO2
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME 2008-01-03 yangxu-P-Br
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080103
Time 17.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 303
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 456
DW 20.800 usec
DE 6.50 usec
TE 294.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 12.05 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128733 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

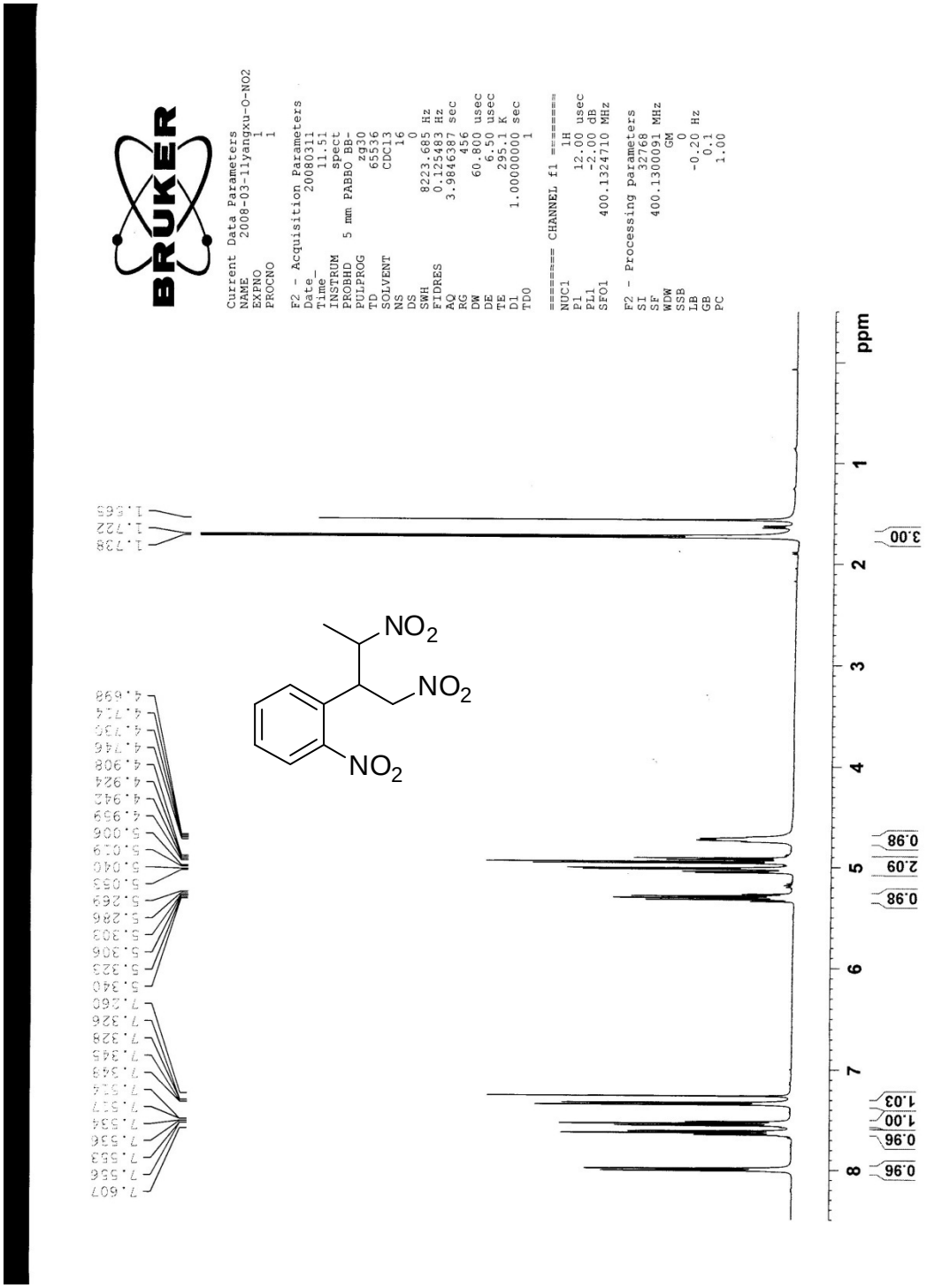

Current Data Parameters
NAME 2008-01-03 yangxu-M-Cl
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080103
Time_ 15.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300084 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NO2
NO2
Cl
ppm
3.06
1.00
1.07
2.01
0.99
1.00
2.00

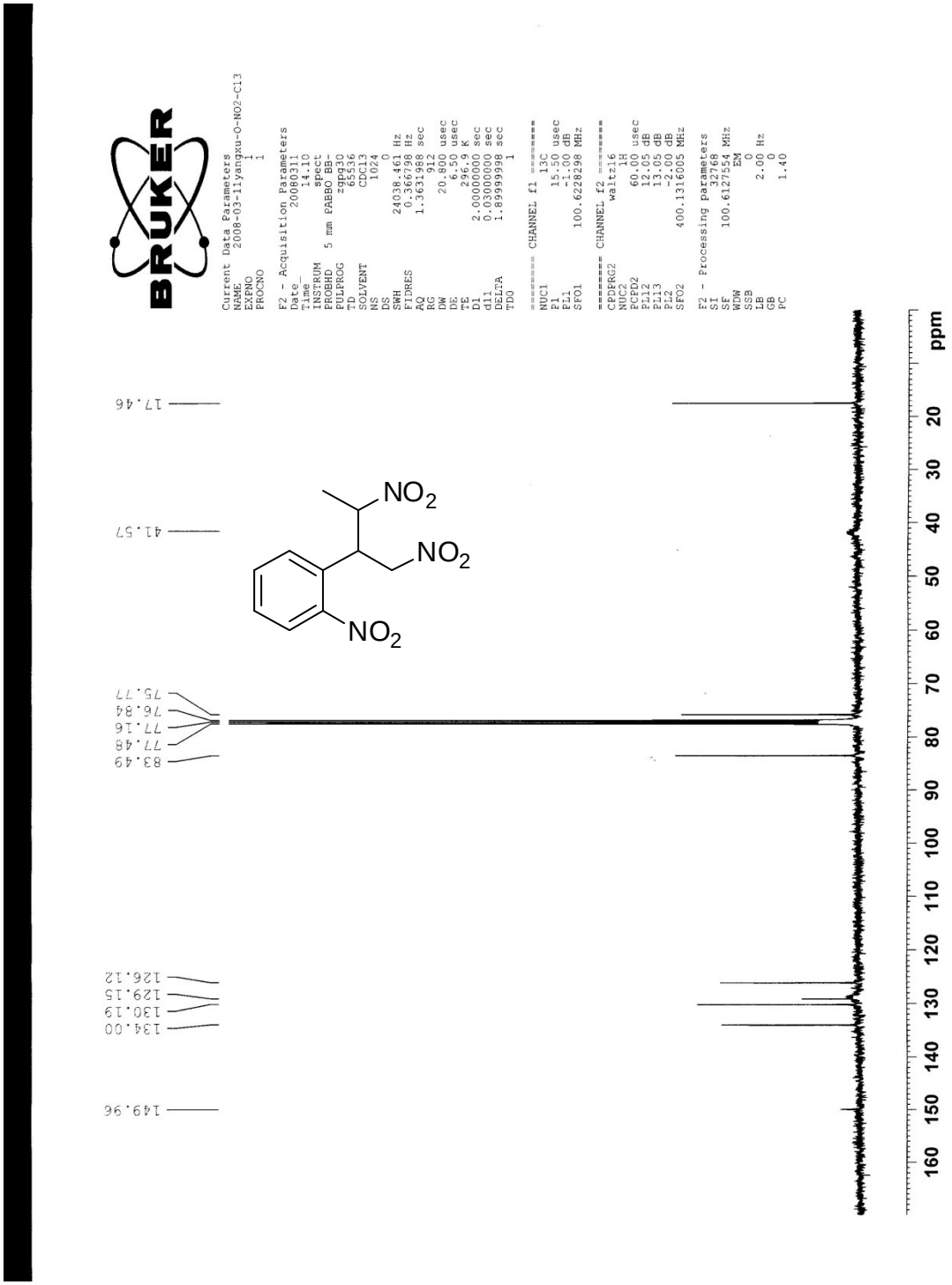

Current Data Parameters
NAME 2008-01-03 yangxu-M-Cl
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080103
Time 16.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 516
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 456
DW 20.800 usec
DE 6.50 usec
TE 294.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 12.05 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128735 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
15.78
45.32
74.78
75.68
75.89
76.51
82.77
124.94
127.36
128.44
129.60
134.01
134.45
NO2
NO2
Cl
200
180
160
140
120
100
80
60
40
20
0
ppm

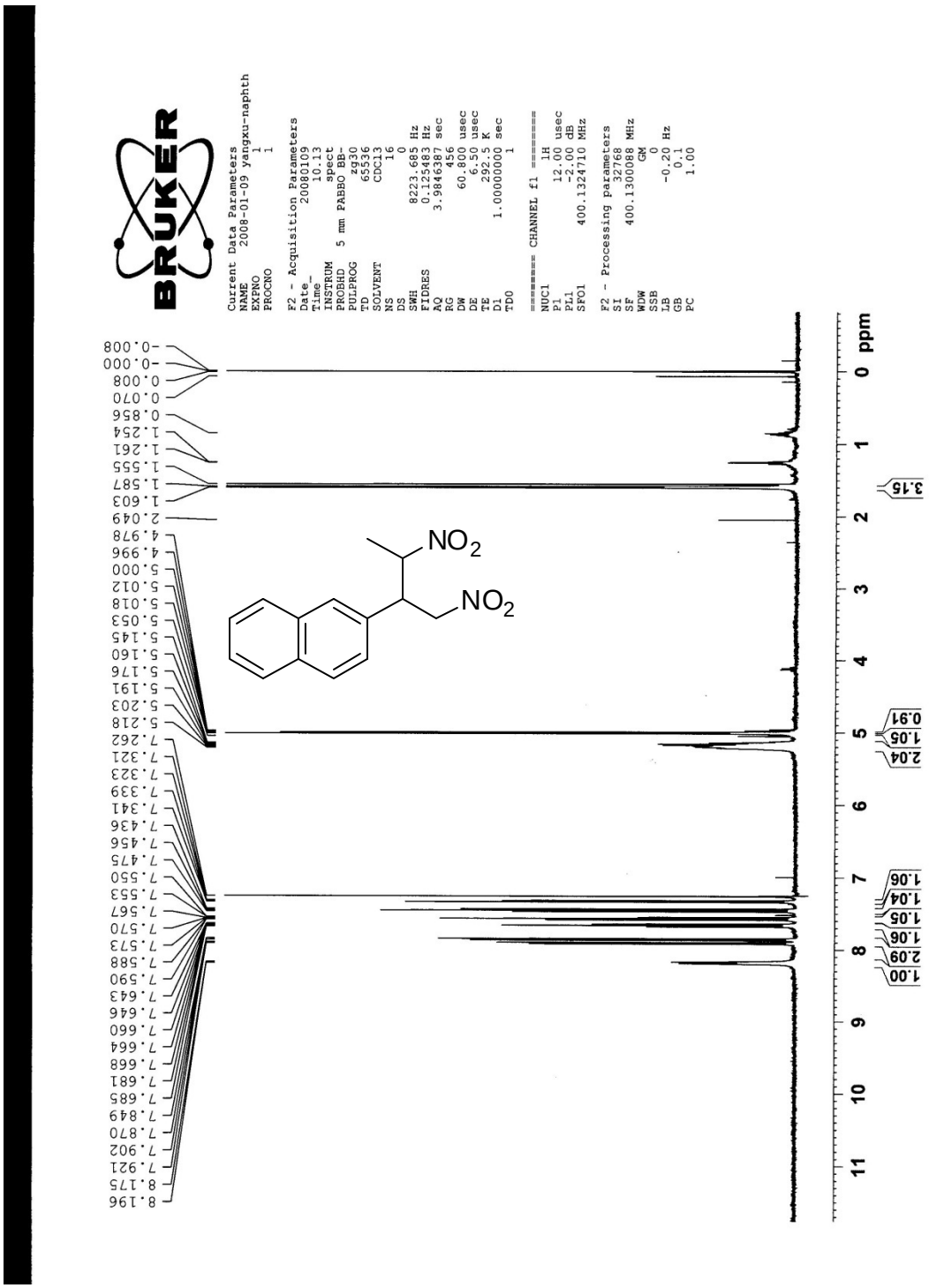

BRUKER
Current Data Parameters
NAME 2008-01-09 yangxu-o-cl
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080109
Time_ 12.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 322
DW 60.800 usec
DE 6.50 usec
TE 292.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300082 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
0.000
0.071
0.855
0.871
0.880
1.257
1.261
1.558
1.590
1.615
1.626
1.632
1.642
2.045
4.679
4.695
4.712
4.729
4.851
4.870
4.886
4.905
4.915
4.923
4.930
4.942
4.957
5.145
5.162
5.171
5.178
5.187
7.127
7.132
7.145
7.150
7.247
7.250
7.263
7.269
7.275
7.282
7.288
7.294
7.299
7.306
7.313
7.318
7.439
7.445
7.457
7.462
7.468
NO2
NO2
Cl
ppm
0 1 2 3 4 5 6 7 8
3.00
0.98
2.04
0.98
1.01
1.02
0.99

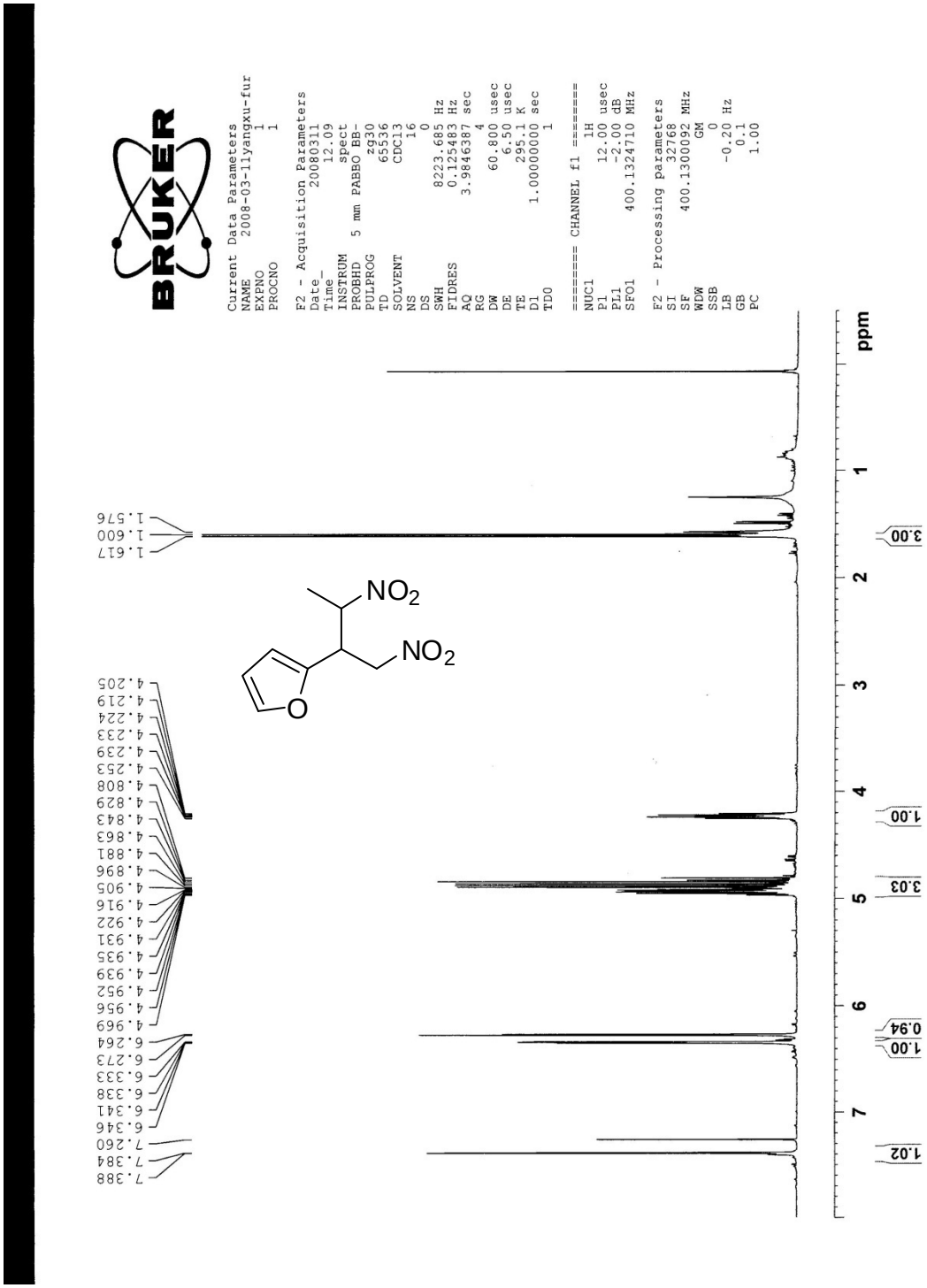

Current Data Parameters
NAME 2008-01-09 yangxu-op-cl
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080109
Time_ 12.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846397 sec
RG 322
DW 60.800 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300081 MHz
WDW EM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
NO2
NO2
Cl
Cl
ppm
3.00
0.97
2.00
0.95
0.97
0.46
0.43
0.95

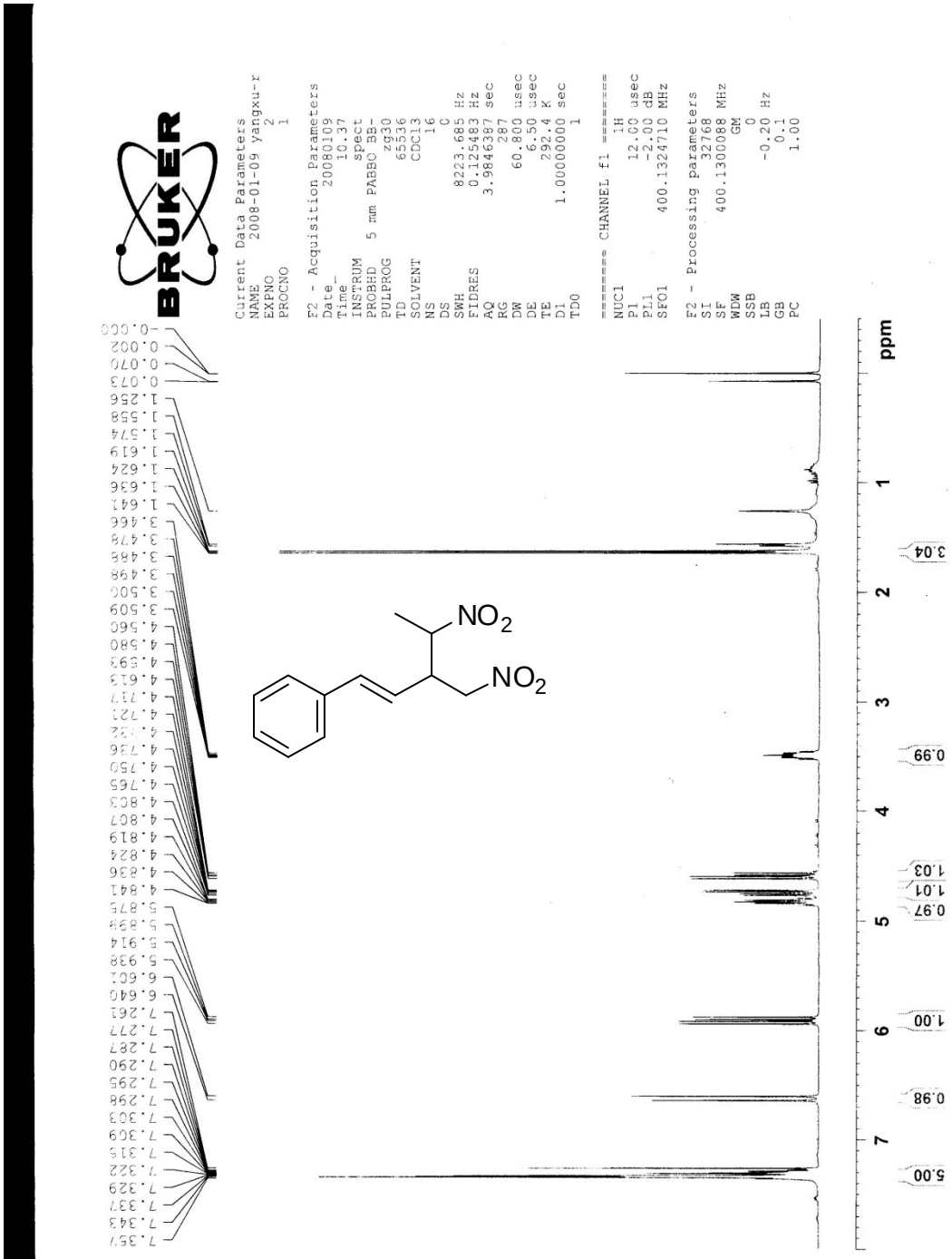

Current Data Parameters
NAME 2008-01-09 yangxu-op-ci
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080109
Time_ 13.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 1440
DW 20.800 usec
DE 6.50 usec
TE 294.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 12.05 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
16.55
42.72
74.90
76.73
77.05
77.36
82.81
128.14
129.11
130.00
130.60
135.27
135.70
NO2
NO2
Cl
Cl
200
180
160
140
120
100
80
60
40
20
0
ppm

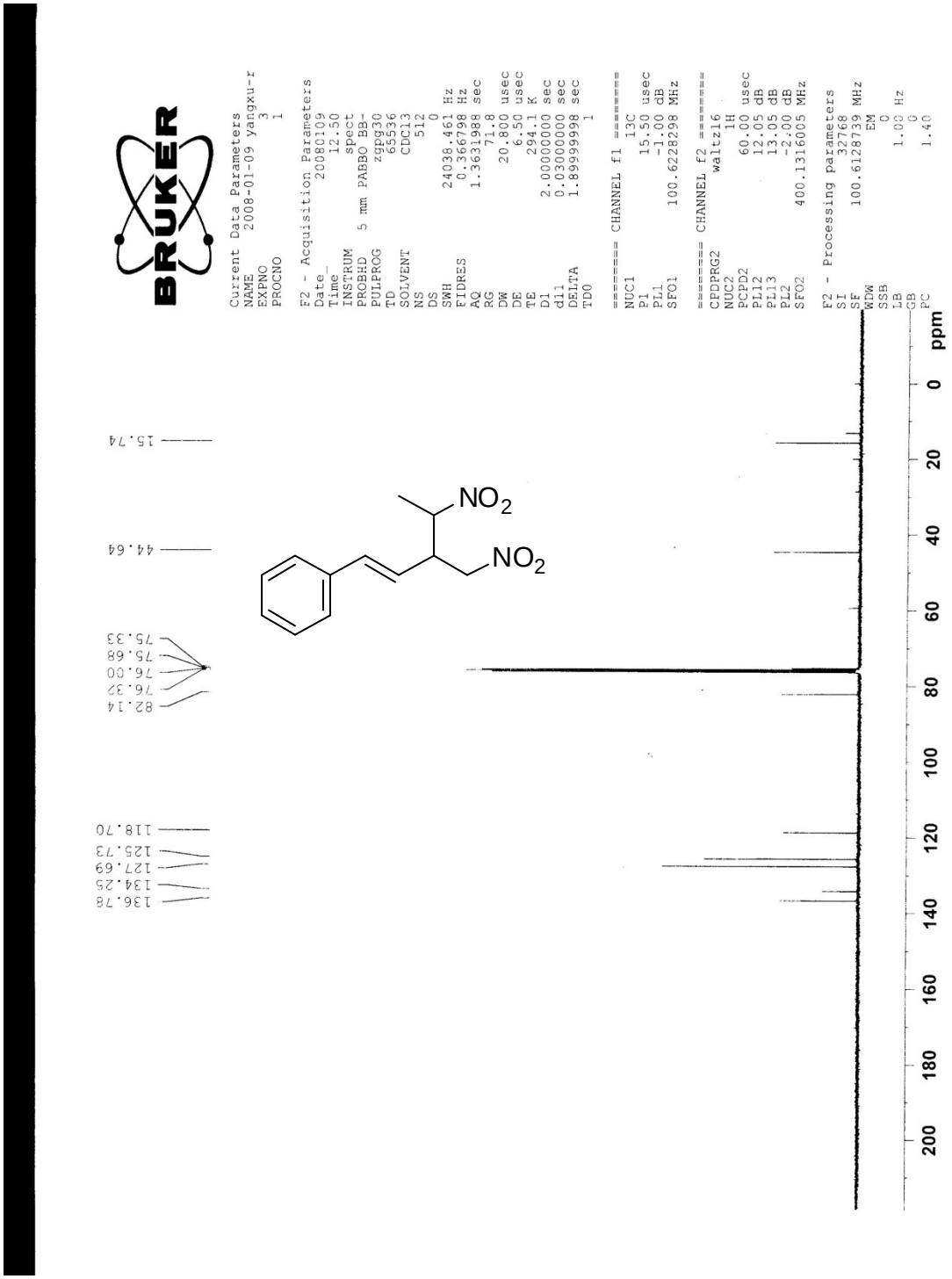

Current Data Parameters
NAME 2008-03-11yangxu-O-NO2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080311
Time_ 11.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 456
DW 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300091 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
NO2
NO2
NO2
1.738
1.722
1.685
4.698
4.714
4.730
4.746
4.908
4.924
4.942
4.969
5.006
5.019
5.040
5.053
5.269
5.286
5.303
5.306
5.323
5.340
7.260
7.326
7.328
7.345
7.349
7.514
7.517
7.534
7.536
7.553
7.556
7.607
ppm
3.00
0.98
2.09
0.98
1.03
1.00
0.96
0.96

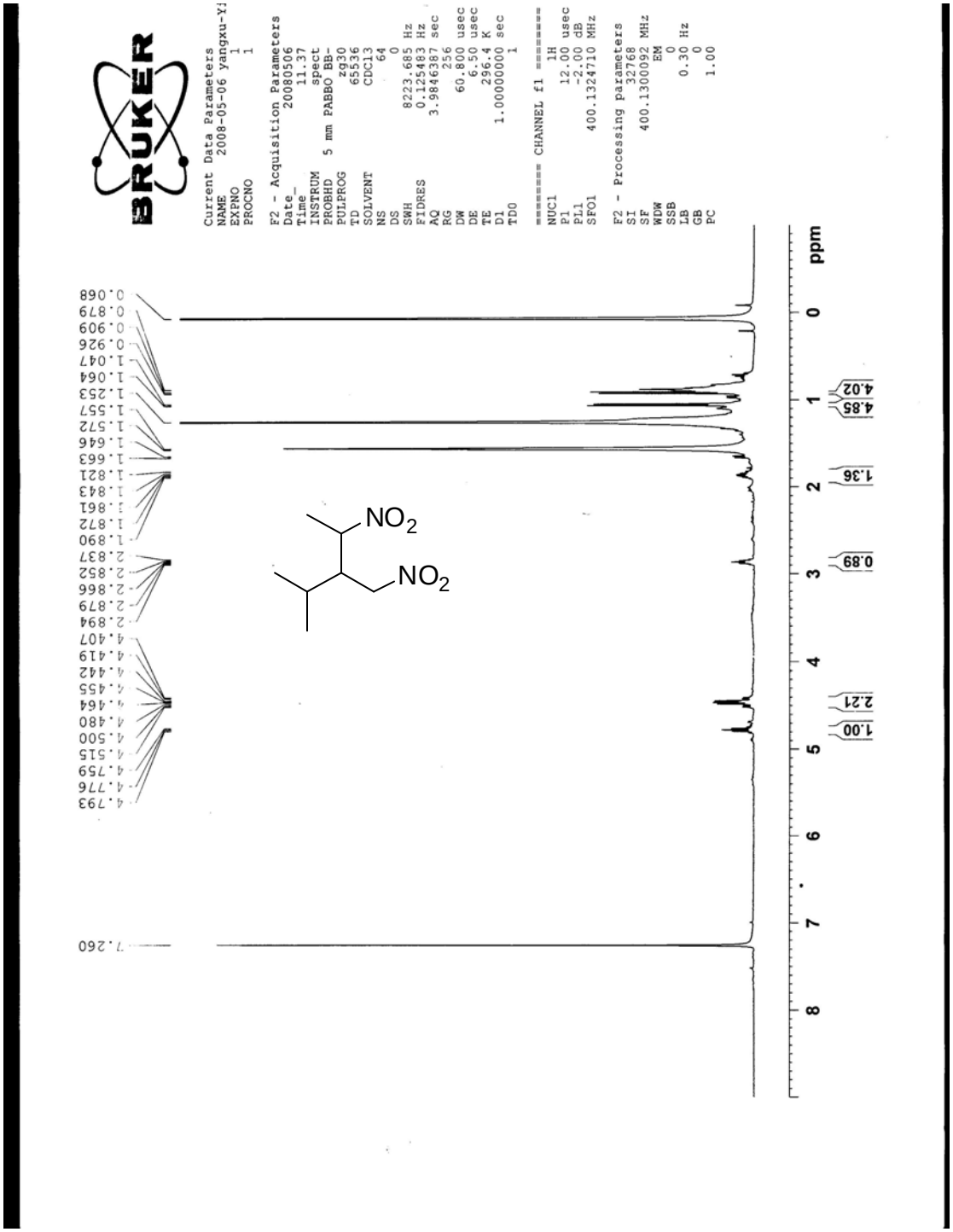

Current Data Parameters
NAME 2008-03-11yangxu-O-NO2-Cl3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080311
Time 14.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 912
DW 20.800 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 12.05 dB
PL13 13.05 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127554 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
17.46
41.57
75.77
76.84
77.16
77.48
83.49
126.12
129.15
130.19
134.00
149.96
NO2
NO2
NO2
ppm
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

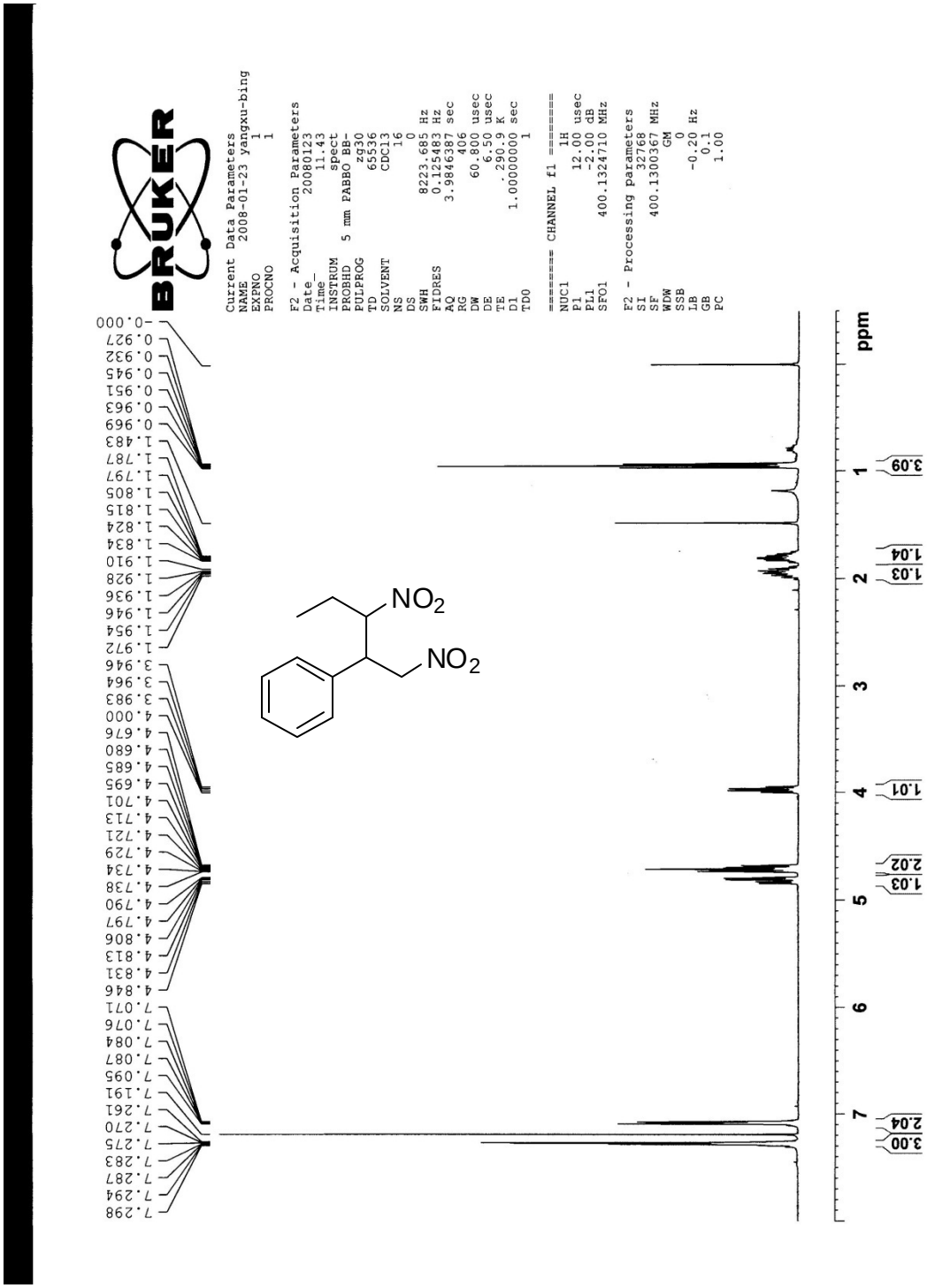

Current Data Parameters
NAME 2008-01-09 yangxu-naphth
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080109
Time_ 10.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 456
DW 60.800 usec
DE 6.50 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300088 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
NO2
NO2
ppm
3.15
0.91
1.05
2.04
1.06
1.04
1.05
1.06
2.09
1.00


Current Data Parameters
NAME 2008-03-11yangxu-fur
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080311
Time 12.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300092 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
NO2
NO2
O
1.576
1.600
1.617
4.205
4.219
4.224
4.233
4.239
4.253
4.808
4.829
4.843
4.863
4.881
4.896
4.905
4.916
4.922
4.931
4.935
4.939
4.952
4.956
4.969
6.264
6.273
6.333
6.338
6.341
6.346
7.260
7.384
7.388
3.00
1.00
3.03
1.00
0.94
1.02
ppm


BRUKER
Current Data Parameters
NAME 2008-01-09 yangxu-r
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080109
Time_ 10.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 287
DW 60.800 usec
DE 6.50 usec
TE 292.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300088 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
NO2
NO2
ppm
3.04
0.99
1.03
1.01
0.97
1.00
0.98
5.00


Current Data Parameters
NAME 2008-01-09 yangxu-r
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080109
Time_ 12.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 71.8
DW 20.800 usec
DE 6.50 usec
TE 294.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 12.05 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128739 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
15.74
44.64
75.33
75.68
76.00
76.37
82.14
118.70
125.73
127.69
134.25
136.78
NO2
NO2
200
180
160
140
120
100
80
60
40
20
0
ppm


Current Data Parameters
NAME 2008-05-06 yangxu-Yi
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080506
Time 11.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 64
DS 0
SWH 8223.685 Hz
FIDRES 0.125463 Hz
AQ 3.9846387 sec
RG 256
DW 60.800 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NO2
NO2
ppm
4.85
4.02
1.36
0.89
2.21
1.00


Current Data Parameters
NAME 2008-01-23 yangxu-bing
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080123
Time_ 11.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125403 Hz
AQ 3.9846387 sec
RG 406
DW 60.800 usec
DE 6.50 usec
TE 290.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300367 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
NO2
NO2
ppm
3.09
1.04
1.03
1.01
1.03
2.02
2.04
3.00