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Enantioselective synthesis of 4*H*-pyranonaphthoquinones *via* sequential squaramide and silver catalysis

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Supporting Information

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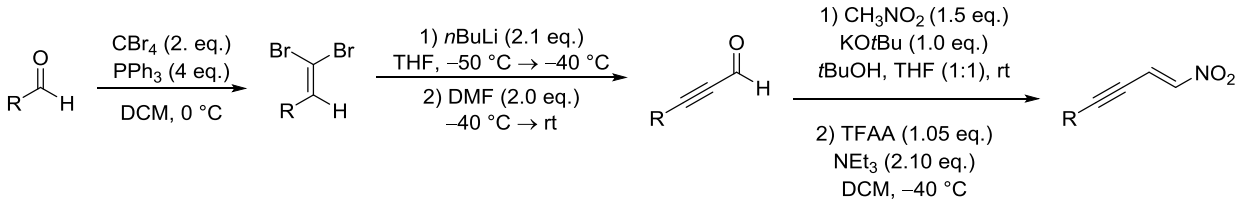
General Methods and Materials

Unless otherwise noted, all commercially available chemicals were used without purification. Analytical TLC were performed using SIL G-25 UV252 from *Macherey&Nagel* (particle size 0.040-0.063 nm; 230-240 mesh. flash) and visualized with ultraviolet radiation at 254 nm. ^{1}H and ^{13}C NMR spectra were recorded at ambient temperature on a *Varian* Innova 400 or Innova 600. Chemical shifts for ^{1}H NMR and ^{13}C NMR are reported in parts per million (ppm) and coupling constants were reported in Hertz (Hz). The following abbreviations are used for the spin multiplicity: s = singlet, d = doublet, dd = doublet of doublet, t = triplet and m = multiplet. Optical rotations were measured on a *Perkin-Elmer* 241 polarimeter. Mass spectra were acquired on a *ThermoFinnigan* LCQ Deca XP plus (ESI) spectrometer and high resolution ESI spectra on a *ThermoFisher* Scientific LTQ Orbitrap XL. Analytical HPLC was performed on a *Aligent* 1100, *Aligent* 1260 or *Hewlett-Packard* 1100 Series instrument using chiral stationary phases (Chiralpak IC, IB and AS; *Daicel*; Whelk 01). During optimization of the organocatalytic step for the determination of the enantiomeric excess of the Michael adduct **3a**, the corresponding OTBS protected derivative **3a'** was synthesized in order to achieve a higher resolution on the HPLC. Catalyst **K**, nitroalkenes **2** and the substituted 2-hydroxy-1,4-naphthoquinones were prepared according to known procedures.[1, 2, 3]

[1] W. Yang and D.-M. Du, *Adv. Synth. Catal.*, 2011, **353**, 1241
[2] D. Hack, P. Chauhan, K. Deckers, Y. Mizutani, G. Raabe and D. Enders, *Chem. Commun.*, 2015, **51**, 2266
[3] R.-Y. Yang, D. Kizer, H. Wu, E. Volckova, X.-S. Miao, S. M. Ali, M. Tandon, R. E. Savage, T. C. K. Chan and M. A. Ashwell, *Bioorganic & Medicinal Chemistry*, 2008, **16**, 5635.

General Procedure

Synthesis of nitroalkenes[1]:



General procedure for the preparation of *gem*-dibromoolefines (first step):

Under an atmosphere of argon, a solution of triphenylphosphine (4.0 eq.) and tetrabromomethane (2.0 eq.) in abs. DCM (0.15 M) was stirred at 0 °C for 30 minutes. Then the aldehyde was added over a period of five minutes, and the mixture as stirred at 0 °C for one hour. After addition of water, the layers were separated, and the aqueous layer was extracted with DCM (three times). The combined organic layers were dried over $MgSO_4$ and the solvent was removed under reduced pressure. The crude product was dry-loaded on silica and subjected to flash chromatography (silica, *n*-pentane/DCM).

General procedure for the Corey-Fuchs homologisation (second step):

Under an atmosphere of argon, *n*-BuLi (2.1 eq., 1.6 M in *n*-hexane) was added over a period of 30 minutes *via* syringe pump to a solution of *gem*-dibromoolefine (1.0 eq.) in abs THF (0.4 M) at −50 °C, and the mixture was stirred at −40 °C for 15 minutes. After addition of DMF (2.0 eq.) at once, the mixture was allowed to warm to room temperature and stirred for one hour. The mixture was added to a stirring solution of NaH_2PO_4 (aq.)/MTBE (1:1). After five minutes, the layers were separated and the aqueous layer was extracted with MTBE. The combined organic layers were dried over $MgSO_4$, the solvent was removed under reduced pressure and the crude product was subjected to flash chromatography (silica, *n*-pentane/EtOAc).

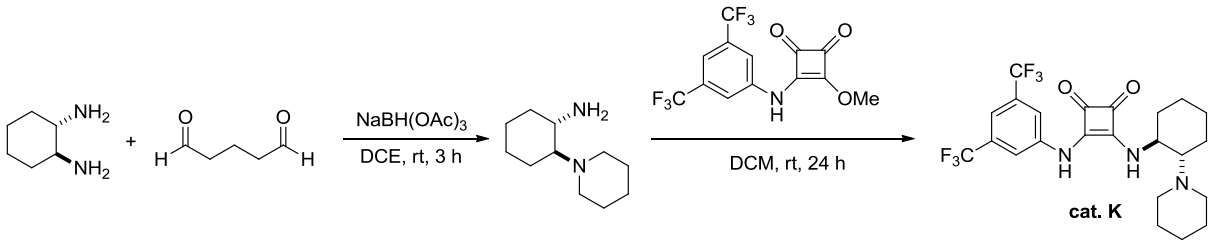
General procedure for the preparation of nitro olefines (third step):

Under an atmosphere of argon, nitromethane (1.5 eq.) and KO*t*Bu (1 eq.) were added to a solution of aldehyde (1.0 eq.) in a solution of THF and *t-BuOH* (1:1, 0.5 M), and the mixture was stirred overnight. Then a solution of NH_4Cl was added, the product was extracted twice with Et_2O, the combined organic phases were washed with brine and dried over $MgSO_4$. The

solvent was removed under reduced pressure and the crude product was subjected to flash chromatography on silica (*n*-pentane/Et_2O 5:1 to 1:1).

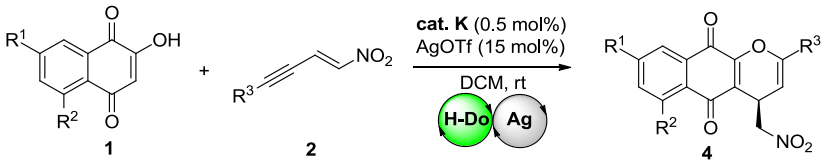
Under an atmosphere of argon, TFAA (1.05 eq.) was added over a period of 30 min to a solution of nitroalcohol (1.0 eq.) in DCM (0.5 M) at −40 °C. Then triethylamine (2.1 eq.) was added, and the mixture was stirred at −0 °C for 45 min. The reaction was quenched with a solution of NH_4Cl was added, the liquid phases were separated, and the aqueous phase was extracted twice with DCM. The combined organic phases were washed with a saturated solution of $NaHCO_3$ and brine, and dried over Na_2SO_4. The solvent was removed under reduced pressure and the product was purified by flash chromatography on silica (*n*-pentane/Et_2O 5:1).

Synthesis of the squaramide catalyst K^2:



Aqueous glutaraldehyde (50%, 1.0 mL) was added dropwise into a mixture of $NaBH(OAc)_3$ (4.24 g, 20.0 mmol) and (1*S*,2*S*)-1,2-diaminocyclohexane (570 mg, 5.0 mmol) in DCE (30 mL) at room temperature. The resulting mixture was stirred at room temperature for 3 h, and then quenched with aq. NaOH (10%, 20 mL). The organic layer was separated and the aq. layer was extracted with DCM (3 x 20 mL). The combined organic layers were washed with brine (20 mL), dried over anhydrous Na_sSO_4, and concentrated to give the crude product as a yellow liquid. To a solution of the crude product (273 mg, 1.5 mmol) in DCM (5 mL) was added 3-((3,5-bis(trifluoromethyl)phenyl)amino)-4-methoxycyclobut-3-ene-1,2-dione (271 mg, 1.0 mmol). The reaction was stirred at room temperature for 24 h. Then the mixture was concentrated and purified by silica gel column chromatography (100 g silica gel was treated with 5 mL $NH_3 \cdot H_2O$, using DCM as eluant) to afford the desired product as a pale yellow solid.

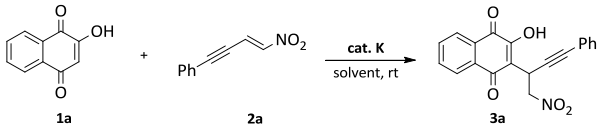
Synthesis of 4*H*-pyranonaphthoquinones:



A solution of catalyst **K** (0.5 mL, 0.5 mol%, 1.2 mg/mL) and nitroalkene **2** (0.275 mmol, 1.1 eq.) in DCM (2 mL) was stirred for 10 min at room temperature under exclusion of light. 2-Hydroxy-1,4-naphthoquinone **1** (0.25 mmol) was added, and the reaction mixture was stirred until complete conversion of the naphthoquinone substrate as indicated by TLC. Then AgOTf (15 mol%) was given to the reaction mixture. After complete conversion, the crude product was directly purified by flash column chromatography to afford the desired product as a yellow solid[4].

Optimization Studies of the Michael Addition

Table 1 Optimization Studies of the Michael addition[a]

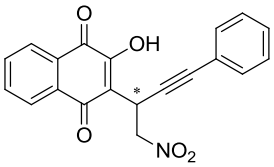


Entry	Solvent	Time (h)	Yield (%)	*ee* (%)[e]
1	DCM	0.5	94	96
2	toluene	1	88	94
3	THF	1	94	95
4	benzene	1	91	94
5	Et_2O	1.5	96	95
6	$CHCl_3$	0.5	92	95
7[b]	DCM	0.5	95	95
8[c]	DCM	1	95	96
9[d]	DCM	1.5	96	97

[a]Reaction conditions: 1,4-naphthoquinone **1a** (0.25 mmol), nitroalkene **2a** (1.1 eq.), **cat K** (5 mol%), solvent (2.5 mL, 0.1 M).[b]Reaction was carried out with **cat K** (1 mol%).[c]Reaction was carried out with **cat K** (0.5 mol%).[d]Reaction was carried out with **cat K** (1 mol%) at 0 °C.[e]Enantiomeric excess was determined by HPLC analysis of the TBS-protected derivative **3a'**.

Analytical Data

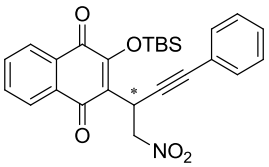
2-Hydroxy-3-(1-nitro-4-phenylbut-3-yn-2-yl)naphthalene-1,4-dione (3a)



[4] The pyranonaphthoquinones **4** are light and heat sensitive.

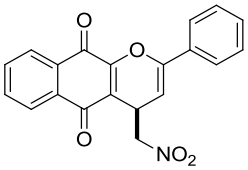
Compound **3a** was isolated after flash chromatography (*n*-pentane : ethyl acetate 4:1) as red solid (82 mg, 94%). **Molecular formula**: $C_{20}H_{13}NO_5$. **Molecular mass**: 347.32 g mol^{-1}. **R_f =** 0.65 (DCM). **Mp**: 140-142 °C. **OR**: $[\alpha]_D^{21}$ = 19.4 (c = 0.5, EtOH, 96% *ee*). **^{1}H NMR** (400 MHz, CD_3OD)[5]: δ = 8.06 (m, 2 H, Ar-H), 7.76 (m, 2 H, Ar-H), 7.36 – 7.31 (m, 2 H, Ar-H), 7.28 – 7.22 (m, 3 H, Ar-H), 5.21 (dd, *J* = 8.3, 7.0 Hz, 1 H, CH_2CH), 5.10 (dd, *J* = 12.7, 8.3 Hz, 1 H, C*H*H), 4.94 (dd, *J* = 12.7, 7.0 Hz, 1 H, CH*H*) ppm. **^{13}C NMR** (101 MHz, CD_3OD): δ = 183.0 (C=O), 180.6 (C=O), 156.6 (C_q), 134.5 (Ar-C), 133.0 (Ar-C), 132.2 (C_q), 131.3 (2 C, Ar-C), 130.0 (C_q), 127.9 (3 C, Ar-C), 126.1 (Ar-C), 125.7 (Ar-C), 122.7 (C_q), 117.1 (C_q), 84.3 (alkyne), 82.3 (alkyne), 75.4 (NO_2CH_2), 26.1 (CH_2CH) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3327, 3040, 2924, 2321, 2095, 1742, 1647, 1541, 1359, 1253, 1041, 894, 729, 689 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 370.07 [M+Na]$^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for [M+Na]$^+$ = 370.0686; found: 370.0686.

2-((tert-Butyldimethylsilyl)oxy)-3-(1-nitro-4-phenylbut-3-yn-2-yl)naphthalene-1,4-dione (3a')



Compound **3a'** was isolated after flash chromatography (*n*-pentane : ethyl acetate 4:1) as yellow oil (76 mg, 70%). **Molecular formula**: $C_{26}H_{27}NO_5Si$. **Molecular mass**: 461.58 g mol^{-1}. **R_f =** 0.68 (*n*-pentane : ethyl acetate 4:1). **HPLC**: Whelk 01, 97:3 *n*-heptane : EtOH, 0.7 ml/min, λ = 254 nm, τ_{minor} = 13.6 min, τ_{major} = 11.9 min. **OR**: $[\alpha]_D^{21}$ = 14.6 (c = 0.5, $CHCl_3$, 96% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 8.13 – 8.10 (m, 1 H, Ar-H), 8.08 – 8.04 (m, 1 H, Ar-H), 7.75 (m, 1 H, Ar-H), 7.71 (m, 1 H, Ar-H), 7.40 – 7.35 (m, 2 H, Ar-H), 7.30 – 7.24 (m, 3 H, Ar-H), 5.29 (m, 1 H, CH_2CH), 5.13 – 5.05 (m, 2 H, CH_2), 1.07 (s, 9 H, ^{t}Bu), 0.42 (s, 3 H, CH_3), 0.39 (s, 3 H, CH_3) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 183.8 (C=O), 181.0 (C=O), 155.7 (C_q), 134.6 (Ar-C), 133.3 (Ar-C), 132.1 (C_q), 131.8 (2 C, Ar-C), 130.2 (C_q), 128.4 (Ar-C), 128.2 (2 C, Ar-C), 126.5 (Ar-C), 126.4 (Ar-C), 123.7 (C_q), 122.5 (C_q), 84.3 (alkyne), 83.4 (alkyne), 76.0 (NO_2CH_2), 27.2 (CH_2CH), 25.9 (3 C, ^{t}Bu), 19.2 (Si-C), -3.7 (2 C, d, CH_3) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3589, 2936, 2323, 2089, 1905, 1578, 1352, 1252, 1005, 814, 585 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 484.15 [M+Na]$^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for [M+Na]$^+$ = 484.1551; found: 484.1542.

(*R*)-4-(Nitromethyl)-2-phenyl-4*H*-benzo[*g*]chromene-5,10-dione (4a)

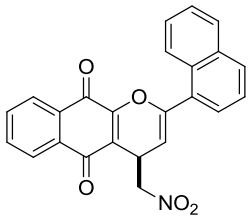


Compound **4a** was isolated after flash chromatography (DCM : *n*-pentane 7:3) as yellow solid (81 mg, 93%). **Molecular formula**: $C_{20}H_{13}NO_5$. **Molecular mass**: 347.08 g mol^{-1}. **R_f =** 0.61 (DCM). **Mp**: Decomposition at 175-177 °C. **HPLC**: IB, 9:1 *n*-heptane : *i*-PrOH , 1.0 ml/min, λ = 254 nm, τ_{minor} = 27.0 min, τ_{major} = 23.5 min. **OR**: $[\alpha]_D^{21}$ = –22.5 (c = 0.2, $CHCl_3$, 98% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 8.17 (m, 1 H, Ar-H), 8.13 (m, 1 H, Ar-H), 7.81 – 7.74 (m, 2 H, Ar-H), 7.72

[5] The hydroxyl substituent was not observed on the NMR spectra.

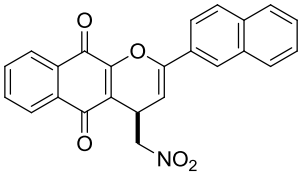
– 7.68 (m, 2 H, Ar-H), 7.45 – 7.37 (m, 3 H, Ar-H), 5.68 (d, *J* = 4.4 Hz, 1 H, ArC=C*H*), 4.78 – 4.69 (m, 2 H, CH_2NO_2), 4.43 (dt, *J* = 6.2, 4.4 Hz, 1 H, CH_2CH) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 183.9 (C=O), 177.5 (C=O), 153.0 (C_q), 150.7 (C_q), 134.5 (Ar-C), 134.0 (Ar-C), 131.6 (C_q), 131.5 (C_q), 130.8 (C_q), 129.9 (Ar-C), 128.6 (2 C, Ar-C), 126.8 (Ar-C), 126.4 (Ar-C), 125.0 (2 C, Ar-C), 117.0 (C_q), 97.1 (ArC=*C*H), 78.7 (CH_2NO_2), 30.4 (CH_2CH) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3069, 2183, 2094, 1979, 1900, 1672, 1644, 1594, 1549, 1493, 1388, 1333, 1242, 1197, 1156, 1120, 1076, 1011, 941, 900, 854, 761, 718, 691 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 370.07 [M+Na]$^+$, 386.04 [M+K]$^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for [M+Na]$^+$ = 370.0686; found: 370.0688.

(*R*)-2-(Naphthalen-1-yl)-4-(nitromethyl)-4*H*-benzo[*g*]chromene-5,10-dione (4b)



Compound **4b** was isolated after flash chromatography (DCM : *n*-pentane 7:3) as yellow solid (93 mg, 94%). **Molecular formula**: $C_{24}H_{15}NO_5$. **Molecular mass**: 397.38 g mol^{-1}. **R_f =** 0.39 (DCM : *n*-pentane 7:3). **Mp**: 175-177 °C. **HPLC**: IC, 7:3 *n*-heptane : EtOH , 0.7 ml/min, λ = 254 nm, τ_{minor} = 9.4 min, τ_{major} = 7.4 min. **OR**: $[\alpha]_D^{21}$ = –19.9 (c = 0.5, $CHCl_3$, 96% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 8.15 (t, *J* = 6.4 Hz, 2 H, Ar-H), 8.06 (d, *J* = 8.2 Hz, 1 H, Ar-H), 7.89 (dd, *J* = 25.3, 8.0 Hz, 2 H, Ar-H), 7.91 (d, *J* = 8.2 Hz, 1 H, Ar-H), 7.87 (d, *J* = 7.9 Hz, 1 H, Ar-H), 7.59 (d, *J* = 6.8 Hz, 1 H, Ar-H), 7.57 – 7.44 (m, 3 H, Ar-H), 5.50 (d, *J* = 4.0 Hz, 1H, ArC=C*H*), 4.94 (dd, *J* = 11.7, 5.7 Hz, 1 H, C*H*H), 4.73 (dd, *J* = 11.7, 3.6 Hz, 1 H, CH*H*), 4.46 (m, 1 H, CH_2CH) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 184.0 (C=O), 177.4 (C=O), 153.2 (C_q), 152.1 (C_q), 134.5 (Ar-C), 134.0 (Ar-C), 133.6 (C_q), 131.6 (C_q), 130.9 (C_q), 130.8 (C_q), 130.6 (Ar-C), 130.4 (C_q), 128.4 (Ar-C), 127.7 (Ar-C), 127.1 (Ar-C), 126.8 (Ar-C), 126.4 (Ar-C), 126.3 (Ar-C), 125.0 (2 C, Ar-C), 117.0 (C_q), 102.5 (ArC=*C*H), 78.8 (CH_2NO_2), 31.0 (CH_2CH) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3070, 2326, 2100, 1742, 1699, 1672, 1627, 1592, 1550, 1509, 1435, 1383, 1335, 1304, 1243, 1190, 1125, 1060, 1036, 997, 954, 903, 860, 771, 718 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 420.09 [M+Na]$^+$, 817.18 [2xM+Na]$^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for [M+Na]$^+$ = 420.0842; found: 420.0854.

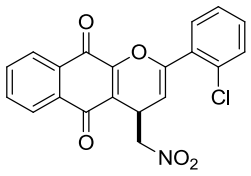
(*R*)-2-(Naphthalen-2-yl)-4-(nitromethyl)-4*H*-benzo[*g*]chromene-5,10-dione (4c)



Compound **4c** was isolated after flash chromatography (DCM : *n*-pentane 7:3) as yellow solid (87 mg, 88%). **Molecular formula**: $C_{24}H_{15}NO_5$. **Molecular mass**: 397.38 g mol^{-1}. **R_f =** 0.50 (DCM : *n*-pentane 7:3). **Mp**: Decomposition at 190-192 °C. **HPLC**: AS, 9:1 *n*-heptan : EtOH , 1.0 ml/min, λ = 254 nm, τ_{minor} = 21.9 min, τ_{major} = 19.3 min. **OR**: $[\alpha]_D^{21}$ = –6.9 (c = 0.5, $CHCl_3$, 97% *ee*). **^{1}H NMR** (600 MHz, DMSO-d_6): δ = 8.26 (s, 1 H, Ar-H), 8.12 – 8.08 (m, 1 H, Ar-H), 8.08 – 8.04 (m, 1 H, Ar-H), 8.01 (m, 2 H, Ar-H), 7.97 – 7.88 (m, 3 H, Ar-H), 7.82 (m, 1 H, Ar-H), 7.57 (m, 2 H, Ar-H), 6.17 (d, *J* = 4.5 Hz, 1 H, ArC=C*H*), 4.87 (m, 2 H, CH_2NO_2), 4.39 (m, 1 H, CH_2CH) ppm. **^{13}C NMR**

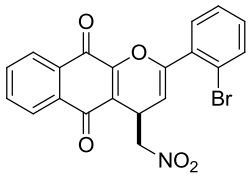
(151 MHz, DMSO-d_6): δ = 183.9 (C=O), 177.9 (C=O), 152.7 (C_q), 149.0 (C_q), 135.3 (Ar-C), 134.7 (Ar-C), 133.6 (C_q), 132.9 (C_q), 131.7 (C_q), 131.1 (C_q), 129.3 (C_q), 129.0 (d, 2 C, Ar-C), 128.1 (Ar-C), 127.6 (Ar-C), 127.4 (Ar-C), 126.6 (Ar-C), 126.4 (Ar-C), 124.1 (Ar-C), 122.4 (Ar-C), 117.9 (C_q), 100.0 (ArC=*C*H), 79.5 (CH_2NO_2), 30.6 (CH_2*C*H) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3063, 2910, 2671, 2324, 2099, 1748, 1664, 1539, 1431, 1380, 1307, 1195, 1017, 904, 865, 810, 709 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 420.09 $[M+Na]^+$. **HR**-**MS** (**ESI**$^+$) *m*/*z* : calcd. for $[M+Na]^+$ = 420.0842; found: 420.0842.

(*R*)-2-(2-Chlorophenyl)-4-(nitromethyl)-4*H*-benzo[*g*]chromene-5,10-dione (4d)



Compound **4d** was isolated after flash chromatography (DCM : *n*-pentane 8:2) as yellow solid (77 mg, 81%). **Molecular formula**: $C_{20}H_{12}ClNO_5$. **Molecular mass**: 381.77 g mol^{-1}. **R_f =** 0.68 (DCM). **Mp**: Decomposition at 175-177 °C. **HPLC**: IC, 7:3 *n*-heptane : EtOH , 1.0 ml/min, λ = 254 nm, τ_{minor} = 5.7 min, τ_{major} = 5.0 min. **OR**: $[\alpha]_D^{21}$ = –20.5 (c = 0.5, $CHCl_3$, 95% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 8.18 – 8.11 (m, 2 H, Ar-H), 7.82 – 7.74 (m, 2 H, Ar-H), 7.51 (m, 1 H, Ar-H), 7.44 (m, 1 H, Ar-H), 7.33 (m, 2 H, Ar-H), 5.56 (d, *J* = 4.3 Hz, 1 H, ArC=C*H*), 4.83 (dd, *J* = 12.0, 6.2 Hz, 1 H, C*H*H), 4.71 (dd, *J* = 12.0, 3.9 Hz, 1 H, CH*H*), 4.45 (dt, *J* = 6.2, 4.1 Hz, 1 H, CH_2C*H*) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 183.9 (C=O), 177.3 (C=O), 153.1 (C_q), 149.5 (C_q), 134.5 (Ar-C), 134.0 (Ar-C), 133.0 (C_q), 131.5 (C_q), 131.5 (C_q), 131.0 (Ar-C), 130.8 (Ar-C), 130.3 (Ar-C), 126.9 (Ar-C), 126.9 (C_q), 126.8 (Ar-C), 126.4 (Ar-C), 116.8 (C_q), 103.2 (ArC=*C*H), 78.6 (CH_2NO_2), 30.5 (CH_2*C*H) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3925, 3787, 3697, 3333, 3072, 2918, 2646, 2312, 2180, 2052, 1819, 1651, 1554, 1443, 1381, 1313, 1185, 1053, 998, 715 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 404.03 $[M+Na, {}^{35}Cl]^+$, 406.03 $[M+Na, {}^{37}Cl]^+$. **HR**-**MS** (**ESI**$^+$) *m*/*z* : calcd. for $[M+Na, {}^{35}Cl]^+$ = 404.0296; found: 404.0296.

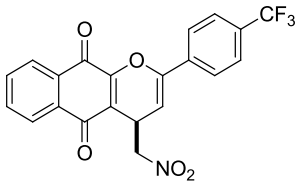
(*R*)-2-(2-Bromophenyl)-4-(nitromethyl)-4*H*-benzo[*g*]chromene-5,10-dione (4e)



Compound **4e** was isolated after flash chromatography (DCM : *n*-pentane 8:2) as yellow solid (88 mg, 83%). **Molecular formula**: $C_{20}H_{12}BrNO_5$. **Molecular mass**: 426.22 g mol^{-1}. **R_f =** 0.69 (DCM). **Mp**: 175-177 °C. **HPLC**: IC, 7:3 *n*-heptane : EtOH , 1.0 ml/min, λ = 254 nm, τ_{minor} = 5.8 min, τ_{major} = 5.0 min. **OR**: $[\alpha]_D^{21}$ = –19.4 (c = 0.5, $CHCl_3$, 96% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 8.19 – 8.11 (m, 2 H, Ar-H), 7.82 – 7.74 (m, 2 H, Ar-H), 7.63 (m, 1 H, Ar-H), 7.45 (m, 1 H, Ar-H), 7.36 (m, 1 H, Ar-H), 7.28 (m, 1 H, Ar-H), 5.48 (d, *J* = 4.3 Hz, 1 H, ArC=C*H*), 4.83 (dd, *J* = 12.0, 6.3 Hz, 1 H, C*H*H), 4.72 (dd, *J* = 12.0, 3.9 Hz, 1 H, CH*H*), 4.45 (m, 1 H, CH_2C*H*) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 183.9 (C=O), 177.3 (C=O), 153.1 (C_q), 151.0 (C_q), 134.5 (Ar-C), 134.0 (Ar-C), 133.7 (C_q), 133.5 (Ar-C), 131.6 (Ar-C), 131.2 (Ar-C), 131.2 (Ar-C), 130.8 (C_q), 127.5 (Ar-C), 126.8 (Ar-C), 126.4 (Ar-C), 122.5 (C_q), 116.9 (C_q), 103.0 (ArC=*C*H), 78.5 (CH_2NO_2), 30.5 (CH_2*C*H) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3070, 2923, 2314, 2199, 2076, 1823, 1654, 1556, 1439, 1381, 1311, 1190, 1003,

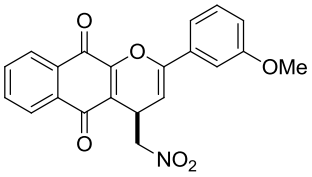
714 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 447.98 [M+Na, ^{79}Br]$^+$, 449.98 [M+Na, ^{81}Br]$^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for [M+Na, ^{79}Br]$^+$ = 447.9791; found: 449.9774.

(*R*)-4-(Nitromethyl)-2-(4-(trifluoromethyl)phenyl)-4*H*-benzo[*g*]chromene-5,10-dione (4f)



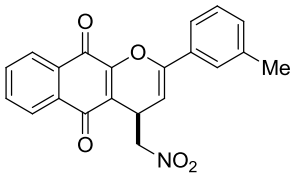
Compound **4f** was isolated after flash chromatography (DCM : *n*-pentane 7:3) as yellow solid (23 mg, 22%). **Molecular formula**: $C_{21}H_{12}F_3NO_5$. **Molecular mass**: 415.32 g mol^{-1}. **R$_f$ =** 0.50 (DCM : *n*-pentane 7:3). **Mp**: Decomposition at 185-187 °C. **HPLC**: IC, 7:3 *n*-heptane : EtOH , 1.0 ml/min, λ = 254 nm, τ_{minor} = 4.4 min, τ_{major} = 3.7 min. **OR**: $[\alpha]_D^{21}$ = –12.9 (c = 0.3, $CHCl_3$, 96% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 8.21 – 8.07 (m, 2 H, Ar-H), 7.88 – 7.73 (m, 4 H, Ar-H), 7.67 (m, 2 H, Ar-H), 5.79 (d, *J* = 4.4 Hz, 1 H, ArC=C*H*), 4.76 (m, 2 H, CH_2NO_2), 4.43 (m, 1 H, CH_2CH) ppm. **^{13}C NMR** (101 MHz, $CDCl_3$): δ = 183.8 (C=O), 177.3 (C=O), 152.7 (C_q), 149.4 (C_q), 134.8 (C_q), 134.7 (Ar-C), 134.1 (Ar-C), 131.5 (C_q), 130.7 (C_q), 126.8 (Ar-C), 126.5 (Ar-C), 125.7 (q, *J* = 3.6, 2 C, Ar-C), 125.3 (2 C, Ar-C), 123.8 (q, *J* = 272.5, CF_3), 122.9 (C_q) 116.9 (C_q), 99.1 (ArC=*C*H), 78.4 (NO_2CH_2), 30.4 (CH_2CH) ppm. **^{19}F {^{1}H} NMR** (376 MHz, $CDCl_3$): δ = – 62.87 (s) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3061, 2916, 2322, 2100, 1670, 1596, 1540, 1430, 1382, 1322, 1246, 1198, 1114, 1069, 1008, 944, 906, 873, 843, 784, 710 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 438.06 [M+Na]$^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for [M+Na]$^+$ = 438.0560; found: 438.0570.

(*R*)-2-(3-Methoxyphenyl)-4-(nitromethyl)-4*H*-benzo[*g*]chromene-5,10-dione (4g)



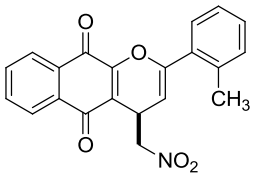
Compound **4g** was isolated after flash chromatography (DCM : *n*-pentane 7:3) as yellow solid (73 mg, 77%). **Molecular formula**: $C_{21}H_{15}NO_6$. **Molecular mass**: 377.35 g mol^{-1}. **R$_f$ =** 0.36 (DCM : *n*-pentane 7:3). **Mp**: Decomposition at 165-167 °C. **HPLC**: AS, 7:3 *n*-heptane : EtOH , 0.7 ml/min, λ = 254 nm, τ_{minor} = 15.9 min, τ_{major} = 14.0 min. **OR**: $[\alpha]_D^{21}$ = –13.6 (c = 0.5, $CHCl_3$, 96% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 8.20 – 8.10 (m, 2 H, Ar-H), 7.83 – 7.74 (m, 2 H, Ar-H), 7.35 – 7.27 (m, 2 H, Ar-H), 7.25 – 7.23 (m, 1 H, Ar-H), 6.98 – 6.92 (m, 1 H, Ar-H), 5.68 (d, *J* = 4.4 Hz, 1 H, ArC=C*H*), 4.79 – 4.70 (m, 2 H, CH_2NO_2), 4.43 (m, 1 H, CH_2CH), 3.86 (s, 3 H, OCH_3) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 183.9 (C=O), 177.4 (C=O), 159.8 (C_q), 152.9 (C_q), 150.5 (C_q), 134.5 (Ar-C), 134.0 (Ar-C), 132.9 (C_q), 131.6 (C_q), 130.8 (C_q), 129.7 (Ar-C), 126.7 (Ar-C), 126.4 (Ar-C), 117.5 (Ar-C), 116.9 (C_q), 115.6 (Ar-C), 110.5 (Ar-C), 97.4 (ArC=*C*H), 78.6 (CH_2NO_2), 55.4 (OCH_3), 30.4 (CH_2CH) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 2940, 2842, 1675, 1649, 1624, 1582, 1543, 1492, 1432, 1379, 1330, 1288, 1247, 1207, 1107, 1046, 1006, 971, 914, 886, 785, 719, 691, 662 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 400.08 [M+Na]$^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for [M+Na]$^+$ = 400.0792; found: 400.0801.

(*R*)-4-(Nitromethyl)-2-(*m*-tolyl)-4*H*-benzo[*g*]chromene-5,10-dione (4h)



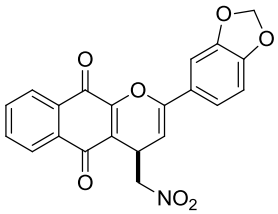
Compound **4h** was isolated after flash chromatography (DCM : *n*-pentane 7:3) as yellow solid (82 mg, 91%). **Molecular formula**: $C_{21}H_{15}NO_5$. **Molecular mass**: 361.35 g mol^{-1}. **R_f =** 0.44 (DCM : *n*-pentane 7:3). **Mp**: Decomposition at 177-179 °C. **HPLC**: AS, 7:3 *n*-heptane : EtOH , 0.7 ml/min, λ = 254 nm, τ_{minor} = 12.2 min, τ_{major} = 10.7 min. **OR**: $[\alpha]_D^{21}$ = –15.9 (c = 0.5, $CHCl_3$, 96% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 8.20 – 8.08 (m, 2 H, Ar-H), 7.81 – 7.73 (m, 2 H, Ar-H), 7.50 (m, 2 H, Ar-H), 7.29 (m, 1 H, Ar-H), 7.21 (m, 1 H, Ar-H), 5.66 (d, *J* = 4.4 Hz, 1 H, ArC=C*H*), 4.78 – 4.69 (m, 2 H, CH_2NO_2), 4.42 (m, 1 H, CH_2CH), 2.39 (s, 3 H, CH_3) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 183.9 (C=O), 177.5 (C=O), 153.0 (C_q), 150.8 (C_q), 138.4 (C_q), 134.5 (Ar-C), 134.0 (Ar-C), 131.6 (C_q), 131.5 (C_q), 130.8 (C_q), 130.7 (Ar-C), 128.5 (Ar-C), 126.7 (Ar-C), 126.4 (Ar-C), 125.6 (Ar-C), 122.2 (Ar-C), 116.9 (C_q), 96.9 (ArC=*C*H), 78.7 (CH_2NO_2), 30.4 (CH_2CH), 21.5 (CH_3) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3057, 2916, 2320, 2096, 1665, 1537, 1433, 1383, 1323, 1245, 1196, 1108, 1010, 945, 875, 783, 705 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 384.09 $[M+Na]^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for $[M+Na]^+$ = 384.0842; found: 384.0852.

(*R*)-4-(Nitromethyl)-2-(*o*-tolyl)-4*H*-benzo[*g*]chromene-5,10-dione (4i)



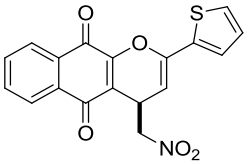
Compound **4i** was isolated after flash chromatography (DCM : *n*-pentane 8:2) as yellow solid (84 mg, 93%). **Molecular formula**: $C_{21}H_{15}NO_5$. **Molecular mass**: 361.35 g mol^{-1}. **R_f =** 0.74 (DCM). **Mp**: 145-147 °C. **HPLC**: AS, 7:3 *n*-heptane : EtOH, 1.0 ml/min, λ = 254 nm, τ_{minor} = 9.6 min, τ_{major} = 7.0 min. **OR**: $[\alpha]_D^{21}$ = –22.0 (c = 0.5, $CHCl_3$, 95% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 8.14 (m, 2 H, Ar-H), 7.78 (m, 2 H, Ar-H), 7.33 (m, 2 H, Ar-H), 7.22 (m, 2 H, Ar-H), 5.30 (d, *J* = 4.4 Hz, 1 H, ArC=C*H*), 4.85 (dd, *J* = 11.7, 5.8 Hz, 1 H, C*H*H), 4.69 (dd, *J* = 11.7, 3.9 Hz, 1 H, CH*H*), 4.41 (m, 1 H, CH_2CH), 2.41 (s, 3 H, CH_3) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 184.0 (C=O), 177.5 (C=O), 153.0 (C_q), 153.0 (C_q), 137.0 (C_q), 134.5 (Ar-C), 133.9 (Ar-C), 132.4 (C_q), 131.6 (C_q), 130.8 (C_q), 130.7 (Ar-C), 129.9 (Ar-C), 129.3 (Ar-C), 126.8 (Ar-C), 126.4 (Ar-C), 125.9 (Ar-C), 116.8 (C_q), 101.1 (ArC=*C*H), 78.9 (CH_2NO_2), 30.7 (CH_2CH), 20.2 (CH_3) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3068, 3023, 2961, 2107, 1901, 1676, 1628, 1590, 1544, 1489, 1456, 1427, 1379, 1330, 1233, 1200, 1104, 1039, 995, 907, 879, 821, 797, 765, 718, 663 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 384.08 $[M+Na]^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for $[M+Na]^+$ = 384.0842; found: 384.0845.

(*R*)-2-(Benzo[*d*][1,3]dioxol-5-yl)-4-(nitromethyl)-4*H*-benzo[*g*]chromene-5,10-dione (4j)



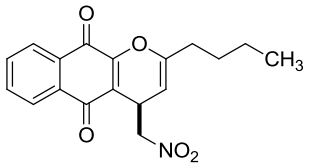
Compound **4j** was isolated after flash chromatography (DCM : *n*-pentane 8:2) as yellow solid (89 mg, 91%). **Molecular formula**: $C_{21}H_{13}NO_7$. **Molecular mass**: 391.33 g mol^{-1}. **R_f =** 0.56 (DCM). **Mp**: Decomposition at 173-175 °C. **HPLC**: IC, 7:3 *n*-heptane : EtOH , 0.7 ml/min, λ = 254 nm, τ_{minor} = 14.0 min, τ_{major} = 12.6 min. **OR**: $[\alpha]_D^{21}$ = –12.4 (c = 0.5, $CHCl_3$, 92% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 8.13 (m, 2 H, Ar-H), 7.77 (m, 2 H, Ar-H), 7.27 – 7.21 (m, 1 H, Ar-H), 7.13 (m, 1 H, Ar-H), 6.82 (d, *J* = 8.2 Hz, 1 H, Ar-H), 6.00 (s, 2 H, OCH_2), 5.51 (d, *J* = 4.4 Hz, 1 H, ArC=C*H*), 4.72 (m, 2 H, CH_2NO_2), 4.39 (m, 1 H, CH_2CH) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 183.9 (C=O), 177.5 (C=O), 152.8 (C_q), 150.3 (C_q), 149.0 (C_q), 148.0 (C_q), 134.5 (Ar-C), 133.9 (Ar-C), 131.6 (C_q), 130.8 (C_q), 126.7 (Ar-C), 126.4 (Ar-C), 125.7 (C_q), 119.6 (Ar-C), 117.0 (C_q), 108.3 (Ar-C), 105.5 (Ar-C), 101.5 (OCH_2), 95.7 (ArC=*C*H), 78.7 (CH_2NO_2), 30.4 (CH_2CH) ppm. **IR** (ATR): $\tilde{\nu}$ = 3064, 2905, 2310, 2061, 1862, 1659, 1541, 1493, 1440, 1331, 1246, 1116, 1021, 930, 804, 713 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 414.06 [M+Na]$^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for [M+Na]$^+$ = 414.0584; found: 414.0587.

(*R*)-4-(Nitromethyl)-2-(thiophen-2-yl)-4*H*-benzo[*g*]chromene-5,10-dione (4l)



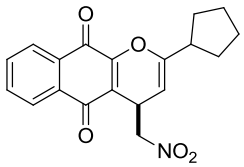
Compound **4l** was isolated after flash chromatography (DCM : *n*-pentane 8:2) as yellow solid (20 mg, 23%). **Molecular formula**: $C_{18}H_{11}NO_5S$. **Molecular mass**: 353.35 g mol^{-1}. **R_f =** 0.58 (DCM : *n*-pentane 4:1). **Mp**: 85-87 °C. **HPLC**: AS, 7:3 *n*-heptane : EtOH, 0.7 ml/min, λ = 254 nm, τ_{minor} = 16.2 min, τ_{major} = 14.1 min. **OR**: $[\alpha]_D^{21}$ = –12.5 (c = 0.5, $CHCl_3$, 99% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 8.17 – 8.09 (m, 2 H, Ar-H), 7.76 (m, 2 H, Ar-H), 7.45 (dd, *J* = 3.6, 1.0 Hz, 1 H, CH), 7.33 (dd, *J* = 5.0, 1.1 Hz, 1 H, CH), 7.05 (dd, *J* = 5.0, 3.7 Hz, 1 H, CH), 5.56 (d, *J* = 4.6 Hz, 1 H, ArC=C*H*), 4.78 – 4.67 (m, 2 H, NO_2CH_2), 4.39 (m, 1 H, CH_2CH) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 183.9 (C=O), 177.2 (C=O), 152.7 (C_q), 146.5 (C_q), 134.6 (Ar-C), 134.1 (Ar-C), 131.5 (C_q), 130.7 (C_q), 127.7 (CH), 127.5 (C_q), 126.8 (2 C, Ar-C), 126.5 (CH), 125.9 (CH), 117.0 (C_q), 96.0 (ArC=*C*H), 78.4 (CH_2NO_2), 30.2 (CH_2CH) ppm. **IR** (ATR): $\tilde{\nu}$ = 3103, 2918, 2853, 2223, 2101, 1993, 1943, 1745, 1668, 1592, 1542, 1430, 1381, 1334, 1305, 1235, 1194, 1109, 1043, 1005, 946, 900, 850, 797, 715 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 376.03 [M+Na]$^+$, 729,06 [2M+Na]$^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for [M+Na]$^+$ = 376.0250; found: 376.0255.

(*R*)-2-Butyl-4-(nitromethyl)-4*H*-benzo[*g*]chromene-5,10-dione (4m)



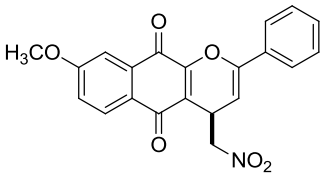
Compound **4m** was isolated after flash chromatography (DCM : *n*-pentane 7:3) as yellow solid (41 mg, 50%). **Molecular formula**: $C_{18}H_{17}NO_5$. **Molecular mass**: 327.33 g mol^{-1}. **R_f =** 0.38 (DCM : *n*-pentane 7:3). **Mp**: Decomposition at 82-84 °C. **HPLC**: IC, 7:3 *n*-heptane : ^{i}PrOH , 1.0 ml/min, λ = 254 nm, τ_{minor} = 6.8 min, τ_{major} = 5.1 min. **OR**: $[\alpha]_D^{21}$ = –21.5 (c = 0.3 , $CHCl_3$, 95% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 8.16 – 8.08 (m, 2 H, Ar-H), 7.80 – 7.72 (m, 2 H, Ar-H), 4.94 (d, *J* = 4.0 Hz, 1 H, *n*-but-C=C*H*), 4.70 (dd, *J* = 11.8, 6.2 Hz, 1 H, C*H*H), 4.60 (dd, *J* = 11.8, 4.0 Hz, 1 H, CH*H*), 4.25 – 4.19 (m, 1 H, CH_2CH), 2.29 (t, *J* = 7.6 Hz, 2 H, CH_2), 1.60 – 1.52 (m, 2 H, CH_2), 1.41 – 1.32 (m, 2H, C H_2), 0.93 (t, *J* = 7.4 Hz, 3 H, CH_3) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 184.1 (C=O), 177.7 (C=O), 154.1 (C_q), 153.1 (C_q), 134.4 (Ar-C), 133.9 (Ar-C), 131.6 (C_q), 130.8 (C_q), 126.7 (Ar-C), 126.3 (Ar-C), 116.9 (C_q), 97.0 (*n*-but-C=*C*H), 78.9 (CH_2NO_2), 32.3 (CH_2), 30.2 (CH_2CH), 28.4 (CH_3), 21.9 (CH_2), 13.7 (CH_2) ppm. **IR** (ATR): $\tilde{\nu}$ = 3082, 2956, 2868, 2332, 2112, 1674, 1647, 1624, 1590, 1553, 1439, 1382, 1331, 1304, 1254, 1199, 1107, 1035, 1001, 949, 905, 867, 799, 719 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 350.10 [M+Na]$^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for [M+Na]$^+$ = 350.0999; found: 350.1006.

(*R*)-2-Cyclopentyl-4-(nitromethyl)-4*H*-benzo[*g*]chromene-5,10-dione (4n)



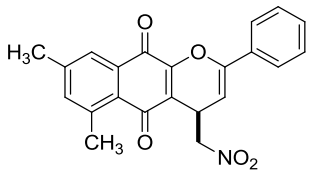
Compound **4n** was isolated after flash chromatography (DCM : *n*-pentane 7:3) as yellow solid (16 mg, 19%). **Molecular formula**: $C_{19}H_{17}NO_5$. **Molecular mass**: 339.34 g mol^{-1}. **R_f =** 0.54 (DCM : *n*-pentane 7:3). **Mp**: 90-92 °C. **HPLC**: IC, 7:3 *n*-heptane : ^{i}PrOH , 1.0 ml/min, λ = 254 nm, τ_{minor} = 7.3 min, τ_{major} = 5.2 min. **OR**: $[\alpha]_D^{21}$ = –17.7 (c = 0.3 , $CHCl_3$, 97% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 8.16 – 8.03 (m, 2 H, Ar-H), 7.79 – 7.68 (m, 2 H, Ar-H), 4.94 (d, *J* = 4.0 Hz, 1 H, *c*-pent-C=C*H*), 4.67 (dd, *J* = 11.7, 6.1 Hz, 1 H, CH*H*), 4.58 (dd, *J* = 11.7, 4.0 Hz, 1 H, C*H*H), 4.20 (m, 1 H, CH_2CH), 2.74 – 2.61 (m, 1 H, CH), 1.96 – 1.82 (m, 2 H, CH_2), 1.74 (m, 2 H, CH_2), 1.60 (m, 4 H, CH_2) ppm. **^{13}C NMR** (101 MHz, $CDCl_3$): δ = 184.1 (C=O), 177.7 (C=O), 157.1 (C_q), 153.3 (C_q), 134.4 (Ar-C), 133.8 (Ar-C), 131.6 (C_q), 130.8 (C_q), 126.6 (Ar-C), 126.3 (Ar-C), 116.9 (C_q), 95.3 (*c*-pent-C=*C*H), 79.0 (CH_2NO_2), 42.5 (CH), 30.4 (CH_2), 30.3 (CH_2), 30.1 (CH_2CH), 25.2 (2 C, CH_2) ppm. **IR** (ATR): $\tilde{\nu}$ = 3459, 2955, 2870, 2321, 2208, 2084, 1993, 1741, 1678, 1623, 1544, 1439, 1372, 1204, 1037, 996, 897, 801, 721 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 362.10 [M+Na]$^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for [M+Na]$^+$ = 362.0999; found: 362.1009.

(*R*)-8-Methoxy-4-(nitromethyl)-2-phenyl-4*H*-benzo[*g*]chromene-5,10-dione (4o)



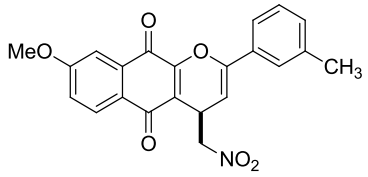
Compound **4o** was isolated after flash chromatography (DCM : *n*-pentane 8:2) as yellow solid (79 mg, 84%). **Molecular formula**: $C_{21}H_{15}NO_6$. **Molecular mass**: 377.35 g mol^{-1}. **R_f =** 0.59 (DCM). **Mp**: Decomposition at 170-172 °C. **HPLC**: IB, 7:3 *n*-heptane : EtOH, 1.0 ml/min, λ = 254 nm, τ_{minor} = 13.6 min, τ_{major} = 12.5 min. **OR**: $[\alpha]_D^{21}$ = –25.0 (c = 0.5, $CHCl_3$, 97% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 8.06 (d, *J* = 8.6 Hz, 1 H, Ar-H), 7.70 (m, 2 H, Ar-H), 7.59 (d, *J* = 2.6 Hz, 1 H, Ar-H), 7.45 – 7.38 (m, 3 H, Ar-H), 7.23 (dd, *J* = 8.6, 2.6 Hz, 1 H, Ar-H), 5.67 (d, *J* = 4.4 Hz, 1 H, ArC=C*H*), 4.77 – 4.72 (m, 2 H, CH_2NO_2), 4.45 – 4.39 (m, 1 H, CH_2CH), 3.96 (s, 3 H, OCH_3) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 183.1 (C=O), 177.6 (C=O), 164.2 (C_q), 152.7 (C_q), 150.6 (C_q), 132.7 (C_q), 131.6 (C_q), 129.8 (Ar-C), 128.8 (Ar-C), 128.6 (2 C, Ar-C), 125.0 (2 C, Ar-C), 125.0 (C_q), 120.7 (Ar-C), 116.8 (C_q), 110.4 (Ar-C), 97.0 (ArC=*C*H), 78.8 (CH_2NO_2), 56.0 (OCH_3), 30.3 (CH_2CH) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3055, 2989, 2942, 2321, 2208, 2080, 1814, 1677, 1638, 1589, 1538, 1492, 1448, 1379, 1316, 1242, 1189, 1083, 1014, 913, 847, 751, 685 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 400.08 $[M+Na]^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for $[M+Na]^+$ = 400.0792; found: 400.0796.

(*R*)-6,8-Dimethyl-4-(nitromethyl)-2-phenyl-4*H*-benzo[*g*]chromene-5,10-dione (4p)



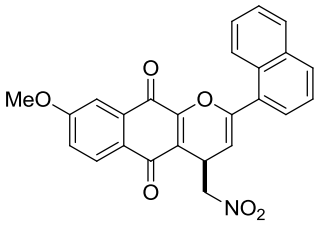
Compound **4p** was isolated after flash chromatography (DCM : *n*-pentane 8:2) as yellow solid (81 mg, 86%). **Molecular formula**: $C_{22}H_{17}NO_5$. **Molecular mass**: 375.37 g mol^{-1}. **R_f =** 0.68 (DCM). **Mp**: Decomposition at 165-167 °C. **HPLC**: IC, 7:3 *n*-heptane : EtOH, 1.0 ml/min, λ = 254 nm, τ_{minor} = 5.9 min, τ_{major} = 5.2 min. **OR**: $[\alpha]_D^{21}$ = –18.7 (c = 0.5, $CHCl_3$, 96% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 7.88 (s, 1 H, Ar-H), 7.72 – 7.67 (m, 2 H, Ar-H), 7.43 – 7.39 (m, 3 H, Ar-H), 7.35 (m, 1 H, Ar-H), 5.66 (d, *J* = 4.4 Hz, 1 H, ArC=C*H*), 4.71 (m, 2 H, CH_2NO_2), 4.41 (m, 1 H, CH_2CH), 2.72 (s, 3 H, CH_3), 2.45 (s, 3 H, CH_3) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 185.5 (C=O), 178.2 (C=O), 151.7 (C_q), 150.5 (C_q), 143.9 (C_q), 141.6 (C_q), 139.3 (Ar-C), 132.2 (C_q), 131.7 (C_q), 129.7 (Ar-C), 128.6 (2 C, Ar-C), 126.8 (C_q), 126.2 (Ar-C), 125.0 (2 C, Ar-C), 117.9 (C_q), 97.0 (ArC=*C*H), 78.9 (CH_2NO_2), 30.4 (CH_2CH), 22.8 (CH_3), 21.5 (CH_3) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3059, 2973, 2922, 2740, 2327, 2199, 2083, 1894, 1672, 1631, 1542, 1434, 1374, 1334, 1273, 1230, 1188, 1140, 1114, 1072, 1031, 998, 966, 902, 867, 815, 755, 685 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 398.10 $[M+Na]^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for $[M+Na]^+$ = 398.0999; found: 398.1007.

(*R*)-8-Methoxy-4-(nitromethyl)-2-(*m*-tolyl)-4*H*-benzo[*g*]chromene-5,10-dione (4q)



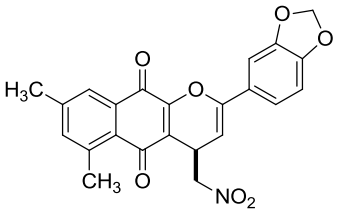
Compound **4q** was isolated after flash chromatography (DCM : *n*-pentane 8:2) as yellow solid (68 mg, 70%). **Molecular formula**: $C_{22}H_{17}NO_6$. **Molecular mass**: 391.37 g mol^{-1}. **R_f =** 0.63 (DCM). **Mp**: Decomposition at 170-172 °C. **HPLC**: IB, 7:3 *n*-heptane : EtOH, 1.0 ml/min, λ = 254 nm, τ_{minor} = 12.2 min, τ_{major} = 10.8 min. **OR**: $[\alpha]_D^{21}$ = –21.5 (c = 0.5, $CHCl_3$, 97% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 8.07 (m, 1 H, Ar-H), 7.59 (m, 1 H, Ar-H), 7.50 (m, 2 H, Ar-H), 7.30 (t, *J* = 7.9 Hz, 1 H, Ar-H), 7.25 – 7.20 (m, 2 H, Ar-H), 5.65 (d, *J* = 4.4 Hz, 1 H, ArC=C*H*), 4.73 (d, *J* = 5.3 Hz, 2 H, NO_2CH_2), 4.44 – 4.39 (m, 1 H, CH_2CH), 3.97 (s, 3 H, OCH_3), 2.40 (s, 3 H, CH_3) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 183.1 (C=O), 177.7 (C=O), 164.2 (C_q), 152.8 (C_q), 150.8 (C_q), 138.3 (C_q), 132.7 (C_q), 131.6 (C_q), 130.6 (Ar-C), 128.8 (Ar-C), 128.5 (Ar-C), 125.6 (Ar-C), 125.0 (C_q), 122.2 (Ar-C), 120.7 (Ar-C), 116.8 (C_q), 110.4 (Ar-C), 96.8 (ArC=*C*H), 78.9 (CH_2NO_2), 56.0 (OCH_3), 30.4 (CH_2CH), 21.5 (CH_3) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 2920, 2834, 2321, 2088, 1909, 1598, 1538, 1445, 1376, 1255, 1192, 1101, 1012, 900, 830, 781, 693 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 414.10 $[M+Na]^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for $[M+Na]^+$ = 414.0948; found: 414.0963.

(*R*)-8-Methoxy-2-(naphthalen-1-yl)-4-(nitromethyl)-4*H*-benzo[*g*]chromene-5,10-dione (4r)



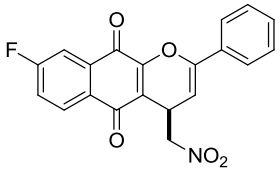
Compound **4r** was isolated after flash chromatography (DCM : *n*-pentane 8:2) as yellow solid (90 mg, 84%). **Molecular formula**: $C_{25}H_{17}NO_6$. **Molecular mass**: 427.41 g mol^{-1}. **R_f =** 0.56 (DCM). **Mp**: 120-122 °C. **HPLC**: IB, 7:3 *n*-heptane : EtOH, 1.0 ml/min, λ = 254 nm, τ_{minor} = 16.9 min, τ_{major} = 15.3 min. **OR**: $[\alpha]_D^{21}$ = –30.7 (c = 0.5, $CHCl_3$, 97% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 8.09 (d, *J* = 8.6 Hz, 1 H, Ar-H), 8.06 (d, *J* = 8.3 Hz, 1 H, Ar-H), 7.91 (d, *J* = 8.2 Hz, 1 H, Ar-H), 7.87 (d, *J* = 7.8 Hz, 1 H, Ar-H), 7.63 – 7.44 (m, 5 H, Ar-H), 7.28 – 7.21 (m, 1 H, Ar-H), 5.48 (d, *J* = 4.1 Hz, 1 H, ArC=C*H*), 4.92 (dd, *J* = 11.7, 5.8 Hz, 1 H, C*H*H), 4.72 (dd, *J* = 11.7, 3.8 Hz, 1 H, CH*H*), 4.48 – 4.42 (m, 1 H, CH_2CH), 3.94 (s, 3 H, OCH_3) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 183.2 (C=O), 177.6 (C=O), 164.2 (C_q), 153.0 (C_q), 152.1 (C_q), 133.6 (C_q), 132.7 (C_q), 130.9 (C_q), 130.5 (Ar-C), 130.5 (C_q), 128.8 (Ar-C), 128.4 (Ar-C), 127.7 (Ar-C), 127.0 (Ar-C), 126.3 (Ar-C), 125.0 (d, 2 C, Ar-C), 125.0 (C_q), 120.8 (Ar-C), 116.9 (C_q), 110.3 (Ar-C), 102.4 (ArC=*C*H), 79.0 (CH_2NO_2), 56.0 (OCH_3), 30.9 (CH_2CH) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3055, 2942, 2311, 2094, 1928, 1739, 1552, 1444, 1316, 1245, 1187, 1008, 853, 778, 693 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 450.10 $[M+Na]^+$, 877,21 $[2M+Na]^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for $[M+Na]^+$ = 450.0948; found: 450.0967.

(*R*)-2-(Benzo[*d*][1,3]dioxol-5-yl)-6,8-dimethyl-4-(nitromethyl)-4*H*-benzo[*g*]chromene-5,10-dione (4s)



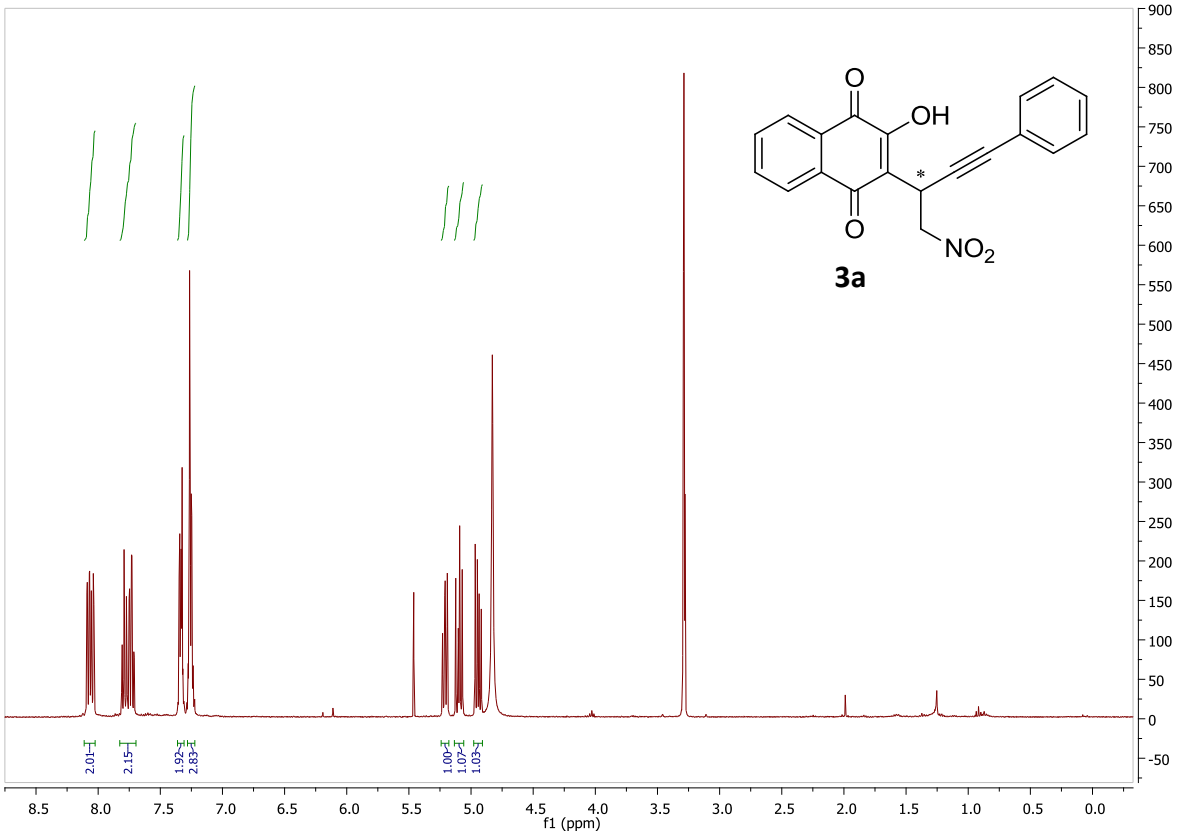
Compound **4s** was isolated after flash chromatography (DCM : *n*-pentane 8:2) as yellow solid (80 mg, 84%). **Molecular formula**: $C_{23}H_{17}NO_7$. **Molecular mass**: 415.32 g mol^{-1}. **R_f =** 0.5 (DCM : *n*-pentane 7:3). **Mp**: Decomposition at 195-197 °C. **HPLC**: IB, 7:3 *n*-heptane : EtOH, 1.0 ml/min, λ = 254 nm, τ_{minor} = 12.1 min, τ_{major} = 10.9 min. **OR**: $[\alpha]_D^{21}$ = –12.4 (c = 0.5, $CHCl_3$, 89% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 7.98 (s, 1 H, Ar-H), 7.45 (s, 1 H, Ar-H), 7.35 (s, 1 H, Ar-H), 7.24 (d, *J* = 1.7 Hz, 1 H, Ar-H), 6.93 (d, *J* = 8.2 Hz, 1 H, Ar-H), 6.10 (s, 2 H, OCH_2), 5.60 (d, *J* = 4.5 Hz, 1 H, ArC=C*H*), 4.83 – 4.74 (m, 2 H, NO_2CH_2), 4.48 (m, 1 H, CH_2CH), 2.82 (s, 3 H, CH_3), 2.55 (s, 3 H, CH_3) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 185.5 (C=O), 178.2 (C=O), 151.7 (C_q), 150.2 (C_q), 148.9 (C_q), 148.0 (C_q), 143.9 (C_q), 141.6 (C_q), 139.3 (Ar-C), 132.2 (C_q), 126.8 (C_q), 126.2 (Ar-C), 125.9 (C_q), 119.6 (Ar-C), 117.9 (C_q), 108.3 (Ar-C), 105.6 (Ar-C), 101.5 (OCH_2), 95.7 (ArC=*C*H), 78.9 (NO_2CH_2), 30.5 (CH_2CH), 22.9 (CH_3), 21.5 (CH_3) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 2917, 2322, 2050, 1955, 1651, 1504, 1352, 1243, 1121, 1024, 923, 820, 746, 687 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 481.20 $[M+Na+K]^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for $[M+Na]^+$ = 442.0900; found: 442.0904.

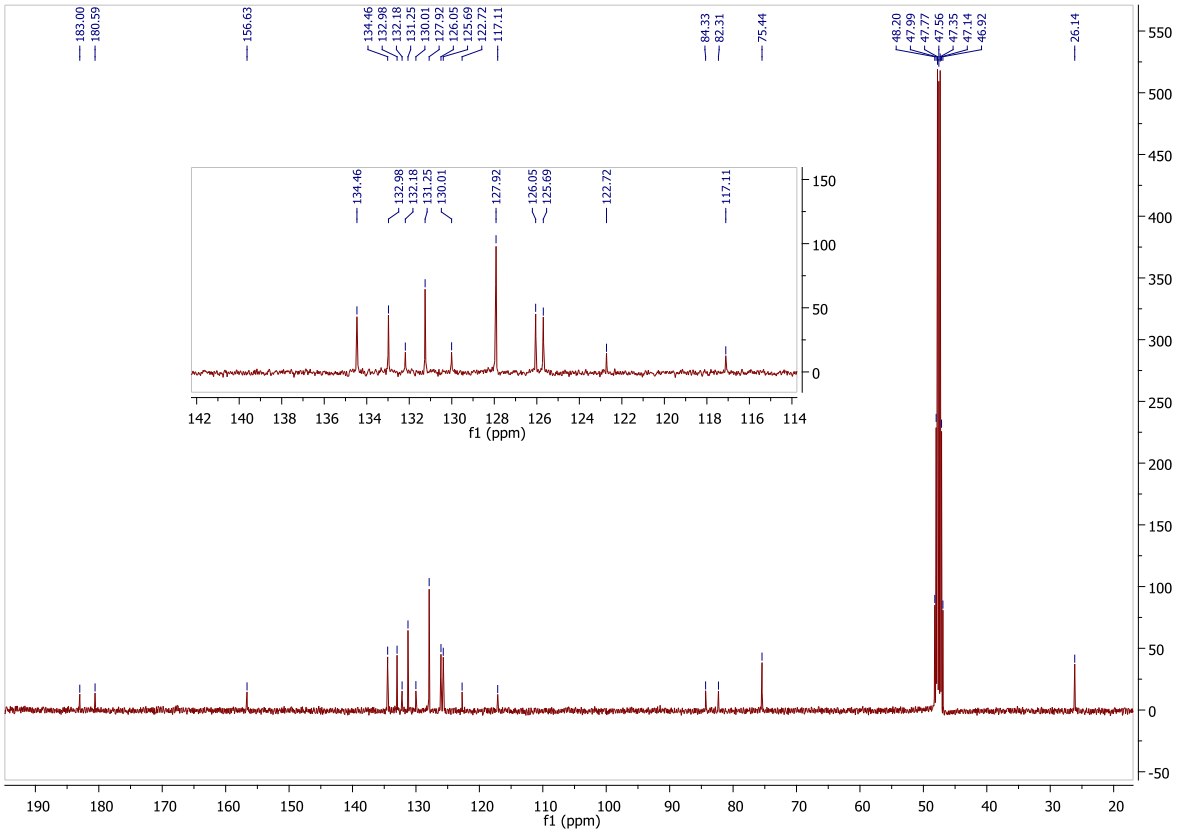
(*R*)-8-Fluoro-4-(nitromethyl)-2-phenyl-4*H*-benzo[*g*]chromene-5,10-dione (4t)

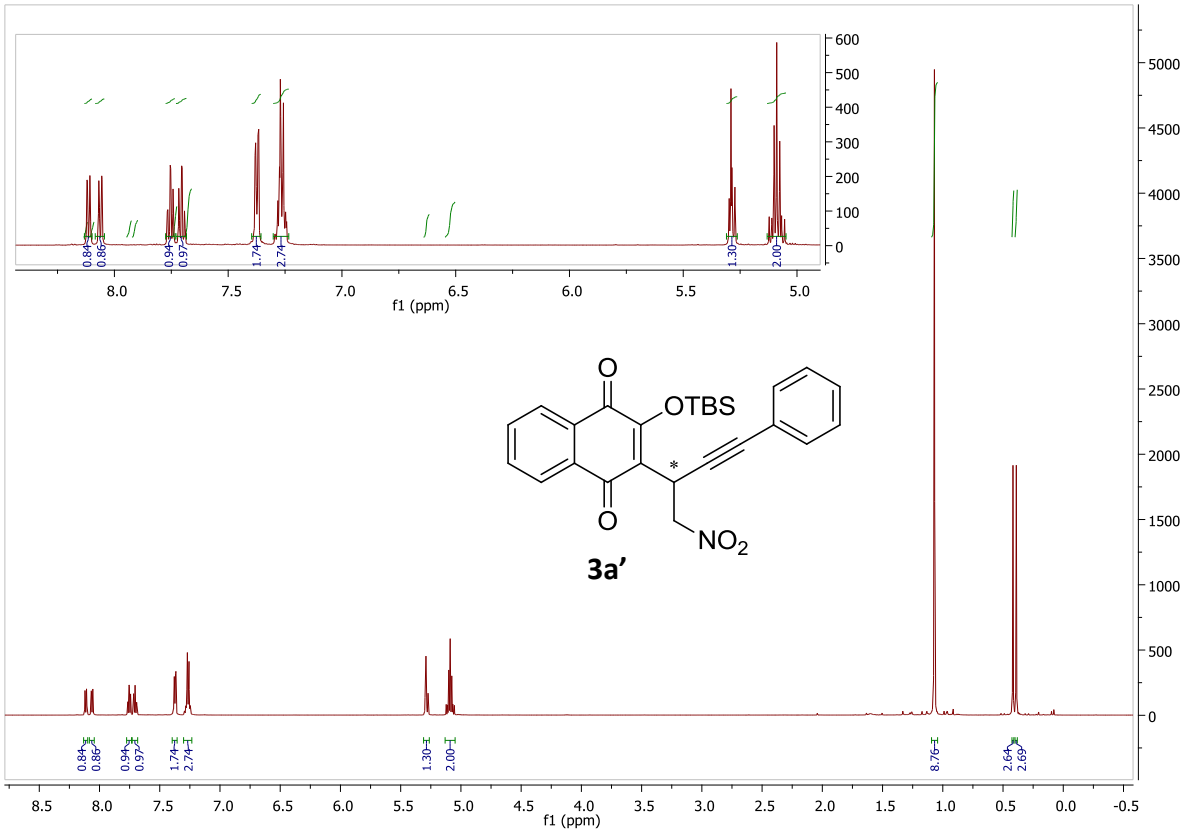


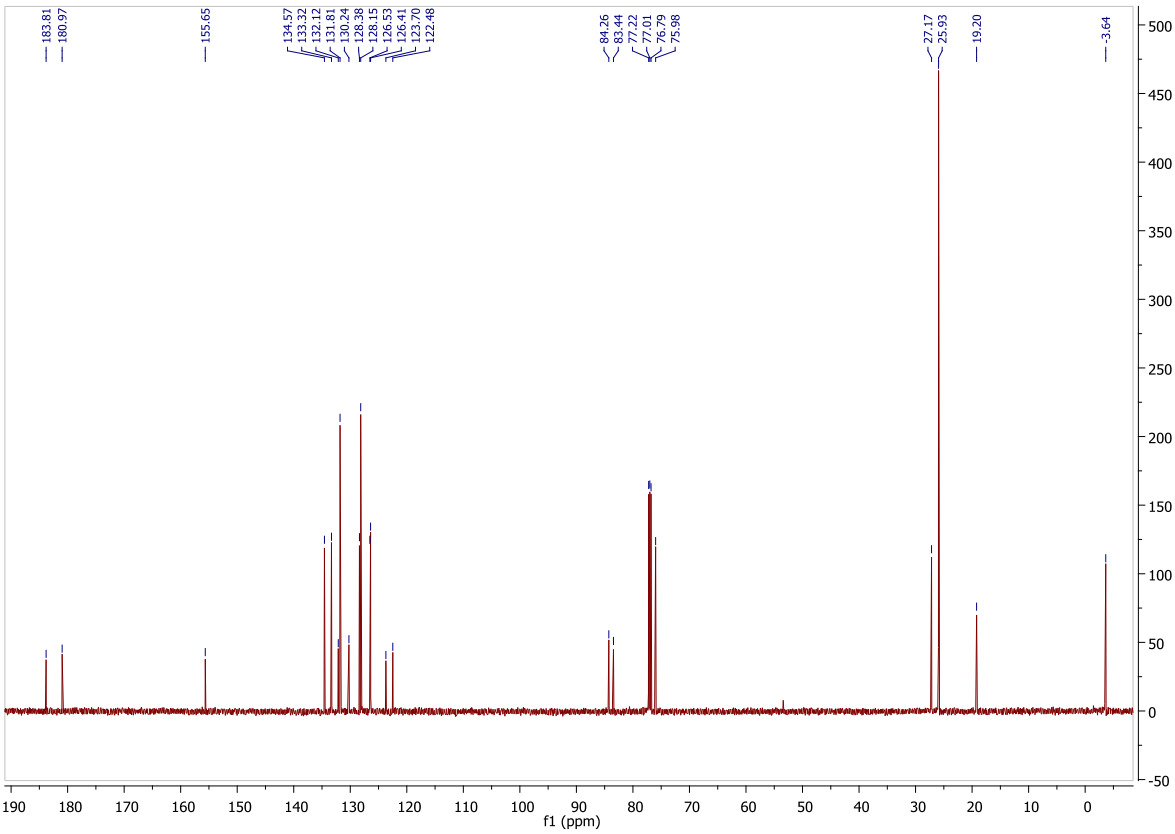
Compound **4t** was isolated after flash chromatography (DCM : *n*-pentane 7:3) as yellow solid (64 mg, 70%). **Molecular formula**: $C_{20}H_{12}FNO_5$. **Molecular mass**: 365.31 g mol^{-1}. **R_f** = 0.4 (DCM : *n*-pentane 7:3). **Mp**: Decomposition at 185-187 °C. **HPLC**: AS, 9:1 *n*-heptane : EtOH, 1.0 ml/min, λ = 254 nm, τ_{minor} = 17.7 min, τ_{major} = 14.4 min. **OR**: $[\alpha]_D^{21}$ = –13.8 (c = 0.5, $CHCl_3$, 97% *ee*). **^{1}H NMR** (600 MHz, $CDCl_3$): δ = 8.18 (m, 1 H, Ar-H), 7.81 (m, 1 H, Ar-H), 7.73 – 7.67 (m, 2 H, Ar-H), 7.49 – 7.39 (m, 4 H, Ar-H), 5.68 (d, *J* = 4.4 Hz, 1 H, ArC=C*H*), 4.78 (dd, *J* = 12.0, 6.4 Hz, 1 H, C*H*H), 4.72 (dd, *J* = 12.0, 4.0 Hz, 1 H, CH*H*), 4.43 (m, 1 H, CH_2CH) ppm. **^{13}C NMR** (151 MHz, $CDCl_3$): δ = 182.7 (C=O), 176.5 (C=O), 167.0 (C_q), 165.3 (C_q), 153.1 (C_q), 150.7 (C_q), 131.4 (C_q), 130.0 (Ar-C), 129.7 (d, J = 8.8 Hz, Ar-C), 128.7 (2 C, Ar-C), 128.1 (C_q), 125.0 (2 C, Ar-C), 121.6 (d, *J* = 22.4 Hz, Ar-H), 117.1 (C_q), 113.6 (d, *J* = 24.0 Hz, Ar-C), 97.0 (ArC=*C*H), 78.7 (NO_2CH_2), 30.4 (CH_2CH) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = –101.42 (ddd, *J* = 8.1, 8.1, 5.2 Hz) ppm. **IR** (ATR): $\tilde{\upsilon}$ = 3037, 2920, 2306, 2089, 1804, 1552, 1438, 1316, 1241, 1176, 1006, 887, 756, 686 cm^{-1}. **MS** (**ESI**$^+$) *m*/*z* = 388.05 $[M+Na]^+$, 753.12 $[2M+Na]^+$. **HR-MS** (**ESI**$^+$) *m*/*z* : calcd. for $[M+Na]^+$ = 388.0592; found: 388.0579.

NMR Spectra and HPLC Chromatograms






O
OTBS
*
O
NO2
3a'
f1 (ppm)


183.81
180.97
155.65
134.57
133.32
132.12
131.81
130.24
128.38
128.15
126.53
126.41
123.70
122.48
84.26
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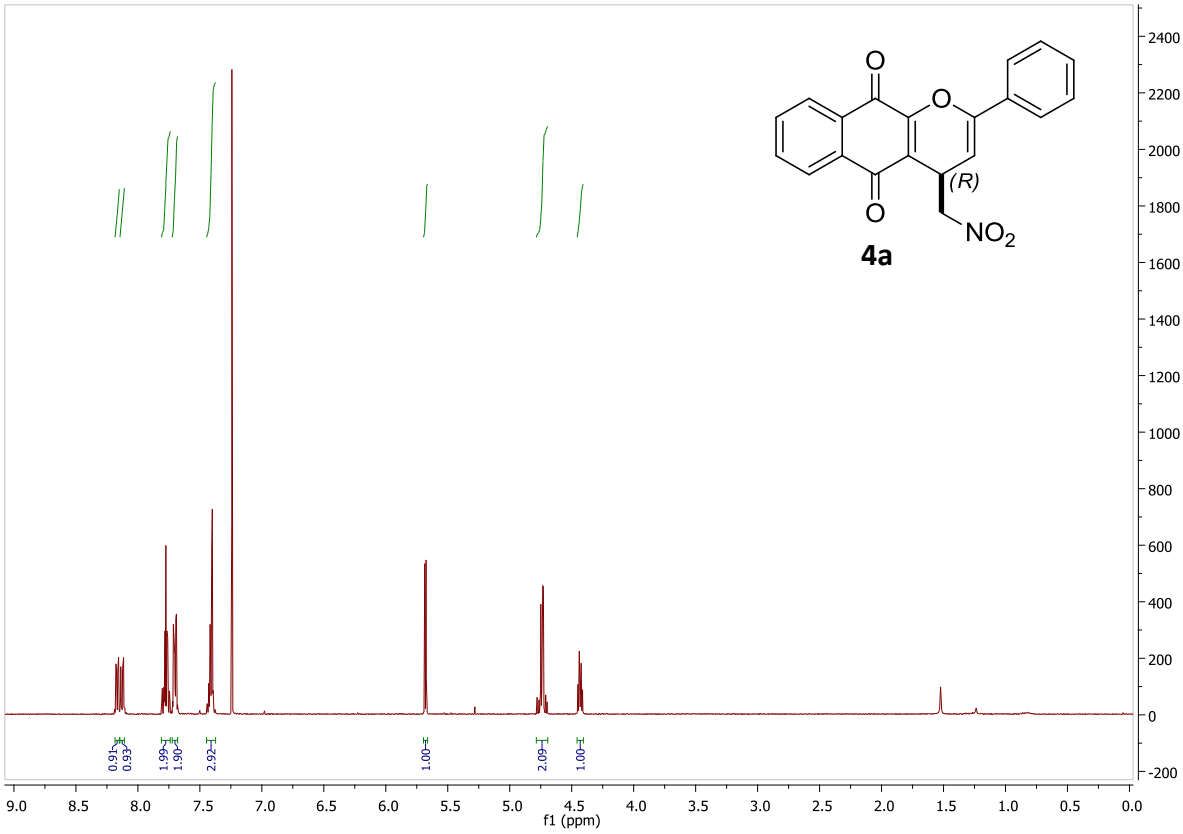
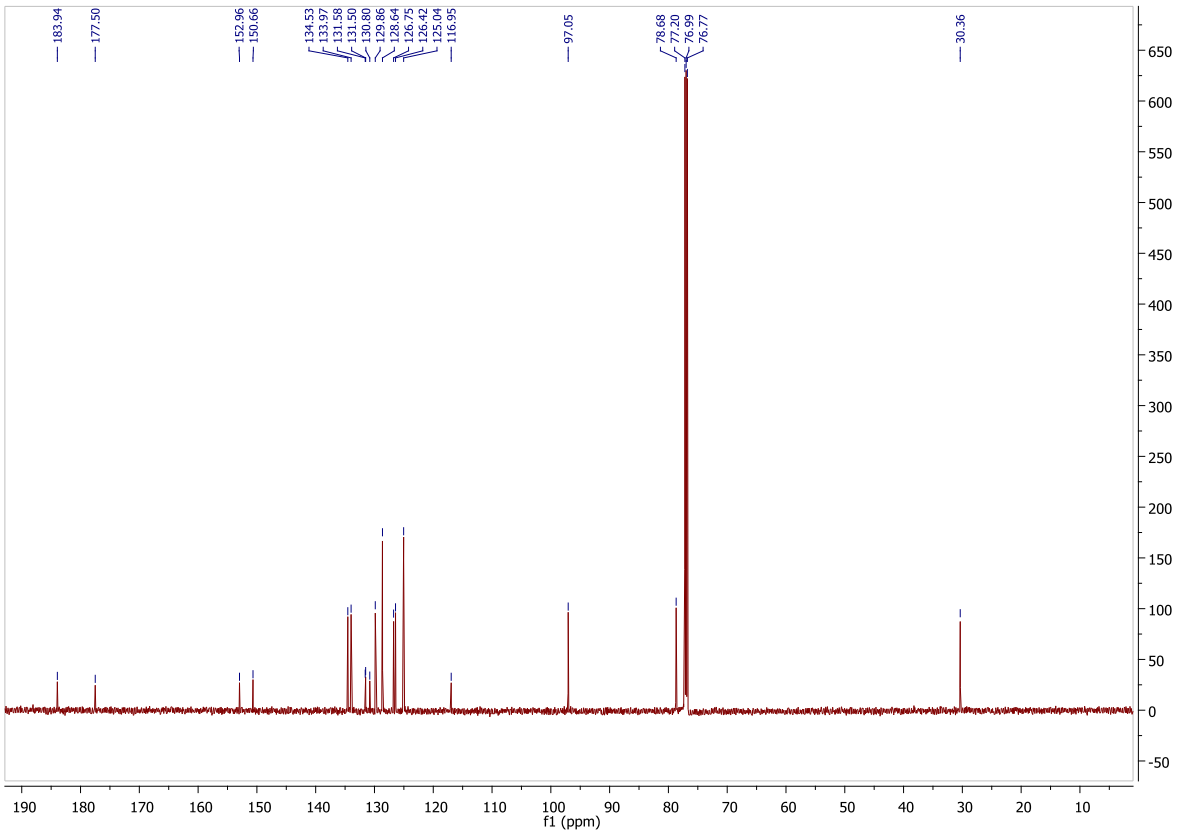
77.22
77.01
76.79
75.98
27.17
25.93
19.20
-3.64
f1 (ppm)


O
O
O
(R)
NO2
4a
0.91
0.93
1.99
1.90
2.92
1.00
2.09
1.00
f1 (ppm)


183.94
177.50
152.96
150.66
134.53
133.97
131.58
131.50
130.80
129.86
128.64
126.75
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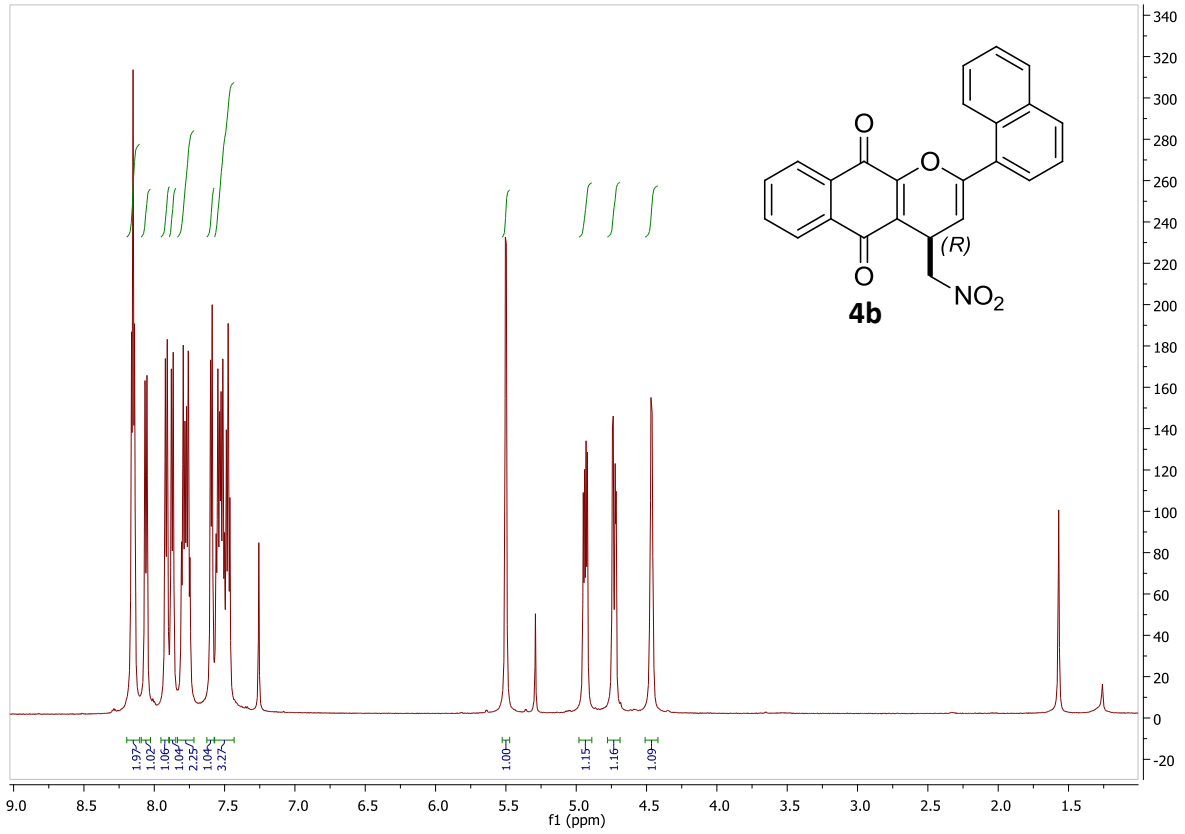
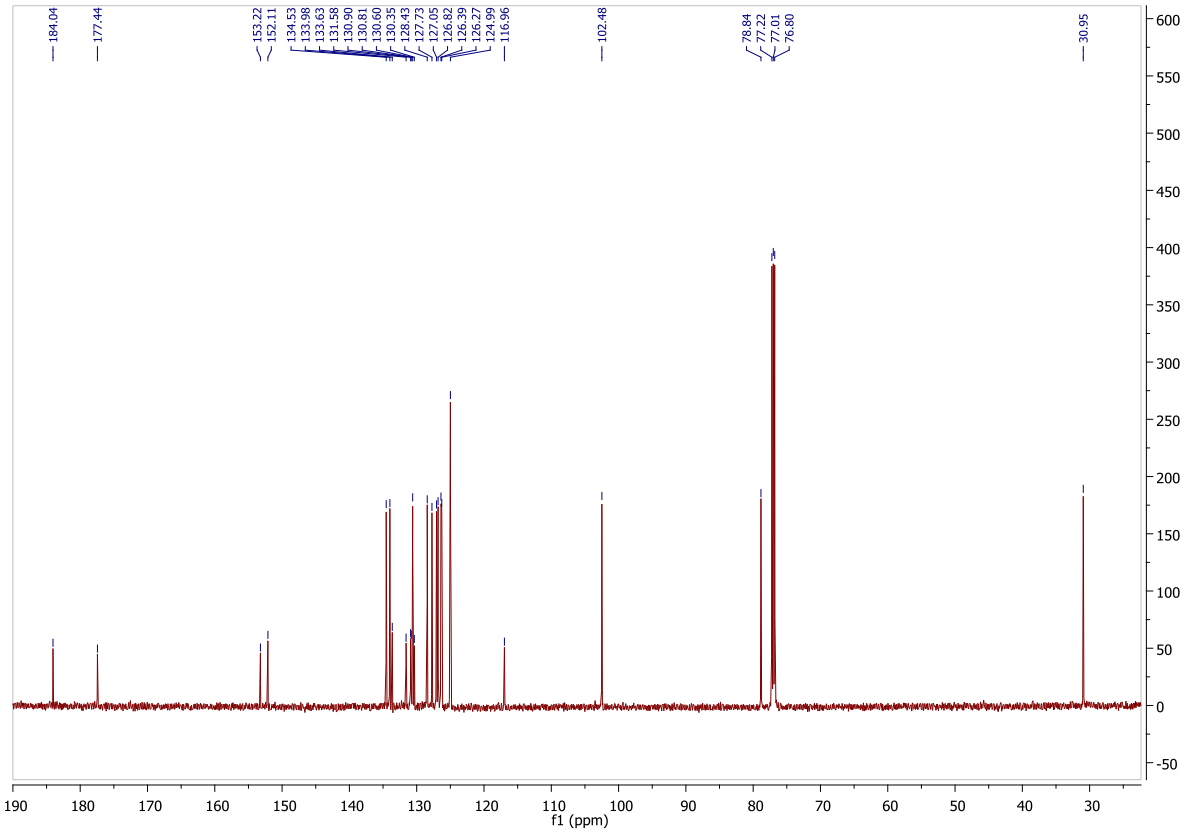
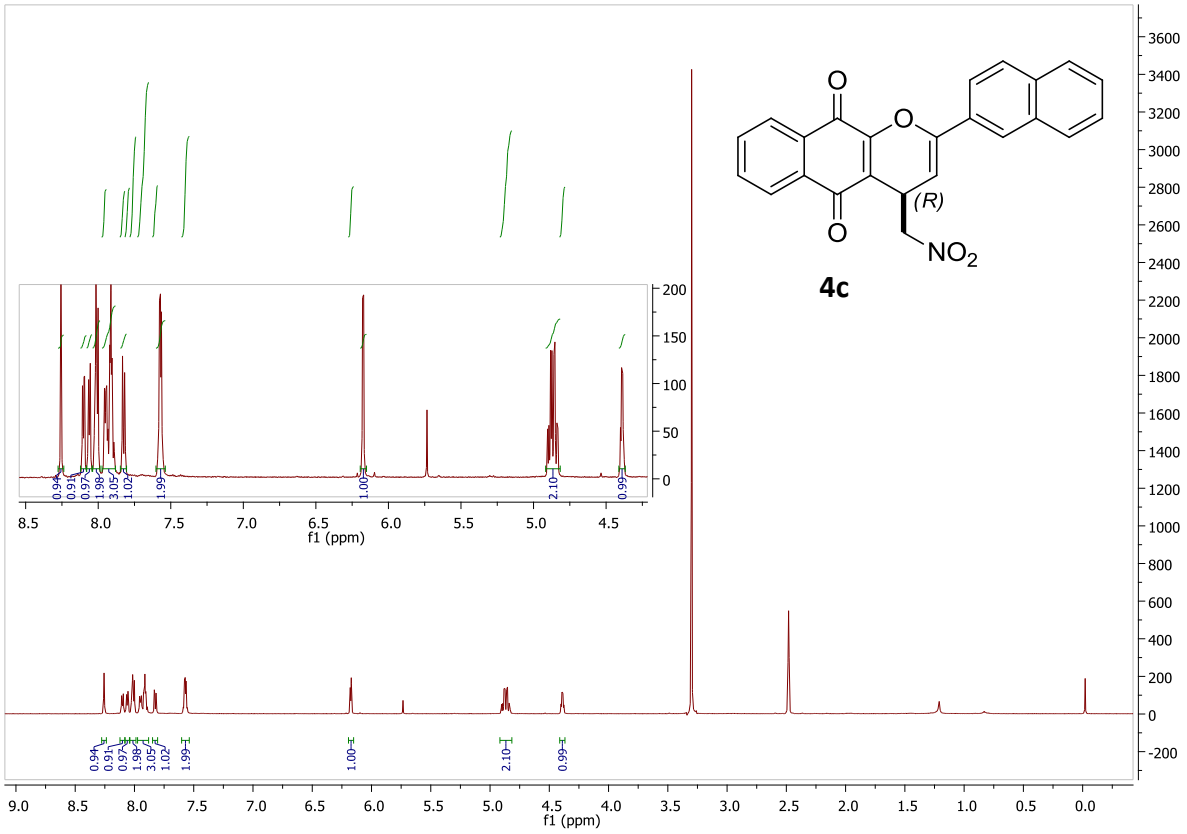
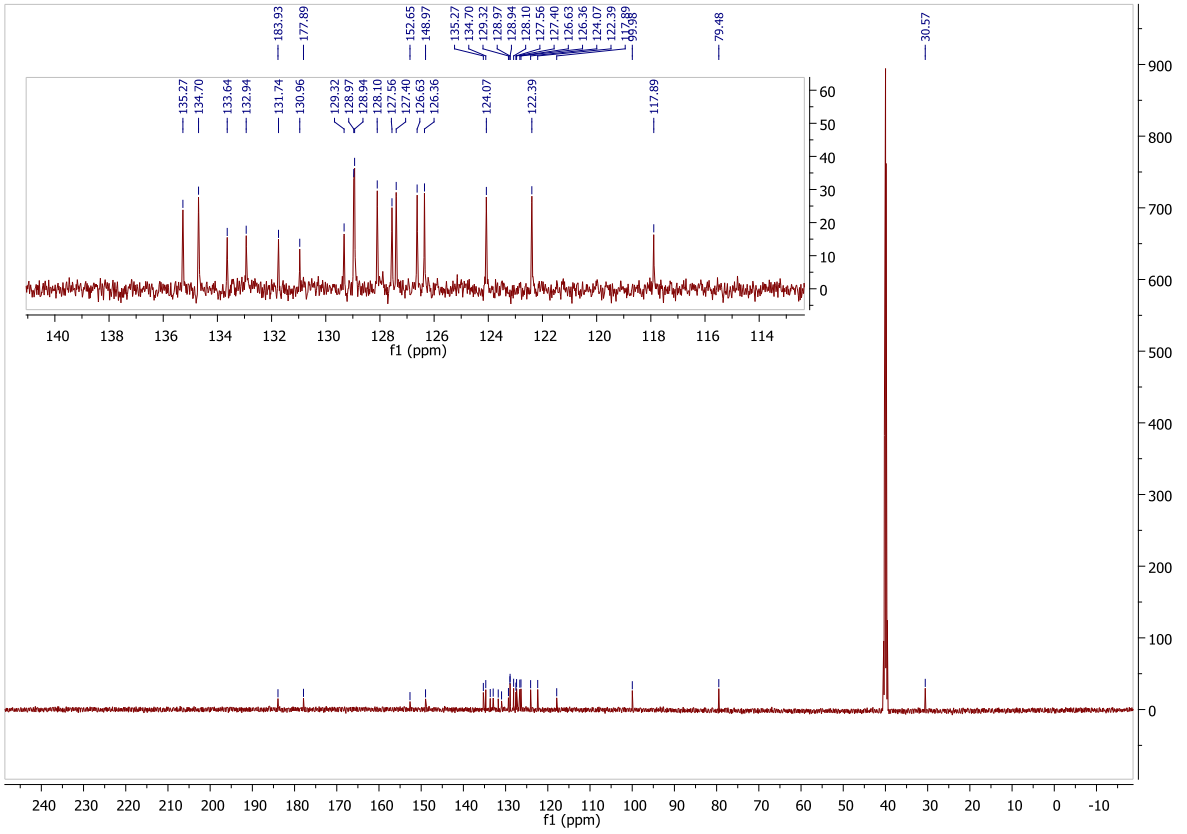
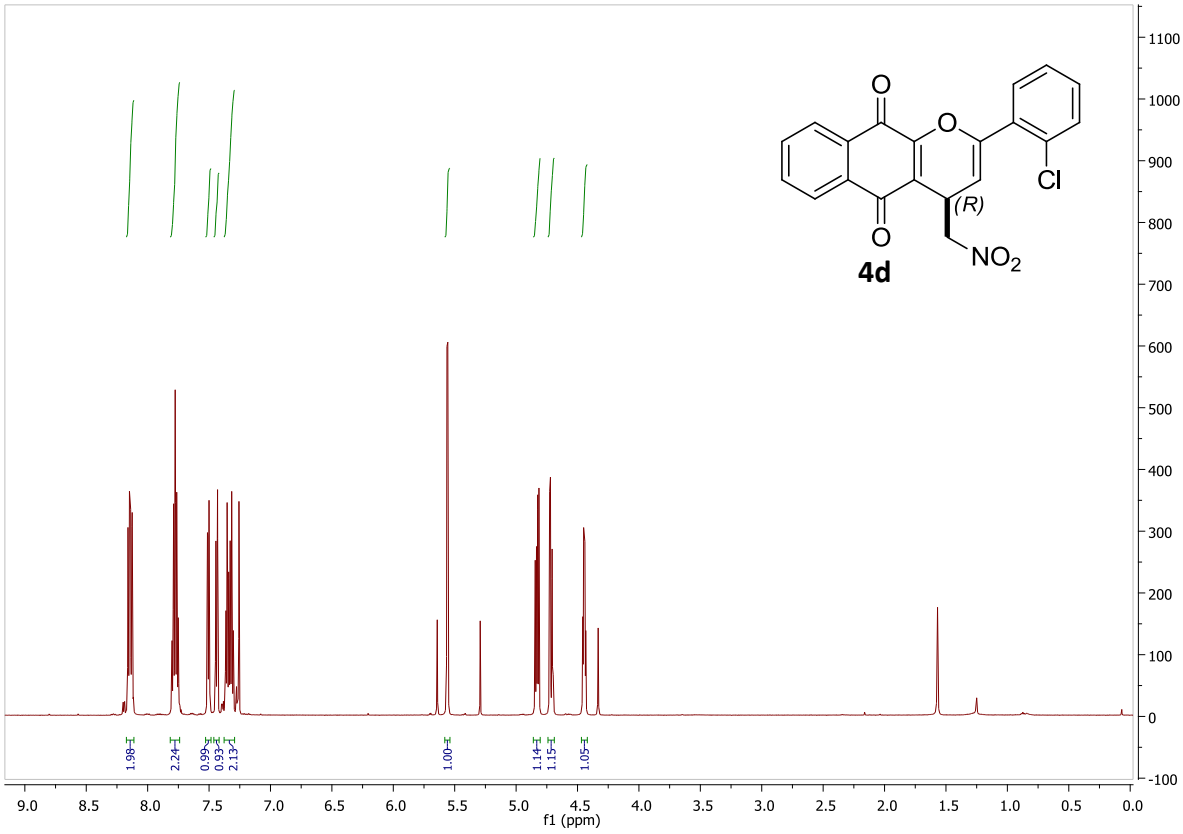
77.20
76.99
76.77
30.36
f1 (ppm)

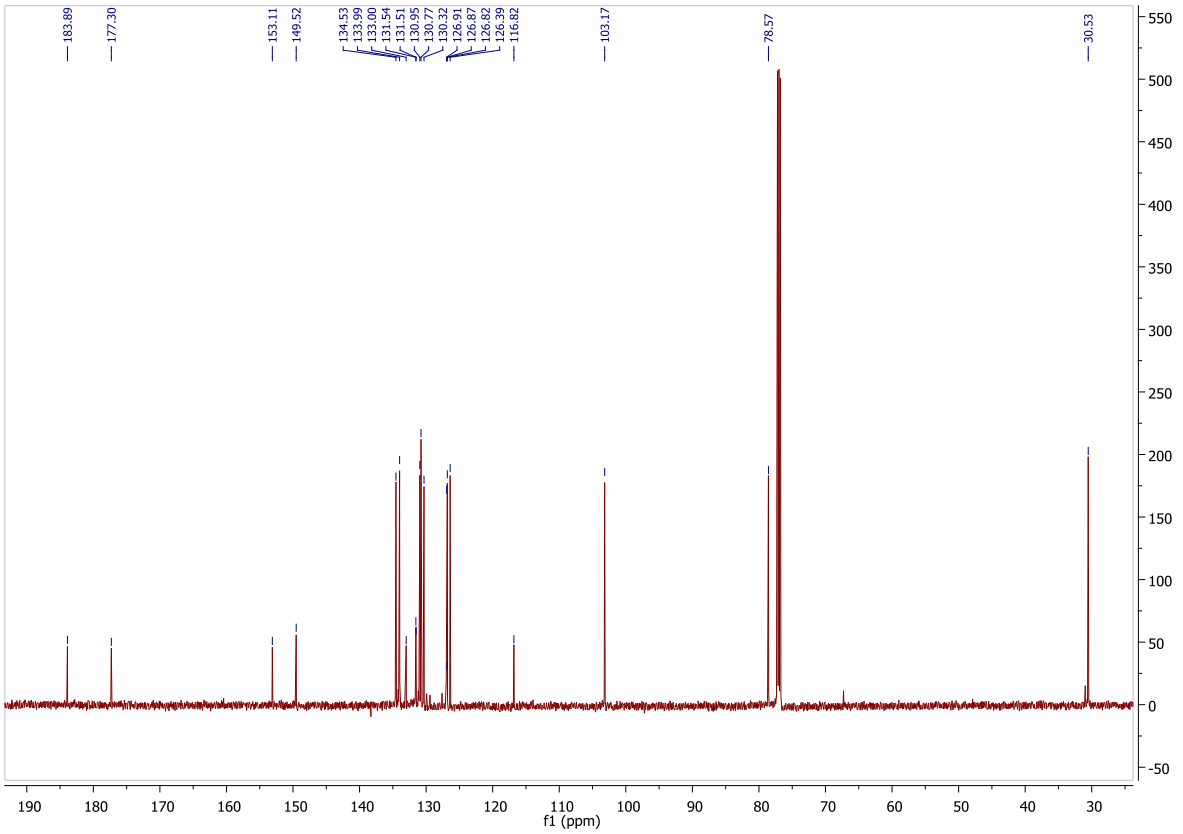

O
O
O
(R)
NO2
4b
1.97
1.02
1.06
1.04
2.25
1.04
3.27
1.00
1.15
1.16
1.09
f1 (ppm)

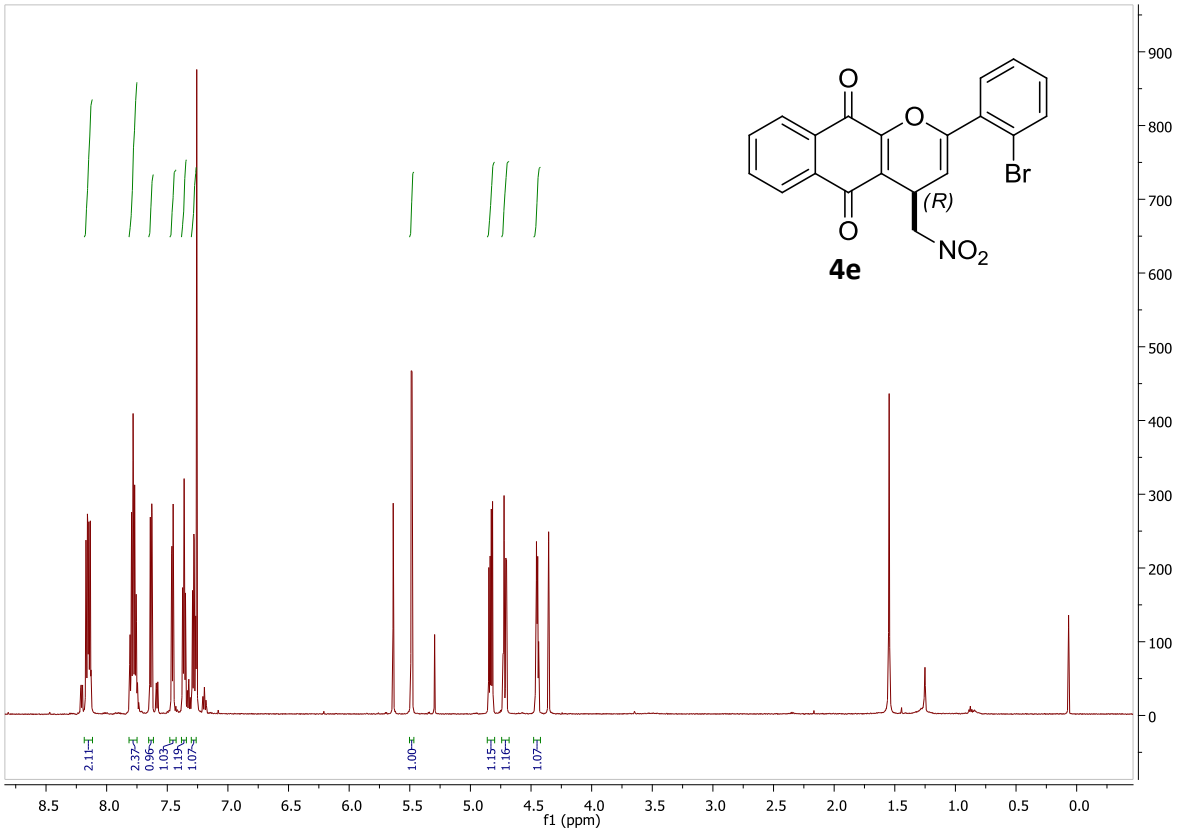

184.04
177.44
153.22
152.11
134.53
133.98
133.63
131.58
130.90
130.81
130.60
130.35
128.43
127.73
127.05
126.82
126.39
126.27
124.99
116.96
102.48
78.84
77.22
77.01
76.80
30.95
f1 (ppm)

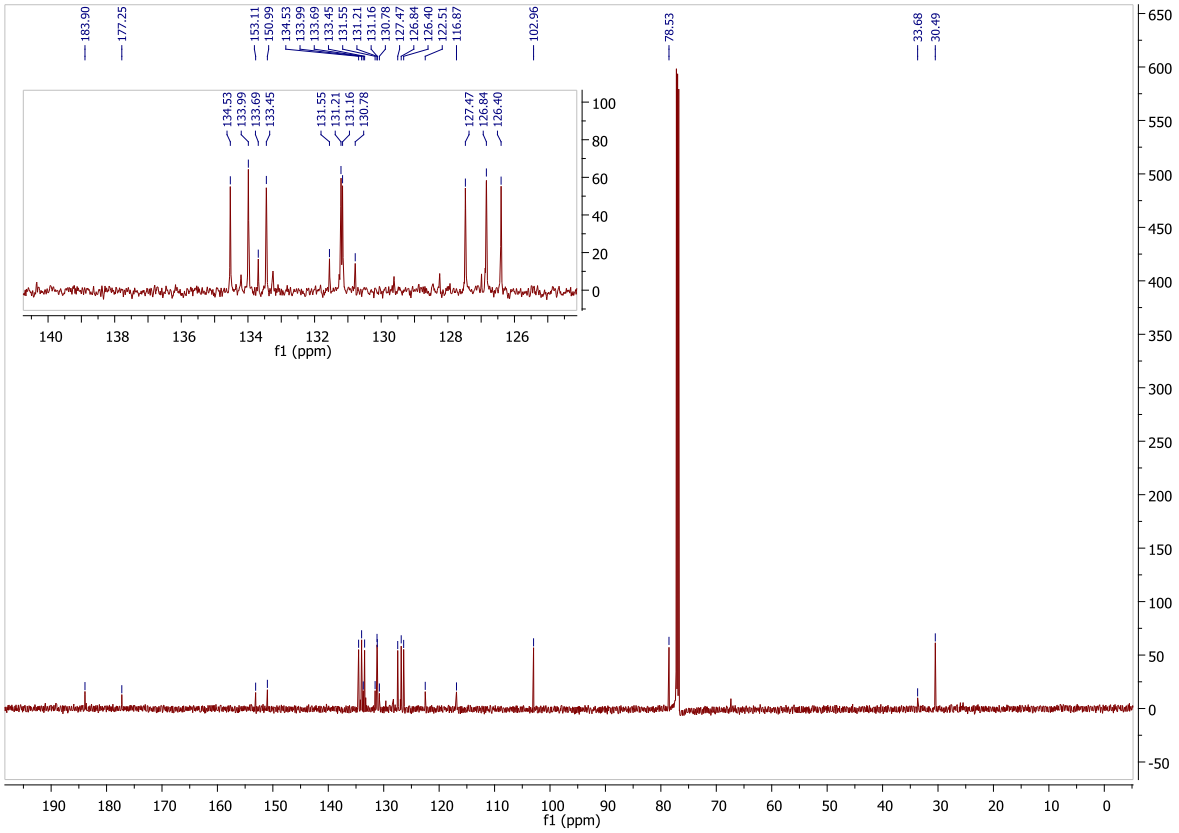

O
O
O
(R)
NO2
4c
f1 (ppm)

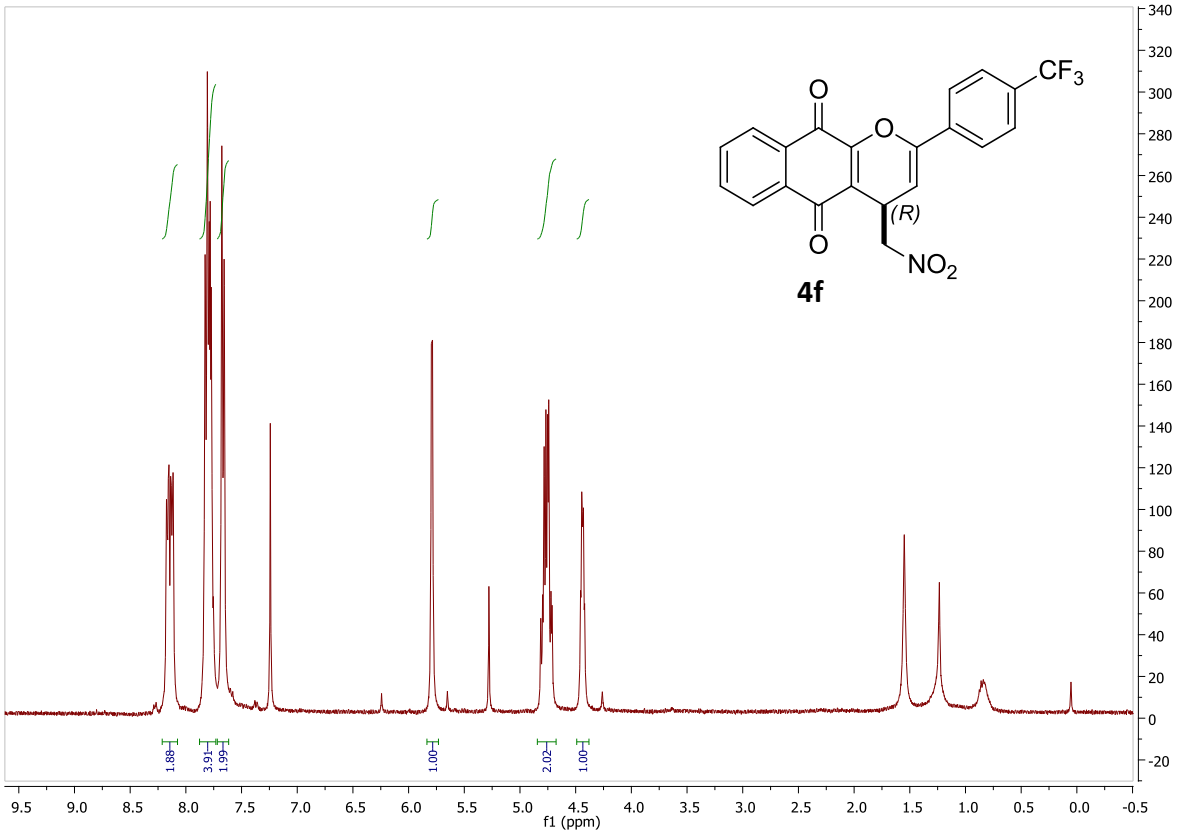

183.93
177.89
152.65
148.97
135.27
134.70
129.32
128.97
128.94
128.10
127.56
127.40
126.63
126.36
124.07
122.39
117.89
79.48
30.57
f1 (ppm)

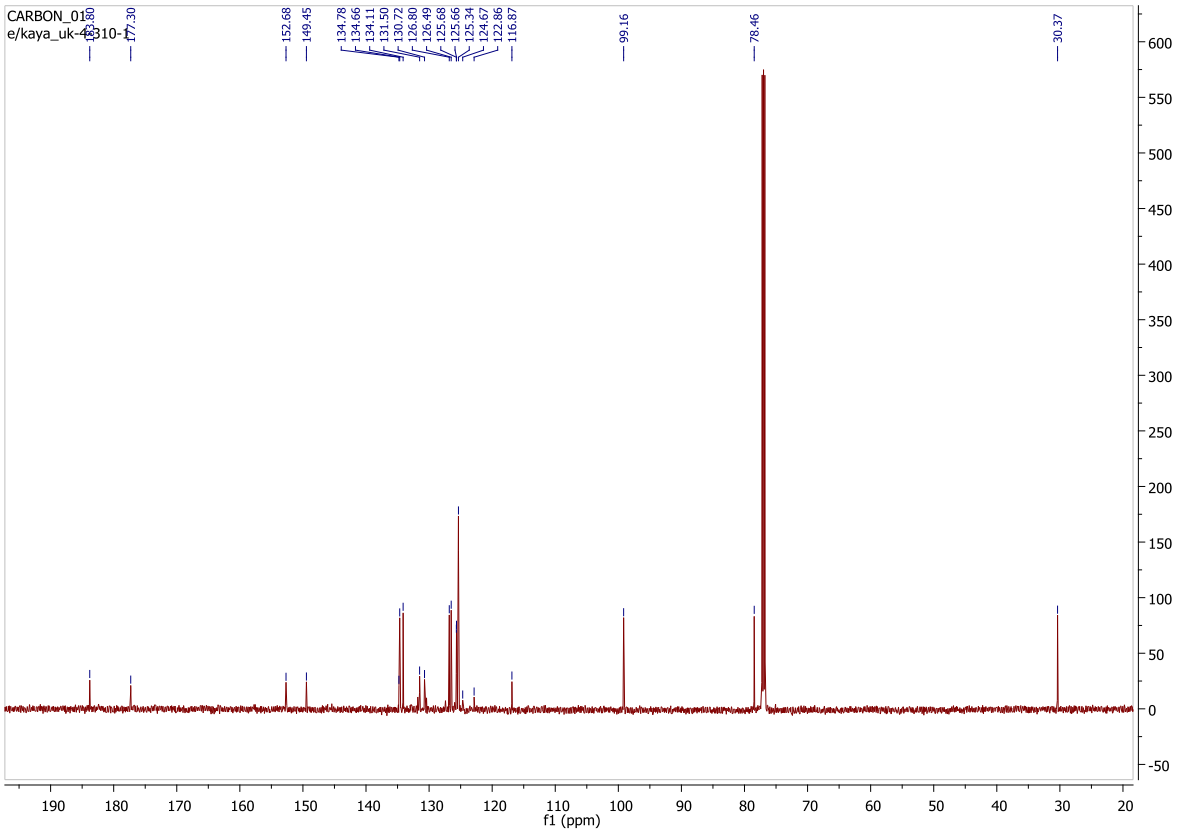

O
O
Cl
(R)
O
NO2
4d
1.98
2.24
0.99
0.93
2.13
1.00
1.14
1.15
1.05
f1 (ppm)

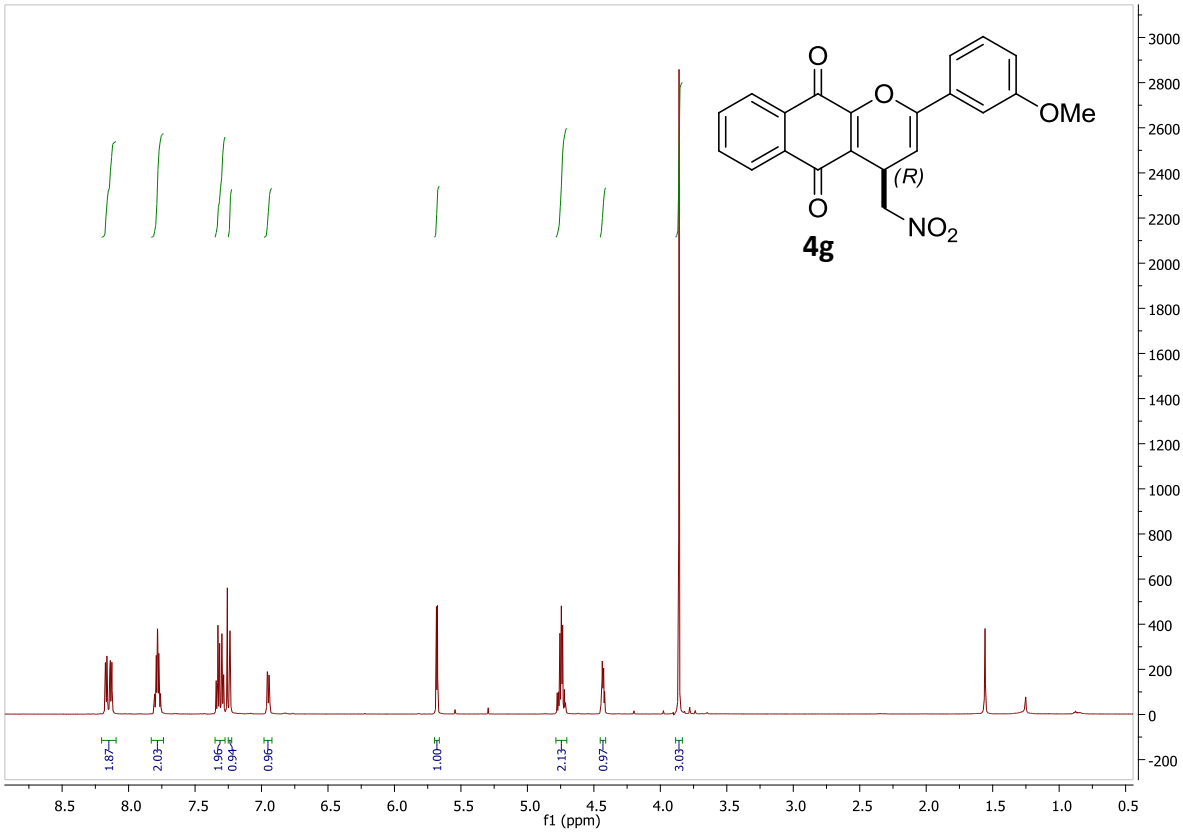

183.89
177.30
153.11
149.52
134.53
133.99
133.00
131.54
131.51
130.95
130.77
130.32
126.91
126.87
126.82
126.39
116.82
103.17
78.57
30.53
f1 (ppm)

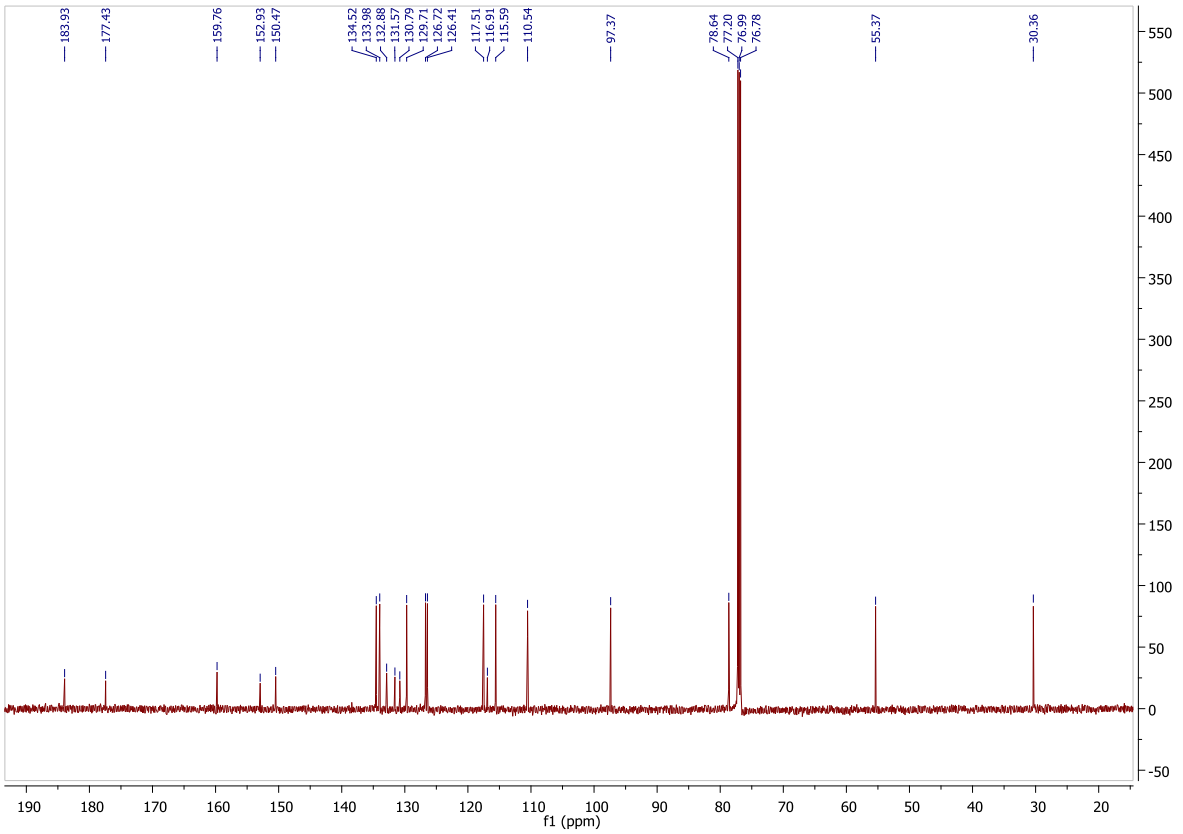

O
O
Br
(R)
O
NO2
4e
2.11
2.37
0.96
1.03
1.19
1.07
1.00
1.15
1.16
1.07
f1 (ppm)

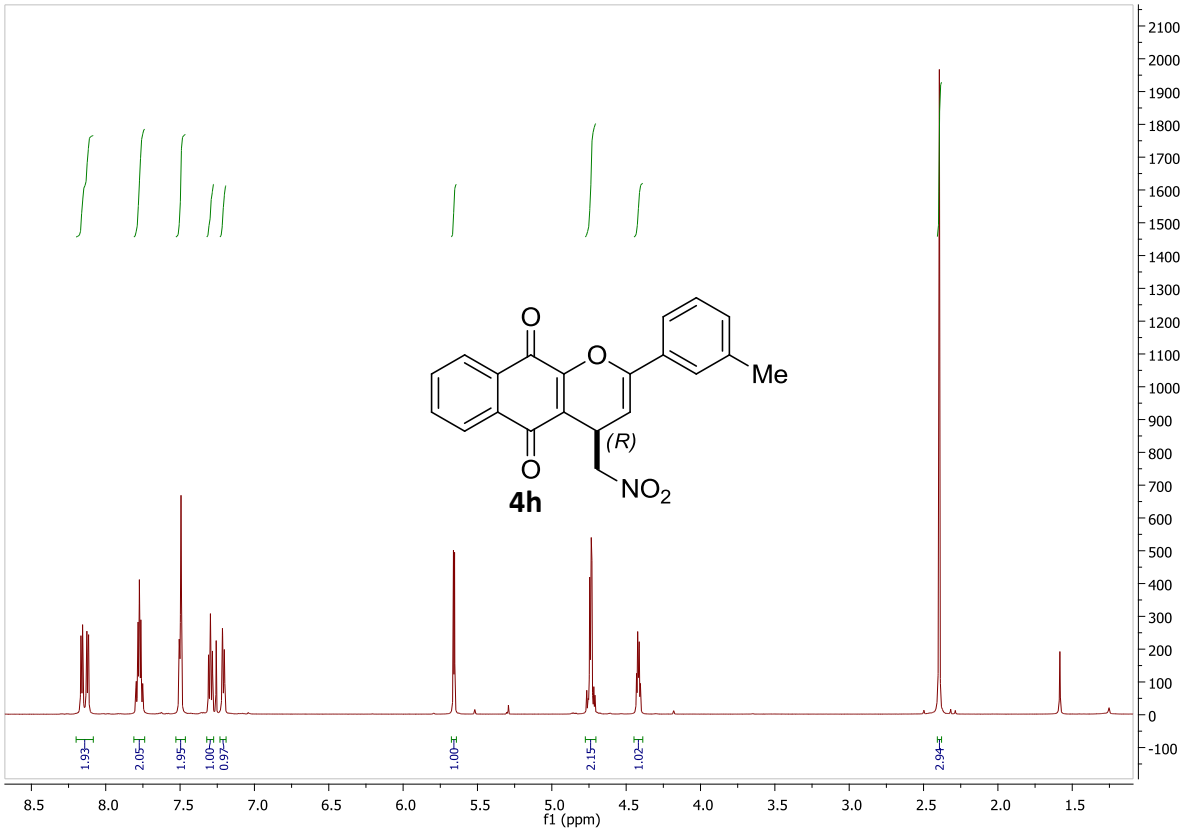

183.90
177.25
153.11
150.99
134.53
133.99
133.69
133.45
131.55
131.21
131.16
130.78
127.47
126.84
126.40
122.51
116.87
102.96
78.53
33.68
30.49
f1 (ppm)

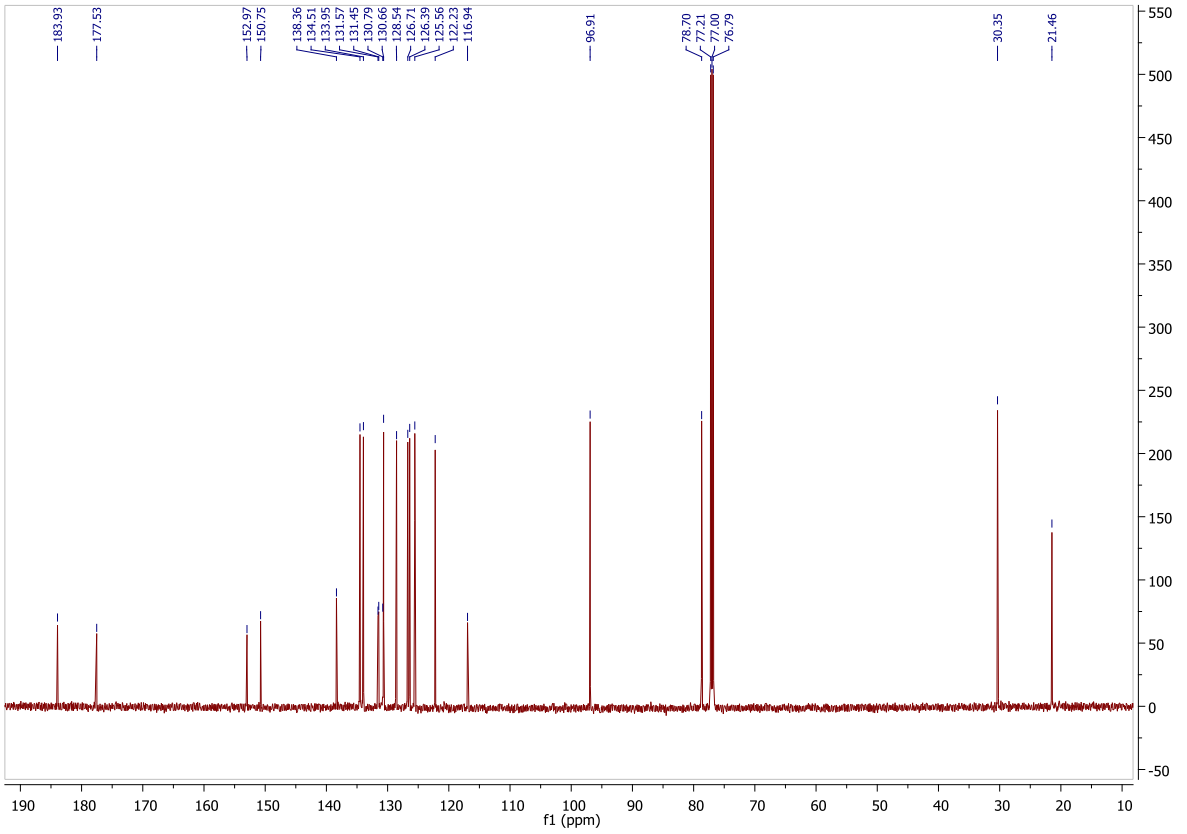

O
O
O
O
CF3
(R)
NO2
4f
1.88
3.91
1.99
1.00
2.02
1.00
f1 (ppm)

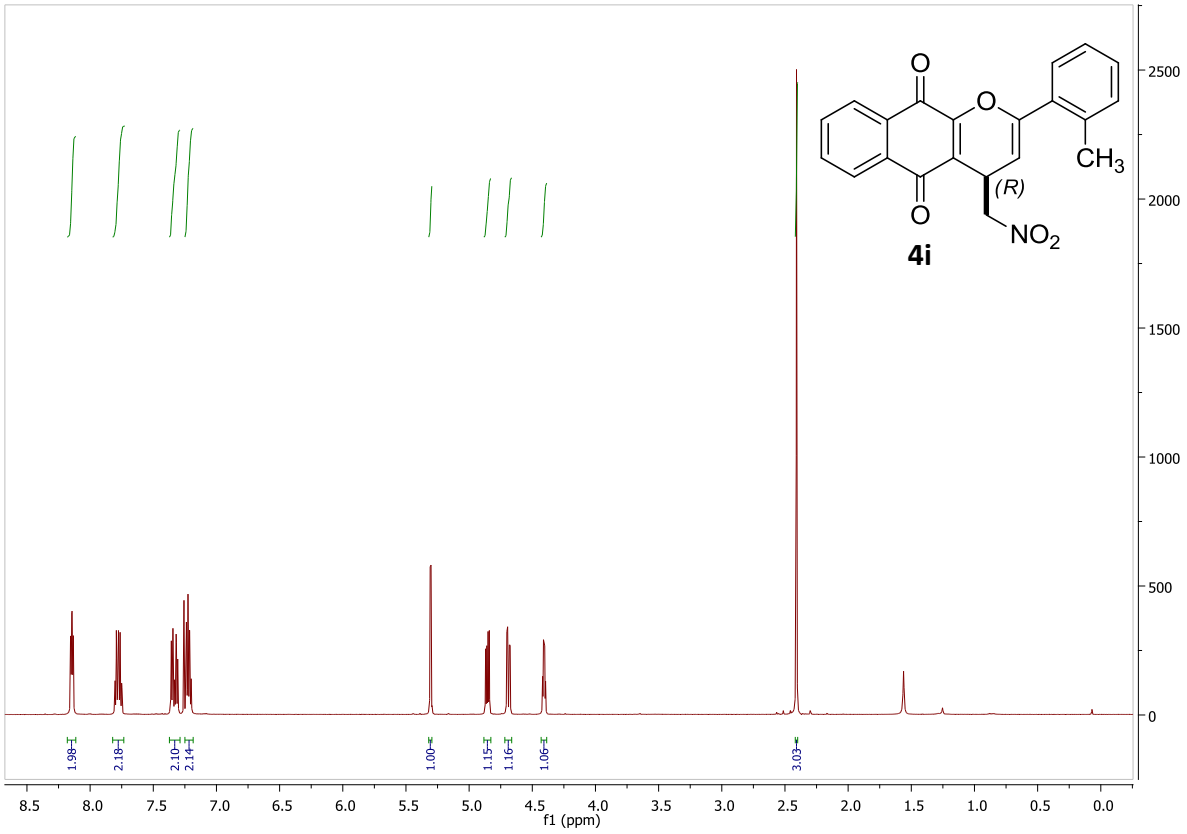

CARBON_01
e/kaya_uk-4310-1
177.30
152.68
149.45
134.78
134.66
134.11
131.50
130.72
126.80
126.49
125.68
125.66
125.34
124.67
122.86
116.87
99.16
78.46
30.37
f1 (ppm)

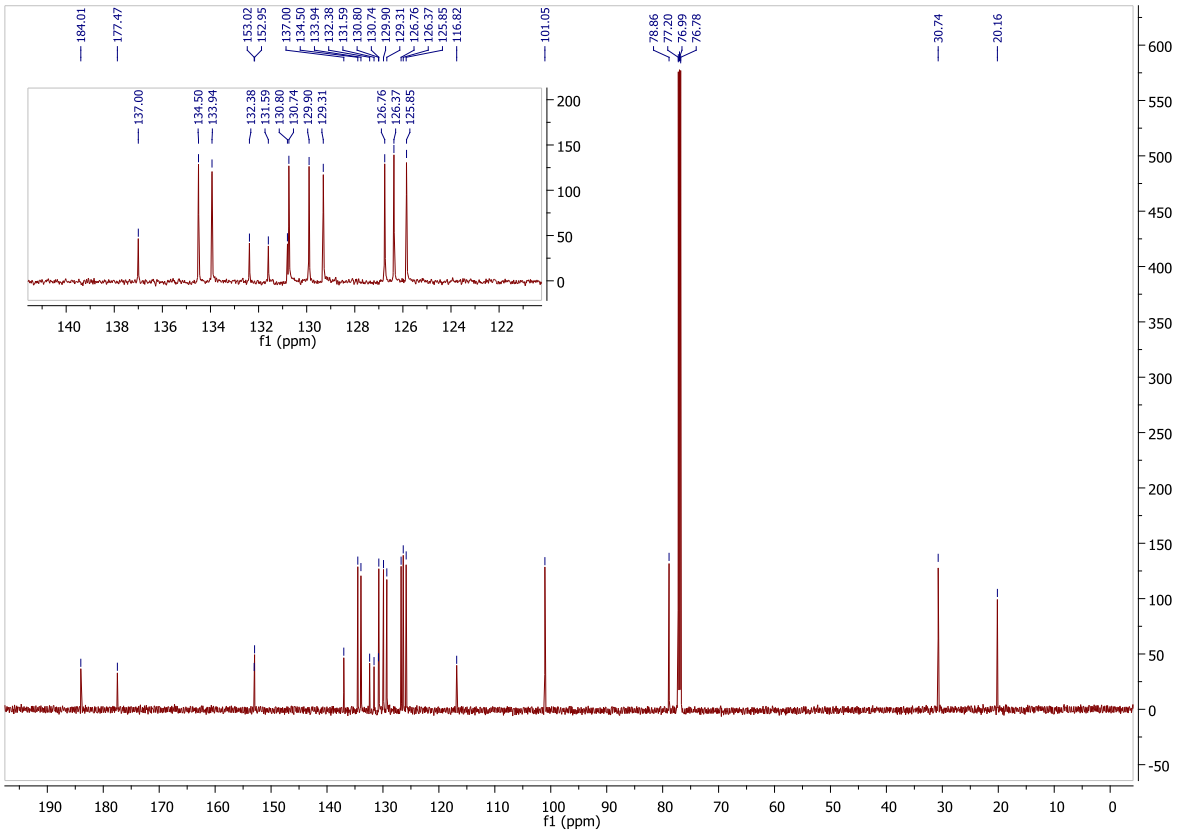

O
O
OMe
(R)
O
NO2
4g
1.87
2.03
1.96
0.94
0.96
1.00
2.13
0.97
3.03
f1 (ppm)

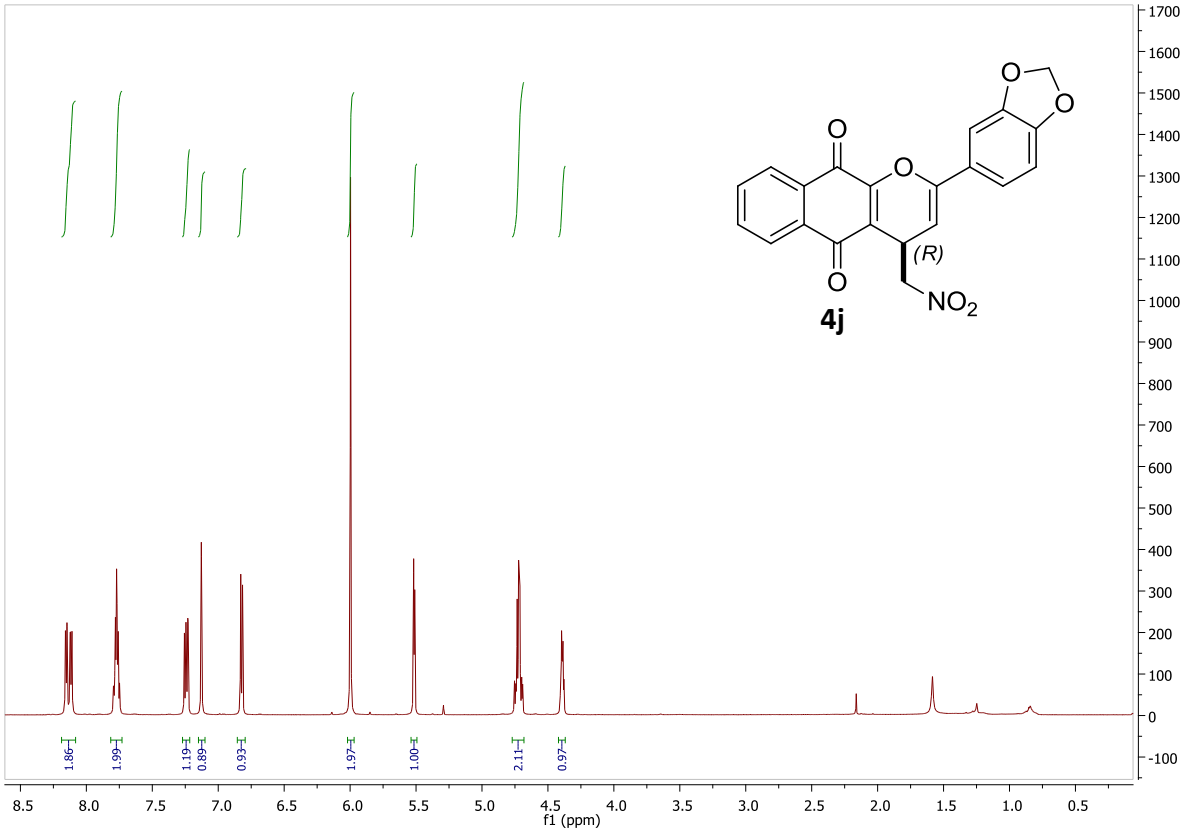

183.93
177.43
159.76
152.93
150.47
134.52
133.98
132.88
131.57
130.79
129.71
126.72
126.41
117.51
116.91
115.59
110.54
97.37
78.64
77.20
76.99
76.78
55.37
30.36
f1 (ppm)

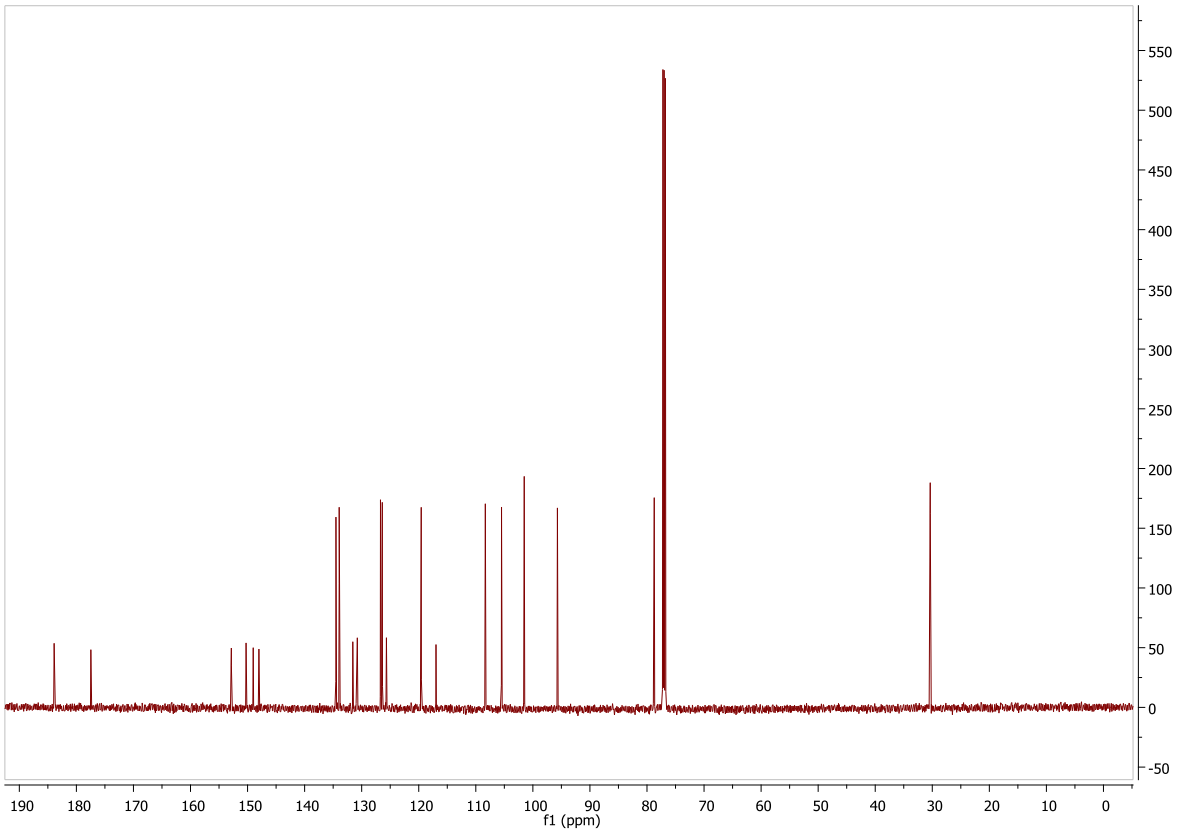

O
O
Me
(R)
O
NO2
4h
1.93
2.05
1.95
1.00
0.97
1.00
2.15
1.02
2.94
f1 (ppm)

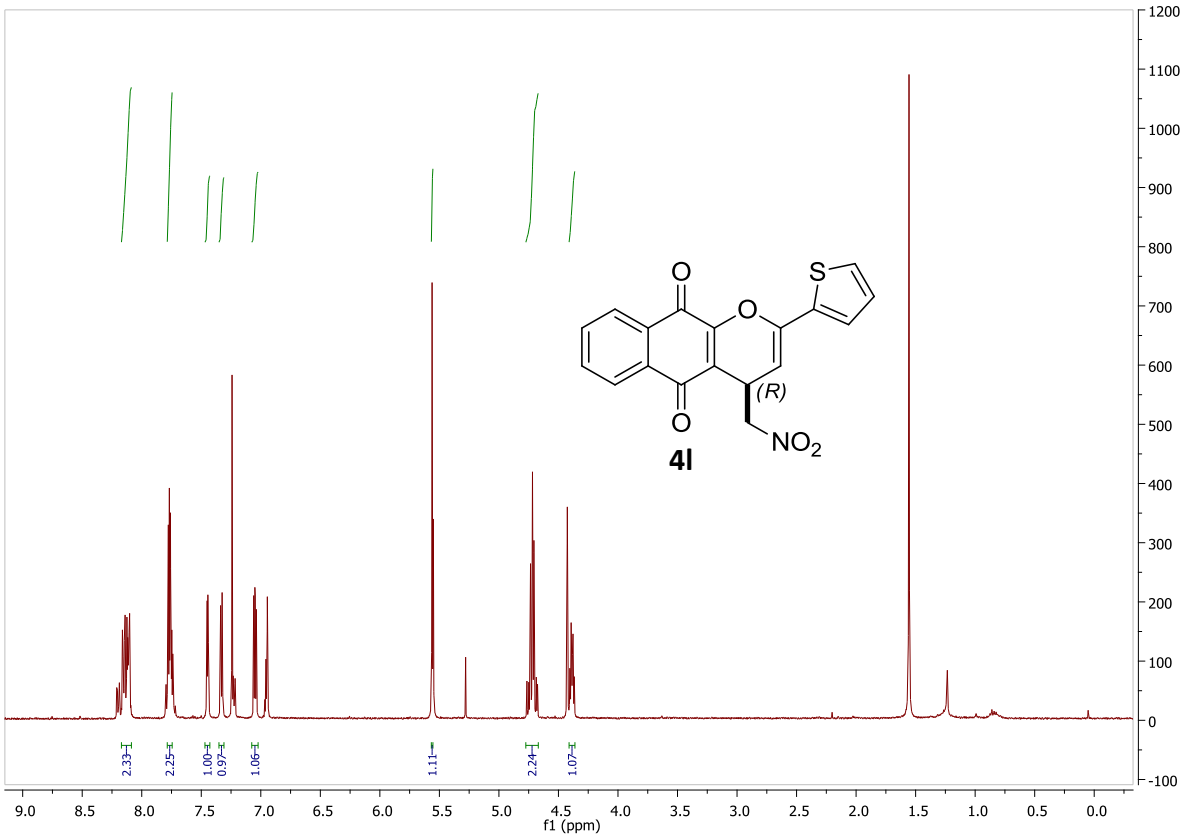

183.93
177.53
152.97
150.75
138.36
134.51
133.95
131.57
131.45
130.79
130.66
128.54
126.71
126.39
125.56
122.23
116.94
96.91
78.70
77.21
77.00
76.79
30.35
21.46
f1 (ppm)

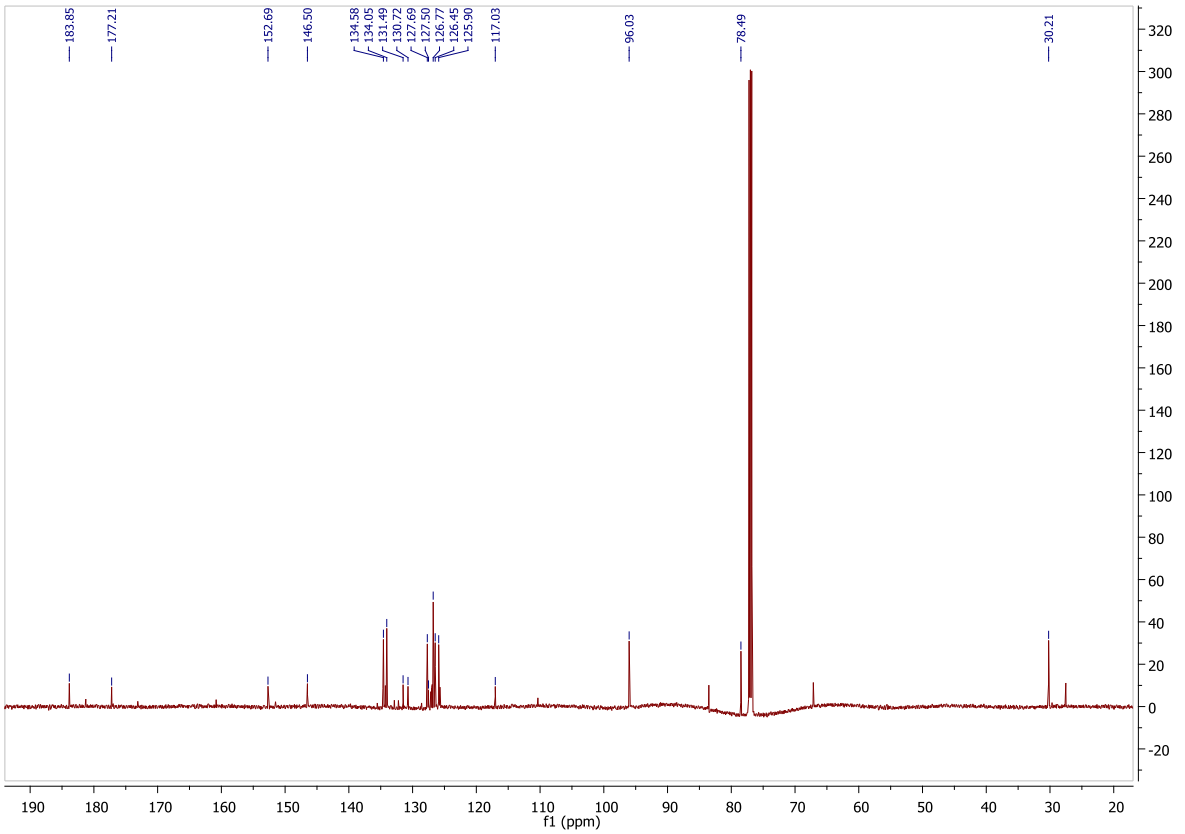

O
O
O
CH3
(R)
NO2
4i
1.98
2.18
2.10
2.14
1.00
1.15
1.16
1.06
3.03
f1 (ppm)

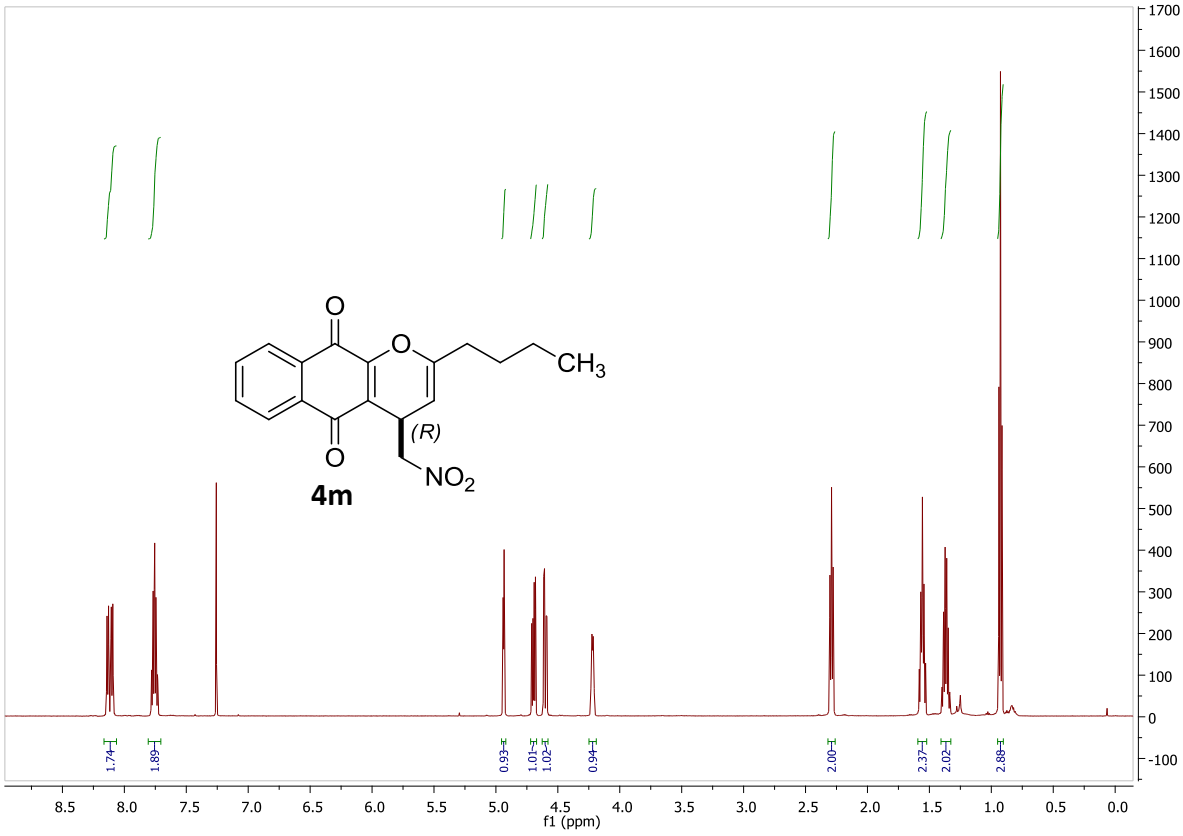

184.01
177.47
153.02
152.95
137.00
134.50
133.94
132.38
131.59
130.80
130.74
129.90
129.31
126.76
126.37
125.85
116.82
101.05
78.86
77.20
76.99
76.78
30.74
20.16
f1 (ppm)

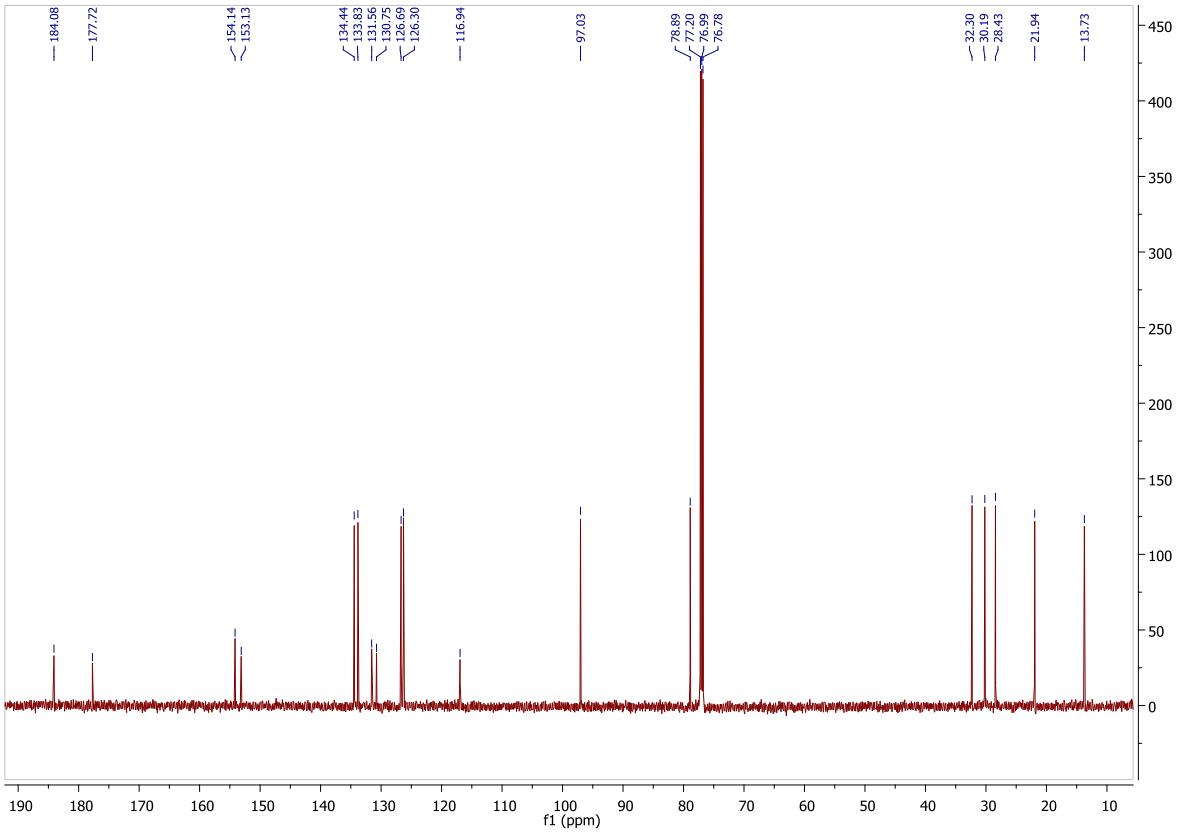

O
O
O
O
O
(R)
NO2
4j
1.86
1.99
1.19
0.89
0.93
1.97
1.00
2.11
0.97
f1 (ppm)

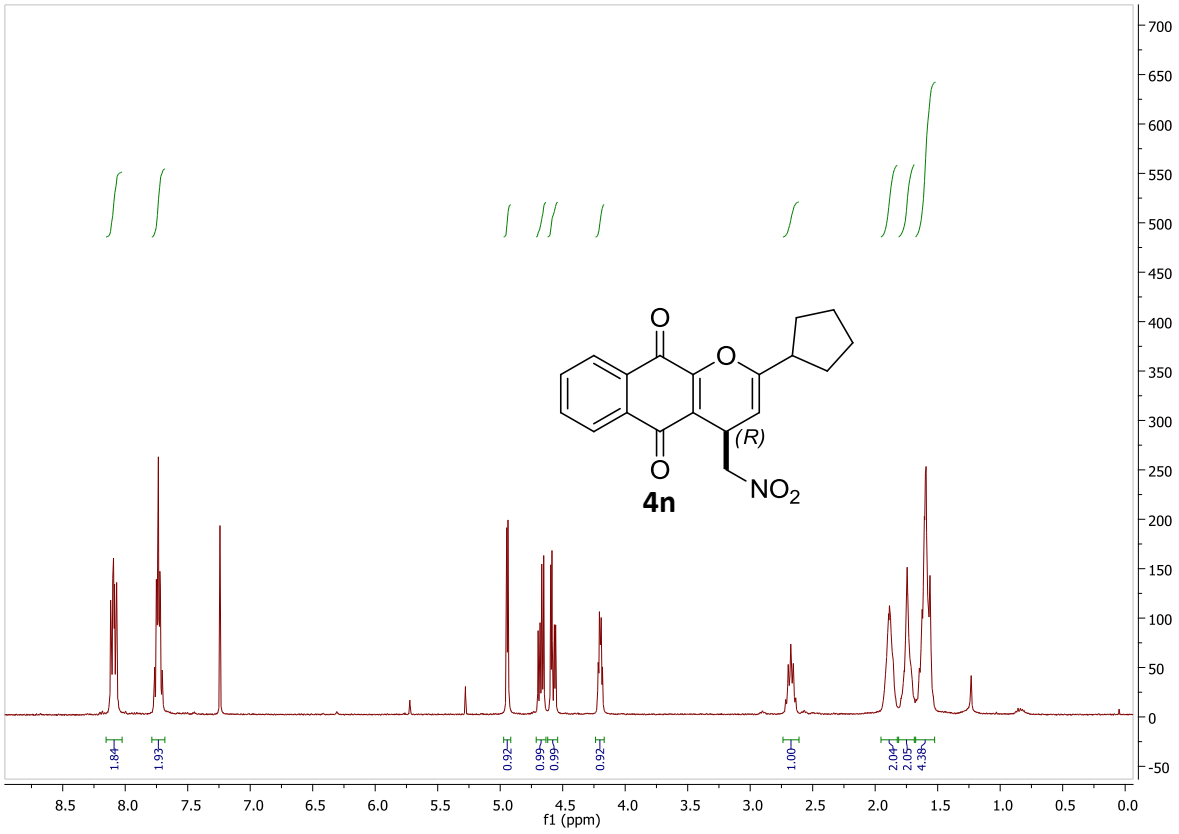

f1 (ppm)

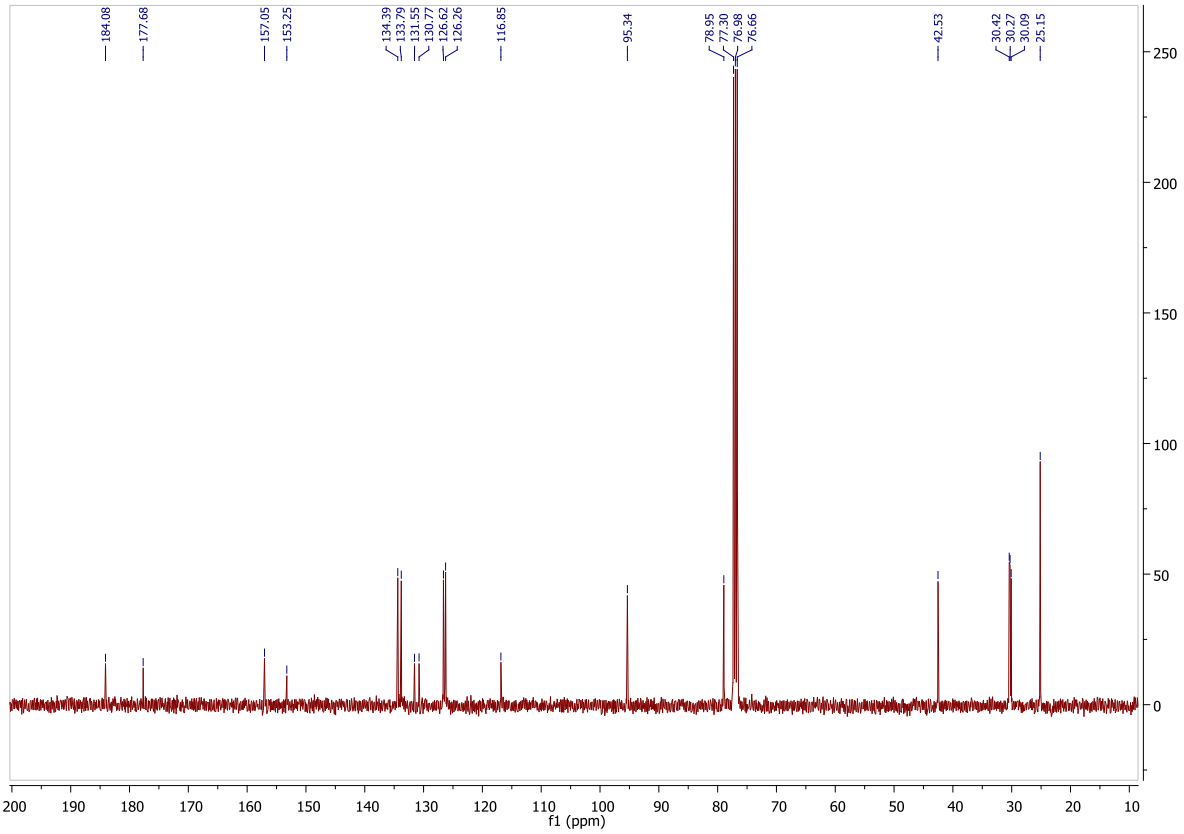

O
S
O
(R)
O
NO2
4l
2.33
2.25
1.00
0.97
1.06
1.11
2.24
1.07
f1 (ppm)

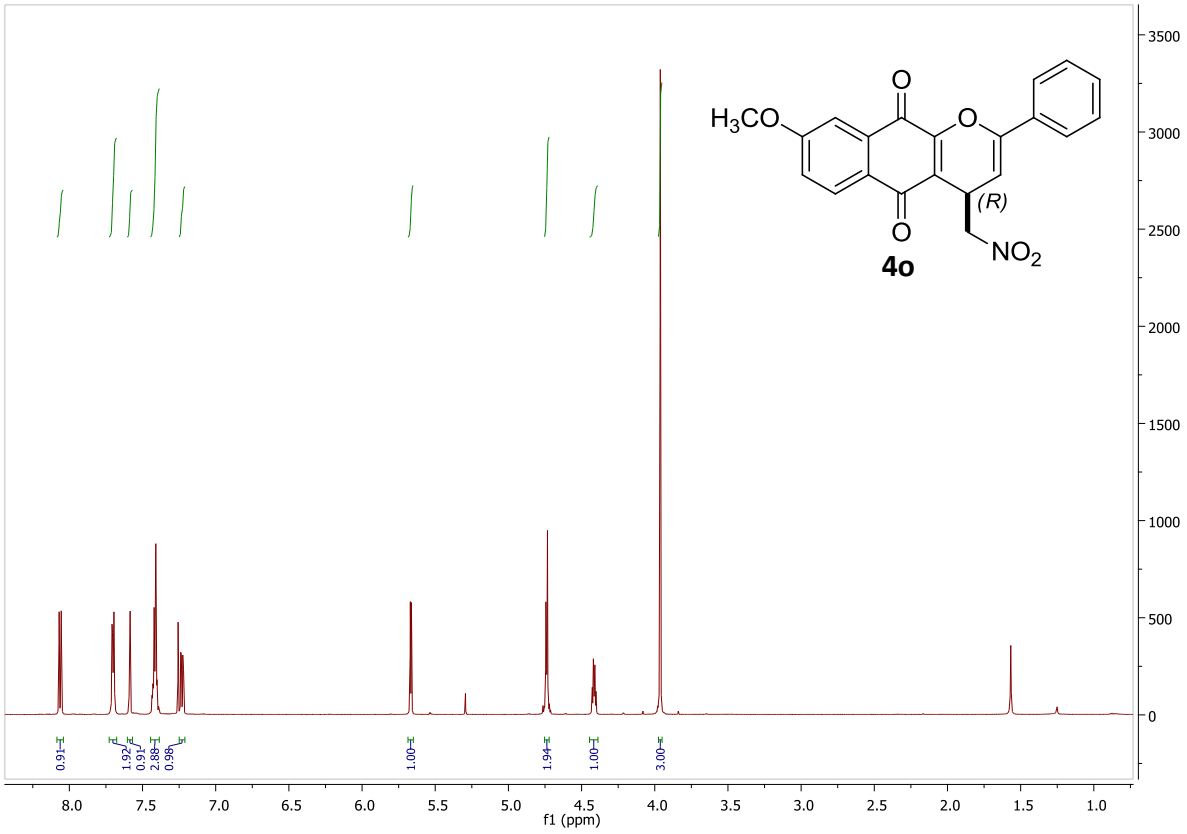

183.85
177.21
152.69
146.50
134.58
134.05
131.49
130.72
127.69
127.50
126.77
126.45
125.90
117.03
96.03
78.49
30.21
f1 (ppm)

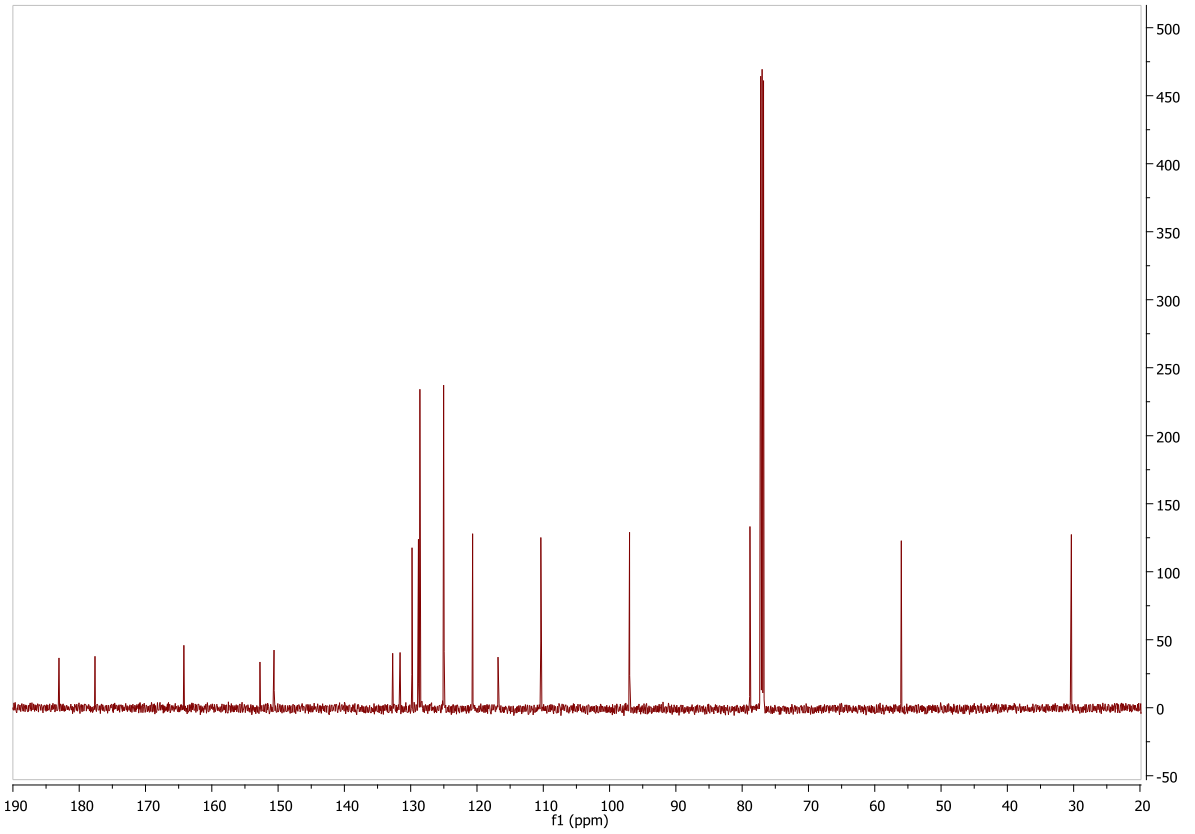

O
O
CH3
(R)
O
NO2
4m
1.74
1.89
0.93
1.01
1.02
0.94
2.00
2.37
2.02
2.88
f1 (ppm)

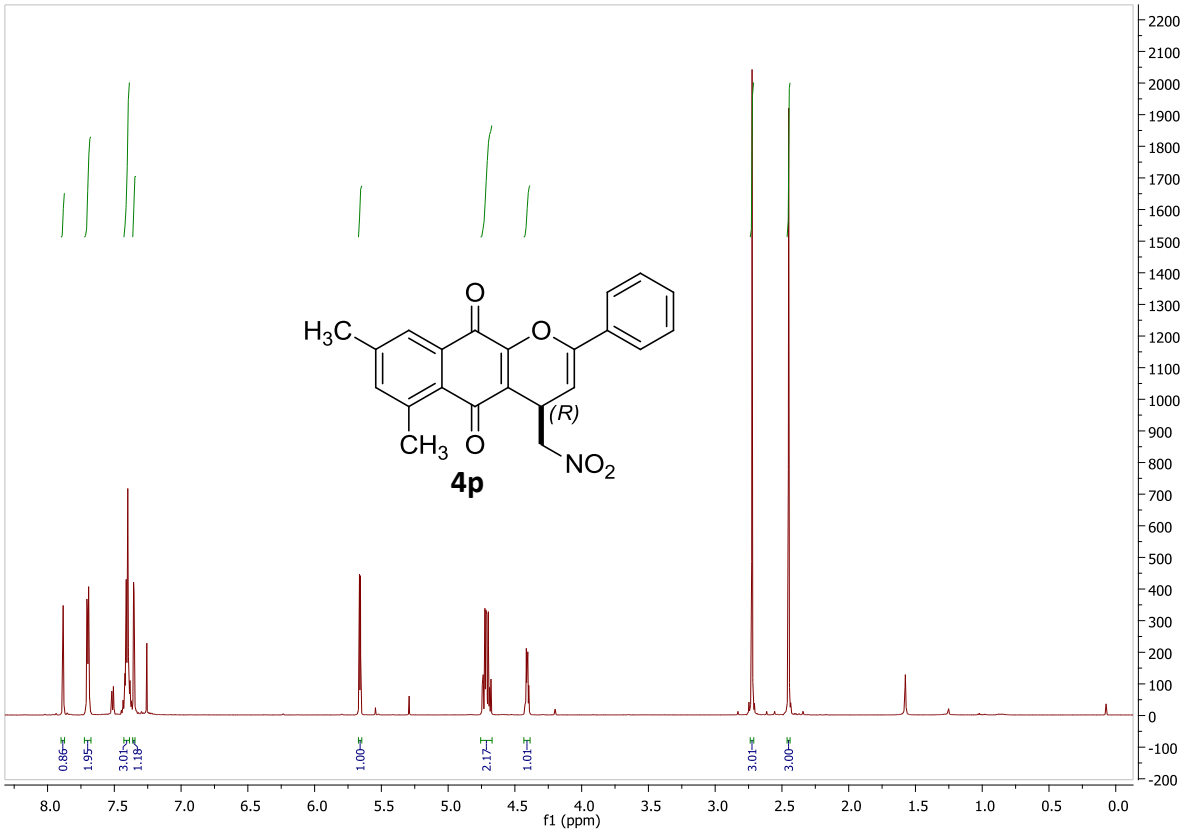

184.08
177.72
154.14
153.13
134.44
133.83
131.56
130.75
126.69
126.30
116.94
97.03
78.89
77.20
76.99
76.78
32.30
30.19
28.43
21.94
13.73
f1 (ppm)

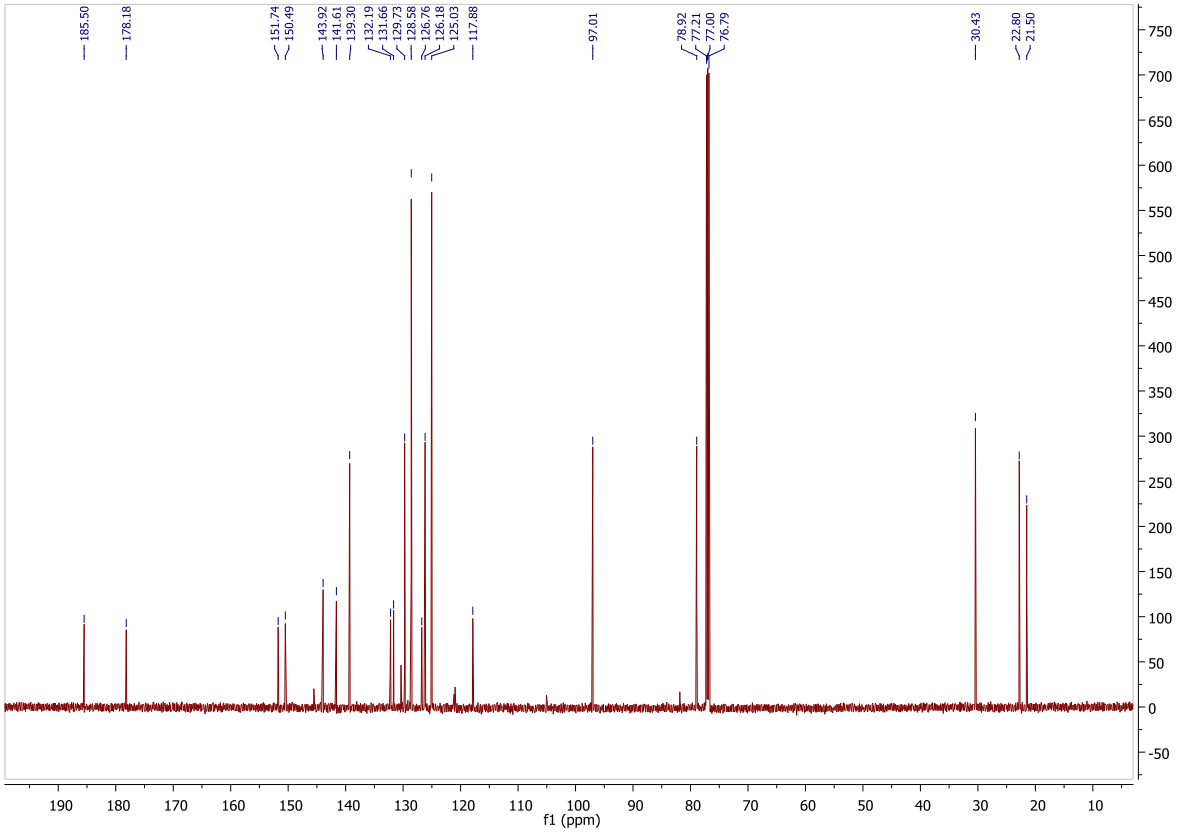

O
O
O
(R)
NO2
4n
1.84
1.93
0.92
0.99
0.99
0.92
1.00
2.04
2.05
4.38
f1 (ppm)

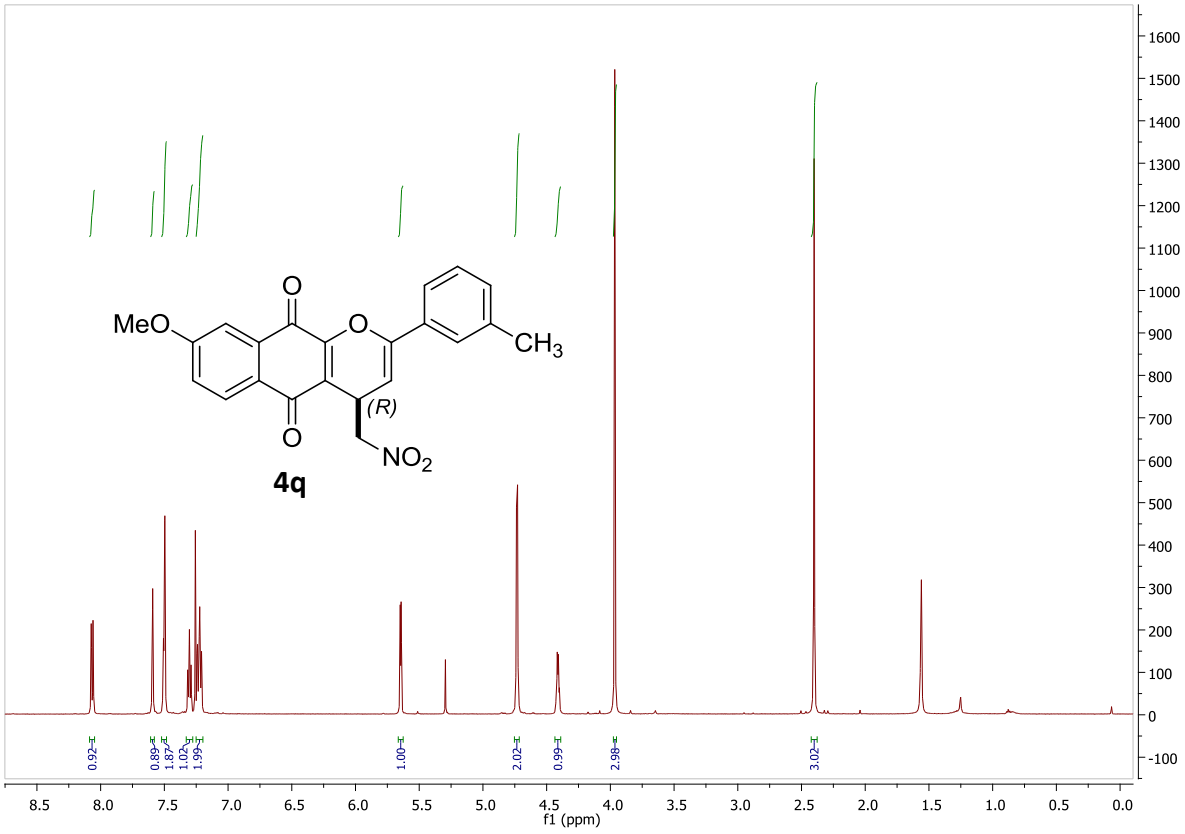

184.08
177.68
157.05
153.25
134.39
133.79
131.55
130.77
126.62
126.26
116.85
95.34
78.95
77.30
76.98
76.66
42.53
30.42
30.27
30.09
25.15
f1 (ppm)

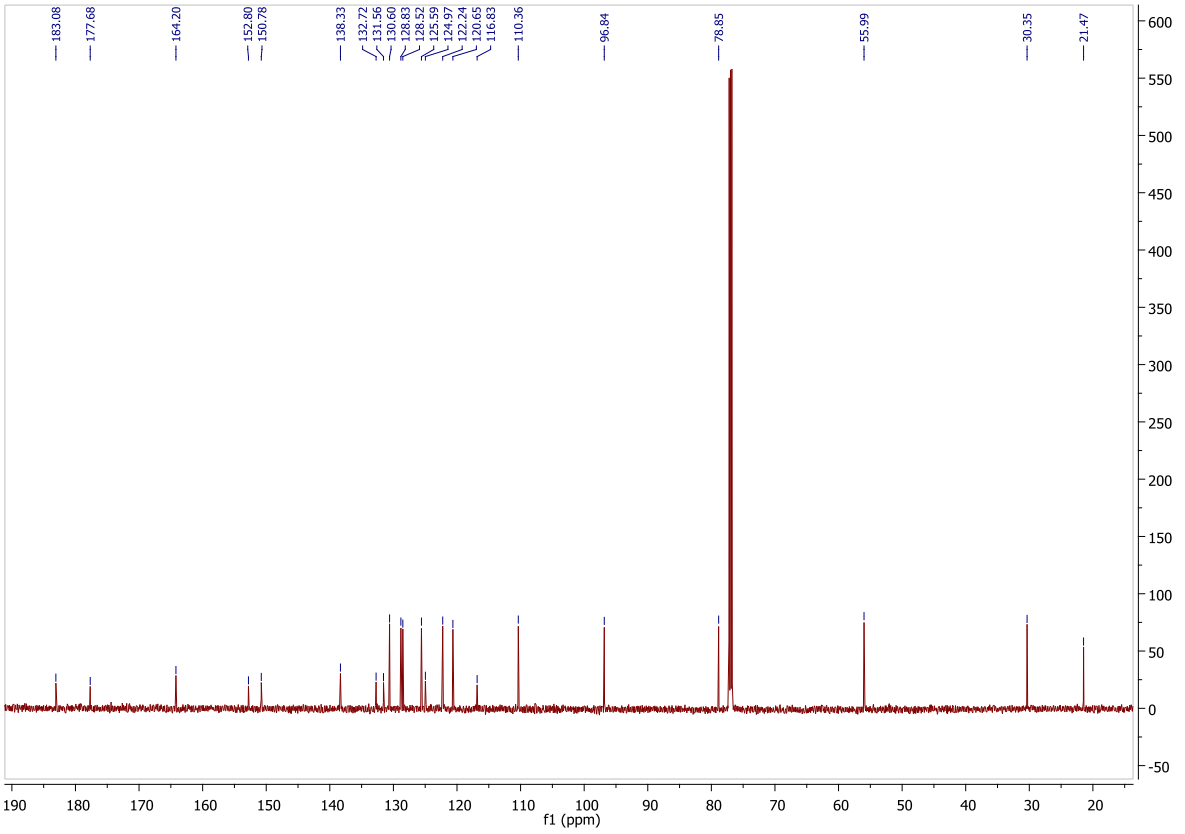

H3CO
O
O
O
(R)
NO2
4o
0.91
1.92
0.91
2.88
0.98
1.00
1.94
1.00
3.00
f1 (ppm)

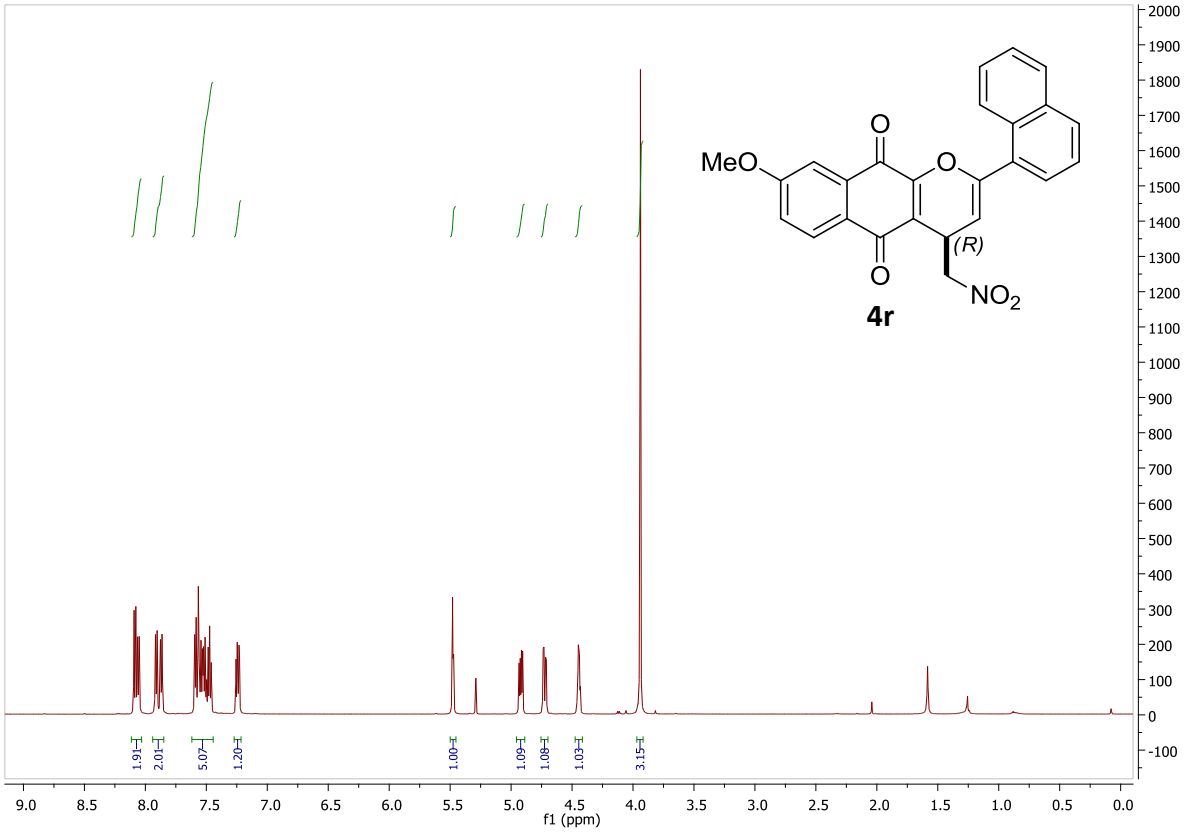

f1 (ppm)

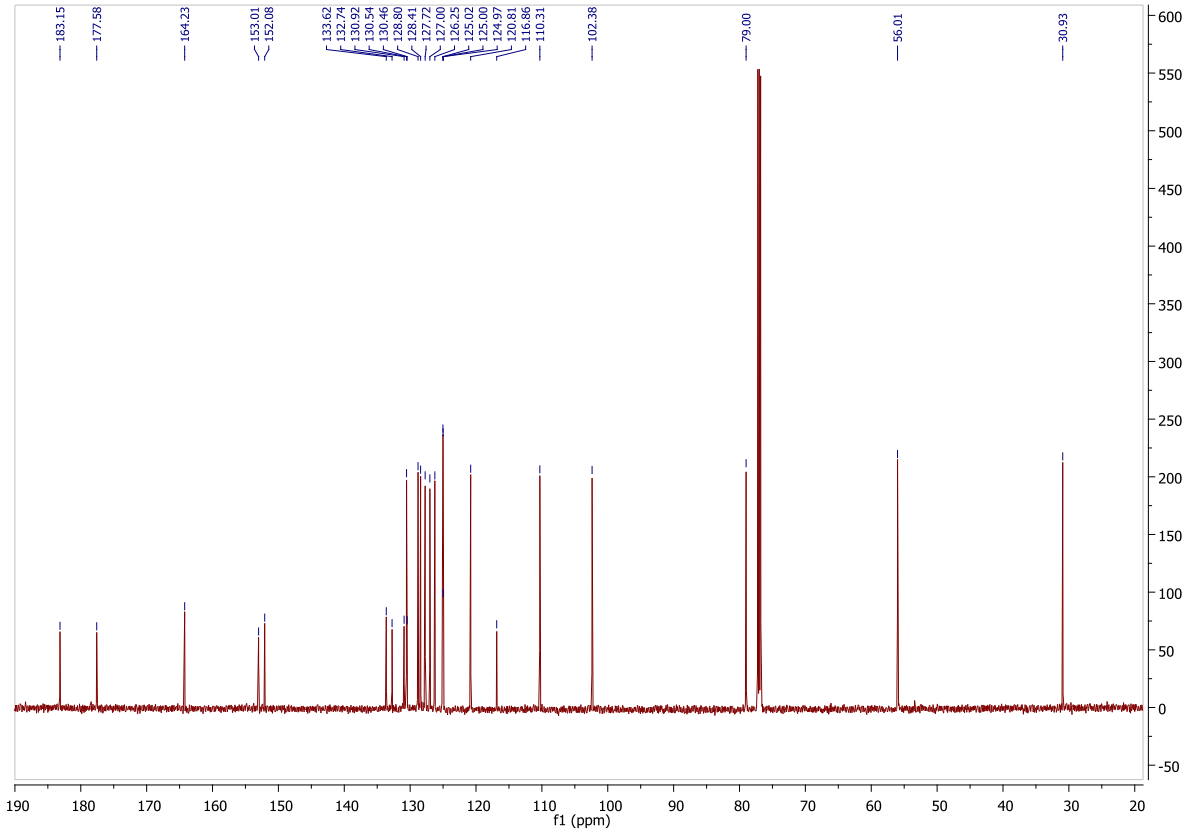

O
H3C
O
(R)
CH3
O
NO2
4p
0.86
1.95
3.01
1.18
1.00
2.17
1.01
3.01
3.00
f1 (ppm)

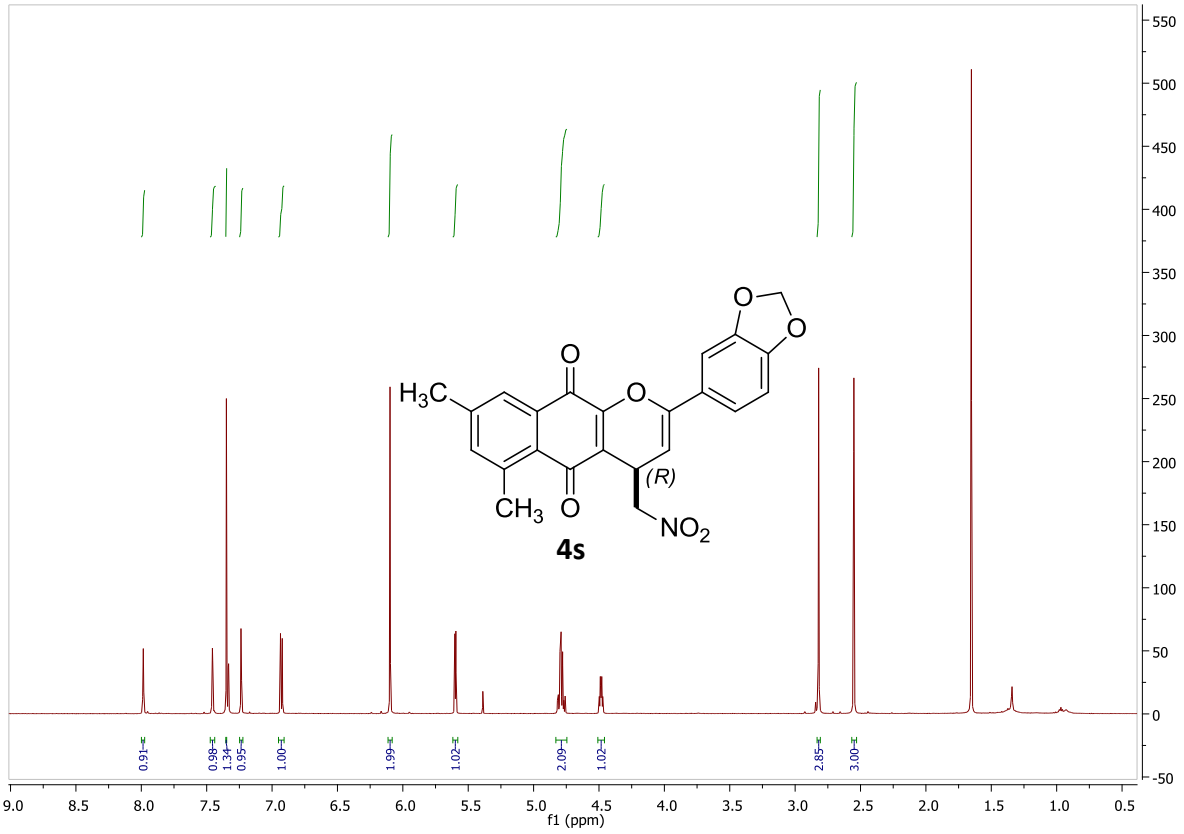

185.50
178.18
151.74
150.49
143.92
141.61
139.30
132.19
131.66
129.73
128.58
126.76
126.18
125.03
117.88
97.01
78.92
77.21
77.00
76.79
30.43
22.80
21.50
f1 (ppm)

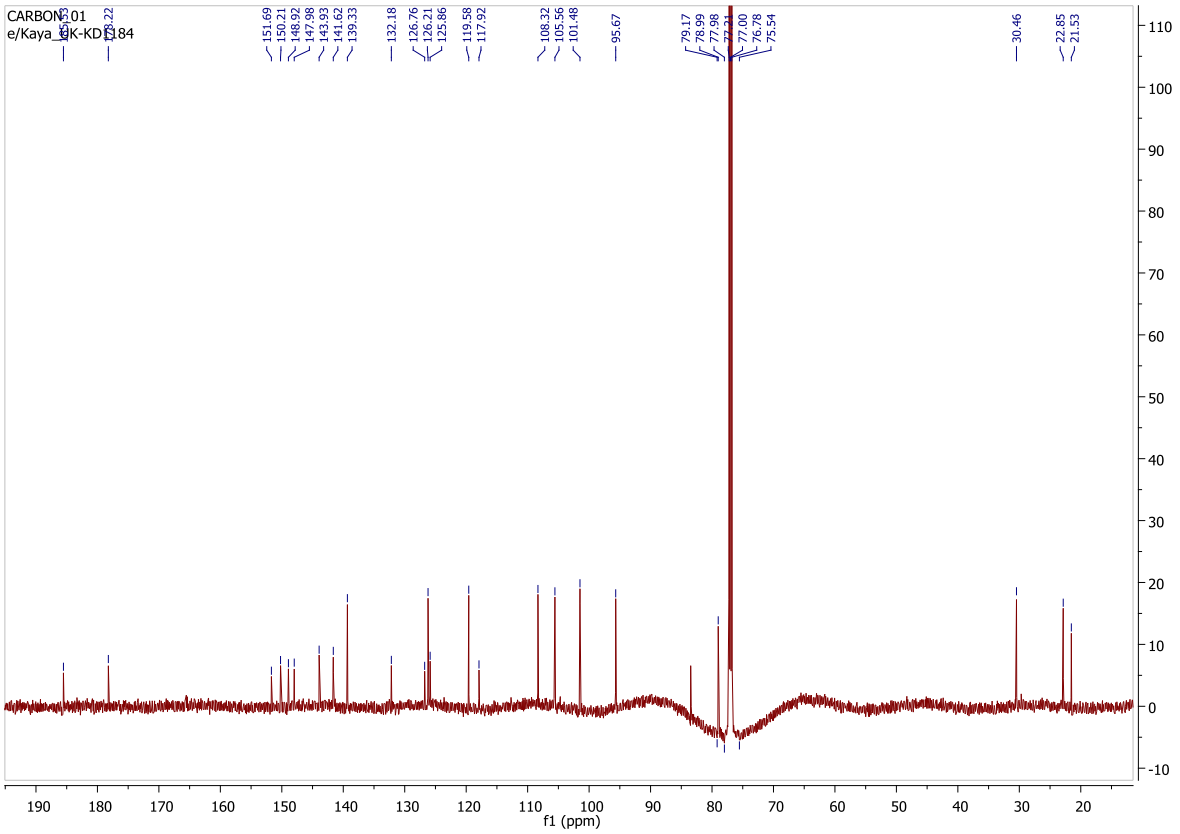

MeO
O
O
O
O
CH3
(R)
NO2
4q
0.92
0.89
1.87
1.02
1.99
1.00
2.02
0.99
2.98
3.02
f1 (ppm)

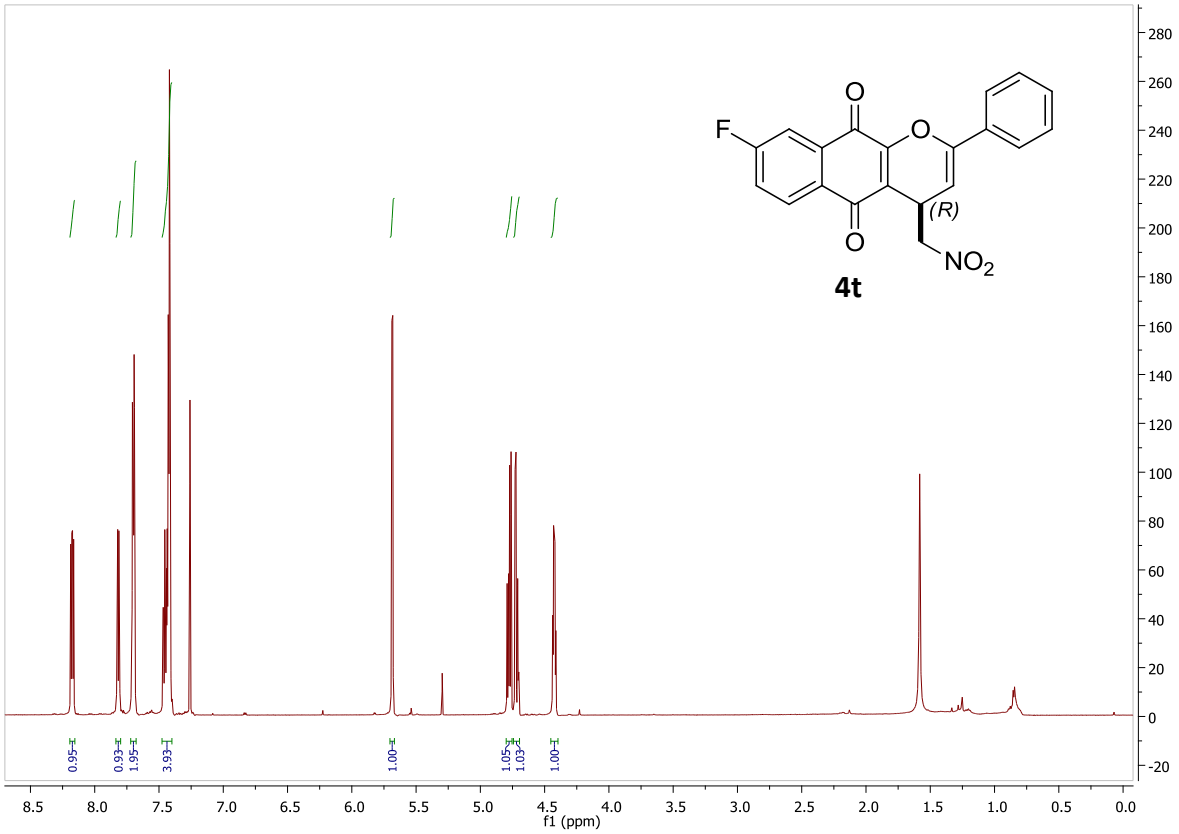

183.08
177.68
164.20
152.80
150.78
138.33
132.72
131.56
130.60
128.83
128.52
125.59
124.97
122.24
120.65
116.83
110.36
96.84
78.85
55.99
30.35
21.47
f1 (ppm)

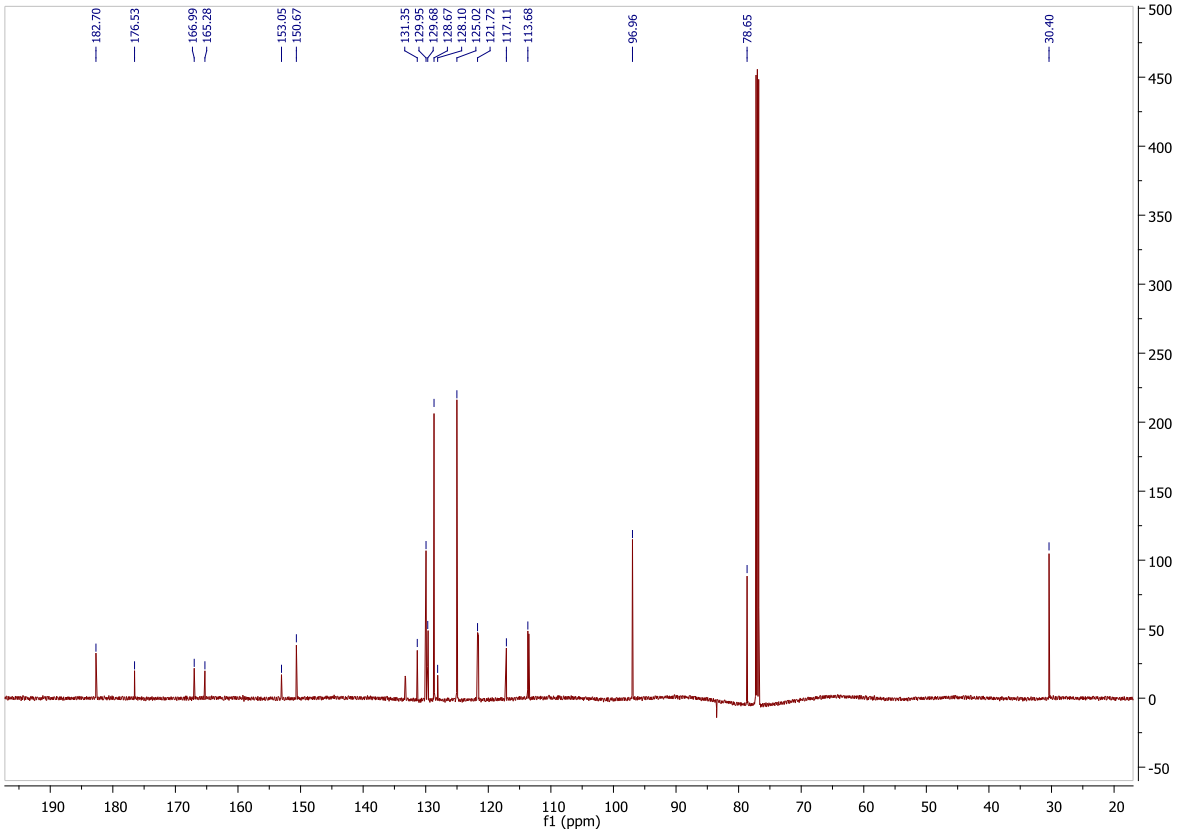

MeO
O
O
O
(R)
NO2
4r
1.91
2.01
5.07
1.20
1.00
1.09
1.08
1.03
3.15
f1 (ppm)


183.15
177.58
164.23
153.01
152.08
133.62
132.74
130.92
130.54
130.46
128.80
128.41
127.72
127.00
126.25
125.02
125.00
124.97
120.81
116.86
110.31
102.38
79.00
56.01
30.93
f1 (ppm)


O
O
O
O
H3C
(R)
CH3
O
NO2
4s
0.91
0.98
1.34
0.95
1.00
1.99
1.02
2.09
1.02
2.85
3.00
f1 (ppm)

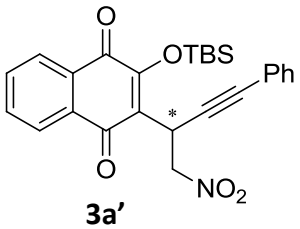

CARBON.01
e/Kaya_GK-KD2184
151.69
150.21
148.92
147.98
143.93
141.62
139.33
132.18
126.76
126.21
125.86
119.58
117.92
108.32
105.56
101.48
95.67
79.17
78.99
77.98
77.00
76.78
75.54
30.46
22.85
21.53
f1 (ppm)


F
O
O
O
(R)
NO2
4t
0.95
0.93
1.95
3.93
1.00
1.05
1.03
1.00
f1 (ppm)


182.70
176.53
166.99
165.28
153.05
150.67
131.35
129.95
129.68
128.67
128.10
125.02
121.72
117.11
113.68
96.96
78.65
30.40
f1 (ppm)

Sample Name: UK 2-165-1 rac
Data file: D:\BERT\UK\165-1R3W.D
Sample Info: Laufmittel: n-Heptan/EtOH 97:3;
Die Probe ist in DCM/LM gelöst

Agilent Technologies



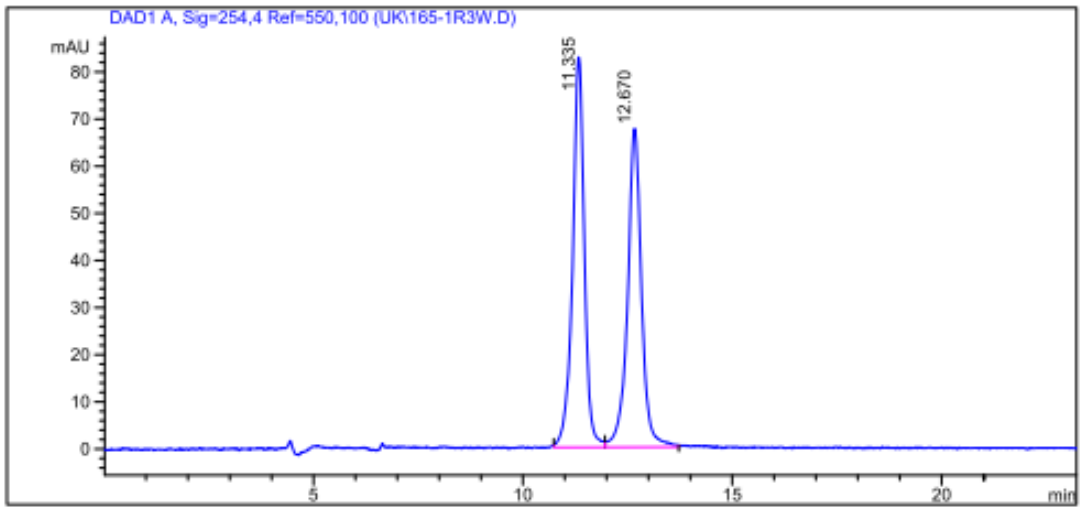
3a'

Säule: WHELK.M
Säuleninfo: (s,s)-Whelk O1 (250x4,6)mm

Operator: Analytik Labor AKEN

Injektion Time: 14:18:19
Injektion Date: 16.03.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	33.6	33.8
Flow in ml/min:	0.7	0.7



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	11.33	0.29	82.73	1617.59	50.85
2	12.67	0.35	67.54	1563.56	49.15
Total				3181.15	100.00

Sample Name: UK 2-276-1
Data file: D:\BERT\UK\276-1W.D
Sample Info: Laufmittel: n-Heptan/EtOH 97:3;
Die Probe ist in DCM/LM gelöst

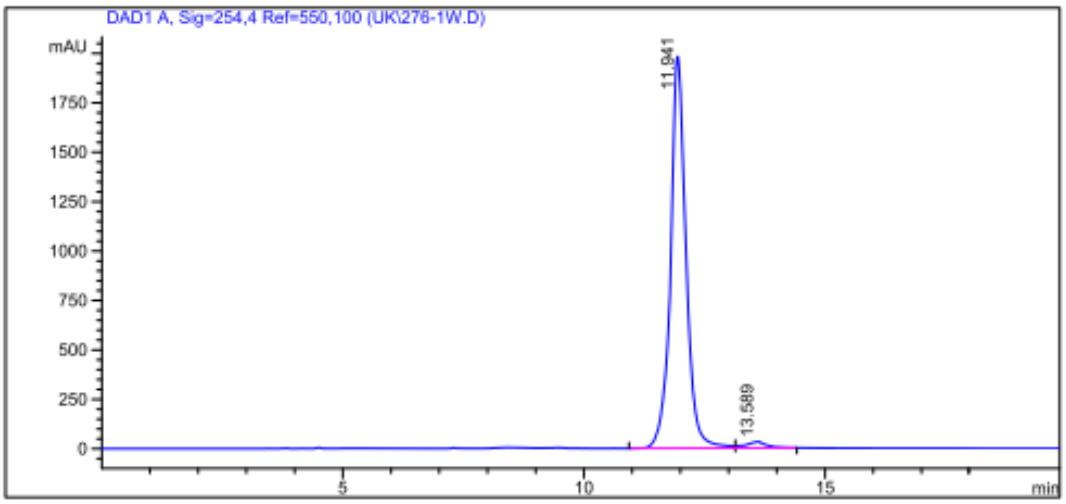
Agilent Technologies

Säule: WHELK.M
Säuleninfo: (s,s)-Whelk O1 (250x4,6)mm

Operator: Analytik Labor AKEN

Injektion Time: 14:36:01
Injektion Date: 24.06.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	34.0	34.4
Flow in ml/min:	0.7	0.7



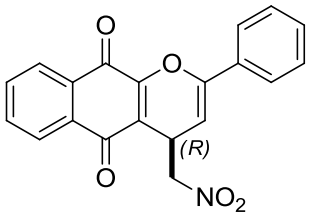
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	11.94	0.33	1981.93	44123.74	97.85
2	13.59	0.44	31.49	970.99	2.15
Total				45094.73	100.00

Sample Name: UK 3-253-1 rac
Data file: D:\GONZO\UK\253-1RIB.D
Sample Info: Laufmittel: n-Heptan/iPrOH 9:1;
Die Probe ist in DCM/LM gelöst.

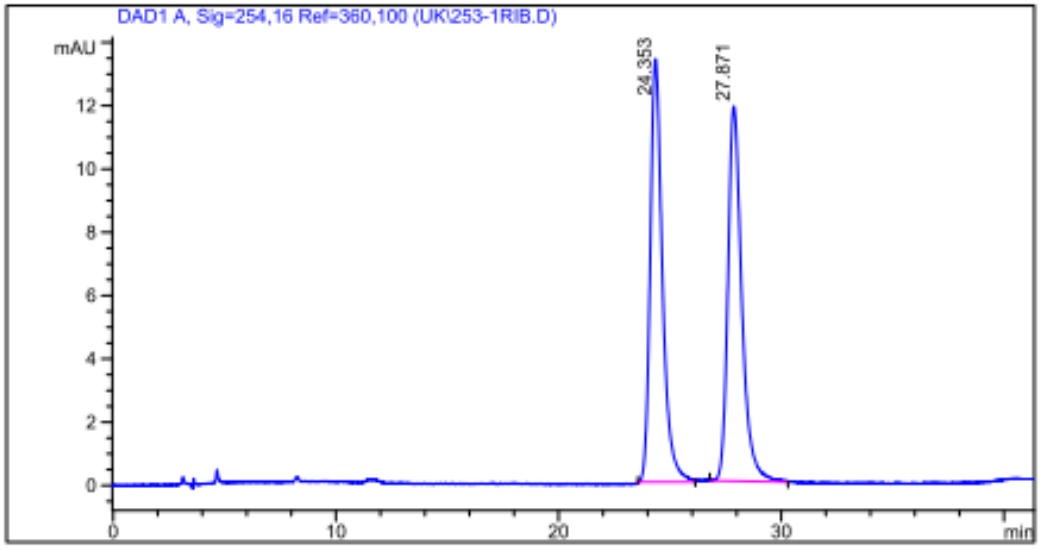
Säule: DAICELIB.M
Säuleninfo: Chiralpak IB (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 10:46:07
Injektion Date: 10.06.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	47.8	48.4
Flow in ml/min:	1.00	1.00



4a



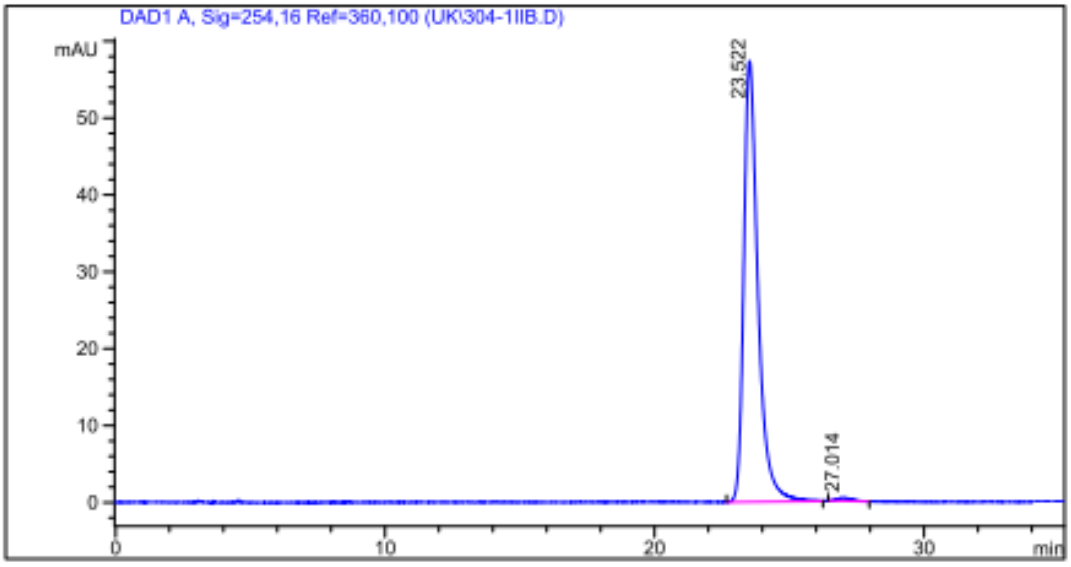
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	24.35	0.63	13.37	508.14	49.82
2	27.87	0.72	11.84	511.88	50.18
Total				1020.01	100.00

Sample Name: UK 4-304-1
Data file: D:\GONZO\UK\304-1IIB.D
Sample Info: Laufmittel: n-Heptan/iPrOH 9:1;
Die Probe ist in DCM/LM gelöst.

Säule: DAICELIB.M
Säuleninfo: Chiralpak IB (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 14:23:59
Injektion Date: 22.07.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	47.0	47.4
Flow in ml/min:	1.00	1.00



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	23.52	0.61	57.37	2101.58	99.23
2	27.01	0.63	0.43	16.28	0.77
Total				2117.86	100.00

Sample name: KD 1146 A rac

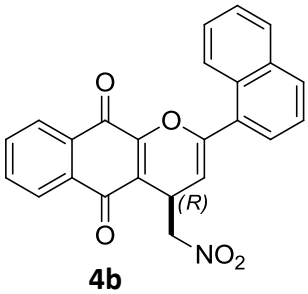
Data file: C:\SNOOPY\KD\KD 1146 A RAC IC.D

Description: Laufmittel: n-Heptan/EtOH 7:3; Die Probe ist DCM/LM gelöst.

Injection date: 6/22/2015 3:19:32 PM

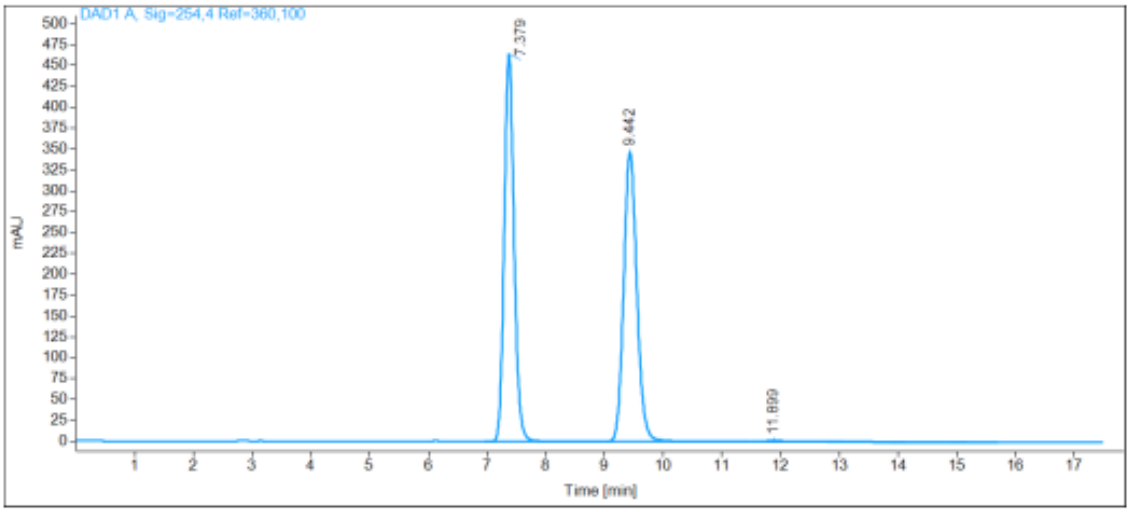
Acq. Analysis method: CHIRALPAKIC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015



4b

Pressure at start: 33 bar **Start flow:** 0.700 ml/min **Column oven:** 30 °C



Name KD 1146 A rac

RT [min]	Type	Area%	Area	Height	Width [min]
7.38	BB	49.75	5405.58	463.90	0.18
9.44	BB	49.70	5401.04	346.59	0.24
11.90	BBA	0.55	59.65	1.61	0.51
	Sum	100.00	10866.28		

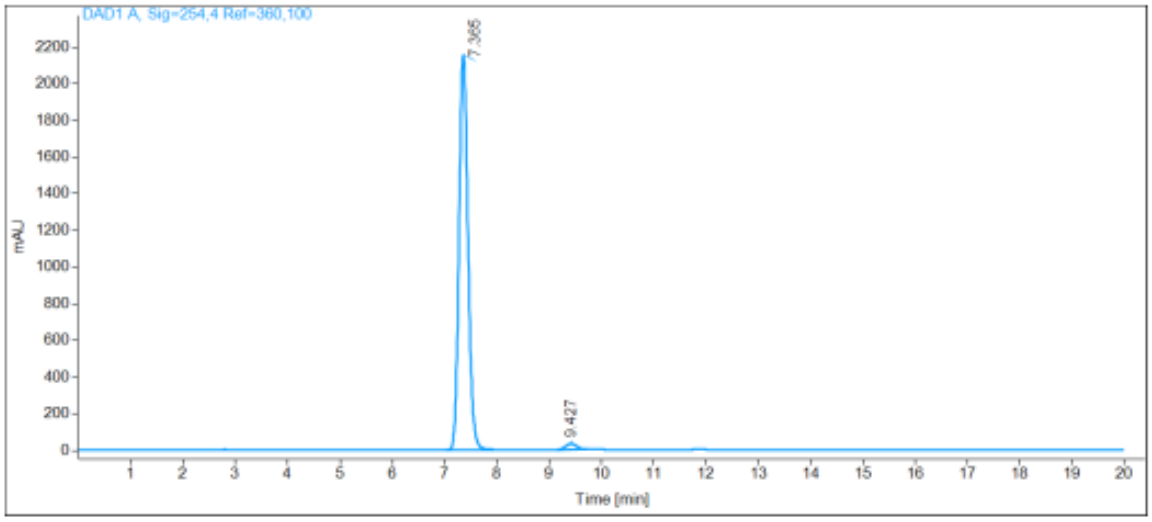
Sample name: **UK 4-312-1**

Data file: C:\SNOOPY\UK\UK 4-312-1 IC.D
Description: Laufmittel: n-Heptant/EtOH 7:3; Die Probe ist DCM/LM gelöst.

Injection date: 7/29/2015 7:47:25 AM
Acq. Analysis method: CHIRALPAKIC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 32 bar **Start flow:** 0.700 ml/min **Column oven:** 29.98 °C

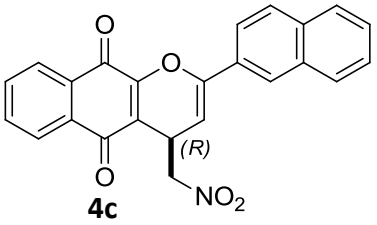


Name UK 4-312-1

RT [min]	Type	Area%	Area	Height	Width [min]
7.36	MM	98.03	25717.09	2159.38	0.20
9.43	MM	1.97	515.91	33.82	0.25
	Sum	100.00	26233.01		

Sample Name: KD 1148 A rac
Data file: D:\ERNIE\KD\1148R2AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

Agilent Technologies



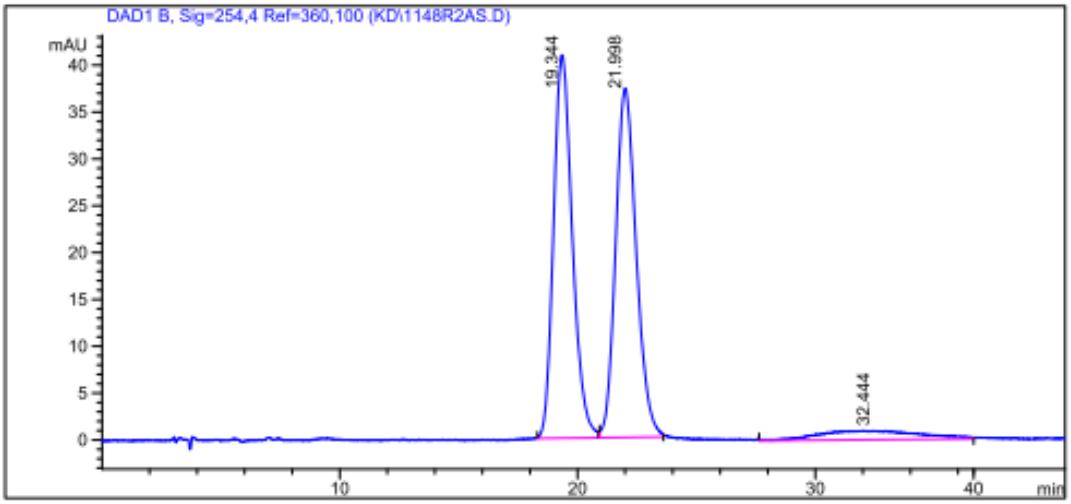
4c

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 14:25:59
Injektion Date: 23.06.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	34.0	34.3
Flow in ml/min:	1.0	1.0



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	19.34	0.72	40.92	2249.72	46.82
2	22.00	0.71	37.26	2244.30	46.71
3	32.44	5.23	0.99	310.83	6.47
Total				4804.85	100.00

Sample Name: UK 4-311-1
Data file: D:\BERT\UK\311-1IAS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

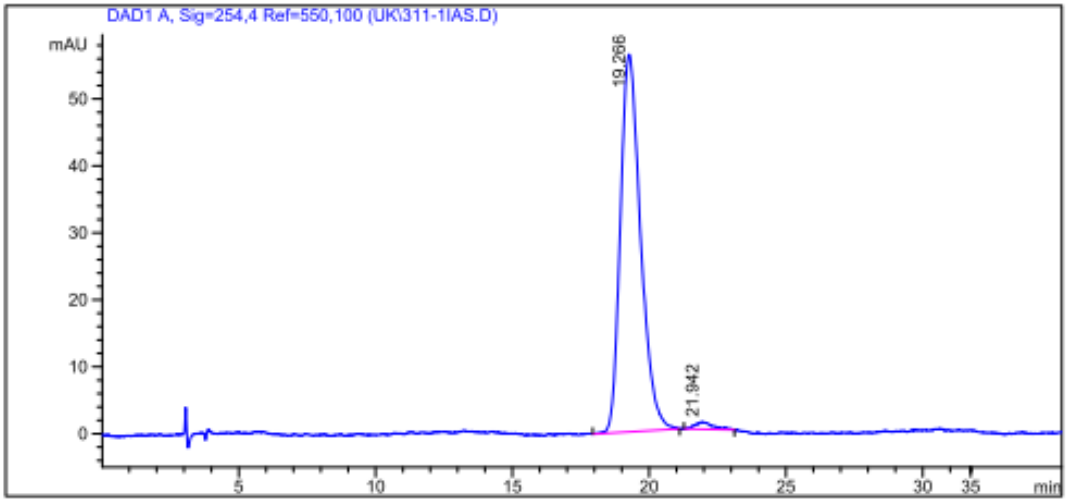
Agilent Technologies

Säule: DAICELAS.M
Säuleninfo: (250x4,6)mm

Operator: Analytik Labor AKEN

Injektion Time: 10:22:57
Injektion Date: 29.07.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	30.1	30.1
Flow in ml/min:	1.0	1.0



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	19.27	0.86	56.39	2894.51	98.32
2	21.94	0.74	1.12	49.42	1.68
Total				2943.92	100.00

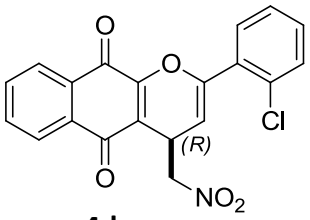
Sample name: KD 1142 A rac

Data file: C:\SNOOPY\KD\1142ARNIC.D

Description: Laufmittel: n-Heptan/EtOH 7:3;
Probe ist in LM/DCM gelöst

Injection date: 6/18/2015 11:29:19 AM

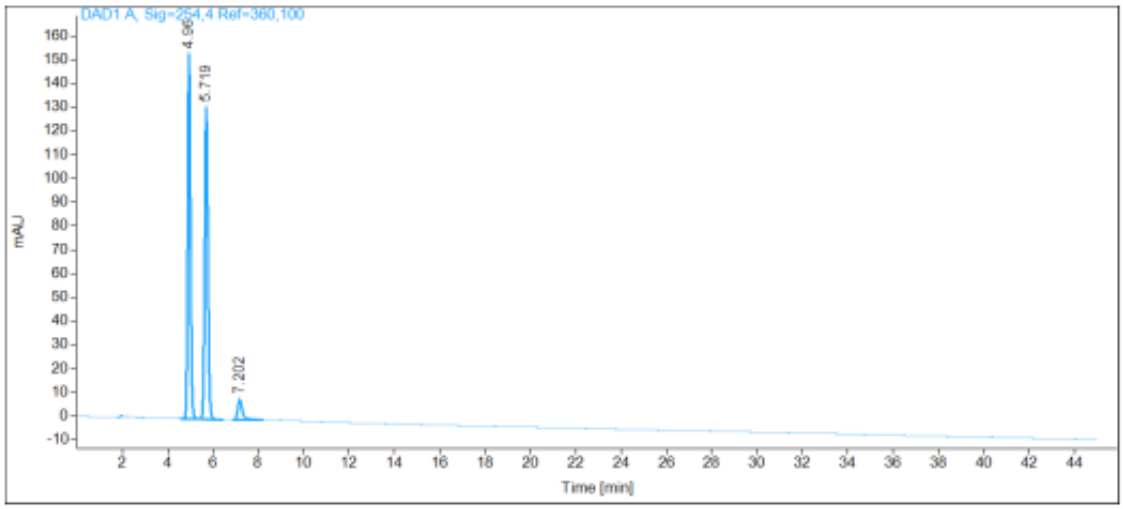
Acq. Analysis method: CHIRALPAKIC1-6LNP.M



4d

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 45 bar **Start flow:** 1.000 ml/min **Column oven:** 29.98 °C



Name KD 1142 A rac

RT [min]	Type	Area%	Area	Height	Width [min]
4.96	BB	47.64	1279.18	153.34	0.13
5.72	BB	47.59	1277.93	130.92	0.15
7.20	BB	4.77	127.99	8.43	0.23
	Sum	100.00	2685.10		

Sample name: **UK 4-317-1**

Data file: C:\SNOOPY\UK\4-317-1IC.D

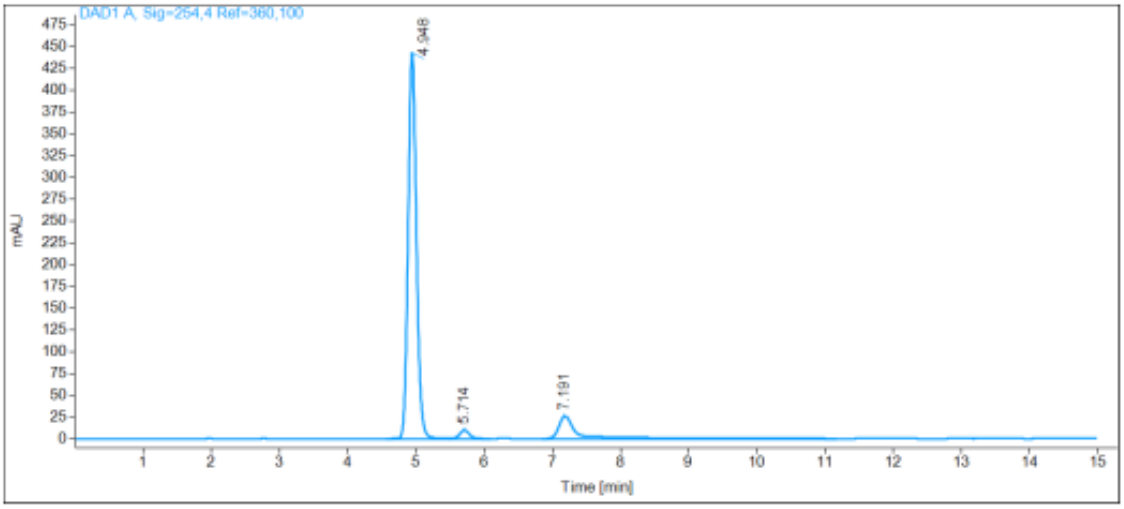
Description: Laufmittel: n-Heptan/EtOH 7:3;
Probe ist in LM/DCM gelöst

Injection date: 8/19/2015 8:20:09 AM

Acq. Analysis method: CHIRALPAKIC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 46 bar **Start flow:** 1.000 ml/min **Column oven:** 30.01 °C



Name UK 4-317-1

RT [min]	Type	Area%	Area	Height	Width [min]
4.95	BV	85.44	3735.24	442.77	0.13
5.71	VB	2.18	95.30	9.57	0.15
7.19	BB	12.38	541.01	26.07	0.29
	Sum	100.00	4371.55		

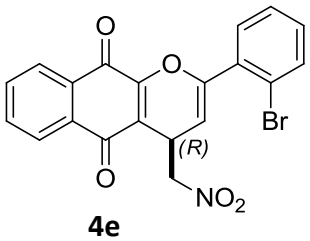
Sample name: KD 1145 A rac

Data file: C:\SNOOPY\KD\KD 1145 A RAC IC.D

Description: Laufmittel: n-Heptan/EtOH 7:3; Die Probe ist DCM/LM gelöst.

Injection date: 6/19/2015 11:47:34 AM

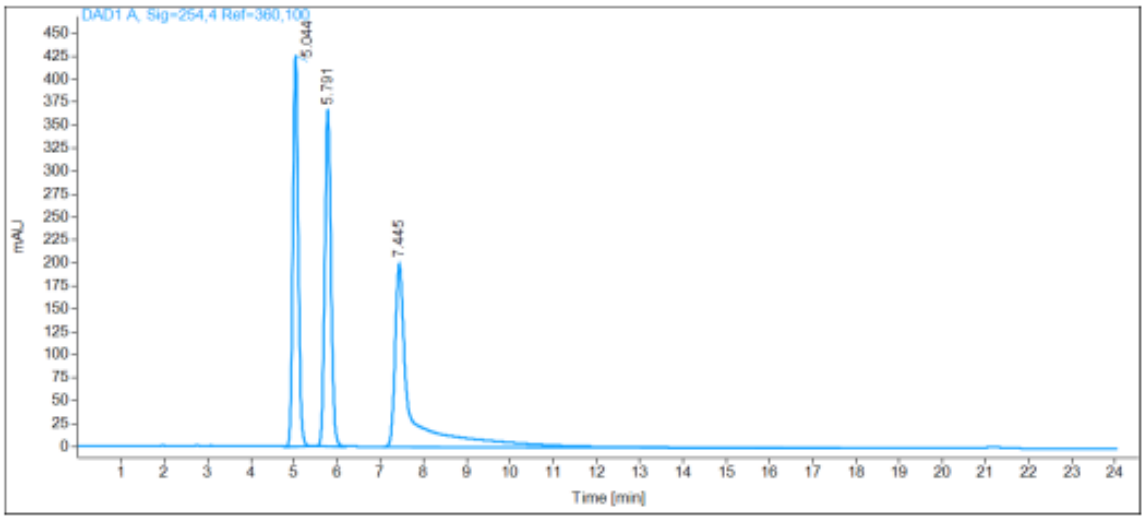
Acq. Analysis method: IC9107.M



4e

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 46 bar **Start flow:** 1.000 ml/min **Column oven:** 29.99 °C



Name KD 1145 A rac

RT [min]	Type	Area%	Area	Height	Width [min]
5.04	BB	30.00	3651.36	426.17	0.13
5.79	BV	29.97	3647.87	366.16	0.16
7.45	VB	40.04	4873.50	199.23	0.34
	Sum	100.00	12172.73		

Sample name: **UK 4-316-1**

Data file: C:\SNOOPY\UK\4-316-1IC.D

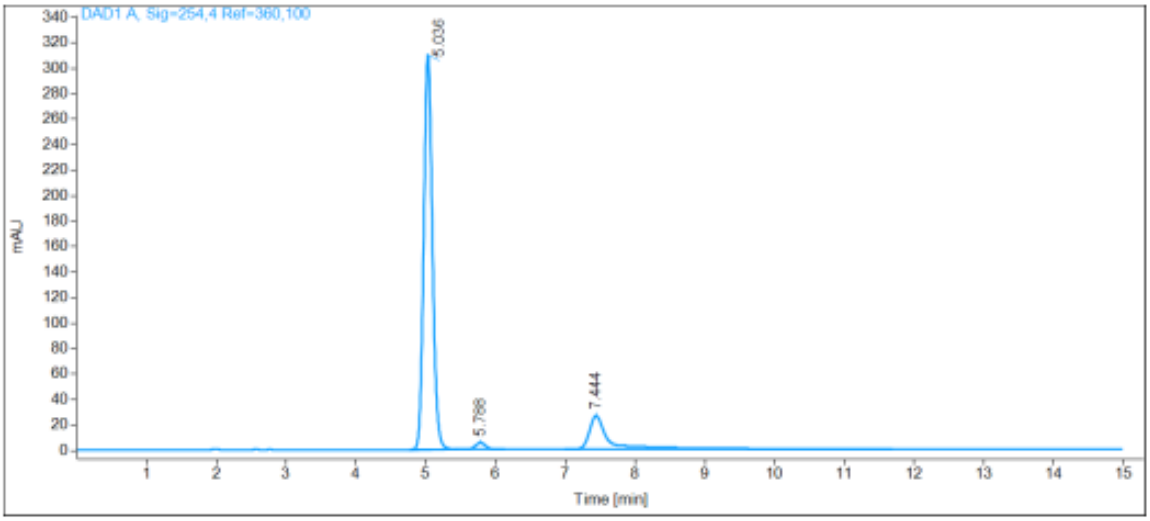
Description: Laufmittel: n-Heptan/EtOH 7:3;
Probe ist in LM/DCM gelöst

Injection date: 8/19/2015 8:03:42 AM

Acq. Analysis method: CHIRALPAKIC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 46 bar **Start flow:** 1.000 ml/min **Column oven:** 30.02 °C



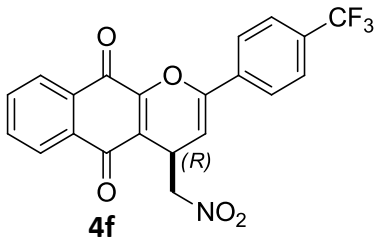
Name UK 4-316-1

RT [min]	Type	Area%	Area	Height	Width [min]
5.04	BB	81.13	2666.35	309.81	0.13
5.79	BB	1.58	51.92	5.15	0.16
7.44	BB	17.29	568.40	26.26	0.30
	Sum	100.00	3286.68		

Sample name: KD 1144 A rac

Data file: C:\SNOOPY\KD\KD 1144 A RAC IC.D

Description: Laufmittel: n-Heptan/EtOH 7:3; Die Probe ist DCM/LM gelöst.



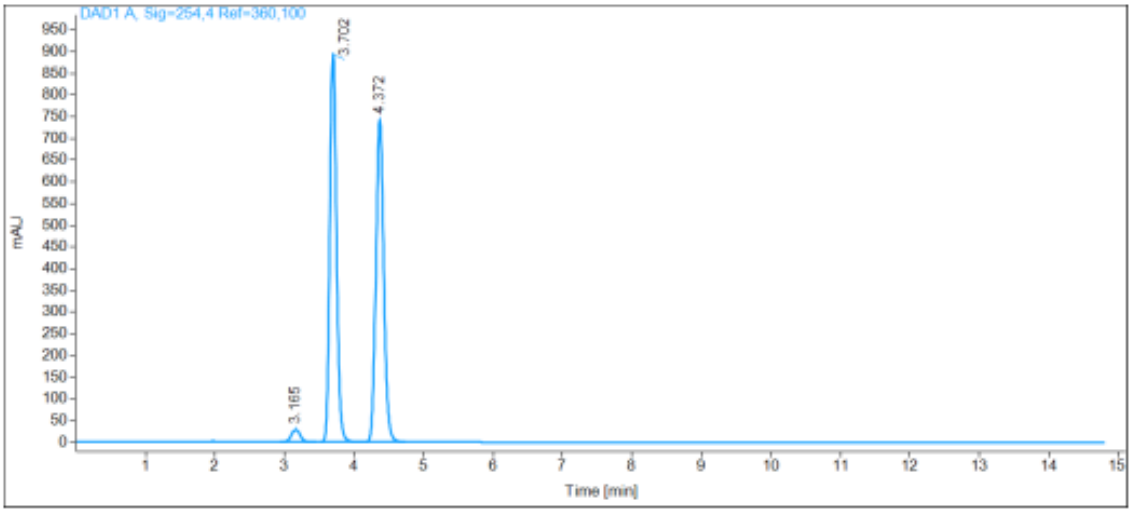
4f

Injection date: 6/19/2015 11:29:50 AM

Acq. Analysis method: IC9107.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 46 bar **Start flow:** 1.000 ml/min **Column oven:** 30.01 °C



Name KD 1144 A rac

RT [min]	Type	Area%	Area	Height	Width [min]
3.17	VB	2.14	251.79	27.26	0.15
3.70	BB	48.97	5755.40	895.27	0.10
4.37	BV	48.89	5746.23	743.51	0.12
	Sum	100.00	11753.42		

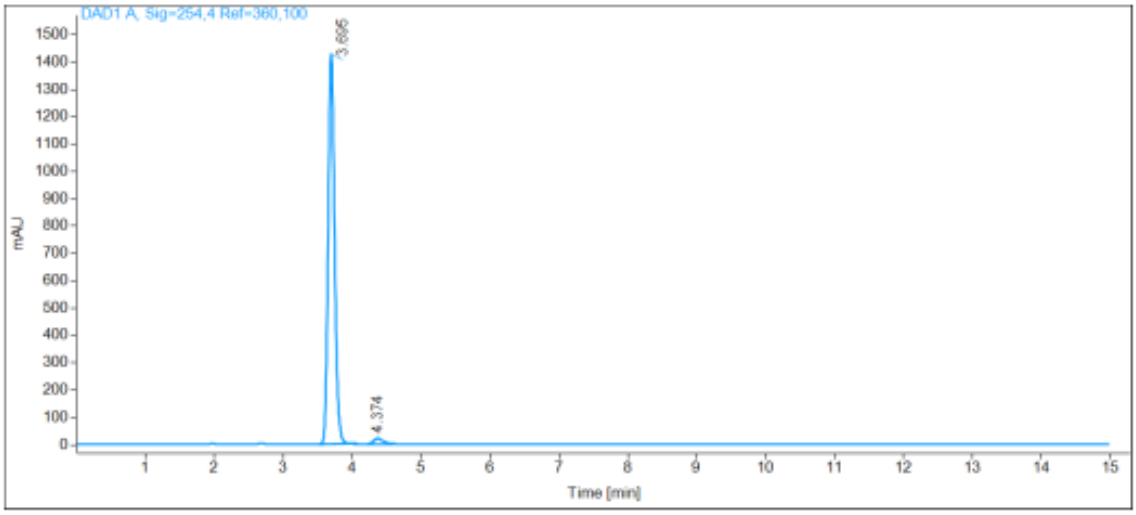
Sample name: **UK 4-310-1**

Data file: C:\SNOOPY\UK\UK 4-310-1 IC.D
Description: Laufmittel: n-Heptant/EtOH 7:3; Die Probe ist DCM/LM gelöst.

Injection date: 7/29/2015 8:19:54 AM
Acq. Analysis method: CHIRALPAKIC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 46 bar **Start flow:** 1.000 ml/min **Column oven:** 29.98 °C



Name UK 4-310-1

RT [min]	Type	Area%	Area	Height	Width [min]
3.70	MM	97.79	9235.16	1432.63	0.11
4.37	MM	2.21	208.40	21.19	0.16
	Sum	100.00	9443.56		

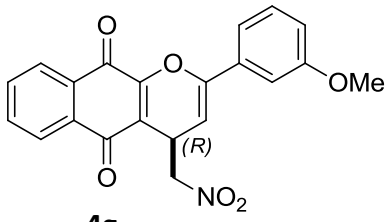
Sample Name: KD 1141 A rac
Data file: D:\ERNIE\KD\1141RAS.D
Sample Info: Laufmittel: n-Heptan/EtOH 7:3;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

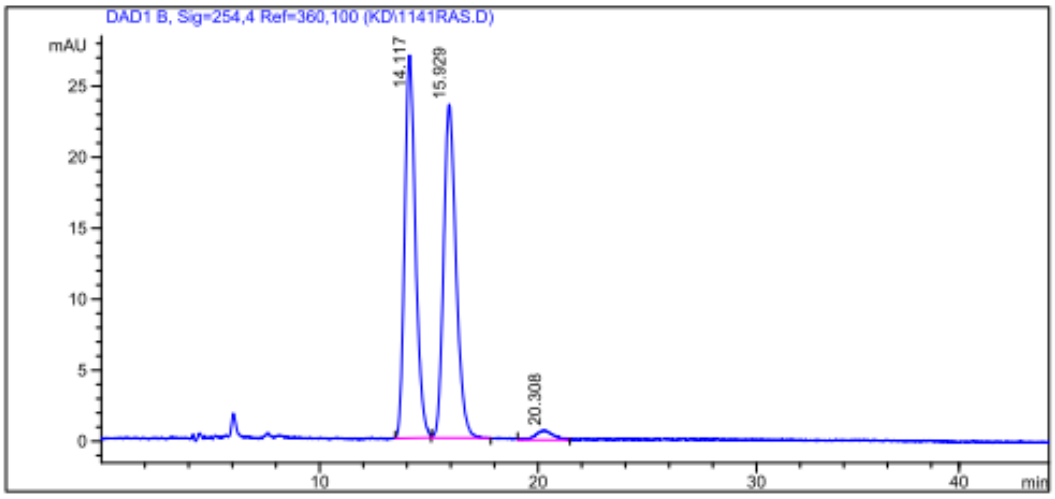
Injektion Time: 10:05:00
Injektion Date: 17.06.2015

Agilent Technologies



4g

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	30.4	30.8
Flow in ml/min:	0.7	0.7



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	14.12	0.50	26.96	924.78	48.84
2	15.93	0.66	23.50	929.35	49.08
3	20.31	0.98	0.67	39.36	2.08
Total				1893.49	100.00

Sample Name: UK 4-309-1
Data file: D:\BERT\UK\309-1AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 7:3;
Die Probe ist in DCM/LM gelöst

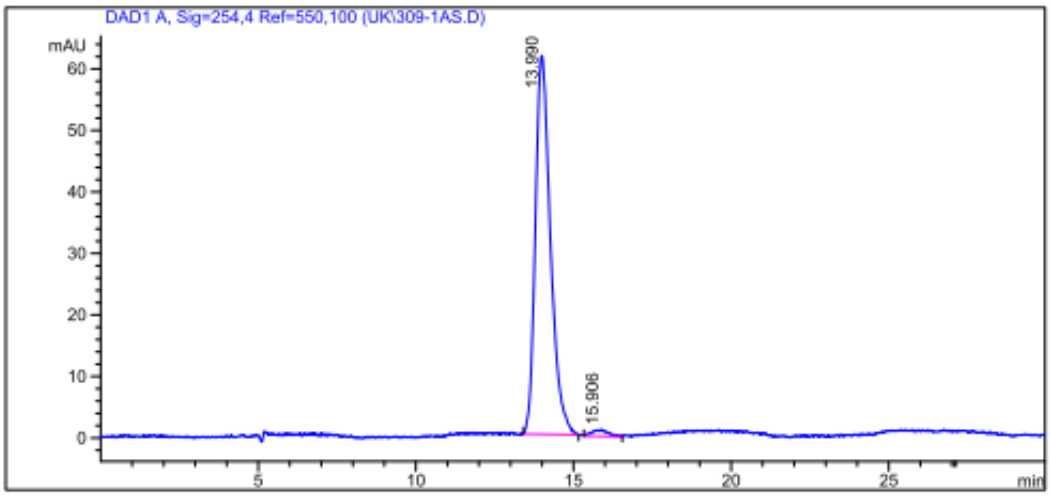
Agilent Technologies

Säule: DAICELAS.M
Säuleninfo: (250x4,6)mm

Operator: Analytik Labor AKEN

Injektion Time: 07:39:47
Injektion Date: 28.07.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	28.2	28.0
Flow in ml/min:	0.7	0.7



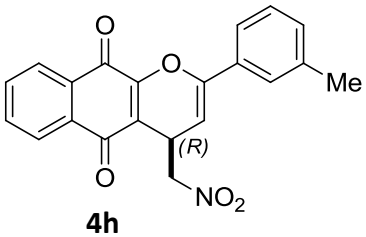
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	13.99	0.46	61.59	2047.00	98.12
2	15.91	0.59	1.10	39.12	1.88
Total				2086.12	100.00

Sample Name: KD 1140 A rac
Data file: D:\ERNIE\KD\1140RAS.D
Sample Info: Laufmittel: n-Heptan/EtOH 7:3;
Die Probe ist in DCM/LM gelöst

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

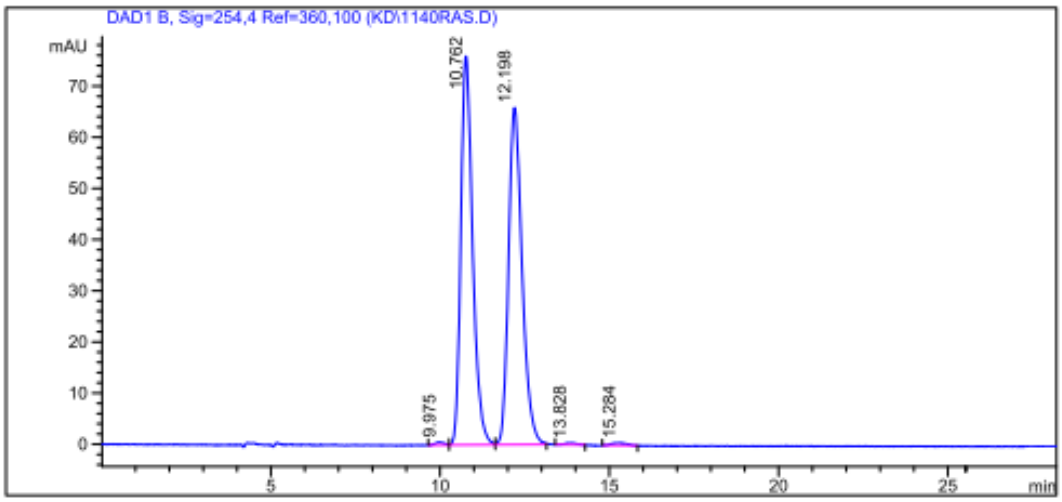
Operator: Analytik Labor AKEN

Injektion Time: 08:46:19
Injektion Date: 16.06.2015



4h

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	33.2	32.0
Flow in ml/min:	0.7	0.7



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	9.98	0.25	0.56	10.43	0.28
2	10.76	0.36	76.02	1834.66	49.53
3	12.20	0.42	65.87	1835.39	49.55
4	13.83	0.41	0.35	8.56	0.23
5	15.28	0.50	0.51	15.33	0.41
Total				3704.36	100.00

Sample Name: UK 4-308-1
Data file: D:\BERT\UK\308-1AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 7:3;
Die Probe ist in DCM/LM gelöst

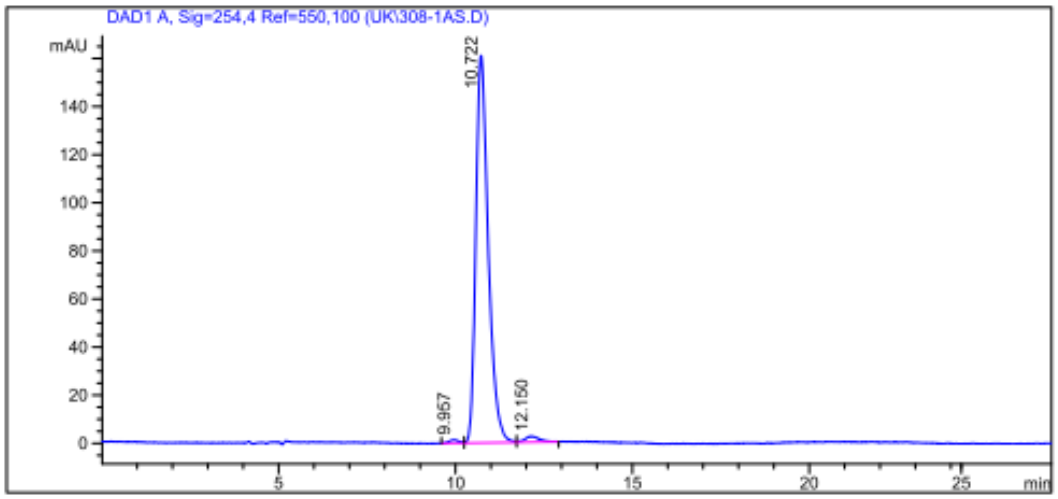
Agilent Technologies

Säule: DAICELAS.M
Säuleninfo: (250x4,6)mm

Operator: Analytik Labor AKEN

Injektion Time: 10:20:29
Injektion Date: 27.07.2015

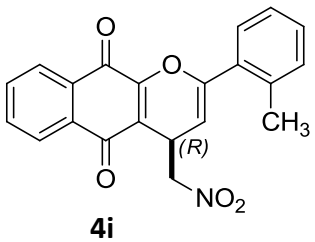
Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	28.2	28.4
Flow in ml/min:	0.7	0.7



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	9.96	0.24	1.45	26.87	0.67
2	10.72	0.37	161.10	3921.00	97.54
3	12.15	0.36	2.43	72.11	1.79
Total				4019.98	100.00

Sample Name: UK KD 1178 rac
Data file: D:\BERT\UK\1178RAS.D
Sample Info: Laufmittel: n-Heptan/EtOH 7:3;
Die Probe ist in DCM/LM gelöst

Agilent Technologies



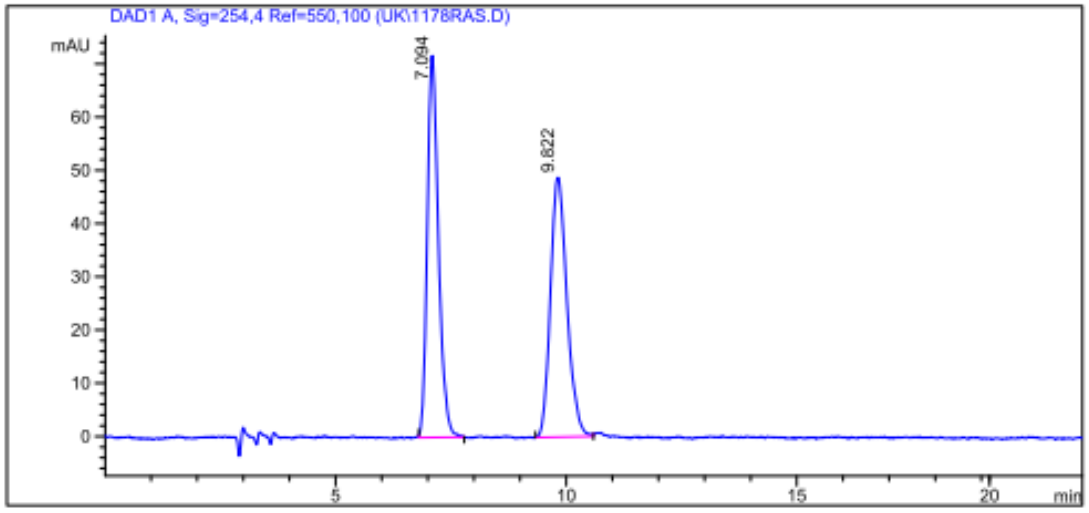
4i

Säule: DAICELAS.M
Säuleninfo: (250x4,6)mm

Operator: Analytik Labor AKEN

Injektion Time: 09:53:56
Injektion Date: 18.08.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	37.0	36.8
Flow in ml/min:	1.0	1.0



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	7.09	0.25	71.84	1209.65	49.95
2	9.82	0.37	48.77	1212.15	50.05
Total				2421.79	100.00

Sample Name: UK 4-320-1
Data file: D:\BERT\UK\4-320AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 7:3;
Die Probe ist in DCM/LM gelöst

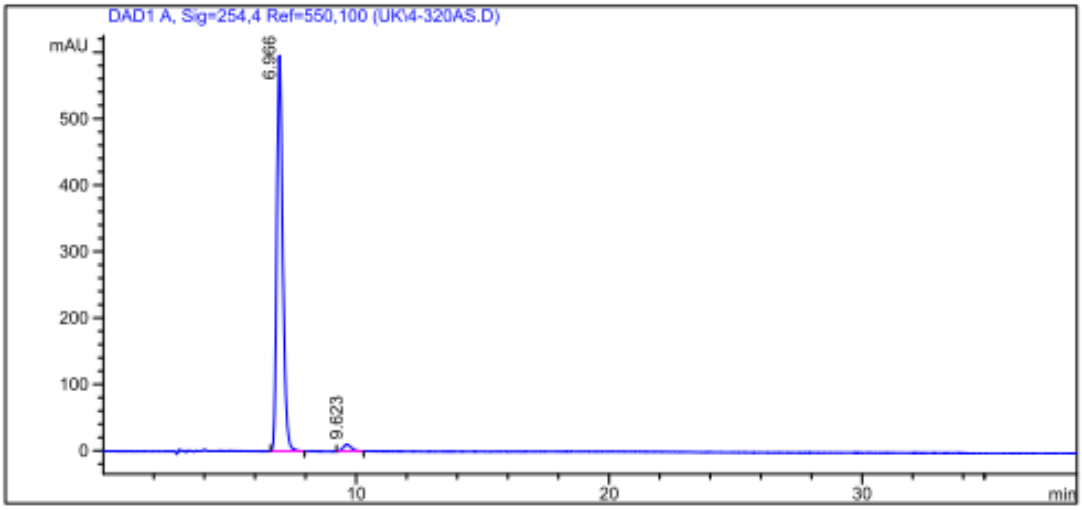
Agilent Technologies

Säule: DAICELAS.M
Säuleninfo: (250x4,6)mm

Operator: Analytik Labor AKEN

Injektion Time: 07:55:15
Injektion Date: 25.08.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	36.5	36.3
Flow in ml/min:	1.0	1.0

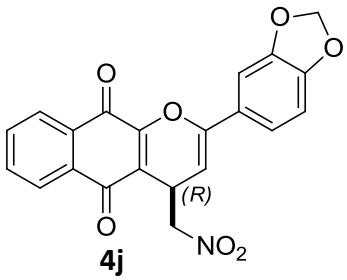


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	6.97	0.25	596.16	9711.88	97.60
2	9.62	0.28	10.26	239.25	2.40
Total				9951.12	100.00

Sample name: KD 1143 A rac

Data file: C:\SNOOPY\KD\KD 1143 A RAC IC.D

Description: Laufmittel: n-Heptan/EtOH 7:3; Die Probe ist DCM/LM gelöst.



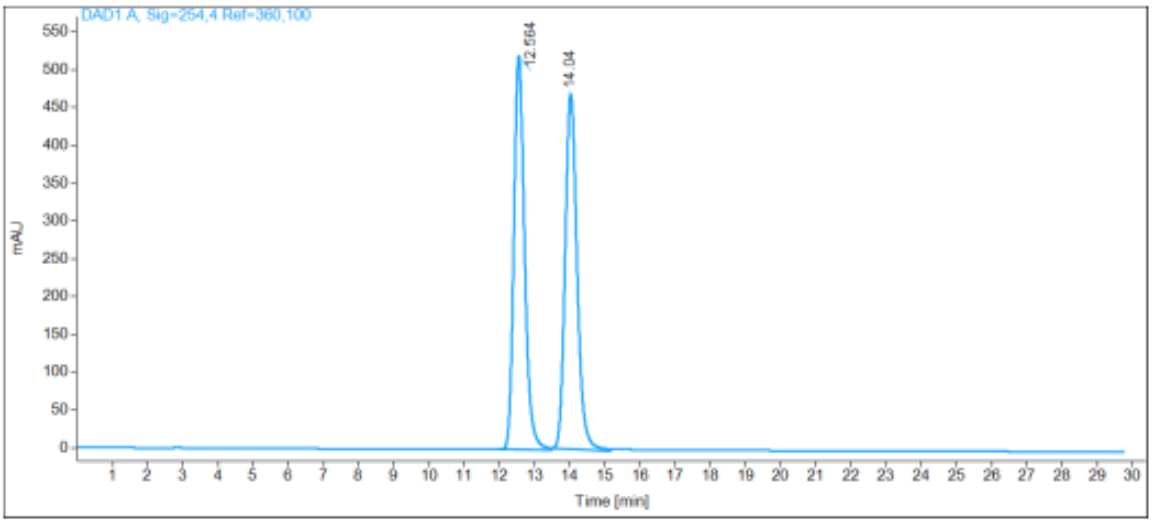
4j

Injection date: 6/18/2015 8:04:02 AM

Acq. Analysis method: CHIRALPAKIC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 32 bar **Start flow:** 0.700 ml/min **Column oven:** 29.97 °C



Name KD 1143 A rac

RT [min]	Type	Area%	Area	Height	Width [min]
12.56	BV	49.88	10891.71	520.58	0.32
14.04	MM	50.12	10946.24	469.21	0.39
	Sum	100.00	21837.95		

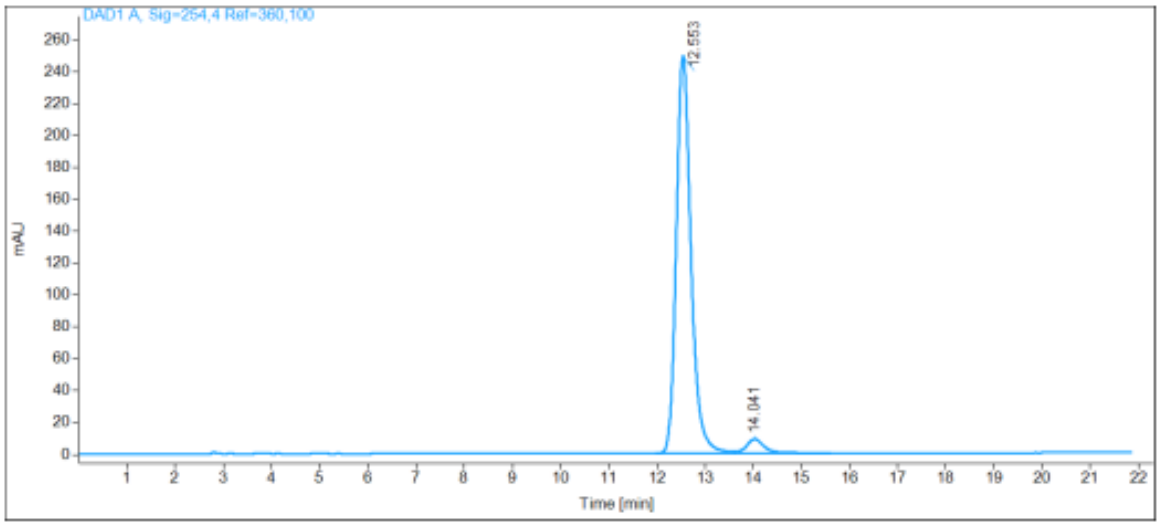
Sample name: **UK 4-318-1**

Data file: C:\SNOOPY\UK\UK 4-318-1 IC.D
Description: Laufmittel: n-Heptant/EtOH 7:3; Die Probe ist DCM/LM gelöst.

Injection date: 8/20/2015 8:40:58 AM
Acq. Analysis method: CHIRALPAKIC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 32 bar **Start flow:** 0.700 ml/min **Column oven:** 30 °C

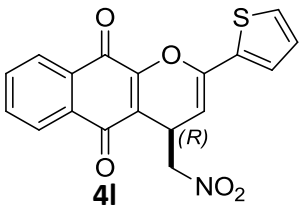


Name UK 4-318-1

RT [min]	Type	Area%	Area	Height	Width [min]
12.55	BV	95.91	5286.02	249.01	0.32
14.04	VB	4.09	225.50	8.66	0.39
	Sum	100.00	5511.53		

Sample Name: KD 1149 A rac
Data file: D:\ERNIE\KD\1149RAS.D
Sample Info: Laufmittel: n-Heptan/EtOH 7:3;
Die Probe ist in DCM/LM gelöst

Agilent Technologies



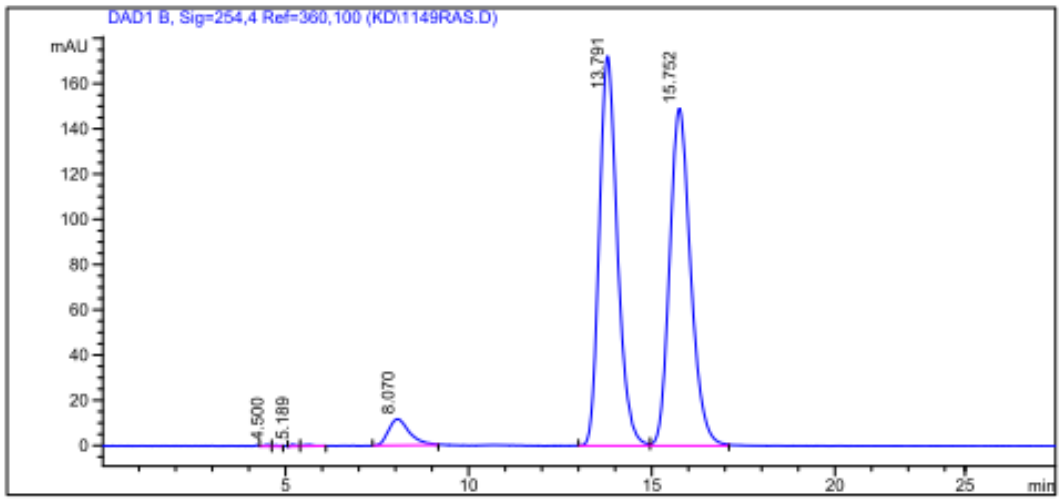
4l

Säule: DAICELAS.M
Säuleninfo: Chiralpak AS (250 x 4.6)mm 10µ

Operator: Analytik Labor AKEN

Injektion Time: 10:50:05
Injektion Date: 24.06.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	32.2	31.0
Flow in ml/min:	0.7	0.7



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.50	0.17	0.69	8.46	0.07
2	4.73	0.17	0.54	7.09	0.06
3	5.19	0.15	0.90	9.87	0.08
4	5.61	0.27	0.59	13.35	0.11
5	8.07	0.55	11.63	467.40	3.78
6	13.79	0.52	172.29	5912.24	47.81
7	15.75	0.61	149.08	5947.13	48.09
Total				12365.55	100.00

Sample Name: UK 4-326-1
Data file: D:\BERT\UK\4-326AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 7:3;
Die Probe ist in DCM/LM gelöst

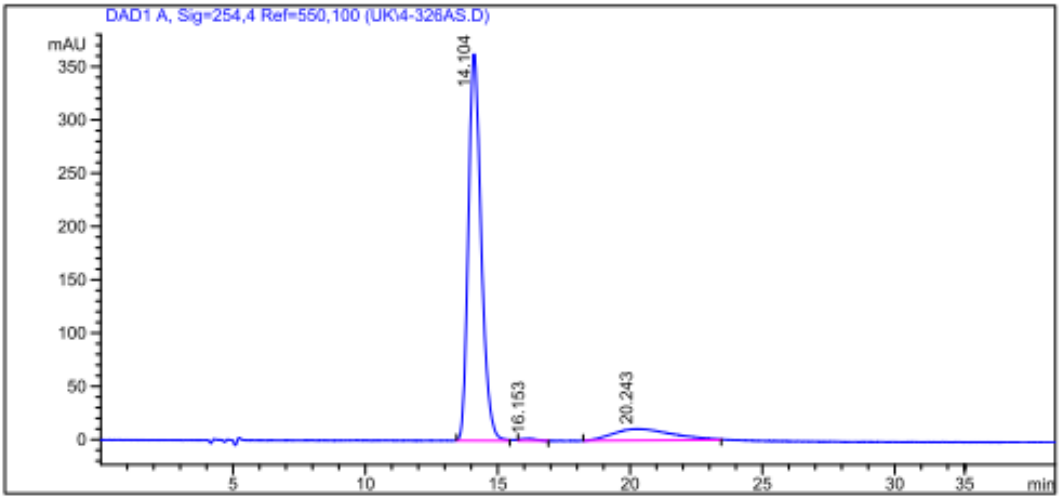
Agilent Technologies

Säule: DAICELAS.M
Säuleninfo: (250x4,6)mm

Operator: Analytik Labor AKEN

Injektion Time: 07:55:29
Injektion Date: 06.10.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	27.2	26.8
Flow in ml/min:	0.7	0.7



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	14.10	0.52	362.86	12270.03	87.33
2	16.15	0.50	1.95	80.04	0.57
3	20.24	1.84	10.83	1700.41	12.10
Total				14050.47	100.00

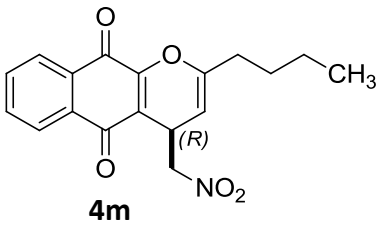
Sample name: KD 1151 A rac

Data file: C:\SNOOPY\KD\1151ARIC.D

Description: Laufmittel: n-Heptan/iPrOH 7:3; Probe ist in LM/DCM gelöst

Injection date: 6/24/2015 11:50:58 AM

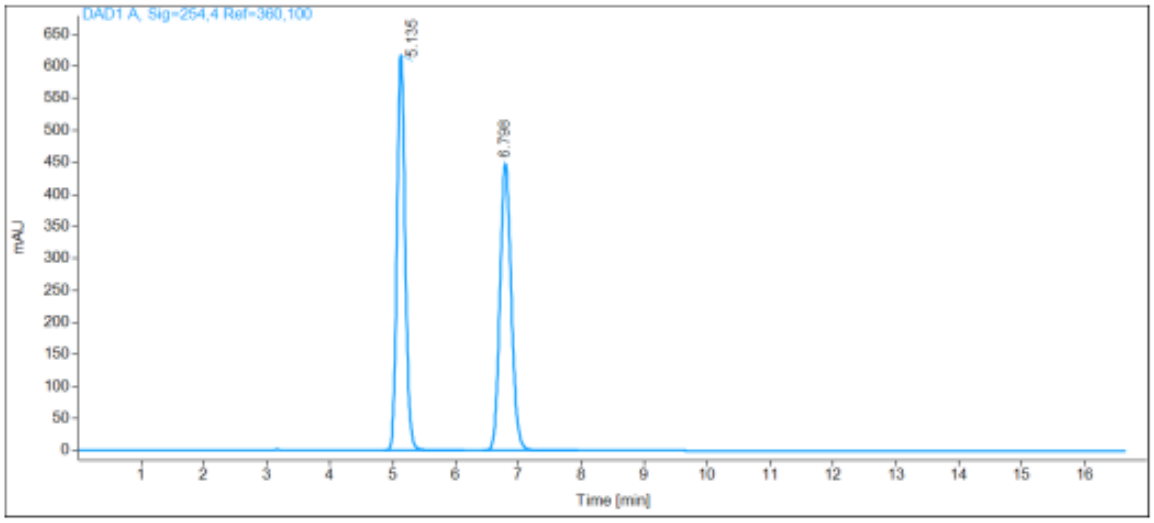
Acq. Analysis method: CHIRALPAKIC1-6LNP.M



4m

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 45 bar **Start flow:** 1.000 ml/min **Column oven:** 29.99 °C



Name KD 1151 A rac

RT [min]	Type	Area%	Area	Height	Width [min]
5.14	BB	50.01	5588.79	617.95	0.14
6.80	BPA	49.99	5587.12	447.92	0.19
	Sum	100.00	11175.91		

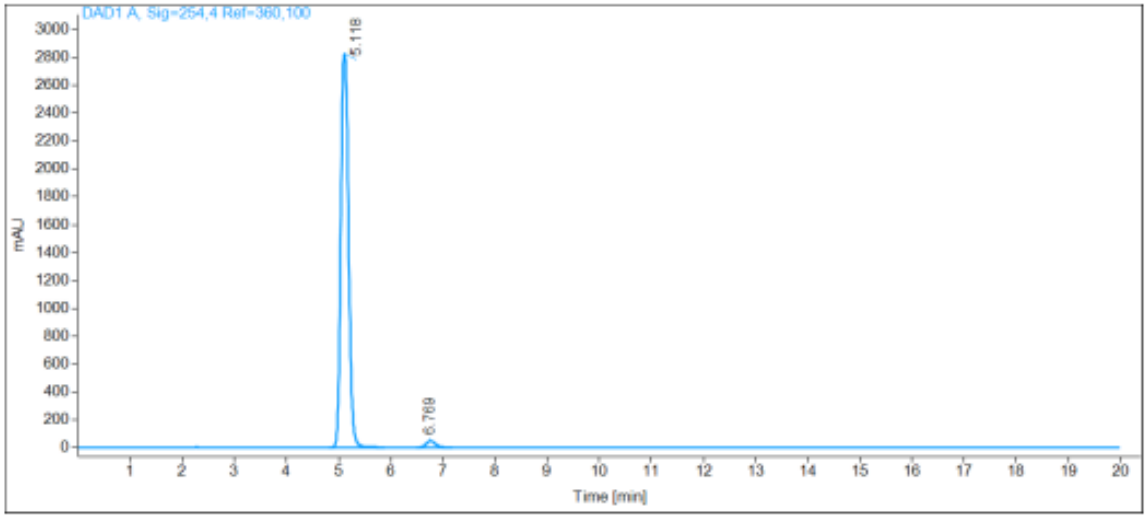
Sample name: UK 4-314-1

Data file: C:\SNOOPY\UK\UK 4-314-1 IC.D
Description: Laufmittel: n-Heptant/iPrOH 7:3; Die Probe ist DCM/LM gelöst.

Injection date: 7/30/2015 8:29:49 AM
Acq. Analysis method: CHIRALPAKIC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 46 bar **Start flow:** 1.000 ml/min **Column oven:** 30 °C



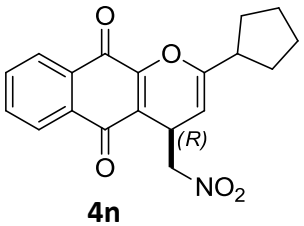
Name UK 4-314-1

RT [min]	Type	Area%	Area	Height	Width [min]
5.12	MM	97.57	29172.92	2825.76	0.17
6.77	MM	2.43	726.94	50.00	0.24
	Sum	100.00	29899.86		

Sample name: KD 1150 A rac

Data file: C:\SNOOPY\KD\KD 1150 A RAC IC.D

Description: Laufmittel: n-Heptan/EtOH 7:3; Die Probe ist DCM/LM gelöst.



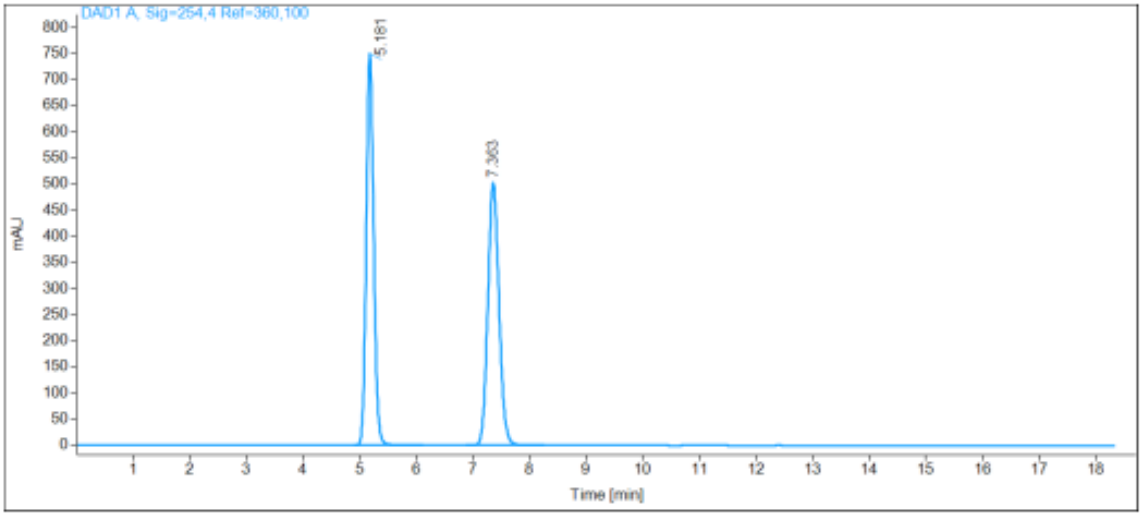
4n

Injection date: 6/24/2015 11:29:29 AM

Acq. Analysis method: CHIRALPAKIC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 45 bar **Start flow:** 1.000 ml/min **Column oven:** 30 °C



Name KD 1150 A rac

RT [min]	Type	Area%	Area	Height	Width [min]
5.18	BB	50.01	6865.90	749.74	0.14
7.36	BP	49.99	6862.79	501.39	0.21
	Sum	100.00	13728.68		

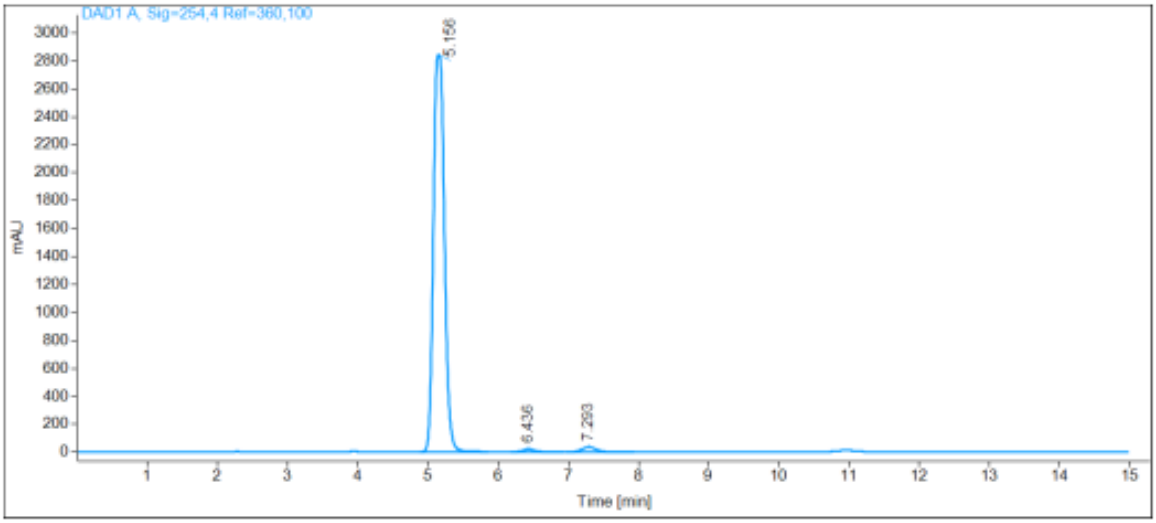
Sample name: **UK 4-313-1**

Data file: C:\SNOOPY\UK\UK 4-313-1 IC.D
Description: Laufmittel: n-Heptant/iPrOH 7:3; Die Probe ist DCM/LM gelöst.

Injection date: 7/30/2015 3:38:24 PM
Acq. Analysis method: CHIRALPAKIC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 46 bar **Start flow:** 1.000 ml/min **Column oven:** 30 °C



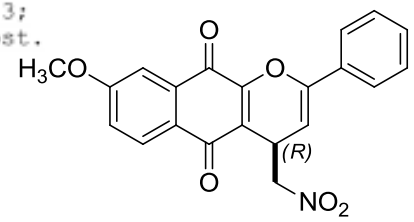
Name UK 4-313-1

RT [min]	Type	Area%	Area	Height	Width [min]
5.16	BB	97.82	30770.85	2841.94	0.17
6.44	BB	0.67	209.83	17.61	0.18
7.29	BP	1.52	477.51	34.73	0.21
	Sum	100.00	31458.19		

Sample Name: UK KD 1179 rac
Data file: D:\GONZO\UK\1179RIB.D
Sample Info: Laufmittel: n-Heptan/EtOH 7:3;
Die Probe ist in DCM/LM gelöst.

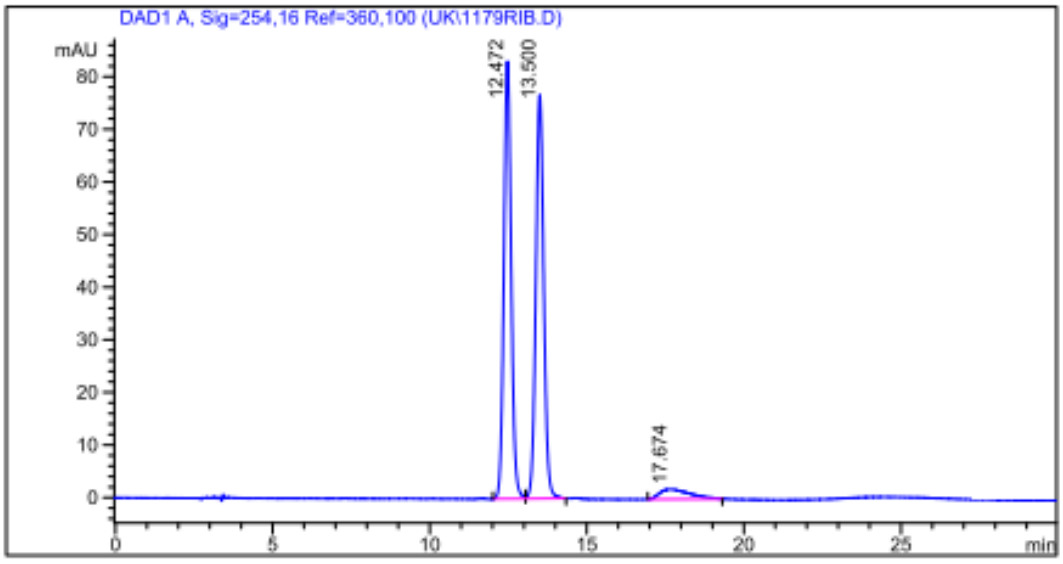
Säule: DAICELIB.M
Säuleninfo: Chiralpak IB (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 10:29:38
Injektion Date: 18.08.2015



4o

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	59.9	60.6
Flow in ml/min:	1.00	1.00



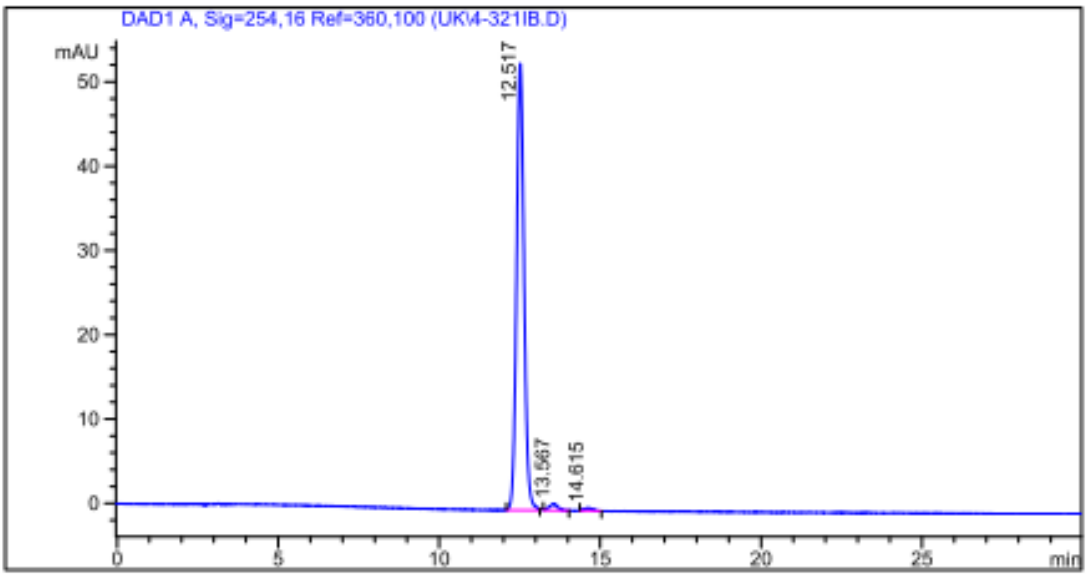
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	12.47	0.25	83.17	1349.47	47.80
2	13.50	0.27	76.74	1342.79	47.56
3	17.67	0.85	1.89	130.88	4.64
Total				2823.15	100.00

Sample Name: UK 4-321-1
Data file: D:\GONZO\UK\4-321IB.D
Sample Info: Laufmittel: n-Heptan/EtOH 7:3;
Die Probe ist in DCM/LM gelöst.

Säule: DAICELIB.M
Säuleninfo: Chiralpak IB (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 08:05:58
Injektion Date: 25.08.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	58.8	60.4
Flow in ml/min:	1.00	1.00

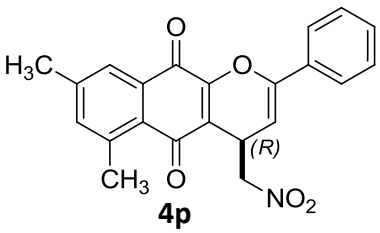


#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	12.52	0.25	53.01	856.34	97.66
2	13.57	0.26	0.76	14.63	1.67
3	14.62	0.23	0.34	5.87	0.67
Total				876.84	100.00

Sample name: **UK KD 1171 rac**

Data file: C:\SNOOPY\UK\UK KD 1171 RAC IC.D

Description: Laufmittel: n-Heptant/EtOH 7:3; Die Probe ist DCM/LM gelöst.



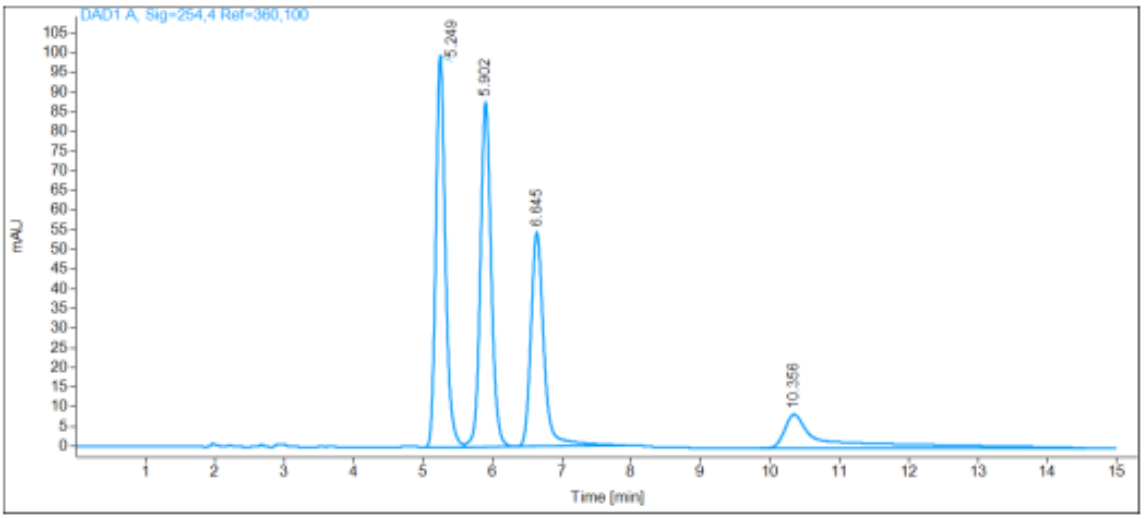
4p

Injection date: 8/18/2015 10:44:40 AM

Acq. Analysis method: CHIRALPAKIC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 46 bar **Start flow:** 1.000 ml/min **Column oven:** 29.99 °C



Name UK KD 1171 rac

RT [min]	Type	Area%	Area	Height	Width [min]
5.25	BV	32.02	947.24	99.54	0.15
5.90	VB	32.01	947.08	87.57	0.17
6.65	BB	23.97	709.03	54.29	0.20
10.36	BB	12.00	354.93	8.51	0.56
	Sum	100.00	2958.28		

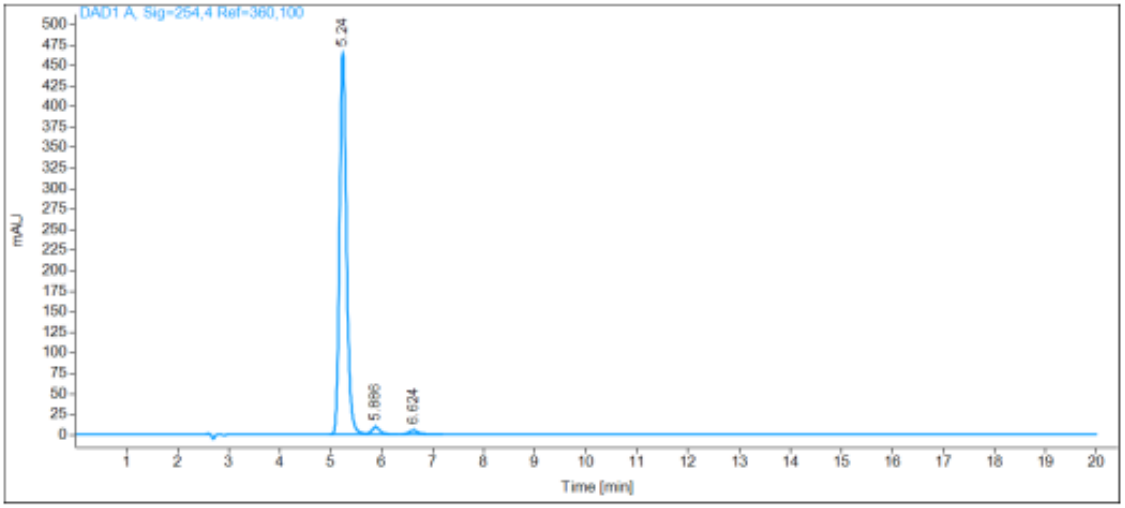
Sample name: **UK 4-322-1**

Data file: C:\SNOOPY\UK\UK 4-322-1 IC.D
Description: Laufmittel: n-Heptant/EtOH 7:3; Die Probe ist DCM/LM gelöst.

Injection date: 8/26/2015 11:01:27 AM
Acq. Analysis method: CHIRALPAKIC1-6LNP.M

Column: Chiralpak IC, (150 x 4,6) mm, 5µ, SN: IC00CD-QF015

Pressure at start: 45 bar **Start flow:** 1.000 ml/min **Column oven:** 29.99 °C



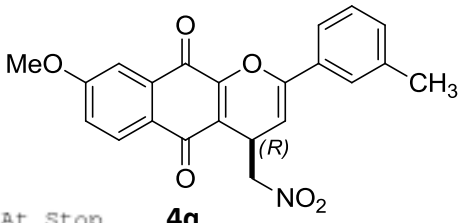
Name UK 4-322-1

RT [min]	Type	Area%	Area	Height	Width [min]
5.24	BV	96.79	4352.81	465.13	0.15
5.89	VB	2.09	93.85	8.41	0.17
6.62	BB	1.13	50.66	4.19	0.19
	Sum	100.00	4497.32		

Sample Name: UK KD 1180 rac
Data file: D:\GONZO\UK\1180R1IB.D
Sample Info: Laufmittel: n-Heptan/EtOH 7:3;
Die Probe ist in DCM/LM gelöst.

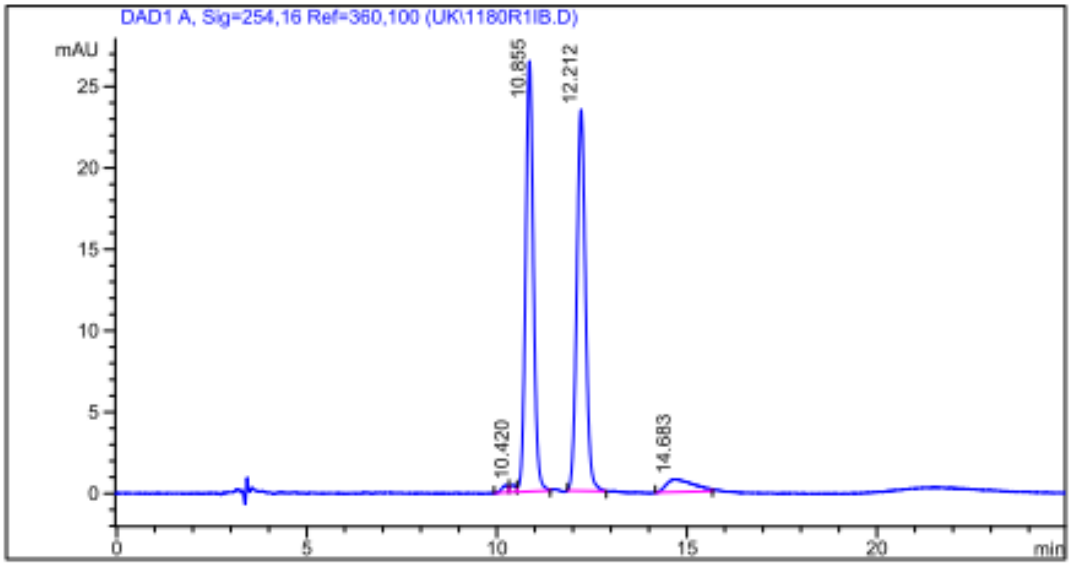
Säule: DAICELIB.M
Säuleninfo: Chiralpak IB (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 09:34:44
Injektion Date: 25.09.2015



4q

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	60.2	60.3
Flow in ml/min:	1.00	1.00



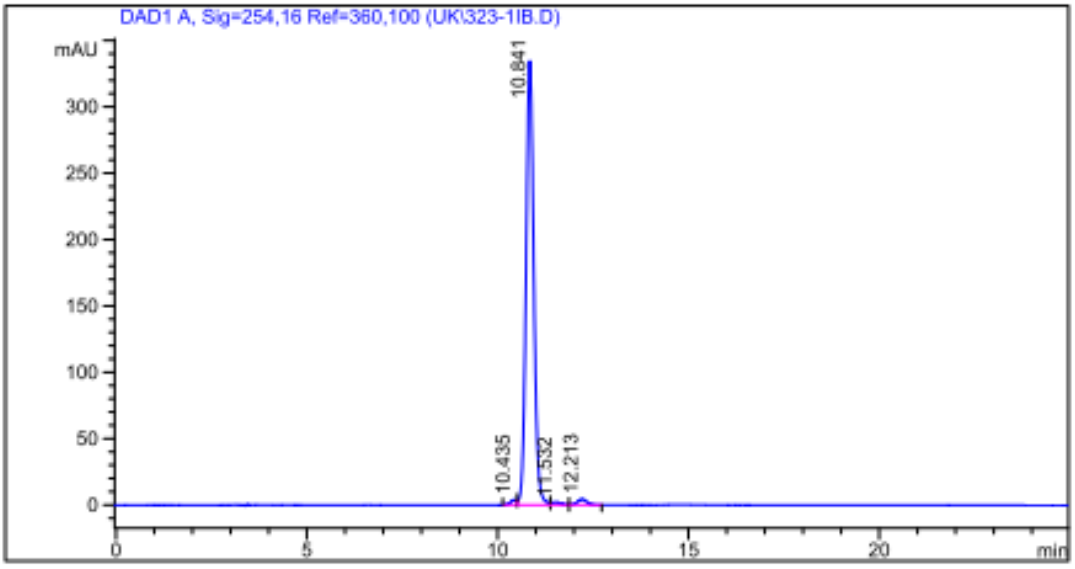
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	10.24	0.18	0.45	5.39	0.68
2	10.42	0.14	0.45	4.97	0.62
3	10.86	0.22	26.47	375.09	47.04
4	12.21	0.24	23.41	372.82	46.76
5	14.68	0.60	0.79	39.05	4.90
Total				797.31	100.00

Sample Name: UK 4-323-1
Data file: D:\GONZO\UK\323-1IB.D
Sample Info: Laufmittel: n-Heptan/EtOH 7:3;
Die Probe ist in DCM/LM gelöst.

Säule: DAICELIB.M
Säuleninfo: Chiralpak IB (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 10:00:56
Injektion Date: 25.09.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	60.2	60.5
Flow in ml/min:	1.00	1.00



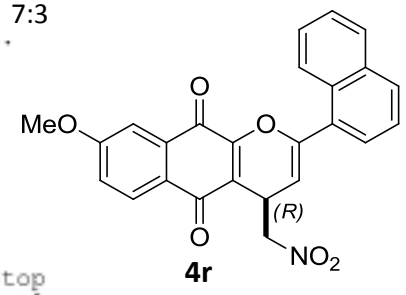
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	10.43	0.17	3.87	42.45	0.87
2	10.84	0.22	334.69	4723.46	96.85
3	11.53	0.26	2.04	36.05	0.74
4	12.21	0.26	4.45	75.02	1.54
Total				4876.98	100.00

Sample Name: UK KD 1181 rac
Data file: D:\GONZO\UK\1181RIB.D
Sample Info: Laufmittel: n-Heptan/EtOH ~~9+1~~; 7:3
Die Probe ist in DCM/LM gelöst.

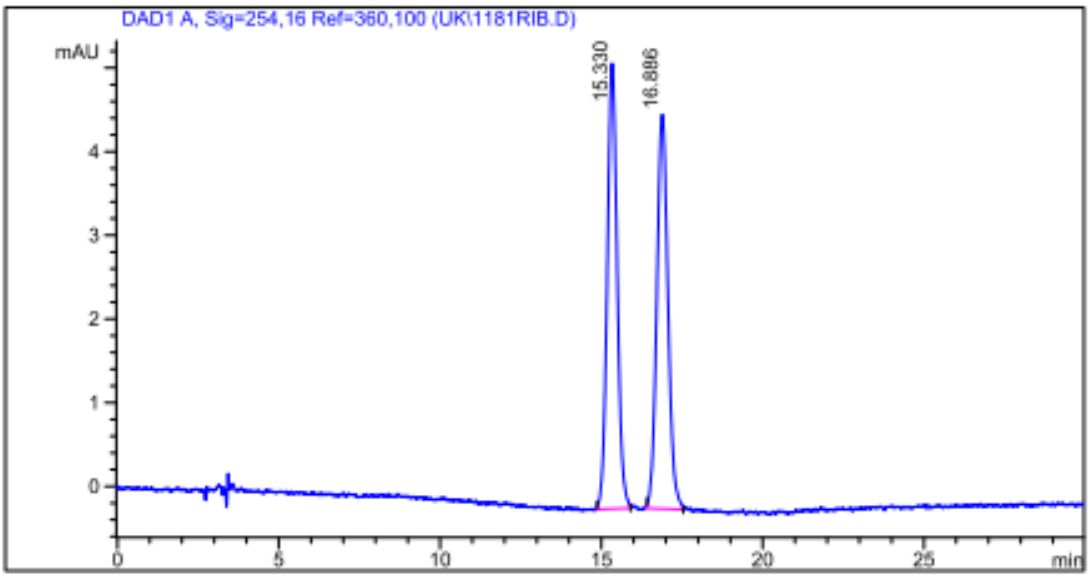
Säule: DAICELIB.M
Säuleninfo: Chiralpak IB (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 08:06:08
Injektion Date: 25.09.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	59.9	60.9
Flow in ml/min:	1.00	1.00



4r



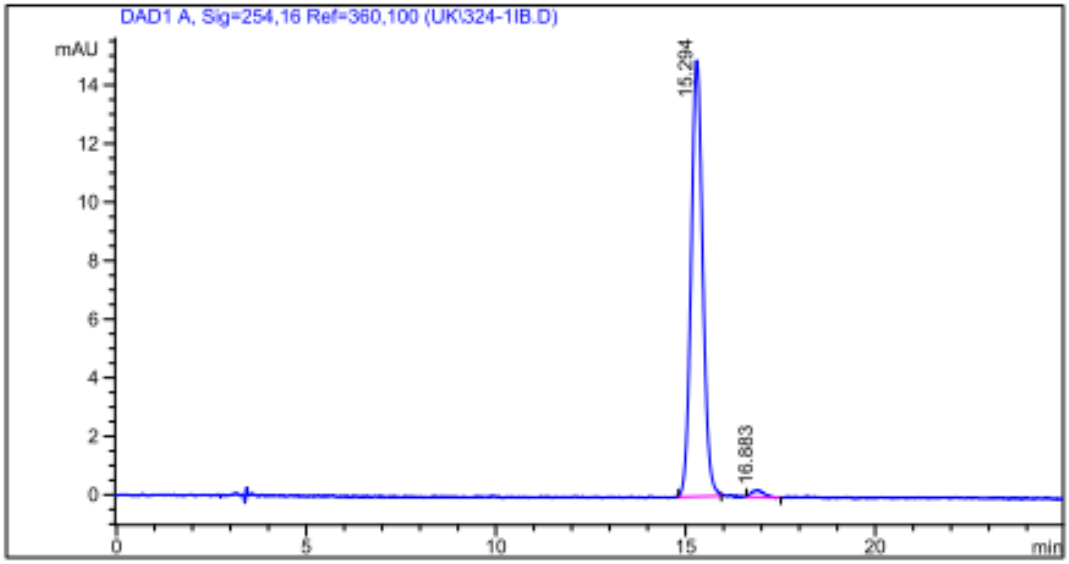
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	15.33	0.33	5.32	113.79	50.25
2	16.89	0.35	4.71	112.64	49.75
Total				226.43	100.00

Sample Name: UK 4-324-1
Data file: D:\GONZO\UK\324-1IB.D
Sample Info: Laufmittel: n-Heptan/EtOH ~~9:1~~; 7:3
Die Probe ist in DCM/LM gelöst.

Säule: DAICELIB.M
Säuleninfo: Chiralpak IB (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 09:08:32
Injektion Date: 25.09.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	60.0	60.6
Flow in ml/min:	1.00	1.00



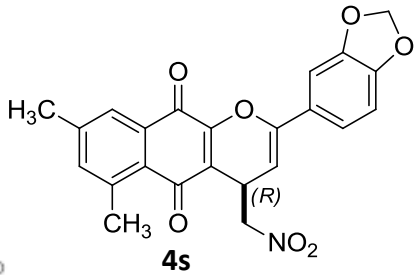
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	15.29	0.33	14.90	316.39	98.40
2	16.88	0.28	0.23	5.15	1.60
Total				321.53	100.00

Sample Name: UK KD 1183 rac
Data file: D:\GONZO\UK\1183RIB.D
Sample Info: Laufmittel: n-Heptan/EtOH ~~9:1~~; 7:3
Die Probe ist in DCM/LM gelöst.

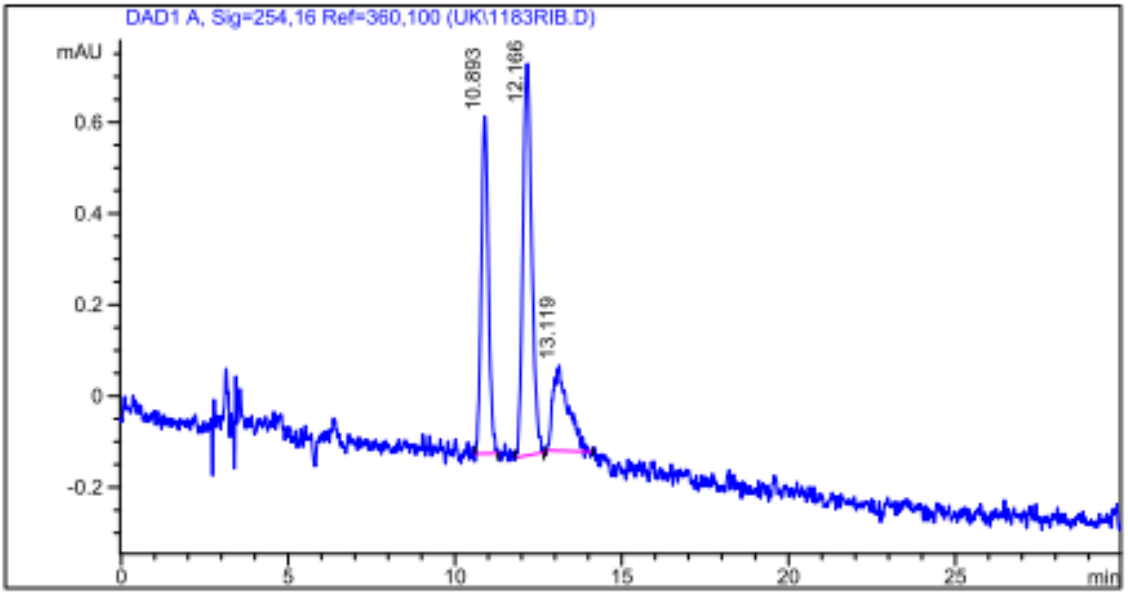
Säule: DAICELIB.M
Säuleninfo: Chiralpak IB (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 08:37:22
Injektion Date: 25.09.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	59.9	60.5
Flow in ml/min:	1.00	1.00



4s



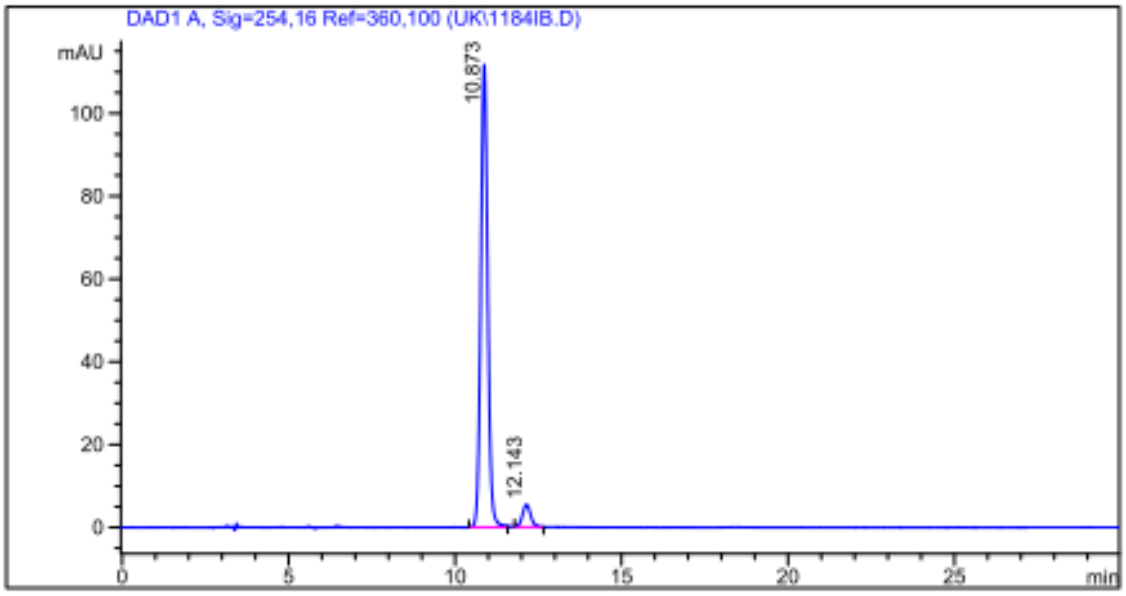
#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	10.89	0.22	0.74	10.95	33.42
2	12.17	0.26	0.86	15.88	48.49
3	13.12	0.53	0.19	5.93	18.09
Total				32.76	100.00

Sample Name: UK KD 1184
Data file: D:\GONZO\UK\1184IB.D
Sample Info: Laufmittel: n-Heptan/EtOH 7:3;
Die Probe ist in DCM/LM gelöst.

Säule: DAICELIB.M
Säuleninfo: Chiralpak IB (250x4,6)mm
Operator: Analytik Labor AKEN

Injektion Time: 10:27:07
Injektion Date: 25.09.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0°C	30.0°C
Pressure in bar:	59.6	60.5
Flow in ml/min:	1.00	1.00



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	10.87	0.22	111.74	1608.28	94.53
2	12.14	0.27	5.39	92.99	5.47
Total				1701.27	100.00

Sample Name: UK 4-332-1 rac
Data file: D:\BERT\UK\332R1AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

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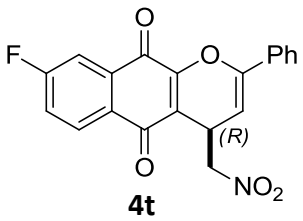


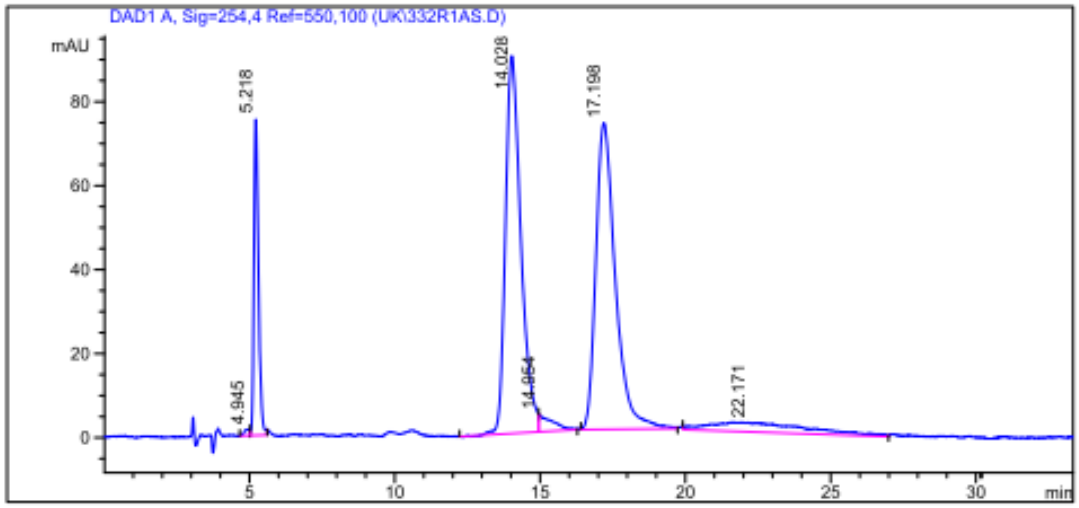
4t

Säule: DAICELAS.M
Säuleninfo: (250x4,6)mm

Operator: Analytik Labor AKEN

Injektion Time: 15:00:20
Injektion Date: 04.11.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	28.5	28.3
Flow in ml/min:	1.0	1.0



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	4.94	0.20	1.52	18.84	0.23
2	5.22	0.17	75.60	817.09	9.89
3	14.03	0.62	89.95	3330.43	40.33
4	14.95	0.52	4.22	132.98	1.61
5	17.20	0.78	72.94	3411.91	41.32
6	22.17	4.09	2.23	546.62	6.62
Total				8257.87	100.00

Sample Name: UK 4-330-1
Data file: D:\BERT\UK\330AS.D
Sample Info: Laufmittel: n-Heptan/EtOH 9:1;
Die Probe ist in DCM/LM gelöst

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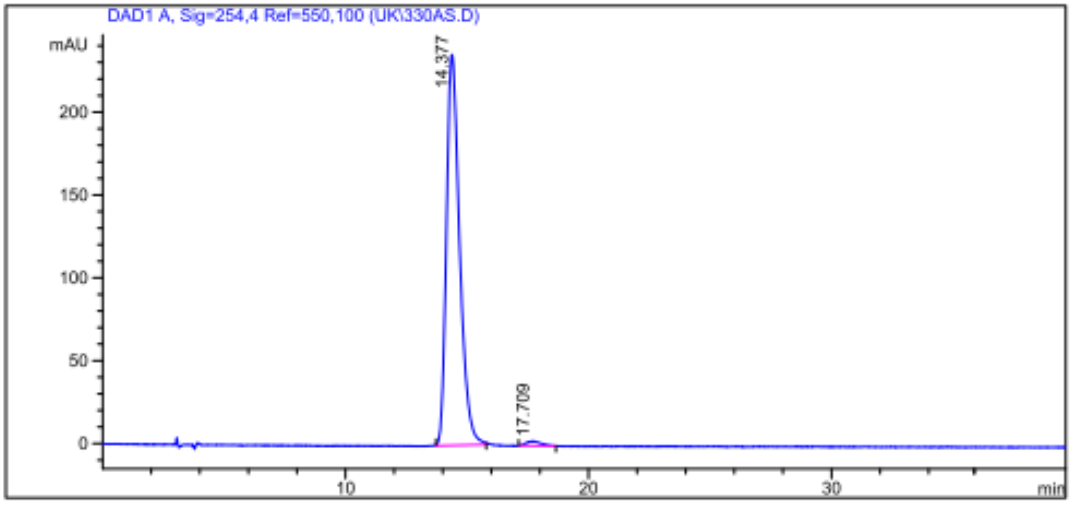
Säule: DAICELAS.M
Säuleninfo: (250x4,6)mm

Operator: Analytik Labor AKEN

Injektion Time: 08:13:36
Injektion Date: 05.11.2015

Instrument Conditions:	At Start	At Stop
Temperature in°C:	30.0	30.0
Pressure in bar:	29.4	29.0
Flow in ml/min:	1.0	1.0



#	Ret. Time (min)	Width	Height (mAU)	Area (mAU*s)	Area %
1	14.38	0.56	235.79	8850.82	98.70
2	17.71	0.50	2.74	116.79	1.30
Total				8967.61	100.00