Enantioselective Synthesis of Cyclohexenol Derivatives from γ-Aryl-substituted Enals via an Organocatalyzed Three-Component Reaction

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Supporting Information

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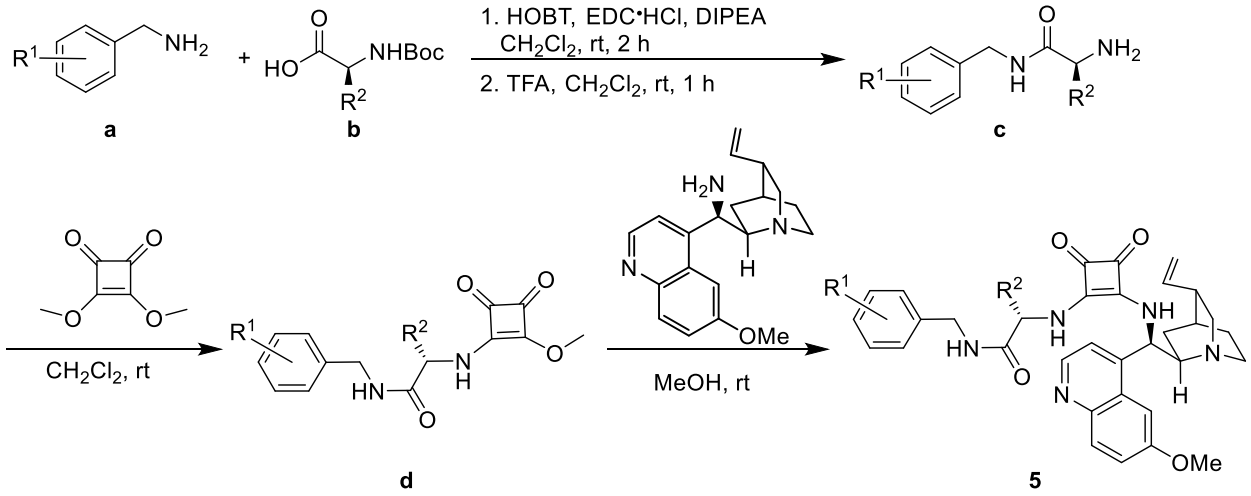
General Information

All reactions were carried out in closed vial and monitored by TLC and the products were visualized by UV detection. Flash column chromatography was performed with silica gel (32-63 μ). ^{1}H and ^{13}C NMR spectra were recorded on a 500 MHz (125 MHz for ^{13}C NMR) or a 300 MHz spectrometer (75 MHz for ^{13}C NMR). The following abbreviations were used to designate chemical shift mutiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. All first-order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted are designated as multiplet (m) or broad (br). FTIR spectra were measured on a Bruker Vector 22 instrument. Enantiomeric excesses (ee) were determined by HPLC analysis using a Shimadzu instrument with chiral HPLC columns. ChiralPak IB, IC, and ID columns (4.6 mm × 250 mm) were purchased from Daicel Chemical Industries. Melting points were recorded on MEL-TEMP melting point apparatus in open capillaries and uncorrected. HRMS analyses were conducted at the RCMI Proteomics and Protein Biomarkers Core Facility of the Department of Chemistry, UTSA.

Materials: All the α,β-unsaturated aldehydes were prepared by following the literature procedure.[12] All the nitroalkenes were either purchased from commercial sources or synthesized by following known literature procedures.[13] All catalysts, except for **5e**, **5f**, **5g**, and **5y**, are known compounds and were prepared by following the known procedures.[14] Tetrahydrofuran was dried over sodium metal under argon atmosphere and distilled before prior to use.

Catalyst Preparation



Scheme S-1. Synthesis of the squaramide catalysts **5**.

Preparation of intermediate c: Intermediate **c** was prepared according to the literature procedure.[15] In a 250-mL round-bottom flask, the Boc-protected amino acid **b** (5.0 mmol), hydroxybenzotriazole (HOBT, 676 mg, 5.0 mmol, 1.0 equiv.), and EDC hydrochloride (959 mg, 5.0 mmol, 1.0 equiv.) were suspended in dry CH_2Cl_2 (35 mL) at room temperature. After 2 min., DIPEA (1.3 g, 10 mmol, 2.0 equiv.) was added, which was followed immediately by the benzylamine derivative **a** (5.0 mmol, 1.0 equiv.). The reaction mixture was allowed to stir further for 2 h at the same temperature. During the reaction progress, due to the consumption of HOBT, the mixture changed from turbid to a clear solution. The reaction was then quenched by adding 1N HCl (20 mL) and the mixture was extracted with CH_2Cl_2 (3 × 30 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The resulting colorless liquid was further dissolved in CH_2Cl_2 (25 mL) and treated with trifluoroacetic acid (75 mmol, 15 equiv.) under stirring. After 1 h the reaction mixture was diluted with CH_2Cl_2 (30 mL) and quenched with chilled saturated solution of sodium carbonate (50 mL). After neutralization by adding more saturated sodium carbonate solution, the aqueous layer was extracted with CH_2Cl_2 (3 × 20 mL), and the combined organic layers were dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure to give a white solid that was used without further purification.

Synthesis of intermediate d: The crude intermediate **c** (5.0 mmol) was dissolved in CH_2Cl_2 (25 mL) and dimethylsquarate (710.6 mg, 5.0 mmol) was added portion wise over 10 min. at room temperature. The mixture was further stirred for 72 h to complete the reaction (monitored by TLC). The solvent was then removed under reduced pressure and the crude product was purified by in flash column chromatography using 98:2 CH_2Cl_2/MeOH as the eluent to give the intermediate **d** (63-72%).

Synthesis of 5: In an oven-dried round-bottom flask, the quinine- or quinidine-derived 9-amino(9-deoxy)cinchona alkaloid (255.5 mg, 0.79 mmol) was dissolved in dry methanol (10 mL) at room temperature. To that solution, **d** (0.79 mmol) was added in one portion and the mixture was further stirred for 48-90 h. During the progress, a white precipitate was observed. The precipitate was filtered and washed with cold MeOH/hexane (1:4), which gave the pure catalyst **5**. The filtrate was further concentrated and purified by silica gel flash column chromatography using MeOH/CH_2Cl_2 (0:100 to 5:95) as a mobile phase to give an additional portion of the catalyst. Both the portions were combined for catalytic use (total yield 55-60%).

Detailed Experimental Procedures

General procedure for the three-component reaction: To a stirred solution of the appropriate nitroalkene (0.60 mmol, 3.0 equiv.) and the squaramide catalyst **5f** (24.9 mg, 0.040 mmol, 20 mol %) in freshly distilled THF (0.7 mL) at room temperature, the γ-aryl-α,β-unsaturated aldehyde (0.20 mmol) was added. The mixture was further stirred at room temperature for 72 h (the progress of the reaction was monitored by TLC). After the completion of the reaction, the solvent was evaporated in a rotary evaporator under the reduced pressure and the crude product obtained was purified by flash column chromatography using 90:10 to 95:5 hexane/EtOAc as an eluent.

1.0 mmol-scale reaction

To a stirred solution of *trans*-β-nitrostyrene (**2a**, 447.5 mg, 3.0 mmol, 3.0 equiv.) and the squaramide catalyst **5f** (124.4 mg, 0.20 mmol, 0.2 equiv) in freshly distilled THF (3.5 mL) at room temperature, 4-phenyl-2-butenal (**1a**, 146.2 mg, 1.0 mmol) was added. The mixture was further stirred at room temperature for 72 h (the progress of the reaction was monitored by TLC). After the completion of the reaction, the solvent was evaporated in a rotary evaporator under reduced pressure and the crude product obtained was purified by flash column chromatography using 95:5 hexanes/EtOAc as an eluent to give **4a** as white color solid (310.5 mg, 70% yield, 87:13 dr, 99% ee).

Acetylation of the adduct 4a

To a solution of the adduct **4a** (44.5 mg, 0.10 mmol) in CH_2Cl_2 (3.0 mL), acetic anhydride (0.10 mL, 1.0 mmol) and DMAP (2.5 mg, 0.020 mmol) were added at 0 °C and the reaction mixture was further stirred for 30 min at room temperature. After the completion the reaction, the solvent was evaporated under reduced pressure, and the crude product was purified by flash chromatography using 93:7 hexanes/EtOAc as an eluent to give **6** as white solid (38.7 mg, 80% yield).

Oxidation of the adduct 4a

To a solution of the adduct **4a** (44.5 mg, 0.10 mmol) in CH_2Cl_2 (3.0 mL), 100 mg Celite was suspended and then PCC (64.7 mg, 0.30 mmol) were added at room temperature and the reaction mixture was further stirred for 12 h at the same temperature. After the completion the reaction, the mixture was filtered through a Celite pad and the crude product was purified by flash chromatography using 92:8 hexane/EtOAc as an eluent to give **7** as white solid. (37.6 mg, 85% yield).

Detailed Catalyst Screening and Condition Optimizations

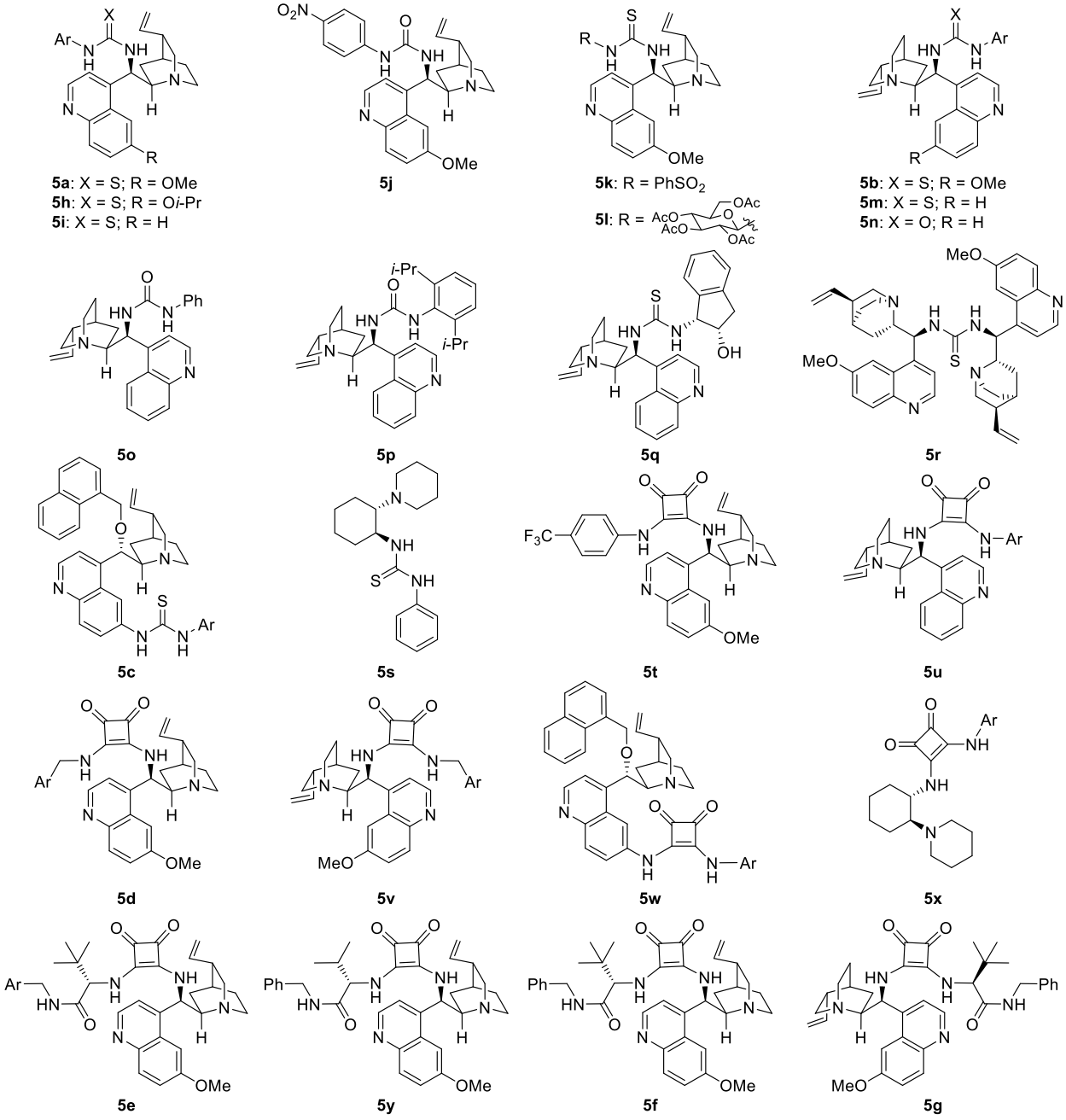
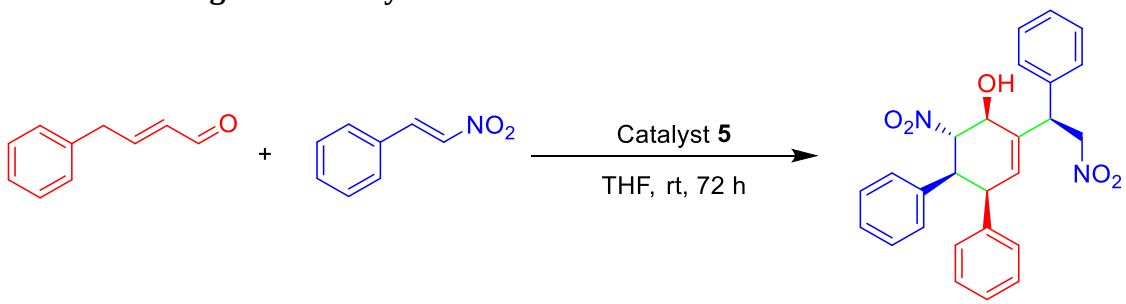


Figure S-1. Structure of catalyst used in the screening

Table S-1: Screening of the catalysts[a]



1a **2a** **4a**

Entry	Catalyst	Yield (%)[b]	dr (%)[c]	ee (%)[d]
1	**5a**	41	60:40	53
2	**5h**	42	59:41	56
3	**5i**	60	61:39	58
4	**5j**	28	61:39	79
5	**5k**	0	-	-
6	**5l**	32	50:50	54
7	**5b**	53	65:35	64[e]
8	**5m**	47	64:36	56[e]
9	**5n**	37	66:34	70[e]
10	**5o**	49	70:30	83[e]
11	**5p**	55	80:20	90[e]
12	**5q**	35	59:41	62[e]
13	**5r**	23	61:39	61[e]
14	**5c**	0	-	-
15	**5s**	0	-	-
16	**5t**	56	77:23	89
17	**5u**	49	80:20	94[e]
18	**5d**	51	79:21	95
19	**5v**	48	85:15	96[e]
20	**5w**	0	-	-
21	**5x**	0	-	-
22	**5e**	34	70:30	97
23	**5f**	80	88:12	99
24	**5g**	75	81:19	97[e]
25	**5y**	67	79:21	96
26[f]	**5f**	34	87:13	99

[a]Unless otherwise noted, all reactions were carried out with **1a** (0.20 mmol) and **2a** (0.6 mmol) in dry THF (0.7 mL) at room temperature for 72 h, using **5** (20 mol %) as the catalyst. [b]Yield of the isolated product. [c]Diastereomeric ratio was determined by ^{1}H NMR analysis of the crude reaction mixture. [d]Determined by chiral HPLC analysis. [e]The opposite enantiomer was obtained as the major product. [f]The catalyst loading was 10 mol %.

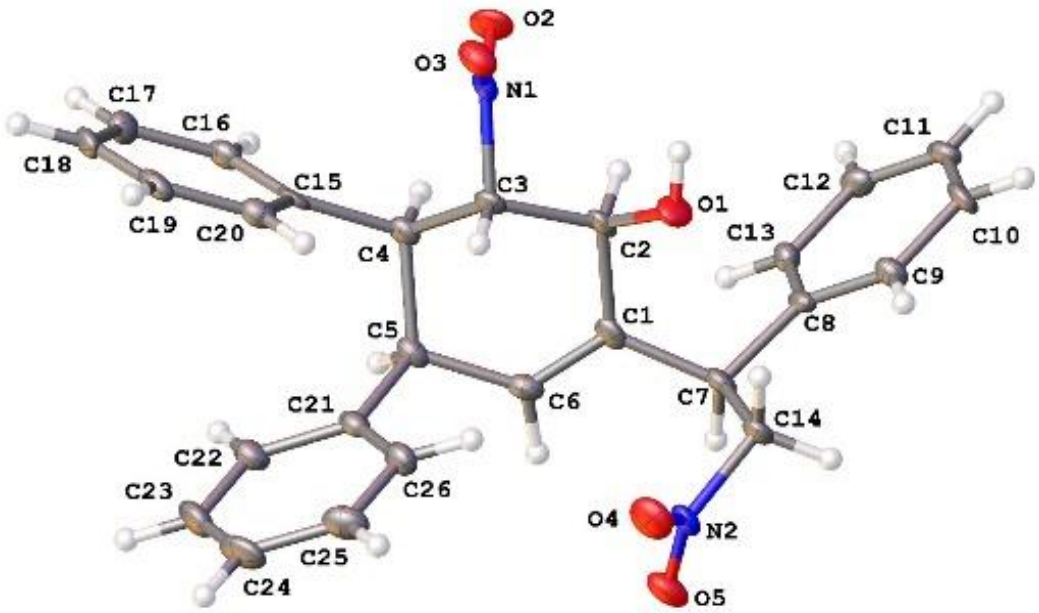
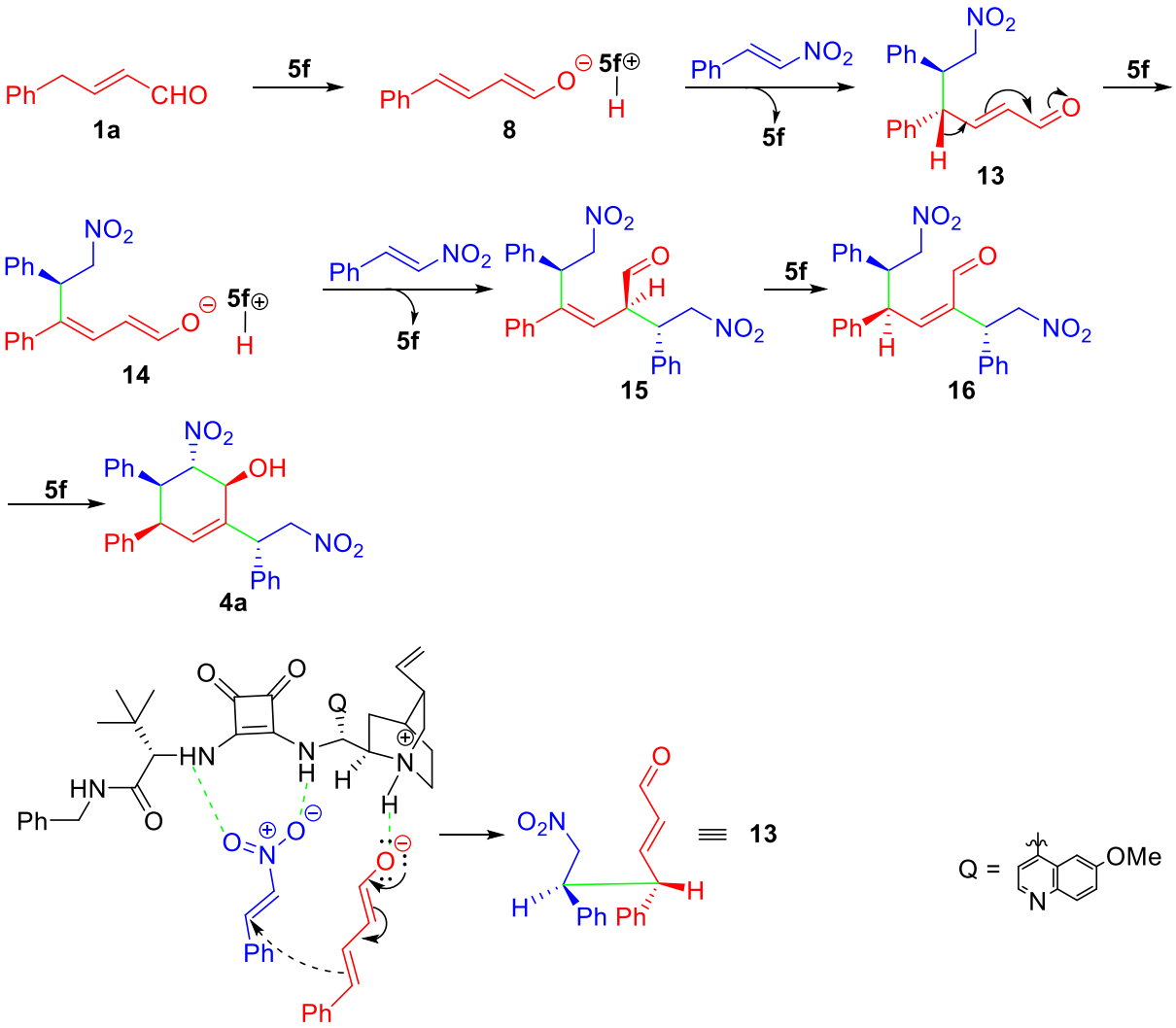


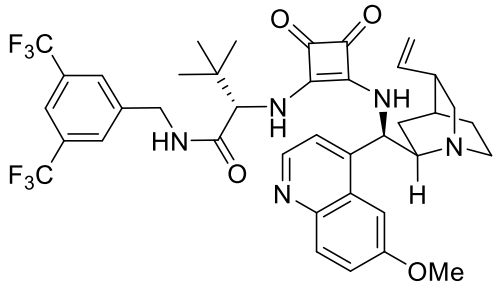
Figure S-2. ORTEP drawing of compound **4a**



Scheme S-2. Alternative reaction mechanism for the three-component reaction

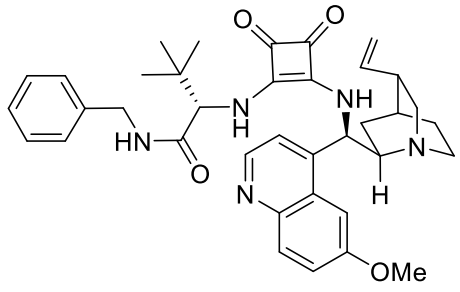
Compound Characterization Data

3-{(*S*)-*N*'-[3,5-Bis(trifluoromethyl)benzyl]-3,3-dimethylbutanamid-2-ylamino}-4-{[(9*R*)-6'-methoxycinchonan-9-yl]amino}-3-cyclobutene-1,2-dione (5e)



The crude product was purified by silica gel flash column chromatography using MeOH/CH_2Cl_2 (0:100 to 5:95) to get **5e** as a white solid, 2.01 g, 55% yield, m.p. 178-180 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.88 (s, 2H), 8.56 (s, 2H), 8.06 (d, *J* = 9.2 Hz, 1H), 7.95 (s, 1H), 7.70 (s, 1H), 7.44-7.40 (m, 2H), 7.27 (s, 1H), 7.12 (br, 1H), 6.51 (br, 1H), 5.97 (d, *J* = 14.3 Hz, 1H), 5.29 (d, *J* = 16.9 Hz, 1H), 5.13 (d, *J* = 10.0 Hz, 1H), 4.47 (s, 2H), 4.14 (br, 1H), 3.89 (s, 3H), 3.42 (br, 2H), 3.06 (s, 3H), 2.41 (s, 1H), 1.92 (s, 3H), 1.72 (s, 3H), 1.16-1.04 (m, 2H), 0.85 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 183.0, 181.7, 171.8, 169.2, 165.5, 159.0, 148.1, 145.2, 144.7, 141.0, 140.8, 132.2, 132.1 (q, $J_{C\text{-}F}$ = 33.7 Hz), 125.8, 125.3 (q, $J_{C\text{-}F}$ = 273.0 Hz), 122.7, 121.0, 119.8 (br.), 119.5, 115.1, 101.9, 64.8, 59.3, 56.0, 53.6, 49.5, 47.1, 42.1, 39.7, 35.6, 28.1, 27.4, 26.5, 26.1. ν_{max} (neat, cm^{-1}): 3234, 2936, 1660, 1622, 1582, 1509, 1434, 1379, 1349, 1275, 1243, 1170, 1128, 1021. HRMS (ESI) *m/z* calcd for $C_{39}H_{42}F_6N_5O_4^+$ ([M+H]): 758.3136; found: 758.3146.

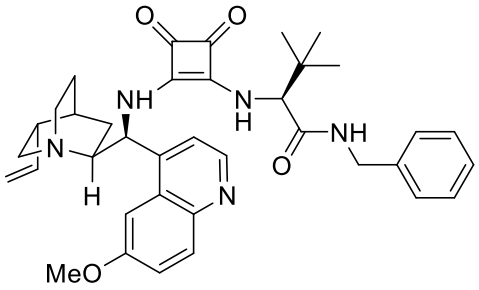
3-[(*S*)-*N*'-Benzyl-3,3-dimethylbutanamid-2-ylamino]-4-{[(9*R*)-6'-methoxycinchonan-9-yl]amino}-3-cyclobutene-1,2-dione (5f)



The crude product was purified by silica gel flash column chromatography using MeOH/CH_2Cl_2 (0:100 to 3:97) to get **5f** as a white solid, 1.86 g, 60% yield, m.p. 190-192 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.70 (br, 1H), 8.54-8.62 (m, 1H), 8.42 (br, 1H), 8.05 (d, *J* = 9.1 Hz, 1H), 7.98 (s, 1H), 7.81 (s, 1H), 7.43 (d, *J* = 9.3 Hz, 1H), 7.26 (br, 1H), 7.08-7.06 (m, 3H), 6.84-6.83 (m, 2H), 6.51 (br, 1H), 5.90 (ddd, J_1 = 17.0 Hz, J_2 = 10.5, J_3 = 6.2 Hz, 1H), 5.20 (d, *J* = 17.1 Hz, 1H), 5.05 (d, *J* = 10.3 Hz, 1H), 4.54 (d, *J* = 9.5 Hz, 1H), 4.13 (s, 2H), 3.92 (s, 3H), 3.78-3.75 (m, 1H), 3.36 (s, 1H), 3.24 (s, 1H), 2.97-2.93 (m, 2H), 2.30-2.28 (m, 2H), 1.64-1.57 (m, 3H), 1.11 (s, 1H), 0.99 (s, 1H), 0.81 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 183.0,

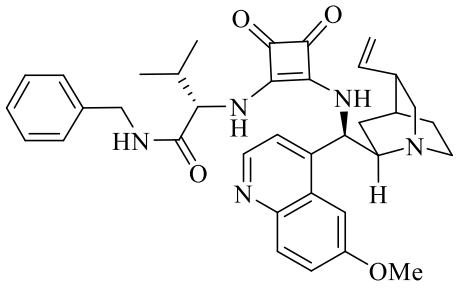
182.1, 170.8, 168.9, 166.1, 158.8, 148.2, 145.1, 140.8, 137.7, 132.0, 128.8, 128.4, 127.6, 122.7, 119.9, 115.1, 101.9, 64.9, 59.3, 56.1, 53.3, 49.4, 47.2, 43.4, 39.8, 35.9, 28.2, 27.3, 26.7, 25.8, 23.0, 14.5, 12.4 ν_{max} (neat, cm^{-1}): 3301, 2932, 1795, 1652, 1621, 1582, 1509, 1432, 1368, 1227, 1128. HRMS (ESI) *m/z* calcd for $C_{37}H_{44}N_5O_4$ ($[M+H]^+$): 622.3388; found: 622.3390.

3-[(*S*)-*N*'-Benzyl-3,3-dimethylbutanamid-2-ylamino]-4-{[(8α,9*S*)-6'-methoxycinchonan-9-yl]amino}-3-cyclobutene-1,2-dione (5g)



The crude product was purified by silica gel flash column chromatography using MeOH/CH_2Cl_2 (0:100 to 4:96) to get **5g** as a white solid, 1.71 g, 55% yield, m.p. 182-184°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.45 (br, 1H), 8.01-7.98 (m, 3H), 7.57 (s, 2H), 7.38-7.36 (m, 1H), 7.02-6.94 (m, 4H), 6.28 (br, 1H), 5.89 (br, 1H), 5.03-4.93 (m, 2H), 4.58 (s, 1H), 3.94-3.88 (m, 5H), 3.40 (s, 1H), 3.24 (t, *J* = 10 Hz, 1H), 2.85-2.76 (m, 2H), 2.29 (d, *J* = 7.2 Hz, 1H), 1.64-1.44 (m, 4H), 0.88 (br, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 183.3, 170.8, 168.8, 167.2, 159.0, 148.2, 145.1, 144.8, 142.1, 137.9, 132.2, 129.4, 128.7, 127.6, 122.6, 120.0, 114.9, 102.0, 64.9, 56.2, 53.9, 43.5, 41.2, 40.1, 35.4, 28.3, 26.5. ν_{max} (neat, cm^{-1}): 33264, 2942, 1657, 1621, 1581, 1524, 1454, 1367, 1259, 1228, 1176, 1132, 1028. HRMS (ESI) *m/z* calcd for $C_{37}H_{44}N_5O_4$ ($[M+H]^+$): 622.3388; found: 622.3401.

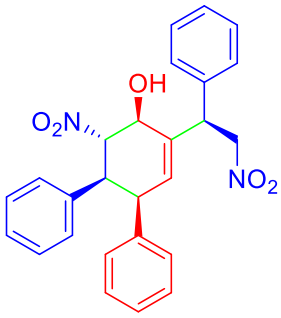
3-[(*S*)-*N*'-Benzyl-3-methylbutanamid-2-ylamino]-4-{[(9*R*)-6'-methoxycinchonan-9-yl]amino}-3-cyclobutene-1,2-dione (5y)



The crude product was purified by silica gel flash column chromatography using MeOH/CH_2Cl_2 (0:100 to 4:96) to get **5y** as a white solid, 1.67 g, 58% yield, m.p. 170-172 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.75 (br, 2H), 8.50 (s, 1H), 8.09-8.04 (m, 2H), 7.81 (s, 1H), 7.46 (d, *J* = 9.3 Hz, 2H), 7.11 (s, 3H), 6.88 (s, 2H), 6.54 (s, 1H), 5.93 (td, J_1 = 11.0 Hz, J_2 = 10.3 Hz, J_3 = 5.7 Hz, 1H), 5.25 (d, *J* = 17.2 Hz, 1H), 5.09 (d, *J* = 10.4 Hz, 1H), 4.60 (s, 1H), 4.10-3.94 (m, 6H), 3.72 (s, 1H), 3.35 (s, 1H), 3.22 (s, 1H), 3.03-3.00 (m, 2H), 2.33 (d, *J* = 8.1 Hz, 1H), 1.94-1.86 (m, 2H), 1.67-1.62 (m, 3H), 1.14 (s, 1H), 1.07-0.89 (m, 2H), 0.81 (d, *J* = 6.4 Hz, 3H),

0.73 (d, J = 5 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 183.2, 181.55, 171.7, 169.2, 165.6, 158.7, 148.2, 145.3, 145.1, 140.9, 137.6, 132.0, 128.8, 128.3, 127.6, 127.4, 122.8, 119.9, 115.0, 101.9, 63.0, 59.2, 56.0, 53.4, 49.4, 47.1, 43.2, 39.8, 33.3, 28.2, 27.3, 25.8, 18.9, 18.7, 12.3. ν_{max} (neat, cm^{-1}): 3232, 2933, 1655, 1621, 1509, 1452, 1367, 1260, 1241, 1228, 1159, 1080, 1027. HRMS (ESI) *m/z* calcd for $C_{36}H_{42}N_5O_4^+$ ([M+H]): 608.3231; found: 608.3249.

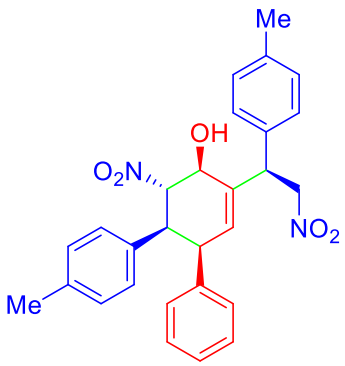
(1'*R*,2'*S*,3'*S*,4'*S*)-3'-Nitro-5'-[(*R*)-2-nitro-1-phenylethyl]-1',2',3',4'-tetrahydro-[1,1':2',1''-terphenyl]-4'-ol (4a)



The crude material was purified by flash column chromatography using hexanes/EtOAc (95:5) as an eluent to get **4a** as a white solid, 71 mg, 80% yield, m.p. 140-142 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.46-7.31 (m, 5H), 7.21-7.19 (m, 1H), 7.17-7.09 (m, 3H), 7.05 (t, J = 7.4 Hz, 2H), 6.61-6.55 (m, 4H), 6.02 (d, J = 4.2 Hz, 1H), 5.24 (dd, J_1 = 12.3 Hz, J_2 = 9.7 Hz, 1H), 5.11 (dd, J_1 = 12.0 Hz, J_2 = 8.5 Hz, 1H), 4.95 (dd, J_1 = 12.2 Hz, J_2 = 6.9 Hz, 1H), 4.80 (t, J = 8.0 Hz, 1H), 4.63 (dd, J_1 = 9.6 Hz, J_2 = 7.0 Hz, 1H), 3.79-3.76 (m, 2H), 2.63 (d, J = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 137.23, 137.0, 136.5, 136.2, 130.8, 130.2, 129.6, 128.5, 128.1, 128.0, 79.0, 73.4, 49.0, 48.7, 47.8. ν_{max} (neat, cm^{-1}): 3586, 3029, 1550, 1453, 1433, 1378, 1246, 1060, 1033, 970. HRMS (ESI) *m/z* calcd for $C_{26}H_{24}N_2NaO_5$ ($[M+Na]^+$): 467.1577; found: 467.1573. Enantiomeric excess of **4a** was determined by chiral stationary phase HPLC analysis using a ChiralPak IC column (90:10 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 18.0 min, major enantiomer: t_R = 50.0 min.

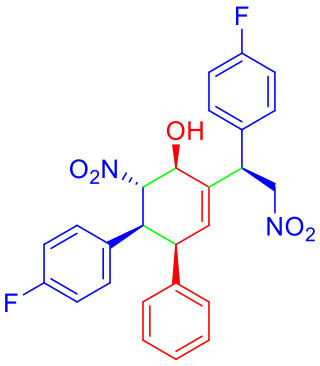
(1'*R*,2'*S*,3'*S*,4'*S*)-4''-Methyl-3'-nitro-5'-[(*R*)-2-nitro-1-(*p*-tolyl)ethyl]-1',2',3',4'-tetrahydro-[1,1':2',1''-terphenyl]-4'-ol (4b)

The crude material was purified by flash column chromatography using hexanes/EtOAc (93:7) as an eluent to get **4b** as a white solid, 68.9 mg, 73% yield, m.p. 130-132 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.29-7.24 (m, 3H), 7.23-7.19 (m, 4H), 7.17-7.14 (m, 2H), 6.85 (d, J = 7.7 Hz, 3H), 6.63 (d, J = 7.4 Hz, 2H), 6.44 (br, 2H), 6.00 (d, J = 3.4 Hz, 1H), 5.21 (dd, J_1 = 12.2 Hz, J_2 = 9.7 Hz, 1H), 5.08 (dd, J_1 = 12.6 Hz, J_2 = 8.5 Hz, 1H), 4.91 (dd, J_1 = 12.3 Hz, J_2 = 7.0 Hz, 1H), 4.79 (t, J = 8.1 Hz, 1H), 4.59 (d, J = 7.3 Hz, 1H), 3.80-3.65 (m, 2H), 2.55 (d, J = 7.6 Hz, 1H), 2.36 (s, 3H),



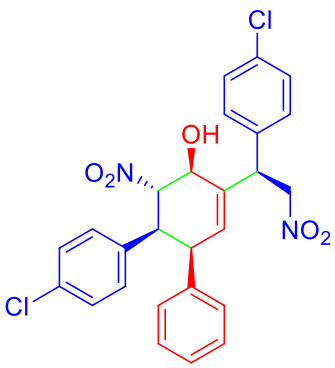
2.21 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 138.3, 137.6, 137.1, 136.7, 134.2, 133.1, 130.7, 130.3, 130.3, 129.1, 128.4, 128.4, 127.9, 127.9, 90.8, 79.1, 73.5, 48.7, 47.6, 21.4, 21.4. ν_{max} (neat, cm^{-1}): 3542, 2922, 1548, 1514, 1452, 1374, 1265, 1050, 976, 899. HRMS (ESI) *m/z* calcd for $C_{28}H_{27}N_2O_5$ ([M-H]$^-$): 471.1925; found: 471.1938. Enantiomeric excess of **4b** was determined by chiral stationary phase HPLC analysis using a ChiralPak IB column (90:10 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 21.1 min, major enantiomer: t_R = 22.9 min.

(1'*R*,2'*S*,3'*S*,4'*S*)-4''-Fluoro-5'-[(*R*)-1-(4-fluorophenyl)-2-nitroethyl]-3'-nitro-1',2',3',4'-tetrahydro-[1,1':2',1''-terphenyl]-4'-ol (4c)



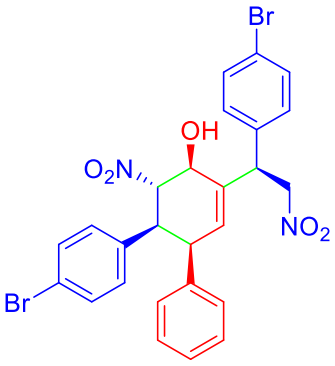
The crude material was purified by flash column chromatography using hexanes/EtOAc (91:9) as an eluent to get **4c** as a white solid, 67.2 mg, 70% yield, m.p. 134-136 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.36-7.33 (m, 2H), 7.24-7.21 (m, 1H), 7.18-7.15 (m, 2H), 7.08-7.05 (m, 2H), 6.76 (t, *J* = 8.5 Hz, 2H), 6.64-6.57 (m, 3H), 6.52 (br, 2H), 5.98 (d, *J* = 3.3 Hz, 1H), 5.18 (dd, J_1 = 12.4 Hz, J_2 = 9.5 Hz, 1H), 5.09-5.02 (m, 1H), 4.91 (dd, J_1 = 12.3 Hz, J_2 = 7.1 Hz, 1H), 4.78 (t, *J* = 7.4 Hz, 1H), 4.62 (dd, J_1 = 9.3, J_2 = 7.2 Hz, 1H), 3.77-3.73 (m, 2H), 2.81 (d, *J* = 7.3 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ: 162.7 (d, $J_{C\text{-}F}$ = 239.4 Hz), 162.5 (d, $J_{C\text{-}F}$ = 239.4 Hz), 137.0, 136.3, 133.0, 131.9, 130.7, 130.2, 130.1 (d, $J_{C\text{-}F}$ = 7.5 Hz), 129.8 (d, $J_{C\text{-}F}$ = 7.5 Hz), 128.7, 128.2, 116.5 (d, $J_{C\text{-}F}$ = 21.3 Hz), 115.4 (d, $J_{C\text{-}F}$ = 21.3 Hz), 90.7, 79.0, 73.1, 48.6, 48.0, 47.1. ν_{max} (neat, cm^{-1}): 3526, 2922, 1603, 1548, 1507, 1452, 1347, 1224, 1160, 1050. HRMS (ESI) *m/z* calcd for $C_{26}H_{21}F_2N_2O_5$ ([M-H]$^-$): 479.1424; found: 479.1430. Enantiomeric excess of **4c** was determined by chiral stationary phase HPLC analysis using a ChiralPak IC column (90:10 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 10.3 min, major enantiomer: t_R = 31.8 min.

(1'*R*,2'*S*,3'*S*,4'*S*)-4''-Chloro-5'-[(*R*)-1-(4-chlorophenyl)-2-nitroethyl]-3'-nitro-1',2',3',4'-tetrahydro-[1,1':2',1''-terphenyl]-4'-ol (4d)



The crude material was purified by flash column chromatography using hexanes/EtOAc (92:8) as an eluent to get **4d** as a white solid, 71.8 mg, 70% yield, m.p. 140-142 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.37-7.34 (m, 3H), 7.31-7.29 (m, 2H), 7.23-7.20 (m, 2H), 7.19-7.16 (m, 2H), 7.05 (d, J = 8.2 Hz, 2H), 6.60 (d, J = 7.5 Hz, 2H), 6.49 (br, 2H), 5.97 (d, J = 3.2 Hz, 1H), 5.15 (dd, J_1 = 12.5 Hz, J_2 = 9.2 Hz, 1H), 5.06 (dd, J_1 = 12.1 Hz, J_2 = 8.3 Hz, 1H), 4.92-4.88 (m, 2H), 4.76 (t, J = 7.6 Hz, 1H), 4.61 (dd, J_1 = 9.1 Hz, J_2 = 7.3 Hz, 1H), 3.79-3.69 (m, 2H), 2.92 (d, J = 7.1 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 136.8, 136.1, 135.8, 134.6, 134.3, 134.0, 130.7, 130.2, 129.8, 129.7, 129.5, 128.7, 128.2, 90.5, 78.7, 73.0, 48.4, 47.9, 47.2. ν_{max} (neat, cm^{-1}): 3501, 2918, 1548, 1490, 1452, 1373, 1091, 1051, 1013, 972. HRMS (ESI) *m/z* calcd for $C_{26}H_{21}Cl_2N_2O_5$ ([M-H]$^-$): 511.0833; found: 511.0830. Enantiomeric excess of **4d** was determined by chiral stationary phase HPLC analysis using a ChiralPak IC column (90:10 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 11.0 min, major enantiomer: t_R = 32.5 min.

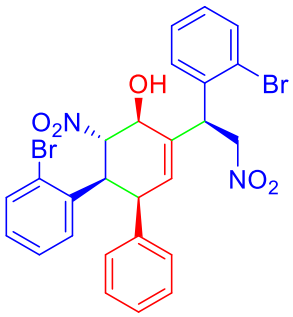
(1'*R*,2'*S*,3'*S*,4'*S*)-4''-Bromo-5'-[(*R*)-1-(4-bromophenyl)-2-nitroethyl]-3'-nitro-1',2',3',4'-tetrahydro-[1,1':2',1''-terphenyl]-4'-ol (4e)



The crude material was purified by flash column chromatography using hexanes/EtOAc (91:9) as an eluent to get **4e** as a light green solid, 90.3 mg, 75% yield, m.p. 150-152 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.51 (d, J = 8.4 Hz, 2H), 7.26-7.16 (m, 9H), 6.60 (d, J = 7.2 Hz, 2H), 6.43 (br, 2H), 5.98 (d, J = 3.5 Hz, 1H), 5.16 (dd, J_1 = 12.5 Hz, J_2 = 9.3 Hz, 1H), 5.05 (dd, J_1 = 12.6 Hz, J_2 = 8.4 Hz, 1H), 4.90 (dd, J_1 = 12.5 Hz, J_2 = 7.2 Hz, 1H), 4.76 (t, J = 7.6 Hz, 1H), 4.63-4.56 (m, 1H), 3.80-3.67 (m, 2H), 2.75 (d, J = 7.2 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 136.7, 136.3, 136.0, 135.1, 132.7, 131.7, 130.8, 130.2, 129.8, 128.8, 128.3, 122.5, 122.2, 90.4, 78.6, 73.1, 48.3, 48.1, 47.3. ν_{max} (neat, cm^{-1}): 3500, 2918, 1547, 1487, 1452, 1432, 1373, 1099, 1073, 1009, 904. HRMS (ESI) *m/z* calcd for $C_{26}H_{21}Br_2N_2O_5$ ([M-H]$^-$): 598.9823; found: 598.9813. Enantiomeric excess of **4e** was determined by chiral stationary phase HPLC analysis using a

ChiralPak IC column (92.5:7.5 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 15.1 min, major enantiomer: t_R = 53.3 min.

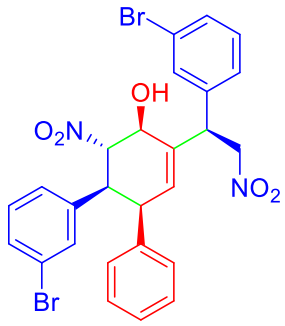
(1'*R*,2'*S*,3'*S*,4'*S*)-2''-Bromo-5'-[(*S*)-1-(2-bromophenyl)-2-nitroethyl]-3'-nitro-1',2',3',4'-tetrahydro-[1,1':2',1''-terphenyl]-4'-ol (4f)



The crude material was purified by flash column chromatography using hexanes/EtOAc (93:7) as an eluent to get **4f** as a brown solid, 78.3 mg, 65% yield, m.p. 125-127 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.66 (d, *J* = 6.7 Hz, 1H), 7.56 (dd, J_1 = 7.9 Hz, J_2 = 1.3 Hz, 1H), 7.43-7.41 (m, 1H), 7.25-7.23 (m, 1H), 7.16 (t, *J* = 7.6 Hz, 2H), 7.11 (t, *J* = 7.1 Hz, 2H), 6.98 (td, J_1 = 7.7 Hz, J_2 = 1.6 Hz, 1H), 6.79 (td, J_1 = 7.6 Hz, J_2 = 1.3 Hz, 1H), 6.61-6.68 (m, 2H), 6.04 (dd, J_1 = 7.9 Hz, J_2 = 1.6 Hz, 1H), 5.93 (d, *J* = 3.7 Hz, 1H), 5.20-5.13 (m, 3H), 4.99-4.94 (m, 1H), 4.76 (dd, J_1 = 8.5 Hz, 6.9 J_2 = Hz, 1H), 4.36 (dd, J_1 = 12.9 Hz, J_2 = 6.1 Hz, 1H), 4.03 (t, *J* = 4.9 Hz, 1H), 2.90 (d, *J* = 7.1 Hz, 1H).^{13}C NMR (125 MHz, $CDCl_3$) δ: 136.7, 135.9, 135.3, 134.7, 134.5, 133.4, 132.1, 129.9, 129.9, 129.3, 128.5, 128.4, 128.3, 128.3, 128.0, 127.2, 125.8, 125.0, 90.2, 77.8, 73.1, 47.4, 45.9, 45.1. ν_{max} (neat, cm^{-1}): 3503, 2919, 2850, 1548, 1489, 1471, 1452, 1373, 1263, 1062, 1021, 973. HRMS (ESI) *m/z* calcd for $C_{26}H_{21}Br_2N_2O_5$ ([M-H]$^-$): 598.9823; found: 598.9822. Enantiomeric excess of **4f** was determined by chiral stationary phase HPLC analysis using a ChiralPak IC column (90:10 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 19.6 min, major enantiomer: t_R = 40.4 min.

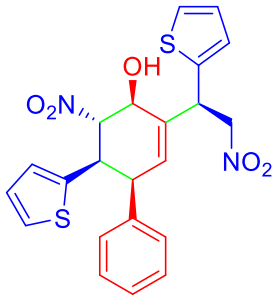
(1'*R*,2'*S*,3'*S*,4'*S*)-3''-Bromo-5'-[(*R*)-1-(3-bromophenyl)-2-nitroethyl]-3'-nitro-1',2',3',4'-tetrahydro-[1,1':2',1''-terphenyl]-4'-ol (4g)

The crude material was purified by flash column chromatography using hexanes/EtOAc (95:5) as an eluent to get **4g** as a light green solid, 86.7 mg, 68% yield, m.p. 125-127 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.52 (s, 1H), 7.48-7.45 (m, 2H), 7.33-7.18 (m, 8H), 6.91 (t, *J* = 7.9 Hz, 1H), 6.80 (br, 1H), 6.61 (d, *J* = 6.8 Hz, 2H), 6.41 (br, 1H), 6.00 (d, *J* = 4.1 Hz, 1H), 5.15 (dd, J_1 = 12.6 Hz,



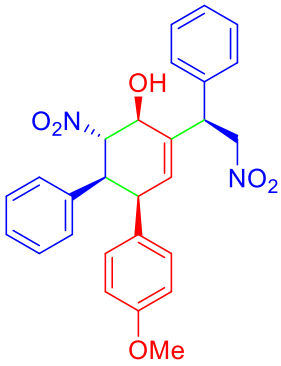
J_2 = 9.4 Hz, 1H), 5.06 (dd, J_1 = 12.3 Hz, J_2 = 8.4 Hz, 1H), 4.93-4.87 (m, 1H), 4.77 (t, J =10.0 Hz, 1H), 4.60 (dd, J_1 = 9.5 Hz, J_2 = 6.9 Hz, 1H), 3.80-3.73 (m, 2H), 2.81 (d, J = 7.1 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 139.6, 138.4, 136.6, 136.0, 132.0, 131.6, 131.3, 131.2, 131.1, 130.8, 130.2, 130.1, 129.9, 128.7, 128.3, 126.7, 123.6, 122.5, 90.2, 78.6, 73.1, 48.4, 48.1, 47.3. ν_{max} (neat, cm^{-1}): 3501, 2921, 2851, 1594, 1547, 1489, 1474, 1452, 1373, 1260, 1199, 996. HRMS (ESI) *m/z* calcd for $C_{26}H_{21}Br_2N_2O_5$ ([M-H]$^-$): 598.9823; found: 598.9833. Enantiomeric excess of **4g** was determined by chiral stationary phase HPLC analysis using a ChiralPak IC column (95:5 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 32.3 min, major enantiomer: t_R = 85.3 min.

(1*R*,2*R*,3*S*,4*S*)-3-Nitro-5-[(*R*)-2-nitro-1-(thiophen-2-yl)ethyl]-2-(thiophen-2-yl)-1,2,3,4-tetrahydro-[1,1'-biphenyl]-4-ol (4h)



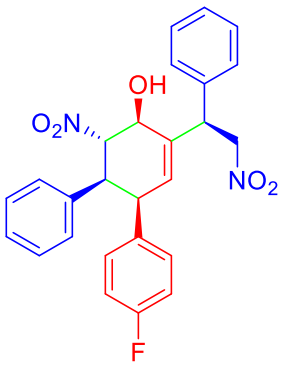
The crude material was purified by flash column chromatography using hexanes/EtOAc (90:10) as an eluent to get **4h** as a white solid, 62.1 mg, 68% yield, m.p. 140-142 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.30-7.28 (m, 1H), 7.25-7.17 (m, 3H), 7.08-6.98 (m, 3H), 6.75 (d, J = 6.7 Hz, 2H), 6.69-6.68 (m, 1H), 6.10 (d, J = 3.5 Hz, 1H), 6.05 (d, J = 4.6 Hz, 1H), 5.19 (dd, J_1 = 12.2 Hz, J_2 = 9.1 Hz, 1H), 5.05 (dd, J_1 = 12.6 Hz, J_2 = 8.5 Hz, 1H), 4.96 (dd, J_1 = 12.2 Hz, J_2 = 6.9 Hz, 1H), 4.92-4.89 (m, 2H), 4.08 (dd, J_1 = 12.7 Hz, J_2 = 6.1 Hz, 1H), 3.86 (t, J = 5.4 Hz, 1H), 2.74 (d, J = 7.3 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 139.9, 138.1, 136.6, 136.6, 130.9, 130.1, 129.9, 128.7, 128.6, 128.2, 127.8, 127.0, 127.0, 126.3, 126.1, 125.9, 124.9, 91.5, 79.5, 72.8, 48.9, 44.3, 42.9. ν_{max} (neat, cm^{-1}): 3581, 2918, 2849, 1550, 1490, 1430, 1379, 1330, 1246, 1201, 1049, 967. HRMS (ESI) *m/z* calcd for $C_{22}H_{19}N_2O_5S_2$ ([M-H]$^-$): 455.0741; found: 455.0750. Enantiomeric excess of **4h** was determined by chiral stationary phase HPLC analysis using a ChiralPak IC column (90:10 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 19.4 min, major enantiomer: t_R = 46.2 min.

(1'*R*,2'*S*,3'*S*,4'*S*)-4-Methoxy-3'-nitro-5'-[(*R*)-2-nitro-1-phenylethyl]-1',2',3',4'-tetrahydro-[1,1':2',1''-terphenyl]-4'-ol (4i)



The crude material was purified by flash column chromatography using hexanes/EtOAc (90:10) as an eluent to get **4i** as a white solid, 64.5 mg, 68% yield, m.p. 120-122 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.48-7.33 (m, 5H), 7.15-7.06 (m, 3H), 6.68 (d, J = 8.8 Hz, 2H), 6.59 (br, 2H), 6.50 (d, J = 8.2 Hz, 2H), 6.01 (d, J = 3.2 Hz, 1H), 5.25 (dd, J_1 = 12.2 Hz, J_2 = 9.8 Hz, 1H), 5.08 (dd, J_1 = 12.0 Hz, J_2 = 8.5 Hz, 1H), 4.94 (dd, J_1 = 12.2 Hz, J_2 = 6.8 Hz, 1H), 4.79-4.76 (m, 1H), 4.61 (dd, J_1 = 9.7 Hz, J_2 = 6.9 Hz, 1H), 3.76 (s, 3H), 3.73-3.70 (m, 2H), 2.62 (d, J = 7.5 Hz, 1H).^{13}C NMR (126 MHz, $CDCl_3$) δ: 159.4, 137.3, 136.6, 136.4, 131.3, 131.3, 129.6, 128.5, 128.5, 128.4, 128.0, 127.9, 113.8, 90.7, 79.1, 77.6, 73.4, 55.6, 49.1, 47.9. ν_{max} (neat, cm^{-1}): 3501, 2920, 2850, 1606, 1547, 1508, 1495, 1454, 1374, 1333, 1247, 1176, 1112, 1030. HRMS (ESI) *m/z* calcd for $C_{27}H_{25}N_2O_6$ ([M-H]$^-$): 473.1718; found: 473.1720. Enantiomeric excess of **4i** was determined by chiral stationary phase HPLC analysis using a ChiralPak IC column (90:10 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 23.4 min, major enantiomer: t_R = 71.7 min.

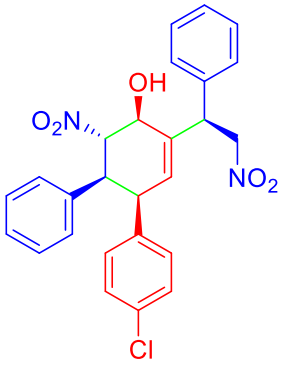
(1'*R*,2'*S*,3'*S*,4'*S*)-4-Fluoro-3'-nitro-5'-[(*R*)-2-nitro-1-phenylethyl]-1',2',3',4'-tetrahydro-[1,1':2',1''-terphenyl]-4'-ol (4j)



The crude material was purified by flash column chromatography using hexanes/EtOAc (95:5) as an eluent to get **4j** as a white solid, 58.3 mg, 63% yield, m.p. 130-132 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.37-7.32 (m, 6H), 7.14 (d, J = 7.1 Hz, 1H), 7.09 (t, J = 7.3 Hz, 2H), 6.83 (t, J = 8.5 Hz, 2H), 6.67-6.57 (m, 2H), 6.55 (dd, J_1 = 8.3 Hz, J_2 = 5.3 Hz, 2H), 5.99 (q, J = 2.0 Hz, 1H), 5.26 (dd, J_1 = 12.2 Hz, J_2 = 9.8 Hz, 1H), 5.06 (dd, J_1 = 12.1 Hz, J_2 = 8.5 Hz, 1H), 4.93 (dd, J_1 = 12.2 Hz, J_2 = 6.7 Hz, 1H), 4.77 (t, J = 7.8 Hz, 1H), 4.62 (dd, J_1 = 9.7 Hz, J_2 = 6.7 Hz, 1H), 3.78-3.74 (m, 2H), 2.79 (d, J = 7.4 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ: 162.5 (d, $J_{C\text{-}F}$ = 248.2 Hz), 137.3, 137.2, 136.1, 132.3 (d, $J_{C\text{-}F}$ = 2.5 Hz), 131.6 (d, $J_{C\text{-}F}$ = 7.5 Hz), 130.7, 129.6, 128.6, 128.5, 128.3, 128.3, 128.1, 128.0, 115.3 (d, $J_{C\text{-}F}$

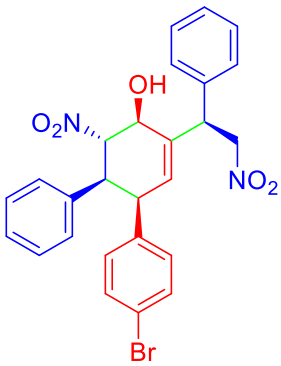
= 21.3 Hz), 79.0, 73.2, 49.0, 47.9, 47.6. ν_{max} (neat, cm^{-1}): 3509, 2918, 1602, 1546, 1506, 1454, 1374, 1331, 1275, 1222, 1158, 1059. HRMS (ESI) *m/z* calcd for $C_{26}H_{22}FN_2O_5$ ($[M-H]^-$): 461.1518; found: 461.1519. Enantiomeric excess of **4j** was determined by chiral stationary phase HPLC analysis using a ChiralPak IC column (90:10 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 15.0 min, major enantiomer: t_R = 44.0 min.

(1'*R*,2'*S*,3'*S*,4'*S*)-4-Chloro-3'-nitro-5'-[(*R*)-2-nitro-1-phenylethyl]-1',2',3',4'-tetrahydro-[1,1':2',1''-terphenyl]-4'-ol (4k)



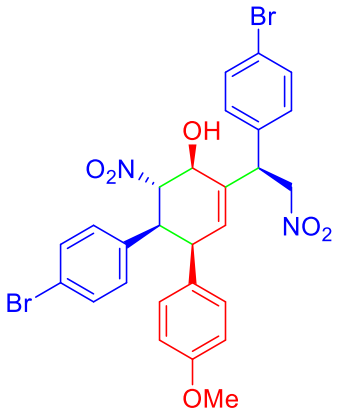
The crude material was purified by flash column chromatography using hexanes/EtOAc (95:5) as an eluent to get **4k** as a pale yellow solid, 58.3 mg, 61% yield, m.p. 135-137 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.38-7.32 (m, 6H), 7.15-7.10 (m, 6H), 6.60 (br, 2H), 6.51 (d, *J* = 8.1 Hz, 2H), 5.96 (d, *J* = 2.9 Hz, 1H), 5.26 (dd, J_1 = 12.2 Hz, J_2 = 9.8 Hz, 1H), 5.05 (dd, J_1 = 12.3 Hz, J_2 = 8.4 Hz, 1H), 4.92 (dd, J_1 = 12.2 Hz, J_2 = 6.7 Hz, 1H), 4.77 (t, *J* = 7.6 Hz, 1H), 4.61 (dd, J_1 = 9.9 Hz, J_2 = 6.7 Hz, 1H), 3.80-3.74 (m, 2H), 2.80 (d, *J* = 7.4 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 137.6, 137.1, 136.0, 135.1, 134.0, 131.4, 130.4, 129.6, 128.7, 128.6, 128.5, 128.3, 128.3, 128.1, 128.0, 90.4, 79.0, 73.2, 48.9, 48.0, 47.5. ν_{max} (neat, cm^{-1}):3523, 3029, 1547, 1489, 1454, 1374, 1274, 1091, 1013. HRMS (ESI) *m/z* calcd for $C_{26}H_{23}ClN_2O_5$ ($[M+Na]^+$): 501.1188; found: 501.1172. Enantiomeric excess of **4k** was determined by chiral stationary phase HPLC analysis using a ChiralPak IC column (90:10 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 15.7 min, major enantiomer: t_R = 49.3 min.

(1'*R*,2'*S*,3'*S*,4'*S*)-4-Bromo-3'-nitro-5'-[(*R*)-2-nitro-1-phenylethyl]-1',2',3',4'-tetrahydro-[1,1':2',1''-terphenyl]-4'-ol (4l)



The crude material was purified by flash column chromatography using hexanes/EtOAc (95:5) as an eluent to get **4l** as a pale yellow solid, 64.9 mg, 62% yield, m.p. 140-142 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.41-7.30 (m, 7H), 7.27-7.25 (m, 4H), 7.18-7.08 (m, 4H), 6.60 (br, 2H), 6.45 (d, J = 8.0 Hz, 2H), 5.96 (d, J = 3.1 Hz, 1H), 5.26 (dd, J_1 = 12.2 Hz, J_2 = 9.9 Hz, 1H), 5.05 (dd, J_1 = 12.1 Hz, J_2 = 8.4 Hz, 1H), 4.92 (dd, J_1 = 12.2 Hz, J_2 = 6.7 Hz, 1H), 4.77 (t, J = 7.9 Hz, 1H), 4.61 (dd, J_1 = 9.9 Hz, J_2 = 6.7 Hz, 1H), 3.79-3.73 (m, 2H), 2.75 (d, J = 7.4 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 137.62, 137.10, 135.96, 135.65, 131.78, 131.54, 130.35, 129.60, 128.67, 128.49, 128.32, 128.29, 128.15, 127.98, 122.14, 90.34, 78.96, 73.21, 48.91, 48.08, 47.44. ν_{max} (neat, cm^{-1}): 2522, 2360, 2340, 2159, 2016, 1868, 1699, 1683, 1652, 1558, 1540, 1456, 1418, 1072. HRMS (ESI) *m/z* calcd for $C_{26}H_{23}BrN_2O_5Na$ ($[M+Na]^+$): 545.0683; found: 545.0663. Enantiomeric excess of **4l** was determined by chiral stationary phase HPLC analysis using a ChiralPak IC column (90:10 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 16.7 min, major enantiomer: t_R =50.9 min.

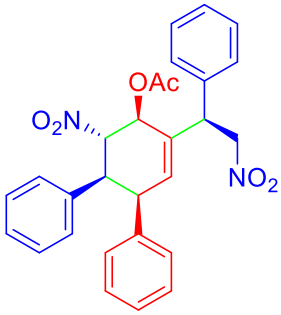
(1'*R*,2'*S*,3'*S*,4'*S*)-4''-Bromo-5'-[(*R*)-1-(4-bromophenyl)-2-nitroethyl]-4-methoxy-3'-nitro-1',2',3',4'-tetrahydro-[1,1':2',1''-terphenyl]-4'-ol (4m)



The crude material was purified by flash column chromatography using hexanes/EtOAc (95:5) as an eluent to get **4m** as a white solid, 82.2 mg, 65% yield, m.p. 135-137 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.51 (d, J = 8.4 Hz, 2H), 7.25-7.21 (m, 6H), 6.71 (d, J = 8.8 Hz, 2H), 6.50 (d, J = 8.2 Hz, 2H), 6.45 (br, 2H), 5.97 (d, J = 3.9 Hz, 1H), 5.18 (dd, J_1 = 12.4 Hz, J_2 = 9.5 Hz, 1H), 5.02 (dd, J_1 = 12.5 Hz, J_2 = 8.4 Hz, 1H), 4.90 (dd, J_1 = 12.4 Hz, J_2 = 7.1 Hz, 1H), 4.74 (t, J = 6.9 Hz, 1H), 4.57 (dd, J_1 = 9.4 Hz, J_2 = 7.1 Hz, 1H), 3.78 (s, 3H), 3.72-3.65 (m, 2H), 2.75 (d, J = 7.2 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 159.6, 136.3, 136.3, 135.3, 132.7, 131.7, 131.3, 131.2, 130.2, 129.8, 127.8, 122.1, 114.1, 90.5, 78.7, 73.1, 55.6, 48.3, 47.6, 47.4. ν_{max} (neat, cm^{-1}): 3420, 2918, 2849, 1703, 1608, 1548, 1508, 1488, 1462, 1373, 1248, 1176, 1112, 1073. HRMS (ESI) *m/z* calcd for $C_{27}H_{23}Br_2N_2O_6$ ($[M-H]^-$): 628.9928; found: 628.9925.

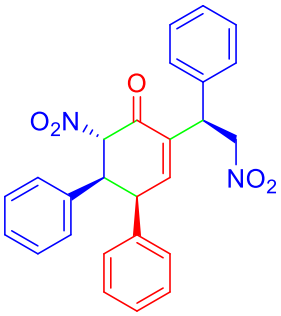
Enantiomeric excess of **4m** was determined by chiral stationary phase HPLC analysis using a ChiralPak IC column (90:10 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 16.0 min, major enantiomer: t_R = 55.5 min.

(1'*R*,2'*S*,3'*S*,4'*S*)-3'-Nitro-5'-[(*R*)-2-nitro-1-phenylethyl]-1',2',3',4'-tetrahydro-[1,1':2',1''-terphenyl]-4'-yl acetate (6)



The crude material was purified by flash column chromatography using hexanes/EtOAc (93:7) as an eluent to get **6** as a white solid, 38.7 mg, 80% yield, m.p. 181-183 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (t, *J* = 7.4 Hz, 2H), 7.36 – 7.31 (m, 1H), 7.29 (d, *J* = 7.1 Hz, 2H), 7.23 (d, *J* = 7.3 Hz, 1H), 7.18 (t, *J* = 10.0 Hz, 2H), 7.13 (t, *J* = 7.4 Hz, 1H), 7.06 (t, *J* = 7.5 Hz, 2H), 6.68 (d, *J* = 7.1 Hz, 2H), 6.59 (br, 2H), 6.34 (d, *J* = 8.4 Hz, 1H), 6.08 (d, *J* = 5.7 Hz, 1H), 5.23 (dd, J_1 = 13.0 Hz, J_2 = 8.4 Hz, 1H), 4.87 (dd, J_1 = 13.4 Hz, J_2 = 8.8 Hz, 1H), 4.68 (dd, J_1 = 13.4 Hz, J_2 = 7.6 Hz, 1H), 4.43 (t, *J* = 8.2 Hz, 1H), 3.94 (dd, J_1 = 13.0 Hz, J_2 = 5.8 Hz, 1H), 3.85 (t, *J* = 5.7 Hz, 1H), 1.84 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.4, 137.4, 136.5, 135.8, 134.3, 131.0, 130.3, 129.6, 128.6, 128.5, 128.4, 128.1, 128.0, 87.3, 78.5, 73.8, 48.4, 47.8, 46.0, 20.7. ν_{max} (neat, cm^{-1}): 3028, 1739, 1547, 1491, 1453, 1427, 1368, 1271, 1209, 1087, 1015. HRMS (ESI) m/z calcd for $C_{28}H_{30}N_3O_6$([M+NH_4]$^+$): 504.2129; found: 504.2132. Enantiomeric excess of **6** was determined by chiral stationary phase HPLC analysis using a ChiralPak IB column (90:10 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 22.2 min, major enantiomer: t_R = 55.3 min.

(1'*R*,2'*S*,3'*S*)-3'-Nitro-5'-((*R*)-2-nitro-1-phenylethyl)-2',3'-dihydro-[1,1':2',1''-terphenyl]-4'(1'*H*)-one (7)



The crude material was purified by flash column chromatography using hexanes/EtOAc (92:8) as an eluent to get **7** as a white solid, 37.6 mg, 85 % yield, m.p. 125-127 °C, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40-7.33 (m, 5H), 7.25 – 7.05 (m, 7H), 6.65 (d, *J* = 7.5 Hz, 2H), 6.56 (d, *J* = 7.6 Hz, 2H), 5.78 (d, *J* = 14.1 Hz, 1H), 5.17 (dd, J_1 = 12.7 Hz, J_2 = 8.9 Hz, 1H), 4.92 (t, *J* = 8.0 Hz, 1H), 4.84 (dd, J_1 = 12.7 Hz, J_2 = 7.1 Hz, 1H), 4.41 (dd, J_1 = 14.1 Hz, J_2 = 5.6 Hz, 1H), 4.11 (t, *J* = 5.8 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 187.4, 148.9, 137.0, 136.9, 135.6, 133.2, 130.0, 129.7, 129.0, 128.9, 128.8, 128.7, 128.5, 128.3, 127.9, 90.4, 76.9, 48.8, 48.5, 44.5. ν_{max} (neat, cm^{-1}): 1689, 1551, 1493, 1453, 1374, 1205, 1085, 1032, 918, 789, 766, 747. HRMS (ESI) m/z calcd for $C_{26}H_{26}N_3O_5$ ($[M+NH_4]^+$): 460.1867; found: 460.1871. Enantiomeric excess of **7** was determined by chiral stationary phase HPLC analysis using a ChiralPak IC column (70:30 hexanes/*i*-PrOH at 1.0 mL/min, λ = 220 nm), minor enantiomer: t_R = 25.6 min, major enantiomer: t_R = 37.3 min.

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12. a) Schönberger, M.; Trauner, D. *Angew. Chem., Int. Ed.* **2014**, *53*, 3264-3267. b) Reddy, B. V. S.; Sundar, C. S.; Reddy, M. R.; Reddy, C. S.; Sridhar B. *Synthesis* **2015**; *47*, 1117-1122.
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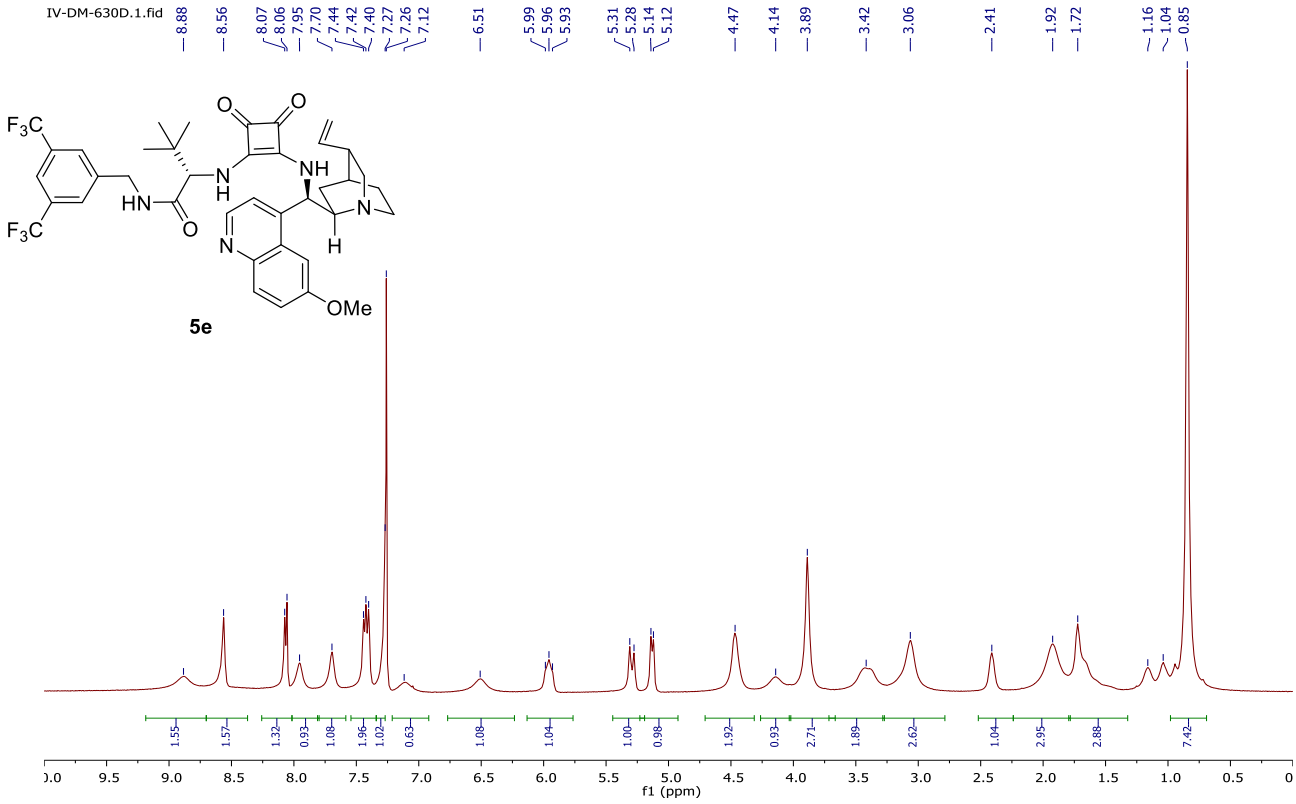
15. Wenzel, A. G.; Jacobsen, E. N. *J. Am. Chem. Soc.* **2002**, *124*, 12964−12965.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5.14
5.12
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4.14
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3.42
3.06
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1.92
1.72
1.16
1.04
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F3C
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O
N
H
NH
N
N
H
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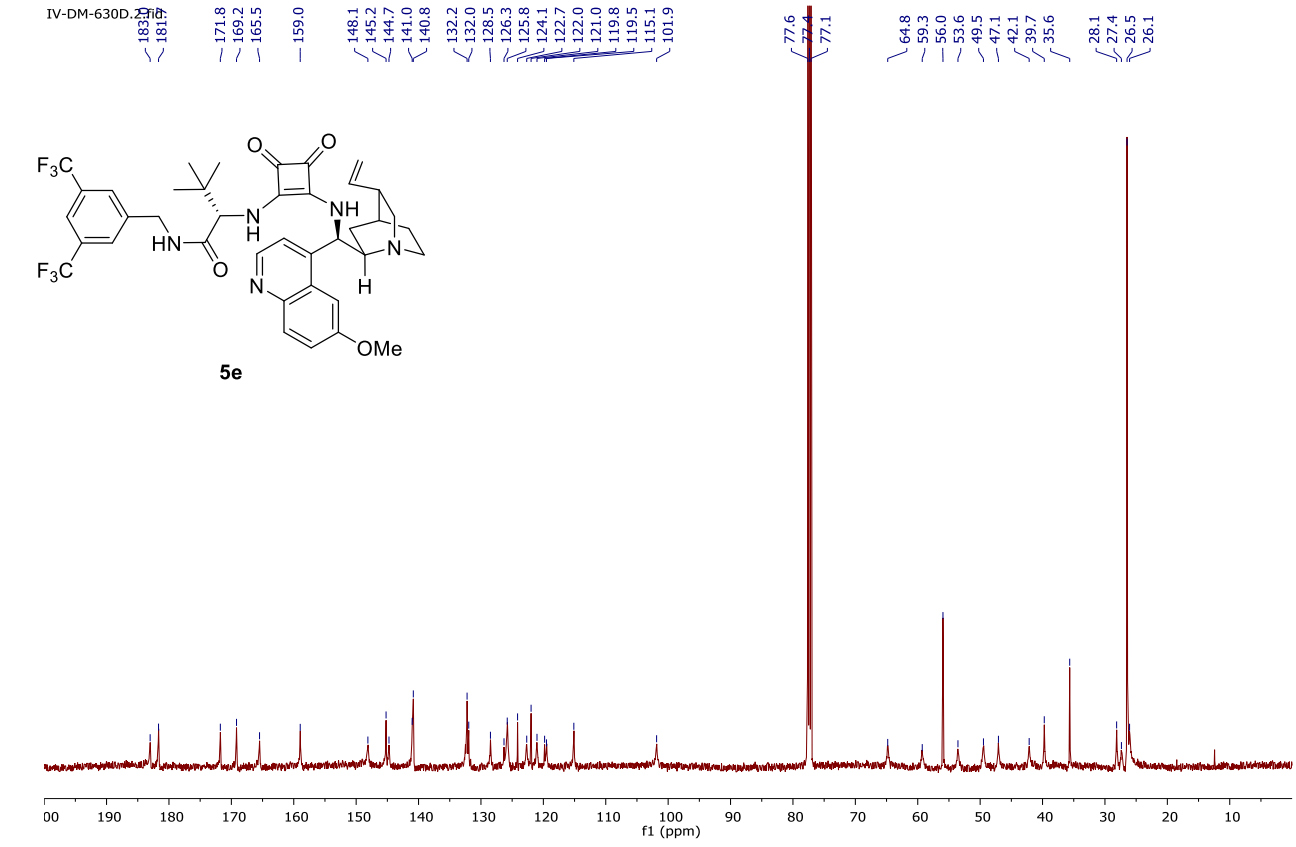
5e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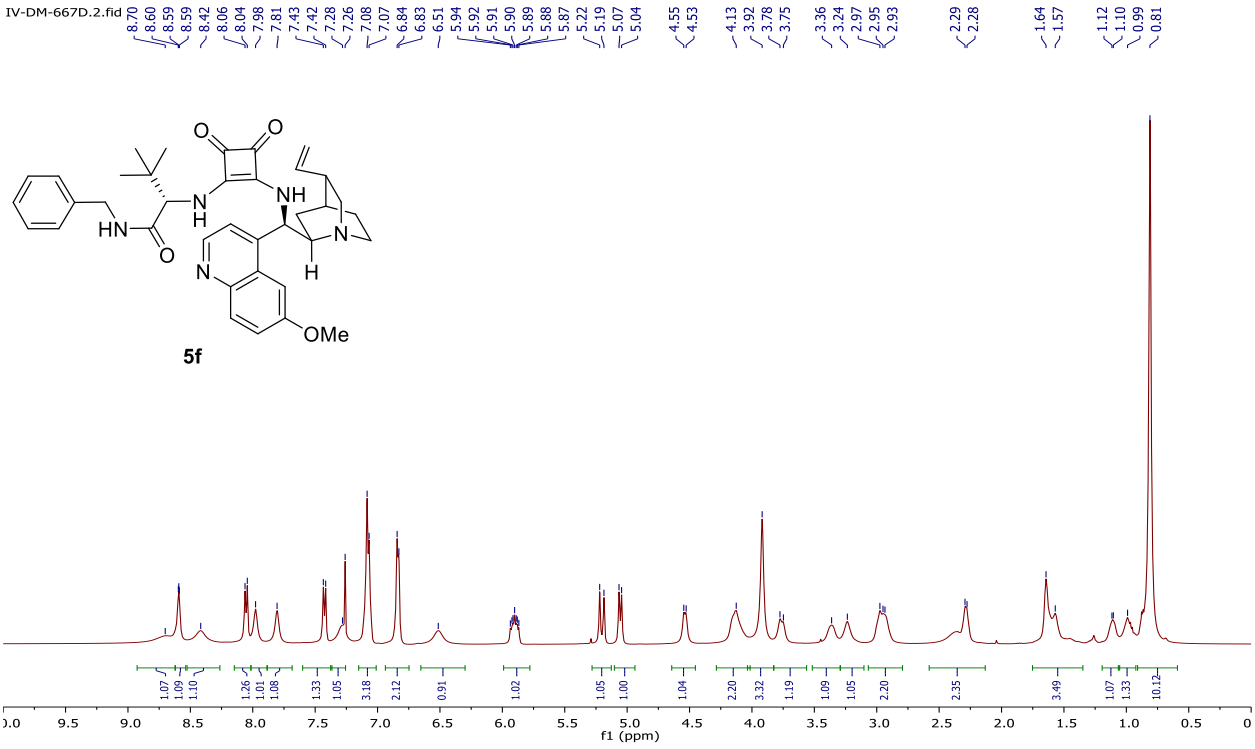
f1 (ppm)

5e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N
H
HN
O
N
H
N
OMe
5f
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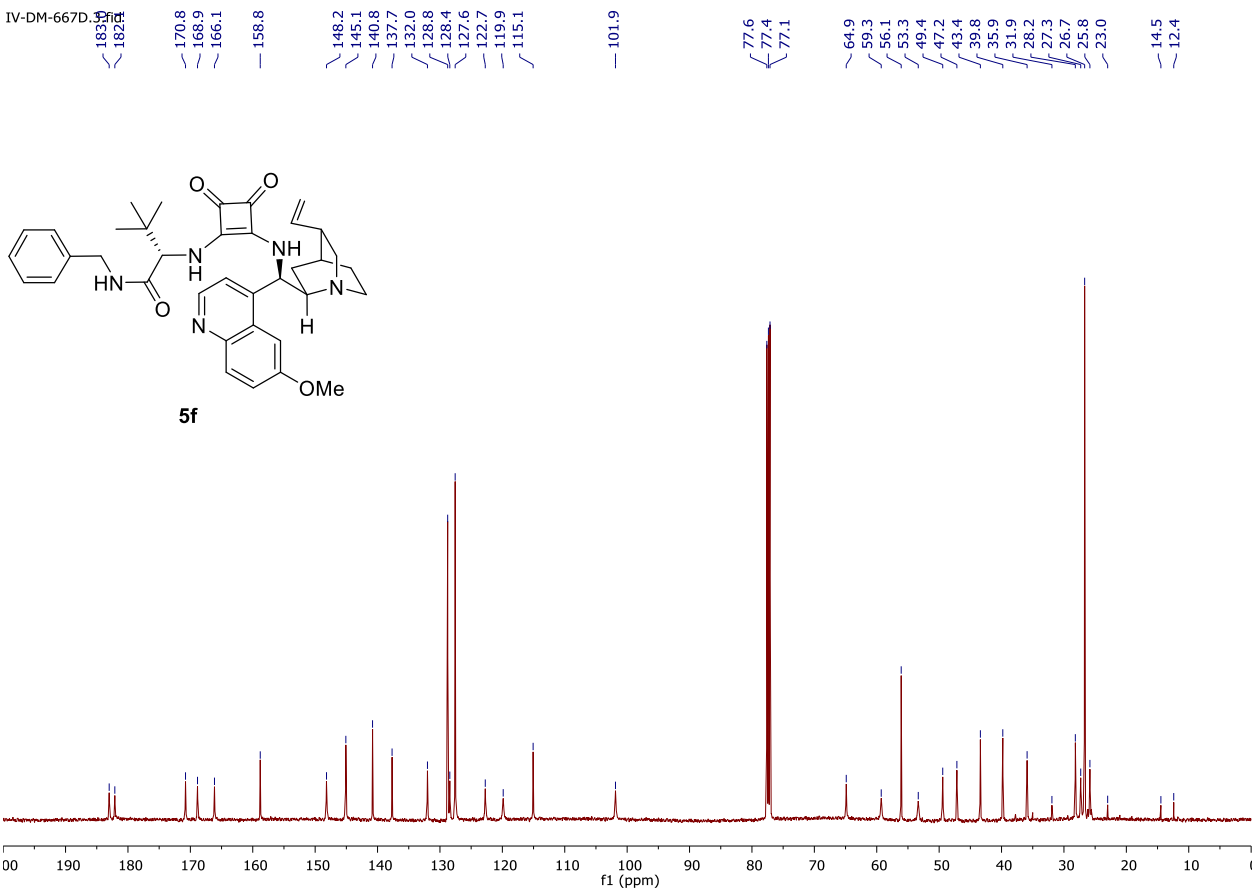
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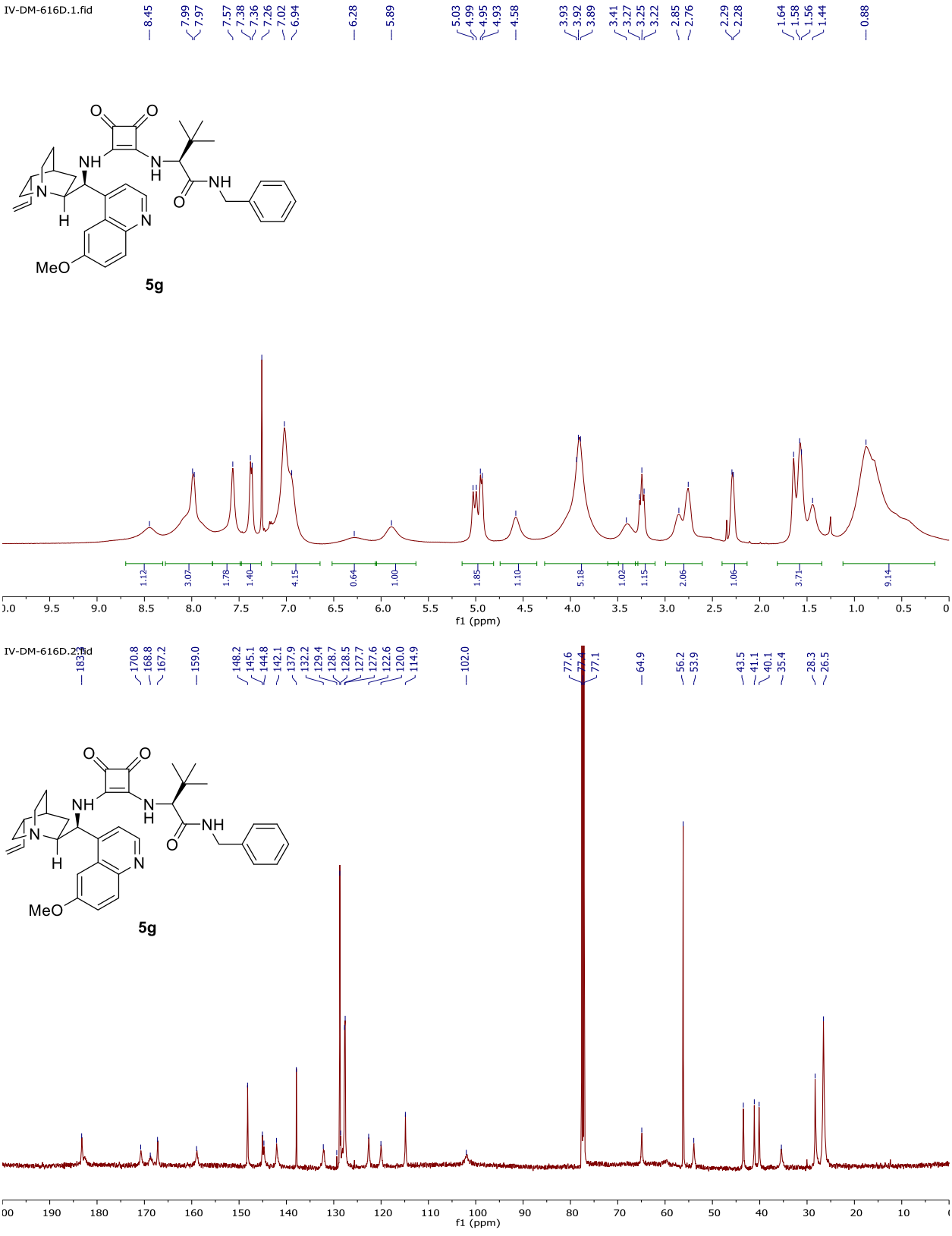
f1 (ppm)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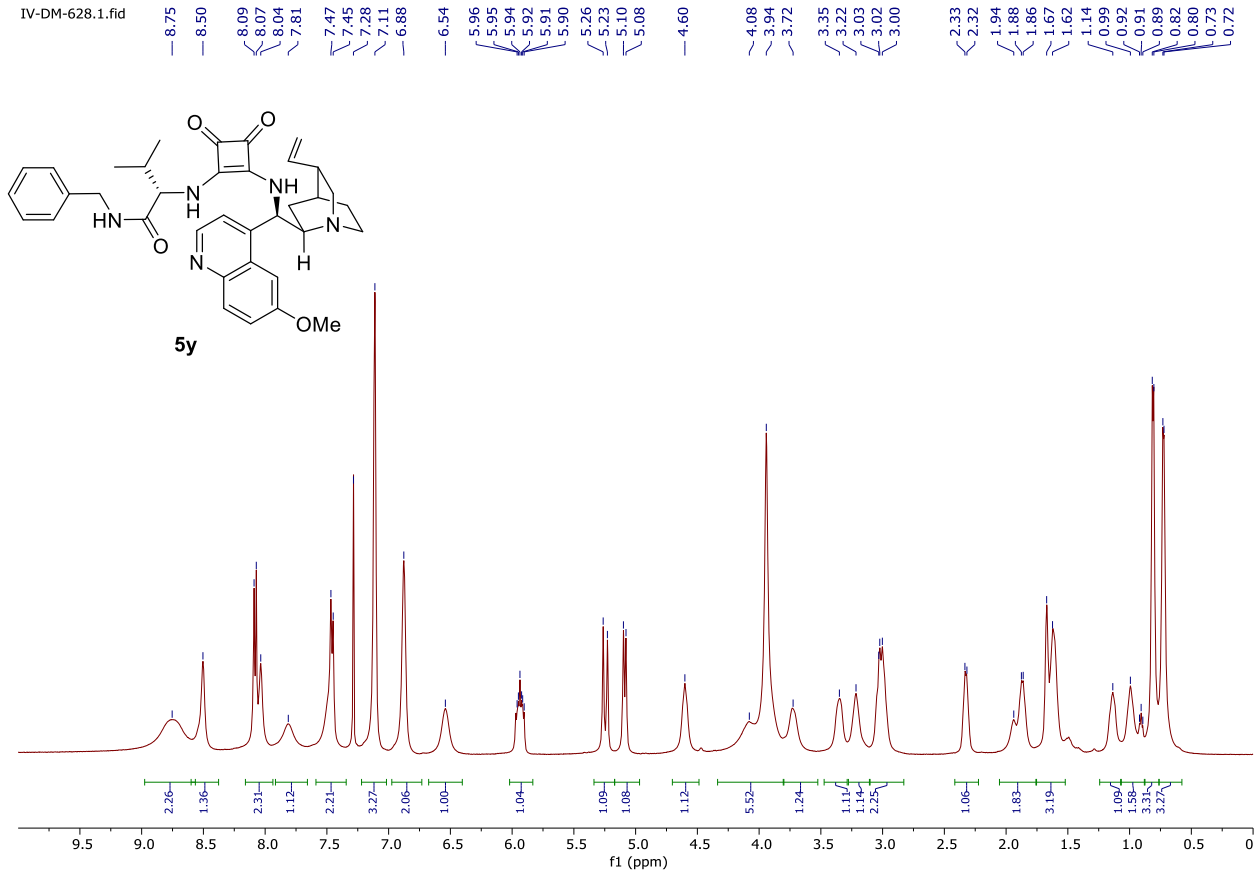
f1 (ppm)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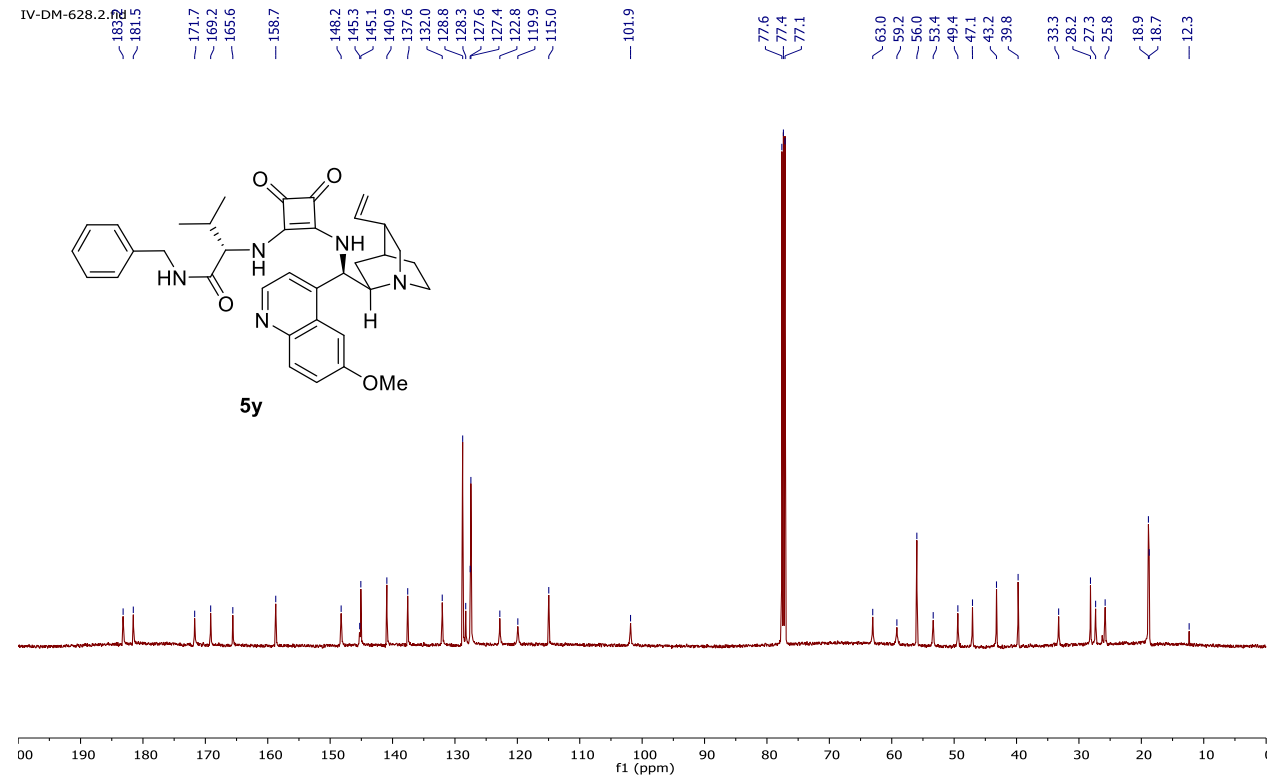
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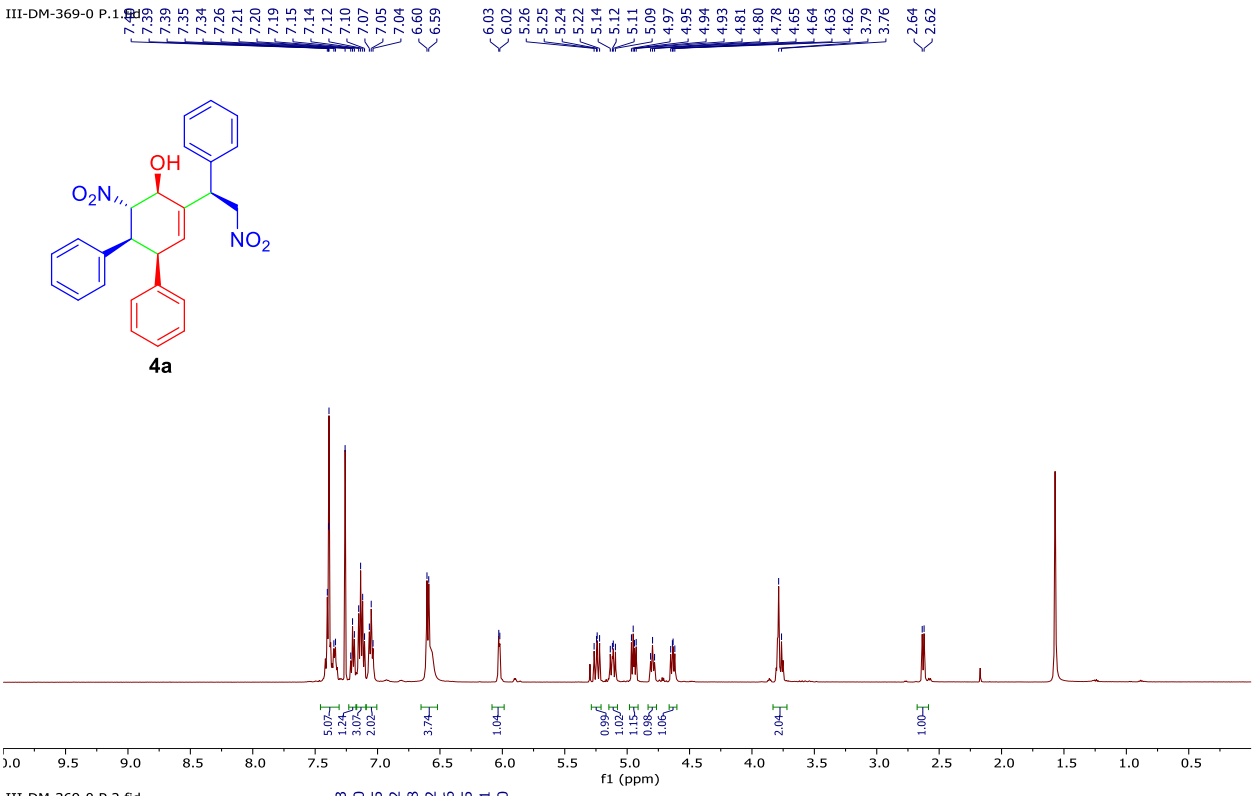
f1 (ppm)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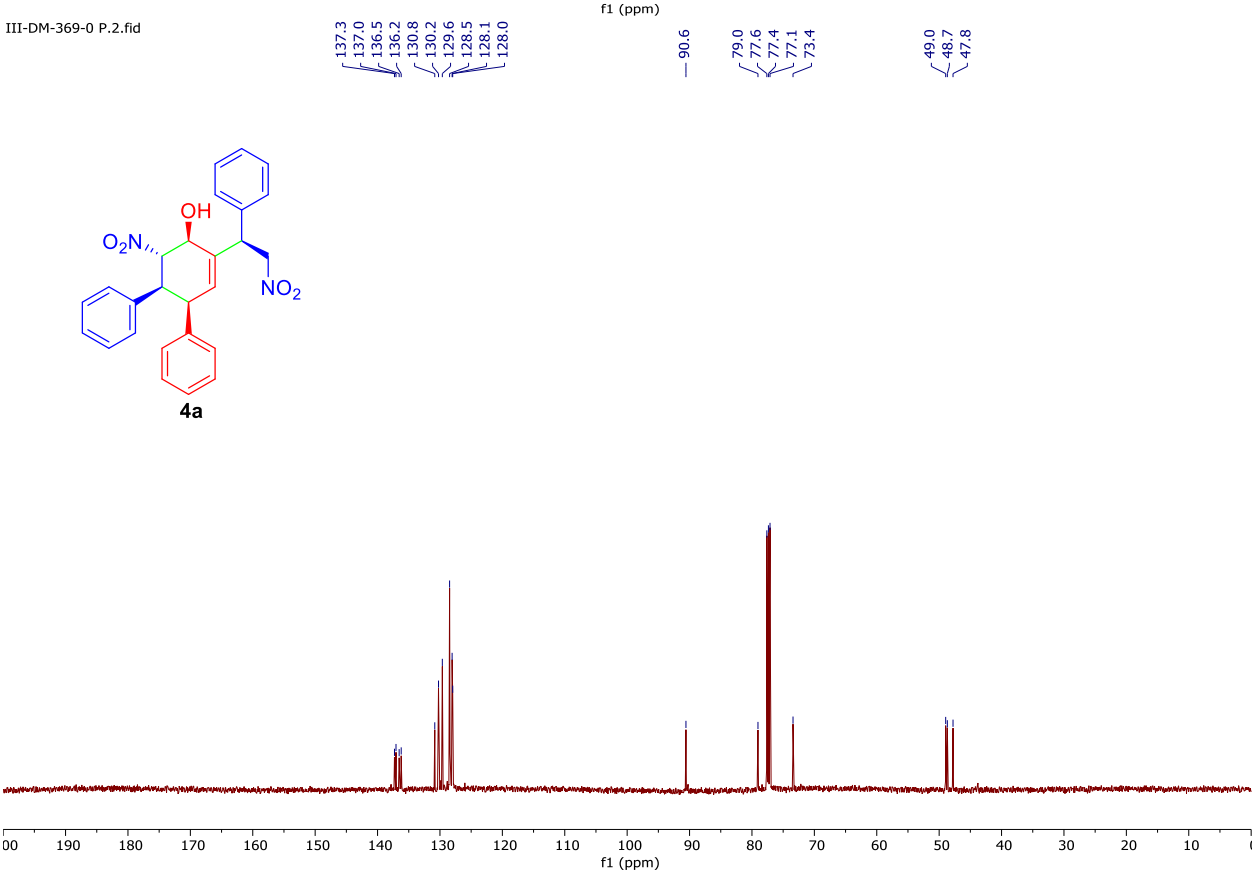
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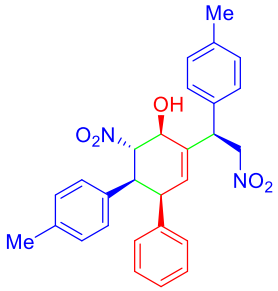

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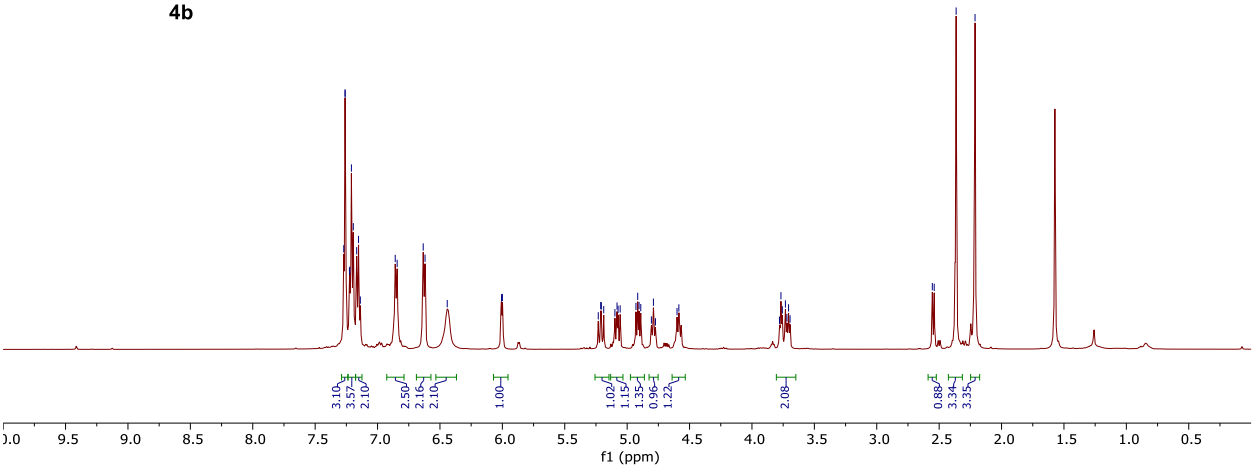

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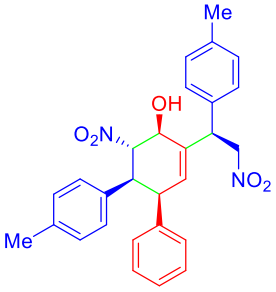
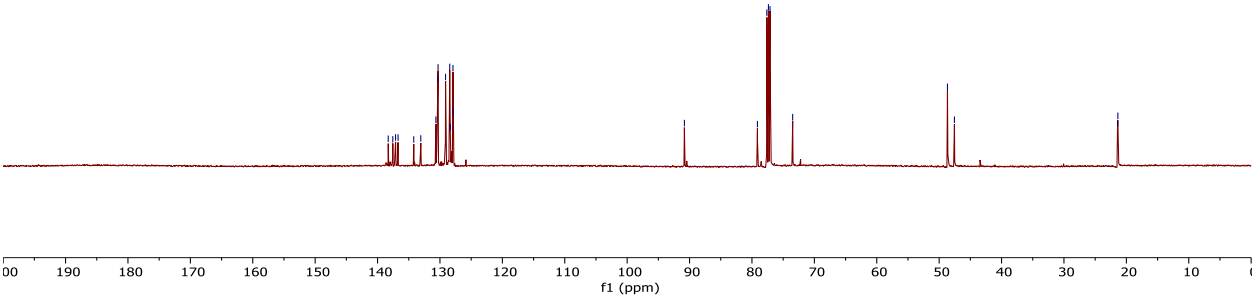
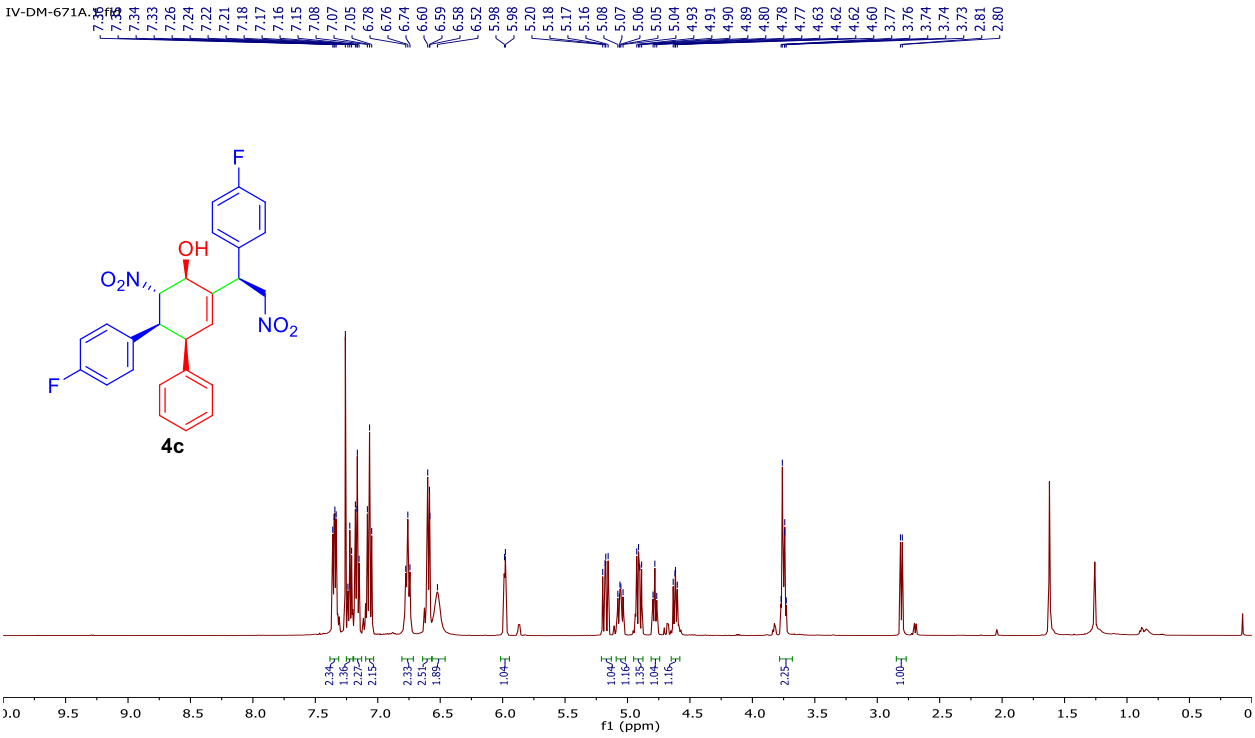
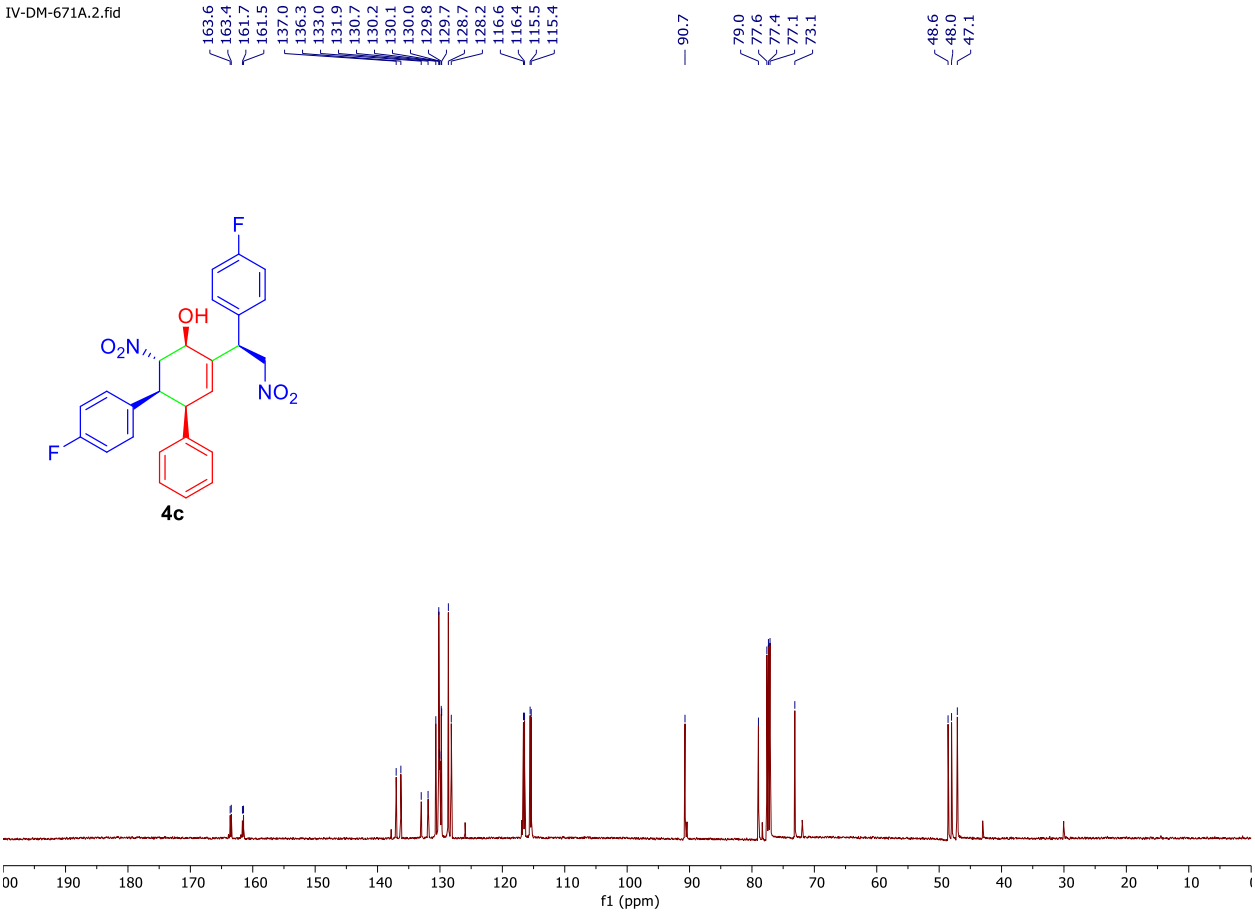


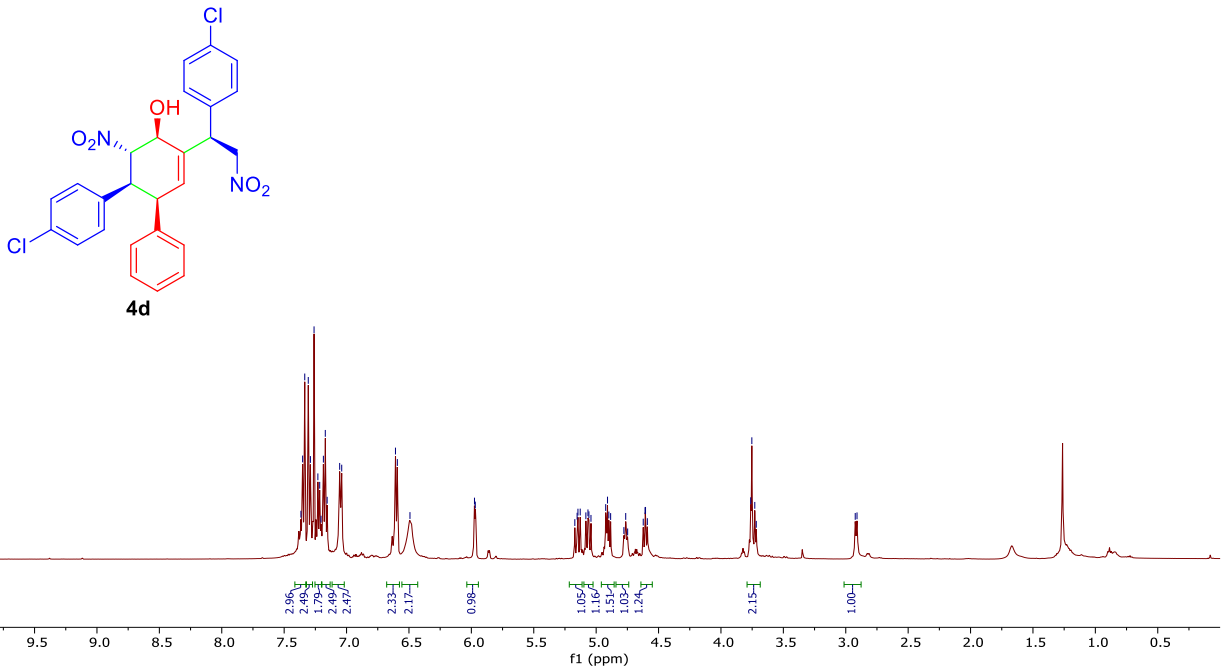
4b



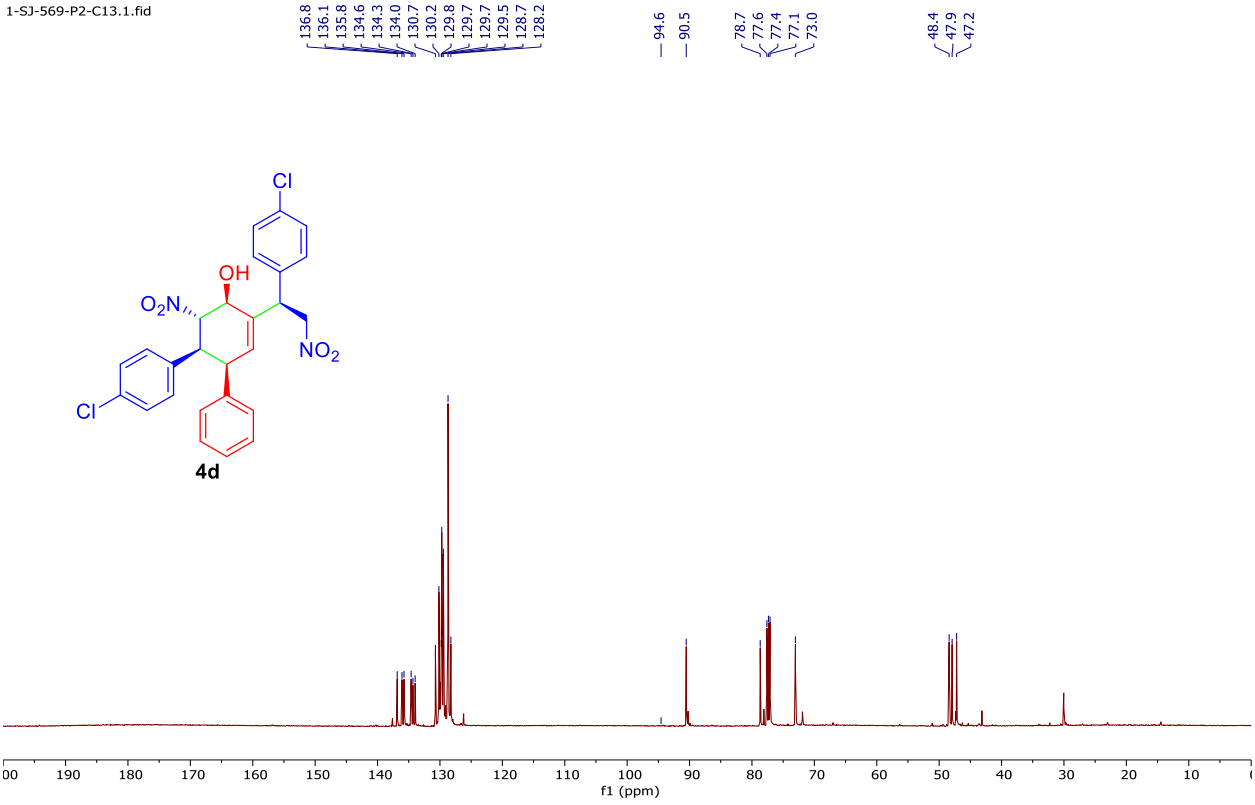

IV-DM-671A.fid
F
OH
O2N
NO2
F
4c
f1 (ppm)


IV-DM-671A.2.fid
F
OH
O2N
NO2
F
4c
f1 (ppm)

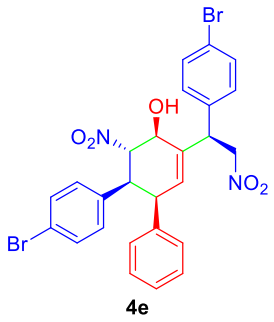
1-SJ-569-P2.1.fid



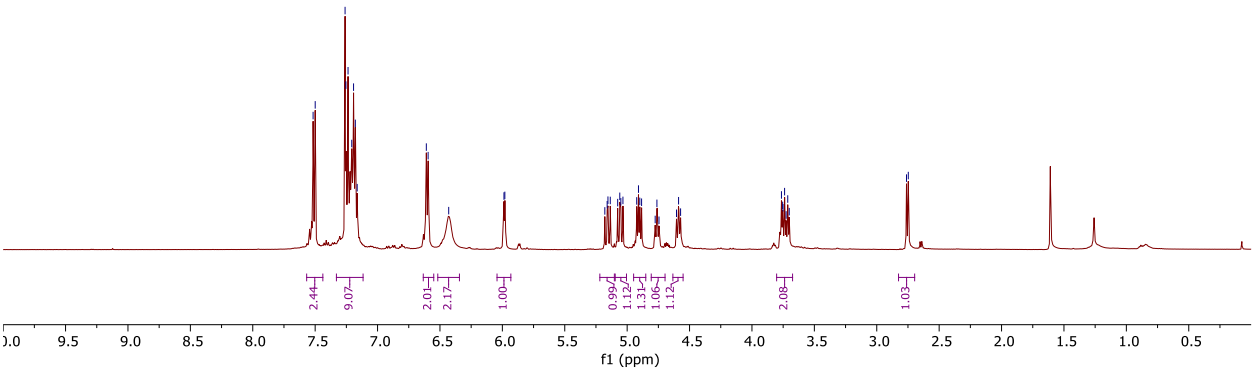
1-SJ-569-P2-C13.1.fid



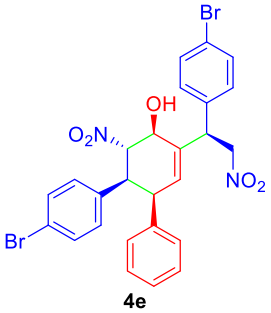
V-DM-714D2.1.fid



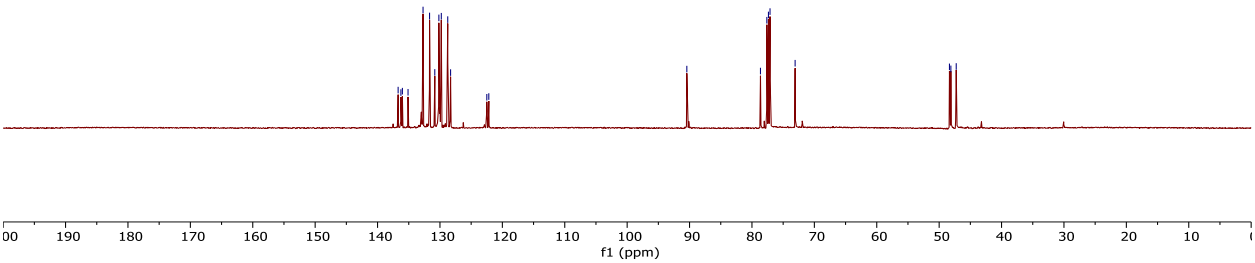
4e

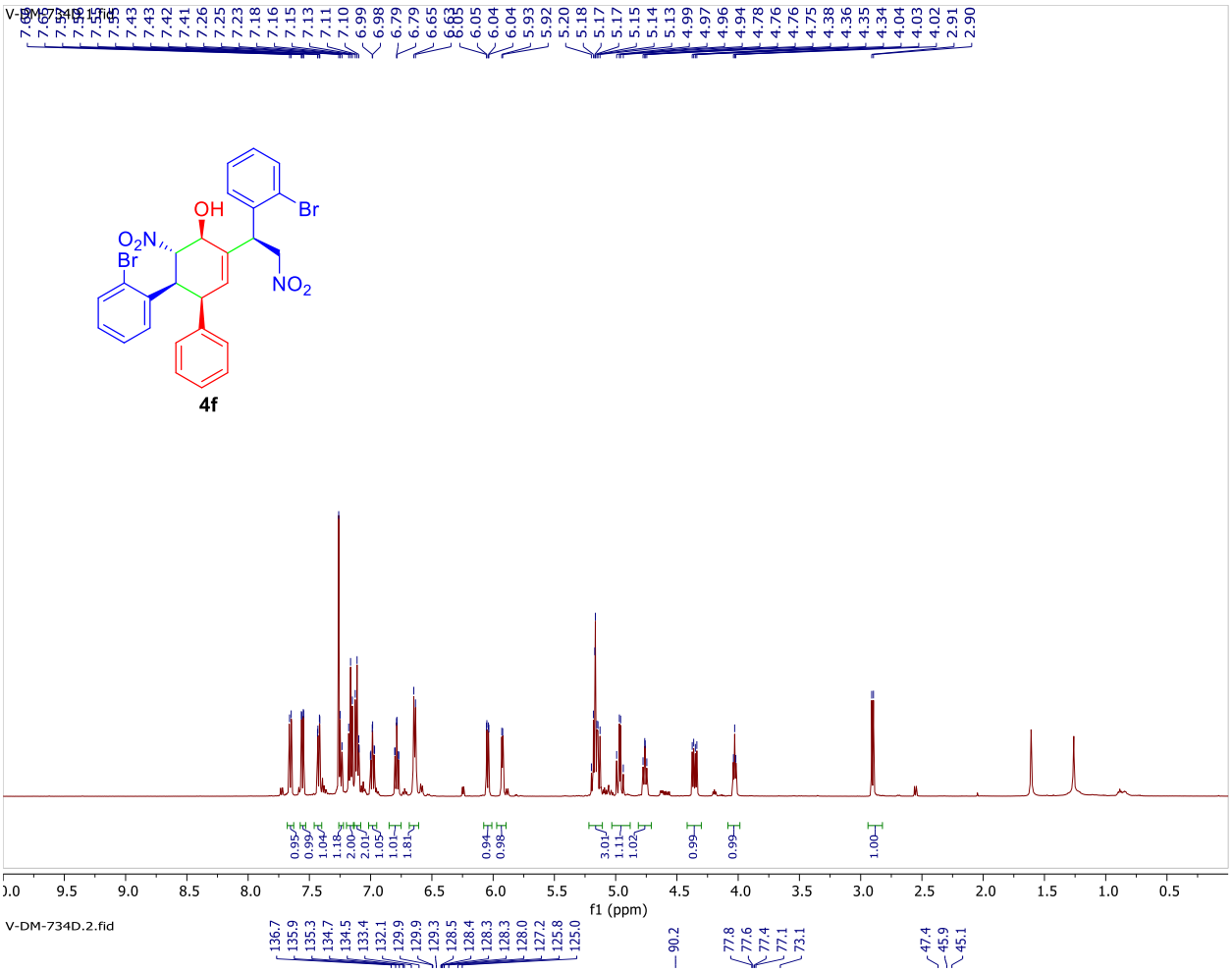


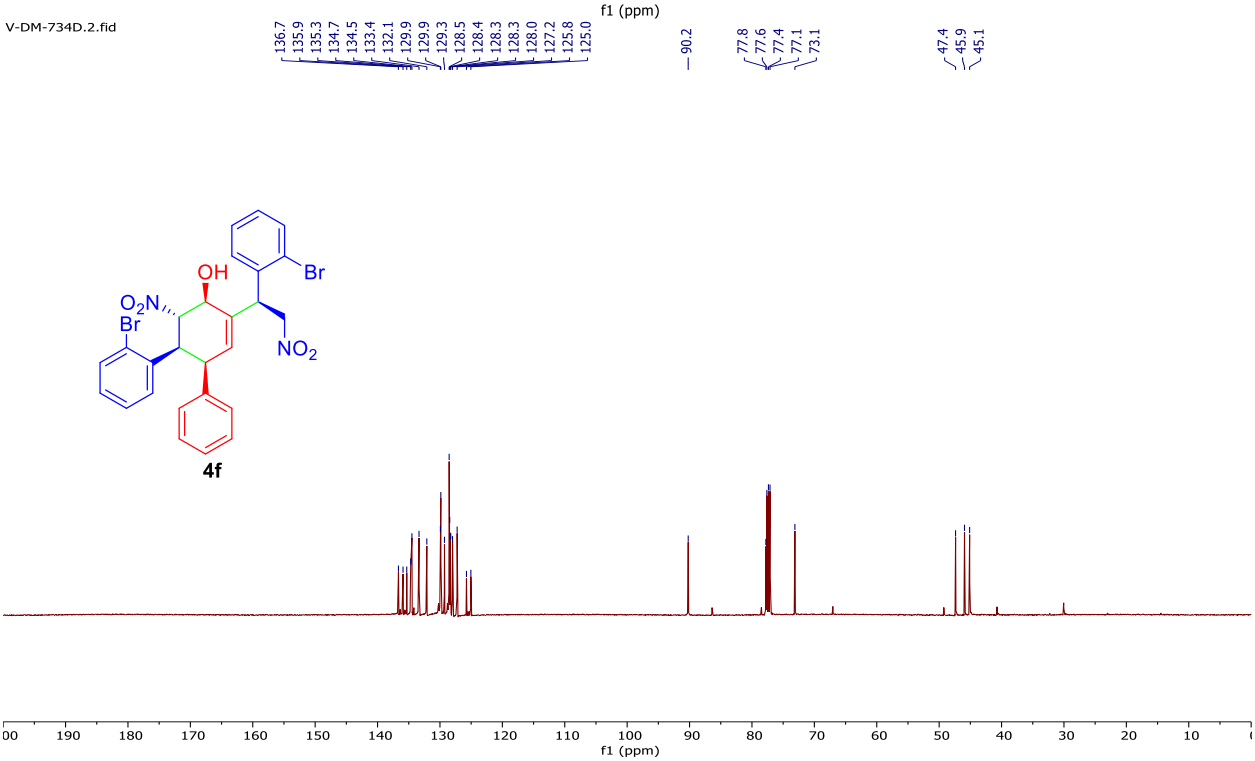
V-DM-714D2.2.fid

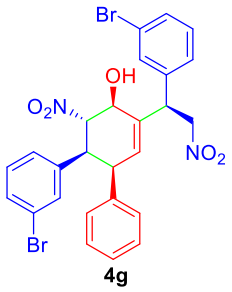


4e

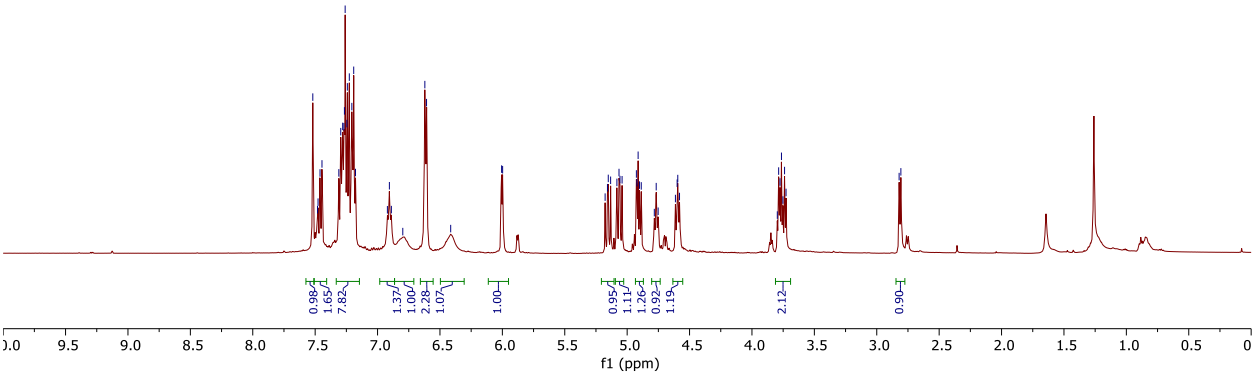



V-DM-734B.1.fid
OH
Br
O_2N
Br
NO_2
4f
f1 (ppm)

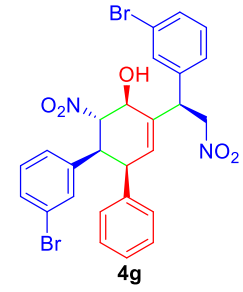

V-DM-734D.2.fid
OH
Br
O_2N
Br
NO_2
4f
f1 (ppm)



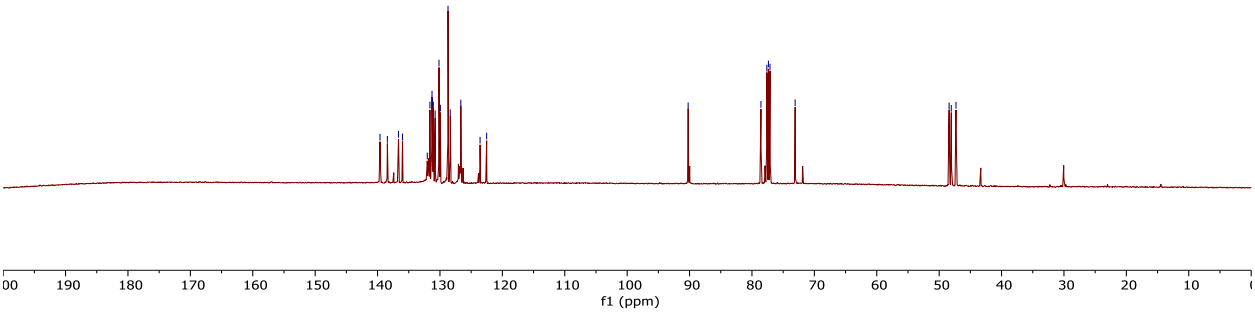
4g

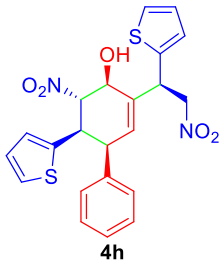


IV-DM-673D.2.fid

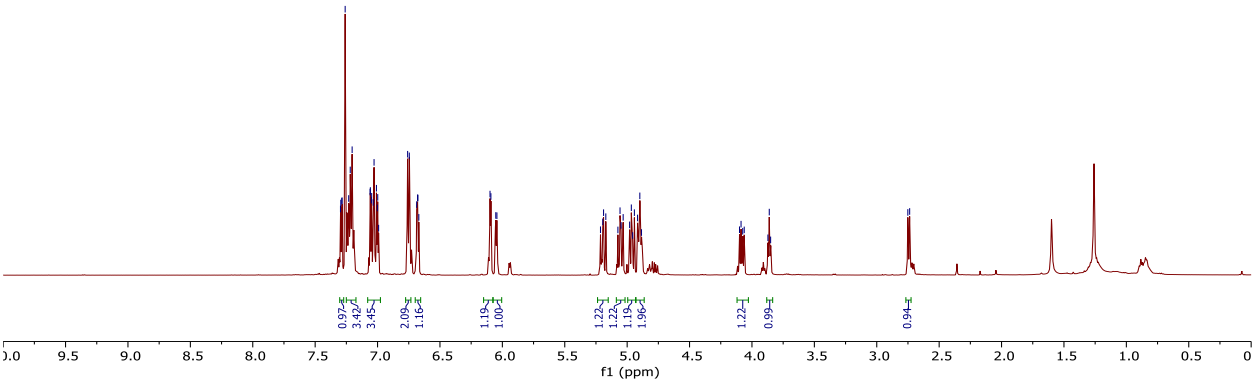


4g

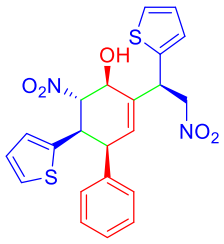




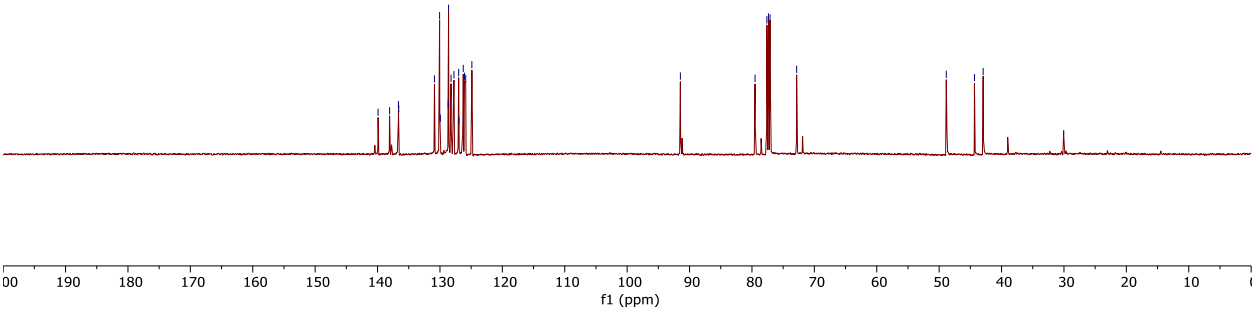
4h

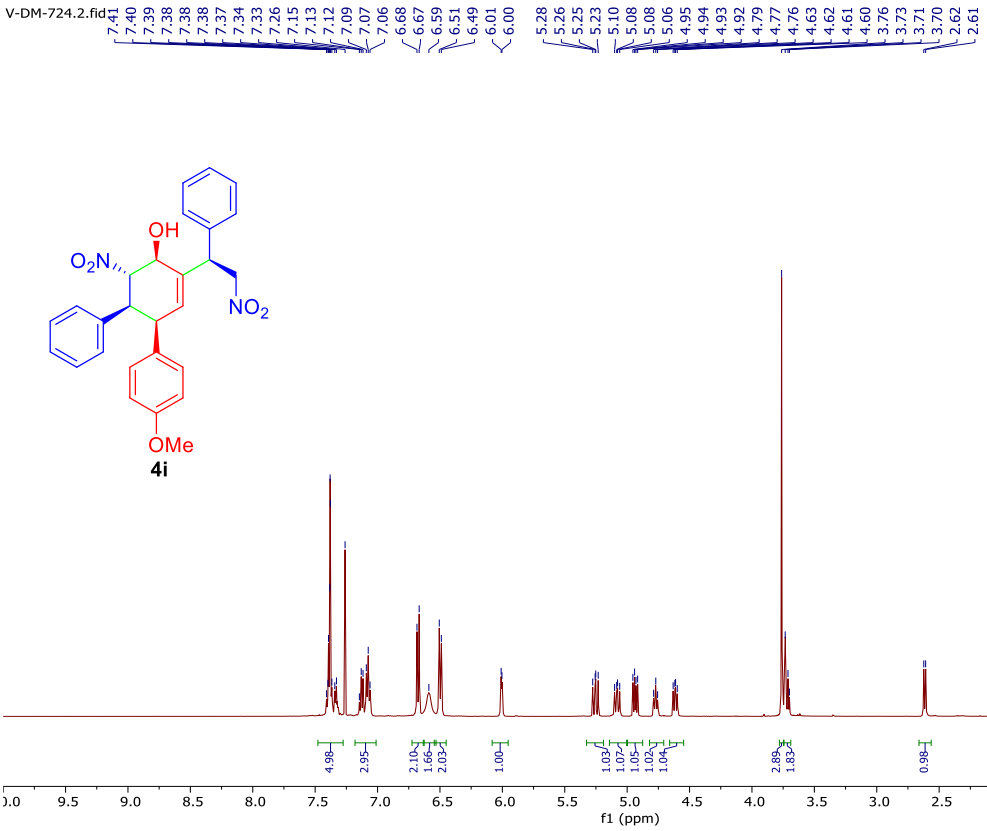


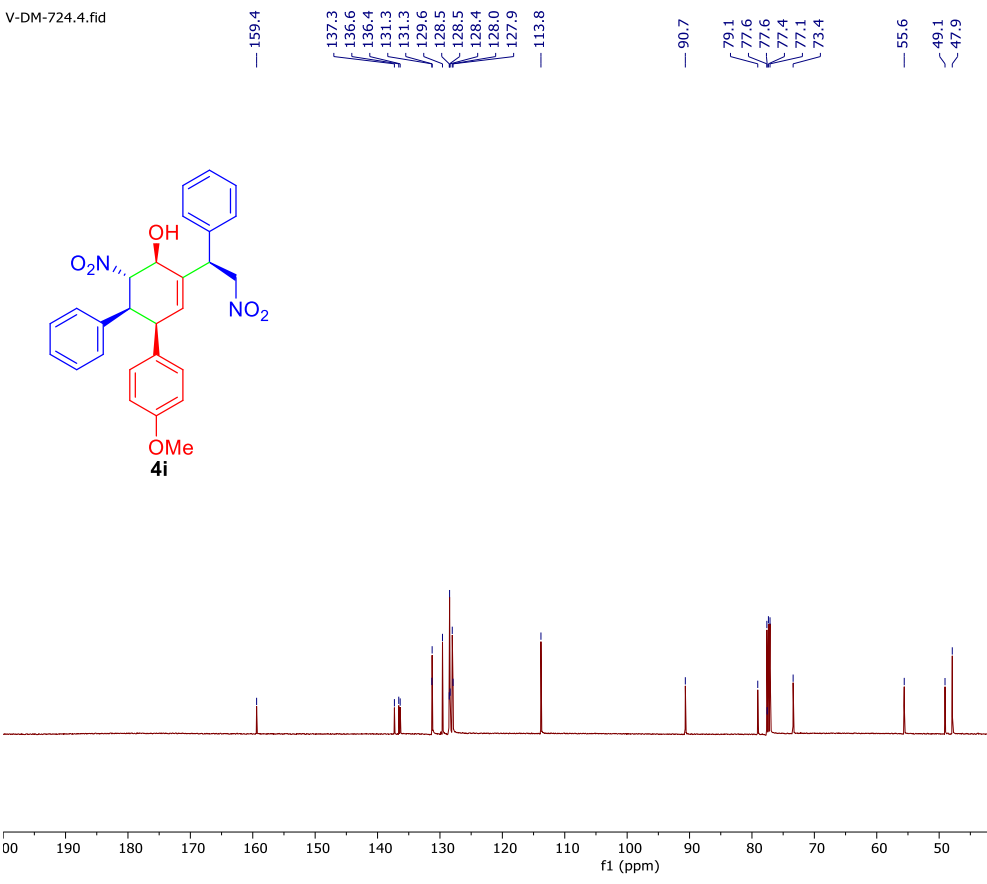
IV-DM-677A.5.fid



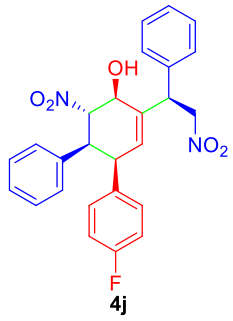
4h



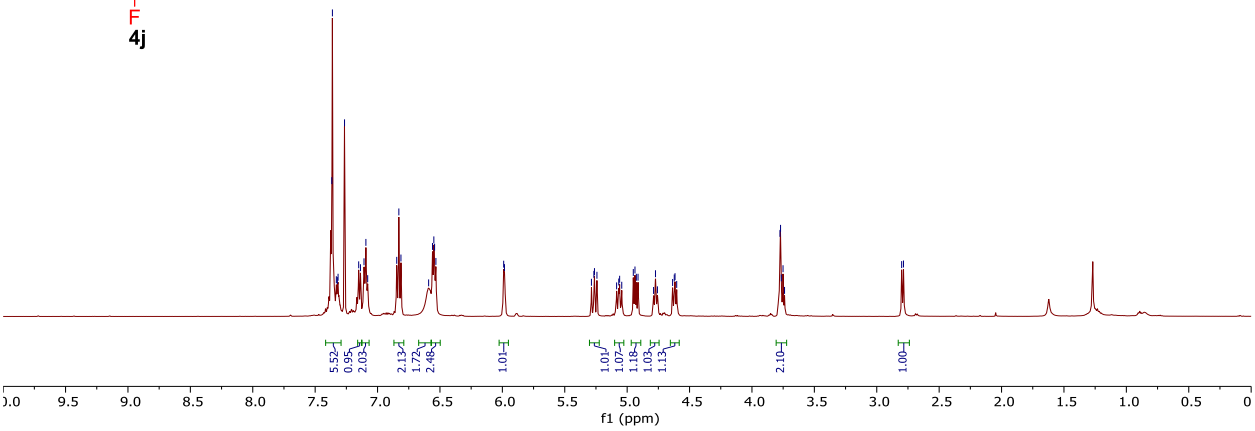

V-DM-724.2.fid
7.41 7.40 7.39 7.38 7.38 7.38 7.37 7.34 7.33 7.26 7.15 7.13 7.12 7.09 7.07 7.06 6.68 6.67 6.59 6.51 6.49 6.01 6.00 5.28 5.26 5.25 5.23 5.10 5.08 5.08 5.06 4.95 4.94 4.93 4.92 4.79 4.77 4.76 4.63 4.62 4.61 4.60 3.76 3.73 3.71 3.70 2.62 2.61
OH
O2N
NO2
OMe
4i
4.98 2.95 2.10 1.66 2.03 1.00 1.03 1.07 1.05 1.02 1.04 2.89 1.83 0.98
f1 (ppm)


V-DM-724.4.fid
159.4 137.3 136.6 136.4 131.3 131.3 129.6 128.5 128.5 128.4 128.0 127.9 113.8 90.7 79.1 77.6 77.6 77.4 77.1 73.4 55.6 49.1 47.9
OH
O2N
NO2
OMe
4i
f1 (ppm)

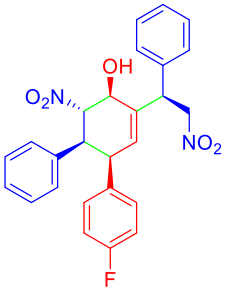
1-SJ-611-1.1.fid



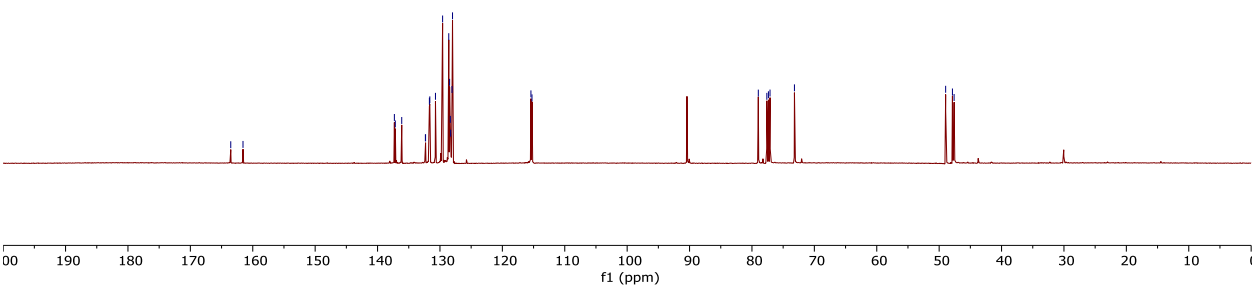
4j



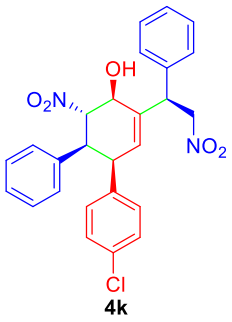
1-SJ-611-1-C13.1.fid



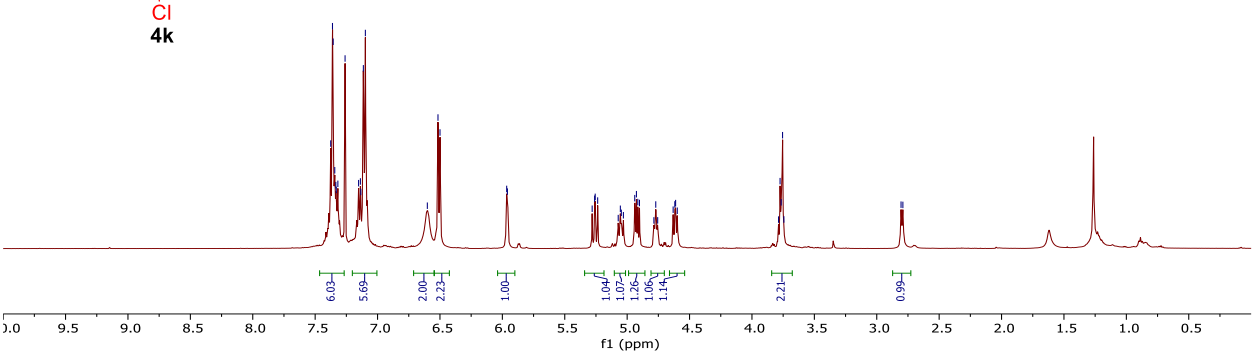
4j



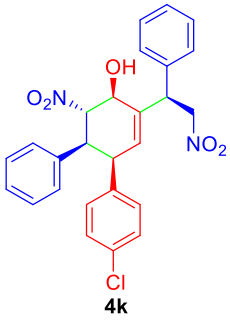
1-SJ-614-PP.2.fid



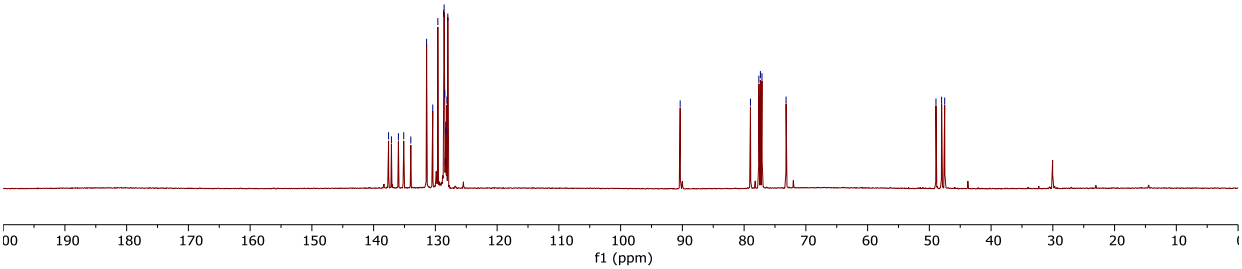
4k

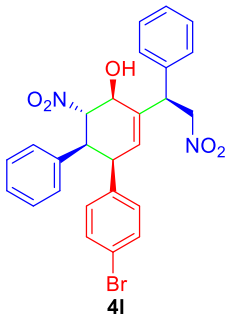


1-SJ-614-PP-C13.1.fid

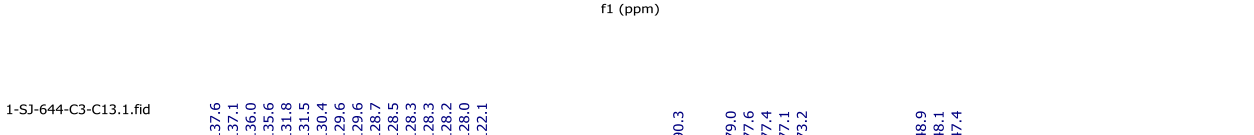


4k

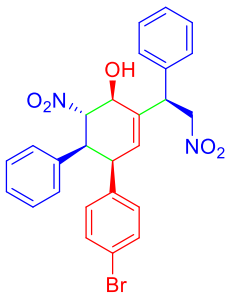




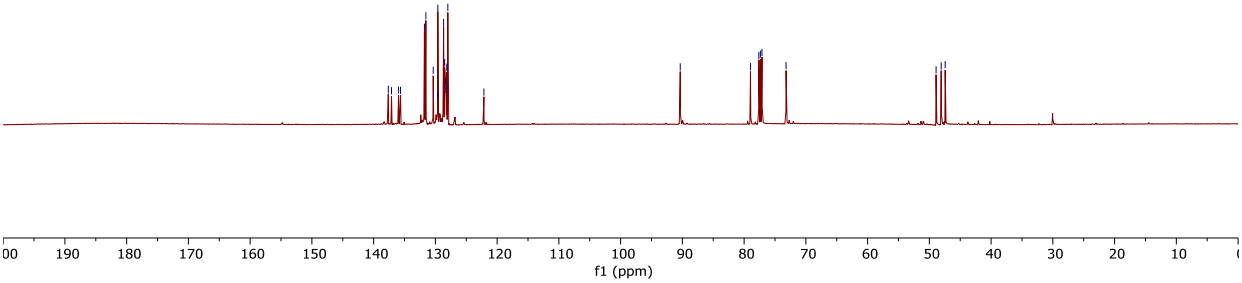
4l

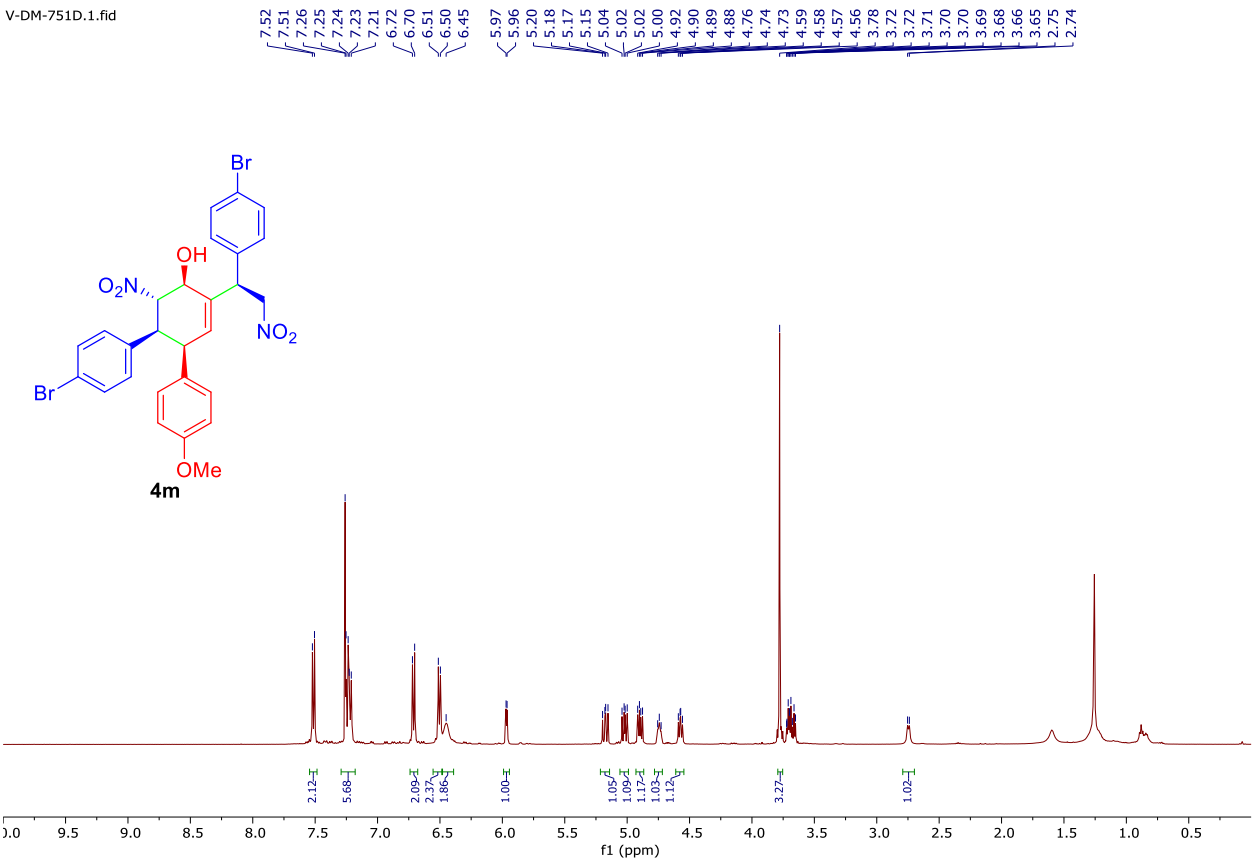


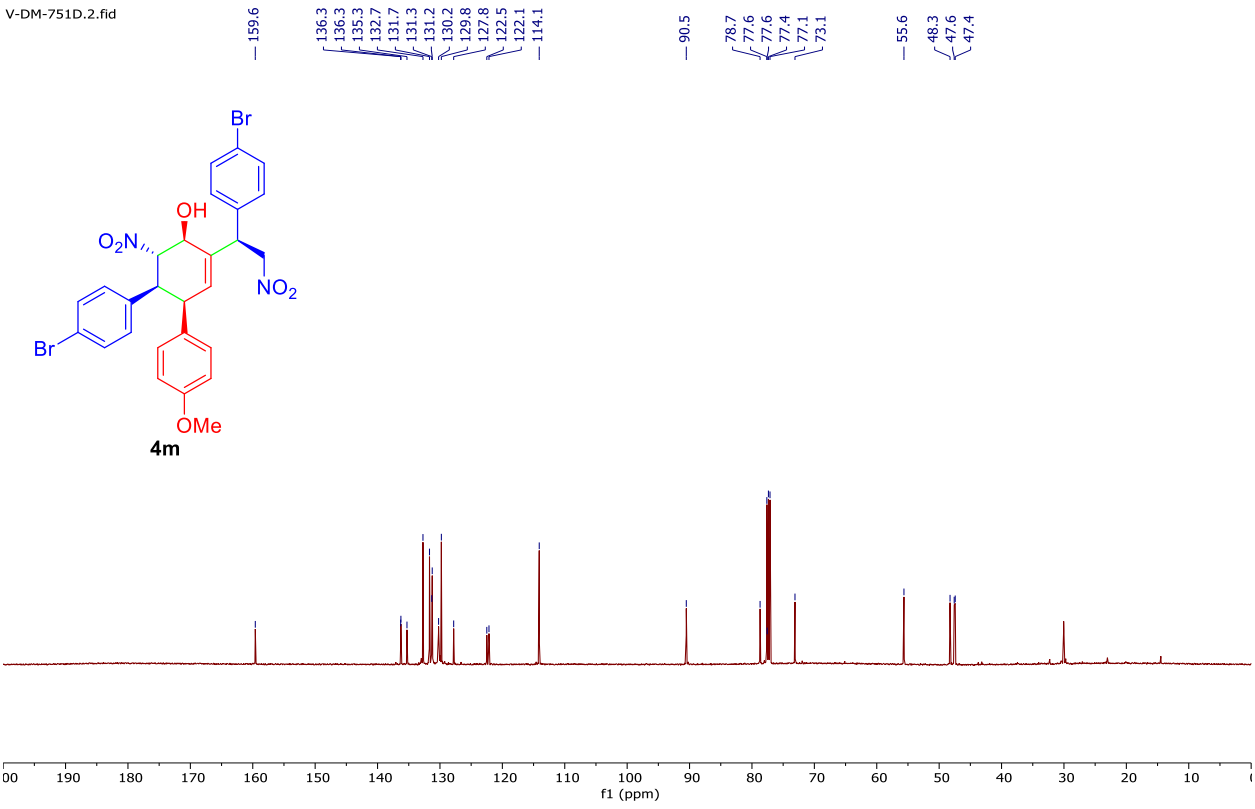
1-SJ-644-C3-C13.1.fid

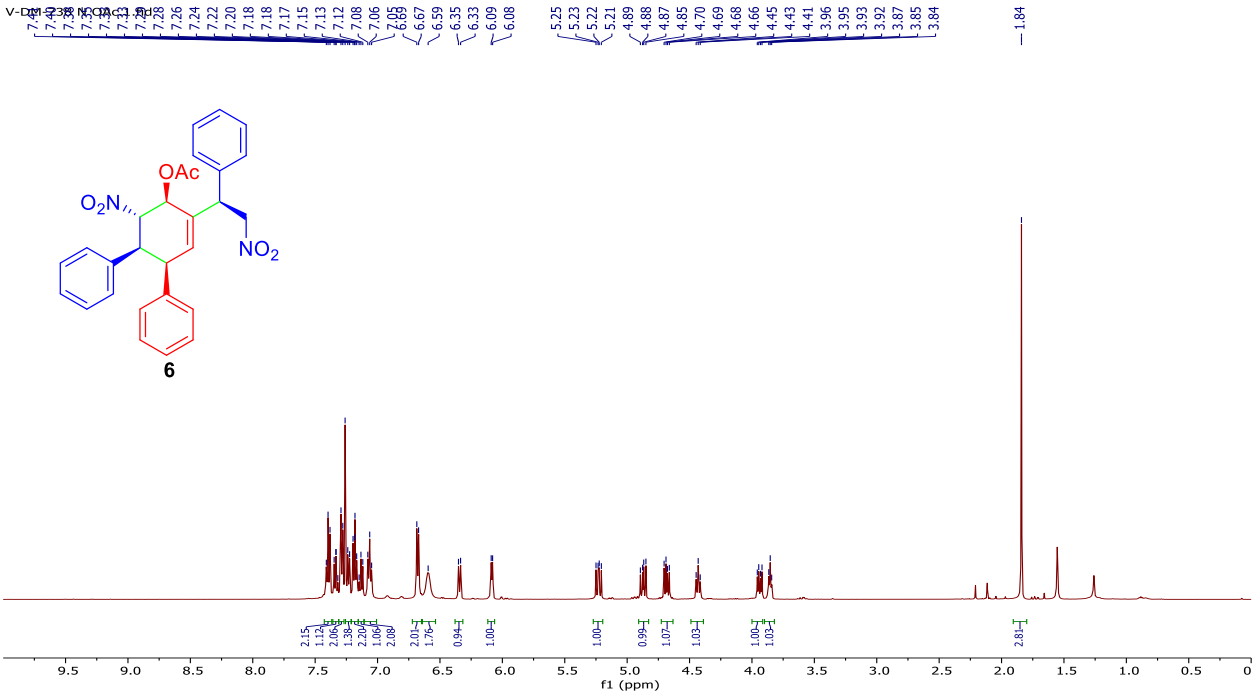


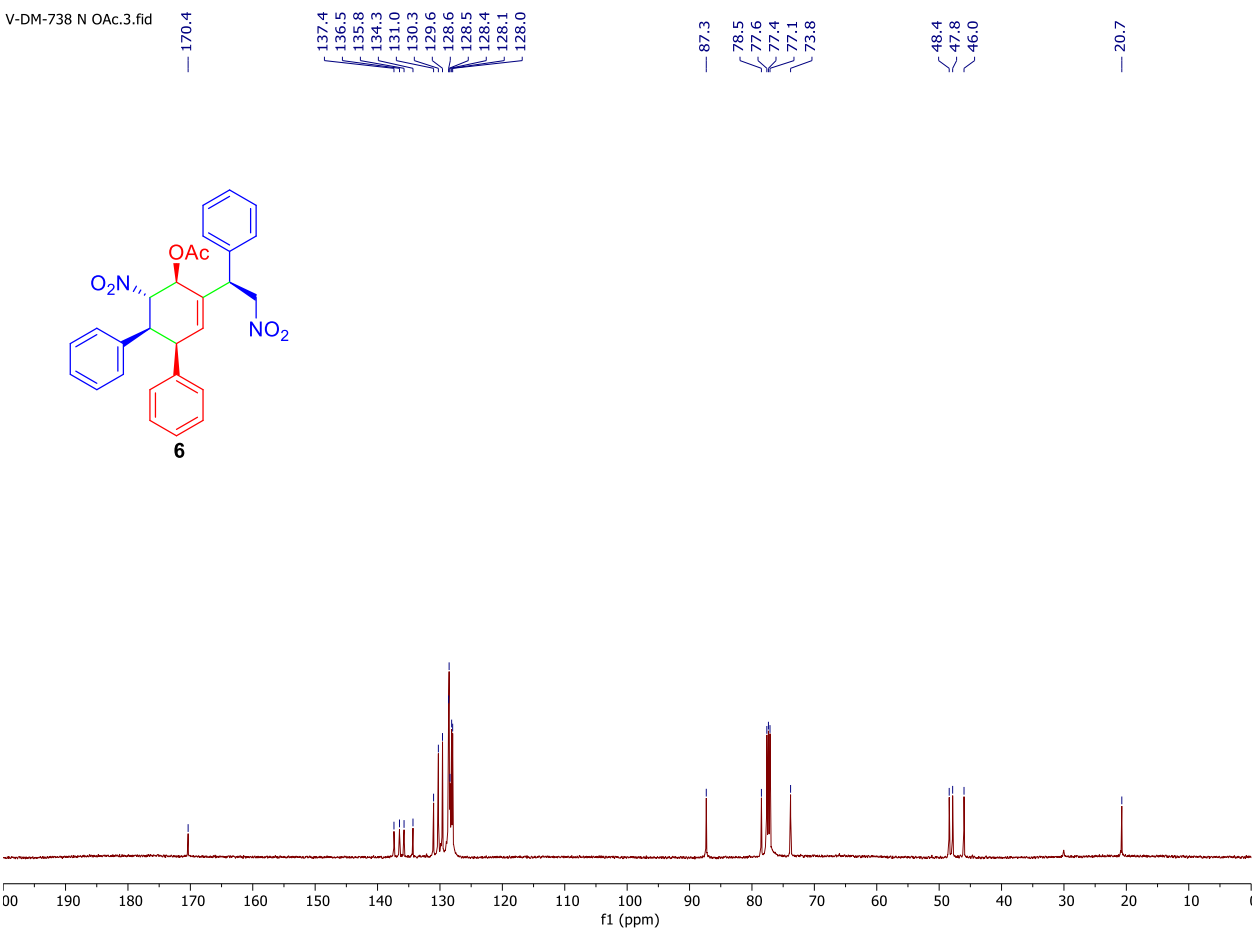
4l



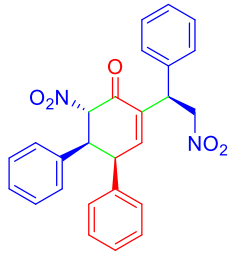

V-DM-751D.1.fid
7.52 7.51 7.26 7.25 7.24 7.23 7.21 6.72 6.70 6.51 6.50 6.45 5.97 5.96 5.20 5.18 5.17 5.15 5.04 5.02 5.02 5.00 4.92 4.90 4.89 4.88 4.76 4.74 4.73 4.59 4.58 4.57 4.56 3.78 3.72 3.72 3.71 3.70 3.70 3.69 3.68 3.66 3.65 2.75 2.74
Br
OH
O2N
NO2
Br
OMe
4m
2.12 5.68 2.09 2.37 1.86 1.00 1.05 1.09 1.17 1.03 1.12 3.27 1.02
f1 (ppm)


V-DM-751D.2.fid
159.6 136.3 136.3 135.3 132.7 131.7 131.3 131.2 130.2 129.8 127.8 122.5 122.1 114.1 90.5 78.7 77.6 77.6 77.4 77.1 73.1 55.6 48.3 47.6 47.4
Br
OH
O2N
NO2
Br
OMe
4m
f1 (ppm)

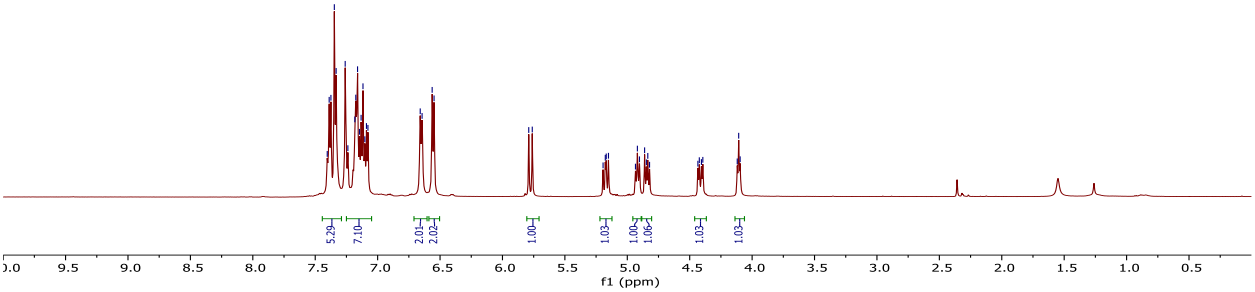

OAc
O_2N
NO_2
6
f1 (ppm)


OAc
O_2N
NO_2
6
f1 (ppm)

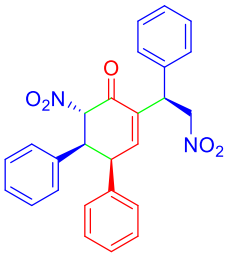

7.40
7.39
7.37
7.35
7.33
7.26
7.24
7.18
7.18
7.16
7.15
7.13
7.12
7.10
7.09
7.08
6.66
6.64
6.56
6.55
5.79
5.76
5.19
5.18
5.17
5.15
4.93
4.92
4.90
4.86
4.85
4.84
4.82
4.43
4.42
4.41
4.39
4.12
4.11
4.10


O
O_2N
NO_2

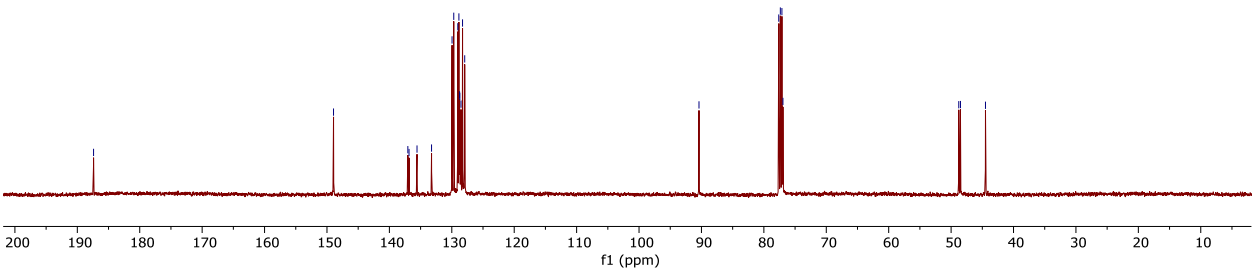
7


5.29
7.10
2.01
2.02
1.00
1.03
1.00
1.06
1.03
1.03
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


V-DM-875 D32.fid
187.2
148.9
137.0
136.9
135.6
133.2
130.0
129.7
129.0
128.9
128.8
128.7
128.5
128.3
127.9
90.4
77.6
77.4
77.1
76.9
48.8
48.5
44.5


O
O_2N
NO_2

7

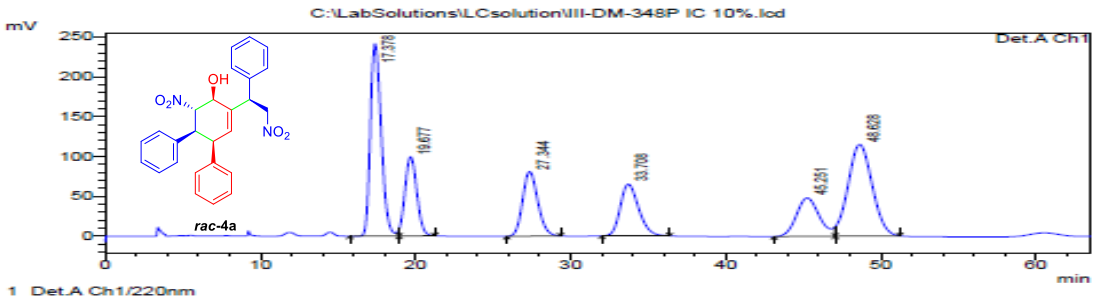

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\III-DM-348P IC 10%.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: III-DM-348P IC 10%.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 8/9/2018 6:13:54 PM
Data Processed	: 8/9/2018 7:17:27 PM

<Chromatogram>



PeakTable

Detector A Ch1 220nm

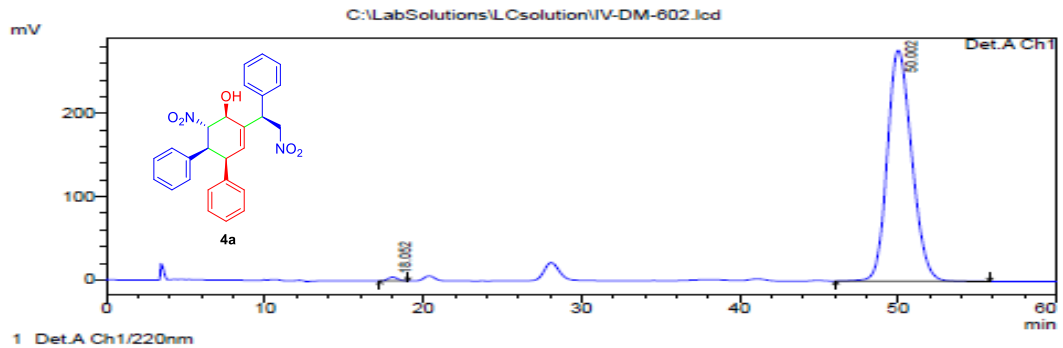
Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.378	11766105	240970	26.003	37.145
2	19.677	5271424	99123	11.650	15.280
3	27.344	5516646	80884	12.192	12.468
4	33.708	5325206	64786	11.769	9.987
5	45.251	4964602	48121	10.972	7.418
6	48.628	12405483	114842	27.416	17.703
Total		45249465	648726	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\IV-DM-602.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: IV-DM-602.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 8/25/2018 4:58:04 PM
Data Processed	: 8/25/2018 5:58:07 PM

<Chromatogram>



PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.052	194273	4598	0.646	1.631
2	50.002	29867160	277344	99.354	98.369
Total		30061434	281943	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\IV-DM-694P IB10%.lcd

Acquired by : Admin
Sample Name :
Sample ID :
Vail # :
Injection Volume : 1 uL
Data File Name : IV-DM-694P IB10%.lcd
Method File Name : ChiralPak IB-10%-1.0 mL-220nm.lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 10/4/2018 11:13:08 AM
Data Processed : 10/4/2018 4:20:22 PM

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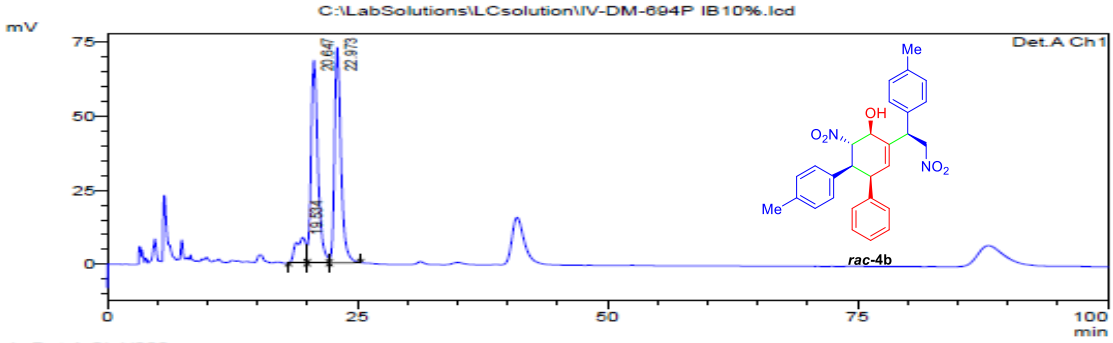

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.534	587153	8555	8.254	5.722
2	20.647	3263077	68319	45.869	45.693
3	22.973	3263743	72642	45.878	48.585
Total		7113973	149517	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\IV-DM-669A IB 10%.lcd

Acquired by : Admin
Sample Name :
Sample ID :
Vail # :
Injection Volume : 1 uL
Data File Name : IV-DM-669A IB 10%.lcd
Method File Name : ChiralPak IB-10%-1.0 mL-220nm.lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 10/4/2018 12:55:44 PM
Data Processed : 10/4/2018 2:35:47 PM

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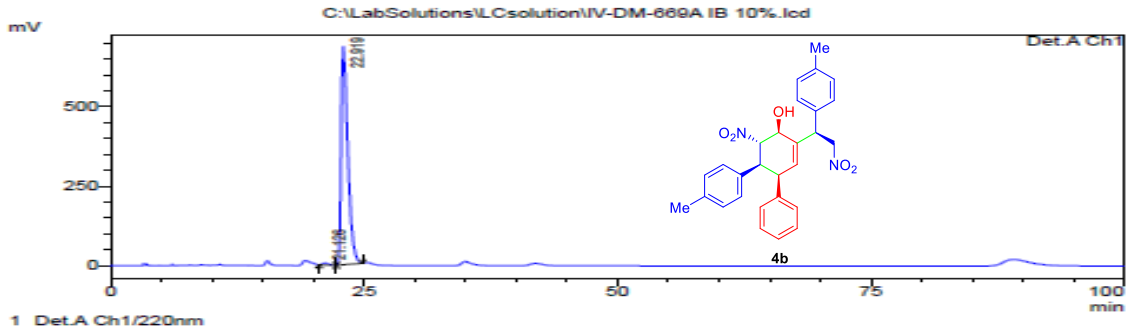

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.126	225215	5938	0.708	0.860
2	22.919	31606241	684760	99.292	99.140
Total		31831457	690697	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\V-DM-711 IC 10%.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: V-DM-711 IC 10%.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/8/2018 3:46:36 PM
Data Processed	: 10/8/2018 4:46:39 PM

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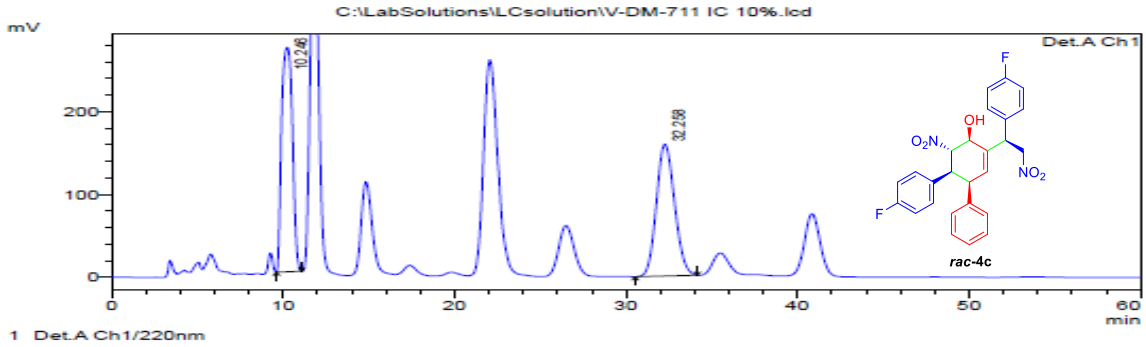

1 Det.A Ch1/220nm

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.246	11963015	271375	50.522	63.019
2	32.258	11715869	159250	49.478	36.981
Total		23678884	430625	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\IV-DM-671 4F CHI.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: IV-DM-671 4F CHI.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/8/2018 5:03:51 PM
Data Processed	: 10/8/2018 6:03:55 PM

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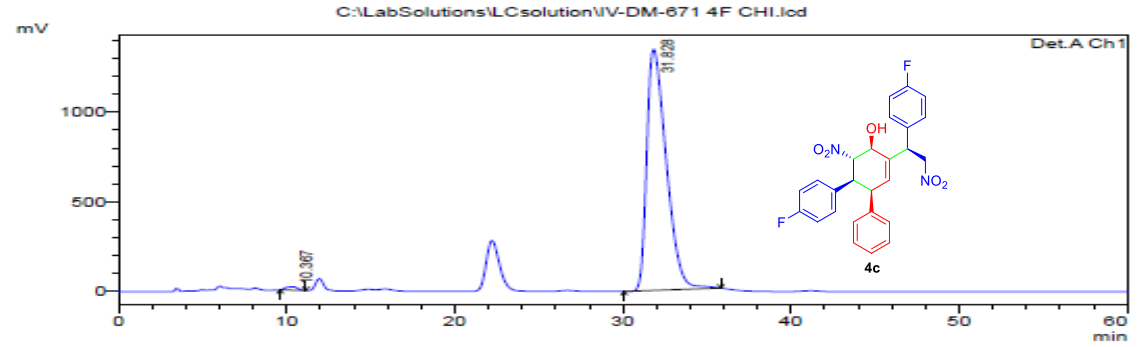

1 Det.A Ch1/220nm

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.367	898940	19002	0.823	1.393
2	31.828	108278107	1344869	99.177	98.607
Total		109177047	1363871	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\1-SJ-594.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: 1-SJ-594.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/10/2018 6:17:54 PM
Data Processed	: 10/10/2018 7:17:58 PM

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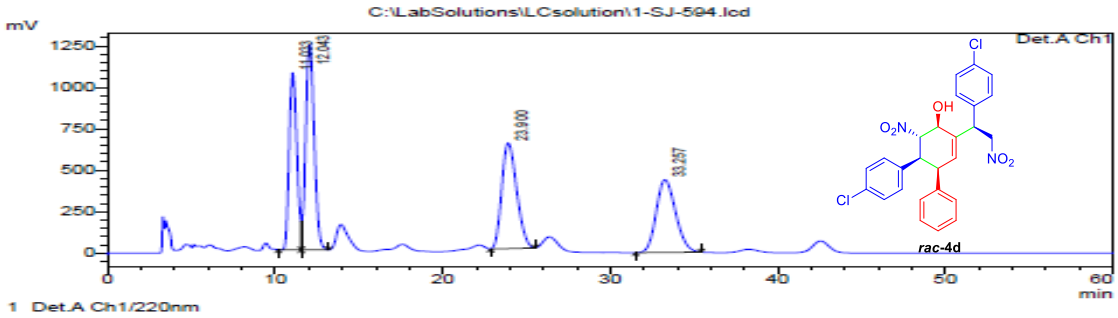

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.033	34291318	1068470	22.350	31.549
2	12.043	44135861	1240781	28.767	36.637
3	23.900	39627253	638539	25.828	18.854
4	33.257	35371075	438918	23.054	12.960
Total		153425508	3386709	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\1-SJ-569.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: 1-SJ-569.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/10/2018 1:06:15 PM
Data Processed	: 10/10/2018 2:26:37 PM

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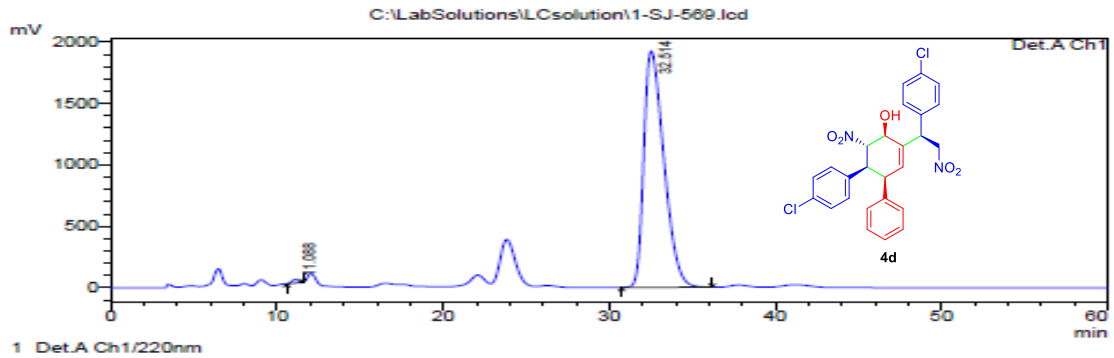

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.088	793819	29296	0.488	1.500
2	32.514	161842480	1923341	99.512	98.500
Total		162636299	1952637	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\V-DM-715 IC 7.5%.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: V-DM-715 IC 7.5%.lcd
Method File Name	: ChiralPak IC-7.5%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/9/2018 9:12:53 AM
Data Processed	: 10/9/2018 10:48:51 AM

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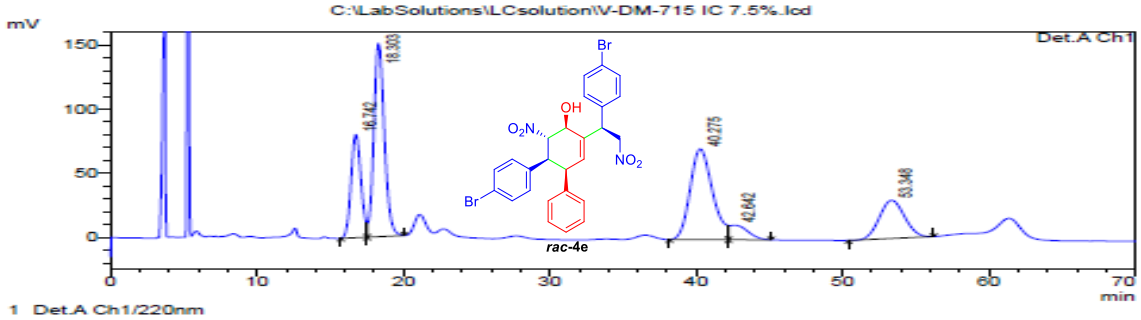


PeakTable

Detector A Ch1 220nm

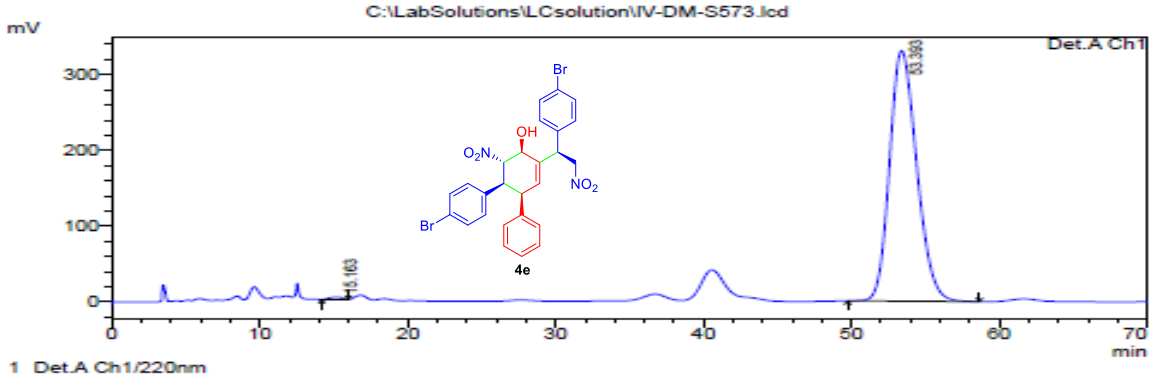
Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.742	3796840	80413	16.222	23.464
2	18.303	7538566	150689	32.208	43.970
3	40.275	7541959	70688	32.223	20.626
4	42.642	983898	11183	4.204	3.263
5	53.348	3544348	29737	15.143	8.677
Total		23405612	342710	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\IV-DM-S573.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: IV-DM-S573.lcd
Method File Name	: ChiralPak IC-7.5%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/9/2018 10:45:37 AM
Data Processed	: 10/9/2018 12:08:29 PM

<Chromatogram>



PeakTable

Detector A Ch1 220nm

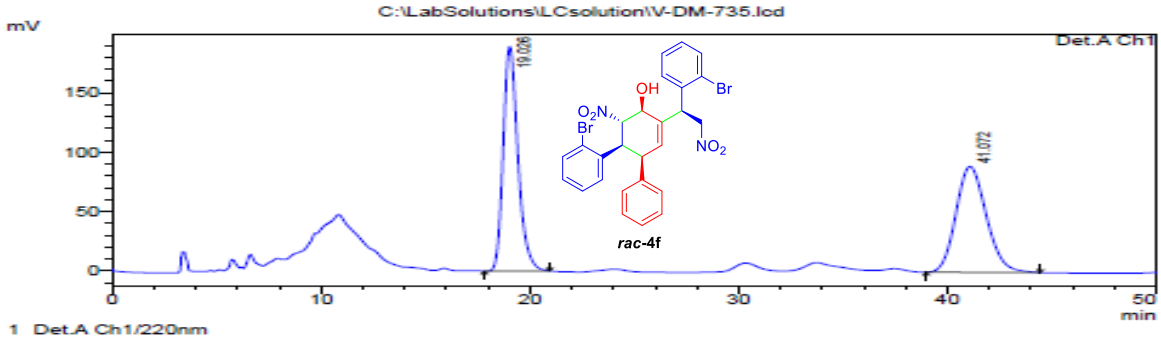
Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.163	192709	3208	0.454	0.961
2	53.393	42210372	330623	99.546	99.039
Total		42403081	333831	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\V-DM-735.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: V-DM-735.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/18/2018 4:30:30 PM
Data Processed	: 10/18/2018 5:23:41 PM

<Chromatogram>



PeakTable

Detector A Ch1 220nm

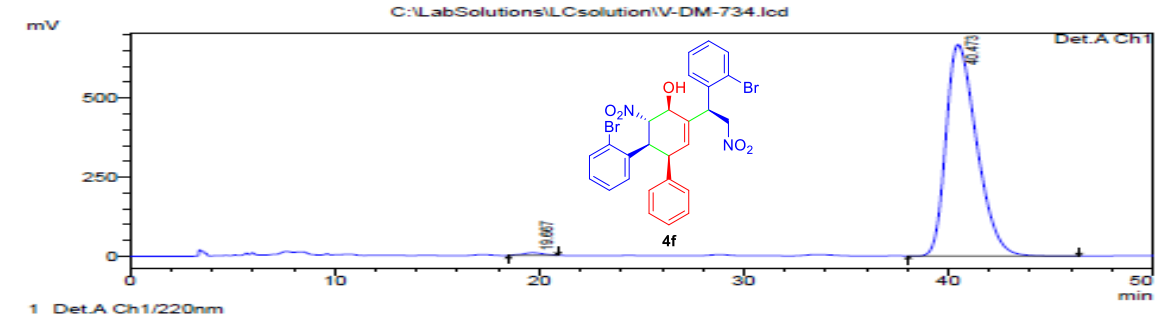
Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.026	9280395	189305	50.699	67.958
2	41.072	9024612	89256	49.301	32.042
Total		18305007	278561	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\V-DM-734.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: V-DM-734.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/18/2018 3:28:32 PM
Data Processed	: 10/18/2018 4:19:41 PM

<Chromatogram>



PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.667	588389	8374	0.833	1.237
2	40.473	70067258	668486	99.167	98.763
Total		70655647	676860	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\IV-DM-712 R IC 5%.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: IV-DM-712 R IC 5%.lcd
Method File Name	: ChiralPak IC-5%-1 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/6/2018 4:23:30 PM
Data Processed	: 10/8/2018 11:07:19 AM

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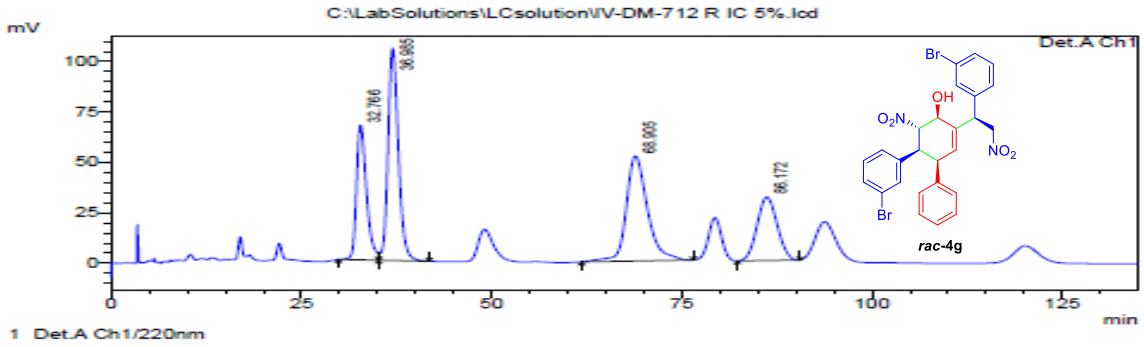

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	32.766	6089510	66851	19.081	26.172
2	36.985	10006213	105190	31.354	41.182
3	68.905	10067276	51993	31.545	20.356
4	86.172	5751168	31391	18.021	12.290
Total		31914167	255425	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\IV-DM-673 IC 5%.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: IV-DM-673 IC 5%.lcd
Method File Name	: ChiralPak IC-5%-1 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/8/2018 9:50:01 AM
Data Processed	: 10/8/2018 4:24:07 PM

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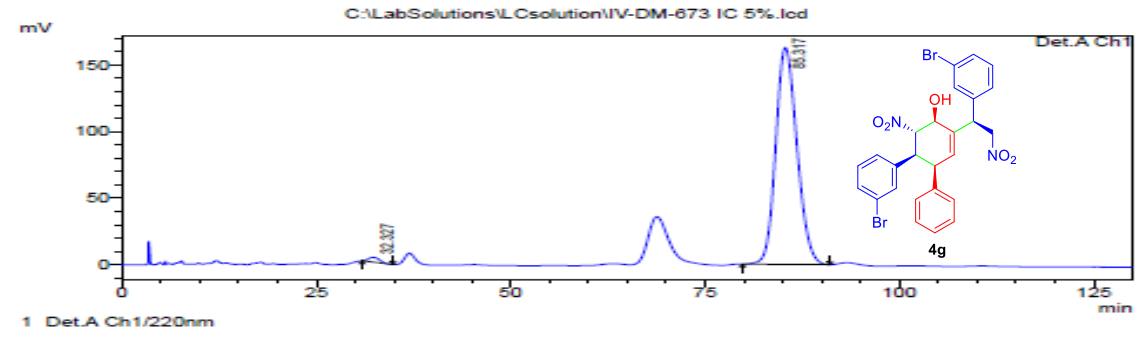

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	32.327	353945	3596	1.130	2.160
2	85.317	30959935	162916	98.870	97.840
Total		31313880	166512	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\V-DM-717 IC 10%.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: V-DM-717 IC 10%.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/9/2018 3:04:32 PM
Data Processed	: 10/9/2018 5:29:26 PM

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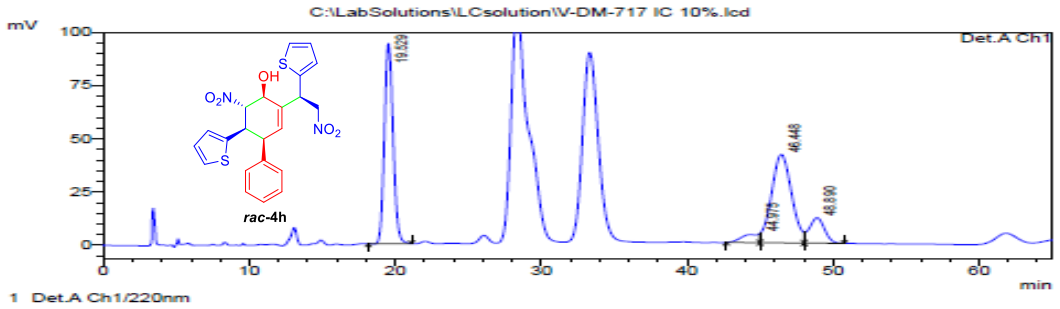

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.529	3883238	94045	42.925	61.968
2	44.975	324279	4239	3.585	2.793
3	46.448	3974710	41527	43.936	27.363
4	48.890	864311	11954	9.554	7.877
Total		9046537	151765	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\IV-DM-677 IC 10%.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: IV-DM-677 IC 10%.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/9/2018 4:36:37 PM
Data Processed	: 10/18/2018 7:14:53 PM

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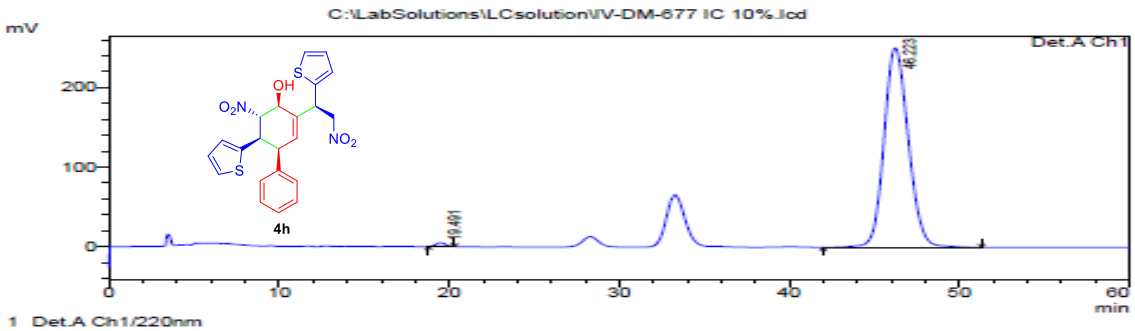

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.491	191888	4634	0.765	1.815
2	46.223	24900111	250692	99.235	98.185
Total		25092000	255326	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\V-DM-725 RAC.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: V-DM-725 RAC.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/15/2018 4:27:21 PM
Data Processed	: 10/15/2018 5:57:24 PM

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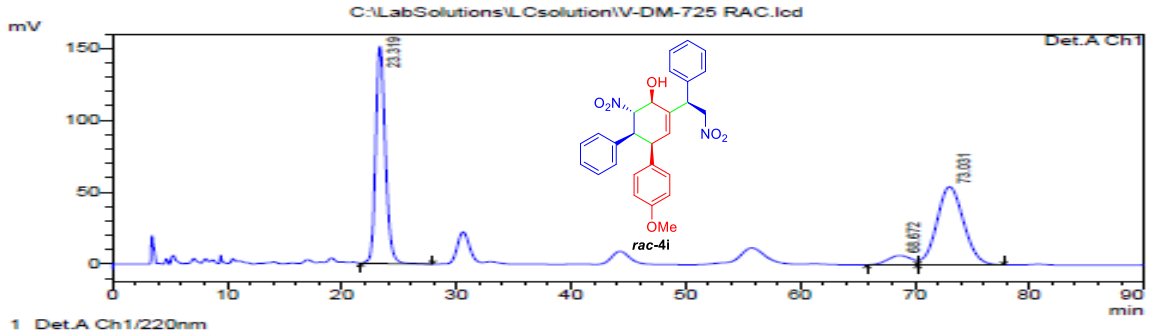

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.319	8853793	151019	47.578	71.320
2	68.672	917401	6473	4.930	3.057
3	73.031	8837952	54255	47.493	25.623
Total		18609147	211748	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\V-DM-724 IC 10%.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: V-DM-724 IC 10%.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/15/2018 6:01:26 PM
Data Processed	: 10/15/2018 7:31:29 PM

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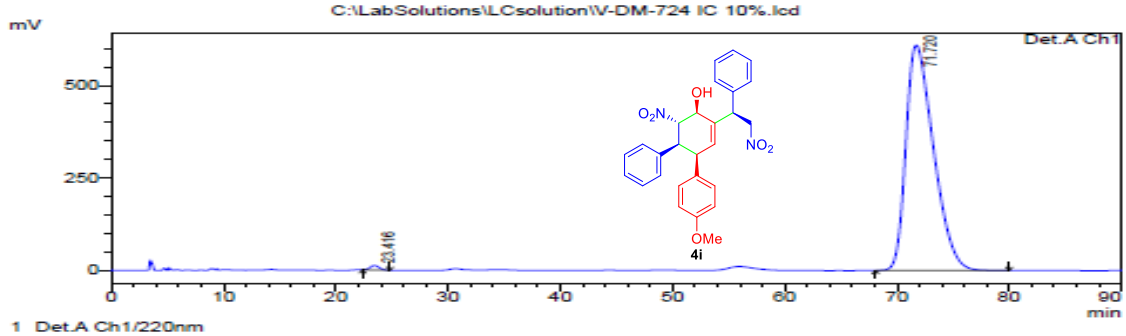

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.416	679467	12187	0.647	1.962
2	71.720	104407112	608993	99.353	98.038
Total		105086579	621181	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\1-SJ-618-10%.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: 1-SJ-618-10%.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/20/2018 2:57:47 PM
Data Processed	: 10/20/2018 3:50:05 PM

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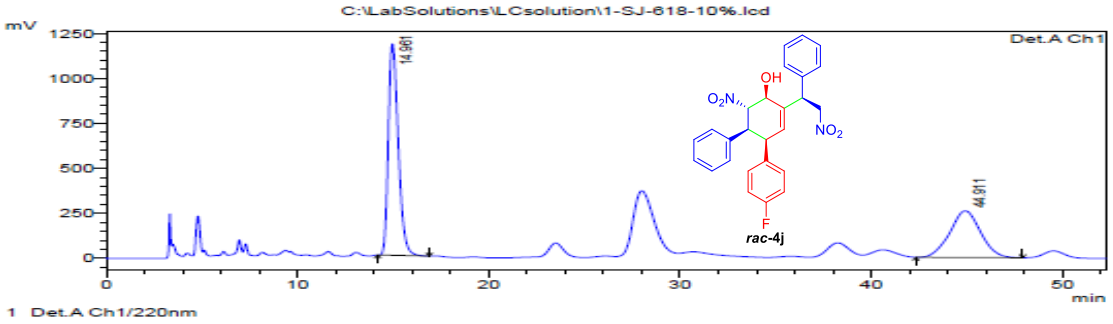


PeakTable

Detector A Ch1 220nm

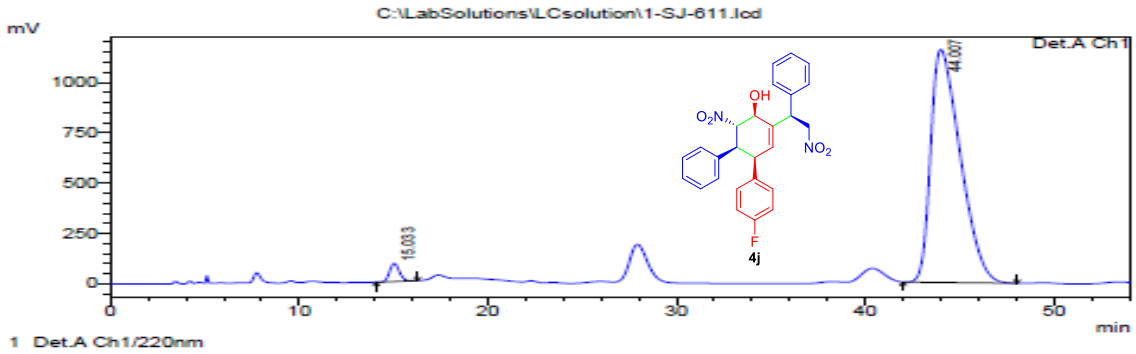
Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.961	42474210	1174565	59.477	81.936
2	44.911	28938325	258955	40.523	18.064
Total		71412534	1433521	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\1-SJ-611.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: 1-SJ-611.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/20/2018 2:01:49 PM
Data Processed	: 10/20/2018 2:55:55 PM

<Chromatogram>



PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.033	3229125	90107	2.487	7.223
2	44.007	126627979	1157355	97.513	92.777
Total		129857105	1247462	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\1-SJ-604.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: 1-SJ-604.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/28/2018 12:27:13 PM
Data Processed	: 10/28/2018 2:47:20 PM

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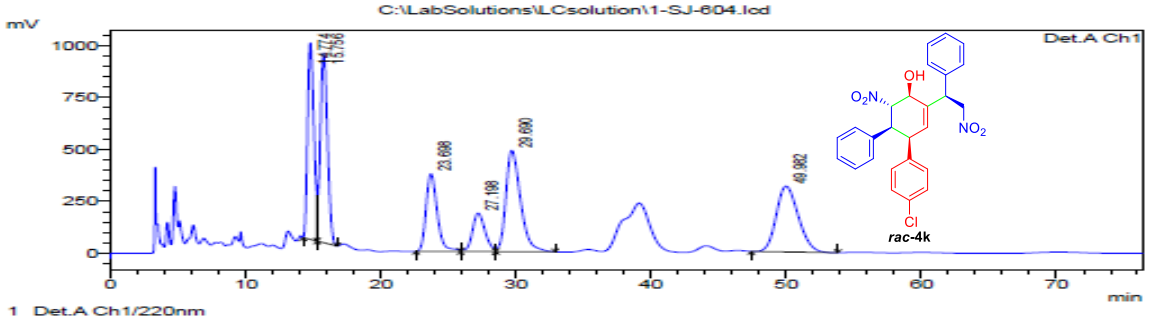

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.774	30818795	944832	18.027	29.356
2	15.756	34035482	911980	19.908	28.335
3	23.698	21527587	373322	12.592	11.599
4	27.198	11950783	184651	6.990	5.737
5	29.690	36775739	487298	21.511	15.140
6	49.982	35853756	316460	20.972	9.832
Total		170962142	3218542	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\1-SJ-614.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: 1-SJ-614.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/28/2018 1:45:10 PM
Data Processed	: 10/28/2018 2:50:11 PM

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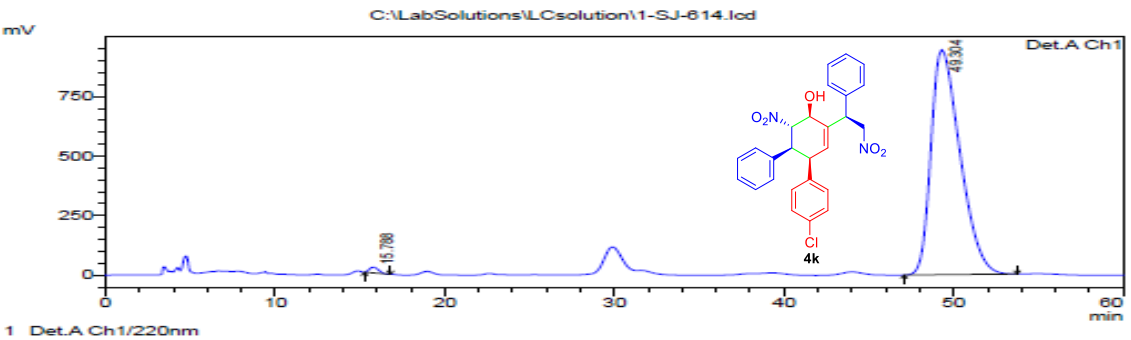

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.788	882003	24028	0.776	2.480
2	49.304	112808883	945004	99.224	97.520
Total		113690887	969032	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\1-SJ-646.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vial #	:
Injection Volume	: 1 uL
Data File Name	: 1-SJ-646.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/12/2018 1:26:01 PM
Data Processed	: 11/12/2018 4:00:17 PM

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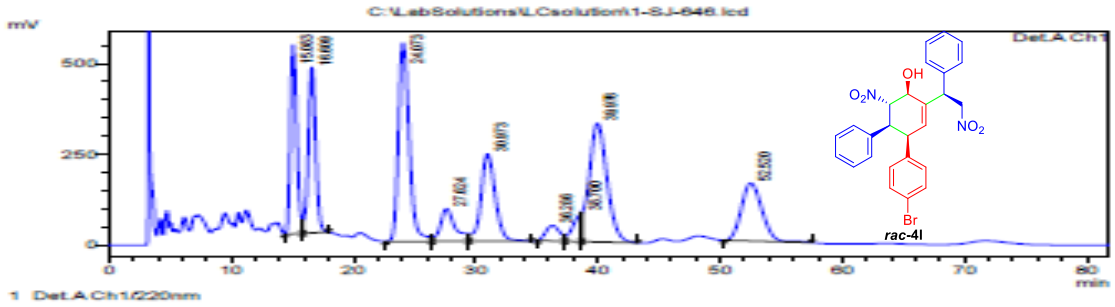

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.083	19085350	520094	12.120	21.126
2	16.609	18947981	456215	12.033	18.531
3	24.073	33544601	546321	21.303	22.191
4	27.624	7537926	90094	4.787	3.660
5	30.973	19496234	240057	12.381	9.751
6	36.286	3321221	42431	2.109	1.724
7	38.700	3792219	80365	2.408	3.264
8	39.976	39044894	326666	20.985	13.269
9	52.520	18696358	159612	11.873	6.483
Total		157466625	2461855	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\1-SJ-644.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vial #	:
Injection Volume	: 1 uL
Data File Name	: 1-SJ-644.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/12/2018 2:53:00 PM
Data Processed	: 11/12/2018 4:04:33 PM

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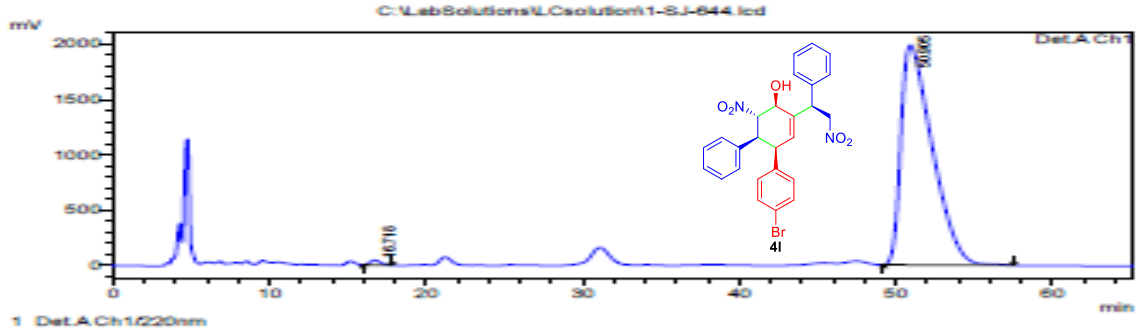


PeakTable

Detector A Ch1 220nm

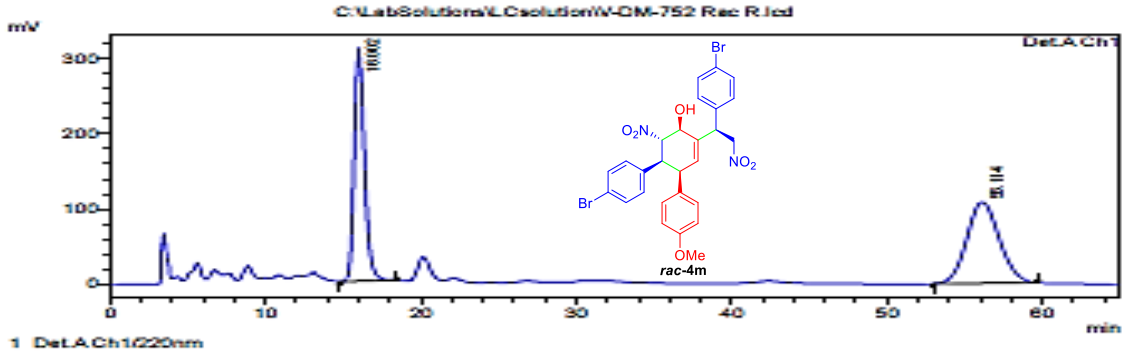
Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.718	1946296	43136	0.701	2.127
2	30.905	275893538	1985038	99.299	97.873
Total		277839834	2028174	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\V-DM-752 Rac R.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: V-DM-752 Rac R.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/24/2018 1:11:25 PM
Data Processed	: 10/24/2018 2:16:27 PM

<Chromatogram>



PeakTable

Detector A Ch1 220nm

Peak #	Ret. Time	Area	Height	Area %	Height %
1	16.002	14240049	310023	47.513	74.156
2	56.114	15718246	106082	52.467	25.844
Total		29958295	416105	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\V-DM-751 R.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: V-DM-751 R.lcd
Method File Name	: ChiralPak IC-10%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/23/2018 4:50:08 PM
Data Processed	: 10/23/2018 5:55:11 PM

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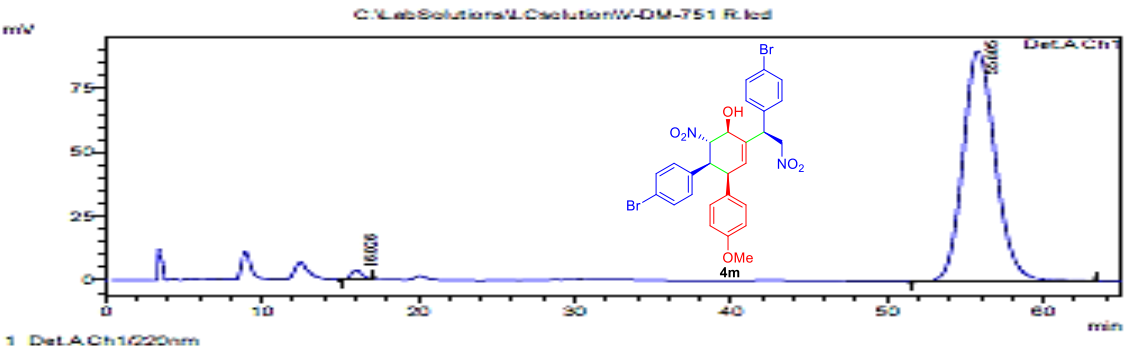


PeakTable

Detector A Ch1 220nm

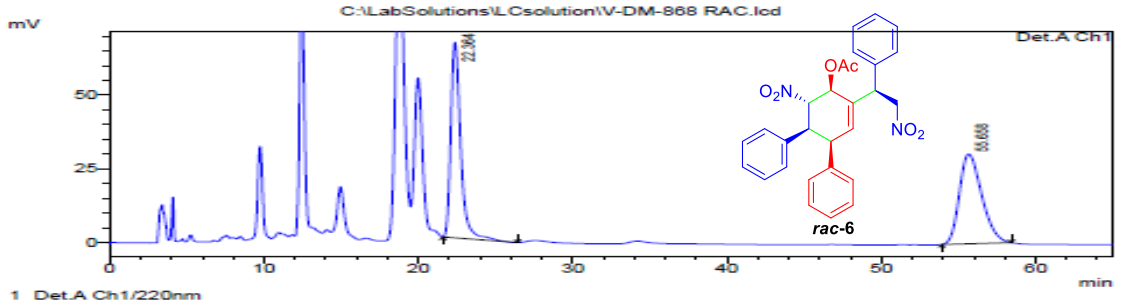
Peak #	Ret. Time	Area	Height	Area %	Height %
1	16.026	163001	3586	1.245	3.823
2	55.806	12929916	90289	98.755	96.177
Total		13092917	93875	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\V-DM-868 RAC.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: V-DM-868 RAC.lcd
Method File Name	: ChiralPak ID-15.0%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 12/19/2018 4:38:24 PM
Data Processed	: 12/19/2018 5:45:54 PM

<Chromatogram>



PeakTable

Detector A Ch1 220nm

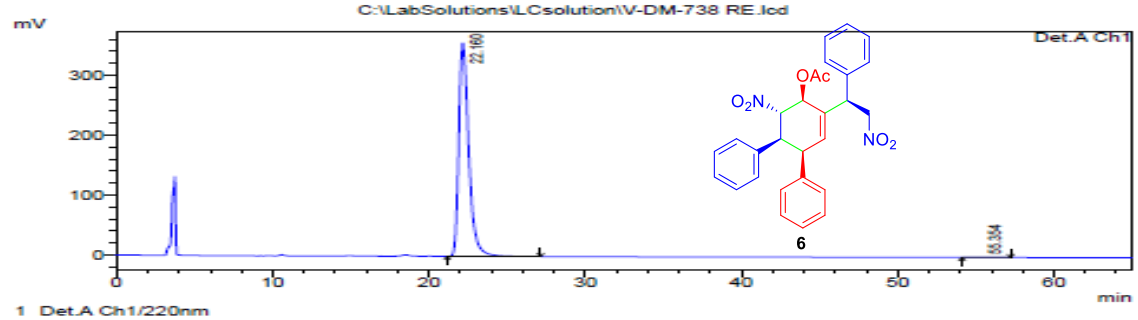
Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.364	2954339	66365	49.052	68.549
2	55.658	3068489	30450	50.948	31.451
Total		6022827	96815	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\V-DM-738 RE.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: V-DM-738 RE.lcd
Method File Name	: ChiralPak ID-15.0%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 12/19/2018 3:14:12 PM
Data Processed	: 12/19/2018 4:21:54 PM

<Chromatogram>



PeakTable

Detector A Ch1 220nm

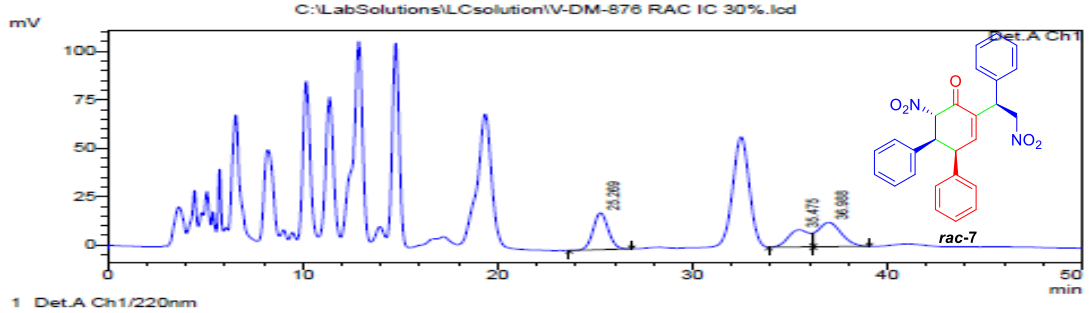
Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.160	15855045	356185	99.816	99.907
2	55.354	29215	333	0.184	0.093
Total		15884261	356518	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\V-DM-876 RAC IC 30%.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: V-DM-876 RAC IC 30%.lcd
Method File Name	: ChiralPak IC-30%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 12/31/2018 2:29:46 PM
Data Processed	: 12/31/2018 6:10:59 PM

<Chromatogram>



PeakTable

Detector A Ch1 220nm

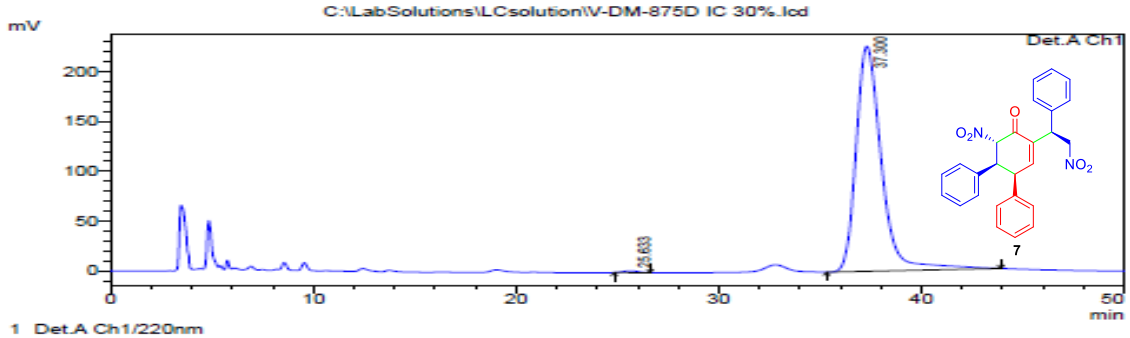
Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.269	1023779	18956	37.358	47.112
2	35.475	676890	8835	24.700	21.957
3	36.988	1039798	12446	37.942	30.931
Total		2740467	40236	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\LCsolution\V-DM-875D IC 30%.lcd

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail #	:
Injection Volume	: 1 uL
Data File Name	: V-DM-875D IC 30%.lcd
Method File Name	: ChiralPak IC-30%-1.0 mL-220nm.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 12/31/2018 4:14:49 PM
Data Processed	: 12/31/2018 6:05:23 PM

<Chromatogram>



PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.633	91002	1820	0.460	0.798
2	37.300	19679493	226138	99.540	99.202
Total		19770494	227957	100.000	100.000