Supporting Information

Diastereoslective and Enantioselective Capture of Chiral Zinc Enolate Using Nitroolefins: A Rapid Acess to Chiral γ-Nitro Carbonyl Compounds

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General information: All non-aqueous reactions and manipulations were performed in an argon atmosphere. All solvents before use were dried and degassed by standard methods. All reactions were monitored by TLC with silica gel-coated plates.

NMR spectra were recorded on a Bruker AVANCE-400 spectrometer. Chemical shifts are reported in parts per million (ppm) down field from TMS with the solvent resonance as the internal standard. Coupling constants (J) are reported in Hz and refer to apparent peak multiplications. IR was recorded on a Nicolet 6700. Enantiomeric excess (ee) were determined by HPLC analysis on a Shimadzu LC-20A. The chiral ligands were prepared according to the known method. [1] All of the chalcones and nitrooelfins used here are known compounds and prepared according to the reported procedure. [2, 3]

Optimization of conditions:

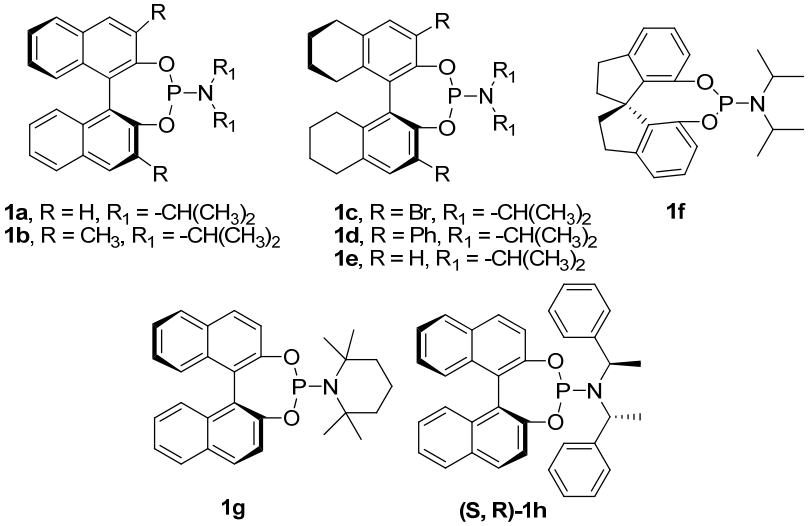
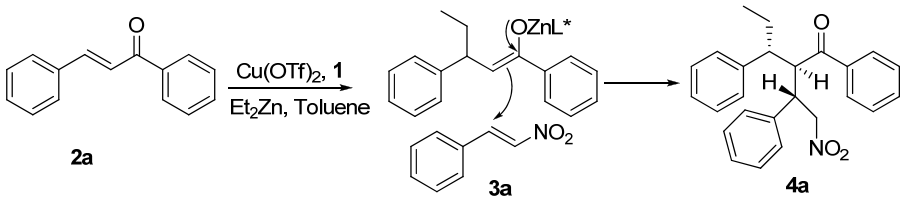


Figure 1 The Ligand investgated in the work.

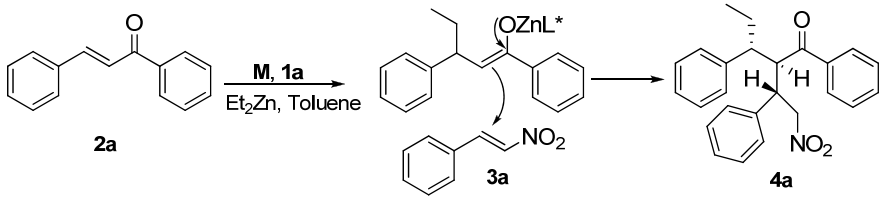
Table 1 Phosporamidites Ligands screening



Entry[a]	Ligand	Yield(%)[b]	Dr[c]	Ee(%)[d]
1	**1a**	79	>20:1	85
2	**1b**	29	>20:1	76
3	**1c**	68	>20:1	42
4	**1d**	66	>20:1	5.0
5	**1e**	63	>20:1	70
6	**1f**	59	>20:1	52
7	**1g**	34	>20:1	13
8	**S,R-1h**	62	>20:1	73
9	**R,R-1h**	60	>20:1	48

[a] Unless otherwise stated, 0.1 mmol of chalcone was exposed to 0.12 mmol of diethylzinc (1.0 M in hexane) in the presence of 2 mmol% $Cu(OTf)_2$ and 4 mmol% of chiral phosphoramidite using toluene as solvent; [b] Isolated yields; [c] The diastereoselectivity was determined by ^1H NMR analysis of crude product; [d] Enantioselecvities were determined by chiral HPLC.

Table 2 Lewis acids, reaction medium and temperature screening

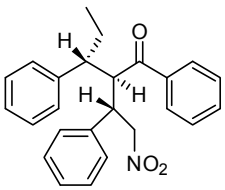


Entry[a]	Metal	Solvent	Yield(%)[b]	Dr[c]	Ee(%)[d]
1	$Cu(OTf)_2$	Toluene	79	>20:1	85
2	$(CuOTf)_2 \cdot$Toluene	Toluene	16	>20:1	76
3	CuCl	Toluene	29	>20:1	80
4	CuBr	Toluene	64	>20:1	88
5	CuI	Toluene	17	>20:1	17
6	$CuBr_2$	Toluene	24	>20:1	24
7	AgCl	Toluene	11	>20:1	0
8	$Pd(OAc)_2$	Toluene	<5	--	--
9	$CuCl_2$	Toluene	40	>20:1	83
10	$Cu(OAc)_2$	Toluene	46	>20:1	0
11	$Cu(OTf)_2$	THF	<5	--	--
12	$Cu(OTf)_2$	$CHCl_3$	<5	--	--
13	$Cu(OTf)_2$	CH_2Cl_2	32	>20:1	74
14	$Cu(OTf)_2$	Et_2O	21	>20:1	66
15[e]	$Cu(OTf)_2$	Toluene	66	>20:1	88

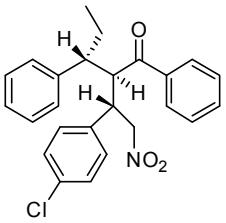
16[f]	$Cu(OTf)_2$	Toluene	57	>20:1	82
17[g]	$Cu(OTf)_2$	Toluene	71	>20:1	89
18[h]	$Cu(OTf)_2$	Toluene	72	>20:1	85

[a] Unless otherwise stated, the reaction was carried out the presence of 2 mmol% of Lewis acid and 4 mmol% of **1a** at -20 °C; [b] Isolated yields; [c] Drs were evaluated according the ^{1}H NMR analysis of crude product; [d] Ees were determined by chiral HPLC; [e] The reaction was carried out at -40 °C; [f] The reaction was carried out at -60 °C; [g] The reaction was carried out at -40 °C in the presence of 3 mmol% of $Cu(OTf)_2$ and 6 mmol% of **1a**. [h] The reaction was carried out at -40 °C in the presence of 4 mmol% of $Cu(OTf)_2$ and 8 mmol% of **1a**.

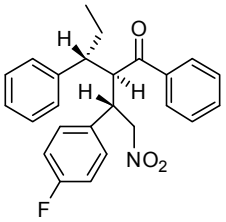
General procedure for the Copper-Catalyzed Enantioselective Tandem Conjugate Addition Reaction: Under an argon atmosphere, a solution of $Cu(OTf)_2$ (2.2 mg, 0.006 mmol) and the monodentate phosphoramidite **1a** (5.0 mg, 0.012 mmol) in toluene (1.0 mL) was stirred at room temperature for 1 h. The colorless solution was cooled (- 40 °C) and the chalcone (41.6 mg, 0.20 mmol) and the diethylzinc solution (1.2 equiv) in hexane (1.0 M, 0.24 mL, 0.24 mmol) were added. After 10 h at - 40 °C, trans-β-nitrostyrene (44.7 mg, 0.30 mmol, 1.5 equiv) in anhydrous toluene (1.0 mL) was added. After stirring the mixture for 12 h, sat. aq NH_4Cl (10 mL) was added to the reaction mixture, and then the reaction mixture was extracted with EtOAc (3 × 10 mL). The combined organic phase were washed with brine (3 × 5 mL) and dried over anhydrous Na_2SO_4. The solvent was then removed under vacuum. Purification by flash chromatography on silica gel (PE/EtOAc = 50:1V/V) afforded the corresponding addition product.



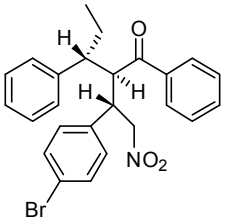
(**4**a) R_f= 0.26 (ethyl acetate/PE = 1:20), yield: 71%, white solid, mp: 99-100 °C, $[\alpha]_D^{25}$ = +28.4° (EtOAc, *c* 0.24). Single diastereoisomer (dr: >20:1), 89% *ee* determined by HPLC analysis (chiral AS-H column, 10% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 6.3 min, t (minor) = 7.4 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.59 (t, *J* = 7.2 Hz, 3H), 1.43-1.56 (m, 2H), 2.95 (td, *J* = 10.6, 10.6, 3.6 Hz, 1H), 3.62 (dt, *J* = 11.6, 4.4, 4.4 Hz, 1H), 4.14 (dd, *J* = 10.0, 4.8 Hz, 1H), 4.93 (dd, *J* = 13.6, 3.6 Hz, 1H), 5.04 (dd, *J* = 13.6, 11.6 Hz, 1H), 6.96-6.98 (m, 2H), 7.10-7.13 (m, 3H), 7.25-7.34 (m, 5H), 7.39-7.49 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.9, 140.8, 138.6, 138.1, 133.1, 129.0, 128.7, 128.3, 128.0, 127.9, 127.6, 127.3, 75.2, 56.0, 49.4, 44.0, 26.9 and 12.1. HRMS calcd for $C_{25}H_{26}NO_3$ [M+H]: 388.1913, found: 388.1907. IR (KBr): 3064, 3027, 2965, 2928, 1671, 1548, 1450, 1374, 759, 703 cm^{-1}.



(**4b**) R_f = 0.27 (ethyl acetate/PE = 1:20), yield: 76%, white solid, mp: 86-88°C, $[\alpha]_D^{25}$ = +30° (EtOAc, *c* 0.15). Single diastereoisomer (dr: >20:1), 91% *ee* determined by HPLC analysis (chiral AS-H column, 10% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 6.9 min, t (minor) = 10.1 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.59 (t, *J* = 7.2 Hz, 3H), 1.41-1.54 (m, 2H), 2.92 (td, *J* = 10.4, 10.4, 3.6 Hz, 1H), 3.58 (dt, *J* = 11.2, 4.4, 4.4 Hz, 1H), 4.10 (dd, *J* = 10.0, 4.8 Hz, 1H), 4.92 (dd, *J* = 14.0, 4.4 Hz, 1H), 4.99 (dd, *J* = 14.0, 11.6 Hz, 1H), 6.88-6.91 (m, 2H), 7.08-7.12 (m, 2H), 7.24-7.26 (m, 2H), 7.30-7.34 (m, 3H), 7.39-7.43 (m, 2H), 7.48-7.54 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.6, 140.6, 138.4, 136.7, 133.5, 133.4, 129.1, 128.9, 128.7, 128.5, 128.0, 127.9, 127.4, 75.1, 55.7, 49.4, 43.5, 27.0 and 12.1. HRMS calcd for $C_{25}H_{25}ClNO_3$ [M+H]: 422.1523, found: 422.1526. IR (KBr): 3065, 3031, 2966, 2925, 2856, 1673, 1550, 1448, 1383, 843, 756, 706 cm^{-1}.

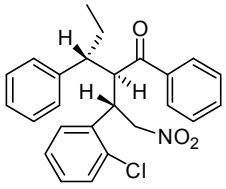


(**4c**) R_f = 0.25 (ethyl acetate/PE = 1:20), yield: 56%, white solid, mp: 124-126 °C, $[\alpha]_D^{25}$ = +15.9° (EtOAc, *c* 0.10). Single diastereoisomer (dr: >20:1), 86% *ee* determined by HPLC analysis (chiral AS-H column, 10% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 7.0 min, t (minor) = 9.5 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.59 (t, *J* = 7.2 Hz, 3H), 1.39-1.55 (m, 2H), 2.93 (td, *J* = 10.6, 10.6, 4.0 Hz, 1H), 3.59 (dt, *J* = 11.2, 4.4, 4.4 Hz, 1H), 4.11 (dd, *J* = 10.4 , 4.8 Hz, 1H), 4.92 (dd, *J* = 13.6, 4.0 Hz, 1H), 5.00 (dd, *J* = 13.6, 11.2 Hz, 1H), 6.79-6.84 (m, 2H), 6.92-6.95 (m, 2H), 7.25-7.34 (m, 5H), 7.40-7.53 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.7, 163.1, 160.7, 140.7, 138.4, 133.9, 133.8, 133.4, 129.1, 128.9, 128.9, 128.5, 128.0, 127.9, 127.4, 115.8, 115.6, 75.3, 55.9, 49.4, 43.4, 27.1 and 12.1. HRMS calcd for $C_{25}H_{25}FNO_3$ [M+H]: 406.1818, found: 406.1812. IR (KBr): 3062, 2965, 2929, 2874, 1668, 1552, 1448, 1379, 843, 733, 702 cm^{-1}.

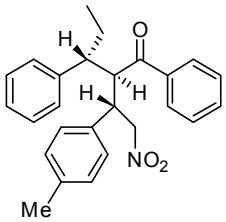


(**4d**) R_f = 0.24 (ethyl acetate/PE = 1:20), yield: 60%, white solid, mp: 68-70°C, $[\alpha]_D^{25}$ = +11.1° (EtOAc, *c* 0.22). Single diastereoisomer (dr: >20:1), 90% *ee* determined by HPLC analysis (chiral AS-H column, 10% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 7.2 min, t (minor) = 10.5 min. Recrystallization from a mixed solvent of ethyl acetate and petroleum afforded a single crystal (>99% *ee*) for X-ray analysis. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.59

(t, J = 7.2 Hz, 3H), 1.41-1.55 (m, 2H), 2.92 (td, J = 10.6, 10.6, 4.0 Hz, 1H), 3.57 (dt, J = 11.2, 4.4, 4.4 Hz, 1H), 4.10 (dd, J = 10.0, 4.8 Hz, 1H), 4.91 (dd, J = 13.6, 4.0 Hz, 1H), 4.98 (dd, J = 13.6, 11.2 Hz, 1H), 6.82-6.85 (m, 2H), 7.24-7.26 (m, 4H), 7.30-7.34 (m, 3H), 7.39-7.43 (m, 2H), 7.48-7.54 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.5, 140.7, 138.4, 137.2, 133.4, 131.9, 129.1, 129.0, 128.5, 128.0, 127.9, 127.4, 121.5, 75.1, 55.6, 49.4, 43.6, 27.0 and 12.1. HRMS calcd for $C_{25}H_{25}BrNO_3$ [M+H]: 466.1018, found: 466.1022. IR (KBr): 3064, 3029, 2963, 2924, 2855, 1672, 1551, 1450, 1381, 842, 742, 705 cm^{-1}.

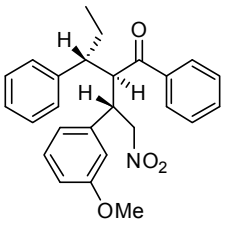


(**4e**) R_f = 0.28 (ethyl acetate/PE = 1:20), yield: 27%, colorless sticky oil, $[\alpha]_D^{25}$ = +21.7° (EtOAc, c 0.13). Major diastereoisomer (dr: 5:1), 89% *ee* determined by HPLC analysis (chiral AD-H column, 3% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 19.9 min, t (minor) = 41.3 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.60 (t, J = 7.6 Hz, 3H), 1.43-1.50 (m, 2H), 2.94-3.00 (m, 1H), 4.09 (dt, J = 12.0, 4.0, 4.0 Hz, 1H), 4.35 (dd, J = 10.4, 4.8 Hz, 1H), 4.96 (dd, J = 14.0, 3.2 Hz, 1H), 5.25 (dd, J =14.0, 12.0 Hz, 1H), 6.83-7.08 (m, 5H), 7.25-7.41 (m, 5H), 7.42-7.45 (m, 2H), 7.52-7.54 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.9, 139.9, 138.4, 134.9, 133.8, 133.2, 130.1, 128.8, 128.6, 128.4, 128.2, 127.8, 127.5, 127.3, 126.9, 73.6, 51.6, 49.2, 48.3, 24.9 and 12.0. HRMS calcd for $C_{25}H_{25}ClNO_3$ [M+H]: 422.1523, found: 422.1530. IR (KBr): 3063, 3029, 2966, 2929, 2873, 1670, 1553, 1446, 1380, 756, 703 cm^{-1}.

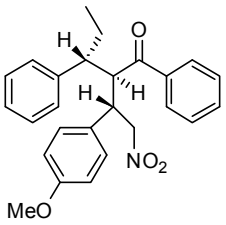


(**4f**) R_f = 0.31 (ethyl acetate/PE = 1:20), yield: 81%, white solid, mp: 97-99 °C, $[\alpha]_D^{25}$ = +23.5° (EtOAc, c 0.27). Single diastereoisomer (dr: >20:1), 88% *ee* determined by HPLC analysis (chiral AS-H column, 10% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 5.8 min, t (minor) = 7.3 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.59 (t, J = 7.2 Hz, 3H), 1.41-1.56 (m, 2H), 2.20 (s, 3H), 2.92 (td, J = 10.6, 10.6, 3.6 Hz, 1H), 3.60 (dt, J = 11.6, 4.4, 4.4 Hz, 1H), 4.12 (dd, J = 9.6, 4.8 Hz, 1H), 4.89 (dd, J = 13.6, 4.0 Hz, 1H), 4.98 (dd, J = 13.2, 11.2 Hz, 1H), 6.85-6.95 (m, 4H), 7.23-7.32 (m, 5H), 7.37-7.41 (m, 2H), 7.43-7.50 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 204.0, 140.9, 138.7, 137.2, 135.0, 133.1, 129.4, 129.0, 128.3, 128.1, 128.0, 127.2, 127.2, 75.6, 56.0, 49.3, 43.8, 26.6, 20.8 and 12.1. HRMS calcd for $C_{26}H_{28}NO_3$ [M+H]: 402.2069, found: 402.2062. IR (KBr): 3030, 2962, 1660, 1548, 1447, 1378, 834, 734, 700 cm^{-1}.

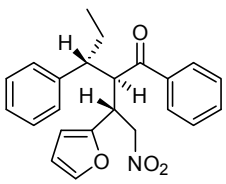
(**4g**) R_f = 0.17 (ethyl acetate/PE = 1:20), yield: 74%, white solid, mp: 60-62 °C, $[\alpha]_D^{25}$ = +30.1°



(EtOAc, *c* 0.17). Single diastereoisomer (dr: >20:1), 96% *ee* determined by HPLC analysis (chiral AS-H column, 3% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 13.7 min, t (minor) = 16.6 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.59 (t, *J* = 7.2 Hz, 3H), 1.41-1.53 (m, 2H), 2.95 (td, *J* = 10.6, 10.6, 3.6 Hz, 1H), 3.55-3.59 (m, 1H), 3.61 (s, 3H), 4.13 (dd, *J* = 10.0, 4.4 Hz, 1H), 4.91 (dd, *J* = 13.6, 4.0 Hz, 1H), 4.99 (dd, *J* = 13.6, 11.2 Hz, 1H), 6.47 (t, *J* = 2.0 Hz, 1H), 6.54-6.56 (m, 1H), 6.65 (2dd, *J* = 8.2, 2.8, 0.8 Hz, 1H), 7.04 (t, *J* = 7.6 Hz, 1H), 7.25-7.33 (m, 5H), 7.38-7.54 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.8, 159.7, 140.9, 139.7, 138.6, 133.2, 129.8, 129.0, 128.3, 128.1, 128.0, 127.3, 119.6, 113.2, 113.0, 75.2, 55.8, 55.0, 49.4, 44.1, 26.9 and 12.1. HRMS calcd for $C_{26}H_{28}NO_4$ [M+H]: 418.2018, found: 418.2014. IR (KBr): 3059, 3026, 2962, 2874, 1662, 1551, 1449, 1378, 873, 779, 731, 701 cm^{-1}.

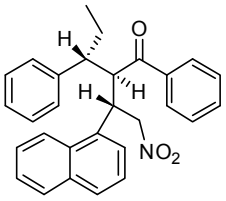


(**4h**) R_f = 0.16 (ethyl acetate/PE = 1:20), yield: 89%, white solid, mp: 63-65 °C, $[\alpha]_D^{25}$ = +25.9° (EtOAc, *c* 0.15). Single diastereoisomer (dr: >20:1), 90% *ee* determined by HPLC analysis (chiral AS-H column, 3% IPA in hexane, rate: 0.5 mL/min, 254 nm). Retention time: t (major) = 28.1min, t (minor) = 33.5 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.58 (t, *J* = 7.2 Hz, 3H), 1.41-1.56 (m, 2H), 2.91 (td, *J* = 10.6, 10.6, 3.2 Hz, 1H), 3.58 (dt, *J* = 11.2, 4.4, 4.4 Hz, 1H), 3.69 (s, 3H), 4.11 (dd, *J* = 10.0, 5.2 Hz, 1H), 4.88 (dd, *J* = 13.2, 4.0 Hz, 1H), 4.96 (dd, *J* = 13.2, 11.2 Hz, 1H), 6.64-6.68 (m, 2H), 6.87-6.90 (m, 2H), 7.23-7.25 (m, 2H), 7.29-7.32 (m, 3H), 7.37-7.52 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 204.0, 158.8, 140.9, 138.6, 133.1, 130.0, 129.0, 128.4, 128.3, 128.1, 128.0, 127.2, 114.1, 75.7, 56.1, 55.1, 49.3, 43.4, 26.7 and 12.1. HRMS calcd for $C_{26}H_{28}NO_4$ [M+H]: 418.2018, found: 418.2013. IR (KBr): 3027, 2965, 2927, 2872, 1669, 1552, 1449, 1379, 811, 757, 702 cm^{-1}.

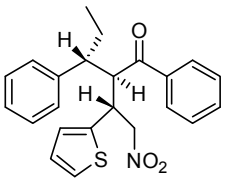


(**4i**) R_f = 0.30 (ethyl acetate/PE = 1:20), yield: 64%, colorless sticky oil, $[\alpha]_D^{25}$ = +65.3° (EtOAc, *c* 0.21). Single diastereoisomer (dr: >20:1), 92% *ee* determined by HPLC analysis (chiral OD-H column, 5% IPA in hexane, rate: 0.5 mL/min, 254 nm). Retention time: t (major) = 26.4 min, t (minor) = 38.0 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.62 (t, *J* = 7.2 Hz, 3H), 1.45-1.55 (m, 2H), 2.94 (td, *J* = 10.4, 10.4, 4.0 Hz, 1H), 3.71 (dt, *J* = 10.8, 4.0, 4.0 Hz, 1H), 4.34 (dd, *J* = 10.4, 4.8 Hz, 1H), 4.81 (dd, *J* = 13.6, 10.8 Hz, 1H), 4.88 (dd, *J* = 13.2, 4.0 Hz, 1H), 5.94 (dt, *J* = 3.2, 0.4, 0.4 Hz, 1H), 6.06 (dd, *J* = 3.2, 1.6 Hz, 1H), 7.12 (dd, *J* = 2.0, 0.8 Hz, 1H), 7.26-7.33 (m, 3H), 7.35-7.54 (m,

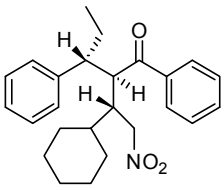
5H), 7.66-7.69 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.2, 151.3, 141.8, 140.4, 138.1, 133.2, 129.0, 128.4, 128.0, 127.9, 127.3, 110.3, 107.1, 74.2, 53.2, 48.8, 37.9, 27.2 and 12.0. HRMS calcd for $C_{23}H_{24}NO_4$ [M+H]: 378.1705, found: 378.1711. IR (KBr): 3062, 2961, 2928, 2871, 1670, 1551, 1449, 1383, 735, 703 cm^{-1}.



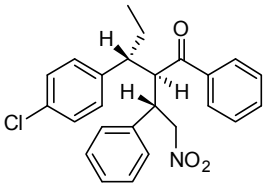
(**4j**) R_f = 0.24 (ethyl acetate/PE = 1:20), yield: 42%, white solid, mp: 96-98 °C, $[\alpha]_D^{25}$ = +19.4° (EtOAc, *c* 0.12). Single diastereoisomer (dr: >20:1), 90% *ee* determined by HPLC analysis (chiral OD-H column, 5% IPA in hexane, rate: 0.5 mL/min, 254 nm). Retention time: t (major) = 23.3 min, t (minor) = 39.4 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.61 (t, *J* = 7.2 Hz, 3H), 1.38-1.50 (m, 2H), 3.10 (td, *J* = 10.8, 10.8, 4.0 Hz, 1H), 4.20 (dd, *J* = 10.8, 4.0 Hz, 1H), 4.44 (dt, *J* = 11.6, 3.6, 3.6 Hz, 1H), 5.24 (dd, *J* = 14.0, 3.6 Hz, 1H), 5.38 (dd, *J* = 14.0, 11.6 Hz, 1H), 6.98-7.04 (m, 2H), 7.07-7.11 (m, 2H), 7.17-7.19 (m, 2H), 7.32-7.37 (m, 1H), 7.45-7.59 (m, 9H), 7.79-7.81 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 204.1, 140.8, 138.5, 134.1, 133.5, 133.0, 130.9, 129.2, 129.1, 128.2, 128.0, 127.7, 126.5, 125.7, 124.7, 123.3, 122.2, 74.5, 54.2, 49.5, 37.8, 27.5 and 12.1. HRMS calcd for $C_{29}H_{28}NO_3$ [M+H]: 438.2069, found: 438.2073. IR (KBr): 3059, 2964, 2930, 1672, 1551, 1450, 1375, 779, 753, 704 cm^{-1}.



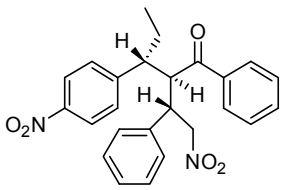
(**4k**) R_f = 0.24 (ethyl acetate/PE = 1:20), yield: 68%, colorless sticky oil, $[\alpha]_D^{25}$ = +18.7° (EtOAc, *c* 0.20). Single diastereoisomer (dr: >20:1), 93% *ee* determined by HPLC analysis (chiral OD-H column, 3% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 18.5 min, t (minor) = 46.0 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.60 (t, *J* = 7.2 Hz, 3H), 1.46-1.55 (m, 2H), 2.93 (td, *J* = 10.4, 10.4, 4.0 Hz, 1H), 3.92 (dt, *J* = 8.4, 5.6, 5.6 Hz, 1H), 4.23 (dd, *J* = 9.6, 5.2 Hz, 1H), 4.92 (d, *J* = 2.4 Hz, 1H), 4.94 (s, 1H), 6.66 (d, *J* = 3.6 Hz, 1H), 6.72 (dd, *J* = 5.2, 3.6 Hz, 1H), 7.04 (dd, *J* = 5.2, 1.2 Hz, 1H), 7.24-7.26 (m, 2H), 7.28-7.41 (m, 5H), 7.47-7.52 (m, 1H), 7.60-7.62 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.6, 141.0, 140.5, 138.6, 133.3, 129.1, 128.4, 128.0, 127.9, 127.4, 126.9, 125.3, 124.4, 76.5, 56.3, 49.2, 39.4, 26.8 and 12.0. HRMS calcd for $C_{23}H_{24}NO_3S$ [M+H]: 394.1477, found: 394.1471. IR (KBr): 2958, 2927, 2869, 1667, 1551, 1448, 1384, 746, 700 cm^{-1}.



(**4l**) R_f = 0.46 (ethyl acetate/PE = 1:20), yield: 25%, colorless sticky oil, $[\alpha]_D^{25}$ = +15.4° (EtOAc, *c* 0.28). Single diastereoisomer (dr: >20:1), 79% *ee* determined by HPLC analysis (chiral OD-H column, 1% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 8.2 min, t (minor) = 11.4 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.58 (t, *J* = 7.2 Hz, 3H), 0.87-0.99 (m, 5H), 1.06-1.12 (m, 1H), 1.24-1.26 (m, 1H), 1.46-1.57 (m, 5H), 1.62-1.65 (m, 1H), 2.21-2.27 (m, 1H), 2.87 (td, *J* = 10.6, 10.6, 4.0 Hz, 1H), 4.18 (dd, *J* = 10.4, 2.8 Hz, 1H), 4.54 (dd, *J* = 14.0, 10.0 Hz, 1H), 4.97 (dd, *J* = 14.0, 2.0 Hz, 1H), 7.24-7.28 (m, 2H), 7.29-7.32 (m, 1H), 7.37-7.40 (m, 2H), 7.54-7.58 (m, 2H), 7.63- 7.67 (m, 1H), 8.05-8.07 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 204.4, 141.2, 138.7, 133.5, 128.9, 128.8, 128.2, 128.1, 127.1, 75.1, 50.2, 48.9, 43.3, 40.1, 30.2, 30.0, 28.0, 26.2, 26.2, 26.0 and 12.4. HRMS calcd for $C_{25}H_{32}NO_3$ [M+H]: 394.2382, found: 394.2375. IR (KBr): 3061, 3028, 2928, 2855, 1672, 1550, 1449, 1380, 751, 702 cm^{-1}.

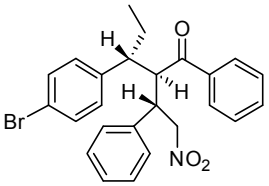


(**4m**) R_f = 0.27 (ethyl acetate/PE = 1:20), yield: 61%, white solid, mp: 34-36 °C, $[\alpha]_D^{25}$ = +16.9° (EtOAc, *c* 0.16). Single diastereoisomer (dr: >20:1), 94% *ee* determined by HPLC analysis (chiral AD-H column, 5% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 12.1 min, t (minor) = 25.6 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.58 (t, *J* = 7.2 Hz, 3H), 1.36-1.56 (m, 2H), 2.92 (td, *J* = 10.6, 10.6, 3.2 Hz, 1H), 3.62 (dt, *J* = 12.0, 4.0, 4.0 Hz, 1H), 4.09 (dd, *J* = 10.0, 5.2 Hz, 1H), 4.87 (dd, *J* = 13.6, 3.6 Hz, 1H), 4.99 (dd, *J* = 13.2, 11.2 Hz, 1H), 6.96-6.99 (m, 2H), 7.12-7.19 (m, 5H), 7.26-7.30 (m, 2H), 7.35-7.38 (m, 2H), 7.44-7.49 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.4, 139.5, 138.4, 137.8, 133.3, 132.9, 129.2, 129.2, 128.8, 128.4, 128.1, 127.7, 127.3, 75.4, 55.7, 48.7, 44.1, 26.6 and 12.0. HRMS calcd for $C_{25}H_{25}ClNO_3$ [M+H]: 422.1523, found: 422.1532. IR (KBr): 2964, 2929, 2873, 1670, 1556, 1451, 1381, 846, 758, 704 cm^{-1}.

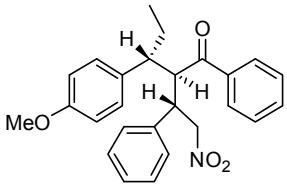


(**4n**) R_f = 0.12 (ethyl acetate/PE = 1:20), yield: 72%, yellow solid, mp: 101-103 °C, $[\alpha]_D^{25}$ = +18.6° (EtOAc, *c* 0.20). Major diastereoisomer (dr: >20:1), 90% *ee* determined by HPLC analysis (chiral AD-H column, 20% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 11.2 min, t (minor) = 21.6 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.58 (t, *J* = 7.2 Hz, 3H), 1.46-1.53 (m, 1H), 1.61-1.69 (m, 1H), 3.03-3.09 (m, 1H), 3.60-3.65 (m, 1H), 4.17 (dd,

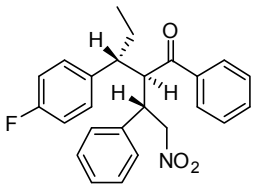
J = 8.8, 5.6 Hz, 1H), 4.83 (dd, *J* = 13.2, 4.0 Hz, 1H), 4.92 (dd, *J* = 13.2, 10.8 Hz, 1H), 6.99-7.01 (m, 2H), 7.16-7.19 (m, 3H), 7.29-7.38 (m, 4H), 7.47-7.52 (m, 3H), 8.20-8.24 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 202.5, 149.0, 147.0, 138.2, 137.4, 133.6, 129.0, 128.9, 128.5, 128.1, 128.0, 127.3, 124.1, 75.8, 55.1, 49.1, 44.4, 25.9 and 11.9. HRMS calcd for $C_{25}H_{25}N_2O_5$ [M+H]: 433.1763, found: 433.1761.IR (KBr): 2972, 2930, 1666, 1560, 1454, 1383, 754, 703 cm^{-1}.



(**4o**) R_f = 0.20 (ethyl acetate/PE = 1:20), yield: 66%, white solid, mp: 55-56 °C, $[\alpha]_D^{25}$ = +35.6° (EtOAc, *c* 0.11). Single diastereoisomer (dr: >20:1), 95% *ee* determined by HPLC analysis (chiral AD-H column, 5% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 12.6 min, t (minor) = 24.8 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.57 (t, *J* = 7.2 Hz, 3H), 1.35-1.54 (m, 2H), 2.91 (td, *J* = 10.6, 10.6, 3.2 Hz, 1H), 3.62 (dt, *J* = 11.6, 4.0, 4.0 Hz, 1H), 4.09 (dd, *J* = 9.6, 5.2 Hz, 1H), 4.87 (dd, *J* = 13.2, 3.6 Hz, 1H), 4.98 (dd, *J* = 13.6, 11.6 Hz, 1H), 6.96-6.99 (m, 2H), 7.11-7.15 (m, 5H), 7.26-7.30 (m, 2H), 7.45-7.53 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.4, 140.0, 138.4, 137.8, 133.3, 132.1, 129.6, 128.8, 128.4, 128.1, 127.7, 127.3, 121.0, 75.4, 55.7, 48.8, 44.1, 26.5 and 12.0. HRMS calcd for $C_{25}H_{25}BrNO_3$ [M+H]: 466.1018, found: 466.1015. IR (KBr): 2967, 2927, 1671, 1554, 1451, 1382, 844, 756, 703 cm^{-1}.

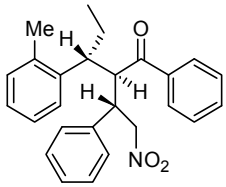


(**4p**) R_f = 0.15 (ethyl acetate/PE = 1:20), yield: 76%, white solid, mp: 46-48 °C, $[\alpha]_D^{25}$ = +21.4° (EtOAc, *c* 0.17). Single diastereoisomer (dr: >20:1), 80% *ee* determined by HPLC analysis (chiral AD-H column, 5% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 13.5 min, t (minor) = 25.9 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.58 (t, *J* = 7.2 Hz, 3H), 1.35-1.53 (m, 2H), 2.89 (td, *J* = 10.6, 10.6, 3.2 Hz, 1H), 3.62 (dt, *J* = 11.6, 4.0, 4.0 Hz, 1H), 3.85 (s, 3H), 4.07 (dd, *J* = 10.0, 4.4 Hz, 1H), 4.93 (dd, *J* = 13.6, 3.6 Hz, 1H), 5.00-5.07 (m, 1H), 6.93-6.98 (m, 4H), 7.09-7.19 (m, 5H), 7.25-7.28 (m, 2H), 7.43-7.48 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 204.1, 158.6, 138.7, 138.2, 133.1, 132.7, 128.8, 128.7, 128.3, 128.0, 127.5, 127.3, 114.4, 75.2, 56.3, 55.2, 48.6, 44.0, 27.1 and 12.1. HRMS calcd for $C_{26}H_{28}NO_4$ [M+H]: 418.2018, found: 418.2023. IR (KBr): 2963, 2925, 1667, 1551, 1455, 1381, 843, 758, 698 cm^{-1}.

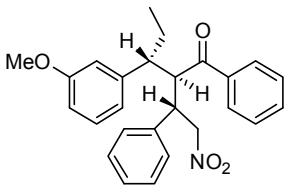


(**4q**) R_f = 0.26 (ethyl acetate/PE = 1:20), yield: 68%, white solid, mp:

88-90 °C, $[\alpha]_D^{25}$ = +14.7° (EtOAc, *c* 0.31). Single diastereoisomer (dr: >20:1), 90% *ee* determined by HPLC analysis (chiral AD-H column, 10% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 9.0 min, t (minor) = 19.6 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.58 (t, *J* = 7.2 Hz, 3H), 1.36-1.56 (m, 2H), 2.93 (td, *J* = 10.6, 10.6, 3.2 Hz, 1H), 3.62 (dt, *J* = 11.6, 4.4, 4.4 Hz, 1H), 4.08 (dd, *J* = 9.6, 5.2 Hz, 1H), 4.88 (dd, *J* = 13.2, 3.6 Hz, 1H), 4.99 (dd, *J* = 13.2, 11.2 Hz, 1H), 6.97-6.99 (m, 2H), 7.06-7.15 (m, 5H), 7.19-7.25 (m, 2H), 7.27-7.29 (m, 2H), 7.46-7.49 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.6, 163.0, 160.6, 138.5, 137.9, 136.6, 136.6, 133.3, 129.4, 129.3, 128.8, 128.4, 128.0, 127.7, 127.3, 116.0, 115.8, 75.4, 55.9, 48.6, 44.1, 26.7 and 12.0. HRMS calcd for $C_{25}H_{25}FNO_3$ [M+H]: 406.1818, found: 406.1813. IR (KBr): 2969, 2928, 1670, 1554, 1451, 1382, 832, 754, 699 cm^{-1}.

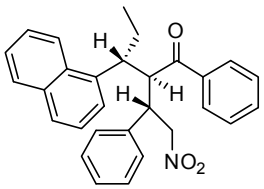


(**4r**) R_f = 0.24 (ethyl acetate/PE = 1:20), yield: 78%, colorless sticky oil, $[\alpha]_D^{25}$ = +9.5° (EtOAc, *c* 0.24). Single diastereoisomer (dr: >20:1), 78% *ee* determined by HPLC analysis (chiral AS-H column, 5% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 6.6 min, t (minor) = 10.5 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.58 (t, *J* = 7.6 Hz, 3H), 1.44-1.54 (m, 1H), 1.62-1.68 (m, 1H), 2.37 (s, 3H), 3.26-3.32 (m, 1H), 3.72 (dt, *J* = 11.6, 4.4, 4.4 Hz, 1H), 4.14 (dd, *J* = 9.2, 5.2 Hz, 1H), 4.81 (dd, *J* = 13.6, 3.6 Hz, 1H), 4.90-4.96 (m, 1H), 6.99-7.01 (m, 2H), 7.15-7.19 (m, 5H), 7.23-7.29 (m, 4H), 7.43-7.49(m, 3H), ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.8, 139.2, 138.6, 138.1, 136.9, 133.2, 130.9, 128.8, 128.3, 128.1, 127.7, 127.3, 126.6, 126.5, 125.8, 76.1, 55.4, 44.2, 43.2, 26.9, 19.8 and 11.5. HRMS calcd for $C_{26}H_{28}NO_3$ [M+H]: 402.2069, found: 402.2067. IR (KBr): 3062, 3027, 2965, 2929, 2874, 1669, 1553, 1448, 1380, 760, 700 cm^{-1}.

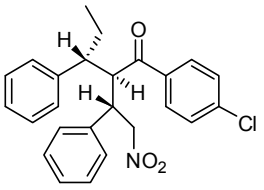


(**4s**) R_f = 0.18 (ethyl acetate/PE = 1:20), yield: 46%, white solid, mp: 79-81 °C, $[\alpha]_D^{25}$ = +12.3° (EtOAc, *c* 0.31). Single diastereoisomer (dr: >20:1), 89% *ee* determined by HPLC analysis (chiral AD-H column, 10% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 10.4 min, t (minor) = 22.2 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.60 (t, *J* = 7.2 Hz, 3H), 1.38-1.55 (m, 2H), 2.91 (td, *J* = 10.6, 10.6, 3.6 Hz, 1H), 3.65 (dt, *J* = 11.6, 4.0, 4.0 Hz, 1H), 3.84 (s, 3H), 4.10 (dd, *J* = 10.0, 4.8 Hz, 1H), 4.94 (dd, *J* = 13.6, 4.0 Hz, 1H), 5.04 (dd, *J* = 13.6, 11.6 Hz, 1H), 6.79-6.99 (m, 5H), 7.09-7.13 (m, 3H), 7.24-7.28 (m, 2H), 7.33 (t, *J* = 7.6 Hz, 1H), 7.43-7.49 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.9, 160.0, 142.6, 138.6, 138.1,

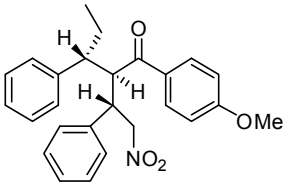
133.1, 130.0, 128.8, 128.3, 128.0, 127.6, 127.3, 120.3, 114.1, 112.0, 75.2, 56.0, 55.2, 49.4, 44.0, 26.9 and 12.1. HRMS calcd for $C_{26}H_{28}NO_4$ [M+H]: 418.2018, found: 418.2020. IR (KBr): 2970, 2929, 1667, 1549, 1452, 1383, 761, 700 cm^{-1}.



(**4t**) R_f= 0.22 (ethyl acetate/PE = 1:20), yield: 53%, colorless sticky oil, $[\alpha]_D^{25}$ = +4.8° (EtOAc, *c* 0.15). Single diastereoisomer (dr: >20:1), 76% *ee* determined by HPLC analysis (chiral AD-H column, 3% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 22.6 min, t (minor) = 25.2 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.61 (t, *J* = 7.6 Hz, 3H), 1.73-1.81 (m, 1H), 1.93-1.99 (m, 1H), 3.76-3.81 (m, 1H), 3.88-3.94 (m, 1H), 4.25 (t, *J* = 7.6 Hz, 1H), 4.66 (dd, *J* = 13.2, 4.0 Hz, 1H), 4.82 (dd, *J* = 13.6, 11.6 Hz, 1H), 7.14-7.20 (m, 4H), 7.24-7.34 (m, 7H), 7.37-7.41 (m, 1H), 7.51-7.55 (m, 2H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.86-7.90 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 203.1, 138.4, 137.7, 136.9, 134.0, 133.1, 132.6, 129.3, 128.9, 128.2, 128.0, 127.9, 127.7, 127.4, 126.4, 125.6, 125.2, 123.9, 122.2, 74.2, 54.6, 44.3, 41.4, 24.9 and 11.8. HRMS calcd for $C_{29}H_{28}NO_3$ [M+H]: 438.2069, found: 438.2077. IR (KBr): 3061, 2965, 2927, 1668, 1553, 1451, 1379, 780, 701 cm^{-1}.



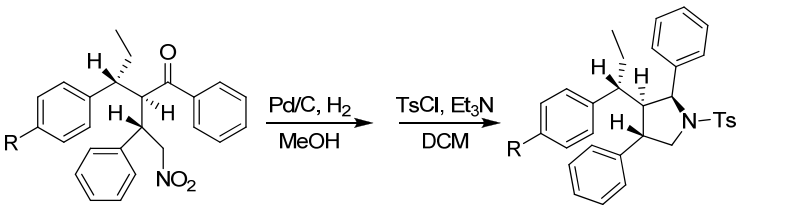
(**4u**) R_f= 0.3 (ethyl acetate/PE = 1:20), yield: 63%, white solid, mp: 133-135 °C, $[\alpha]_D^{25}$ = +20.1° (EtOAc, *c* 0.32). Single diastereoisomer (dr: >20:1), 80% *ee* determined by HPLC analysis (chiral AD-H column, 10% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 9.5 min, t (minor) = 12.7 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.59 (t, *J* = 7.2 Hz, 3H), 1.39-1.55 (m, 2H), 2.93 (td, *J* = 10.4, 10.4, 3.6 Hz, 1H), 3.61 (dt, *J* = 11.6, 4.0, 4.0 Hz, 1H), 4.06 (dd, *J* = 10.0, 5.2 Hz, 1H), 4.90 (dd, *J* = 13.6, 4.0 Hz, 1H), 5.01 (dd, *J* = 13.6, 11.6 Hz, 1H), 6.96-6.98 (m, 2H), 7.12-7.16 (m, 3H), 7.22-7.26 (m, 4H), 7.31-7.43(m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 202.7, 140.6, 139.8, 137.9, 136.9, 129.4, 129.1, 128.9, 128.6, 127.9, 127.8, 127.4, 127.2, 75.0, 56.1, 49.3, 43.9, 26.9 and 12.0. HRMS calcd for $C_{25}H_{25}ClNO_3$ [M+H]: 422.1523, found: 422.1531. IR (KBr): 3064, 3032, 2965, 2928, 2869, 1673, 1550, 1453, 1384, 757, 701 cm^{-1}.



(**4v**) R_f= 0.13 (ethyl acetate/PE = 1:20), yield: 56%, white solid, mp: 105-107 °C, $[\alpha]_D^{25}$ = +23.2° (EtOAc, *c* 0.31). Single diastereoisomer

(dr: >20:1), 87% *ee* determined by HPLC analysis (chiral AD-H column, 20% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 8.4 min, t (minor) = 45.6 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.59 (t, *J* = 7.2 Hz, 3H), 1.39-1.56 (m, 2H), 2.93 (td, *J* = 10.6, 10.6, 3.6 Hz, 1H), 3.59 (dt, *J* = 11.6, 4.0, 4.0 Hz, 1H), 3.81 (s, 3H), 4.05 (dd, *J* = 10.0, 4.8 Hz, 1H), 4.92 (dd, *J* = 13.6, 3.6 Hz, 1H), 5.02 (dd, *J* = 13.6, 11.6 Hz, 1H), 6.71-6.75 (m, 2H), 6.95-6.97 (m, 2H), 7.09-7.14 (m, 3H), 7.25-7.34(m, 3H), 7.39-7.43 (m, 2H), 7.46-7.50 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 201.8, 163.5, 141.1, 138.3, 131.8, 130.5, 129.0, 128.7, 128.0, 127.5, 127.3, 127.2, 113.5, 75.3, 55.6, 55.3, 49.3, 44.0, 26.9 and 12.1. HRMS calcd for $C_{26}H_{28}NO_4$ [M+H]: 418.2018, found: 418.2010. IR (KBr): 3029, 2966, 2930, 1661, 1598, 1551, 1454, 1381, 758, 702 cm^{-1}.

The synthetic procedure of chiral pyrrolidine derivatives:



R = H, **4a**, 89% *ee*
R = OMe, **4p**, 80% *ee*

R = H, **5a**, 90% yield, 90% *ee*
R = OMe, **5p**, 95% yield, 86% *ee*

To a test tube was added of **4a** (114 mg, 0.29 mmol), 10% Pd/C (570 mg) in MeOH (2.0 mL) and the mixture was stirred under a H_2 atmosphere (1.0 atm.) at 40 °C for 2 day. After filtering off the catalyst and evaporating the solvent. The residue was dissolved in dry DCM (2.0 mL). To the mixture was added Et_3N (251 μl) and TsCl (0.87 mmol, 165 mg) dropwise at 0 °C. After the mixture was stirred 24 h at room temperature, cold water was added to the mixture, and it was extracted with DCM. The combined organic phases were washed with brine (3× 10 mL), dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The crude product was subjected to chromatography (PE/EtOAc, 20:1) to obtain pure product **5a** in 90% yield for two steps. R_f= 0.58 (ethyl acetate/PE = 1:5), yield: 90%, white solid, mp: 180-182 °C. $[\alpha]_D^{25}$ = -3.7° (EtOAc, *c* 0.15). 90% *ee* determined by HPLC analysis (chiral AD-H column, 3% IPA in hexane, rate: 1.0 mL/min, 254 nm). Retention time: t (major) = 17.8 min, t (minor) = 23.1 min. Recrystallization from a mixed solvent of ethyl acetate and petroleum afforded a single crystal (>99% *ee*) for X-ray analysis. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.27 (t, *J* = 7.2 Hz, 3H), 1.32-1.40 (m, 1H), 1.68-1.74 (m, 1H), 1.88 (td, *J* = 10.6, 10.6, 2.8 Hz, 1H), 2.40 (s, 3H), 2.89 (td, *J* = 10.4, 10.4, 8.0 Hz, 1H), 3.25 (dt, *J* = 11.6, 8.8, 8.8 Hz, 1H), 3.49 (dd, *J* = 10.4, 8.8 Hz, 1H),

3.85 (dd, *J* = 10.0, 9.6 Hz, 1H), 5.36 (d, *J* = 7.6 Hz, 1H), 6.46-6.55 (m, 4H), 6.70-6.81 (m, 3H), 6.89-6.90 (m, 3H), 7.12 (d, *J* = 8.4 Hz, 2H), 7.20-7.22 (m, 2H), 7.29-7.31 (m, 3H), 7.37-7.39 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 142.7, 141.6, 140.4, 139.0, 135.6, 129.1, 128.6, 128.6, 128.2, 127.9, 127.5, 127.3, 127.2, 127.1, 125.8, 125.5, 67.6, 56.7, 56.3, 48.1, 47.9, 27.6, 21.4 and 11.4. HRMS calcd for $C_{32}H_{34}NO_2S$ [M+H]: 496.2310, found: 496.2303. IR (KBr): 3029, 2945, 2865, 1454, 1339, 1160, 762, 701, 664 cm^{-1}.

(**5p**) R_f = 0.51 (ethyl acetate/PE = 1:5), yield: 95%, white solid, mp: 154-155 ^{o}C, $[\alpha]_D^{25}$ = -0.7^{o} (EtOAc, *c* 0.43). 86% *ee* determined by HPLC analysis (chiral AD-H column, 3% IPA in hexane, rate: 1.0 mL/min, 235 nm). Retention time: t (major) = 27.8 min, t (minor) = 41.5 min. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.29 (t, *J* = 7.2 Hz, 3H), 1.26-1.36 (m, 1H), 1.67-1.78 (m, 1H), 1.81 (td, *J* = 10.8, 10.8, 2.8 Hz, 1H), 2.40 (s, 3H), 2.80-2.87 (m, 1H), 3.21 (dt, *J* = 11.6, 8.8, 8.8 Hz, 1H), 3.49 (dd, *J* = 10.4, 8.8 Hz, 1H), 3.63 (s, 3H), 3.84 (dd, *J* = 10.4, 9.2 Hz, 1H), 5.34 (d, *J* = 8.0 Hz, 1H), 6.23-6.38 (m, 4H), 6.54-6.66 (m, 2H), 6.92-6.93 (m, 3H), 7.12-7.22 (m, 4H), 7.29-7.30 (m, 3H), 7.37-7.40 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 157.5, 142.7, 141.7, 139.1, 135.7, 132.6, 131.4, 130.3, 129.4, 129.1, 128.5, 128.2, 127.8, 127.5, 127.4, 127.1, 125.4, 112.7, 67.6, 56.7, 56.6, 55.0, 48.0, 47.2, 27.7, 21.4 and 11.4. HRMS calcd for $C_{33}H_{36}NO_3S$ [M+H]: 526.2416, found: 526.2415. IR (KBr): 3029, 2926, 1511, 1456, 1348, 1246, 1165, 702, 666 cm^{-1}.

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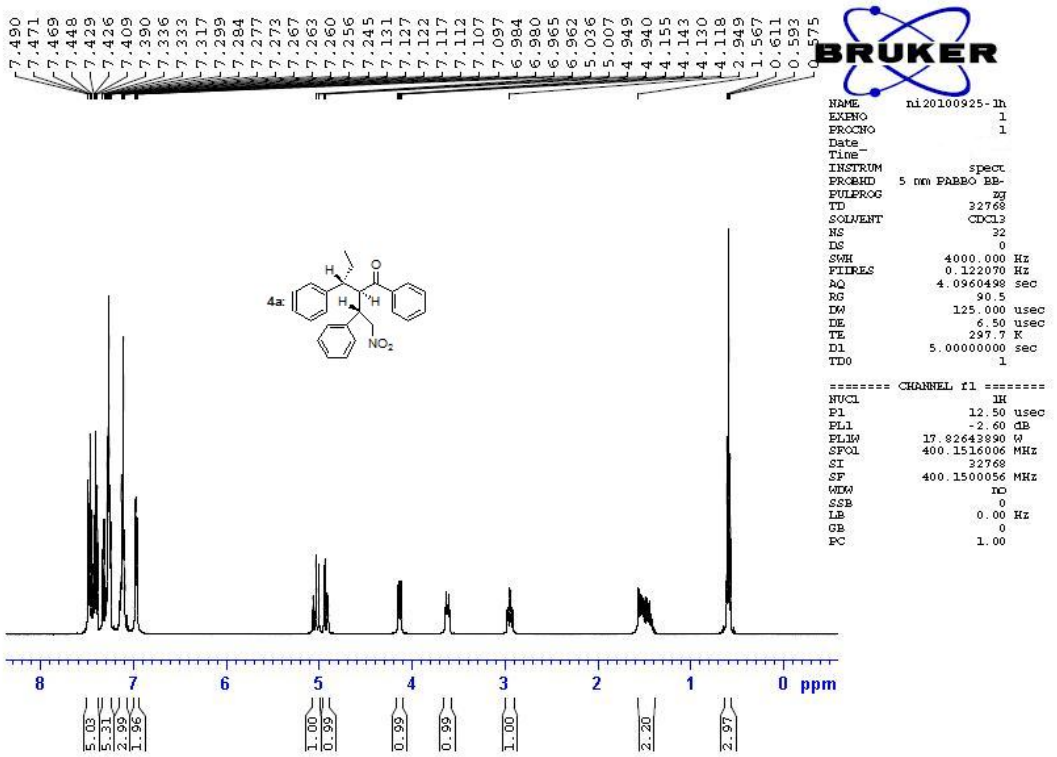
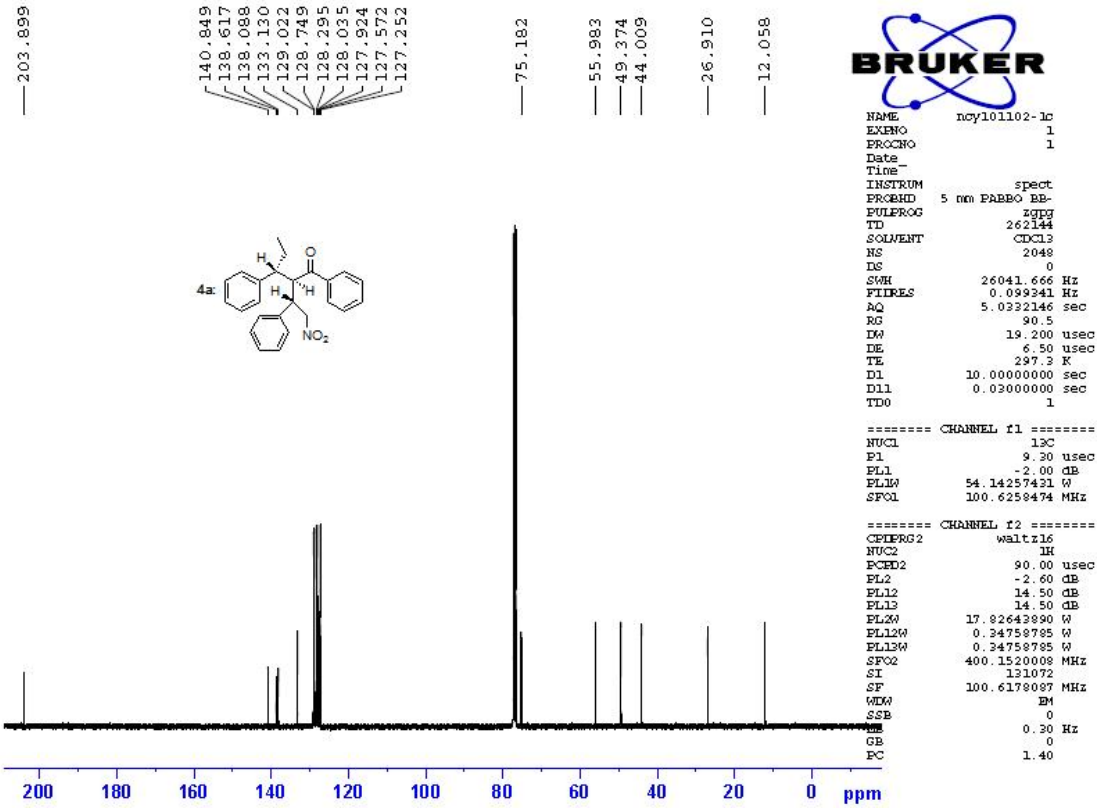
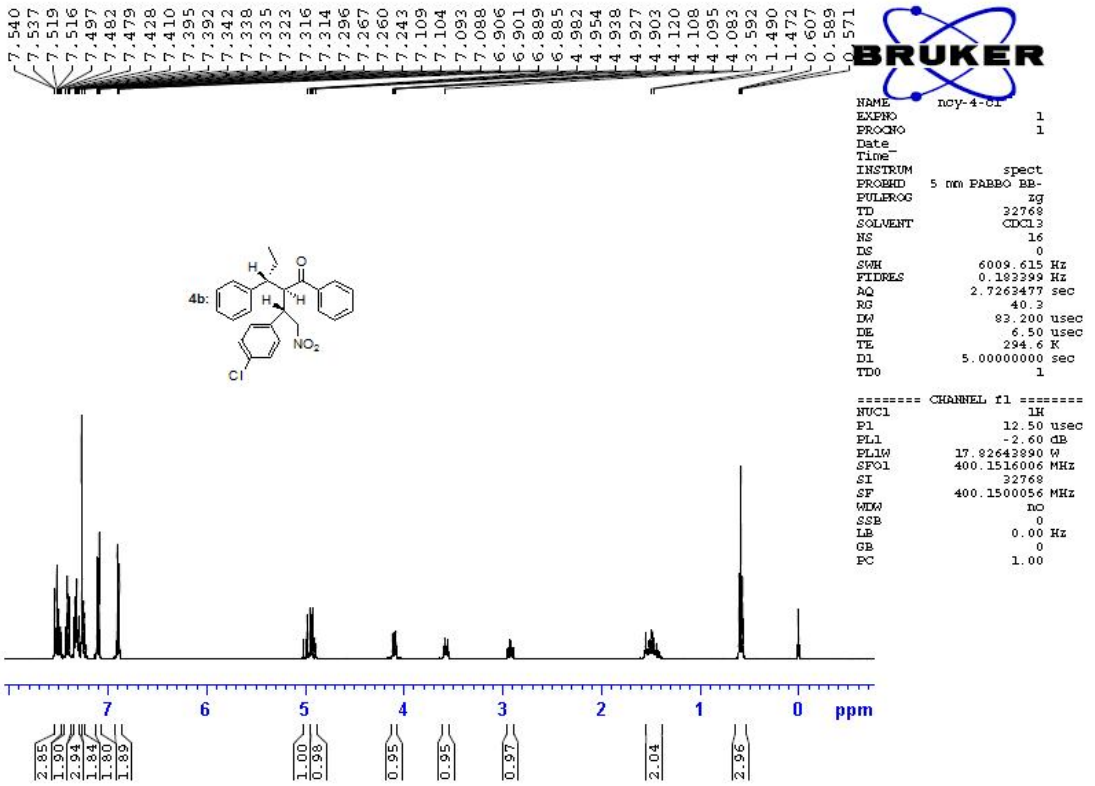
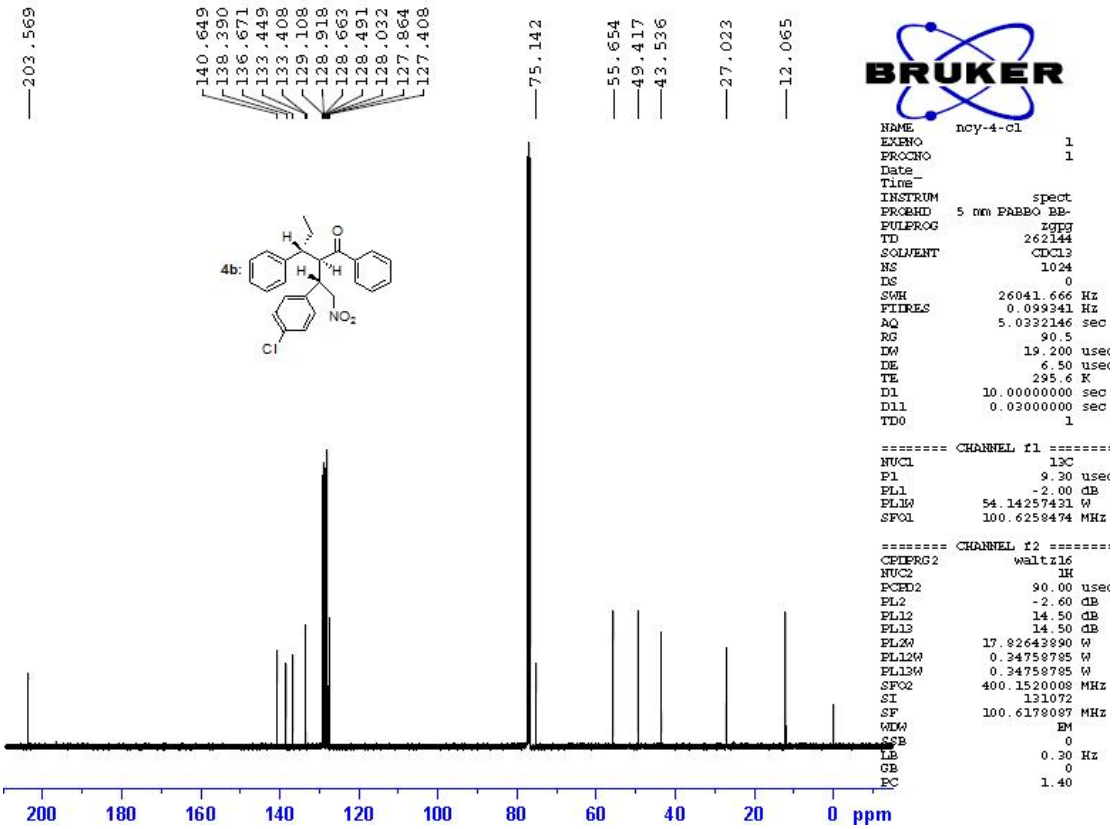
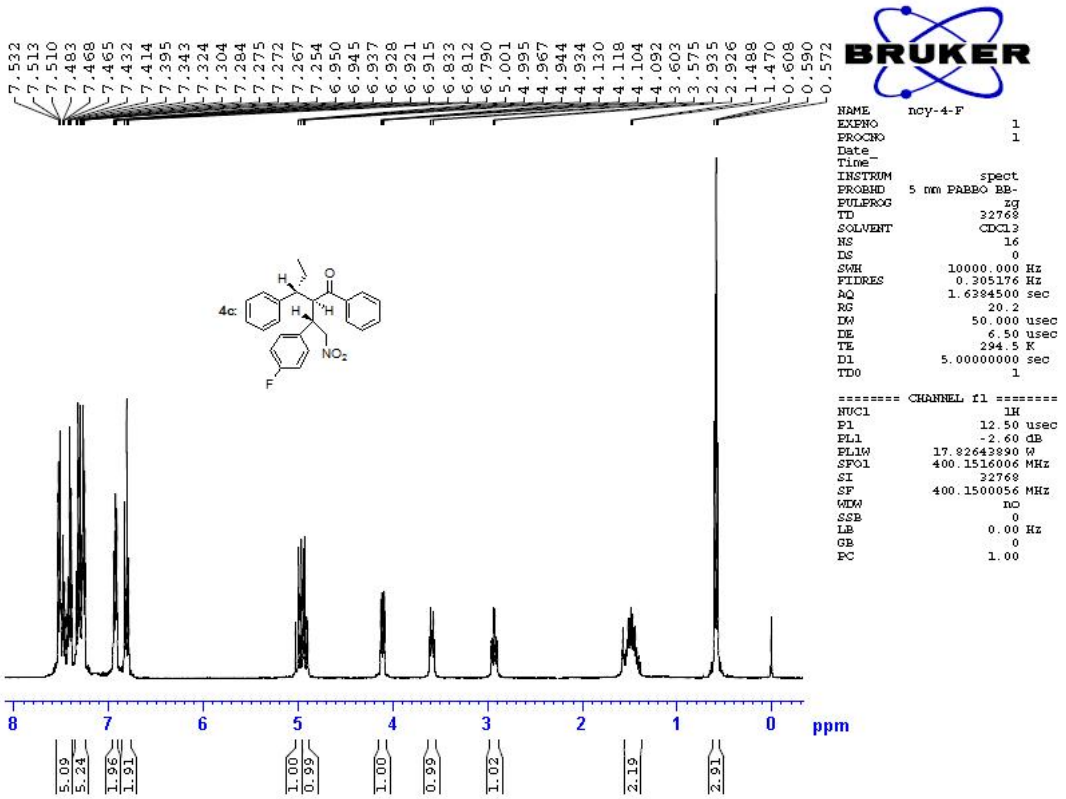
NMR spectra for compounds 4a-v, 5a, 5p







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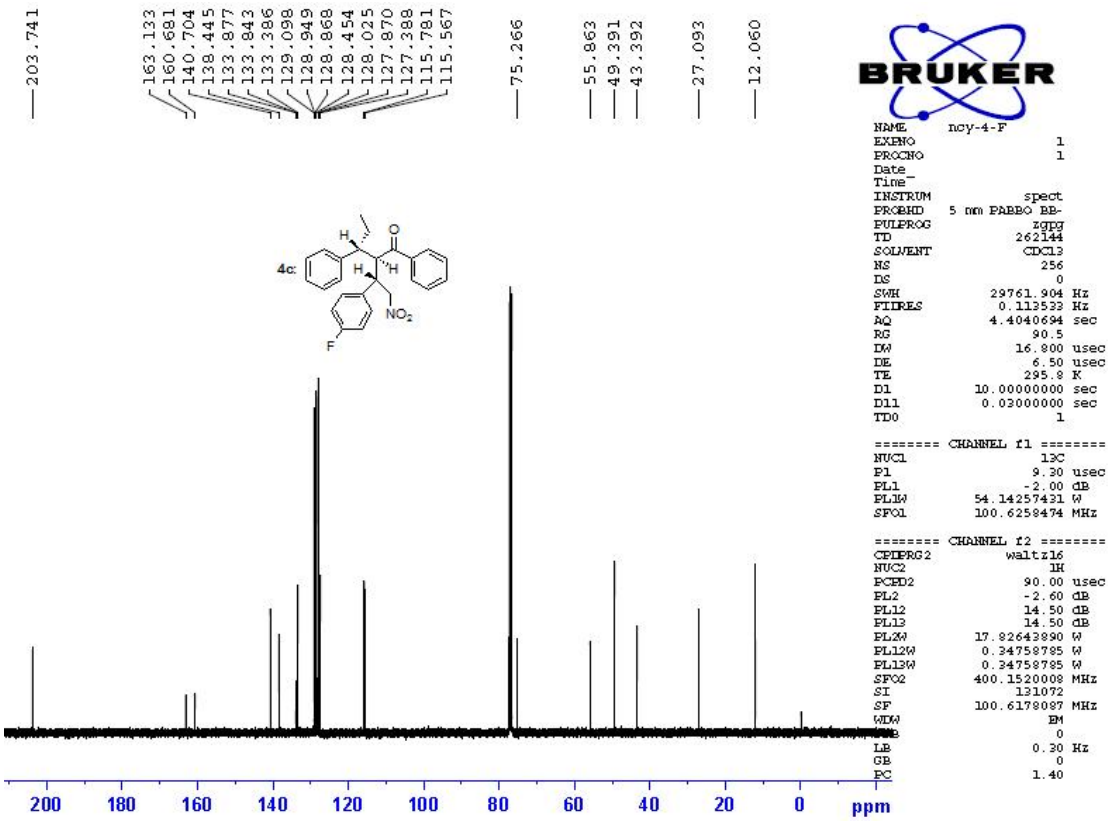
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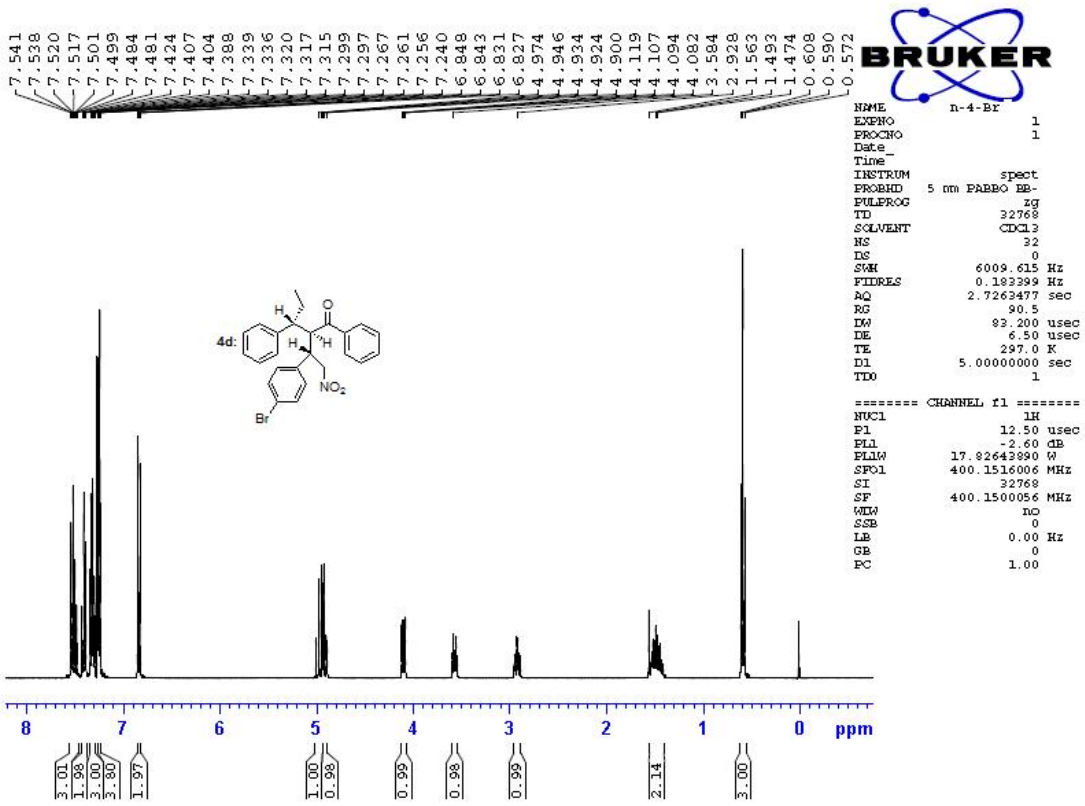
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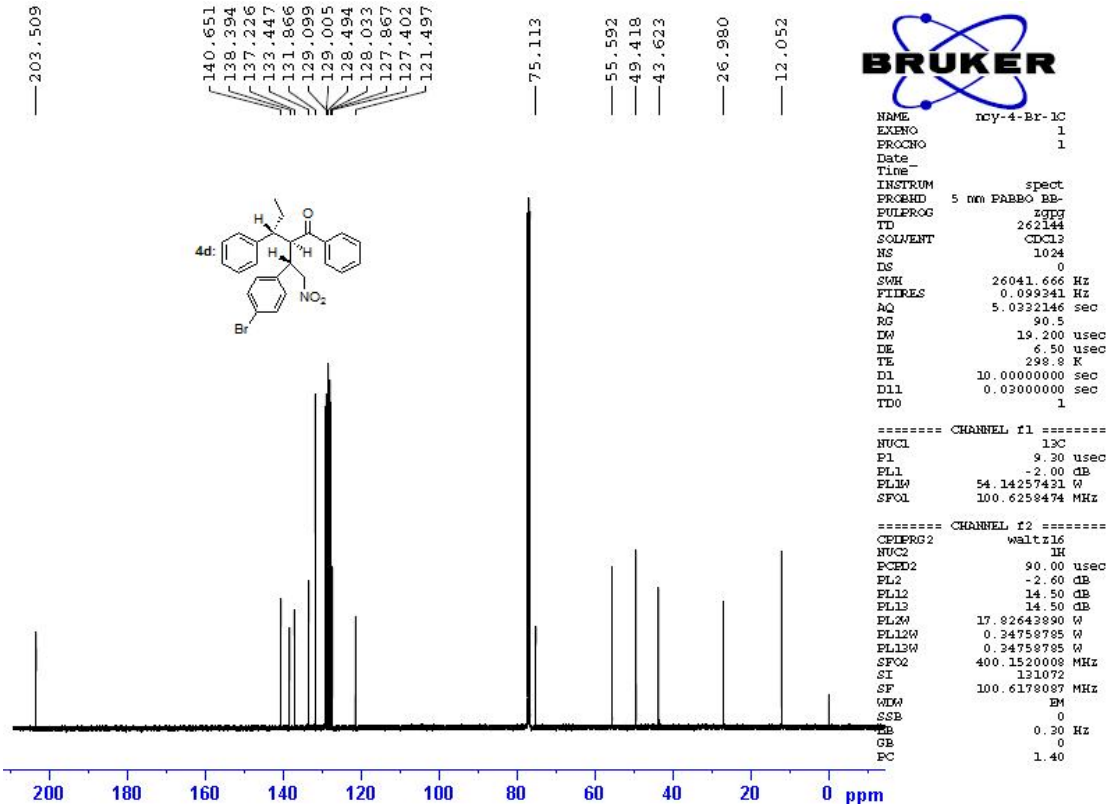
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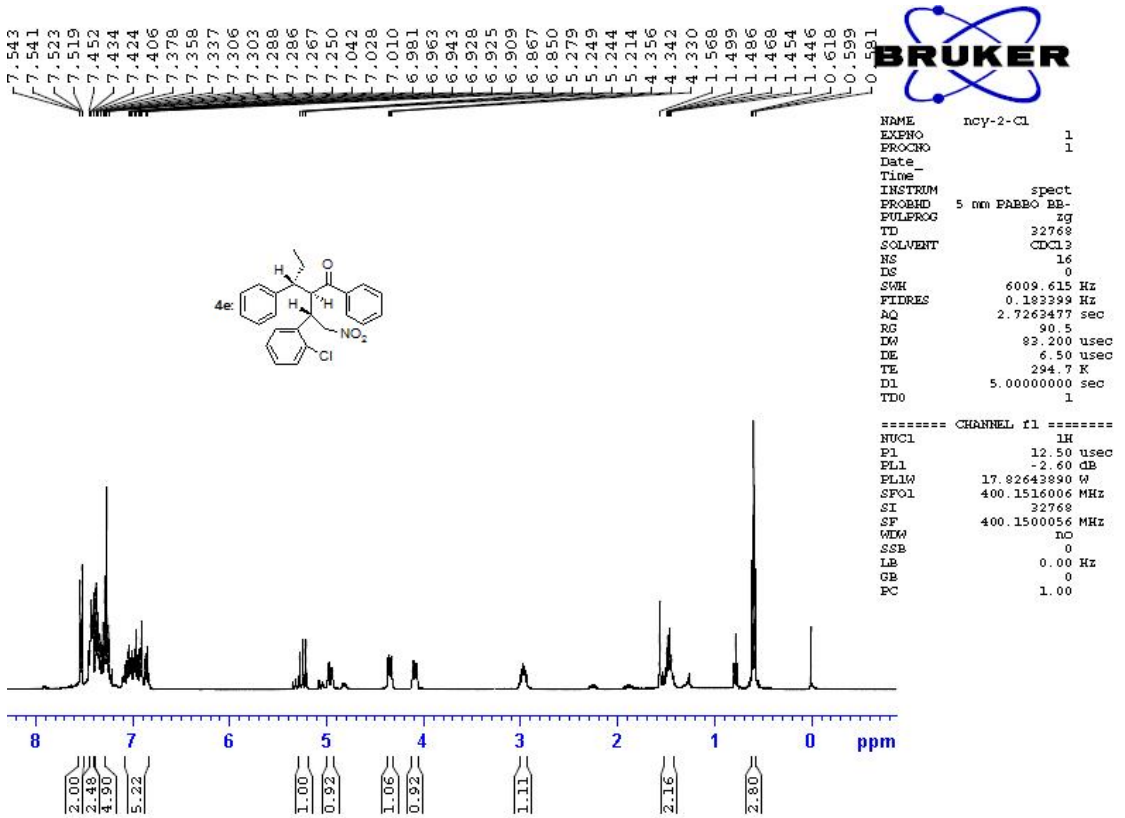
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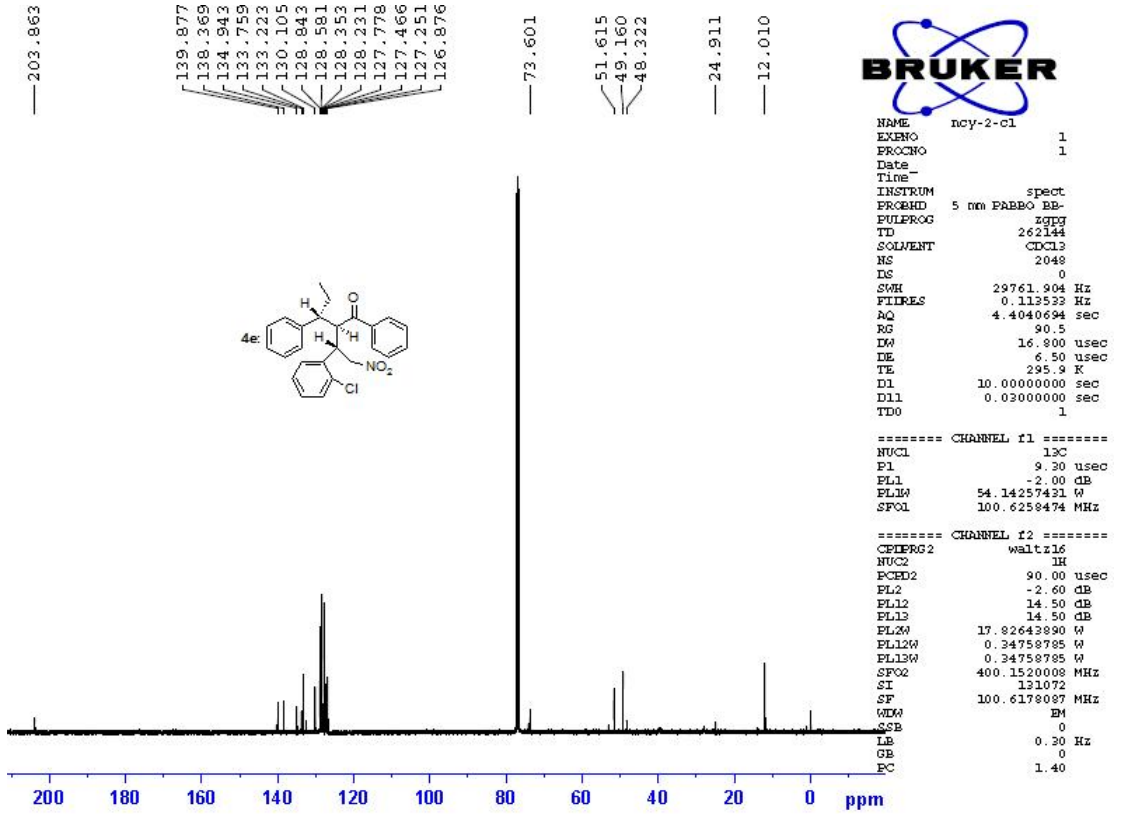
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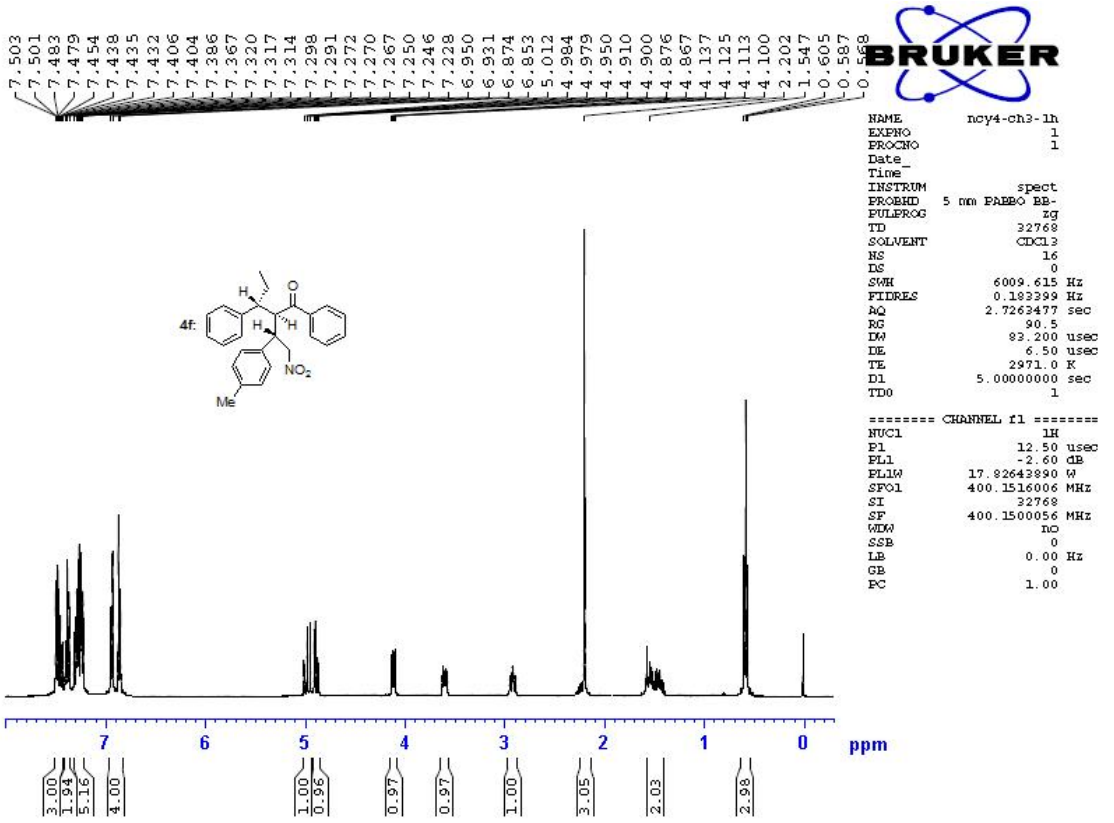
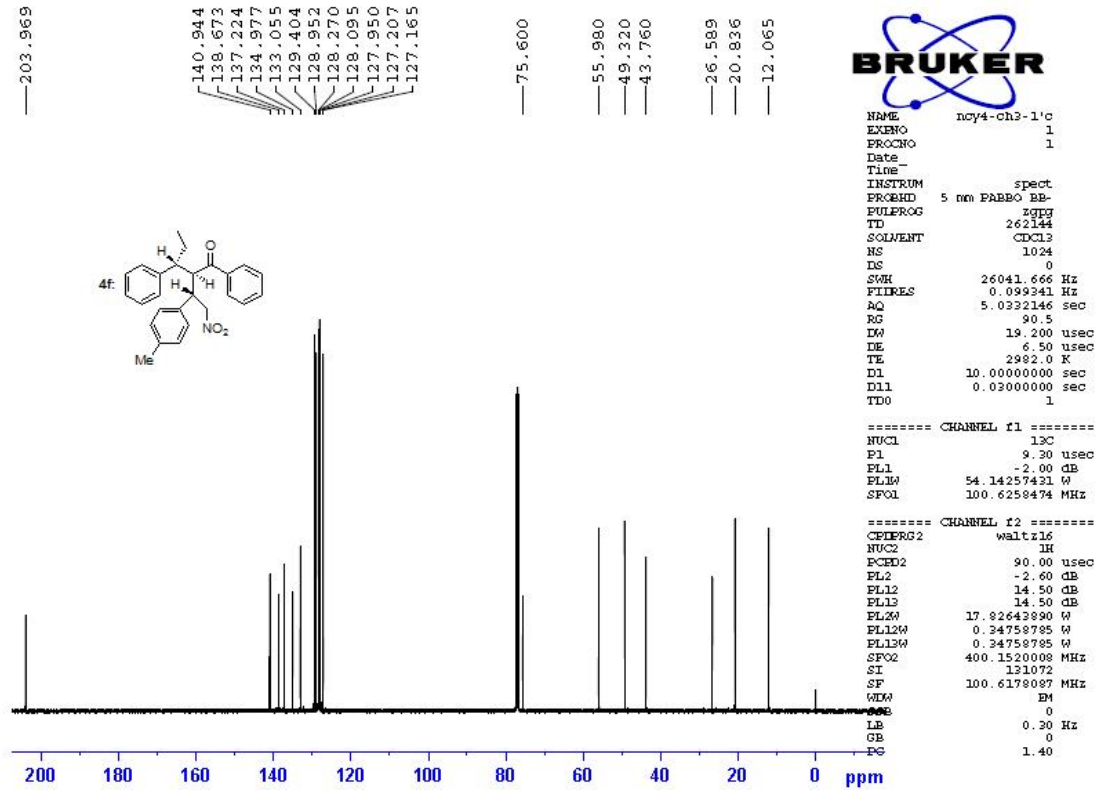
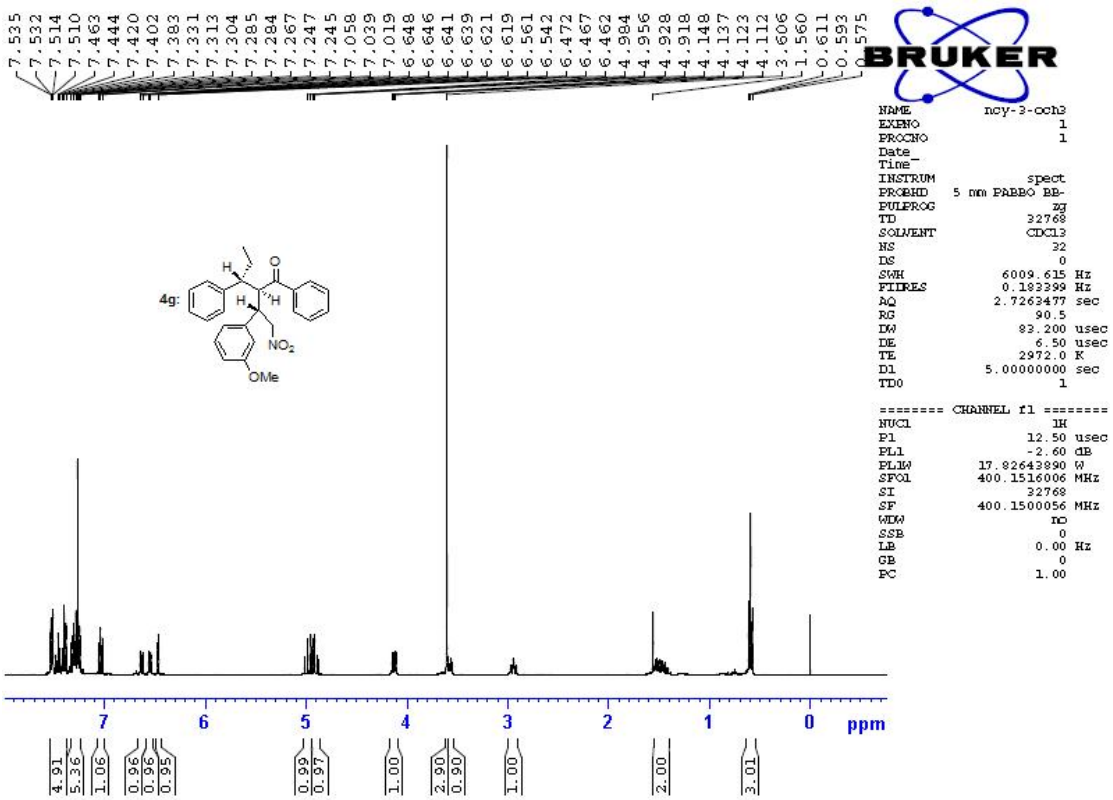
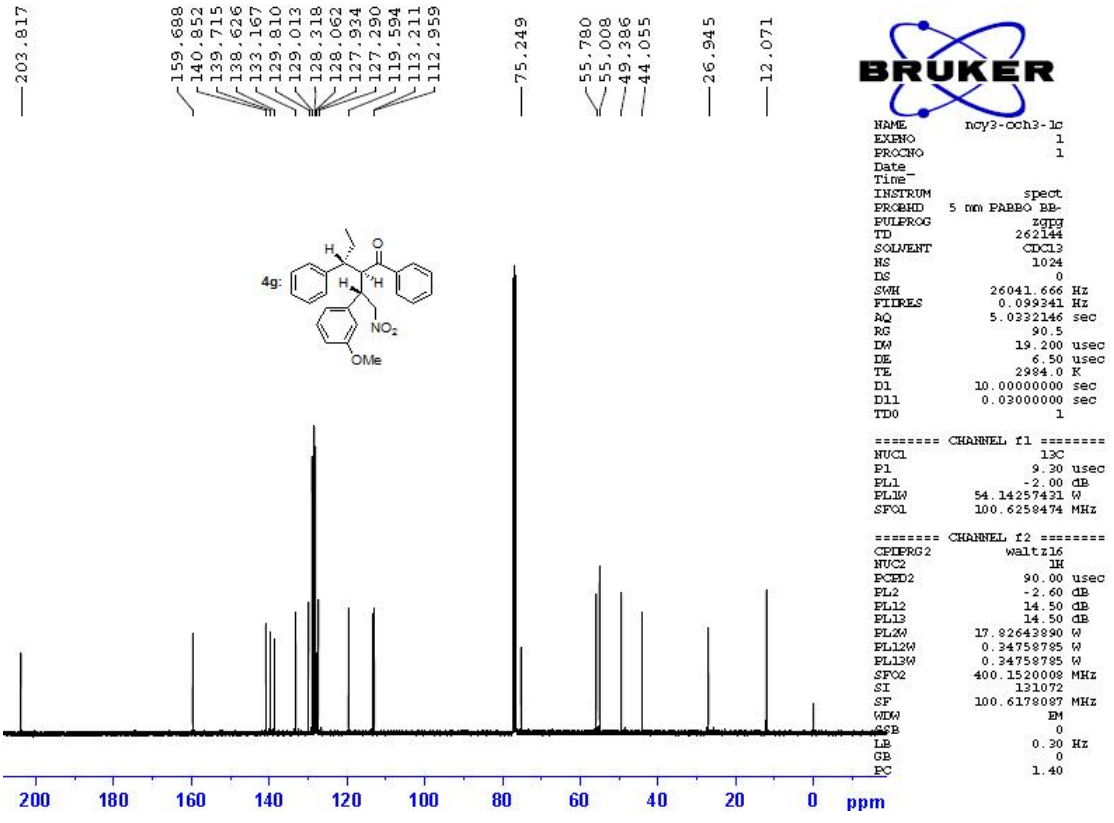
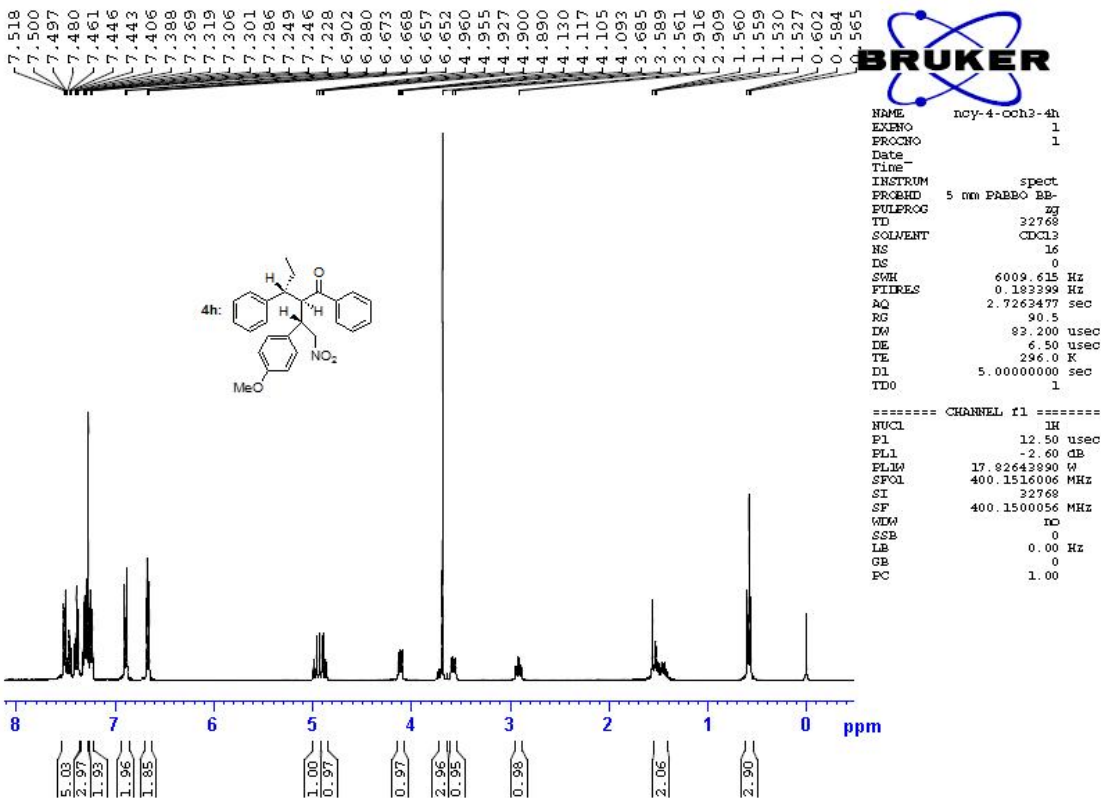
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
4e:
200
180
160
140
120
100
80
60
40
20
0
ppm

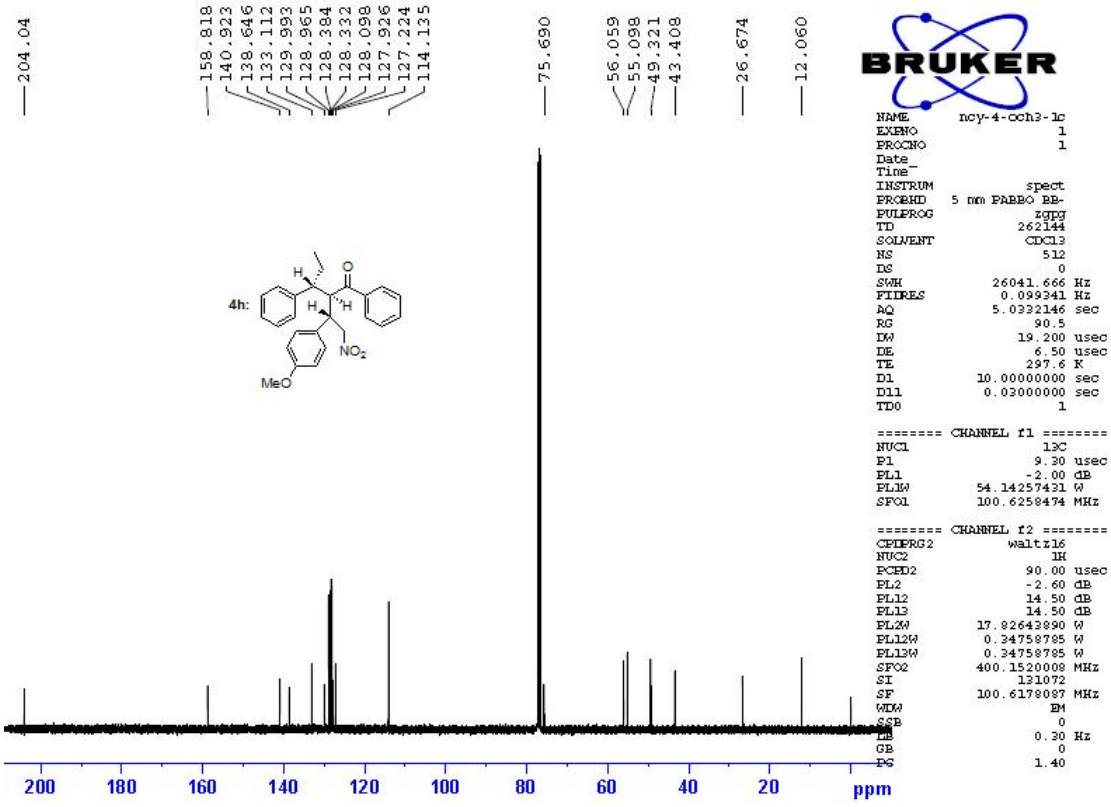

7.503
7.501
7.483
7.479
7.454
7.438
7.435
7.432
7.406
7.404
7.386
7.367
7.320
7.317
7.314
7.298
7.291
7.272
7.270
7.267
7.250
7.246
7.228
6.950
6.931
6.874
6.853
5.012
4.984
4.979
4.950
4.910
4.900
4.876
4.867
4.137
4.125
4.113
4.100
2.202
1.547
0.605
0.587
0.568
BRUKER
NAME ncy4-ch3-1h
EXPNO 1
PROCNO 1
Date_
Time
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 90.5
DW 83.200 usec
DE 6.50 usec
TE 297l.0 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4f:
H
O
H
H
NO2
Me
7
6
5
4
3
2
1
0
ppm
3.00
1.94
5.16
4.00
1.00
0.96
0.97
0.97
1.00
3.05
2.03
2.98

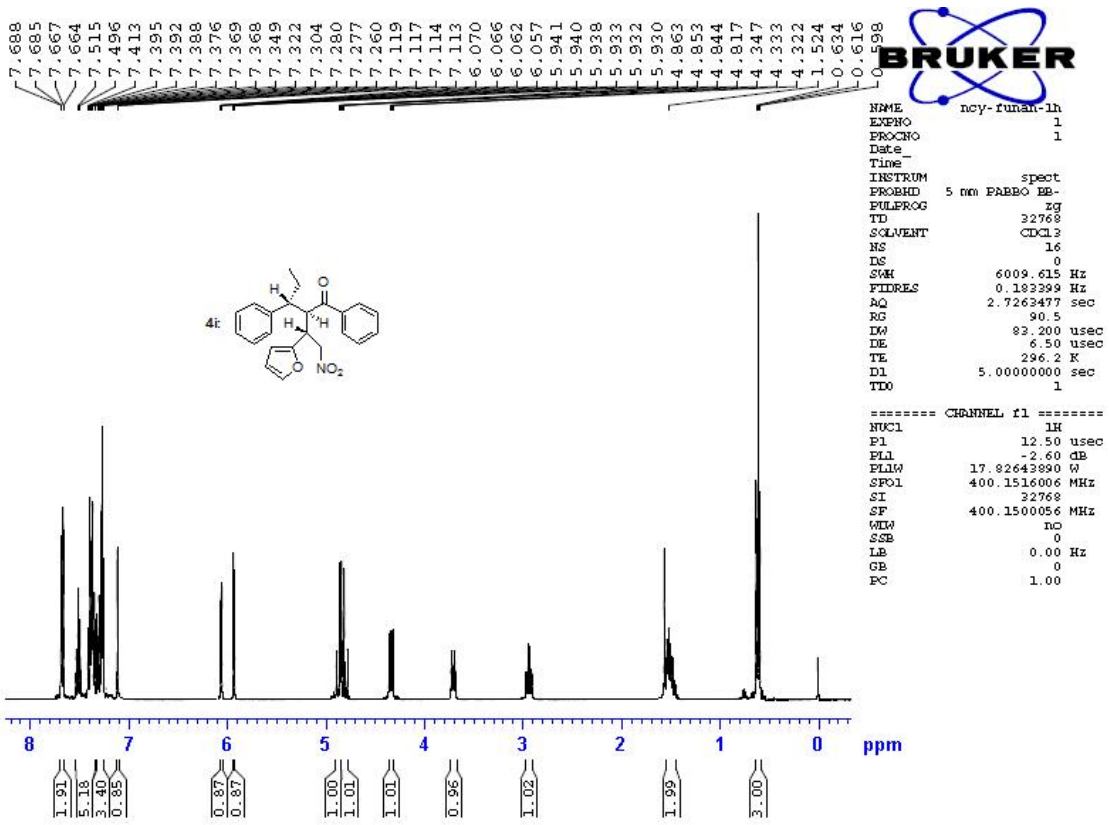

203.969
140.944
138.673
137.224
134.977
133.055
129.404
128.952
128.270
128.095
127.950
127.207
127.165
75.600
55.980
49.320
43.760
26.589
20.836
12.065
BRUKER
NAME ncy4-ch3-1'c
EXPNO 1
PROCNO 1
Date_
Time
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 1024
DS 0
SWH 26041.666 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 90.5
DW 19.200 usec
DE 6.50 usec
TE 2982.0 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
4f:
H
O
H
H
NO2
Me
200
180
160
140
120
100
80
60
40
20
0
ppm

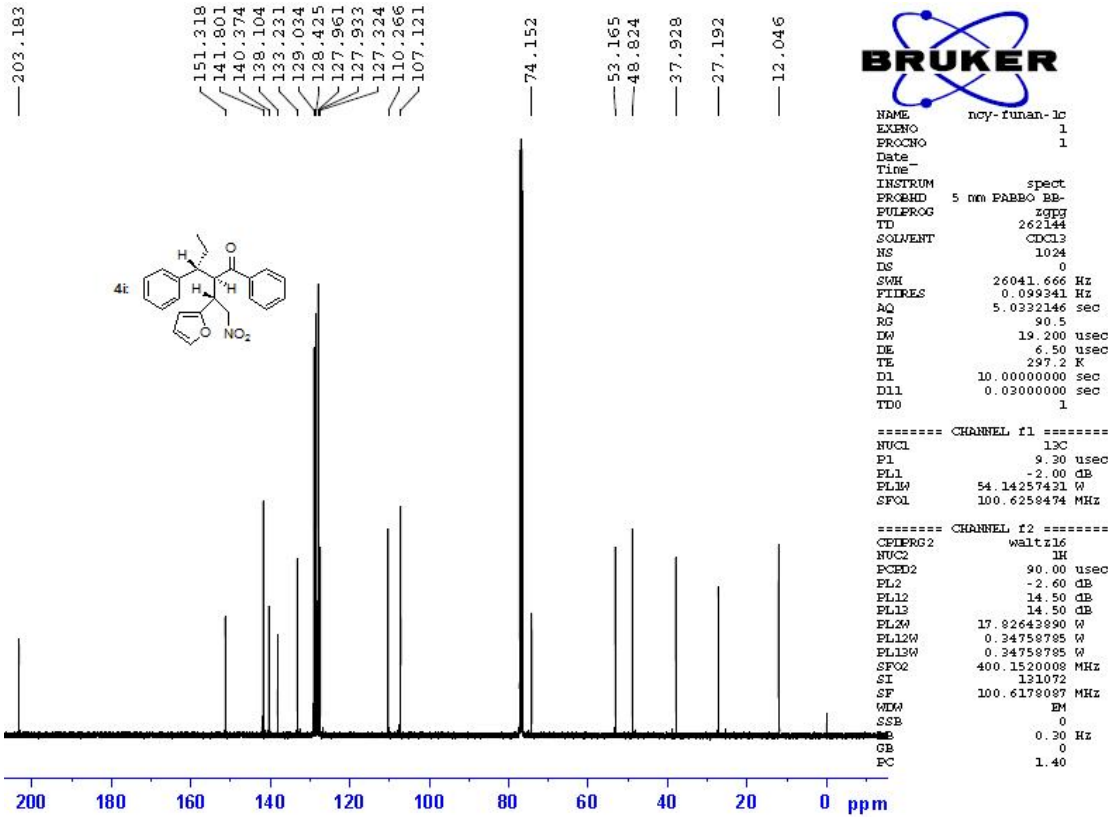


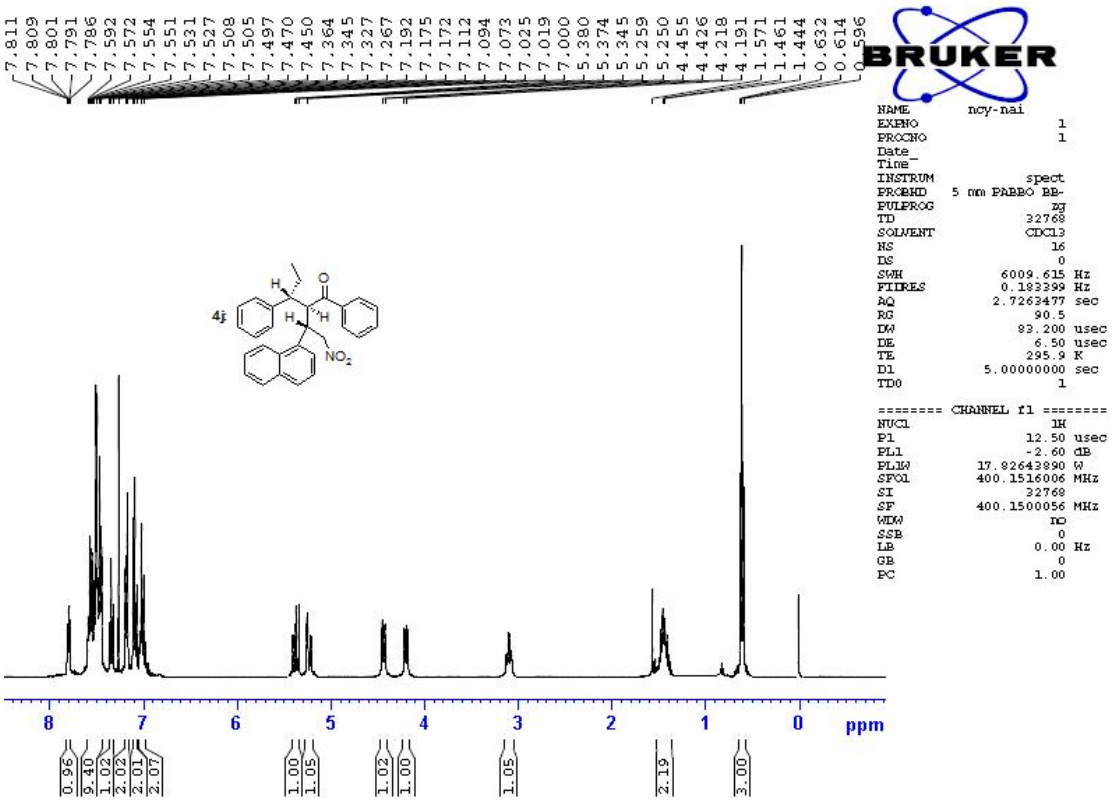



7.518
7.500
7.497
7.480
7.461
7.446
7.443
7.406
7.388
7.369
7.319
7.306
7.301
7.286
7.249
7.246
7.228
6.902
6.880
6.673
6.668
6.657
6.652
4.960
4.955
4.927
4.900
4.890
4.130
4.117
4.105
4.093
3.685
3.589
3.561
2.916
2.909
1.560
1.559
1.530
1.527
0.602
0.584
0.565
BRUKER
NAME ncy-4-och3-4h
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 90.5
DW 83.200 usec
DE 6.50 usec
TE 296.0 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4h:
NO2
MeO
8
7
6
5
4
3
2
1
0
ppm
5.03
2.97
1.93
1.96
1.85
1.00
0.97
0.97
2.96
0.95
0.98
2.06
2.90


204.04
158.818
140.923
138.646
133.112
129.993
128.965
128.384
128.332
128.098
127.926
127.224
114.135
75.690
56.059
55.098
49.321
43.408
26.674
12.060
BRUKER
NAME ncy-4-och3-1c
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 512
DS 0
SWH 26041.666 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 90.5
DW 19.200 usec
DE 6.50 usec
TE 297.6 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
4h:
NO2
MeO
200
180
160
140
120
100
80
60
40
20
ppm

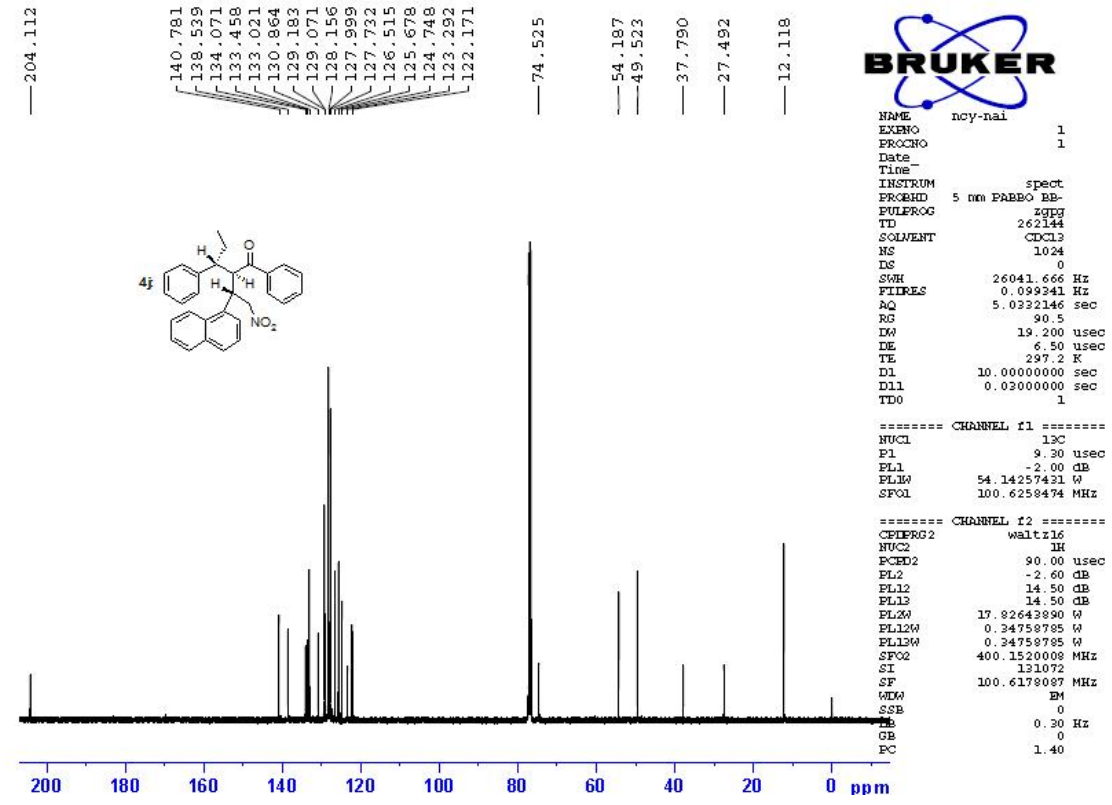

7.688
7.685
7.667
7.664
7.515
7.496
7.413
7.395
7.392
7.388
7.376
7.369
7.368
7.349
7.322
7.304
7.280
7.277
7.260
7.119
7.117
7.114
7.113
6.070
6.066
6.062
6.057
5.941
5.940
5.938
5.933
5.932
5.930
4.863
4.853
4.844
4.817
4.347
4.333
4.322
1.524
0.634
0.616
0.598
BRUKER
NAME ncy-furan-1h
EXPNO 1
PROCNO 1
Date_
Time
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 90.5
DW 83.200 usec
DE 6.50 usec
TE 296.2 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4i
NO2
8
7
6
5
4
3
2
1
0
ppm
1.91
5.18
3.40
0.85
0.87
0.87
1.00
1.01
1.01
0.96
1.02
1.99
3.00


203.183
151.318
141.801
140.374
138.104
133.231
129.034
128.425
127.961
127.933
127.324
110.266
107.121
74.152
53.165
48.824
37.928
27.192
12.046
BRUKER
NAME ncy-furan-1c
EXPNO 1
PROCNO 1
Date_
Time
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 1024
DS 0
SWH 26041.666 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 90.5
DW 19.200 usec
DE 6.50 usec
TE 297.2 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
4i
NO2
200
180
160
140
120
100
80
60
40
20
0
ppm



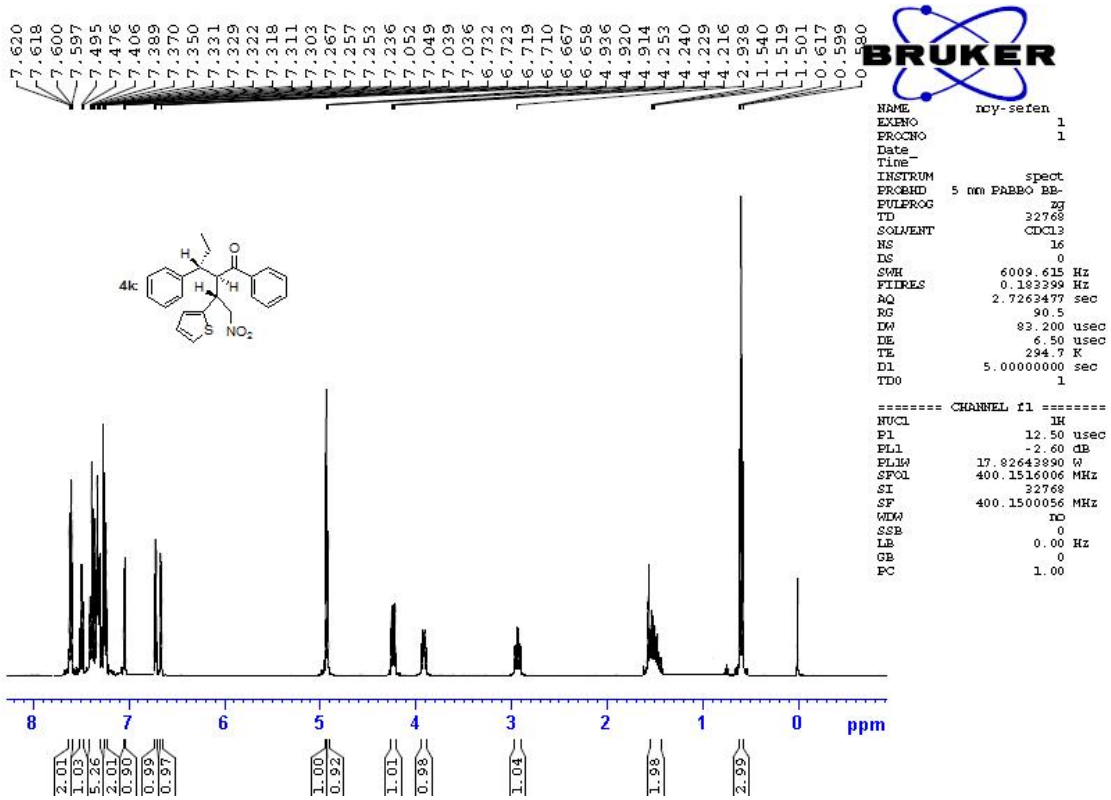
BRUKER

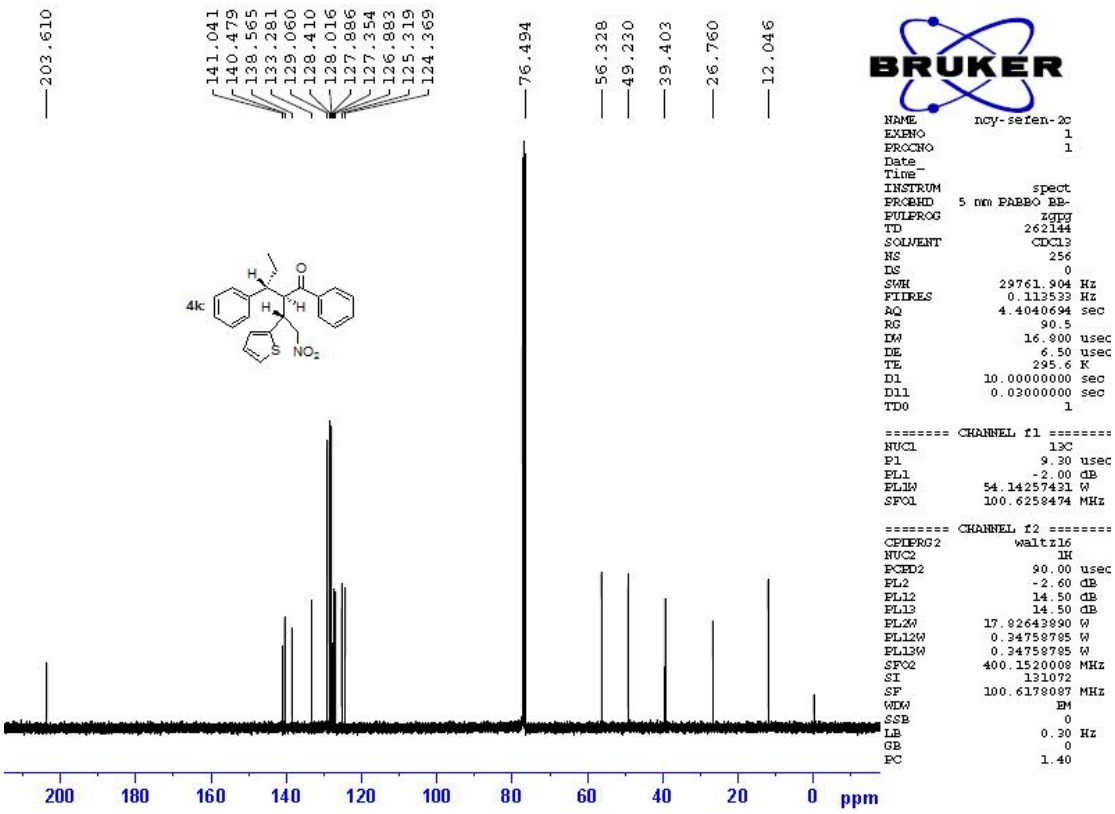
NAME	ncy-nai
EXPNO	1
PROCNO	1
Date_	
Time_	
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7262477 sec
RG	90.5
DW	83.200 usec
DE	6.50 usec
TE	295.9 K
D1	5.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.50 usec
PL1	-2.60 dB
PL1W	17.82643890 W
SFO1	400.1516006 MHz
SI	32768
SF	400.1500056 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

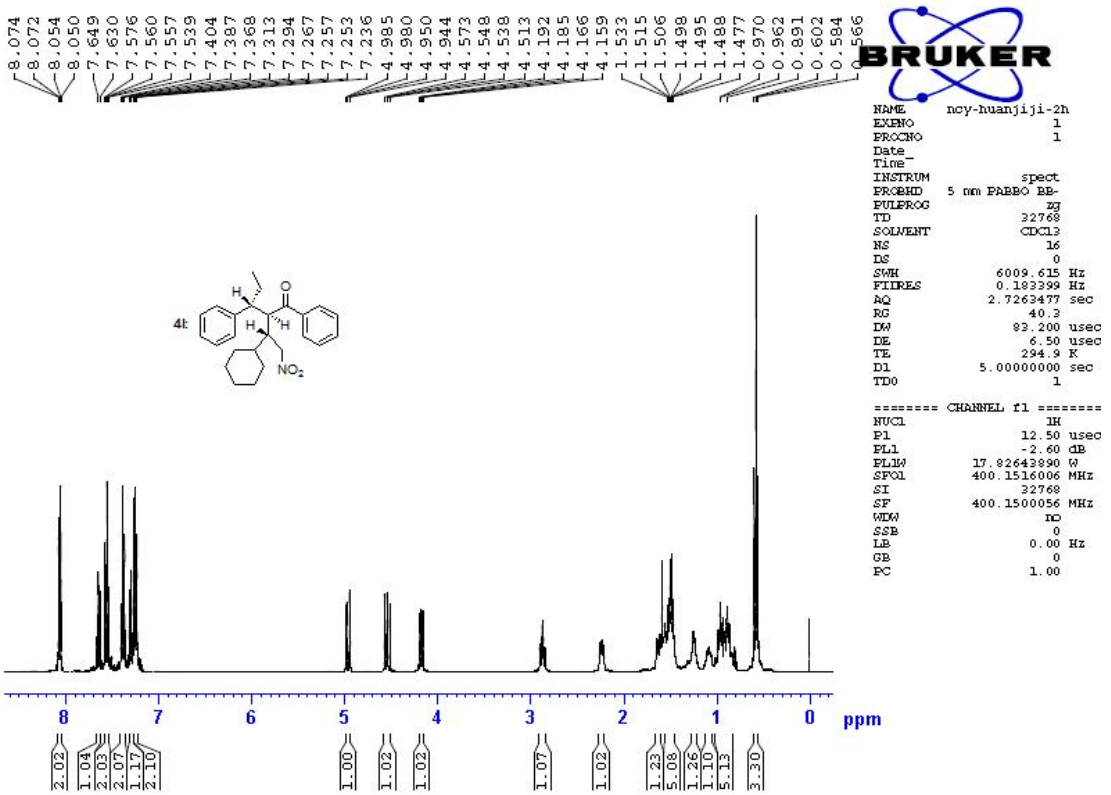


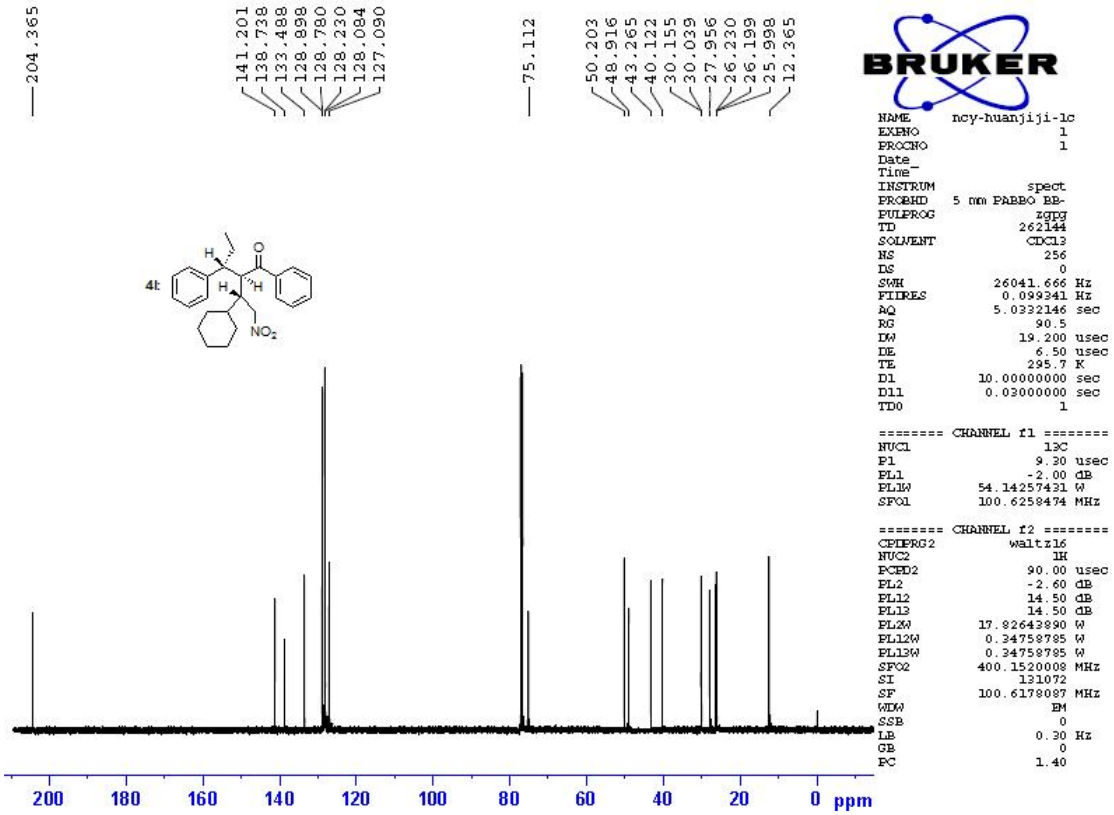
BRUKER

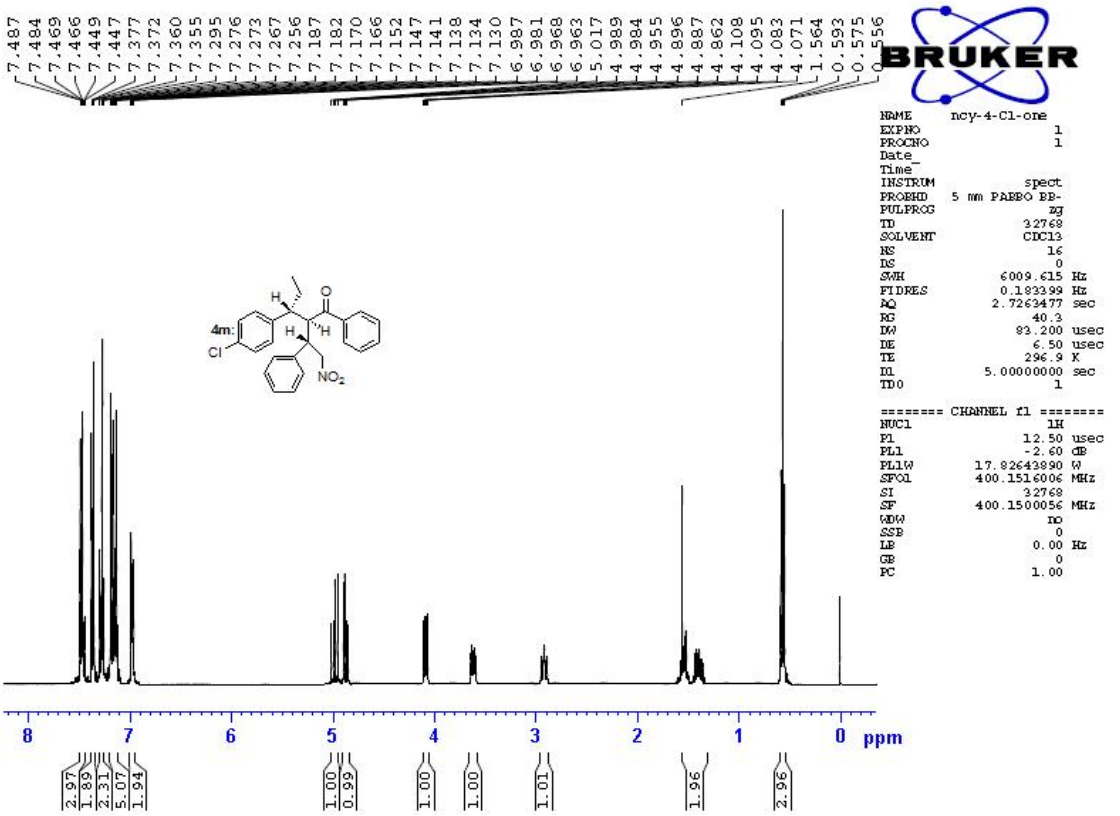
NAME	ncy-nai
EXPNO	1
PROCNO	1
Date_	
Time_	
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg
TD	262144
SOLVENT	CDCl3
NS	1024
DS	0
SWH	26041.666 Hz
FIDRES	0.099341 Hz
AQ	5.0332146 sec
RG	90.5
DW	19.200 usec
DE	6.50 usec
TE	297.2 K
D1	10.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.30 usec
PL1	-2.00 dB
PL1W	54.14257431 W
SFO1	100.6258474 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.60 dB
PL12	14.50 dB
PL13	14.50 dB
PL2W	17.82643890 W
PL12W	0.34758785 W
PL13W	0.34758785 W
SFO2	400.1520008 MHz
SI	131072
SF	100.6178087 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40

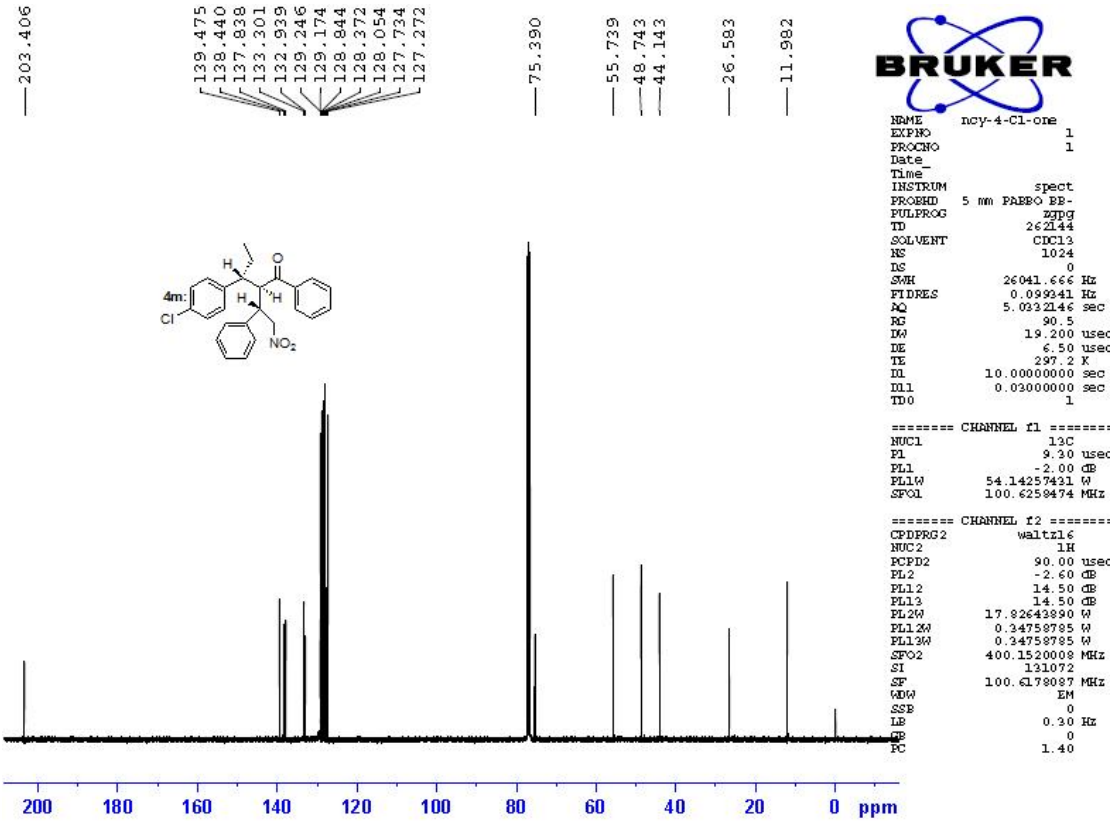

7.620
7.618
7.600
7.597
7.495
7.476
7.406
7.389
7.370
7.350
7.331
7.329
7.322
7.318
7.311
7.303
7.267
7.257
7.253
7.236
7.052
7.049
7.039
7.036
6.732
6.723
6.719
6.710
6.667
6.658
4.936
4.920
4.914
4.253
4.240
4.229
4.216
2.938
1.540
1.519
1.501
0.617
0.599
0.580
BRUKER
NAME ncy-sefen
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 90.5
DW 83.200 usec
DE 6.50 usec
TE 294.7 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4k
8
7
6
5
4
3
2
1
0
ppm
2.01
1.03
5.26
2.01
0.90
0.99
0.97
1.00
0.92
1.01
0.98
1.04
1.98
2.99

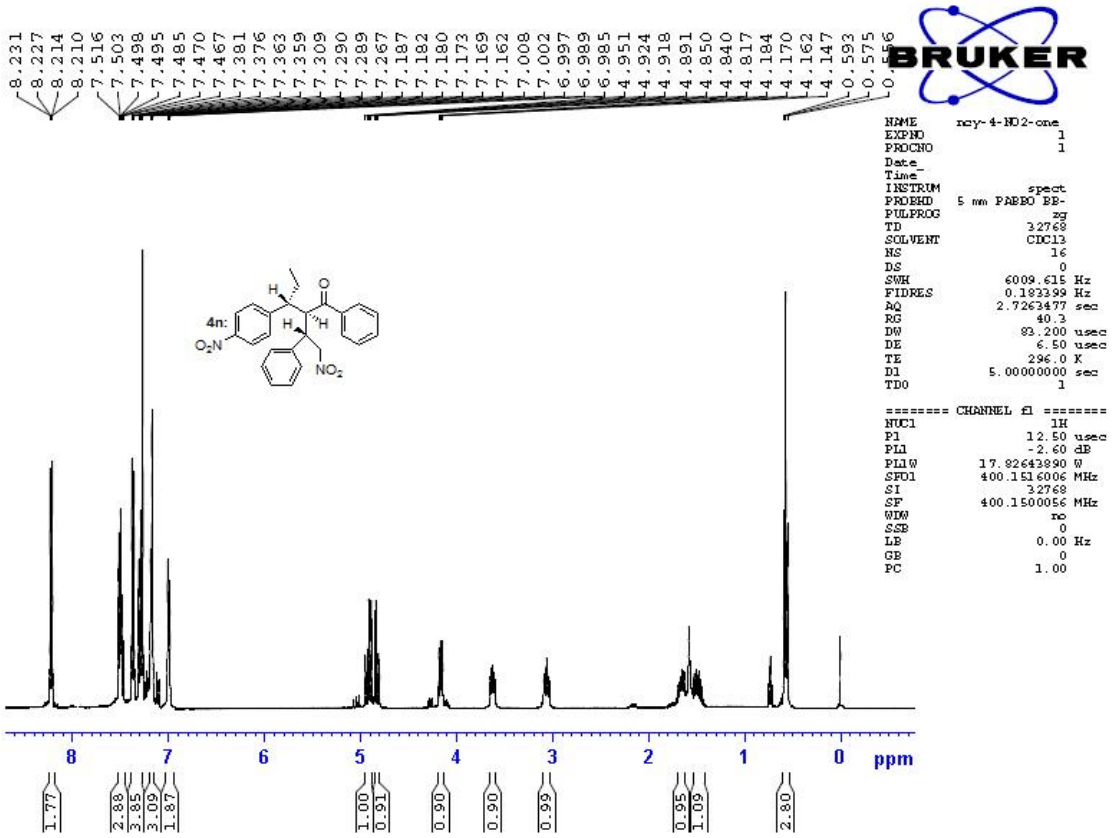

203.610
141.041
140.479
138.565
133.281
129.060
128.410
128.016
127.886
127.354
126.883
125.319
124.369
76.494
56.328
49.230
39.403
26.760
12.046
BRUKER
NAME ncy-sefen-2c
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 256
DS 0
SWH 29761.904 Hz
FIDRES 0.113533 Hz
AQ 4.4040694 sec
RG 90.5
DW 16.800 usec
DE 6.50 usec
TE 295.6 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
4k
200
180
160
140
120
100
80
60
40
20
0
ppm

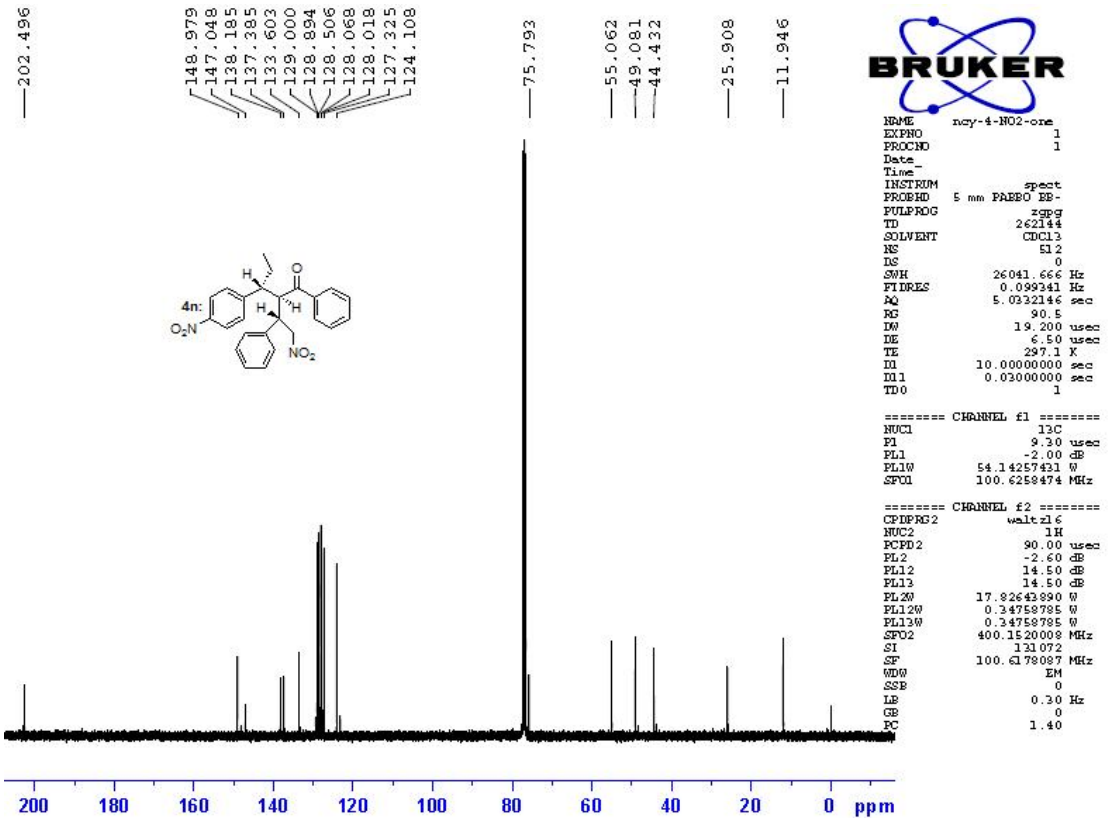

8.074
8.072
8.054
8.050
7.649
7.630
7.576
7.560
7.557
7.539
7.404
7.387
7.368
7.313
7.294
7.267
7.257
7.253
7.236
4.985
4.980
4.950
4.944
4.573
4.548
4.538
4.513
4.192
4.185
4.166
4.159
1.533
1.515
1.506
1.498
1.495
1.488
1.477
0.970
0.962
0.891
0.602
0.584
0.566
BRUKER
NAME ncy-huanjiji-2h
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 40.3
DW 83.200 usec
DE 6.50 usec
TE 294.9 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4t
NO2
8
7
6
5
4
3
2
1
0
ppm
2.02
1.04
2.03
2.07
1.17
2.10
1.00
1.02
1.02
1.07
1.02
1.23
5.08
1.26
1.10
5.13
3.30

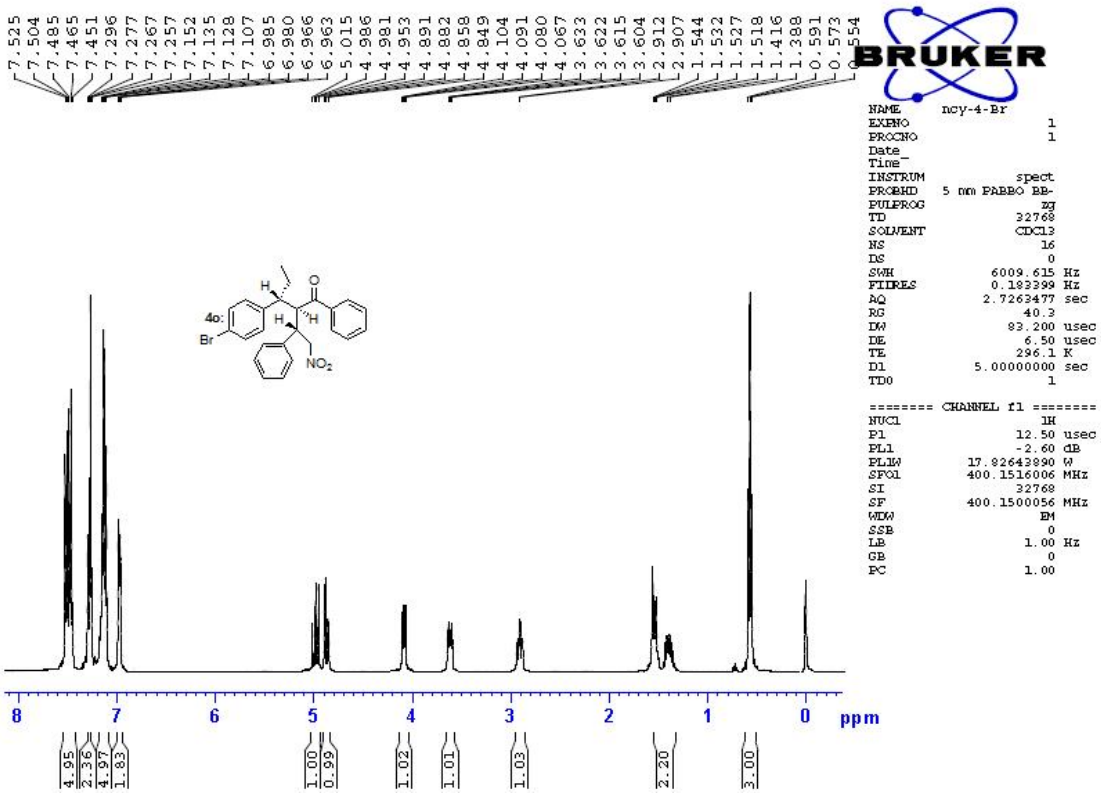

204.365
141.201
138.738
133.488
128.898
128.780
128.230
128.084
127.090
75.112
50.203
48.916
43.265
40.122
30.155
30.039
27.956
26.230
26.199
25.998
12.365
BRUKER
NAME ncy-huanjiji-1c
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 256
DS 0
SWH 26041.666 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 90.5
DW 19.200 usec
DE 6.50 usec
TE 295.7 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
4t
NO2
200
180
160
140
120
100
80
60
40
20
0
ppm

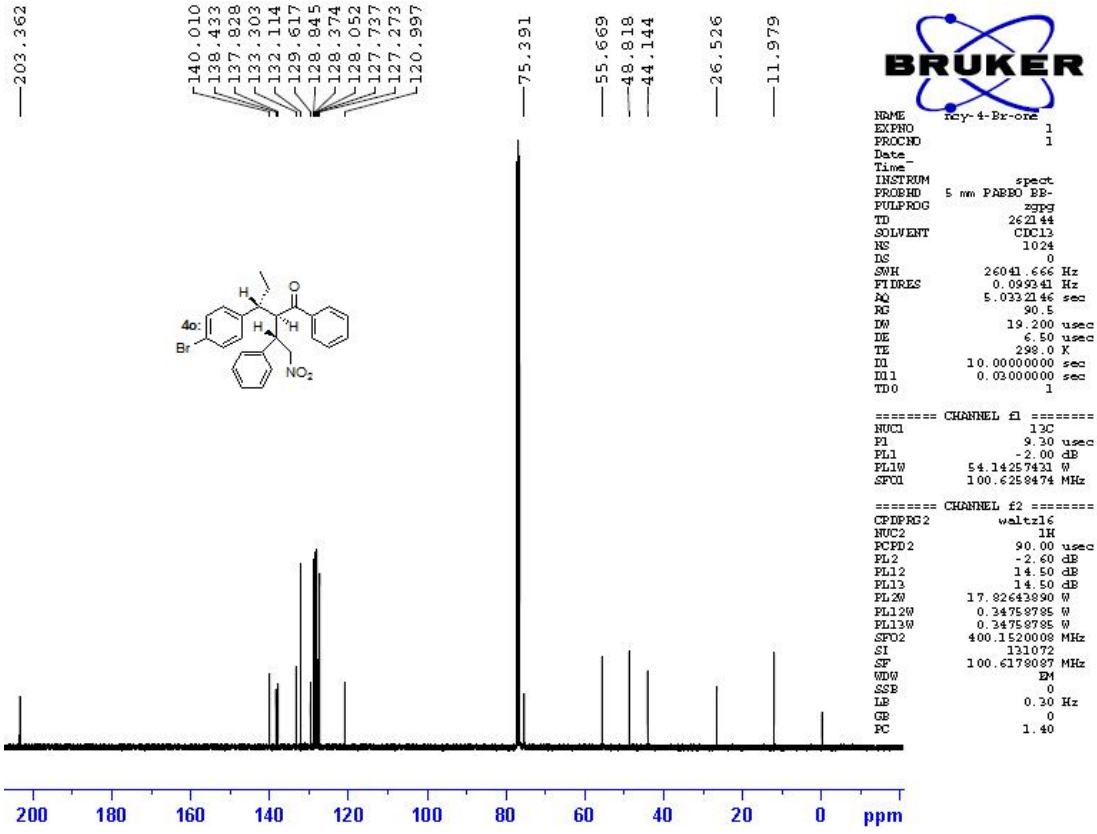

7.487
7.484
7.469
7.466
7.449
7.447
7.377
7.372
7.360
7.355
7.295
7.276
7.273
7.267
7.256
7.187
7.182
7.170
7.166
7.152
7.147
7.141
7.138
7.134
7.130
6.987
6.981
6.968
6.963
5.017
4.989
4.984
4.955
4.896
4.887
4.862
4.108
4.095
4.083
4.071
1.564
0.593
0.575
0.556
BRUKER
NAME ncy-4-Cl-one
EXPNO 1
PROCNO 1
Date_
Time
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 40.3
DW 83.200 usec
DE 6.50 usec
TE 296.9 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4m:
Cl
NO2
8
7
6
5
4
3
2
1
0
ppm
2.97
1.89
2.31
5.07
1.94
1.00
0.99
1.00
1.00
1.01
1.96
2.96

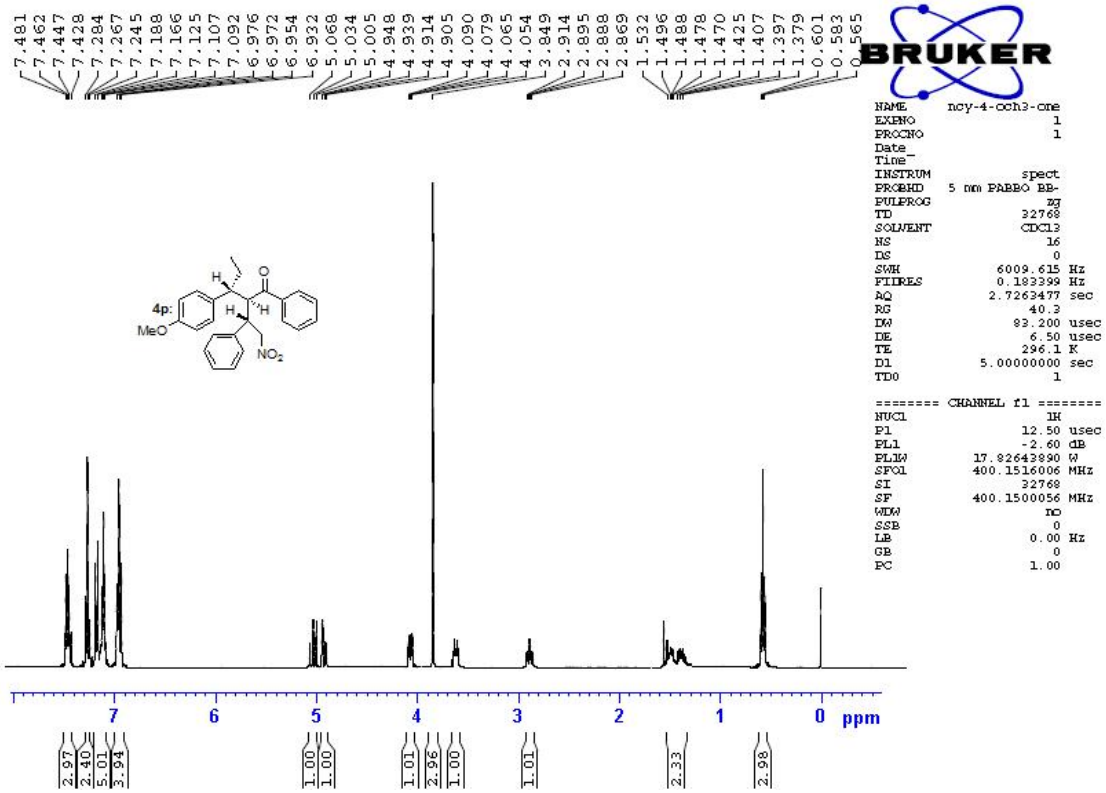

203.406
139.475
138.440
137.838
133.301
132.939
129.246
129.174
128.844
128.372
128.054
127.734
127.272
75.390
55.739
48.743
44.143
26.583
11.982
BRUKER
NAME ncy-4-Cl-one
EXPNO 1
PROCNO 1
Date_
Time
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 1024
DS 0
SWH 26041.666 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 90.5
DW 19.200 usec
DE 6.50 usec
TE 297.2 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
4m:
Cl
NO2
200
180
160
140
120
100
80
60
40
20
0
ppm

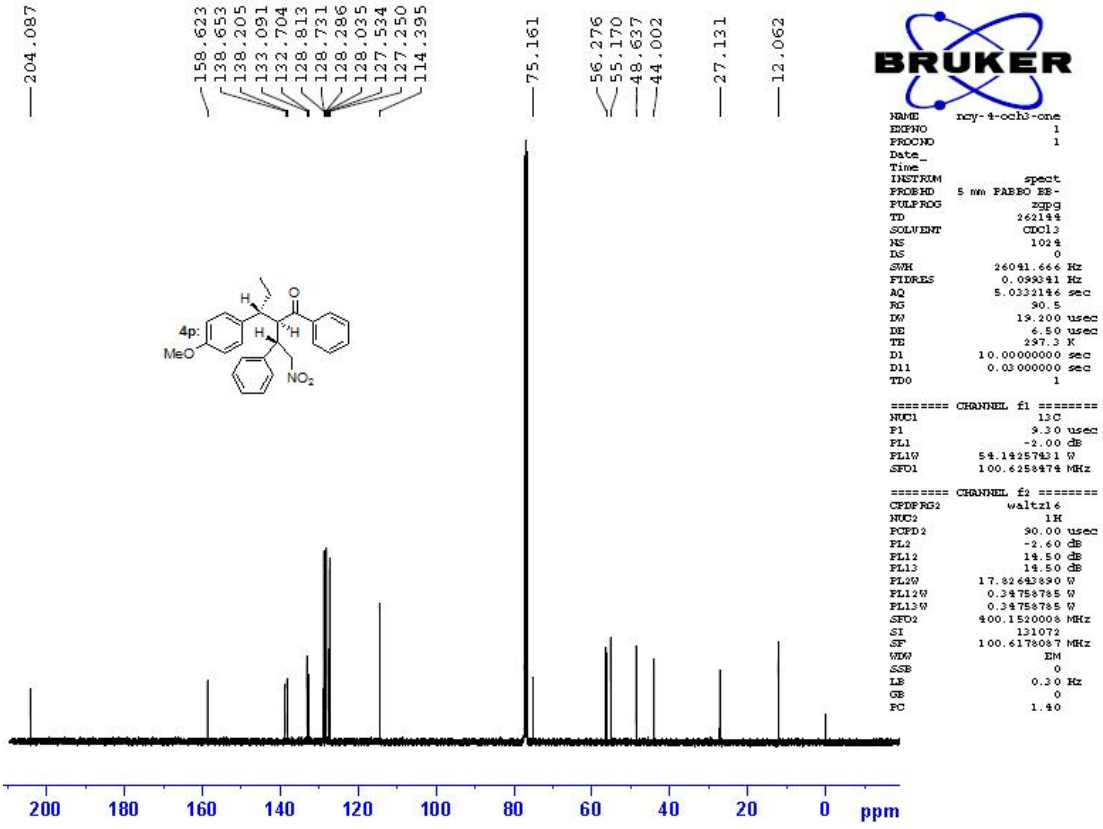

8.231
8.227
8.214
8.210
7.516
7.503
7.498
7.495
7.485
7.470
7.467
7.381
7.376
7.363
7.359
7.309
7.290
7.289
7.267
7.187
7.182
7.180
7.173
7.169
7.162
7.008
7.002
6.997
6.989
6.985
4.951
4.924
4.918
4.891
4.850
4.840
4.817
4.194
4.170
4.162
4.147
0.593
0.575
0.556
BRUKER
NAME ncy-4-NO2-one
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 40.3
DW 83.200 usec
DE 6.50 usec
TE 296.0 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.92643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4n:
O2N
NO2
8
7
6
5
4
3
2
1
0
ppm
1.77
2.88
3.85
3.09
1.87
1.00
0.91
0.90
0.90
0.99
0.95
1.09
2.80

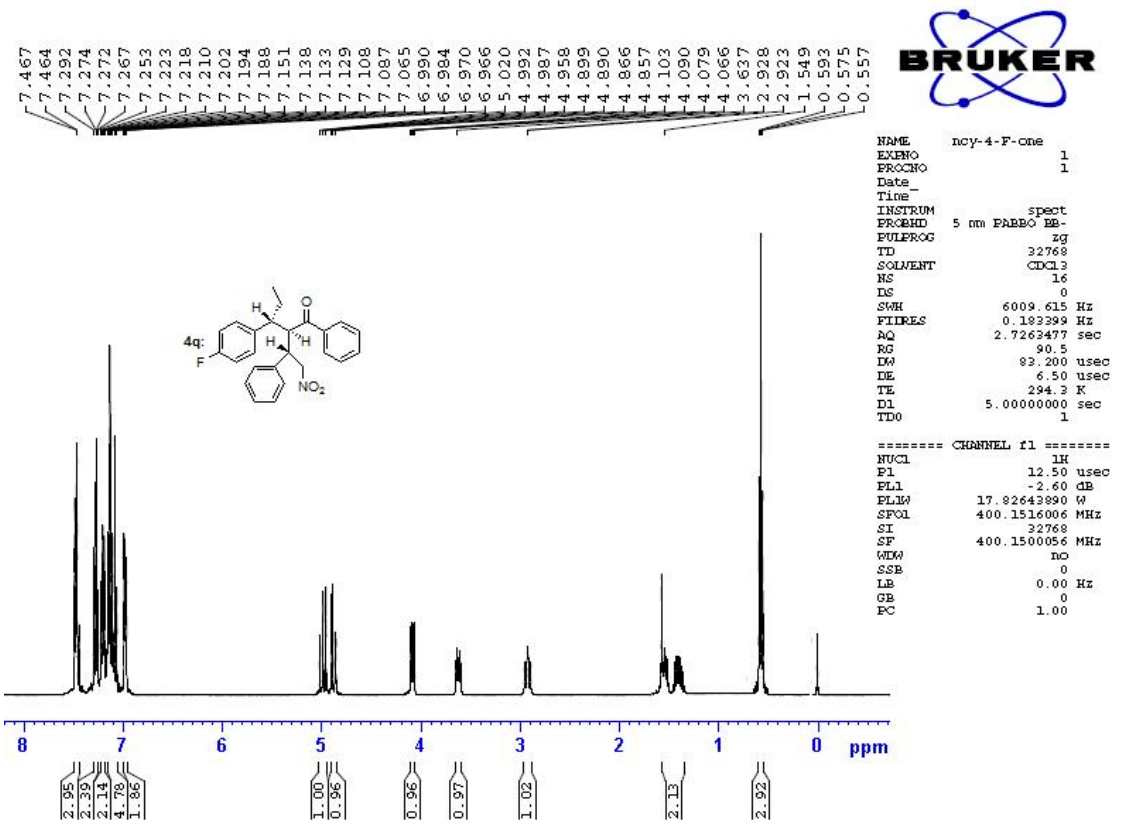

202.496
148.979
147.048
138.185
137.385
133.603
129.000
128.894
128.506
128.068
128.018
127.325
124.108
75.793
55.062
49.081
44.432
25.908
11.946
BRUKER
NAME ncy-4-NO2-one
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 512
DS 0
SWH 26041.666 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 90.5
DW 19.200 usec
DE 6.50 usec
TE 297.1 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257421 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.92643890 W
PL12W 0.34758795 W
PL13W 0.34758795 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
4n:
O2N
NO2
200
180
160
140
120
100
80
60
40
20
0
ppm


7.525
7.504
7.485
7.465
7.451
7.296
7.277
7.267
7.257
7.152
7.135
7.128
7.107
6.985
6.980
6.966
6.963
5.015
4.986
4.981
4.953
4.891
4.882
4.858
4.849
4.104
4.091
4.080
4.067
3.633
3.622
3.615
3.604
2.912
2.907
1.544
1.532
1.527
1.518
1.416
1.388
0.591
0.573
0.554
BRUKER
NAME ncy-4-Br
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183299 Hz
AQ 2.7263477 sec
RG 40.3
DW 83.200 usec
DE 6.50 usec
TE 296.1 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
4o:
Br
NO2
8
7
6
5
4
3
2
1
0
ppm
4.95
2.36
4.97
1.83
1.00
0.99
1.02
1.01
1.03
2.20
3.00


203.362
140.010
138.433
137.828
133.303
132.114
129.617
128.845
128.374
128.052
127.737
127.273
120.997
75.391
55.669
48.818
44.144
26.526
11.979
BRUKER
NAME ncy-4-Br-one
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 1024
DS 0
SWH 26041.666 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 90.5
DW 19.200 usec
DE 6.50 usec
TE 298.0 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
4o:
Br
NO2
200
180
160
140
120
100
80
60
40
20
0
ppm


7.481
7.462
7.447
7.428
7.284
7.267
7.245
7.188
7.166
7.125
7.107
7.092
6.976
6.972
6.954
6.932
5.068
5.034
5.005
4.948
4.939
4.914
4.905
4.090
4.079
4.065
4.054
3.849
2.914
2.895
2.888
2.869
1.532
1.496
1.488
1.478
1.470
1.425
1.407
1.397
1.379
0.601
0.583
0.565
BRUKER
NAME ncy-4-och3-one
EXPNO 1
PROCNO 1
Date_
Time
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7262477 sec
RG 40.3
DW 83.200 usec
DE 6.50 usec
TE 296.1 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4p:
MeO
H
O
H
H
NO2
7
6
5
4
3
2
1
0
ppm
2.97
2.40
5.01
3.94
1.00
1.00
1.01
2.96
1.00
1.01
2.33
2.98


204.087
158.623
138.653
138.205
133.091
132.704
128.813
128.731
128.286
128.035
127.534
127.250
114.395
75.161
56.276
55.170
48.637
44.002
27.131
12.062
BRUKER
NAME ncy-4-och3-one
EXPNO 1
PROCNO 1
Date_
Time
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 1024
DS 0
SWH 26041.666 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 90.5
DW 19.200 usec
DE 6.50 usec
TE 297.3 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
4p:
MeO
H
O
H
H
NO2
200
180
160
140
120
100
80
60
40
20
0
ppm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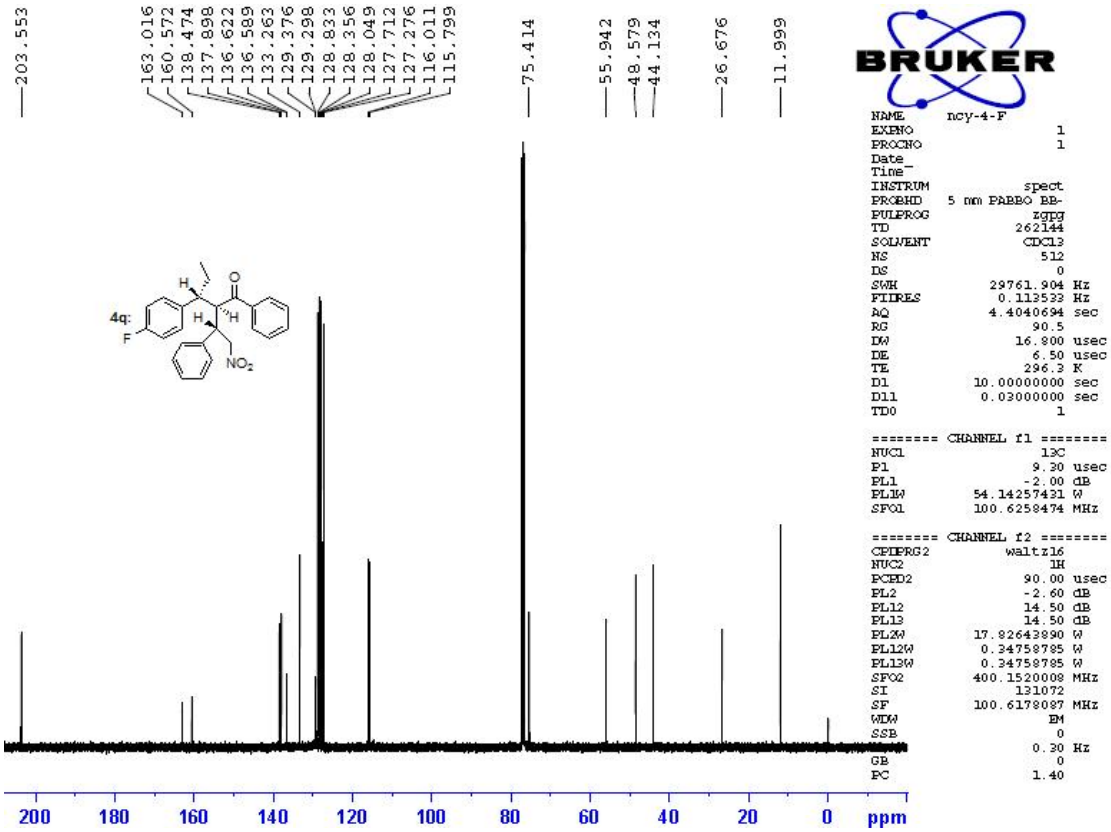
7.464
7.292
7.274
7.272
7.267
7.253
7.223
7.218
7.210
7.202
7.194
7.188
7.151
7.138
7.133
7.129
7.108
7.087
7.065
6.990
6.984
6.970
6.966
5.020
4.992
4.987
4.958
4.899
4.890
4.866
4.857
4.103
4.090
4.079
4.066
3.637
2.928
2.923
1.549
0.593
0.575
0.557
BRUKER
NAME ncy-4-F-one
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 90.5
DW 83.200 usec
DE 6.50 usec
TE 294.3 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4q:
F
NO2
8
7
6
5
4
3
2
1
0
ppm
2.95
2.39
2.14
4.78
1.86
1.00
0.96
0.96
0.97
1.02
2.13
2.92


203.553
163.016
160.572
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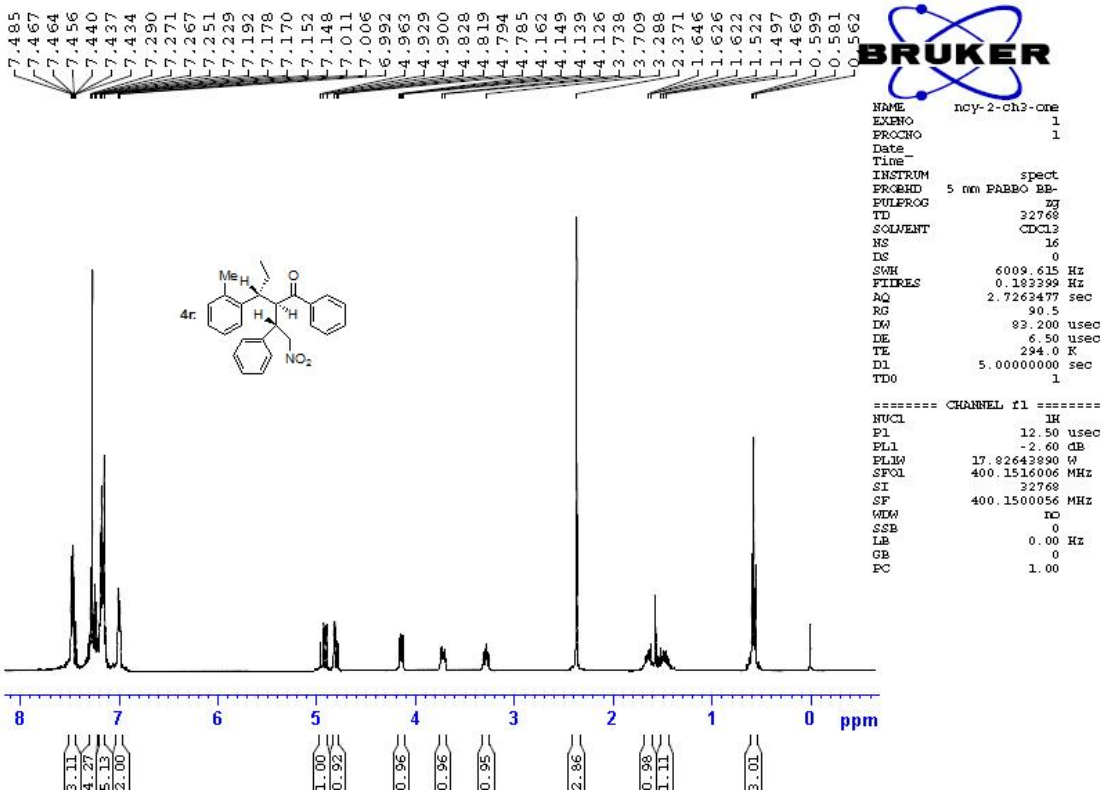
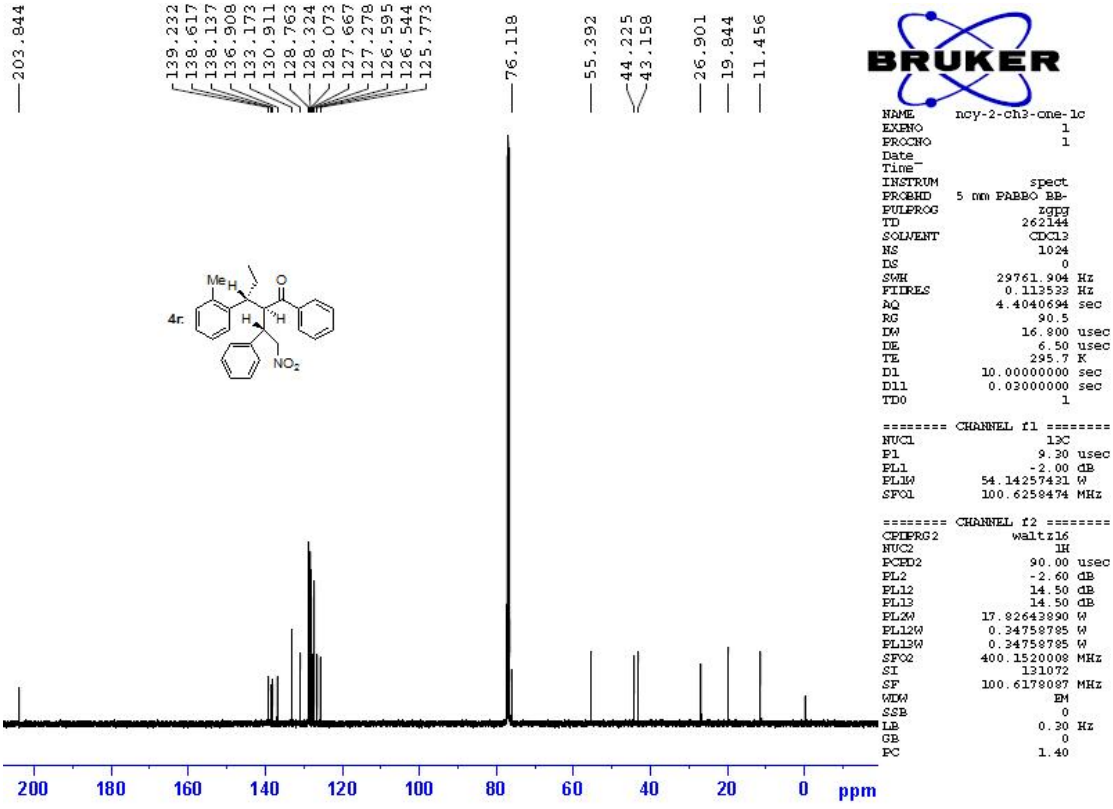
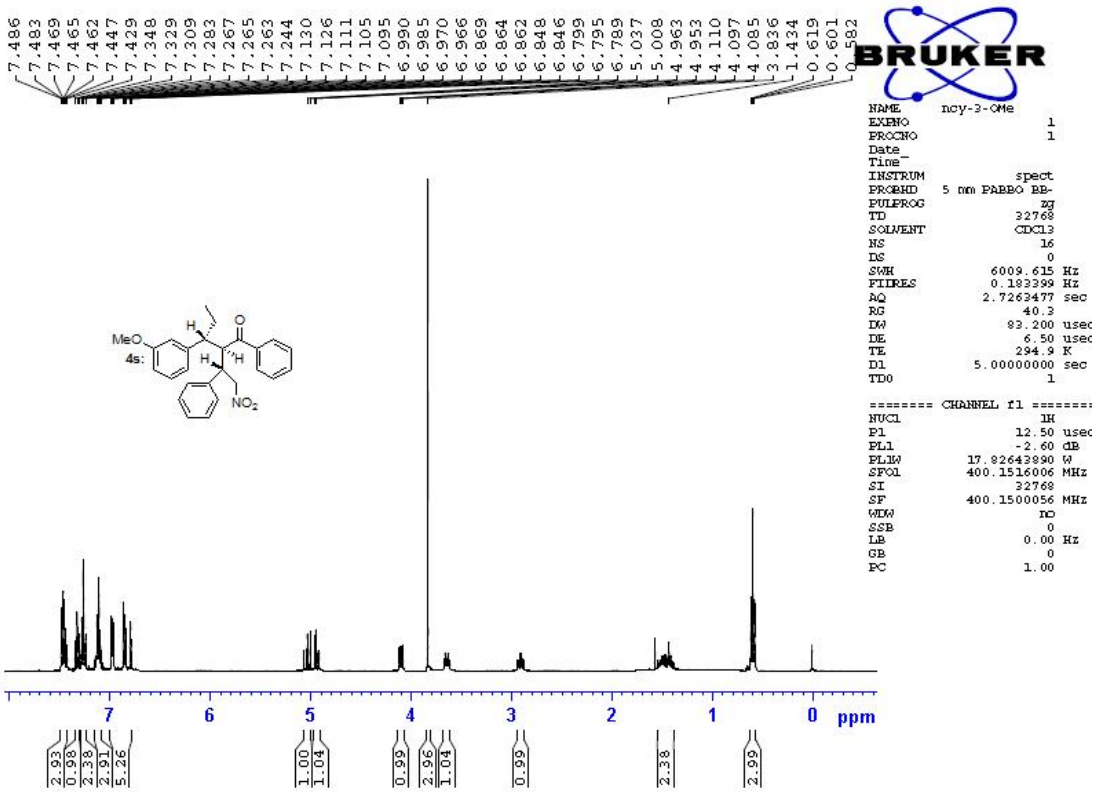
137.898
136.622
136.589
133.263
129.376
129.298
128.833
128.356
128.049
127.712
127.276
116.011
115.799
75.414
55.942
48.579
44.134
26.676
11.999
BRUKER
NAME ncy-4-F
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 512
DS 0
SWH 29761.904 Hz
FIDRES 0.113533 Hz
AQ 4.4040694 sec
RG 90.5
DW 16.800 usec
DE 6.50 usec
TE 296.3 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
4q:
F
NO2
200
180
160
140
120
100
80
60
40
20
0
ppm




```
NAME                ncy-2-ch3-one
EXPNO                           1
PROCNO                          1
Date_
Time
INSTRUM                      spect
PROBHD    5 mm PABBO BB-
PULPROG                        zg
TD                          32768
SOLVENT                     CDCl3
NS                             16
DS                              0
SWH                    6009.615 Hz
FIDRES                 0.183399 Hz
AQ                     2.7263477 sec
RG                          90.5
DW                        83.200 usec
DE                         6.50 usec
TE                        294.0 K
D1                     5.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                        12.50 usec
PL1                       -2.60 dB
PL1W                17.82643890 W
SFO1                400.1516006 MHz
SI                          32768
SF                  400.1500056 MHz
WDW                            no
SSB                             0
LB                         0.00 Hz
GB                              0
PC                         1.00
```




```
NAME             ncy-2-ch3-one-1c
EXPNO                           1
PROCNO                          1
Date_
Time
INSTRUM                      spect
PROBHD    5 mm PABBO BB-
PULPROG                     zgpg
TD                         262144
SOLVENT                     CDCl3
NS                           1024
DS                              0
SWH                   29761.904 Hz
FIDRES                 0.113533 Hz
AQ                     4.4040694 sec
RG                          90.5
DW                        16.800 usec
DE                         6.50 usec
TE                        295.7 K
D1                    10.00000000 sec
D11                    0.03000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                          13C
P1                         9.30 usec
PL1                       -2.00 dB
PL1W                54.14257431 W
SFO1                100.6258474 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                           1H
PCPD2                     90.00 usec
PL2                       -2.60 dB
PL12                      14.50 dB
PL13                      14.50 dB
PL2W                17.82643890 W
PL12W                0.34758785 W
PL13W                0.34758785 W
SFO2                400.1520008 MHz
SI                         121072
SF                  100.6178087 MHz
WDW                            EM
SSB                             0
LB                         0.30 Hz
GB                              0
PC                         1.40
```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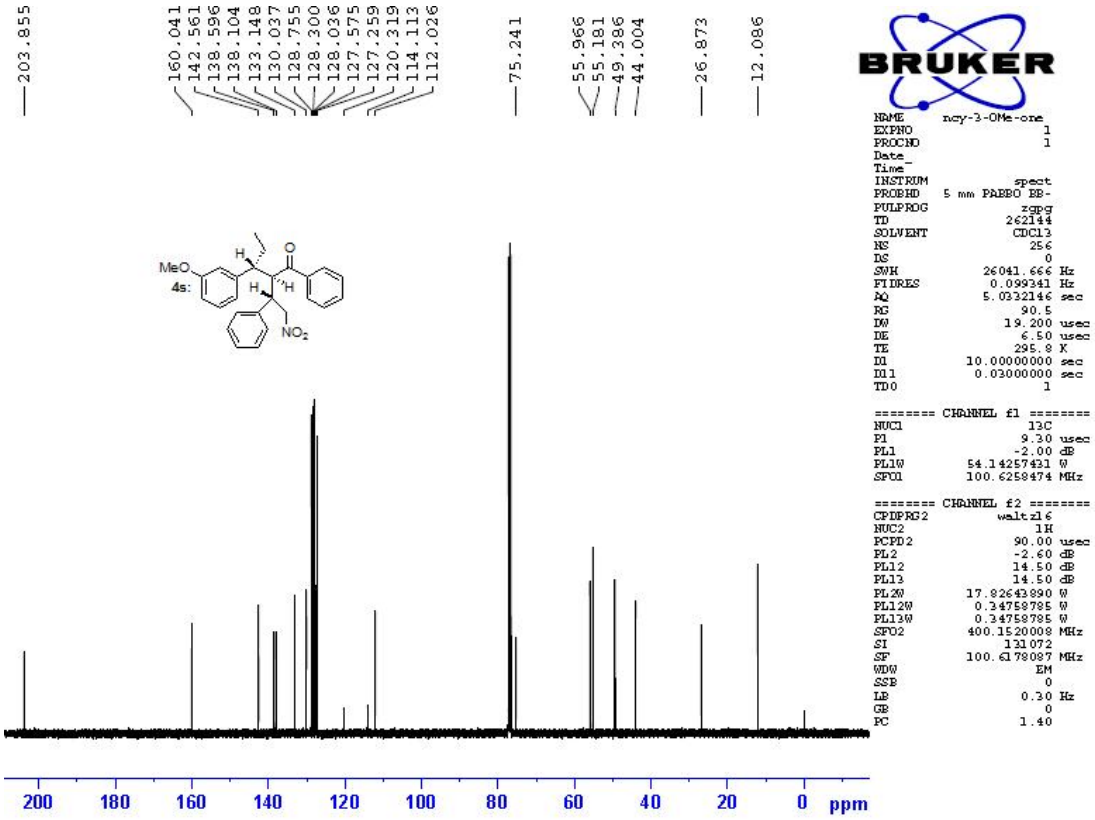
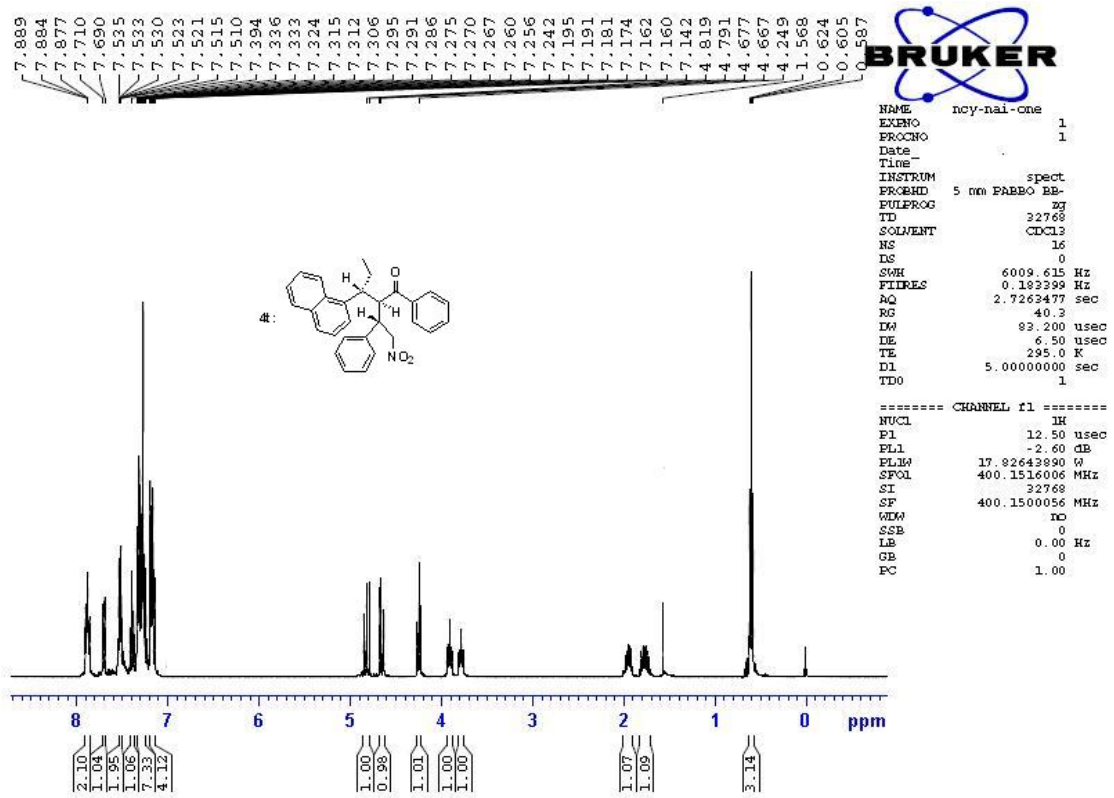
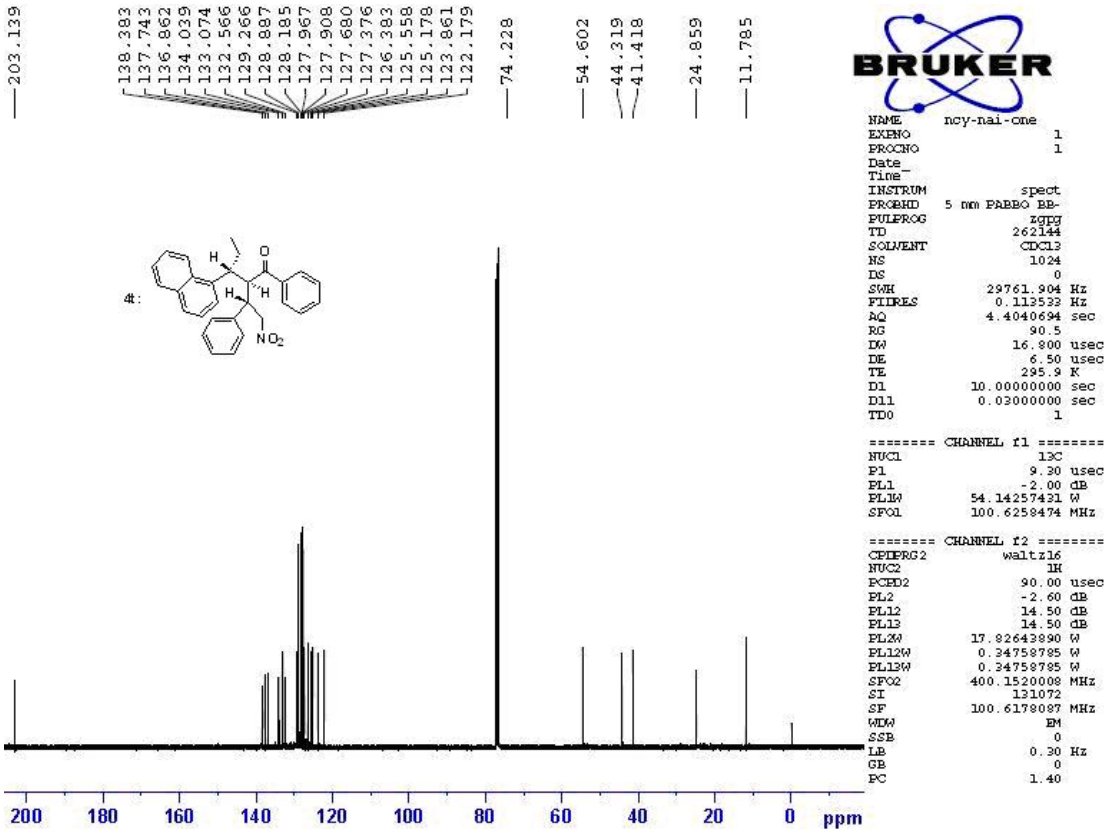
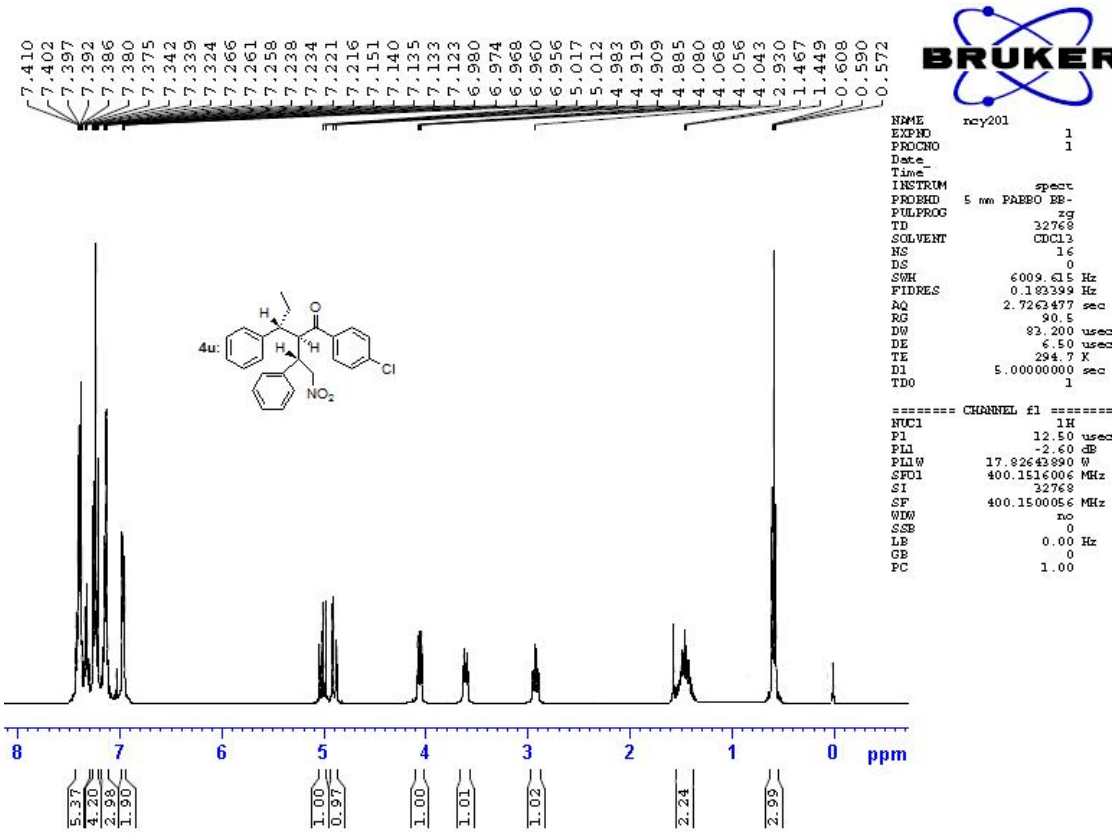
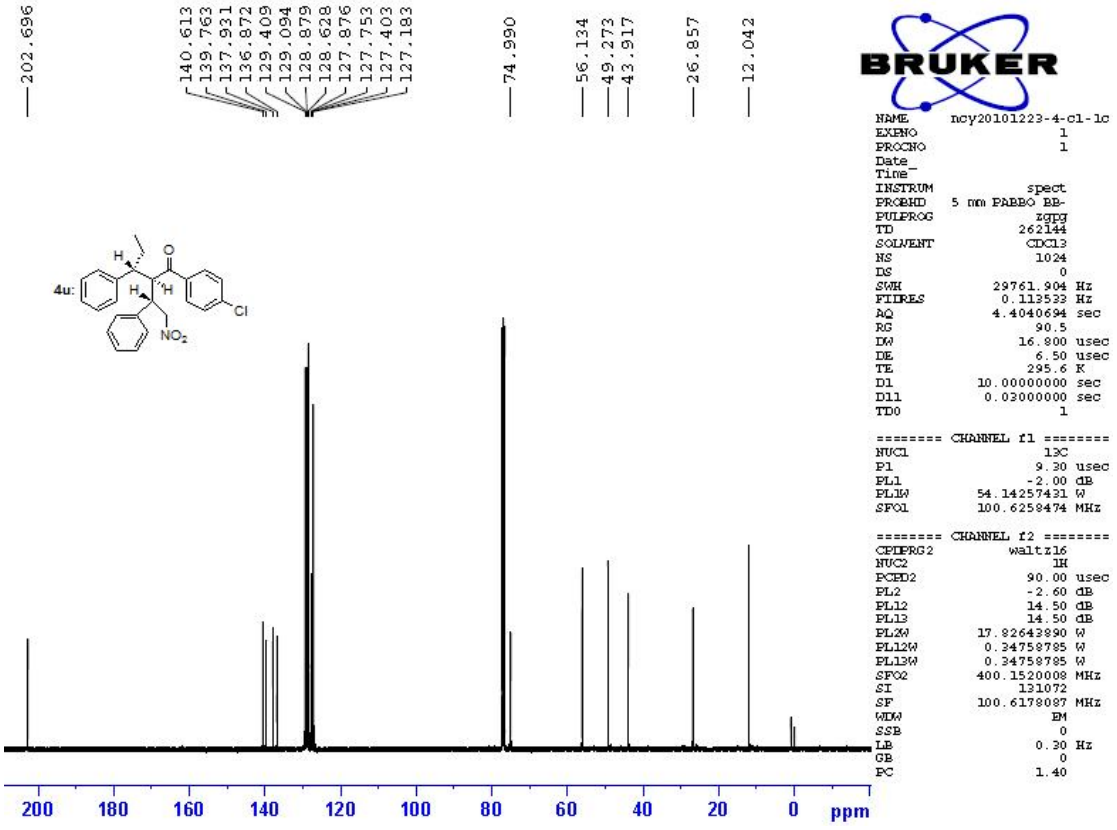
7.483
7.469
7.465
7.462
7.447
7.429
7.348
7.329
7.309
7.283
7.267
7.265
7.263
7.244
7.130
7.126
7.111
7.105
7.095
6.990
6.985
6.970
6.966
6.869
6.864
6.862
6.848
6.846
6.799
6.795
6.789
5.037
5.008
4.963
4.953
4.110
4.097
4.085
3.836
1.434
0.619
0.601
0.582
BRUKER
NAME ncy-3-OMe
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 40.3
DW 83.200 usec
DE 6.50 usec
TE 294.9 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
MeO
4s:
H
O
H
H
NO2
7
6
5
4
3
2
1
0
ppm
2.93
0.98
2.38
2.91
5.26
1.00
1.04
0.99
2.96
1.04
0.99
2.38
2.99

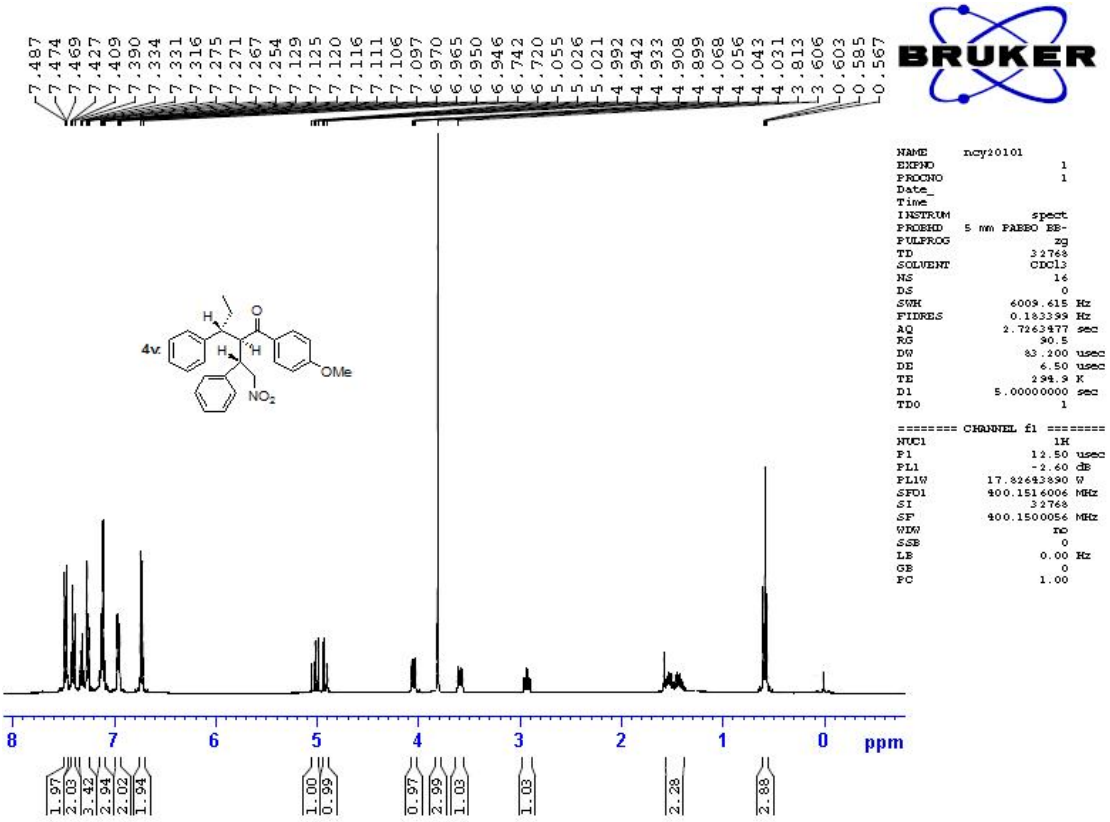

203.855
160.041
142.561
138.596
138.104
133.148
130.037
128.755
128.300
128.036
127.575
127.259
120.319
114.113
112.026
75.241
55.966
55.181
49.386
44.004
26.873
12.086
BRUKER
NAME ncy-3-OMe-one
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 256
DS 0
SWH 26041.666 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 90.5
DW 19.200 usec
DE 6.50 usec
TE 295.8 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
MeO
4s:
H
O
H
H
NO2
200
180
160
140
120
100
80
60
40
20
0
ppm

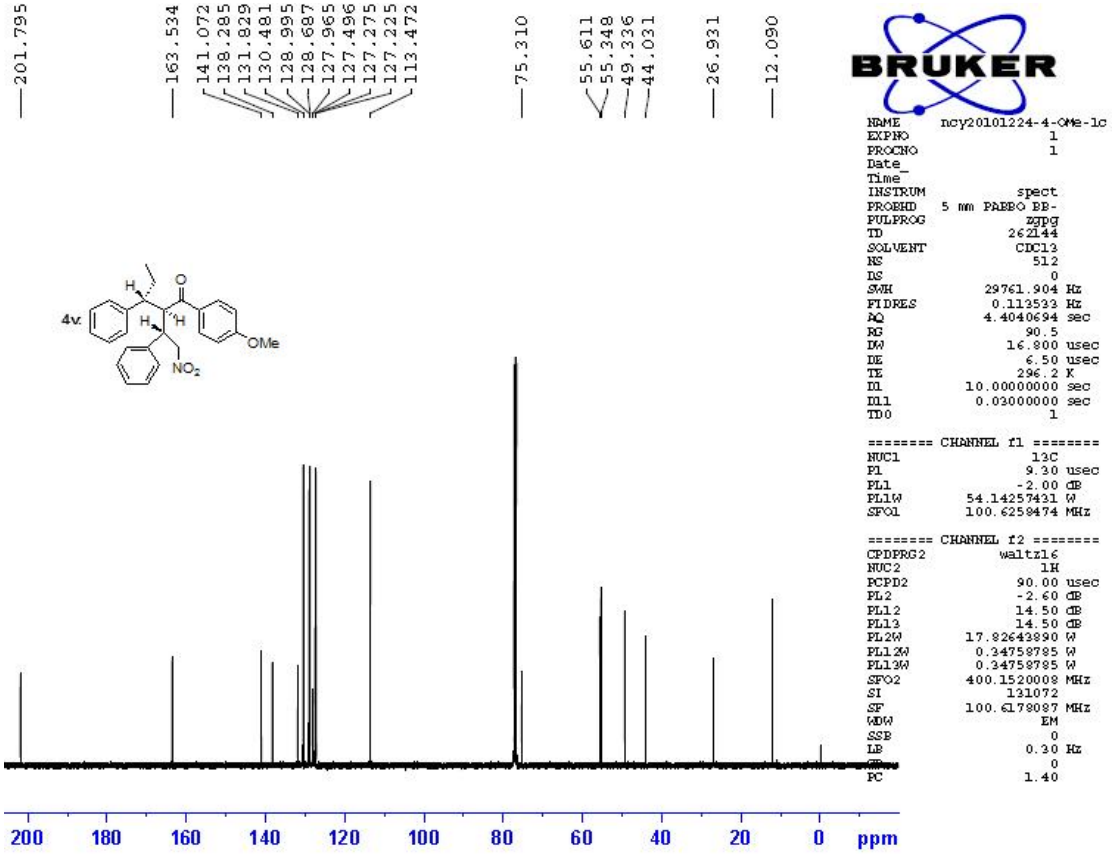

7.889
7.884
7.877
7.710
7.690
7.535
7.533
7.530
7.523
7.521
7.515
7.510
7.394
7.336
7.333
7.324
7.315
7.312
7.306
7.295
7.291
7.286
7.275
7.270
7.267
7.260
7.256
7.242
7.195
7.191
7.181
7.174
7.162
7.160
7.142
4.819
4.791
4.677
4.667
4.249
1.568
0.624
0.605
0.587
BRUKER
NAME ncy-nai-one
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 40.3
DW 83.200 usec
DE 6.50 usec
TE 295.0 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4t:
H
O
H
H
NO2
8
7
6
5
4
3
2
1
0
ppm
2.10
1.04
1.95
1.06
7.33
4.12
1.00
0.98
1.01
1.00
1.00
1.07
1.09
3.14

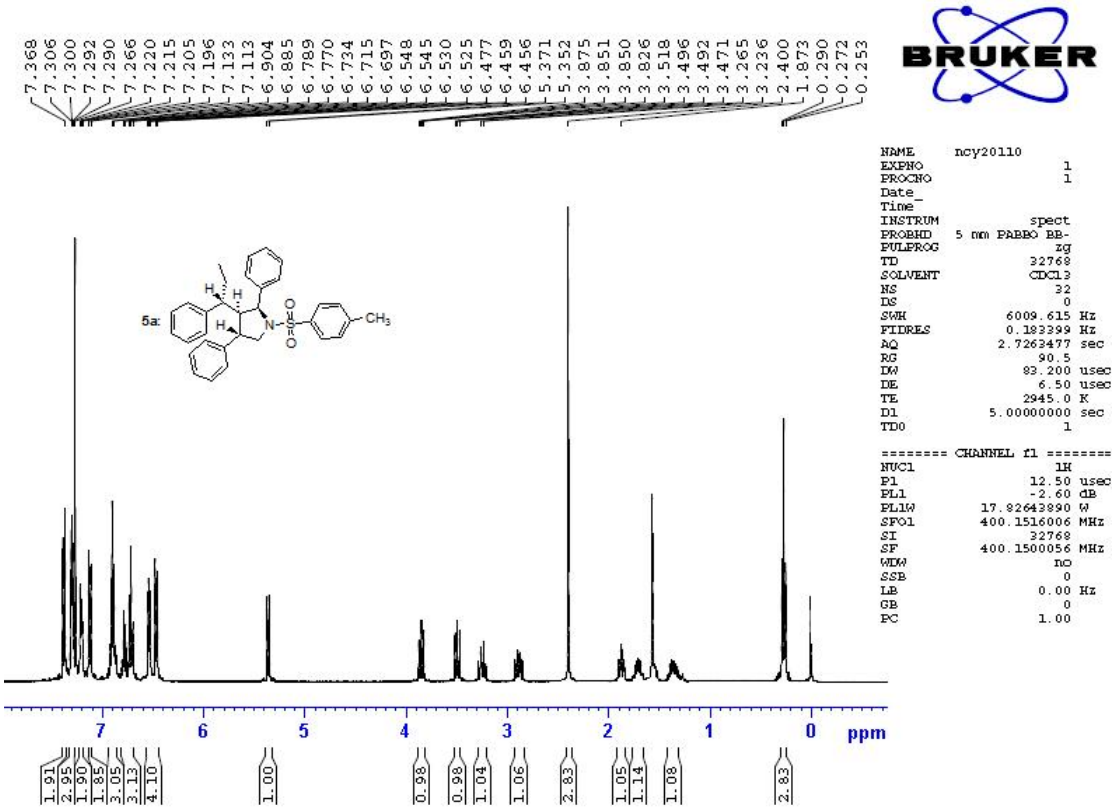

203.139
138.383
137.743
136.862
134.039
133.074
132.566
129.266
128.887
128.185
127.967
127.908
127.680
127.376
126.383
125.558
125.178
123.861
122.179
74.228
54.602
44.319
41.418
24.859
11.785
BRUKER
NAME ncy-nai-one
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 1024
DS 0
SWH 29761.904 Hz
FIDRES 0.113533 Hz
AQ 4.4040694 sec
RG 90.5
DW 16.800 usec
DE 6.50 usec
TE 295.9 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.20 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
4t:
H
O
H
H
NO2
200
180
160
140
120
100
80
60
40
20
0
ppm

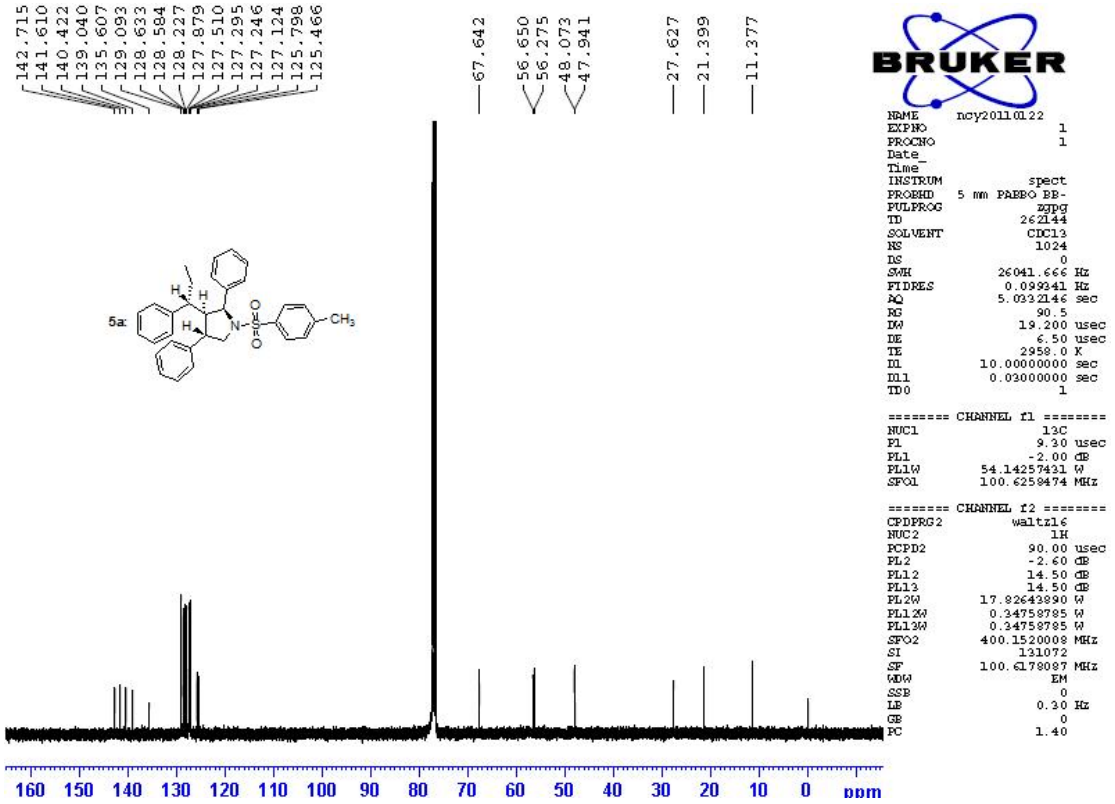

7.410
7.402
7.397
7.392
7.386
7.380
7.375
7.342
7.339
7.324
7.266
7.261
7.258
7.238
7.234
7.221
7.216
7.151
7.140
7.135
7.133
7.123
6.980
6.974
6.968
6.960
6.956
5.017
5.012
4.983
4.919
4.909
4.885
4.080
4.068
4.056
4.043
2.930
1.467
1.449
0.608
0.590
0.572
BRUKER
NAME ncy201
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 90.5
DW 83.200 usec
DE 6.50 usec
TE 294.7 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4u:
Cl
NO2
8
7
6
5
4
3
2
1
0
ppm
5.37
4.20
2.98
1.90
1.00
0.97
1.00
1.01
1.02
2.24
2.99

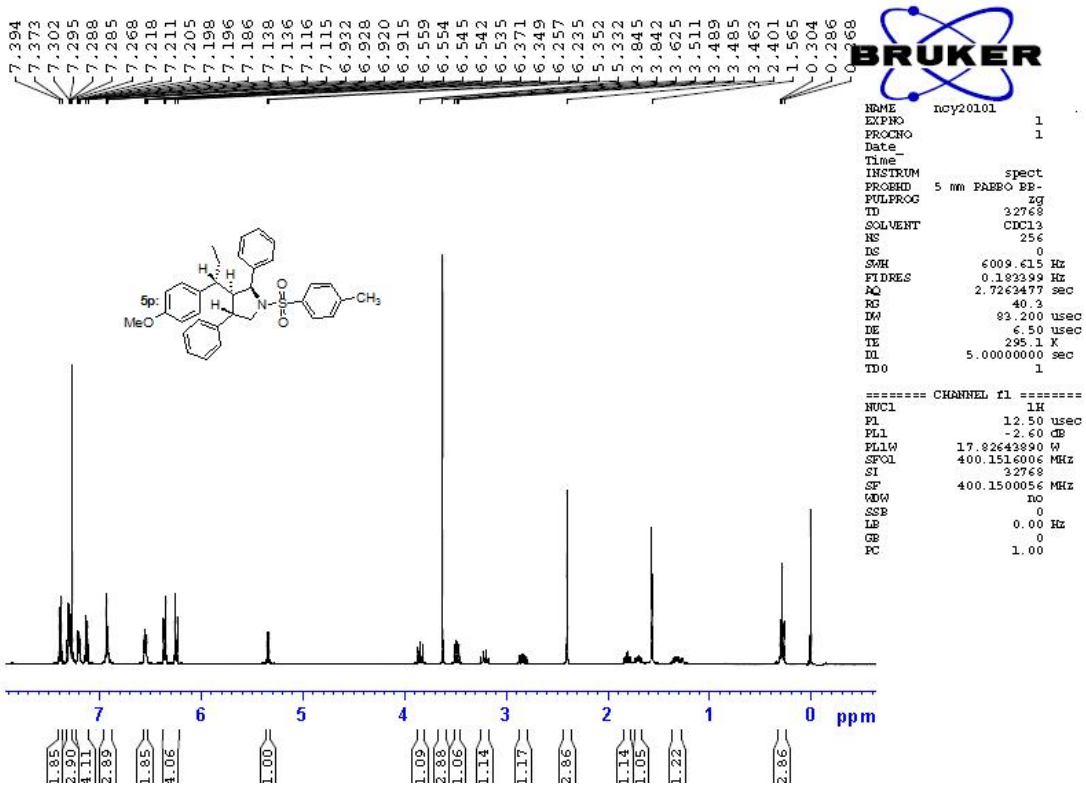

202.696
140.613
139.763
137.931
136.872
129.409
129.094
128.879
128.628
127.876
127.753
127.403
127.183
74.990
56.134
49.273
43.917
26.857
12.042
BRUKER
NAME ncy20101223-4-cl-1c
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 1024
DS 0
SWH 29761.904 Hz
FIDRES 0.113533 Hz
AQ 4.4040694 sec
RG 90.5
DW 16.800 usec
DE 6.50 usec
TE 295.6 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
4u:
Cl
NO2
200
180
160
140
120
100
80
60
40
20
0
ppm

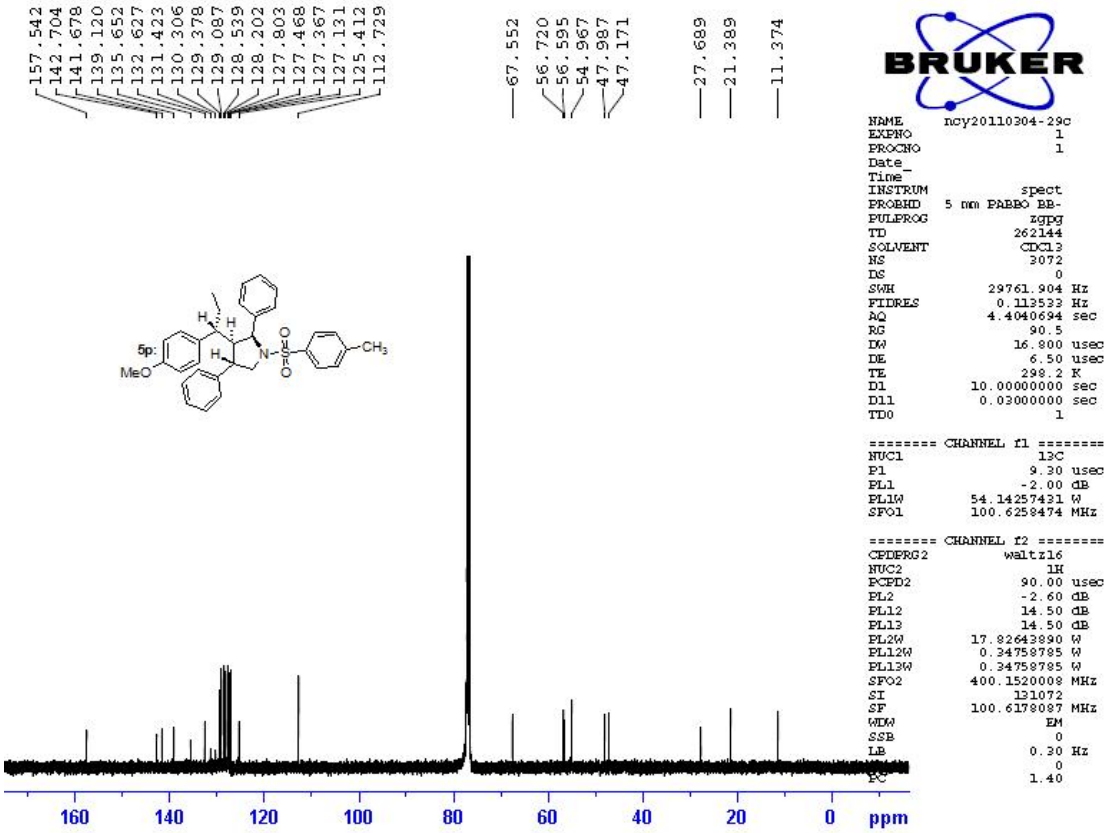

7.487
7.474
7.469
7.427
7.409
7.390
7.334
7.331
7.316
7.275
7.271
7.267
7.254
7.129
7.125
7.120
7.116
7.111
7.106
7.097
6.970
6.965
6.950
6.946
6.742
6.720
5.055
5.026
5.021
4.992
4.942
4.933
4.908
4.899
4.068
4.056
4.043
4.031
3.813
3.606
0.603
0.585
0.567
BRUKER
4v.
OMe
NO2
NAME ncy20101
EXPNO 1
PROCNO 1
Date_
Time
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 90.5
DW 83.200 usec
DE 6.50 usec
TE 294.9 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
0
ppm
1.97
2.03
3.42
2.94
2.02
1.94
1.00
0.99
0.97
2.99
1.03
1.03
2.28
2.88


201.795
163.534
141.072
138.285
131.829
130.481
128.995
128.687
127.965
127.496
127.275
127.225
113.472
75.310
55.611
55.348
49.336
44.031
26.931
12.090
BRUKER
4v.
OMe
NO2
NAME ncy20101224-4-OMe-1c
EXPNO 1
PROCNO 1
Date_
Time
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 512
DS 0
SWH 29761.904 Hz
FIDRES 0.113533 Hz
AQ 4.4040694 sec
RG 90.5
DW 16.800 usec
DE 6.50 usec
TE 296.2 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

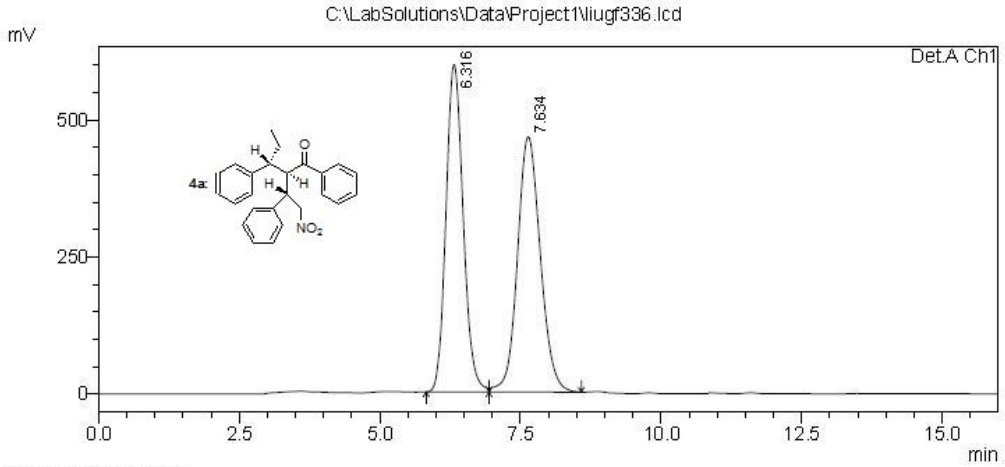

7.368
7.306
7.300
7.292
7.290
7.266
7.220
7.215
7.205
7.196
7.133
7.113
6.904
6.885
6.789
6.770
6.734
6.715
6.697
6.548
6.545
6.530
6.525
6.477
6.459
6.456
5.371
5.352
3.875
3.851
3.850
3.826
3.518
3.496
3.492
3.471
3.265
3.236
2.400
1.873
0.290
0.272
0.253
BRUKER
NAME ncy20110
EXPNO 1
PROCNO 1
Date_
Time
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 90.5
DW 83.200 usec
DE 6.50 usec
TE 2945.0 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
5a:
CH3
7
6
5
4
3
2
1
0
ppm
1.91
2.95
1.90
1.85
3.05
3.13
4.10
1.00
0.98
0.98
1.04
1.06
2.83
1.05
1.14
1.08
2.83


142.715
141.610
140.422
139.040
135.607
129.093
128.633
128.584
128.227
127.879
127.510
127.295
127.246
127.124
125.798
125.466
67.642
56.650
56.275
48.073
47.941
27.627
21.399
11.377
BRUKER
NAME ncy2011022
EXPNO 1
PROCNO 1
Date_
Time
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 1024
DS 0
SWH 26041.666 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 90.5
DW 19.200 usec
DE 6.50 usec
TE 2958.0 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
5a:
CH3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.394 7.373 7.302 7.295 7.288 7.285 7.268 7.218 7.211 7.205 7.198 7.196 7.186 7.138 7.136 7.116 7.115 6.932 6.928 6.920 6.915 6.559 6.554 6.545 6.542 6.535 6.371 6.349 6.257 6.235 5.352 5.332 3.845 3.842 3.625 3.511 3.489 3.485 3.463 2.401 1.565 0.304 0.286
BRUKER
NAME ncy20101
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 256
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7262477 sec
RG 40.3
DW 83.200 usec
DE 6.50 usec
TE 295.1 K
D1 5.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.60 dB
PL1W 17.82643890 W
SFO1 400.1516006 MHz
SI 32768
SF 400.1500056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
5p:
MeO
CH3
7 6 5 4 3 2 1 0 ppm
1.85 2.90 4.11 2.89 1.85 4.06 1.00 1.09 2.88 1.06 1.14 1.17 2.86 1.14 1.05 1.22 2.86


157.542 142.704 141.678 139.120 135.652 132.627 131.423 130.306 129.378 129.087 128.539 128.202 127.803 127.468 127.367 127.131 125.412 112.729
67.552 56.720 56.595 54.967 47.987 47.171 27.689 21.389 11.374
BRUKER
NAME ncy20110304-29c
EXPNO 1
PROCNO 1
Date_
Time_
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 262144
SOLVENT CDCl3
NS 3072
DS 0
SWH 29761.904 Hz
FIDRES 0.113523 Hz
AQ 4.4040694 sec
RG 90.5
DW 16.800 usec
DE 6.50 usec
TE 298.2 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -2.00 dB
PL1W 54.14257431 W
SFO1 100.6258474 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 17.82643890 W
PL12W 0.34758785 W
PL13W 0.34758785 W
SFO2 400.1520008 MHz
SI 131072
SF 100.6178087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
5p:
MeO
CH3
160 140 120 100 80 60 40 20 0 ppm

HPLC spectra for compounds 4a-v, 5a, 5p

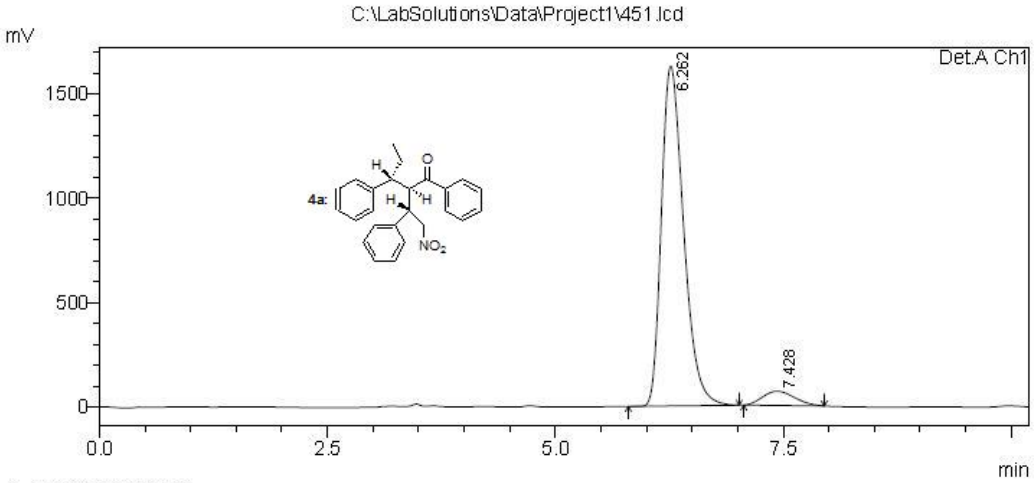


1 Det.A Ch1/254nm

PeakTable

De'ector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	6.316	12703790	597613	49.049
2	7.634	13196419	466159	50.951
Total		25900209	1063771	100.000

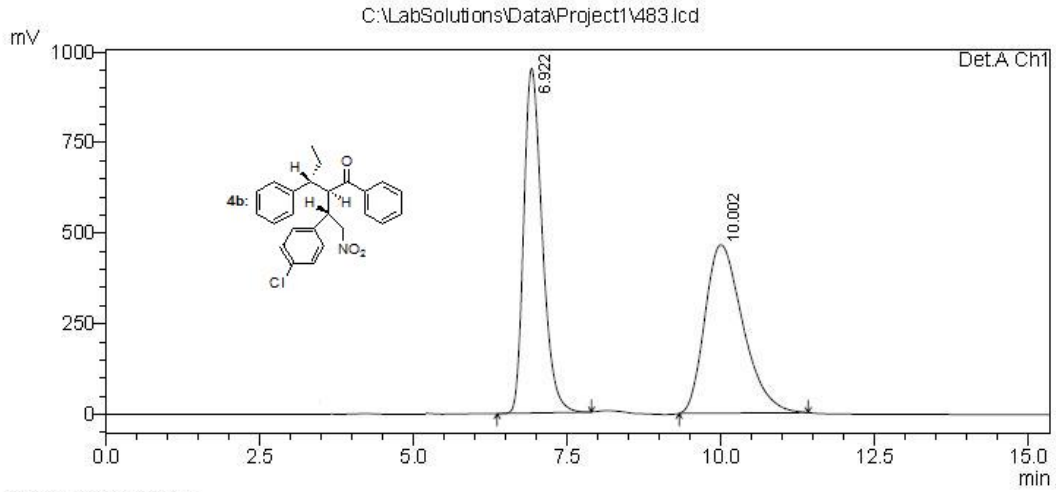


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

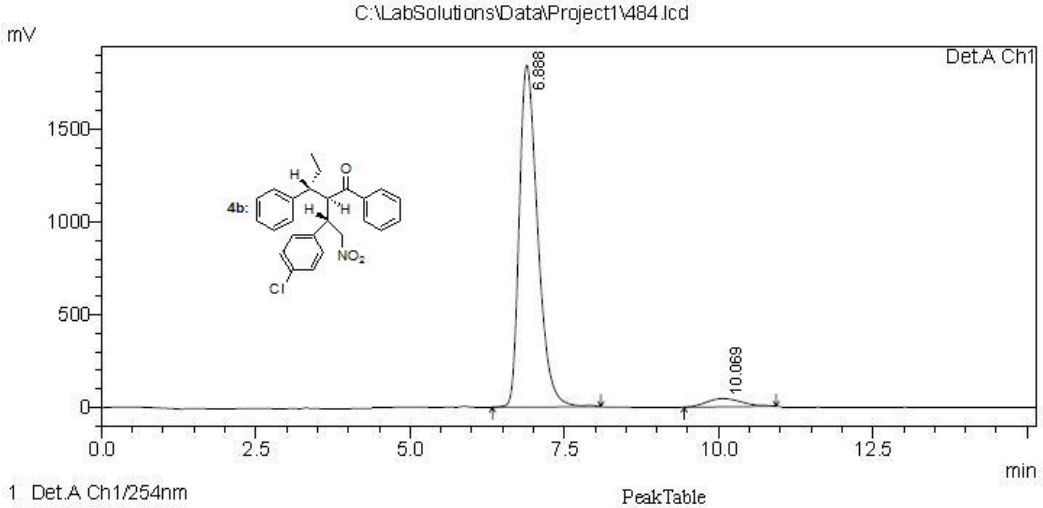
Peak#	Ret. Time	Area	Height	Area %
1	6.262	27582640	1627588	94.315
2	7.428	1662524	67958	5.685
Total		29245164	1695546	100.000



PeakTable

Detector A Ch1 254nm

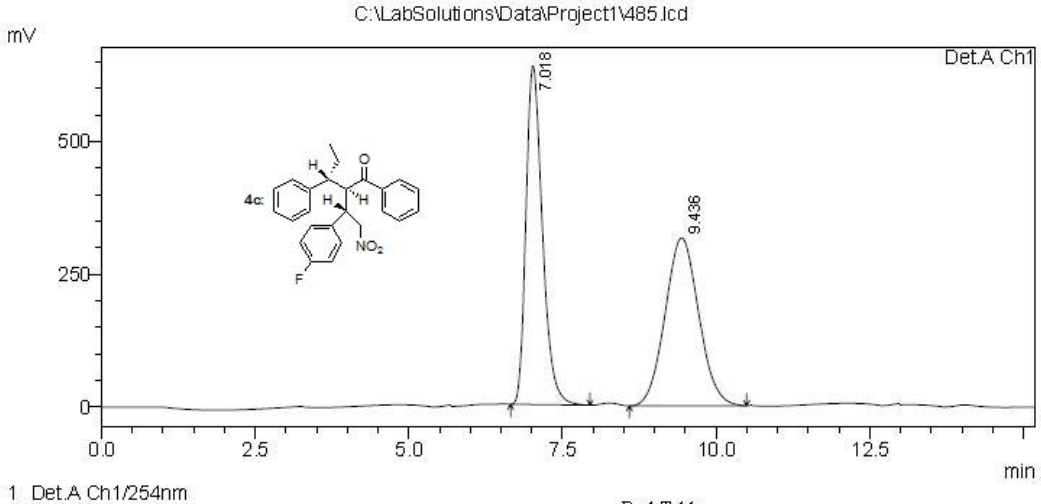
Peak#	Ret. Time	Area	Height	Area %
1	6.922	19829186	950781	49.794
2	10.002	19992956	464422	50.206
Total		39822142	1415203	100.000



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	6.888	38252777	1841067	95.572
2	10.069	1772149	44228	4.428
Total		40024925	1885295	100.000

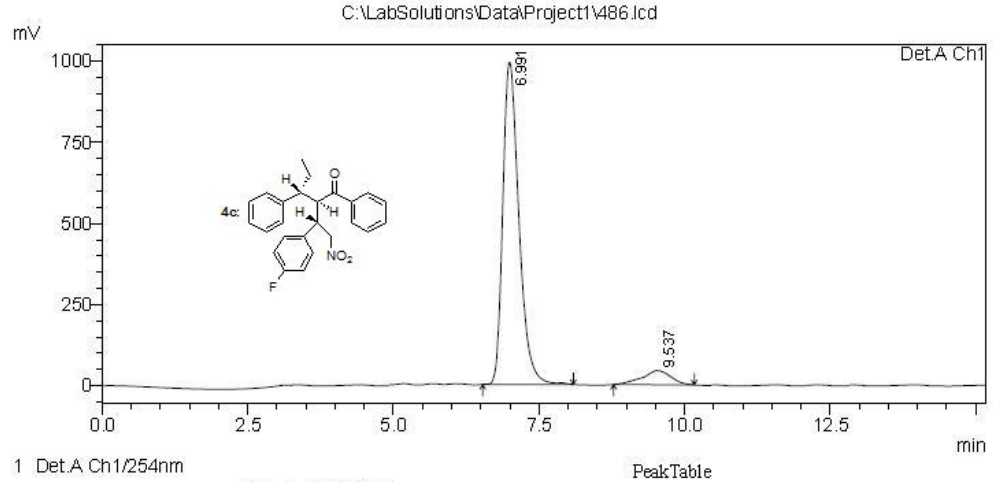


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	7.018	11876958	637190	49.681
2	9.436	12029646	316050	50.319
Total		23906604	953241	100.000

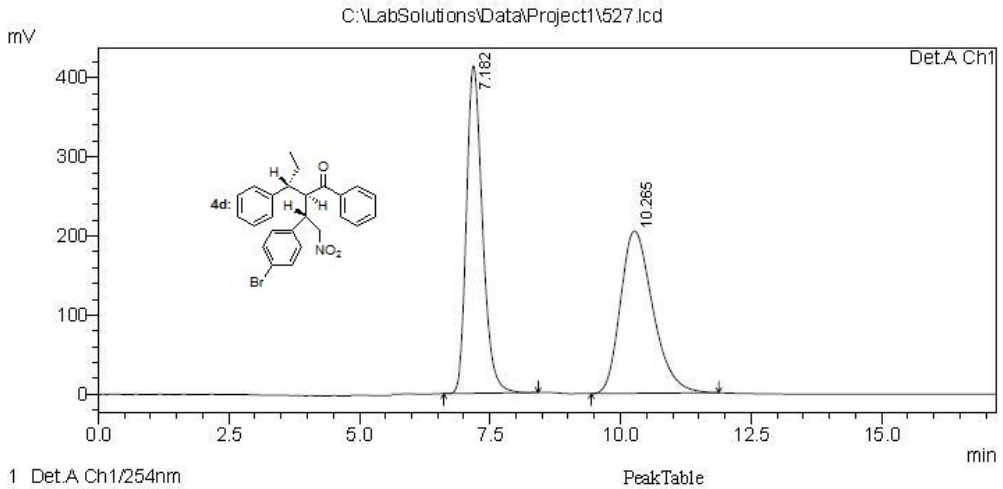


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	6.991	19135744	993658	92.732
2	9.537	1499726	43605	7.268
Total		20635470	1037263	100.000

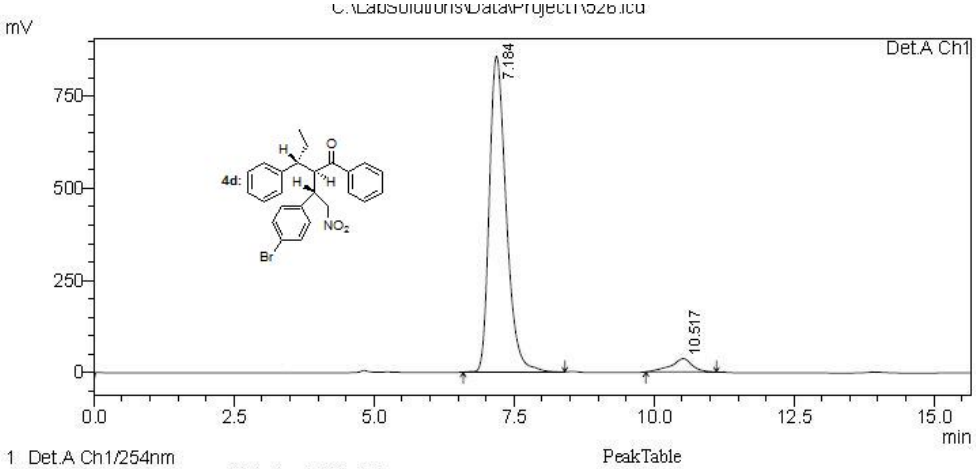


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	7.182	8838201	413596	49.898
2	10.265	8874161	205281	50.102
Total		17712362	618877	100.000

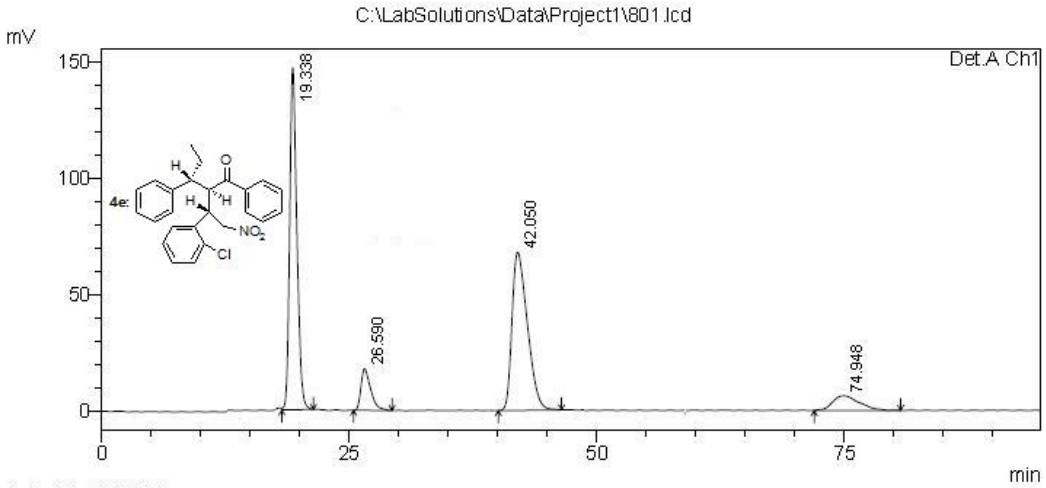


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	7.184	17985525	857000	94.929
2	10.517	960776	36141	5.071
Total		18946300	893141	100.000

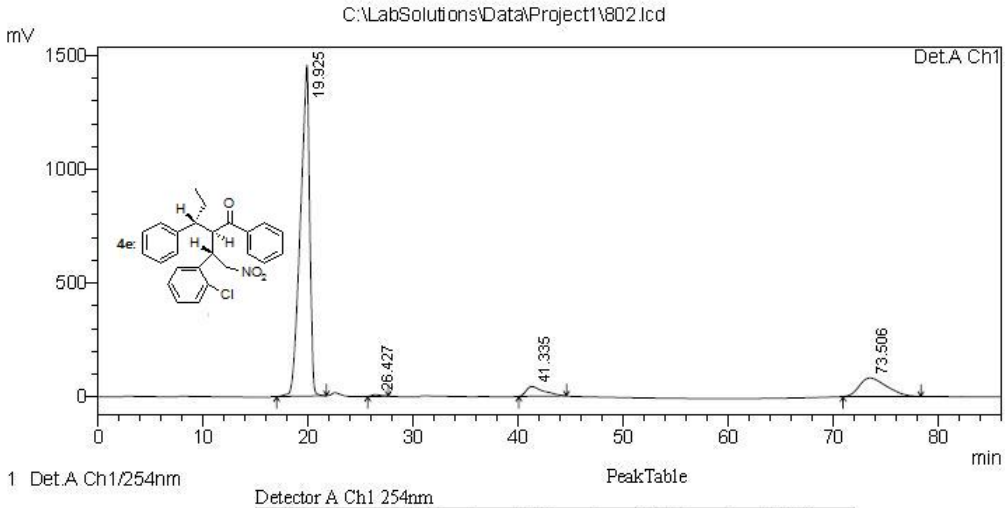


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	19.338	7366582	147017	42.967
2	26.590	1208654	17829	7.050
3	42.050	7361545	67998	42.937
4	74.948	1208151	6368	7.047
Total		17144931	239211	100.000

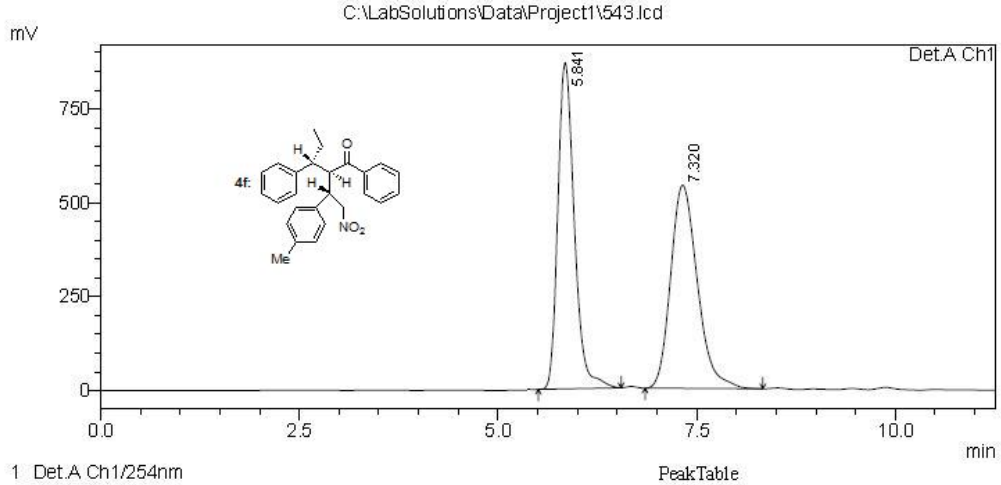


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	19.925	88620123	1453605	81.405
2	26.427	445289	7236	0.409
3	41.335	5042108	43056	4.632
4	73.506	14755715	81543	13.554
Total		108863235	1585440	100.000

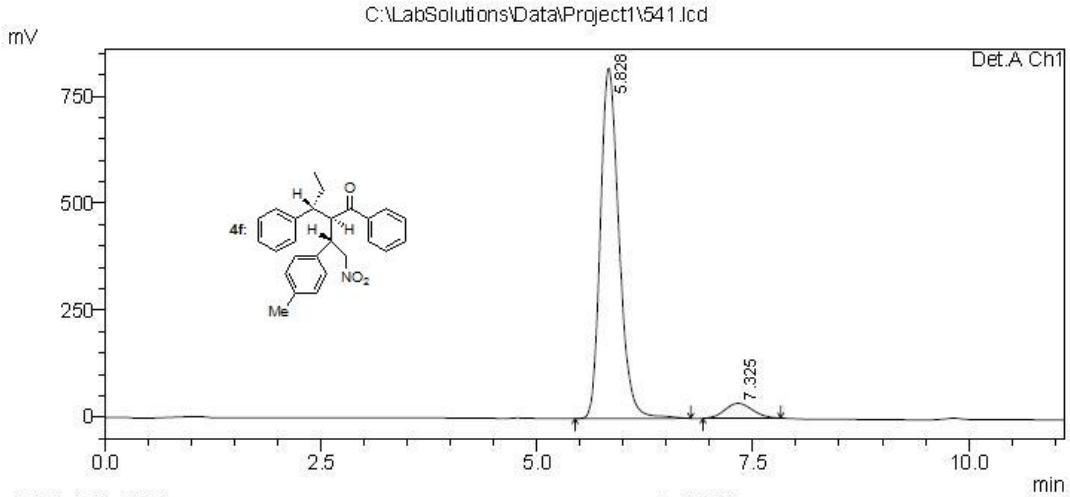


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	5.841	12542357	868366	49.753
2	7.320	12666690	541007	50.247
Total		25209047	1409373	100.000

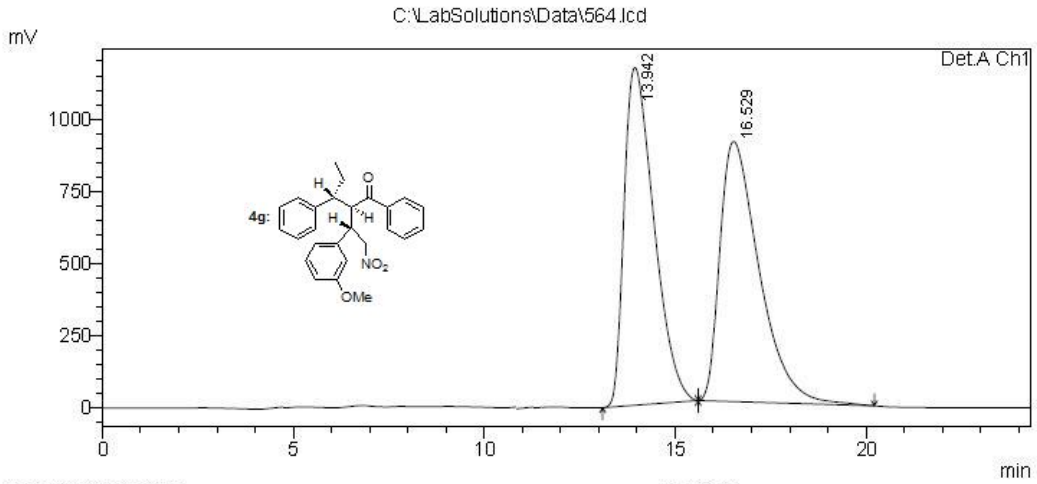


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	5.828	12269479	817998	94.061
2	7.325	774726	34811	5.939
Total		13044205	852809	100.000

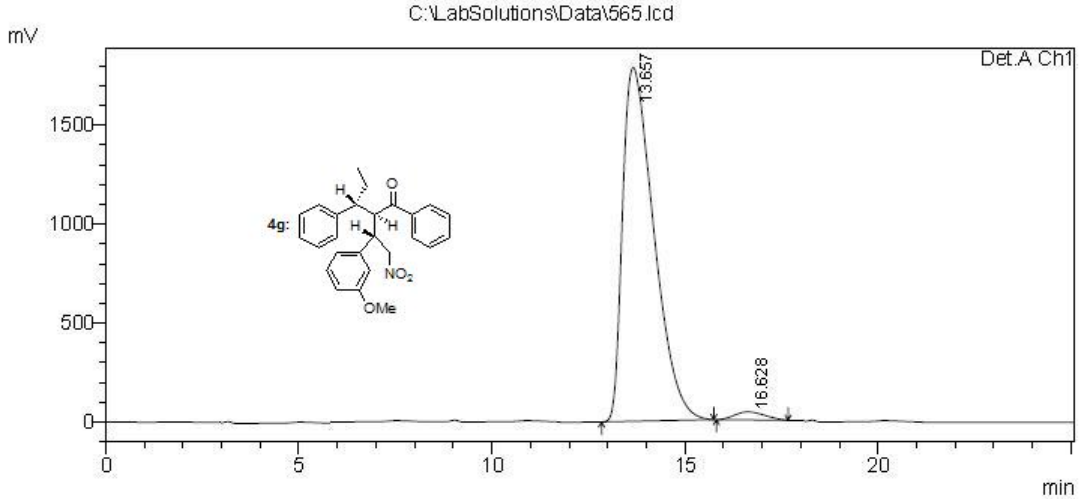


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	13.942	63474297	1169213	49.604
2	16.529	64488503	900371	50.396
Total		127962799	2069585	100.000

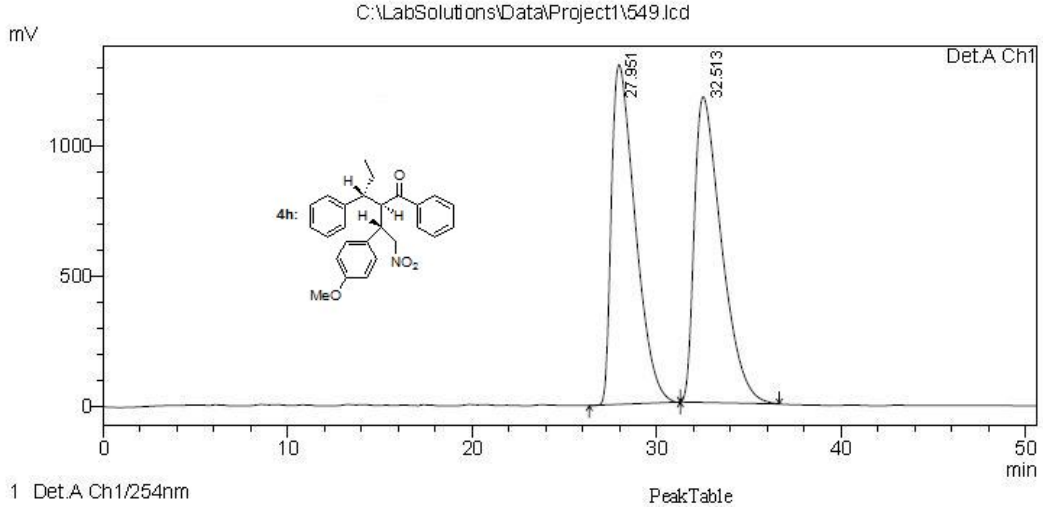


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	13.657	100666946	1787532	97.840
2	16.628	2221942	40502	2.160
Total		102888888	1828035	100.000

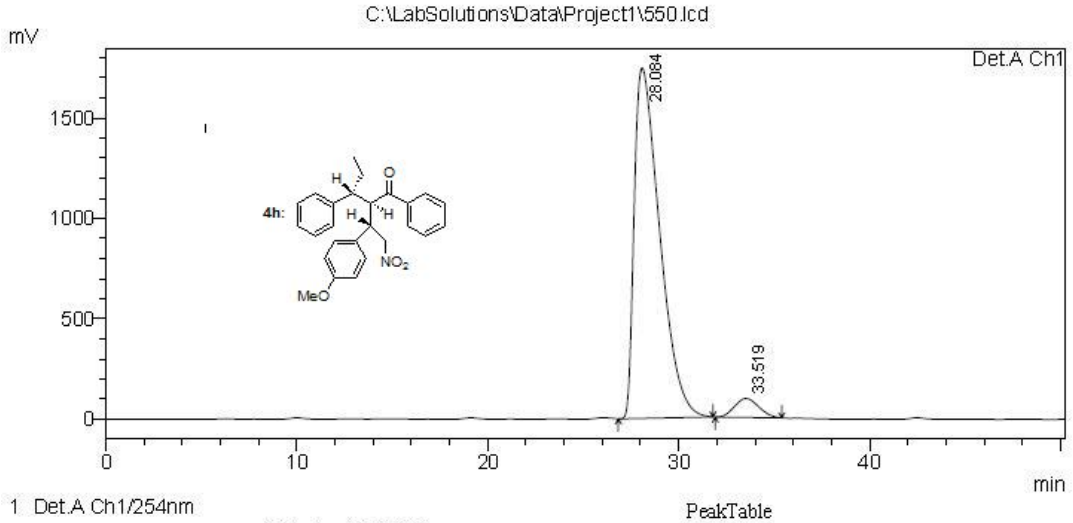


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	27.951	116813693	1301457	49.304
2	32.513	120111682	1170537	50.696
Total		236925375	2471995	100.000

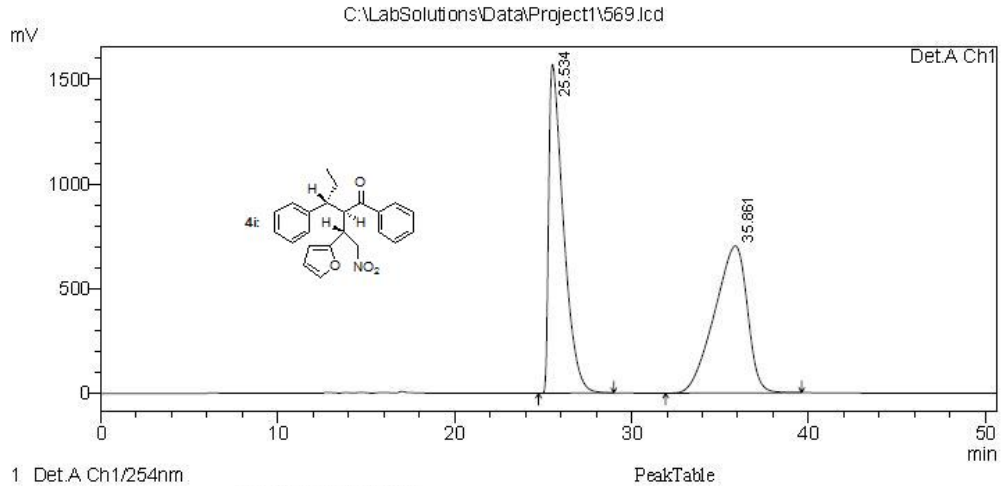


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	28.084	161668722	1745648	95.054
2	33.519	8412242	94815	4.946
Total		170080963	1840463	100.000

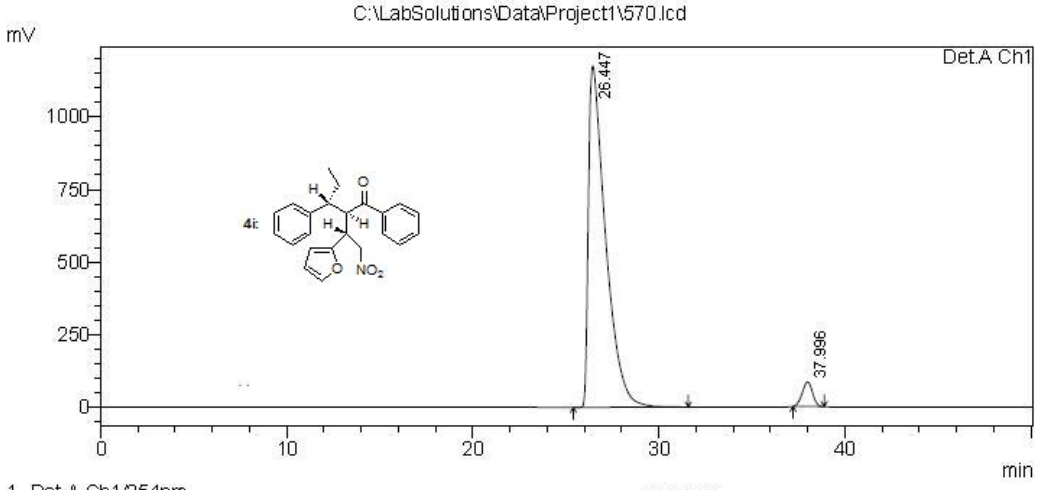


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	25.534	90783731	1568923	50.039
2	35.861	90642521	701698	49.961
Total		181426253	2270621	100.000

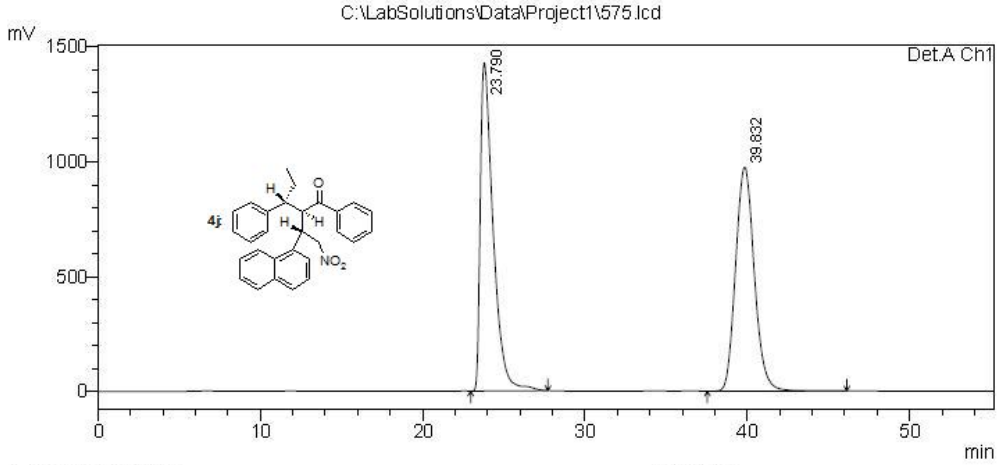


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	26.447	75128946	1176775	95.831
2	37.996	3268608	84250	4.169
Total		78397554	1261026	100.000

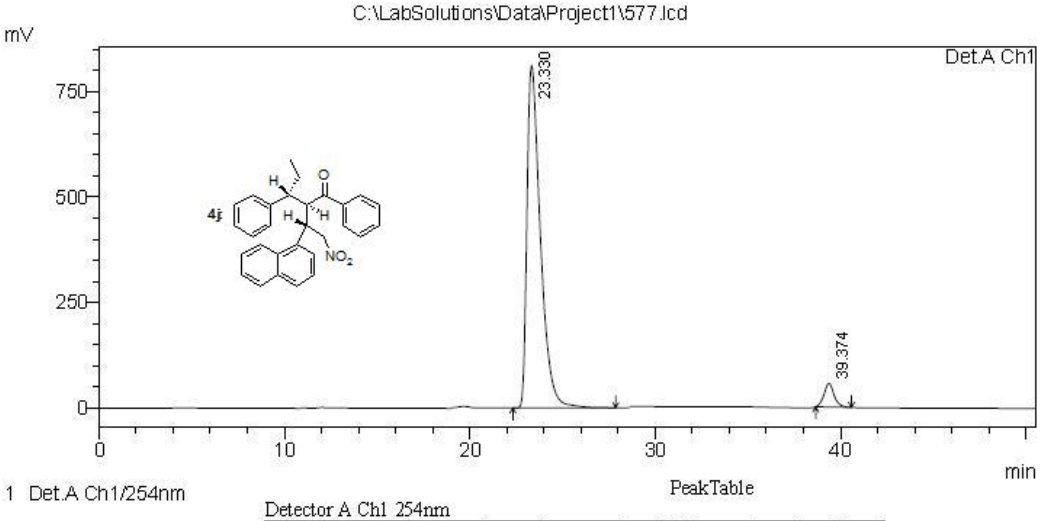


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	23.790	78461903	1428692	50.083
2	39.832	78202849	974811	49.917
Total		156664753	2403504	100.000

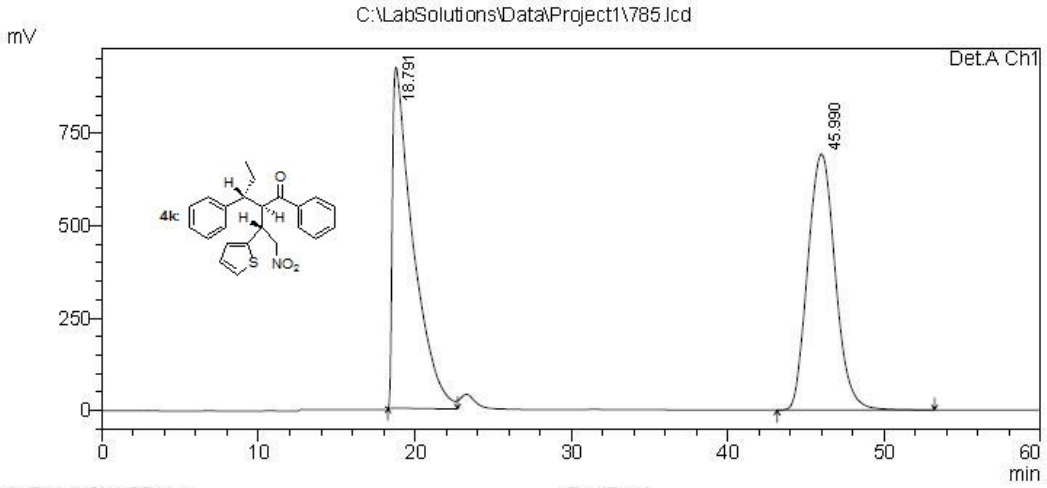


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	23.330	40782985	810992	95.068
2	39.374	2115751	55782	4.932
Total		42898736	866774	100.000

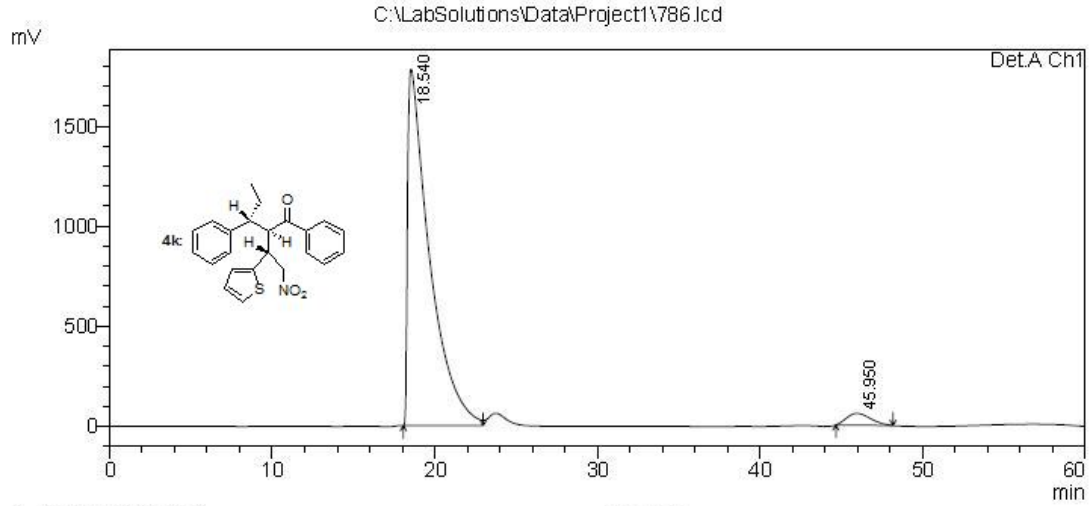


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	18.791	84366483	921475	49.974
2	45.990	84455799	691987	50.026
Total		168822282	1613462	100.000

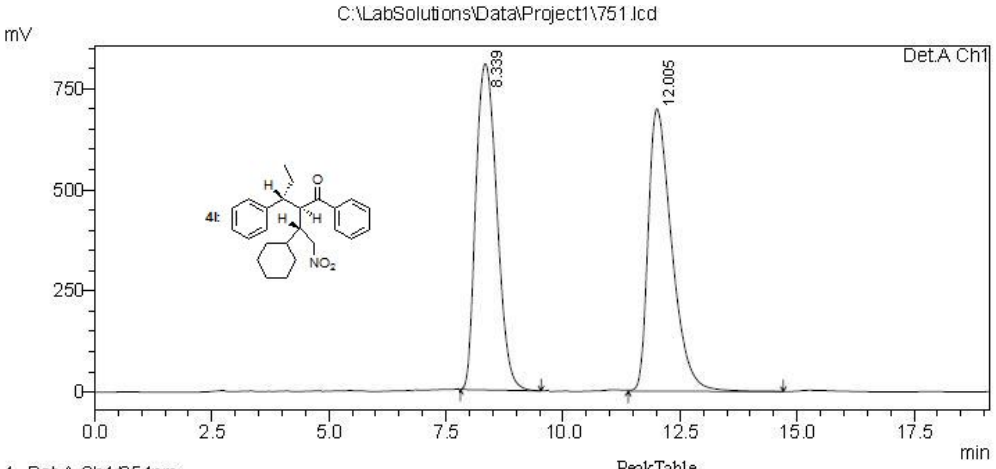


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	18.540	165943088	1782041	96.486
2	45.950	6042867	58808	3.514
Total		171985955	1840848	100.000

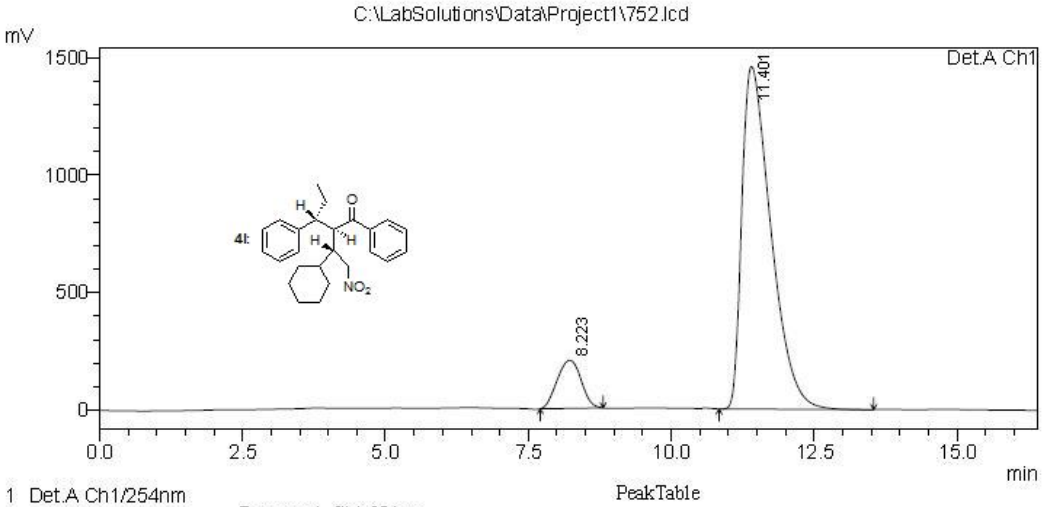


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	8.339	25277257	806872	50.806
2	12.005	24475417	698532	49.194
Total		49752675	1505404	100.000

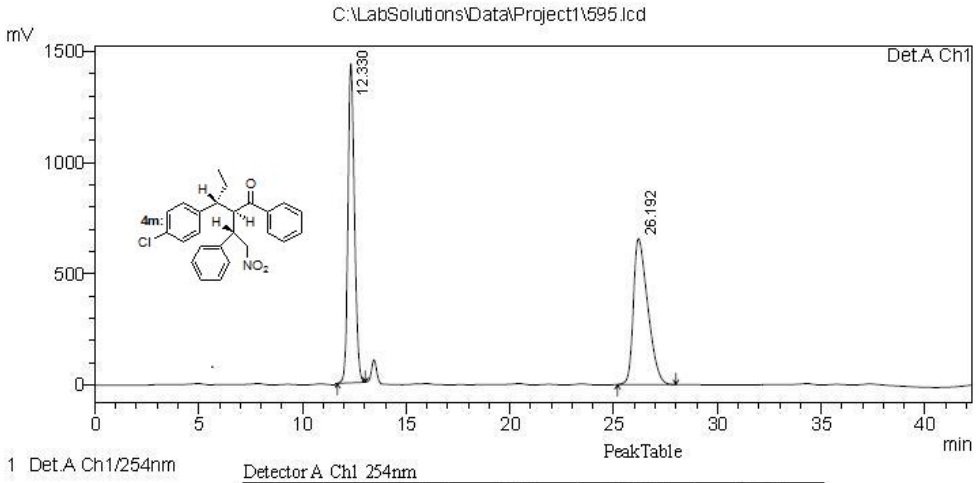


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

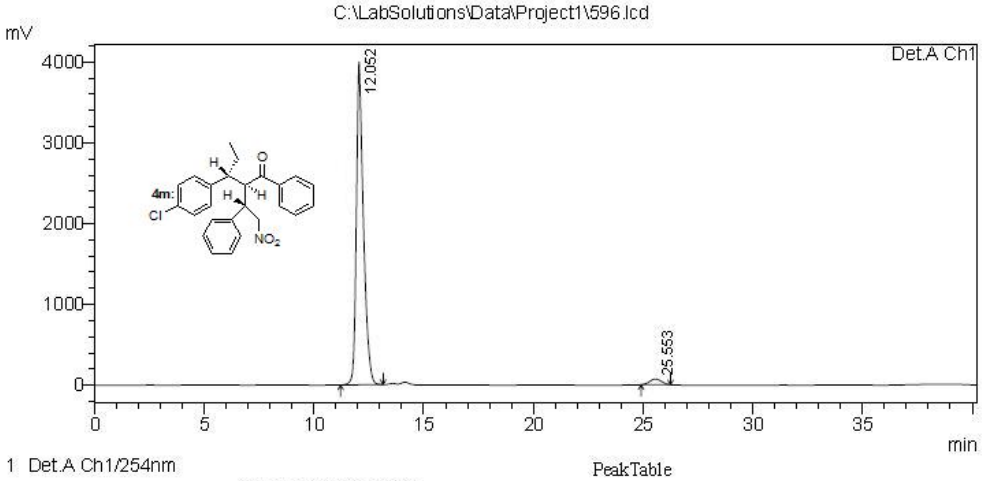
Peak#	Ret. Time	Area	Height	Area %
1	8.223	5944776	204478	10.361
2	11.401	51432770	1455677	89.639
Total		57377546	1660155	100.000



PeakTable

Detector A Ch1 254nm

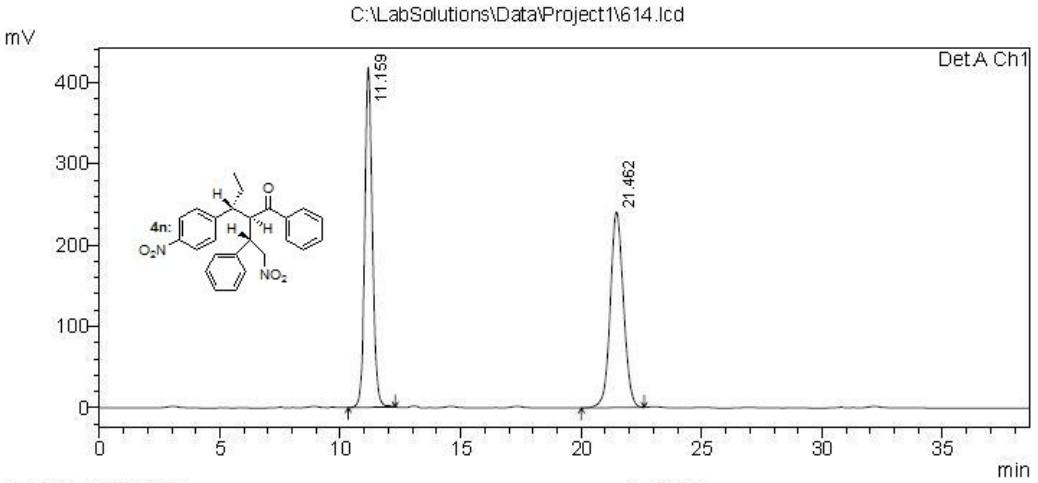
Peak#	Ret. Time	Area	Height	Area %
1	12.330	31228333	1435300	49.692
2	26.192	31615489	656426	50.308
Total		62843822	2091726	100.000



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	12.052	89135597	3994276	97.077
2	25.553	2683419	71000	2.923
Total		91819016	4065276	100.000

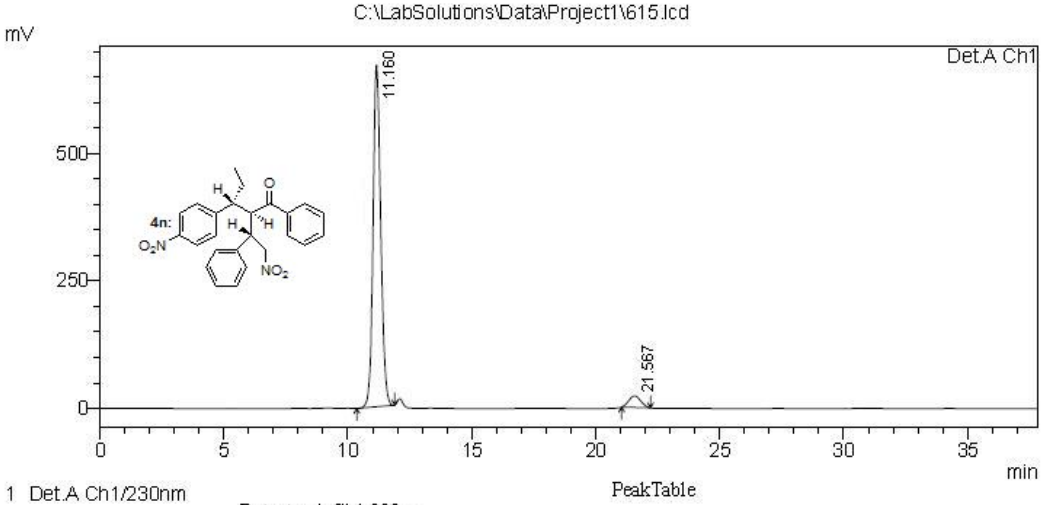


1 Det.A Ch1/230nm

PeakTable

Detector A Ch1 230nm

Peak#	Ret. Time	Area	Height	Area %
1	11.159	9181246	418197	50.103
2	21.462	9143541	240693	49.897
Total		18324787	658890	100.000

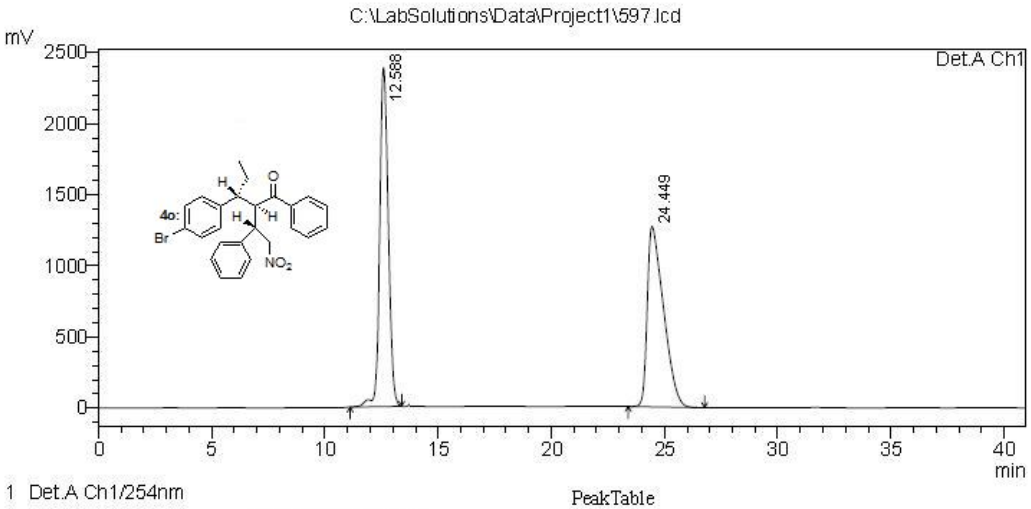


1 Det.A Ch1/230nm

PeakTable

Detector A Ch1 230nm

Peak#	Ret. Time	Area	Height	Area %
1	11.160	14399988	670336	95.020
2	21.567	754694	22673	4.980
Total		15154682	693008	100.000

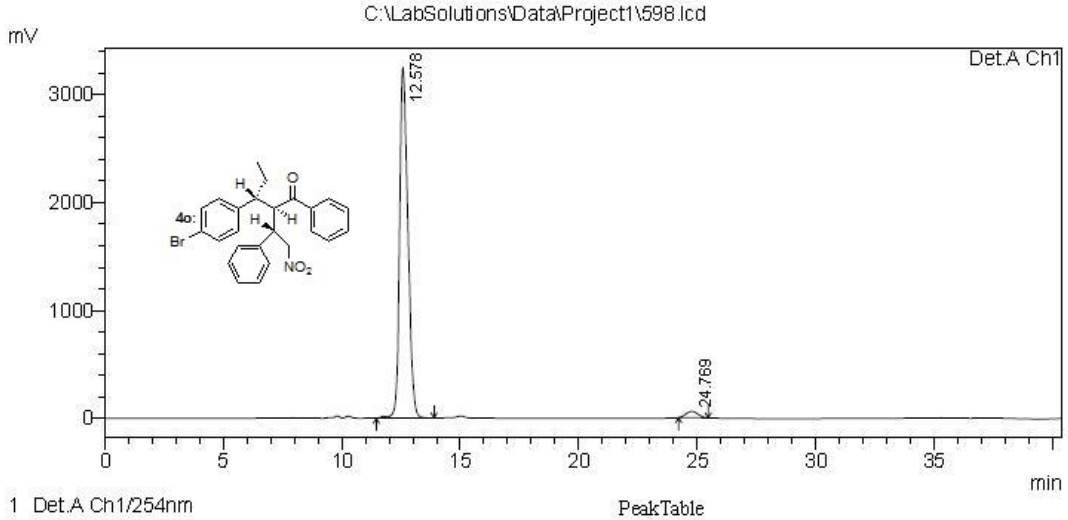


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	12.588	62983230	2384540	49.887
2	24.449	63267983	1268817	50.113
Total		126251213	3653357	100.000

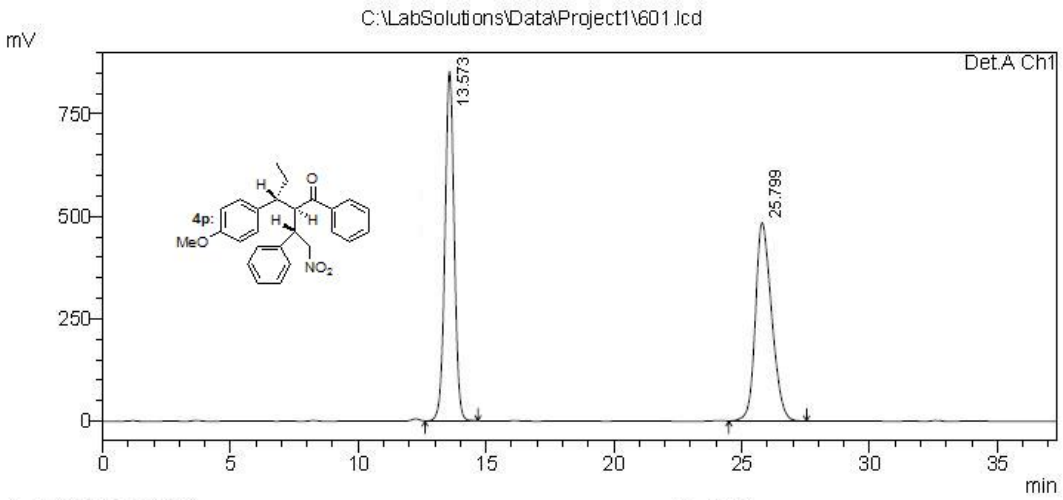


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	12.578	79394439	3254302	97.488
2	24.769	2045586	58414	2.512
Total		81440025	3312717	100.000

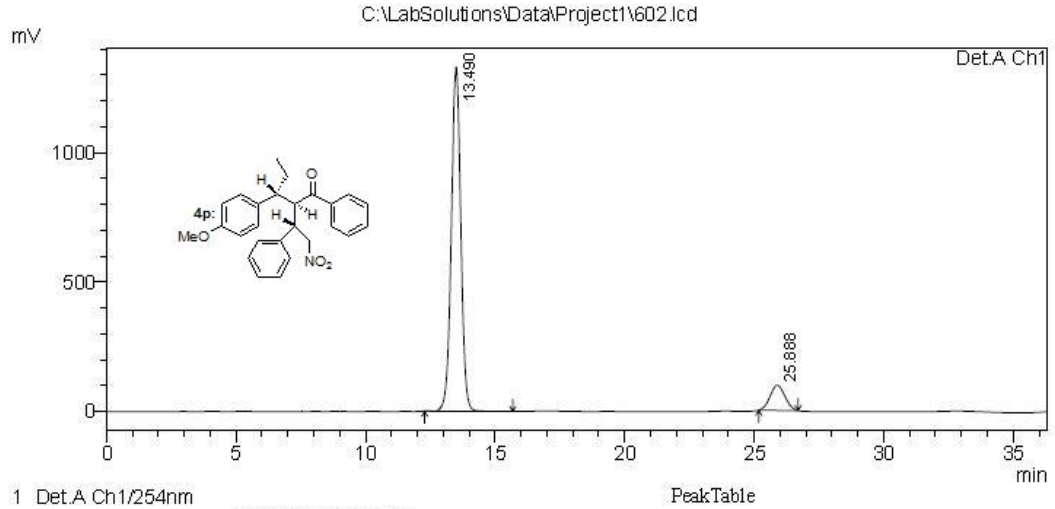


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	13.573	21514598	852702	50.093
2	25.799	21434604	483699	49.907
Total		42949202	1336401	100.000

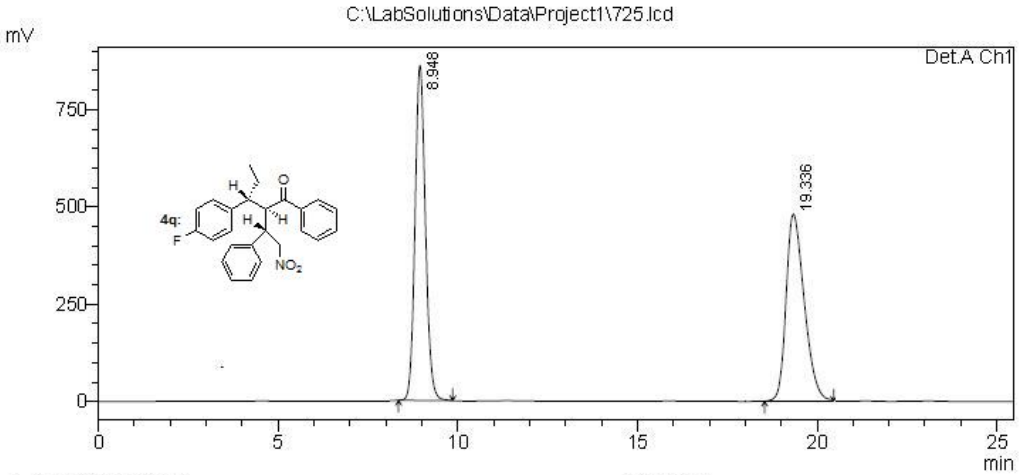


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	13.490	33849058	1330321	89.745
2	25.888	3867826	97135	10.255
Total		37716884	1427456	100.000

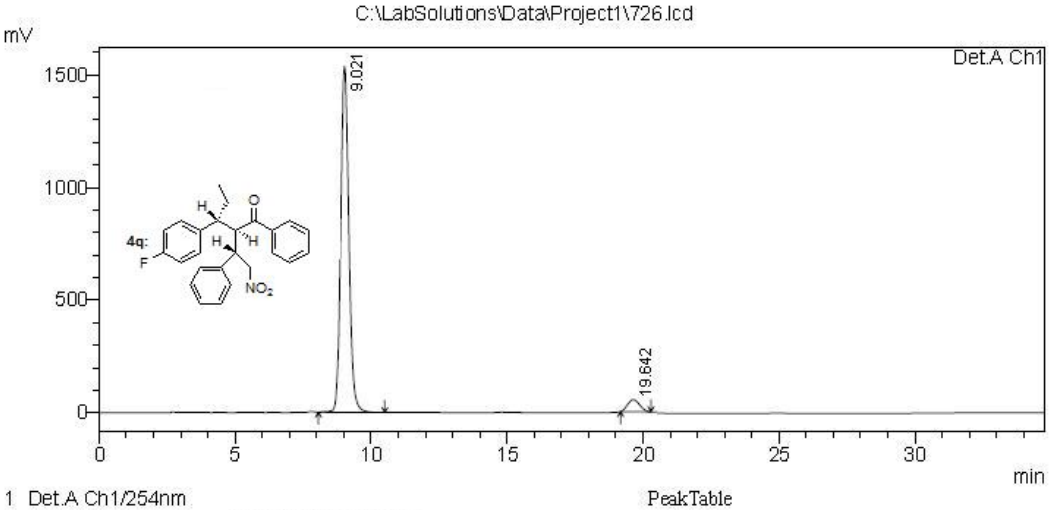


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	8.948	17292808	860091	50.576
2	19.336	16898854	480529	49.424
Total		34191662	1340620	100.000

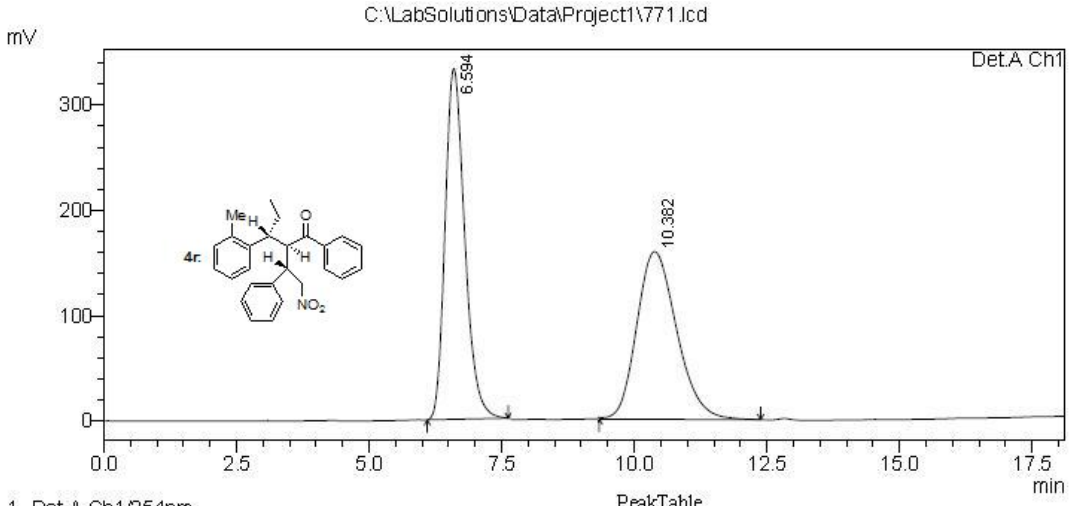


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	9.021	31862947	1536913	95.111
2	19.642	1637904	53278	4.889
Total		33500852	1590191	100.000

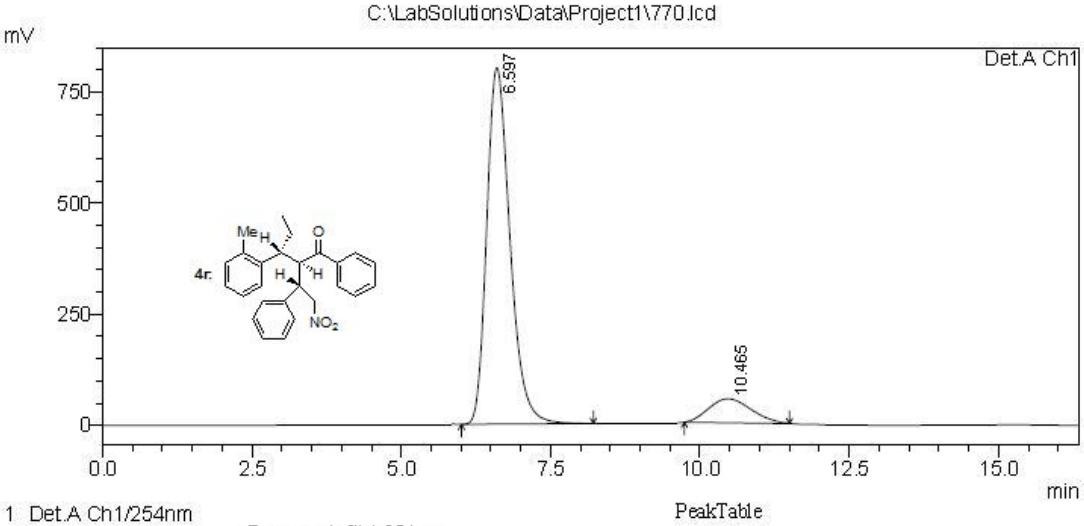


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	6.594	8583076	333220	50.666
2	10.382	8357534	159358	49.334
Total		16940611	492578	100.000

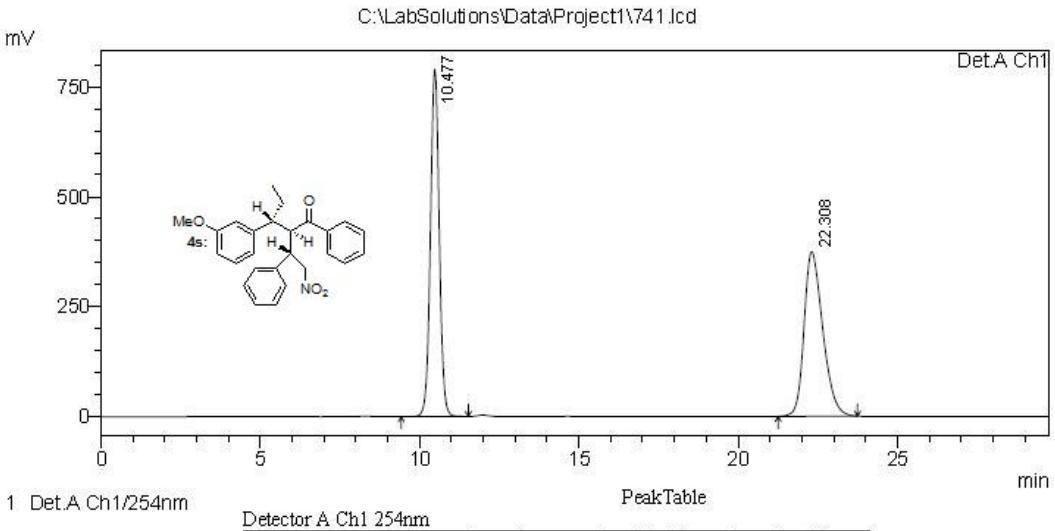


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	6.597	21727797	802396	89.062
2	10.465	2668425	53706	10.938
Total		24396222	856102	100.000

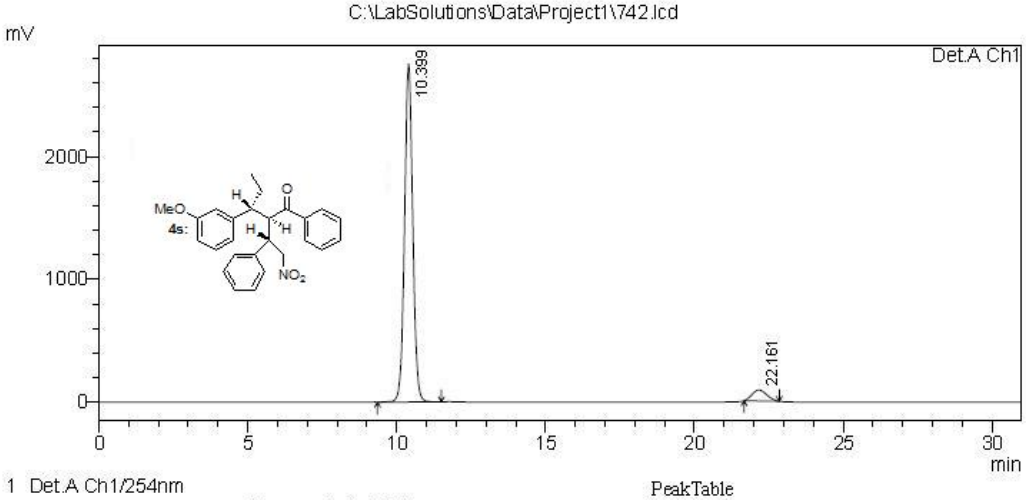


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	10.477	15219599	789065	49.916
2	22.308	15270997	374078	50.084
Total		30490597	1163143	100.000

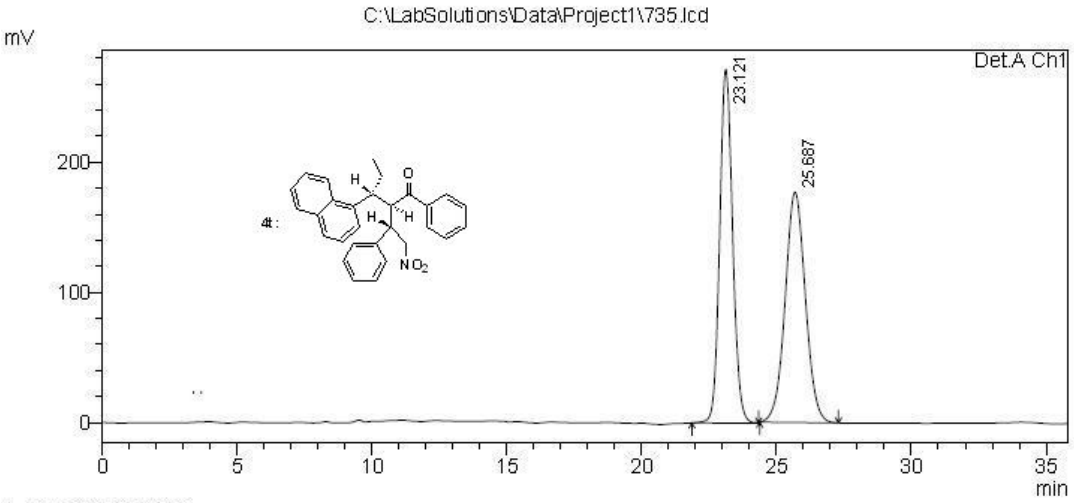


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

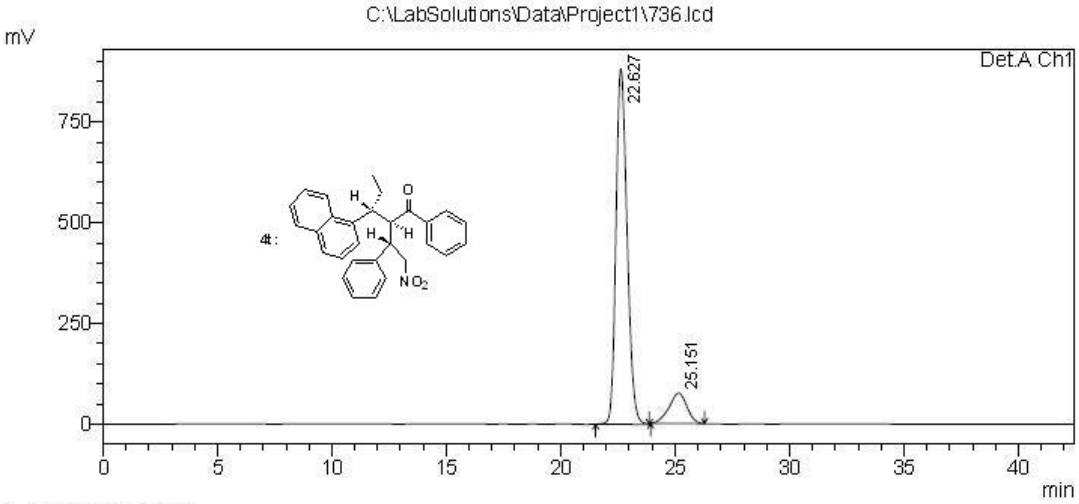
Peak#	Ret. Time	Area	Height	Area %
1	10.399	52615620	2752753	94.489
2	22.161	3068934	89805	5.511
Total		55684554	2842559	100.000



PeakTable

Detector A Ch1 254nm

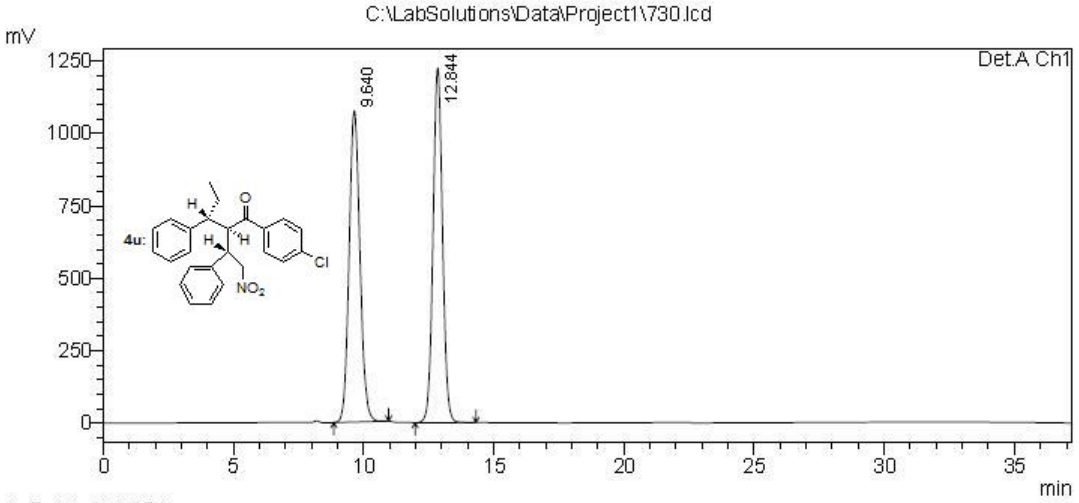
Peak#	Ret. Time	Area	Height	Area %
1	23.121	9047806	271638	49.508
2	25.687	9227494	176946	50.492
Total		18275300	448584	100.000



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	22.627	29719786	882319	87.808
2	25.151	4126405	75529	12.192
Total		33846191	957848	100.000

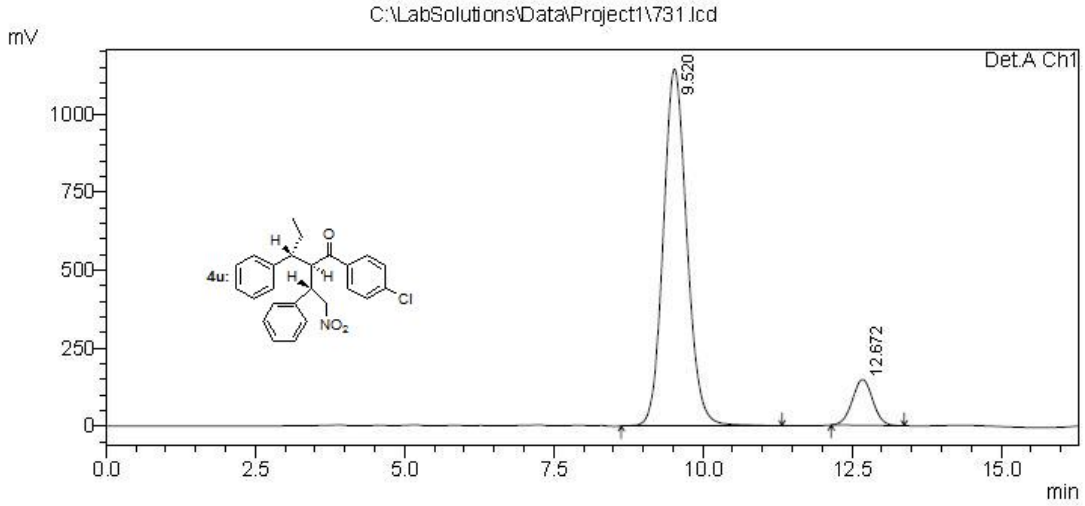


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	9.640	31081776	1074772	50.154
2	12.844	30890326	1224459	49.846
Total		61972103	2299231	100.000

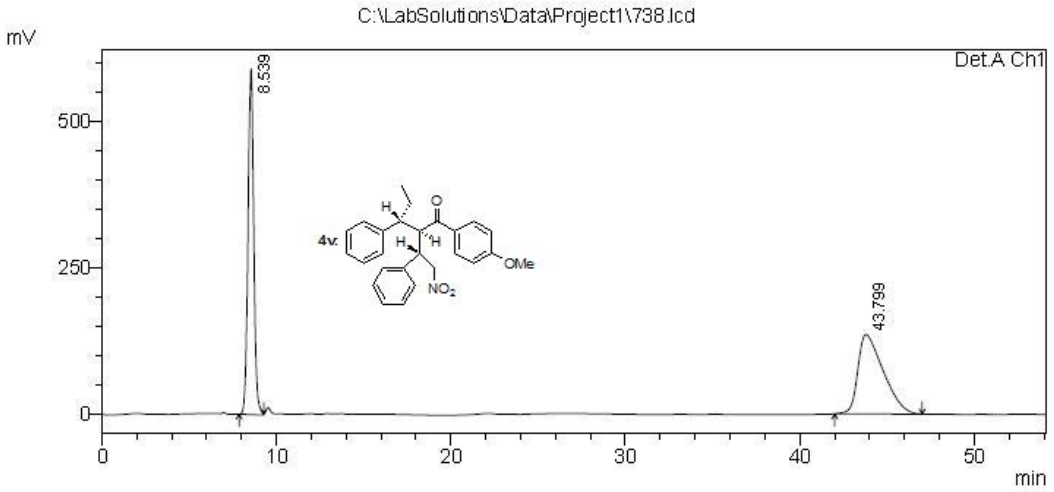


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	9.520	31317445	1143487	89.950
2	12.672	3498887	146923	10.050
Total		34816331	1290409	100.000

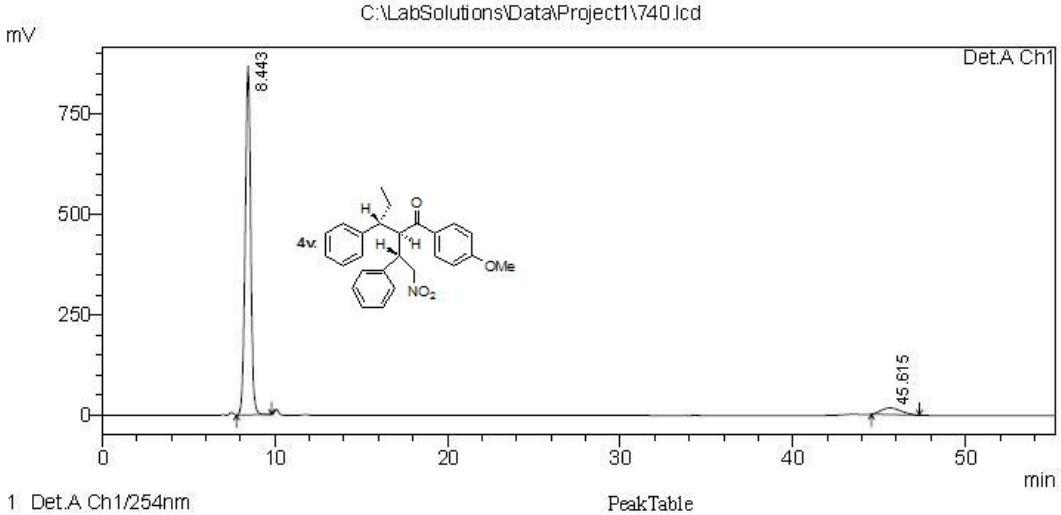


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	8.539	13545894	590742	49.699
2	43.799	13710085	135400	50.301
Total		27255979	726142	100.000

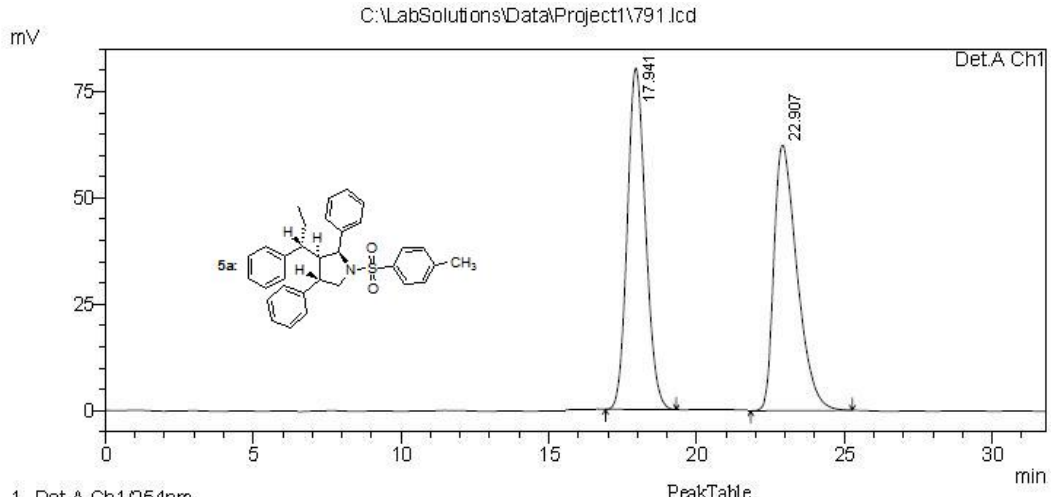


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	8.443	19153108	867488	93.573
2	45.615	1315519	16314	6.427
Total		20468627	883802	100.000

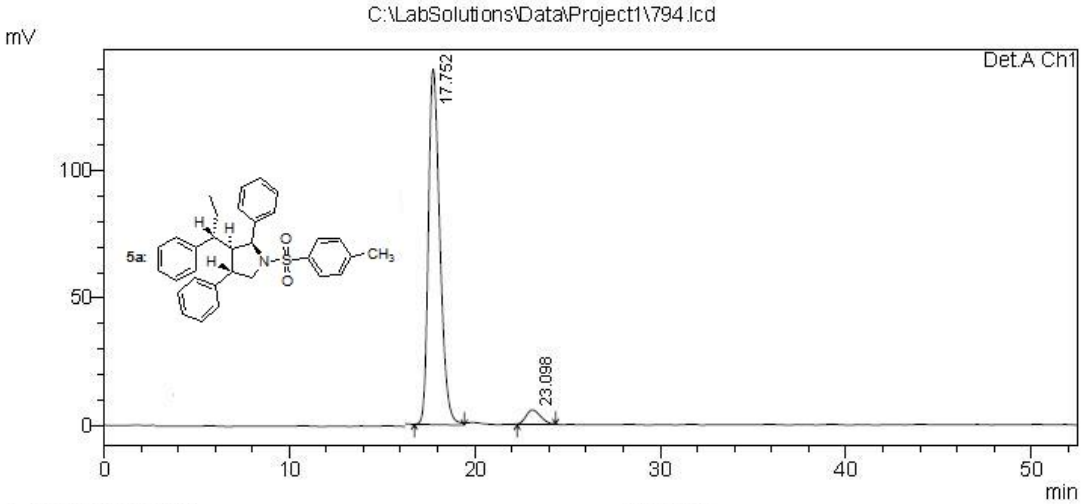


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	17.941	3453528	80338	50.351
2	22.907	3405372	62454	49.649
Total		6858900	142792	100.000

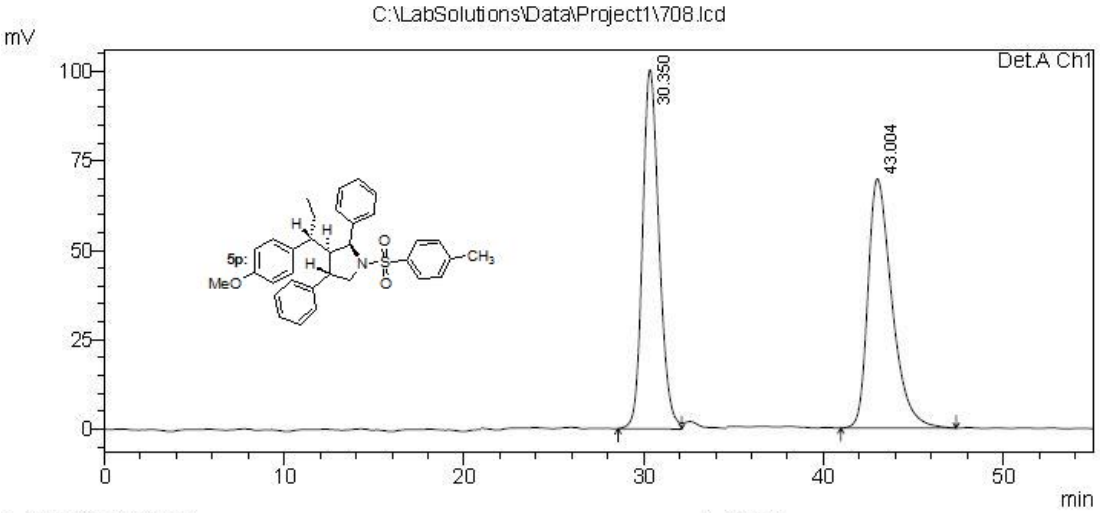


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	17.752	6008138	139667	95.049
2	23.098	312986	5709	4.951
Total		6321124	145376	100.000

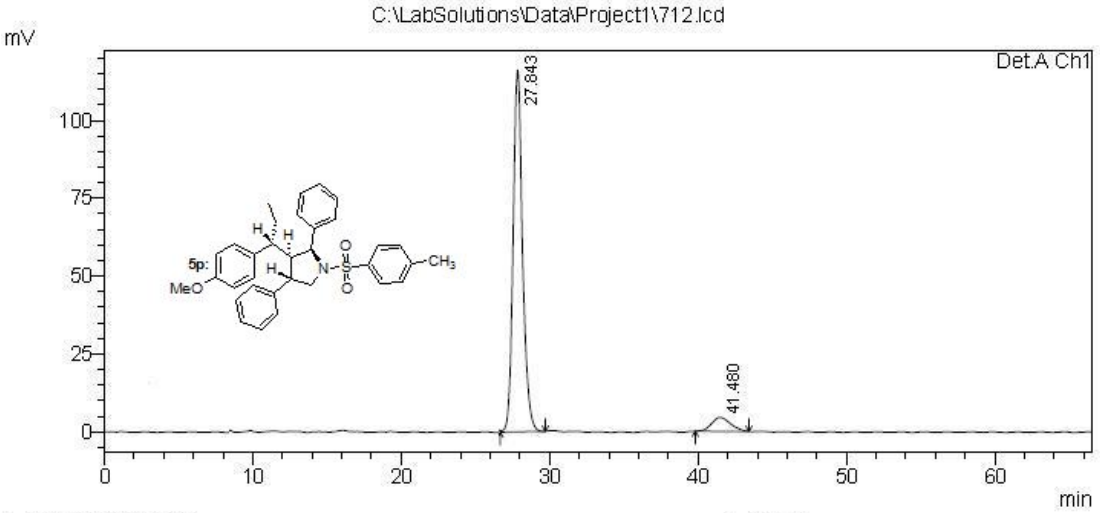


1 Det.A Ch1/235nm

PeakTable

Detector A Ch1 235nm

Peak#	Ret. Time	Area	Height	Area %
1	30.350	6421768	100323	49.786
2	43.004	6476909	69652	50.214
Total		12898677	169976	100.000



1 Det.A Ch1/235nm

PeakTable

Detector A Ch1 235nm

Peak#	Ret. Time	Area	Height	Area %
1	27.843	5134186	116098	92.837
2	41.480	396115	4453	7.163
Total		5530300	120551	100.000

X-ray crystallographic data for compound 4d

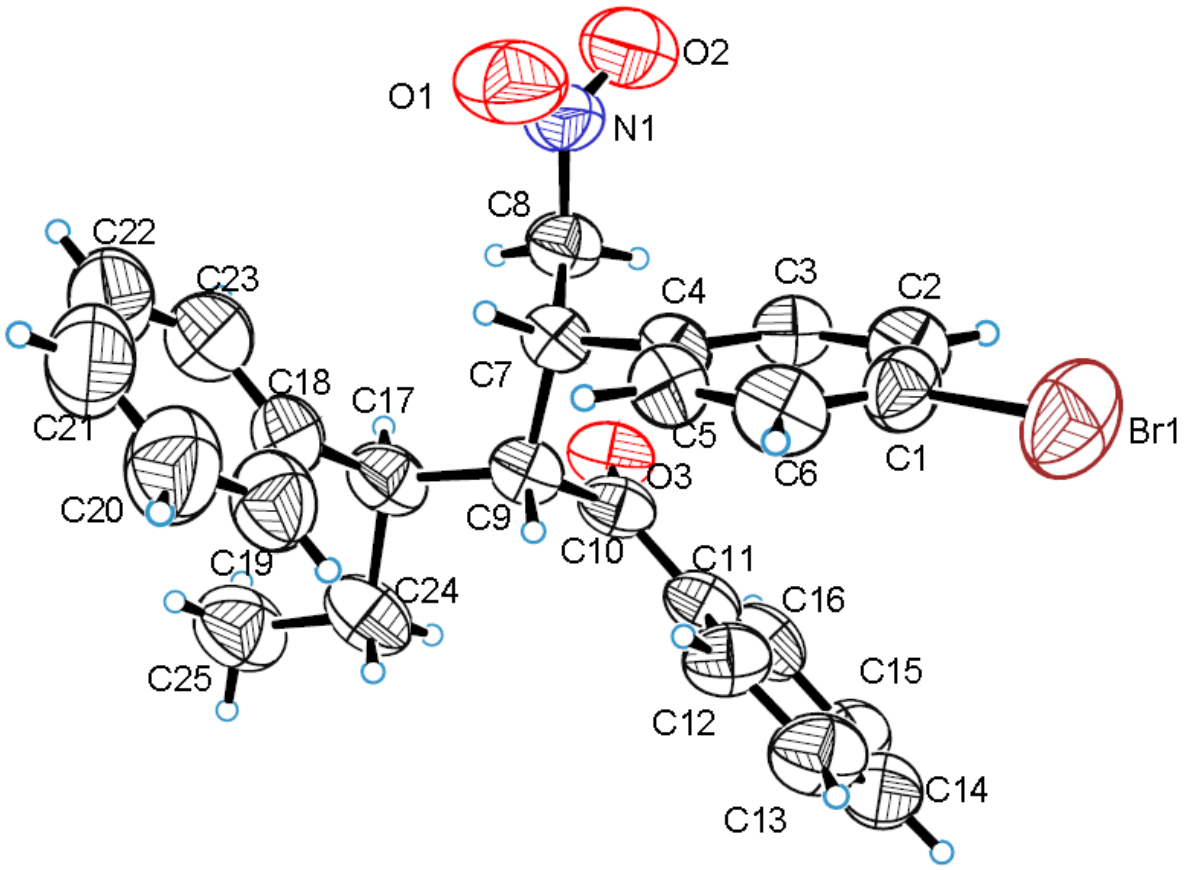


Table 1: Crystal data and structure refinement for Nicy-1

Identification code	Nicy-1
Empirical formula	$C_{25}H_{24}BrNO_3$
Formula weight	466.36
Temperature / K	293(2)
Crystal system	trigonal
Space group	R3
a / Å, b / Å, c / Å	20.3125(12), 20.3125(12), 15.9673(11)
α/°, β/°, γ/°	90.00, 90.00, 120.00
Volume / Å^3	5705.4(6)
Z	9
ρ_{calc} / mg mm^{-3}	1.222
μ / mm^{-1}	2.390
F(000)	2160
Crystal size / mm^3	0.32 × 0.20 × 0.20

Theta range for data collection	3.7346 to 138.979°
Index ranges	$-15 \leq h \leq 20$, $-22 \leq k \leq 19$, $-14 \leq l \leq 12$
Reflections collected	3385
Independent reflections	4702[R(int) = 0.0281]
Data/restraints/parameters	4702/89/261
Goodness-of-fit on F^2	1.082
Final R indexes [I>2σ (I)]	$R_1 = 0.0842$, $wR_2 = 0.1875$
Final R indexes [all data]	$R_1 = 0.1031$, $wR_2 = 0.1980$
Largest diff. peak/hole / e $Å^{-3}$	0.549/-0.247

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for Nicy-1. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Br1	4974.0(11)	10722.3(9)	4176.4(10)	213.0(9)
C4	3457(4)	9035(4)	2092(4)	94.7(17)
O3	2421(3)	6878(3)	2138(4)	132.9(19)
C11	2338(4)	7427(4)	3387(6)	107(2)
C7	2912(4)	8464(4)	1467(5)	98.5(18)
C10	2337(4)	7381(4)	2490(6)	110(2)
C9	2187(4)	7901(4)	1957(5)	106.4(19)
C5	3380(5)	9630(4)	2322(5)	117(2)
C8	3246(4)	8087(4)	947(5)	115(2)
C3	3997(4)	8953(4)	2495(5)	108(2)
C16	2363(4)	6855(5)	3865(8)	132(3)
C2	4466(5)	9438(5)	3113(6)	130(3)
C18	1342(4)	7941(5)	753(5)	153(3)
C19	1172(5)	8460(5)	1118(5)	180(4)
C20	1026(5)	8932(5)	616(7)	234(5)
C21	1051(5)	8885(6)	-251(7)	231(5)
C22	1221(5)	8367(7)	-616(4)	217(5)
C23	1366(5)	7895(6)	-112(6)	203(4)
C6	3807(6)	10122(5)	2939(7)	136(3)
C15	2366(6)	6867(8)	4757(8)	157(4)
C13	2363(6)	8017(7)	4684(9)	167(4)
N1	3812(5)	8628(6)	365(5)	137(3)

C14	2389(6)	7471(7)	5161(7)	144(3)
C1	4346(5)	10008(5)	3323(5)	127(3)
C12	2338(5)	8010(5)	3810(6)	135(3)
O1	3781(6)	9068(6)	38(7)	220(4)
O2	4364(6)	8607(7)	257(6)	221(4)
C24	759(5)	6903(6)	1908(8)	162(4)
C25	21(7)	6364(7)	1338(8)	200(5)
C17	1471(5)	7419(5)	1329(6)	132(2)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for Nicy-1.
The Anisotropic displacement factor exponent takes the form:
$-2\pi^2[h^2a^{*2}U_{11}+...+2hka\times b\times U_{12}]$

Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{23}**	**U_{13}**	**U_{12}**
Br1	254.7(18)	178.3(12)	170.0(11)	-64.7(10)	-47.0(11)	81.1(12)
C4	88(4)	83(4)	117(5)	7(3)	9(3)	46(3)
O3	144(4)	102(3)	166(5)	8(3)	38(4)	71(3)
C11	92(4)	84(4)	142(6)	6(4)	20(4)	41(3)
C7	93(4)	89(4)	115(5)	1(3)	3(4)	46(4)
C10	99(5)	85(4)	136(6)	1(4)	25(4)	39(4)
C9	103(4)	93(4)	124(5)	-1(4)	11(4)	49(4)
C5	117(6)	93(5)	138(6)	-1(5)	-5(5)	50(4)
C8	109(5)	103(5)	118(5)	-12(4)	14(4)	42(4)
C3	120(5)	88(4)	116(5)	-4(4)	10(4)	53(4)
C16	104(5)	104(5)	197(10)	0(6)	4(6)	57(4)
C2	133(6)	113(6)	146(7)	-7(5)	4(5)	64(5)
C18	103(5)	171(7)	148(6)	-6(5)	-16(5)	41(5)
C19	171(8)	171(7)	209(7)	24(6)	-16(6)	94(6)
C20	235(10)	234(9)	240(8)	28(7)	-35(8)	122(8)
C21	203(9)	246(9)	222(8)	25(7)	-45(8)	96(7)
C22	165(8)	245(10)	210(8)	35(6)	-23(7)	81(7)
C23	160(8)	225(9)	187(6)	14(6)	-3(7)	68(6)
C6	149(7)	93(5)	173(8)	-6(5)	9(7)	66(5)
C15	136(7)	176(10)	146(9)	45(8)	16(7)	70(7)
C13	183(10)	139(9)	181(11)	-31(8)	25(9)	81(8)
N1	137(6)	178(7)	122(5)	30(5)	28(4)	98(6)
C14	145(8)	148(9)	148(8)	-2(7)	11(6)	80(7)
C1	131(6)	111(6)	116(6)	-11(5)	-16(5)	42(5)

C12	153(8)	115(6)	138(8)	-6(5)	23(6)	67(5)
O1	226(7)	222(7)	234(8)	87(6)	78(6)	128(6)
O2	173(6)	283(8)	217(7)	70(6)	48(5)	121(6)
C24	108(5)	126(7)	228(11)	7(6)	19(6)	39(5)
C25	148(6)	182(8)	230(9)	-25(7)	7(6)	53(5)
C17	107(4)	120(5)	159(6)	-25(4)	7(4)	49(4)

Table 4 Bond Lengths for Nicy-1.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Br1	C1	1.933(8)	C2	C1	1.341(12)
C4	C7	1.512(9)	C18	C19	1.3900
C4	C5	1.345(10)	C18	C23	1.3900
C4	C3	1.351(10)	C18	C17	1.523(11)
O3	C10	1.251(8)	C19	C20	1.3900
C11	C10	1.435(11)	C20	C21	1.3900
C11	C16	1.411(12)	C21	C22	1.3900
C11	C12	1.366(11)	C22	C23	1.3900
C7	C9	1.551(9)	C6	C1	1.371(12)
C7	C8	1.502(9)	C15	C14	1.367(15)
C10	C9	1.500(10)	C13	C14	1.367(15)
C9	C17	1.630(11)	C13	C12	1.396(15)
C5	C6	1.362(12)	N1	O1	1.064(10)
C8	N1	1.458(10)	N1	O2	1.156(10)
C3	C2	1.383(11)	C24	C25	1.624(15)
C16	C15	1.425(14)	C24	C17	1.591(13)

Table 5 Bond Angles for Nicy-1.

Atom	Atom	Atom	Angle /°	Atom	Atom	Atom	Angle /°
C4	C7	C9	107.1(6)	C19	C18	C23	120.0
C4	C5	C6	122.6(8)	C19	C18	C17	118.1(7)
C4	C3	C2	124.4(7)	C19	C20	C21	120.0
O3	C10	C11	120.3(7)	C20	C19	C18	120.0
O3	C10	C9	118.6(8)	C20	C21	C22	120.0
C11	C10	C9	121.0(7)	C21	C22	C23	120.0
C11	C16	C15	122.0(9)	C22	C23	C18	120.0
C11	C12	C13	119.1(10)	C23	C18	C17	121.9(7)

C7	C9	C17	111.4(6)	C6	C1	Br1	118.2(8)
C10	C9	C7	110.7(6)	C13	C14	C15	117.8(11)
C10	C9	C17	111.1(6)	N1	C8	C7	110.9(7)
C5	C4	C7	120.0(7)	C14	C15	C16	118.9(11)
C5	C4	C3	116.6(7)	C14	C13	C12	124.5(10)
C5	C6	C1	118.0(8)	C1	C2	C3	115.8(9)
C8	C7	C4	114.3(6)	C12	C11	C10	123.3(8)
C8	C7	C9	114.1(6)	C12	C11	C16	117.5(9)
C3	C4	C7	123.3(6)	O1	N1	C8	126.1(10)
C16	C11	C10	119.2(8)	O1	N1	O2	115.0(11)
C2	C1	Br1	119.1(8)	O2	N1	C8	118.8(10)
C2	C1	C6	122.6(8)	C24	C17	C9	106.3(8)
C18	C17	C9	111.5(7)	C17	C24	C25	110.3(9)
C18	C17	C24	113.1(8)				

Table 6 Torsion Angles for Nicy-1.

A	B	C	D	Angle/°
C4	C7	C9	C10	-72.6(7)
C4	C7	C9	C17	163.2(6)
C4	C7	C8	N1	-69.1(8)
C4	C5	C6	C1	1.5(14)
C4	C3	C2	C1	1.8(12)
O3	C10	C9	C7	-71.5(8)
O3	C10	C9	C17	52.8(8)
C11	C10	C9	C7	112.2(7)
C11	C10	C9	C17	-123.4(7)
C11	C16	C15	C14	-3.7(14)
C7	C4	C5	C6	173.6(8)
C7	C4	C3	C2	-175.2(7)
C7	C9	C17	C18	-53.0(8)
C7	C9	C17	C24	-176.8(7)
C7	C8	N1	O1	-37.6(15)
C7	C8	N1	O2	139.6(11)
C10	C11	C16	C15	180.0(8)
C10	C11	C12	C13	-178.0(8)
C10	C9	C17	C18	-177.0(7)

C10	C9	C17	C24	59.3(8)
C9	C7	C8	N1	167.1(7)
C5	C4	C7	C9	-82.0(8)
C5	C4	C7	C8	150.6(7)
C5	C4	C3	C2	-0.4(11)
C5	C6	C1	Br1	178.2(7)
C5	C6	C1	C2	0.1(15)
C8	C7	C9	C10	54.9(8)
C8	C7	C9	C17	-69.3(8)
C3	C4	C7	C9	92.6(8)
C3	C4	C7	C8	-34.7(10)
C3	C4	C5	C6	-1.4(12)
C3	C2	C1	Br1	-179.7(6)
C3	C2	C1	C6	-1.7(14)
C16	C11	C10	O3	-6.0(10)
C16	C11	C10	C9	170.2(7)
C16	C11	C12	C13	0.2(13)
C16	C15	C14	C13	3.7(15)
C18	C19	C20	C21	0.0
C19	C18	C23	C22	0.0
C19	C18	C17	C9	-60.8(8)
C19	C18	C17	C24	59.0(9)
C19	C20	C21	C22	0.0
C20	C21	C22	C23	0.0
C21	C22	C23	C18	0.0
C23	C18	C19	C20	0.0
C23	C18	C17	C9	121.2(6)
C23	C18	C17	C24	-119.0(8)
C14	C13	C12	C11	-0.1(17)
C12	C11	C10	O3	172.2(8)
C12	C11	C10	C9	-11.6(11)
C12	C11	C16	C15	1.7(12)
C12	C13	C14	C15	-1.9(17)
C25	C24	C17	C9	-177.1(8)
C25	C24	C17	C18	60.2(11)
C17	C18	C19	C20	-178.0(7)
C17	C18	C23	C22	178.0(7)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for Nicy-1.

Atom	*x*	*y*	*z*	U(eq)
H7	2764	8742	1081	118
H9	2053	8199	2331	128
H5	3021	9709	2048	140
H8B	3481	7881	1311	138
H8A	2845	7670	635	138
H3	4057	8543	2348	129
H16	2377	6459	3590	159
H2	4842	9373	3367	156
H19	1155	8491	1698	216
H20	913	9279	860	281
H21	954	9201	-587	277
H22	1238	8336	-1195	260
H23	1480	7548	-358	244
H6	3735	10522	3095	163
H15	2353	6469	5060	188
H13	2363	8420	4960	200
H14	2420	7510	5742	173
H12	2321	8399	3520	162
H24B	650	7224	2263	195
H24A	876	6589	2267	195
H25A	108	6006	1037	299
H25C	-66	6672	948	299
H25B	-417	6095	1692	299
H17	1581	7086	983	158

Experimental

Single crystals of $C_{25}H_{24}BrNO_3$ **[Nicy-1]** were recrystallised from **[solvents]** mounted in inert oil and transferred to the cold gas stream of the diffractometer.

Crystal structure determination of [Nicy-1]

Crystal Data. $C_{25}H_{24}BrNO_3$, M =466.36, trigonal, a = 20.3125(12) Å, c = 15.9673(11) Å, U = 5705.4(6) Å^3, T = 293(2), space group R3 (no. 146), Z = 9, μ(Cu Kα) = 2.390, 3385 reflections measured, 4702 unique (R_{int} = 0.0281) which were used in all calculations. The final $wR(F_2)$ was 0.1980 (all data).

X-ray crystallographic data for compound 5a

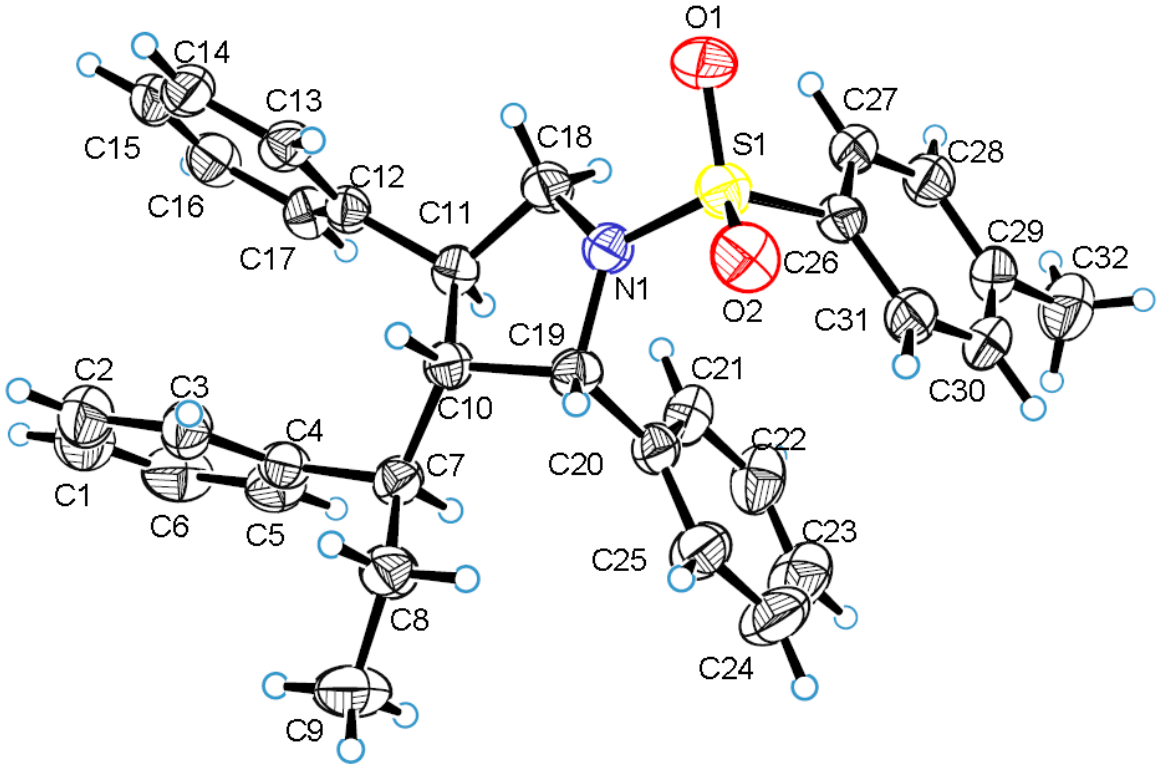


Table 1 Crystal data and structure refinement for ncy-2

Identification code	ncy-2
Empirical formula	$C_{32}H_{33}NSO_2$
Formula weight	495.65
Temperature	291(2)
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
a/Å, b/Å, c/Å	7.4163(3), 14.0432(6), 25.9274(10)
α/°, β/°, γ/°,	90.00, 90.00, 90.00
Volume/Å^3	2700.30(19)
Z	4
ρ_{calc} mg/mm^3	1.219
μ / mm^{-1}	0.149
F(000)	1056
Crystal size	0.40 × 0.30 × 0.24
Theta range for data collection	2.90 to 26.37°
Index ranges	$-9 \le h \le 9$, $-11 \le k \le 17$, $-32 \le l \le 32$
Reflections collected	14601
Independent reflections	5528[R(int) = 0.0442]
Data/restraints/parameters	5528/0/327
Goodness-of-fit on F^2	1.012

Final R indexes [I>2σ (I)]	R_1 = 0.0517, wR_2 = 0.0886
Final R indexes [all data]	R_1 = 0.0890, wR_2 = 0.1012
Largest diff. peak/hole	0.146/-0.208

Table 2 Atomic Coordinates (Å×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for ncy-2. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
S1	5085.9(10)	3890.7(5)	1848.8(2)	51.91(19)
O1	4209(3)	2980.6(12)	1847.9(7)	64.1(5)
N1	5269(3)	4207.1(13)	1251.1(7)	44.7(5)
O2	6841(3)	4001.2(14)	2074.5(7)	65.9(5)
C11	4528(3)	4426.4(18)	362.7(9)	44.9(6)
C12	4241(3)	3774.1(18)	-97.1(9)	44(6)
C29	1316(4)	6132(2)	2494.6(10)	60.1(8)
C10	6500(3)	4722.3(17)	476.4(9)	43.8(6)
C26	3620(4)	4712.7(18)	2146.9(10)	46.8(7)
C7	7294(4)	5519.5(18)	135.9(10)	48.8(7)
C18	3894(4)	3949(2)	868.1(9)	54.5(7)
C20	5886(4)	5964.4(19)	1207.2(9)	49.7(7)
C17	3146(4)	4043(2)	-503.8(10)	53.3(7)
C13	5056(4)	2883.6(17)	-120.4(10)	52.6(7)
C27	1777(4)	4611.2(19)	2087.5(10)	53.3(7)
C4	7099(4)	5305.7(18)	-437(10)	49.6(7)
C19	6482(4)	4959.1(18)	1062.5(9)	45.1(6)
C8	9284(4)	5704(2)	275.5(11)	59.8(8)
C6	5870(5)	5674(3)	-1274(14)	82.2(11)
C31	4309(4)	5504(2)	2394(10)	58.8(8)
C28	636(4)	5324(2)	2260.1(11)	60.4(8)
C15	3713(4)	2584(2)	-941.8(12)	69.4(10)
C14	4787(4)	2297(2)	-537.1(12)	63.2(8)
C21	4113(4)	6265(2)	1157.6(11)	61(8)
C16	2888(4)	3452(2)	-925.1(11)	66(9)
C3	8002(4)	4552(2)	-662(11)	59.7(8)
C25	7100(5)	6588(2)	1418.2(11)	70.3(9)
C30	3164(5)	6209(2)	2566.2(10)	63.4(8)
C5	6031(4)	5865(2)	-748.4(12)	65.2(9)
C9	10103(5)	6543(2)	-2.8(13)	86.2(11)

C32	79(6)	6938(2)	2659.7(12)	86.7(10)
C2	7837(5)	4356(2)	-1181.3(14)	78.2(10)
C1	6788(6)	4925(3)	-1486.3(13)	85.8(12)
C23	4815(7)	7784(2)	1509.7(14)	92.9(12)
C22	3598(5)	7170(2)	1306.9(12)	80.4(10)
C24	6564(6)	7509(3)	1568.2(14)	91.4(12)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for ncy-2. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U11+...+2hka \times b \times U_{12}]$

Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{23}**	**U_{13}**	**U_{12}**
S1	56.3(4)	51.5(4)	47.9(4)	6(3)	-4.1(4)	0.9(4)
O1	79.6(14)	43.6(11)	69(13)	11.2(9)	1.9(11)	-4.3(10)
N1	45.3(13)	46.6(12)	42.2(12)	-0.2(9)	-2.2(11)	-3.1(11)
O2	56.2(12)	81.3(14)	60.3(12)	10.8(11)	-15.2(10)	3.4(11)
C11	40.1(16)	49.6(15)	44.9(15)	-3.2(11)	-2.7(12)	2.8(12)
C12	38.4(14)	50.4(16)	43.1(15)	-4(12)	1.5(12)	-4.5(13)
C29	72(2)	62.8(19)	45.8(17)	-6.4(15)	4.8(15)	-1.4(18)
C10	41.7(15)	42.8(15)	46.8(15)	-2.4(12)	2.8(12)	0.3(12)
C26	51.7(18)	48.8(17)	39.9(15)	1(12)	0.6(12)	-6.9(14)
C7	53.1(17)	43.4(15)	49.9(17)	-2.1(12)	0(13)	-0.5(13)
C18	49(18)	64.7(18)	49.7(17)	-3.6(14)	-1(13)	-12.8(16)
C20	63(18)	48.9(18)	37.2(15)	1.7(12)	-1.4(13)	-3.9(15)
C17	47.9(17)	62.7(18)	49.3(17)	-0.6(14)	-5.1(13)	0.8(14)
C13	42.2(15)	51.5(16)	64.2(17)	-7.4(13)	-2(15)	-3.6(15)
C27	54.8(18)	56.4(17)	48.7(16)	-8.4(13)	5.1(14)	-11.2(15)
C4	49.7(18)	51.8(17)	47.1(16)	3(13)	-0.8(13)	-8.9(14)
C19	43.3(16)	46.9(16)	44.9(15)	0.6(11)	-6.5(12)	-2.5(13)
C8	55.4(18)	71(2)	53(17)	-1.3(14)	-3.7(14)	-14.9(16)
C6	92(3)	85(3)	70(2)	27.5(19)	-26(2)	-17(2)
C31	55.6(18)	74(2)	46.7(17)	-6.8(14)	-3.6(14)	-14.9(17)
C28	50.2(19)	70(2)	60.4(19)	-10.4(15)	7.7(14)	-7.8(16)
C15	72(2)	80(2)	56(2)	-24.5(16)	19.2(17)	-31(2)
C14	50.5(19)	57.4(17)	82(2)	-18.3(16)	15(18)	-8.7(16)
C21	76(2)	57(2)	50.1(18)	-7.6(14)	-5.2(16)	11.7(17)
C16	64(2)	88(2)	45.9(18)	-0.7(16)	-8.2(16)	-20.4(19)
C3	61(2)	67(19)	50.6(18)	-6.3(14)	0.7(15)	-0.7(17)
C25	84(3)	61(2)	66(2)	-7.5(16)	-6.1(18)	-11.8(19)
C30	75(2)	63.8(19)	51(18)	-18.5(15)	2(17)	-15.1(19)
C5	72(2)	56.1(19)	67(2)	11.9(15)	-11(17)	-4.4(17)
C9	82(3)	80(2)	97(2)	10.4(18)	-9(2)	-33(2)

C32	102(3)	80(2)	78(2)	-17.1(17)	2(2)	15(2)
C2	79(3)	88(3)	67(2)	-16.1(19)	7(2)	-11(2)
C1	108(3)	98(3)	51(2)	0(2)	-6(2)	-27(3)
C23	140(4)	48(2)	91(3)	-7.5(17)	-3(3)	17(3)
C22	107(3)	69(2)	66(2)	-2.7(18)	-5(2)	26(2)
C24	114(3)	58(2)	102(3)	-18(19)	-7(3)	-17(2)

Table 4 Bond Lengths for ncy-2.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
S1	O1	1.4341(18)	C20	C25	1.370(4)
S1	O2	1.4355(19)	C20	C21	1.387(4)
S1	N1	1.618(2)	C20	C19	1.526(3)
S1	C26	1.764(3)	C17	C16	1.385(4)
N1	C18	1.469(3)	C13	C14	1.373(3)
N1	C19	1.471(3)	C27	C28	1.384(4)
C11	C12	1.518(3)	C4	C5	1.376(4)
C11	C18	1.545(3)	C4	C3	1.382(3)
C11	C10	1.549(3)	C8	C9	1.509(4)
C12	C17	1.383(3)	C6	C1	1.368(5)
C12	C13	1.391(3)	C6	C5	1.394(4)
C29	C28	1.383(4)	C31	C30	1.379(4)
C29	C30	1.387(4)	C15	C16	1.365(4)
C29	C32	1.518(4)	C15	C14	1.378(4)
C10	C7	1.542(3)	C21	C22	1.382(4)
C10	C19	1.556(3)	C3	C2	1.380(4)
C26	C31	1.380(3)	C25	C24	1.408(4)
C26	C27	1.383(4)	C2	C1	1.368(4)
C7	C4	1.522(3)	C23	C22	1.356(5)
C7	C8	1.542(4)	C23	C24	1.362(5)

Table 5 Bond Angles for ncy-2.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1	S1	O2	120.55(12)	C25	C20	C19	120.0(3)
O1	S1	N1	106.33(11)	C21	C20	C19	122.2(3)
O2	S1	N1	106.55(11)	C12	C17	C16	121.3(3)
O1	S1	C26	107.72(12)	C14	C13	C12	120.7(3)
O2	S1	C26	108.04(13)	C26	C27	C28	119.6(3)
N1	S1	C26	106.95(11)	C5	C4	C3	117.9(3)
C18	N1	C19	112.15(19)	C5	C4	C7	121.0(3)
C18	N1	S1	121.45(16)	C3	C4	C7	121.1(2)

C19	N1	S1	124.53(16)	N1	C19	C20	113.9(2)
C12	C11	C18	111.2(2)	N1	C19	C10	100.16(18)
C12	C11	C10	116.3(2)	C20	C19	C10	116.1(2)
C18	C11	C10	104.0(2)	C9	C8	C7	113.8(3)
C17	C12	C13	117.9(2)	C1	C6	C5	119.9(3)
C17	C12	C11	121.1(2)	C30	C31	C26	120.0(3)
C13	C12	C11	121.1(2)	C29	C28	C27	120.8(3)
C28	C29	C30	118.9(3)	C16	C15	C14	119.7(3)
C28	C29	C32	121.0(3)	C13	C14	C15	120.5(3)
C30	C29	C32	120.1(3)	C22	C21	C20	121.1(3)
C7	C10	C11	116.5(2)	C15	C16	C17	119.9(3)
C7	C10	C19	114.0(2)	C2	C3	C4	121.5(3)
C11	C10	C19	103.60(19)	C20	C25	C24	120.8(4)
C31	C26	C27	120.0(3)	C31	C30	C29	120.6(3)
C31	C26	S1	120.1(2)	C4	C5	C6	120.9(3)
C27	C26	S1	119.5(2)	C1	C2	C3	119.8(3)
C4	C7	C8	110.7(2)	C2	C1	C6	120.0(3)
C4	C7	C10	112.3(2)	C22	C23	C24	119.8(3)
C8	C7	C10	110.7(2)	C23	C22	C21	120.6(4)
N1	C18	C11	104.8(2)	C23	C24	C25	119.9(3)
C25	C20	C21	117.8(3)				

Table 6 Torsion Angles for ncy-2.

A	**B**	**C**	**D**	**Angle/°**
O1	S1	N1	C18	33.5(2)
O2	S1	N1	C18	163.27(19)
C26	S1	N1	C18	-81.4(2)
O1	S1	N1	C19	-163.32(19)
O2	S1	N1	C19	-33.6(2)
C26	S1	N1	C19	81.8(2)
C18	C11	C12	C17	120.0(3)
C10	C11	C12	C17	-121.2(3)
C18	C11	C12	C13	-59.3(3)
C10	C11	C12	C13	59.5(3)
C12	C11	C10	C7	78.8(3)
C18	C11	C10	C7	-158.6(2)
C12	C11	C10	C19	-155.1(2)
C18	C11	C10	C19	-32.5(2)
O1	S1	C26	C31	152.8(2)
O2	S1	C26	C31	21.1(2)

N1	S1	C26	C31	-93.2(2)
O1	S1	C26	C27	-33.6(2)
O2	S1	C26	C27	-165.3(2)
N1	S1	C26	C27	80.4(2)
C11	C10	C7	C4	-53.1(3)
C19	C10	C7	C4	-173.7(2)
C11	C10	C7	C8	-177.3(2)
C19	C10	C7	C8	62.0(3)
C19	N1	C18	C11	12.1(3)
S1	N1	C18	C11	177.18(16)
C12	C11	C18	N1	139.4(2)
C10	C11	C18	N1	13.5(3)
C13	C12	C17	C16	-1.0(4)
C11	C12	C17	C16	179.6(3)
C17	C12	C13	C14	0.5(4)
C11	C12	C13	C14	179.9(2)
C31	C26	C27	C28	2.4(4)
S1	C26	C27	C28	-171.2(2)
C8	C7	C4	C5	-120.8(3)
C10	C7	C4	C5	115.0(3)
C8	C7	C4	C3	58.4(3)
C10	C7	C4	C3	-65.8(3)
C18	N1	C19	C20	92.8(2)
S1	N1	C19	C20	-71.8(3)
C18	N1	C19	C10	-31.9(3)
S1	N1	C19	C10	163.54(17)
C25	C20	C19	N1	131.6(3)
C21	C20	C19	N1	-45.0(3)
C25	C20	C19	C10	-112.8(3)
C21	C20	C19	C10	70.6(3)
C7	C10	C19	N1	166.2(2)
C11	C10	C19	N1	38.5(2)
C7	C10	C19	C20	43.0(3)
C11	C10	C19	C20	-84.6(3)
C4	C7	C8	C9	60.8(3)
C10	C7	C8	C9	-174.0(2)
C27	C26	C31	C30	-2.2(4)
S1	C26	C31	C30	171.3(2)
C30	C29	C28	C27	-1.6(4)
C32	C29	C28	C27	176.8(3)

C26	C27	C28	C29	-0.5(4)
C12	C13	C14	C15	0.4(4)
C16	C15	C14	C13	-0.9(4)
C25	C20	C21	C22	1.2(4)
C19	C20	C21	C22	177.9(3)
C14	C15	C16	C17	0.4(5)
C12	C17	C16	C15	0.5(4)
C5	C4	C3	C2	-0.5(4)
C7	C4	C3	C2	-179.8(3)
C21	C20	C25	C24	-1.4(4)
C19	C20	C25	C24	-178.1(3)
C26	C31	C30	C29	0.1(4)
C28	C29	C30	C31	1.8(4)
C32	C29	C30	C31	-176.7(3)
C3	C4	C5	C6	0.0(4)
C7	C4	C5	C6	179.3(3)
C1	C6	C5	C4	-0.3(5)
C4	C3	C2	C1	1.3(5)
C3	C2	C1	C6	-1.6(5)
C5	C6	C1	C2	1.1(5)
C24	C23	C22	C21	-0.1(6)
C20	C21	C22	C23	-0.5(5)
C22	C23	C24	C25	0.0(6)
C20	C25	C24	C23	0.8(5)

Table 7 Hydrogen Atom Coordinates ($Å \times 10^4$) and Isotropic Displacement Parameters ($Å^2 \times 10^3$) for ncy-2.

Atom	***x***	***y***	***z***	**U(eq)**
H11	3813	5005	310	54
H10	7256	4156	430	53
H7	6619	6104	208	59
H18B	2718	4186	970	65
H18A	3825	3263	828	65
H17	2573	4631	-494	64
H13	5792	2683	149	63
H27	1306	4068	1933	64
H19	7690	4848	1203	54
H8B	9372	5812	644	72
H8A	9981	5138	195	72
H6	5140	6055	-1480	99

H31	5546	5561	2445	71
H28	-603	5258	2218	72
H15	3549	2187	-1225	83
H14	5335	1702	-546	76
H21	3258	5851	1022	73
H16	2155	3647	-1196	79
H3	8737	4169	-459	72
H25	8293	6400	1463	84
H30	3635	6741	2732	76
H5	5409	6376	-606	78
H9A	10106	6421	-367	129
H9C	11318	6636	114	129
H9B	9406	7104	67	129
H32A	-1113	6824	2529	130
H32C	526	7529	2525	130
H32B	42	6969	3029	130
H2	8438	3837	-1324	94
H1	6699	4804	-1838	103
H23	4458	8392	1609	111
H22	2403	7358	1268	96
H24	7404	7929	1707	110

Experimental

Single crystals of $C_{32}H_{33}NSO_2$ were recrystallised from hexane and ethyl acetate mounted in inert oil and transferred to the cold gas stream of the diffractometer.

Crystal structure determination of [ncy-2]

Crystal Data. $C_{32}H_{33}NSO_2$, M =495.65, Orthorhombic, a = 7.4163(3) Å, b = 14.0432(6) Å, c = 25.9274(10) Å, U = 2700.30(19) Å^3, T = 291(2), space group $P2_12_12_1$ (no. 19), Z = 4, μ(Mo-Kα) = 0.149, 14601 reflections measured, 5528 unique (R_{int} = 0.0442) which were used in all calculations. The final $wR(F_2)$ was 0.1012 (all data).