SUPPORTING INFORMATION

Asymmetric Cyclopropanation of Chalcones Using Chiral Phase-Transfer Catalysts

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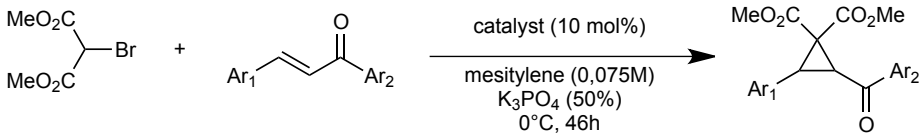
1. Experimental Details and Analytical Data:

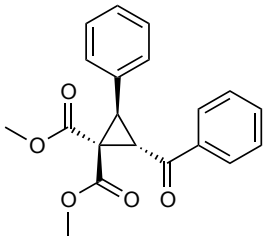
General: Melting points were measured on a Kofler melting point microscope (Reichert, Vienna). ^{1}H- and ^{13}C-NMR spectra were recorded on a Bruker Avance III 300 MHz spectrometer and on a Bruker Avance III 700 MHz spectrometer with TCI cryoprobe. All NMR spectra were referenced on the solvent peak. High resolution mass spectra were obtained using an Agilent 6520 Q-TOF mass spectrometer with an ESI source and an Agilent G1607A coaxial sprayer. All analyses were made in the positive ionization mode. Purine (exact mass for $[M+H]^+$ = 121.050873) and 1,2,3,4,5,6-hexakis(2,2,3,3-tetrafluoropropoxy)-1,3,5,2,4,6-triazatriphosphinane (exact mass for $[M+H]^+$ = 922.009798) were used for internal mass calibration. IR spectra were recorded on a Shimadzu IR Affinity-1 fourier transform infrared spectrometer. Optical rotations were recorded on a Perkin Elmer Polarimeter Model 241 MC. HPLC was performed using a Dionex Summit HPLC system with a Chiralcel OD-H (250 x 4.6 mm, 5 μm) or a Chiralcel OD-R (250 x 4.6 mm, 10 μm) chiral stationary phase. All chemicals were purchased from commercial suppliers and used without further purification unless otherwise stated. Cinchona alkaloid-based catalysts were synthesized according and in analogy to literature-known methods.[1] Bromomalonate-derivatives and chalcone-derivatives were synthesized according and in analogy to literature-known methods.[2]

1) a) Provencher, B. A.; Bartelson, K. J.; Liu, Y.; Foxman, B. M.; Deng, L. *Angew. Chem.* **2011**, *123*, 10753-10757; b) Fiandra, C. D.; Piras, L.; Fini, F.; Disetti, P.; Moccia, M.; Adamo, M. F. A. *Chem. Commun.* **2012**, *48*, 3863-3865; c) Li, H.; Wang, Y.; Tang, L.; Deng, L. *J. Am. Chem. Soc.* **2004**, *126*, 9906-9907; d) Liu, Y.; Provencher, B. A.; Bartelson, K. J.; Deng, L. *Chem. Sci.* **2011**, *2*, 1301-1304; e) Corey, E. J.; Xu, F.; Noe, M. C. *J. Am. Chem. Soc.* **1997**, *119*, 12414-12415; f) Berkessel, A.; Guixà, M.; Schmidt, F.; Neudörfl, J. M.; Lex. J. *Chem. Eur. J.* **2007**, *13*, 4483-4498 g) Lian, M.; Li, Z.; Du, J.; Meng, Q.; Gao, Z. *Eur. J. Org. Chem.* **2010**, 6525-6530.
2) a) Perreault, S.; Spino, C. *Org. Lett.* **2006**, *8*, 4385-4388; b) Lukoyanova, O.; Cardona, C. M.; Rivera, J.; Lugo-Morales, L. Z.; Chancellor, C. J.; Olmstead, M. M.; Rodriguez-Fortea, A.; Poblet, J. M.; Echegoyen, L. *J. Am. Chem. Soc.* **2007**, *129*, 10423-10430



General procedure for the phase-transfer catalysed cyclopropanation using bromomalonates and chalcones: Reactions were usually carried out using less than 0.5 mmol bromomalonate (**1**). First 10 eq. K_2CO_3 (50% aq. solution) were added to a solution of catalyst (10 mol%) in mesitylene (assuring a dilution of 0.075M based on the amount of **1** used in the reaction). After flushing the solution with Argon, the corresponding chalcone derivative (6 eq.) was added. The vigorously stirred solution (>1200 rpm) was cooled to 0°C (Ar-atmosphere). Subsequently, the bromomalonate was added in 3 portions (3x 0.33 eq. over 24 h). The biphasic mixture was stirred for a total of 46 h at 0°C. After extraction with CH_2Cl_2 / H_2O, the combined organic phases were dried over Na_2SO_4, evaporated to dryness and purified by column chromatography using heptanes:EtOAc = 40:1 to 10:1 as the eluent. The excess chalcone could easily be recovered hereby.

(-)-3c. Obtained as a colourless oil in 82% yield (136 mg, 0.40 mmol) and with *er* = 87:13

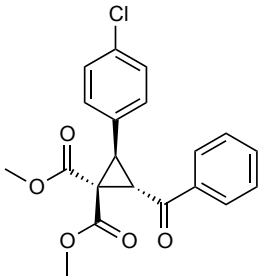


upon reacting chalcone **2** (615 mg, 2.96 mmol) with bromomalonate **1c** (104 mg, 0.49 mmol) in 6.4 mL mesitylene with 0.82 mL aqueous K_2CO_3 solution (50% w/w). Analytical data are in accordance with those reported in literature.[3] $[\alpha]_D^{20}$ -25.9 (*c* 0.44, $CHCl_3$); ^{1}H NMR (300 MHz, δ, $CDCl_3$, 298 K): 3.55 (s, 3H), 3.72 (s, 3H), 3.88 (d, *J* = 7.7 Hz, 1H), 4.14 (d, *J* = 7.7 Hz, 1H), 7.27 – 7.34 (m, 5H), 7.48 – 7.56 (m, 2H), 7.59 – 7.66 (m, 1H), 8.07 – 8.13 (m, 1H) ppm; ^{13}C NMR (75 MHz, δ, $CDCl_3$, 298 K): 35.1, 36.6, 46.0, 53.0, 53.1, 127.8, 128.4, 128.5, 128.6, 128.8, 133.4, 133.8, 136.7, 166.1, 166.6, 193.9 ppm; IR (film): $\bar{\nu}$ = 3064, 3032, 2995, 2953, 2916, 2846, 2358, 2341, 1735, 1678, 1597, 1581, 1449, 1437, 1352, 1267, 1219,

3) a) Kingsbury, C. A.; Durham, D. L.; Hutton, R.; *J. Org. Chem.* **1987**, *43*, 4696-4700; b) Ye, Y.; Zheng, C.; Fan, R. *Org. Lett.* **2009**, *11*, 3156-3159.

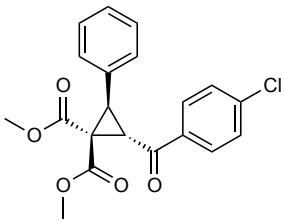
1178, 1117, 1024, 1010, 943, 916, 739 cm^{-1}; The enantioselectivity was determined by HPLC (Chiralcel OD-R, eluent: H_2O:AcN = 55:45, 0.7 mL/min, 10°C, retention times: (+)-enantiomer 36.4 min, (-)-enantiomer 39.6 min); HRMS (ESI): *m/z* calcd for $C_{20}H_{18}O_5$: 339.1227 $[M+H]^+$; found: 339.1227.

(-)-8. Obtained as a colourless oil in 98% yield (168 mg, 0.45 mmol) and with *er* = 88:12



upon reacting chalcone **7** (662 mg, 2.73 mmol) with bromomalonate **1c** (97 mg, 0.46 mmol) in 6.0 mL mesitylene with 0.77 mL aqueous K_2CO_3 solution (50% w/w). $[\alpha]_D^{20}$ -11.2 (*c* 1.30, $CHCl_3$); ^1H NMR (300 MHz, δ, $CDCl_3$, 298 K): 3.59 (s, 3H), 3.71 (s, 3H), 3.84 (d, *J* = 7.7 Hz, 1H), 4.11 (d, *J* = 7.7 Hz, 1H), 7.22 – 7.33 (m, 4H), 7.49 – 7.57 (m, 2H), 7.60 – 7.67 (m, 1H), 8.06 – 8.12 (m, 2H) ppm; ^{13}C NMR (75 MHz, δ, $CDCl_3$, 298 K): 35.1, 35.8, 46.0, 53.1, 53.1, 128.6, 128.6, 128.9, 129.9, 131.9, 133.8, 133.9, 136.6, 166.0, 166.3, 193.6 ppm; IR (film): $\bar{\nu}$ = 3064, 3030, 3003, 2953, 2361, 2339, 2328, 1732, 1678, 1597, 1581, 1494, 1448, 1435, 1400, 1346, 1269, 1217, 1176, 1116, 1093, 1035, 1014, 1002, 921, 835, 769, 736 cm^{-1}; The enantioselectivity was determined by HPLC (Chiralcel OD-R, eluent: H_2O:AcN = 50:50, 0.7 mL/min, 10 °C, retention times: (+)-enantiomer 33.8 min, (-)-enantiomer 37.9 min); HRMS (ESI): *m/z* calcd for $C_{20}H_{17}ClO_5$: 373.0837 $[M+H]^+$; found: 373.0836.

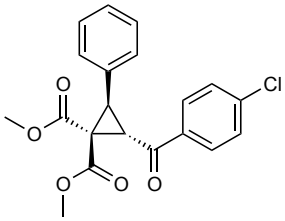
(-)-10. Obtained as a colourless oil in 91% yield (119 mg, 0.32 mmol). and with *er* = 89:11



upon reacting chalcone **9** (497 mg, 2.05 mmol) with bromomalonate **1c** (74 mg, 0.35 mmol) in 4.6 mL mesitylene with 0.58 mL aqueous K_2CO_3 solution (50% w/w). $[\alpha]_D^{20}$ -8.5 (*c* 1.29, $CHCl_3$); ^1H NMR (300 MHz, δ, $CDCl_3$, 298 K): 3.55 (s, 3H), 3.71 (s, 3H), 3.87 (d, *J* = 7.7 Hz, 1H), 4.07 (d, *J* = 7.7 Hz, 1H), 7.27 – 7.33 (m, 5H), 7.47 – 7.52 (m, 2H), 8.01 – 8.06 (m, 2H) ppm; ^{13}C NMR

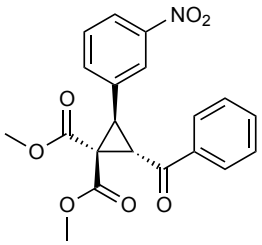
(75 MHz, δ, $CDCl_3$, 298 K): 35.0, 36.5, 46.1, 53.0, 53.1, 127.6, 127.9, 128.1, 128.5, 128.8, 129.5, 130.0, 133.2, 135.0, 140.3, 166.0, 166.4, 192,6 ppm; IR (film): $\bar{\nu}$ = 3057, 3032, 2999, 2953, 2916, 2846, 2333, 2291, 1734, 1678, 1589, 1571, 1489, 1435, 1400, 1350, 1269, 1217, 1195, 1176, 1118, 1091, 1031, 1012, 993, 941, 921, 871, 846, 736 cm^{-1}; The enantioselectivity was determined by HPLC (Chiralcel OD-R, eluent: H_2O:AcN = 55:45, 0.7 mL/min, 10 °C, retention times: (+)-enantiomer 69.7 min, (-)-enantiomer 73.2 min); HRMS (ESI): *m/z* calcd for $C_{20}H_{17}ClO_5$: 373.0837 $[M+H]^+$; found: 373.0837.

(-)-12. Obtained as a colourless oil in 72% yield (132 mg, 0.37 mmol) and with *er* = 87:13



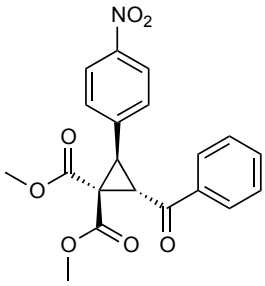
upon reacting chalcone **11** (689 mg, 3.05 mmol) with **1c** (108 mg, 0.51 mmol) in 6.7 mL mesitylene with 0.85 mL aqueous K_2CO_3 solution (50% w/w). $[\alpha]_D^{20}$ -6.1 (*c* 0.80, $CHCl_3$); ^1H NMR (300 MHz, δ, $CDCl_3$, 298 K): 3.55 (s, 3H), 3.71 (s, 3H), 3.87 (d, *J* = 7.7 Hz, 1H), 4.08 (d, *J* = 7.7 Hz, 1H), 7.15 – 7.23 (m, 2H), 7.27 – 7.34 (m, 5H), 8.10 – 8.17 (m, 2H) ppm; ^{13}C NMR (75 MHz, δ, $CDCl_3$, 298 K): 35.0, 36.4, 46.0, 53.0, 53.1, 115.9, 116.1, 127.9, 128.5, 131.2, 131.3, 133.1, 133.1, 133.2, 166.1, 166.5, 192.1 ppm; IR (film): $\bar{\nu}$ = 3062, 3028, 3007, 2953, 2850, 2349, 2301, 1732, 1678, 1597, 1508, 1435, 1350, 1269, 1215, 1197, 1176, 1157, 1116, 1062, 1029, 1010, 995, 941, 923, 871, 852, 817, 700 cm^{-1}; The enantioselectivity was determined by HPLC (Chiralcel OD-H, eluent: *n*-hexane:*i*PrOH = 200:1, 0.5 mL/min, 10 °C, retention times: (-)-enantiomer 77.5 min, (+)-enantiomer 85.2 min); HRMS (ESI): *m/z* calcd for $C_{20}H_{17}FO_5$: 357.1133 $[M+H]^+$; found: 357.1135.

(-)-14. Obtained as a white solid in 59% yield (96 mg, 0.25 mmol) and with *er* = 91:9 upon



reacting chalcone **13** (638 mg, 2.52 mmol) with bromomalonate **1c** (89 mg, 0.42 mmol) in 5.5 mL mesitylene with 0.70 mL aqueous K_2CO_3 solution (50% w/w). $[\alpha]_D^{20}$ -38.4 (*c* 2.14, $CHCl_3$); M.p.: decomp > 180 °C; ^{1}H NMR (300 MHz, δ, $CDCl_3$, 298 K): 3.60 (s, 3H), 3.71 (s, 3H), 3.95 (d, *J* = 7.6 Hz, 1H), 4.18 (d, *J* = 7.6 Hz, 1H), 7.48 – 7.58 (m, 3H), 7.62 – 7.69 (m, 2H), 8.06 – 8.19 (m, 4H) ppm; ^{13}C NMR (75 MHz, δ, $CDCl_3$, 298 K): 35.0, 35.4, 46.0, 53.3, 53.4, 122.9, 123.6, 128.6, 128.9, 129.5, 134.1, 135.0, 135.7, 136.4, 148.2, 165.8, 165.9, 193.1 ppm; IR (film): $\bar{\nu}$ = 3084, 3062, 2954, 2358, 2304, 1732, 1666, 1595, 1579, 1525, 1481, 1446, 1436, 1348, 1319, 1265, 1217, 1178, 1114, 1099, 1085, 1042, 1001, 916, 902, 869, 852, 813, 788, 756, 736, 704 cm^{-1}; The enantioselectivity was determined by HPLC (Chiralcel OD-R, eluent: H_2O:AcN = 55:45, 0.7 mL/min, 10 °C, retention times: (+)-enantiomer 44.5 min, (-)-enantiomer 50.3 min); HRMS (ESI): *m/z* calcd for $C_{20}H_{17}NO_7$: 384.1078 $[M+H]^+$; found: 384.1079.

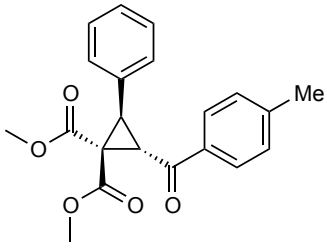
(-)-16. Obtained as a white solid in 58% yield (87 mg, 0.23 mmol) and with *er* = 90:10 upon



reacting chalcone **15** (590 mg, 2.34 mmol) with bromomalonate **1c** (82 mg, 0.39 mmol) in 5.1 mL mesitylene with 0.65 mL aqueous K_2CO_3 solution (50% w/w). $[\alpha]_D^{20}$ -22.8 (*c* 0.32, $CHCl_3$); M.p.: decomp > 180 °C; ^{1}H NMR (300 MHz, δ, $CDCl_3$, 298 K): 3.60 (s, 3H), 3.72 (s, 3H), 3.94 (d, *J* = 7.7 Hz, 1H), 4.18 (d, *J* = 7.7 Hz, 1H), 7.46 – 7.58 (m, 4H), 7.62 – 7.69 (m, 1H), 8.06 – 8.12 (m, 2H), 8.16 – 8.23 (m, 2H) ppm; ^{13}C NMR (75 MHz, δ, $CDCl_3$, 298 K): 35.0, 35.6, 46.2, 53.3, 53.4, 123.7, 128.6, 129.0, 129.6, 134.1, 136.4, 141.0, 147.5, 165.8, 165.8, 193.1 ppm; IR (film): $\bar{\nu}$ = 3061, 3034, 3005, 2953, 2918, 2848, 2364, 2357, 2341, 2337, 1732, 1678, 1598, 1581, 1519, 1496, 1448, 1436, 1406, 1346, 1269, 1217, 1176, 1112, 1035,

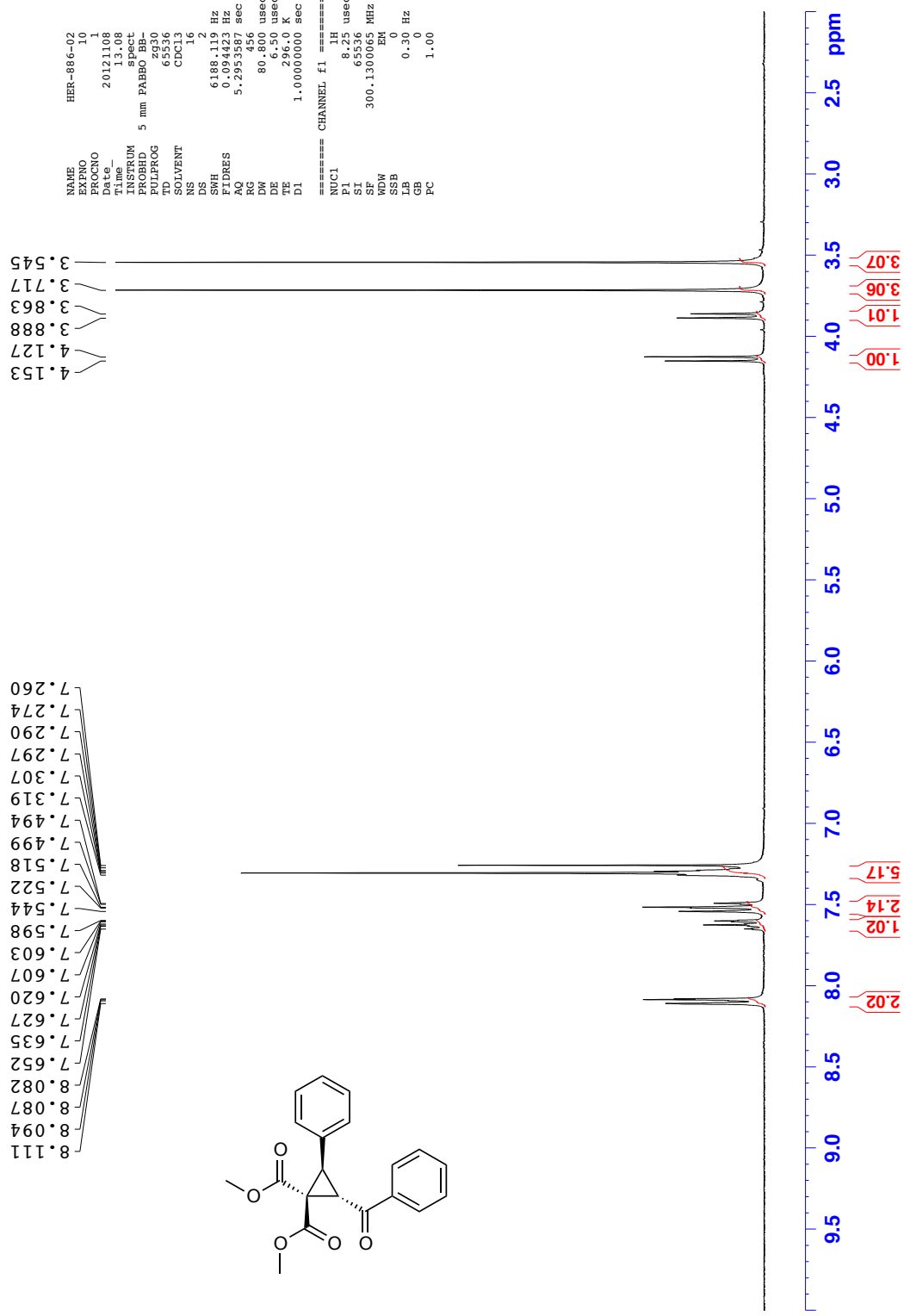
1014, 1002, 960, 939, 923, 877, 854, 738 cm^{-1}; The enantioselectivity was determined by HPLC (Chiralcel OD-R, eluent: H_2O:AcN = 55:45, 0.7 mL/min, 10 °C, retention times: (+)-enantiomer 54.3 min, (-)-enantiomer 70.1 min); HRMS (ESI): *m/z* calcd for $C_{20}H_{17}NO_7$: 384.1078 $[M+H]^+$; found: 384.1080.

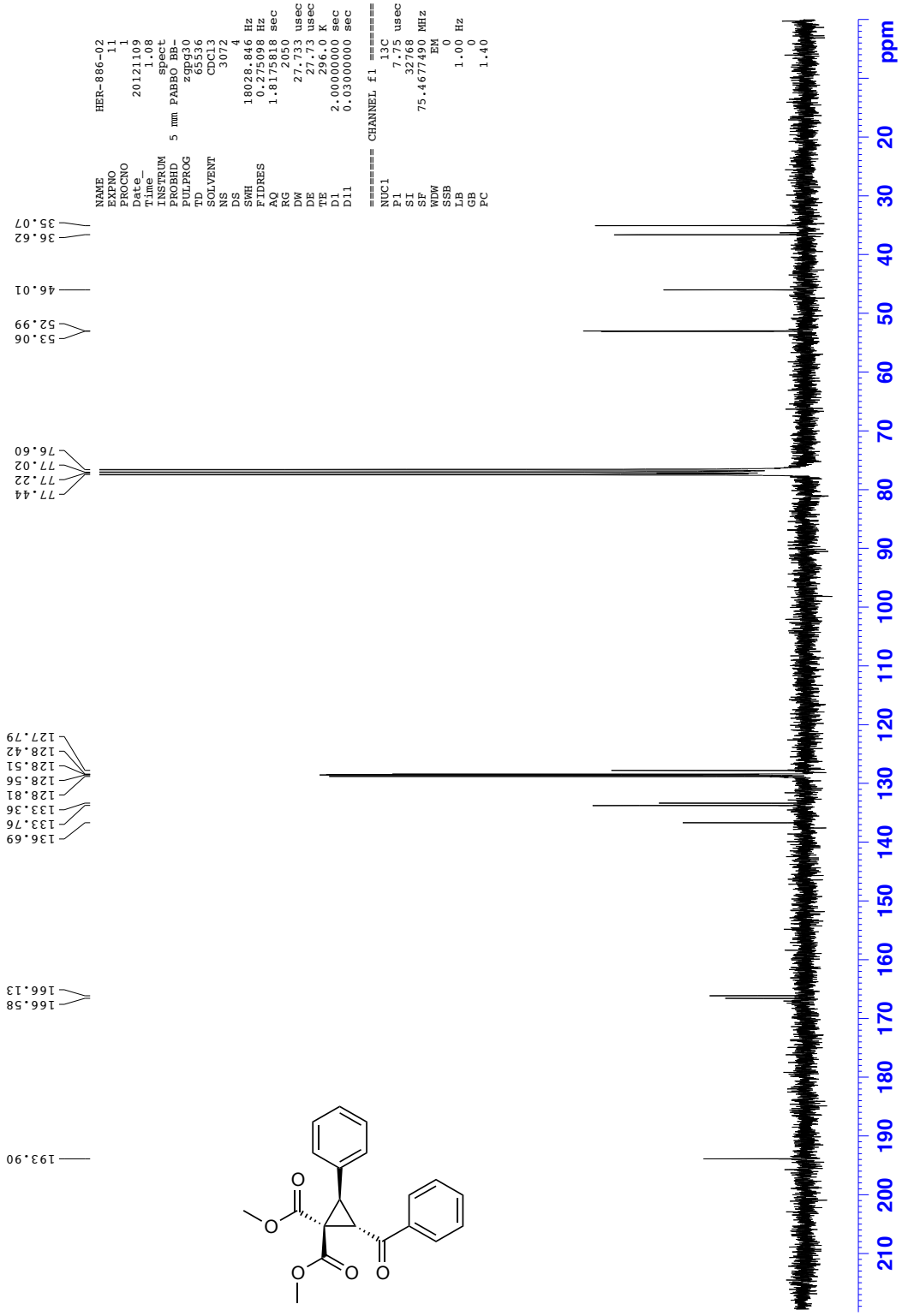
(-)-18. Obtained as a colourless oil in 72% yield (70 mg, 0.20 mmol) and with *er* = 84:16



upon reacting chalcone **17** (369 mg, 1.66 mmol) with bromomalonate **1c** (59 mg, 0.28 mmol) in 3.6 mL mesitylene with 0.47 mL aqueous K_2CO_3 solution (50% w/w). $[\alpha]_D^{20}$ -52.9 (c = 0.09, $CHCl_3$); ^1H NMR (300 MHz, δ, $CDCl_3$, 298 K): 2.44 (s, 3H), 3.54 (s, 3H), 3.72 (s, 3H), 3.86 (d, *J* = 7.7 Hz, 1H), 4.12 (d, *J* = 7.7 Hz, 1H), 7.27 – 7.36 (m, 7H), 7.97 – 8.02 (m, 2H) ppm; ^{13}C NMR (75 MHz, δ, $CDCl_3$, 298 K): 12.8, 35.0, 36.6, 45.9, 52.9, 53.0, 127.7, 128.4, 128.5, 128.7, 129.5, 133.5, 134.3, 144.7, 166.2, 166.7, 193.4 ppm; IR (film): $\bar{\nu}$ = 3059, 3030, 3005, 2953, 2920, 2848, 2358, 2331, 1732, 1674, 1606, 1573, 1533, 1498, 1435, 1352, 1313, 1271, 1220, 1180, 1112, 1035, 1010, 943, 921, 840, 802, 744, 700 cm^{-1}; The enantioselectivity was determined by HPLC (Chiralcel OD-R, eluent: H_2O:AcN = 60:40, 0.7 mL/min, 10 °C, retention times: (+)-enantiomer 100.8 min, (-)-enantiomer 107.9 min); HRMS (ESI): *m/z* calcd for $C_{21}H_{20}O_5$: 353.1384 $[M+H]^+$; found: 353.1386.

2. NMR Spectra:

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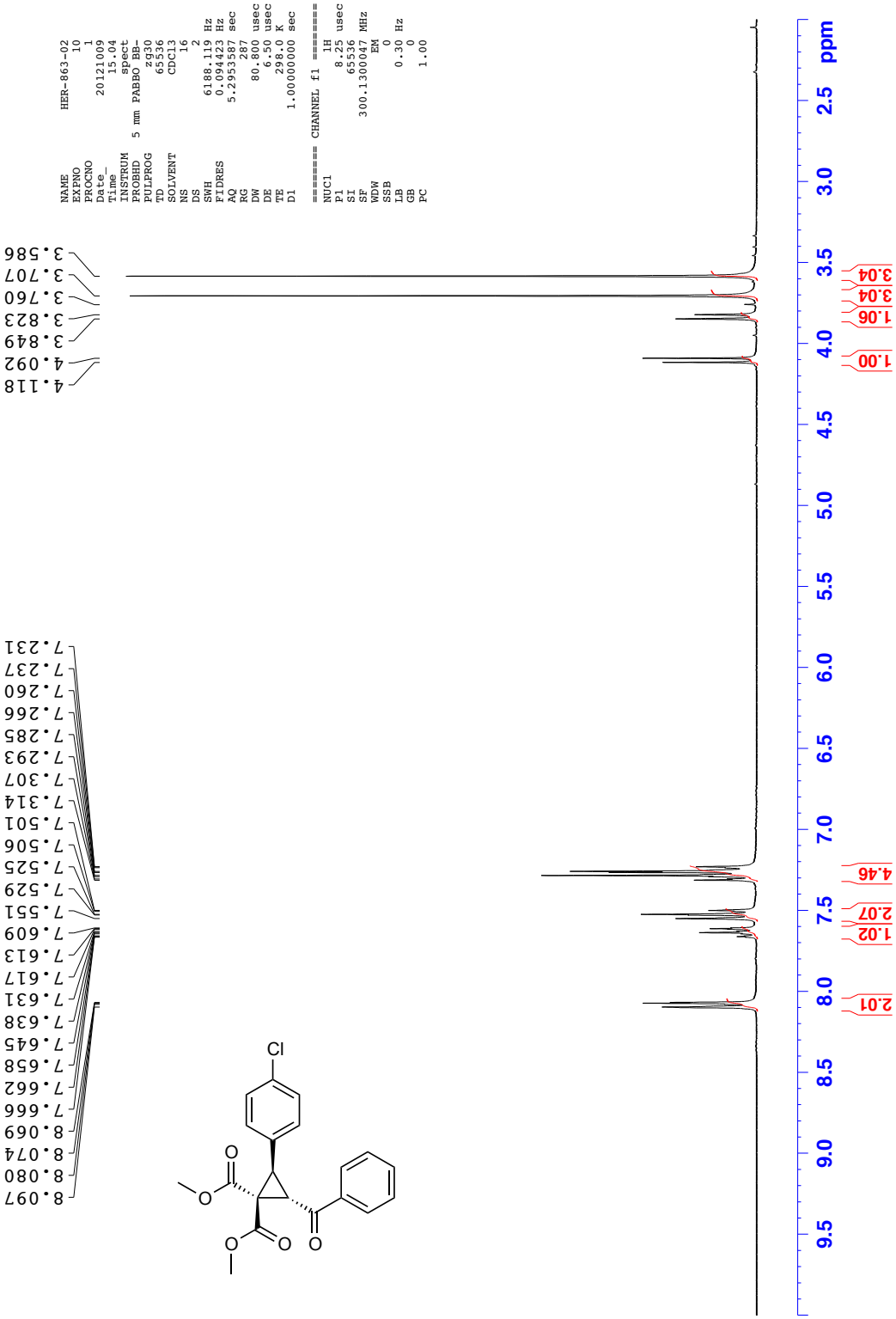
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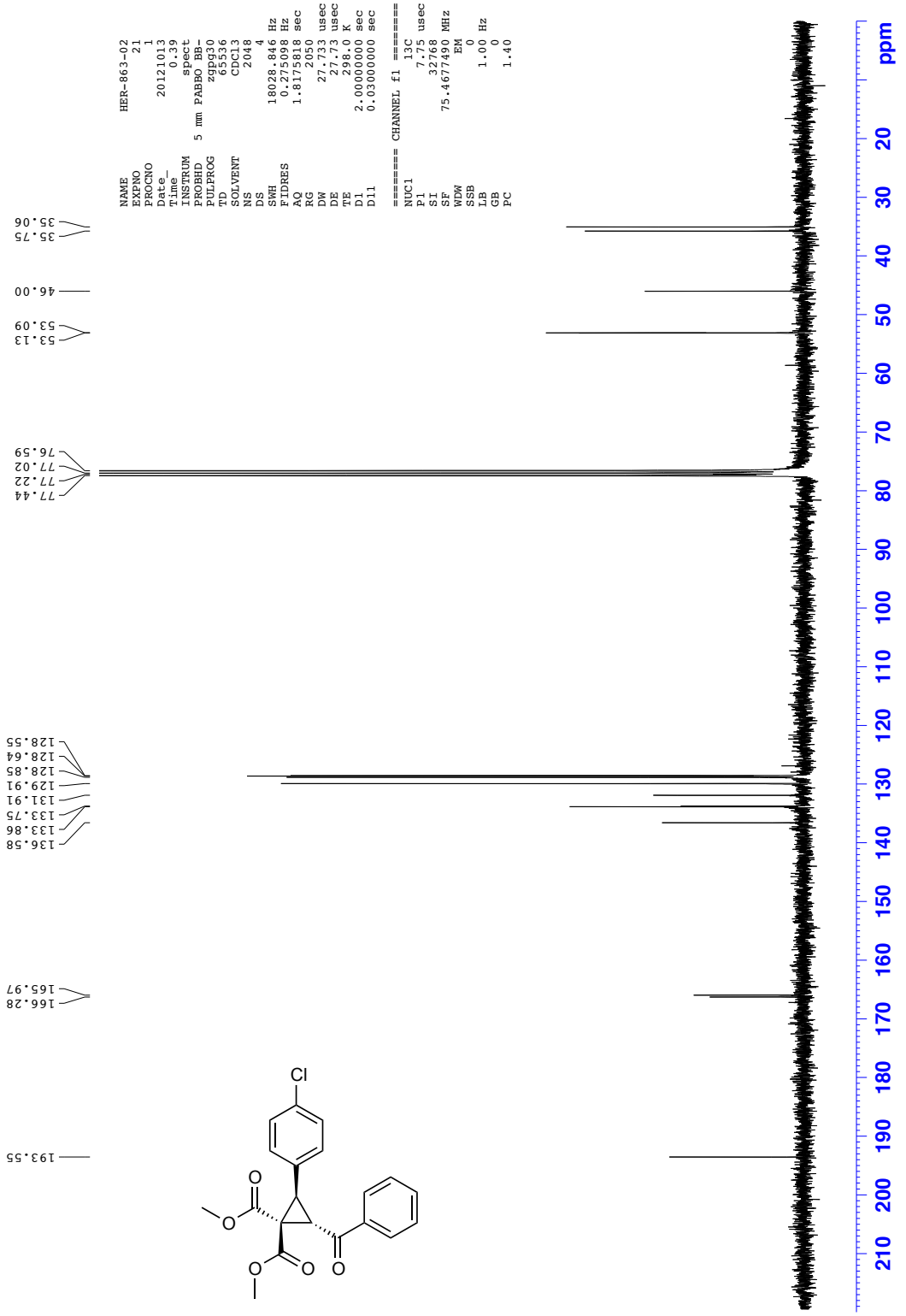
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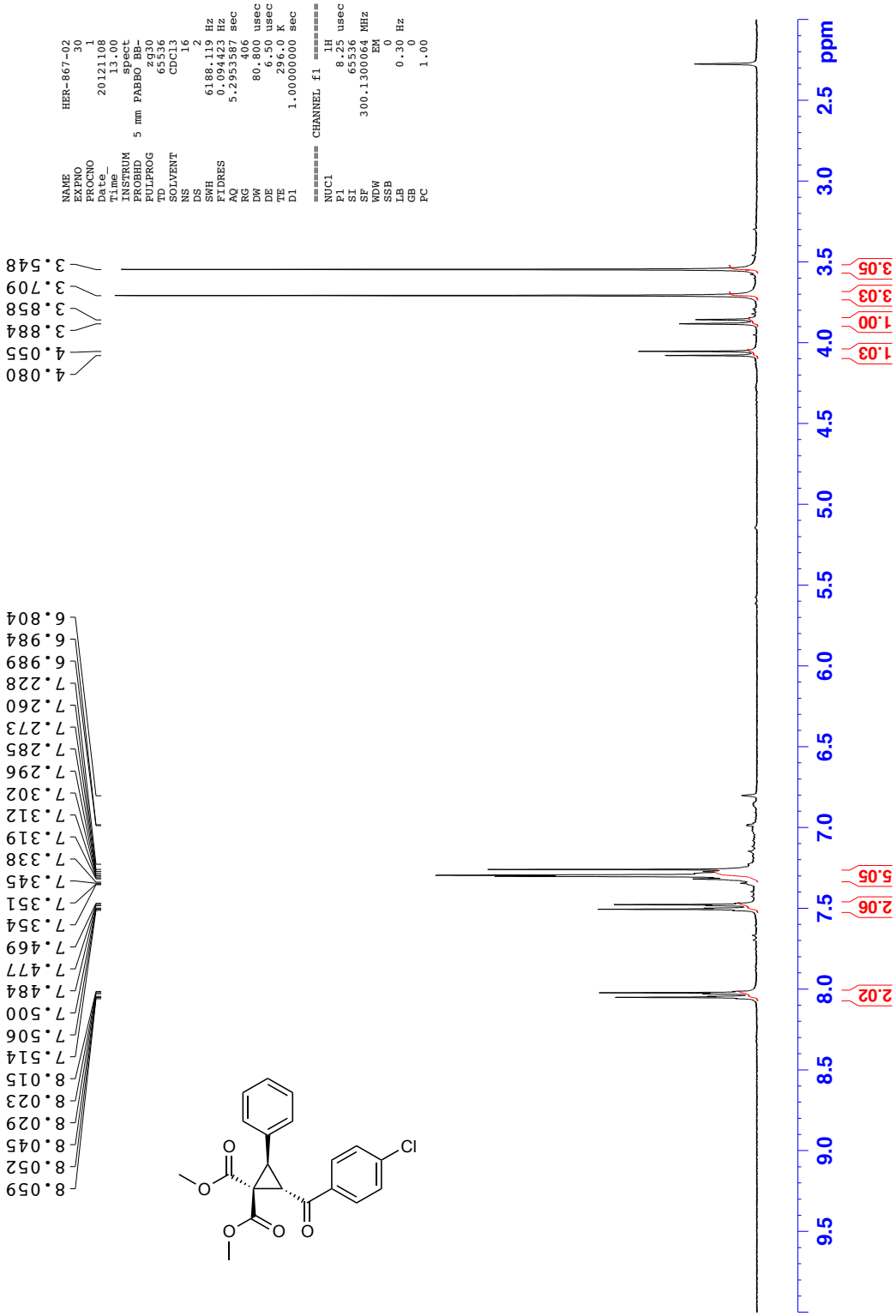
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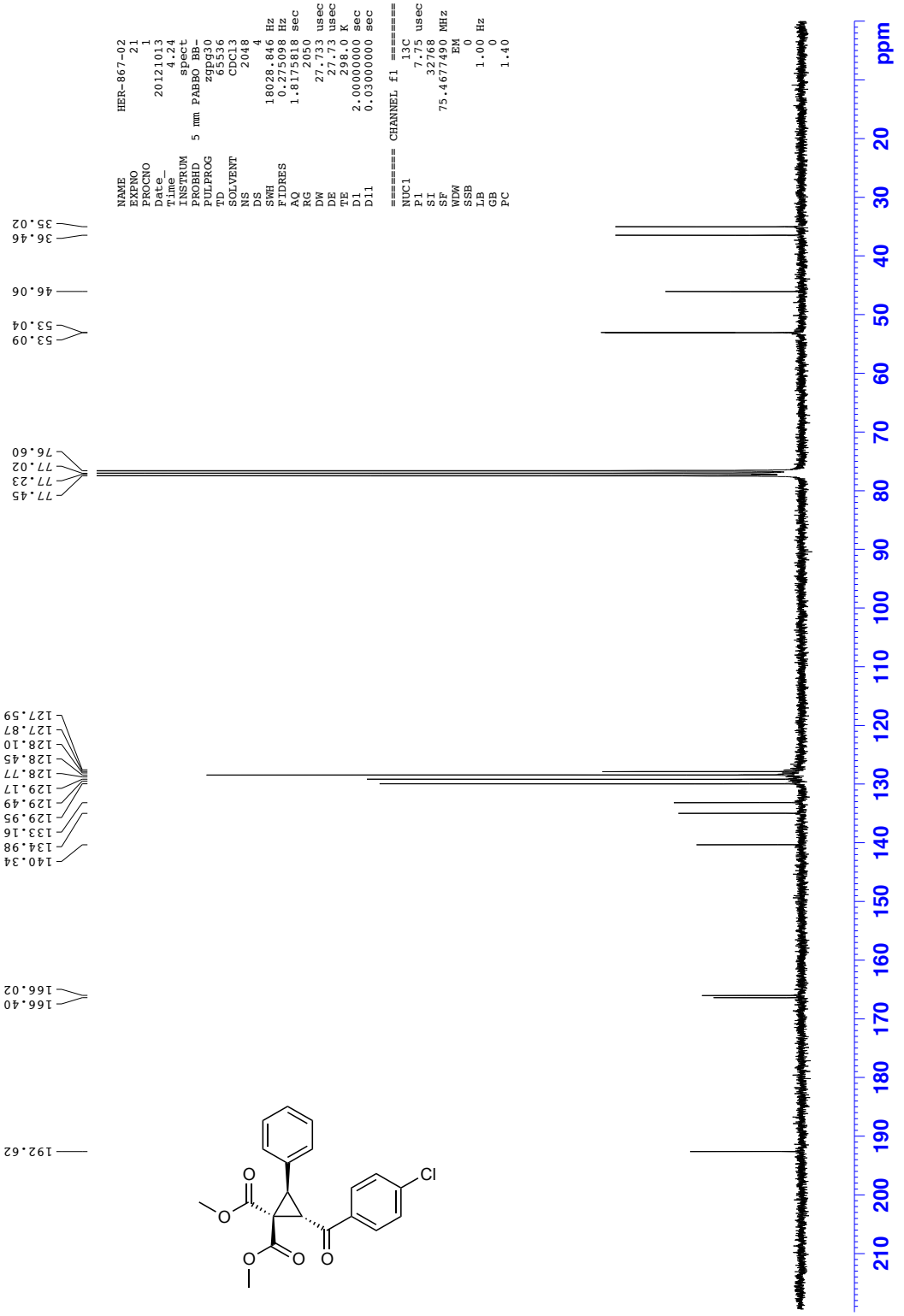
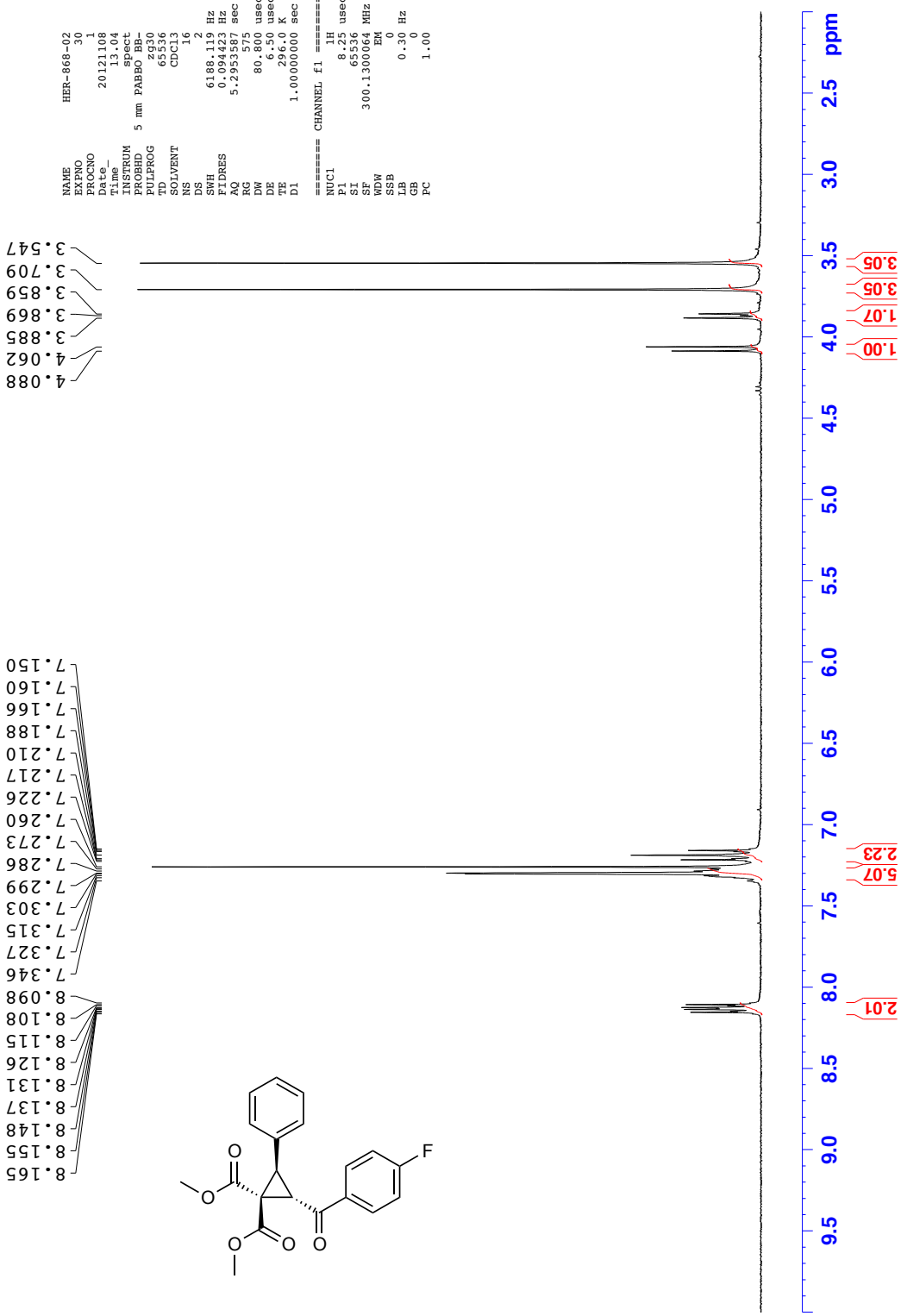
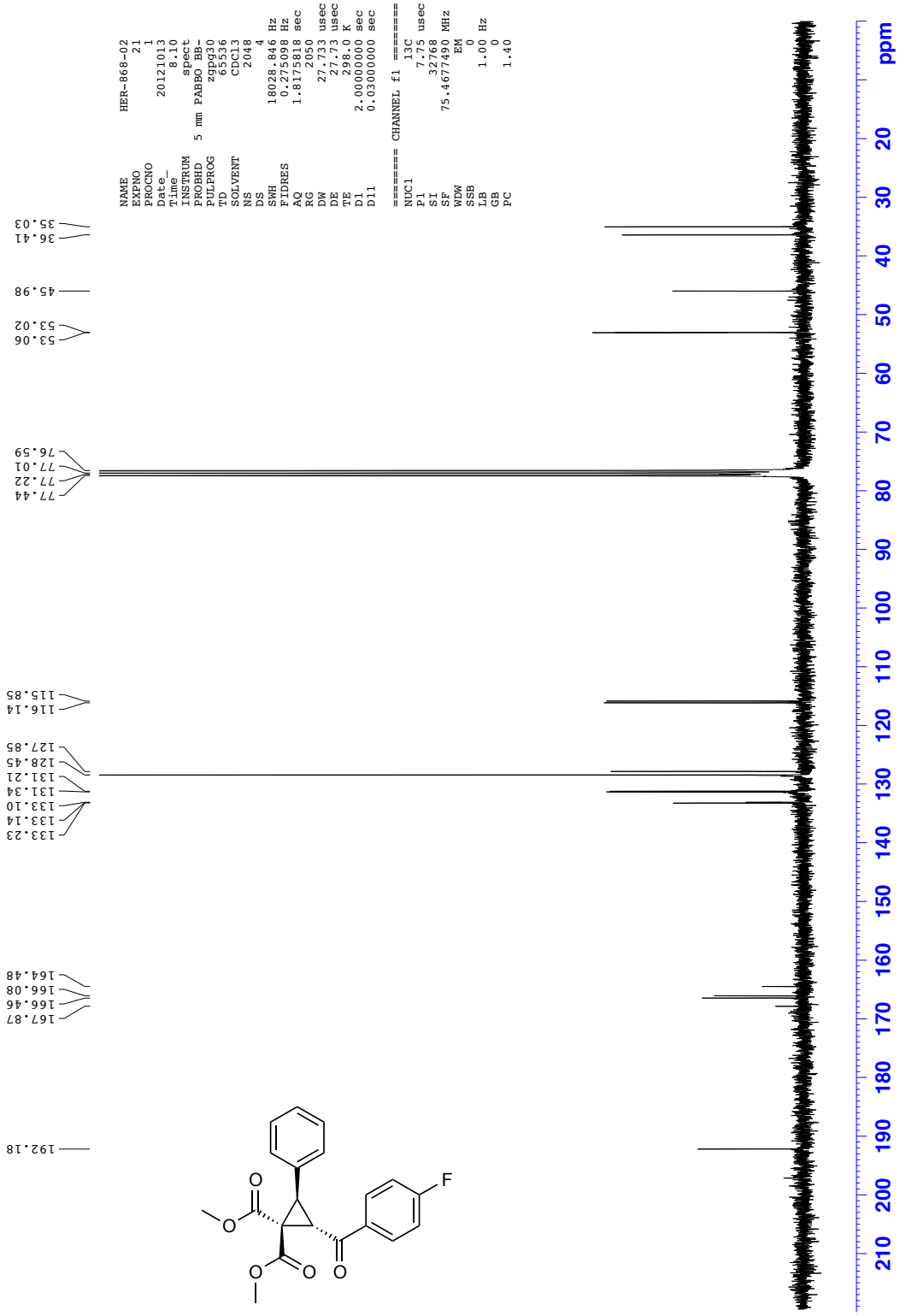
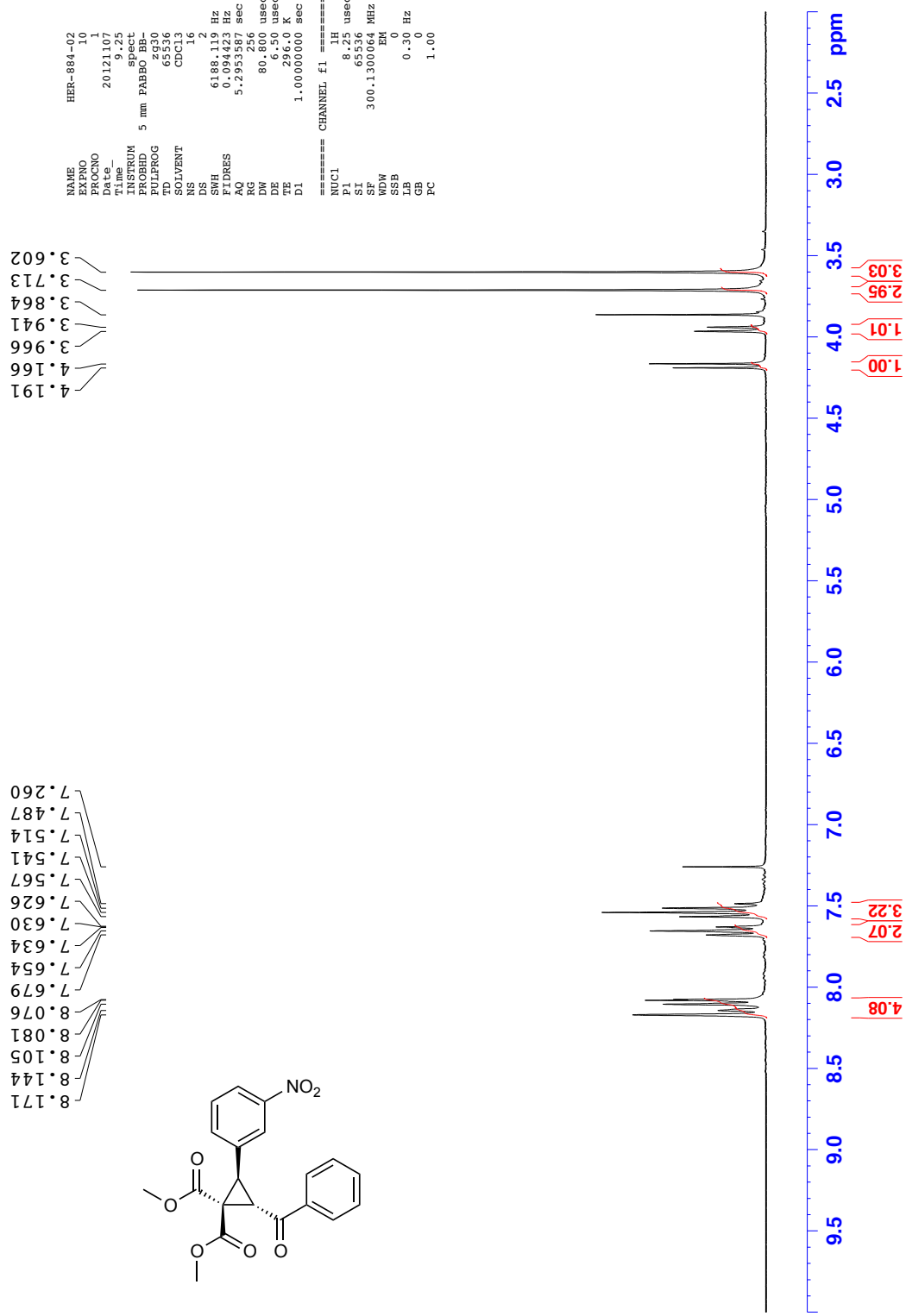
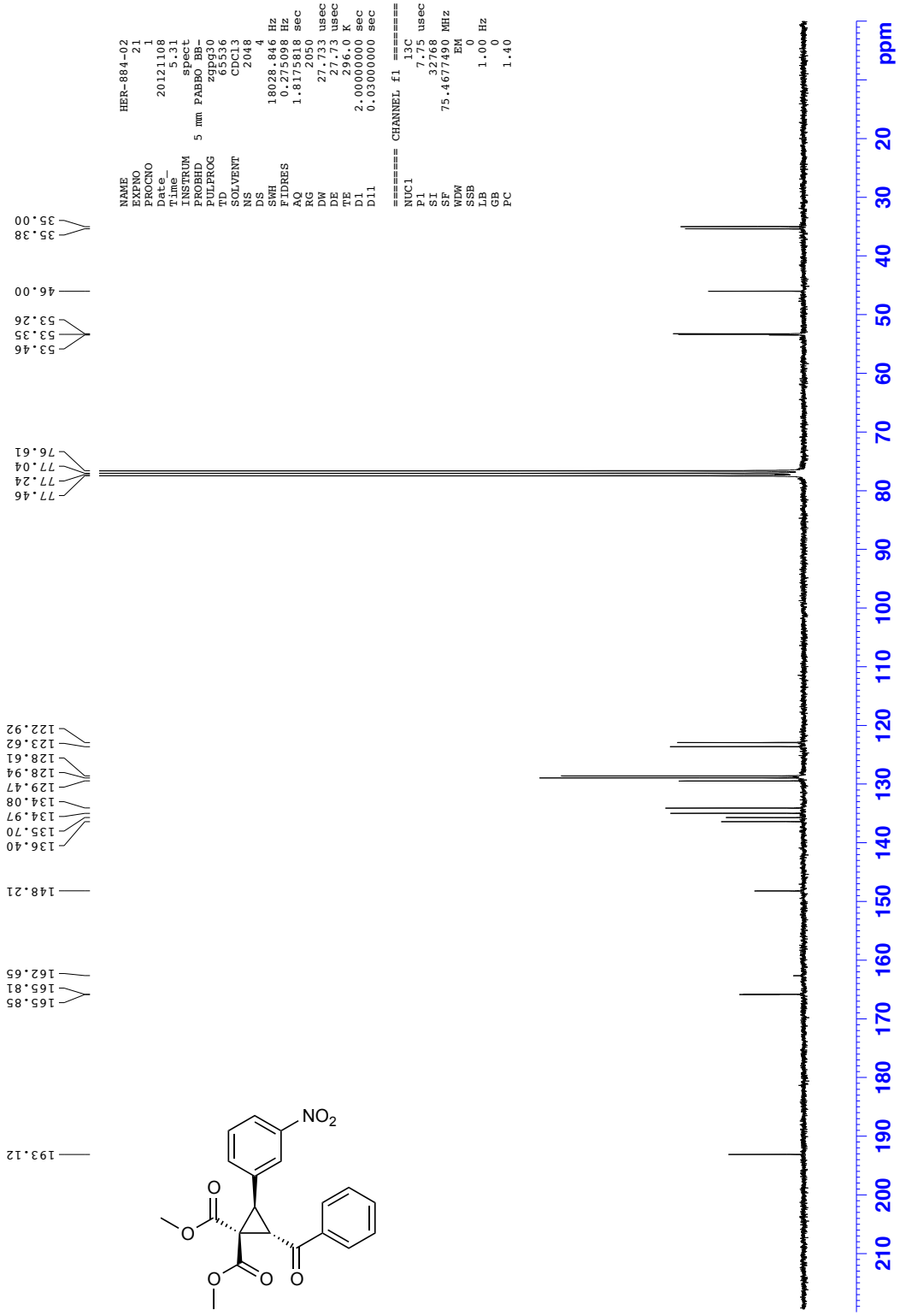
Date_ 20121108
Time 13.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953407 sec
RG 406
DW 80.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.25 usec
SI 65536
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.548
3.709
3.858
3.884
4.055
4.080
6.804
6.984
6.989
7.228
7.260
7.273
7.285
7.296
7.302
7.312
7.319
7.338
7.345
7.351
7.354
7.469
7.477
7.484
7.500
7.506
7.514
8.015
8.023
8.029
8.045
8.052
8.059
O
O
O
O
O
O
Cl
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
3.05
3.03
1.00
1.03
5.05
2.06
2.02

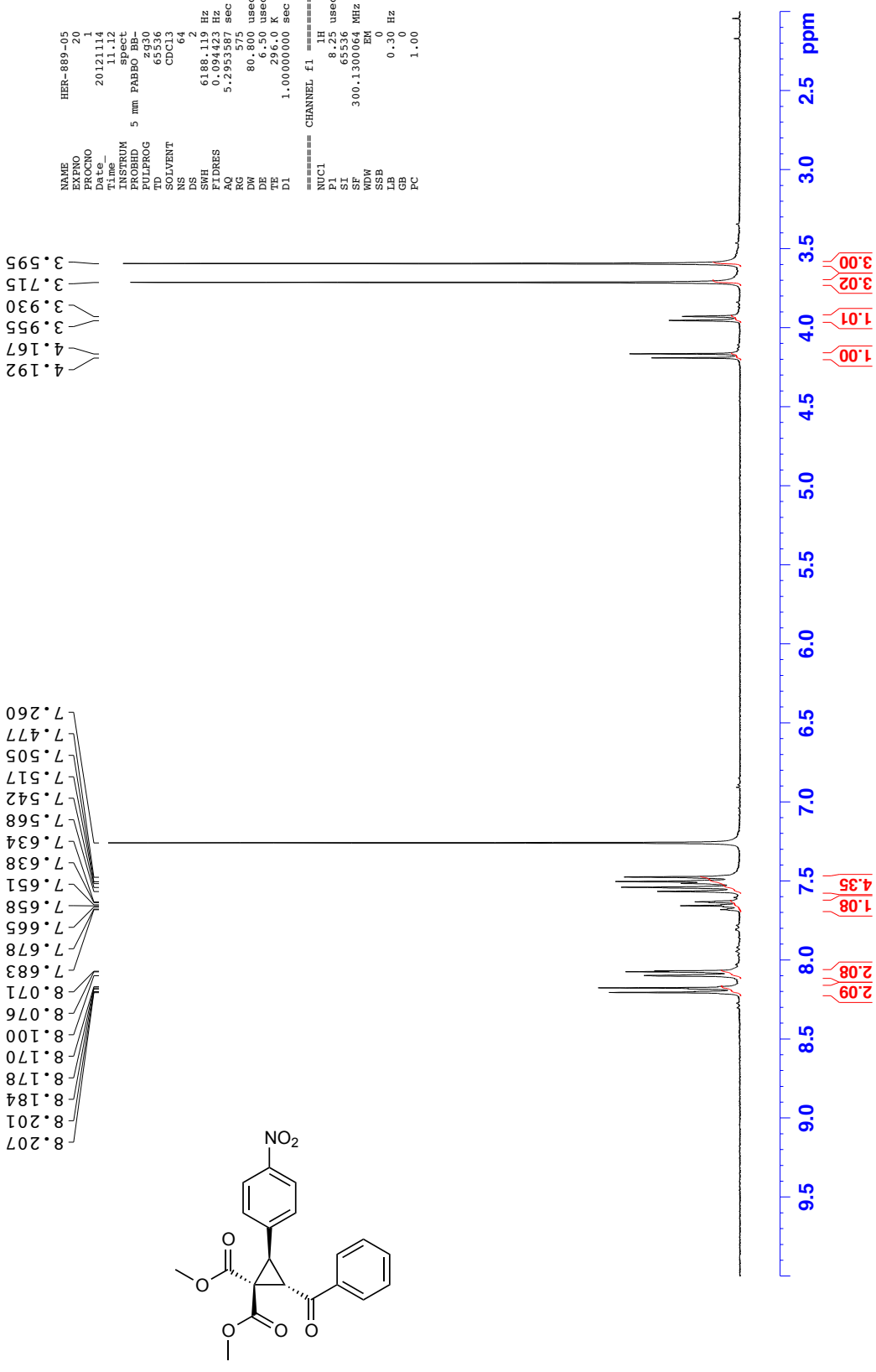

NAME HER-867-02
EXPNO 21
PROCNO 1
Date_ 20121013
Time 4.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 2050
DW 27.733 usec
DE 27.73 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
35.02
36.46
46.06
53.04
53.09
76.60
77.02
77.23
77.45
127.59
127.87
128.10
128.45
128.77
129.17
129.49
129.95
133.16
134.98
140.34
166.02
166.40
192.62
ppm
Cl
O
O
O
O
O

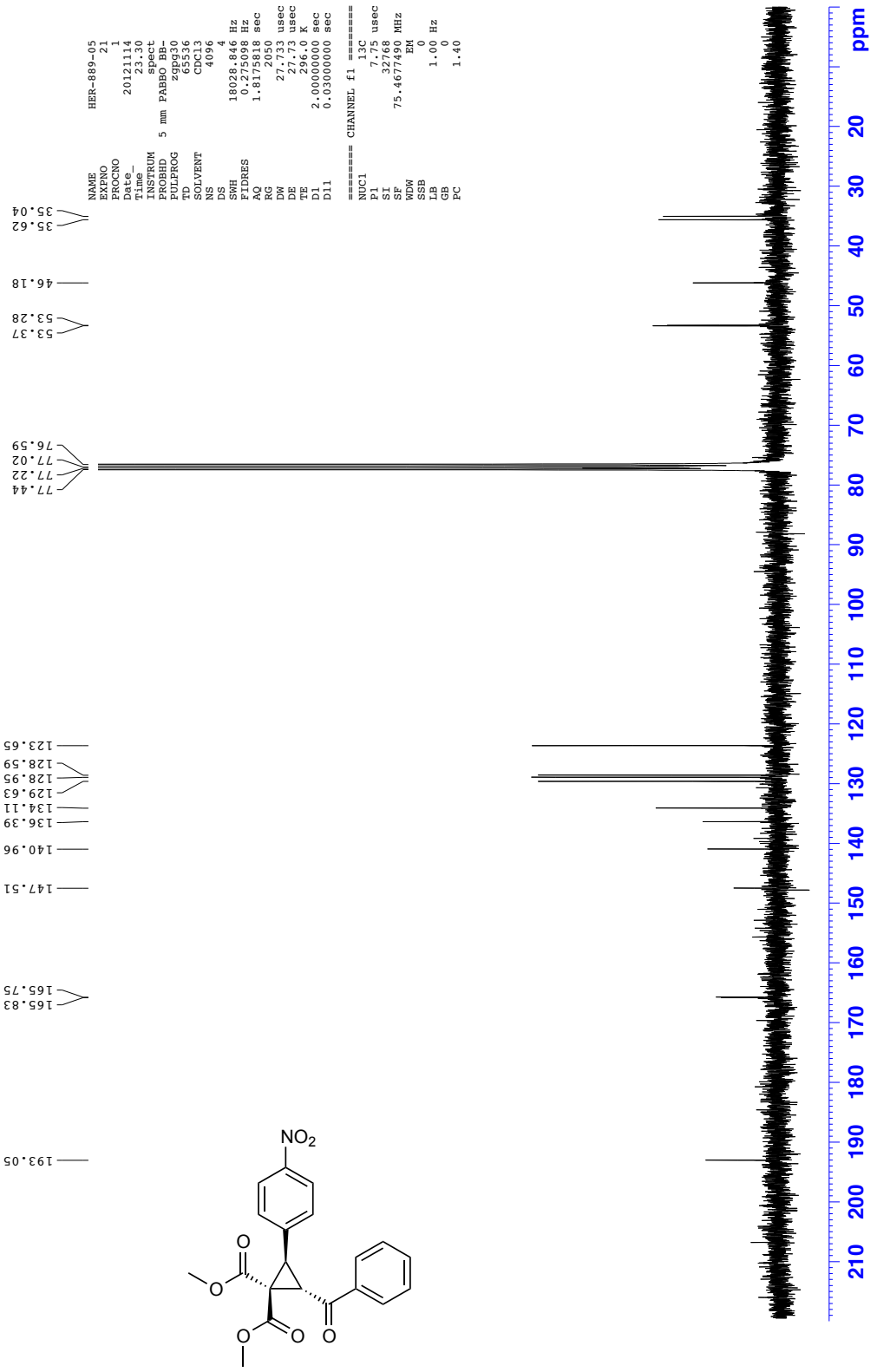

NAME HER-868-02
EXPNO 30
PROCNO 1
Date_ 20121108
Time 13.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953575 sec
RG 575
DW 80.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.25 usec
SI 65536
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.547
3.709
3.859
3.869
3.885
4.062
4.088
7.150
7.160
7.166
7.188
7.210
7.217
7.226
7.260
7.273
7.286
7.299
7.303
7.315
7.327
7.346
8.098
8.108
8.115
8.126
8.131
8.137
8.148
8.155
8.165
3.05
3.05
1.07
1.00
2.23
5.07
2.01
ppm
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
O
O
O
O
O
F

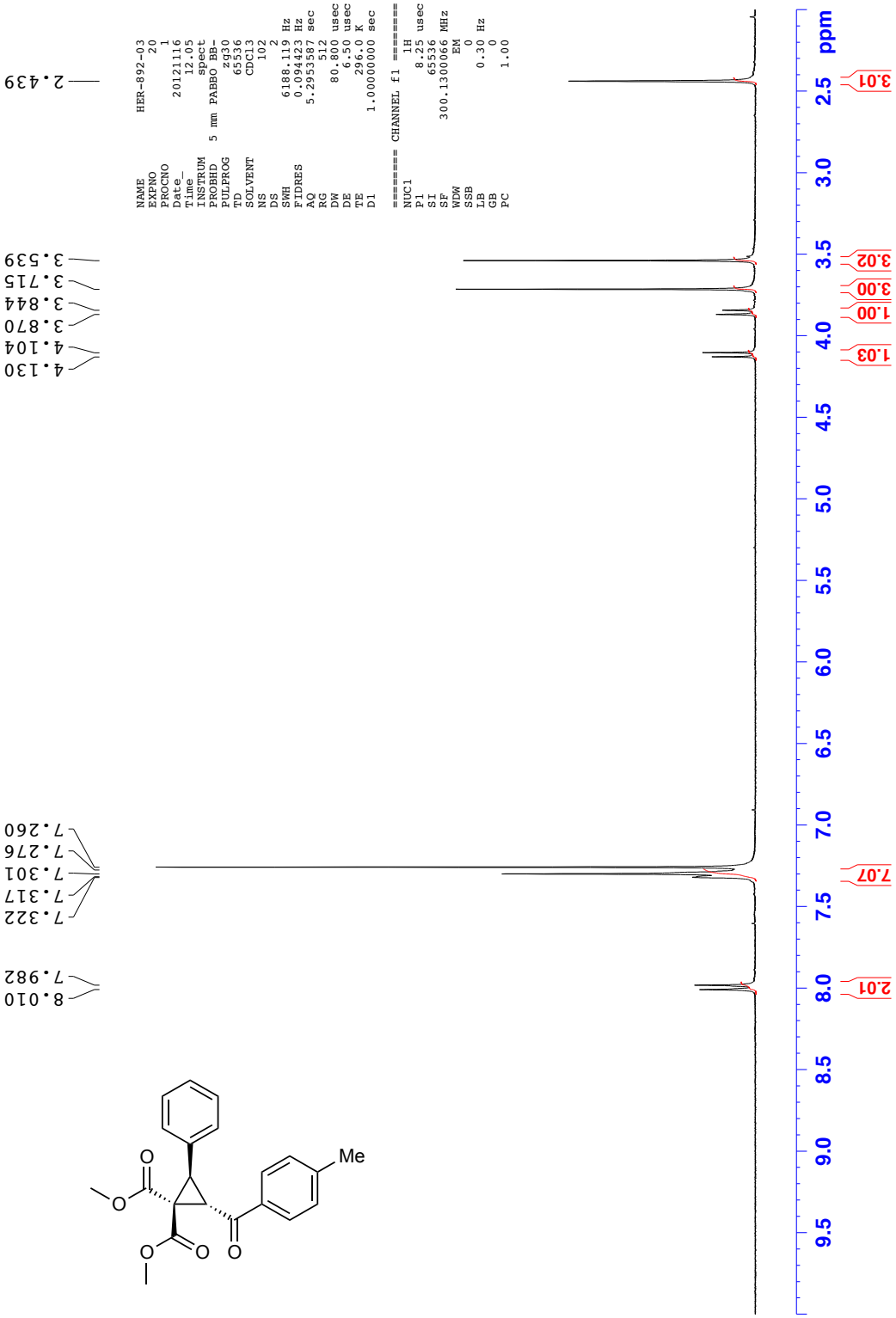

NAME HER-868-02
EXPNO 21
PROCNO 1
Date_ 20121013
Time 8.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 2050
DW 27.733 usec
DE 27.73 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
35.03
36.41
45.98
53.02
53.06
76.59
77.01
77.22
77.44
115.85
116.14
127.85
128.45
131.21
131.34
133.10
133.14
133.23
164.48
166.08
166.46
167.87
192.18
F
O
O
O
O
O
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

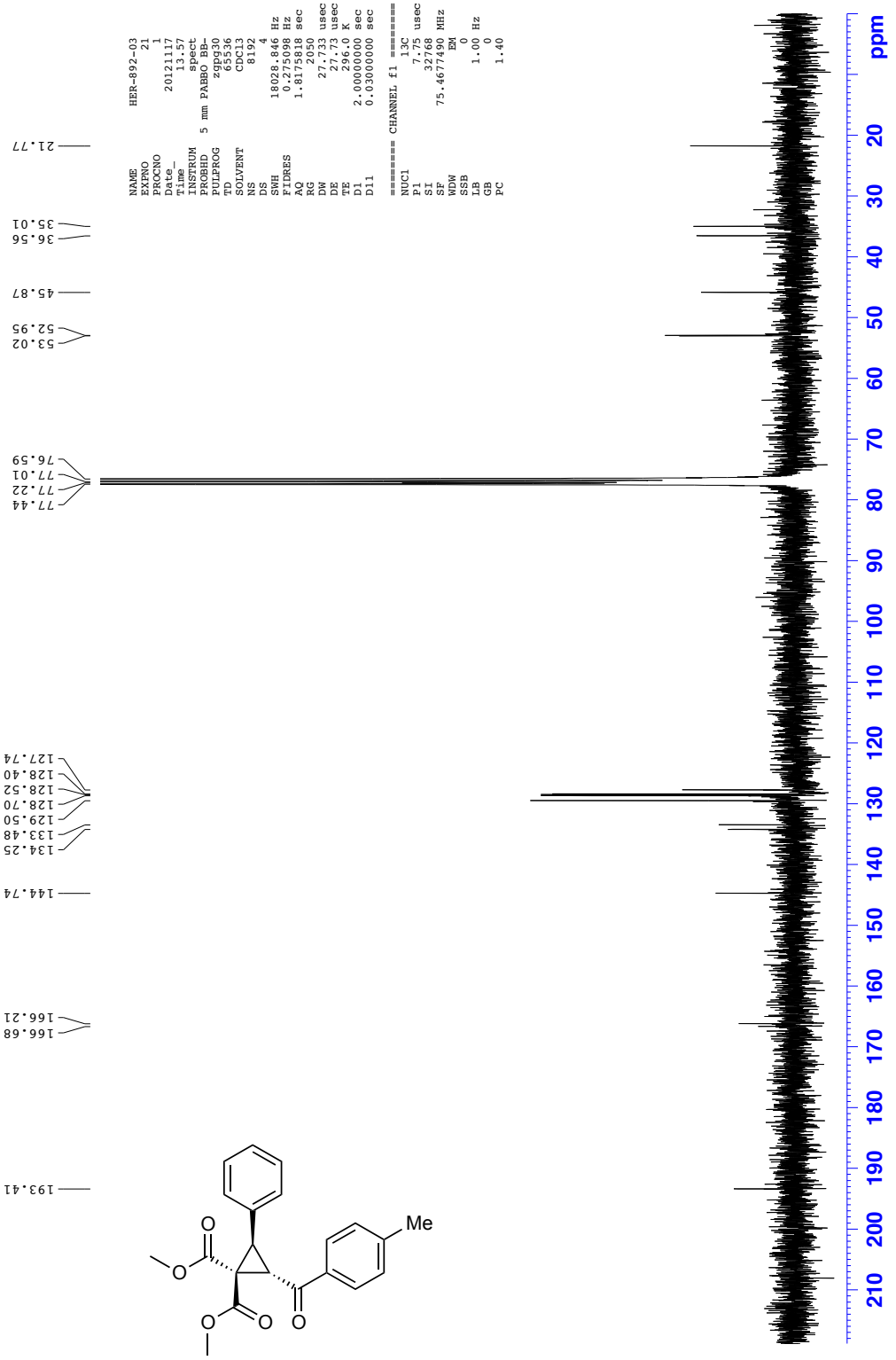

NAME HER-884-02
EXPNO 10
PROCNO 1
Date_ 20121107
Time 9.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953356 sec
RG 256
DW 80.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.25 usec
SI 65536
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.602
3.713
3.864
3.941
3.966
4.166
4.191
7.260
7.487
7.514
7.541
7.567
7.626
7.630
7.634
7.654
7.679
8.076
8.081
8.105
8.144
8.171
NO2
O
O
O
O
O
O
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
4.08
2.07
3.22
1.00
1.01
2.95
3.03


NAME HER-884-02
EXPNO 21
PROCNO 1
Date_ 20121108
Time 5.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 2050
DW 27.733 usec
DE 27.73 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
35.00
35.38
46.00
53.26
53.35
53.46
76.61
77.04
77.24
77.46
122.92
123.62
128.61
128.94
129.47
134.08
134.97
135.70
136.40
148.21
162.65
165.81
165.85
193.12
ppm
NO2
O
O
O
O
O


NAME HER-889-05
EXPNO 20
PROCNO 1
Date_ 20121114
Time 11.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953575 sec
RG 575
DW 80.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.25 usec
SI 65536
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.595
3.715
3.930
3.955
4.167
4.192
7.260
7.477
7.505
7.517
7.542
7.568
7.634
7.638
7.651
7.658
7.665
7.678
7.683
8.071
8.076
8.100
8.170
8.178
8.184
8.201
8.207
ppm
3.00
3.02
1.01
1.00
4.35
1.08
2.08
2.09
NO2
O
O
O
O
O


NAME HER-889-05
EXPNO 21
PROCNO 1
Date_ 20121114
Time_ 23.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4096
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.81775818 sec
RG 2050
DW 27.733 usec
DE 27.73 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.75 usec
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
35.04
35.62
46.18
53.28
53.37
76.59
77.02
77.22
77.44
123.65
128.59
128.95
129.63
134.11
136.39
140.96
147.51
165.75
165.83
193.05
NO2
O
O
O
O
O
ppm

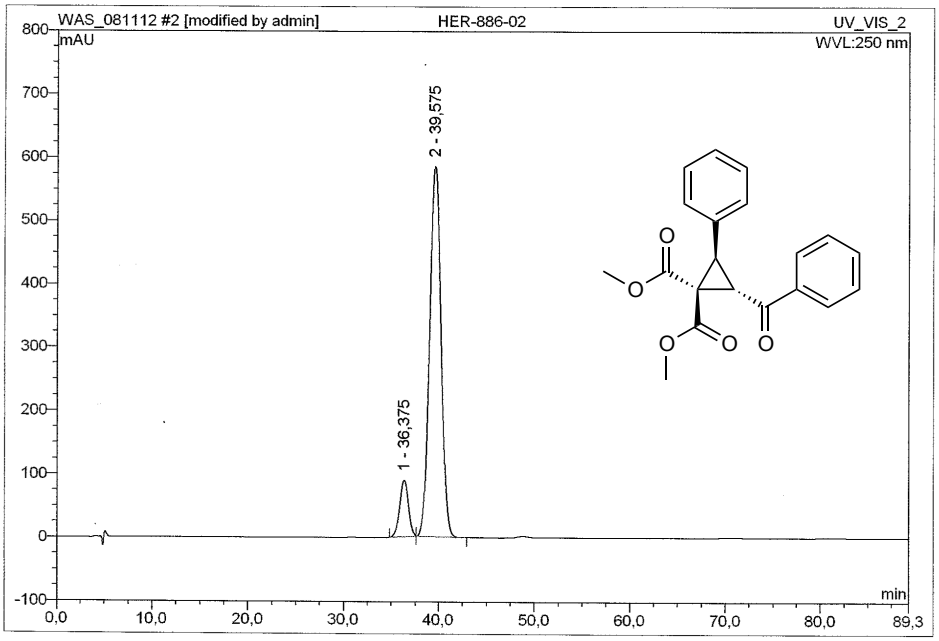

NAME HER-892-03
EXPNO 20
PROCNO 1
Date_ 20121116
Time_ 12.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 102
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 512
DW 80.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.25 usec
SF 65536
SI 300.1300066 MHz
SF EM
WDW 0
SSB 0.30 Hz
LB 0
GB 1.00
PC
2.439
3.539
3.715
3.844
3.870
4.104
4.130
7.260
7.276
7.301
7.317
7.322
7.982
8.010
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
3.01
3.02
3.00
1.00
1.03
7.07
2.01
Me
O
O
O
O
O
O


HER-892-03
NAME
EXPNO
PROCNO
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
D11
======== CHANNEL f1 ========
NUC1
P1
PL1
SF
WDW
SSB
LB
GB
PC
21.77
35.01
36.56
45.87
52.95
53.02
76.59
77.01
77.22
77.44
127.74
128.40
128.52
128.70
129.50
133.48
134.25
144.74
166.21
166.68
193.41
Me
O
O
O
O
O
ppm

3. HPLC-Chromatograms (Chiral Stationary Phase):

2 HER-886-02

Sample Name:	**HER-886-02**	*Injection Volume:*	**10,0**
Vial Number:	**RA2**	*Channel:*	**UV_VIS_2**
Sample Type:	**unknown**	*Wavelength:*	**n.a.**
Control Program:	**100min_55zu45**	*Bandwidth:*	**n.a.**
Quantif. Method:	**default**	*Dilution Factor:*	**1,0000**
Recording Time:	**8.11.2012 11:20**	*Sample Weight:*	**1,0000**
Run Time (min):	**89,26**	*Flow ml/min:*	**0,70**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	36,38	n.a.	88,564	93,816	10,42	n.a.	BMb*
2	39,58	n.a.	584,258	806,212	89,58	n.a.	bMB*
Total:			672,822	900,028	100,00	0,000	

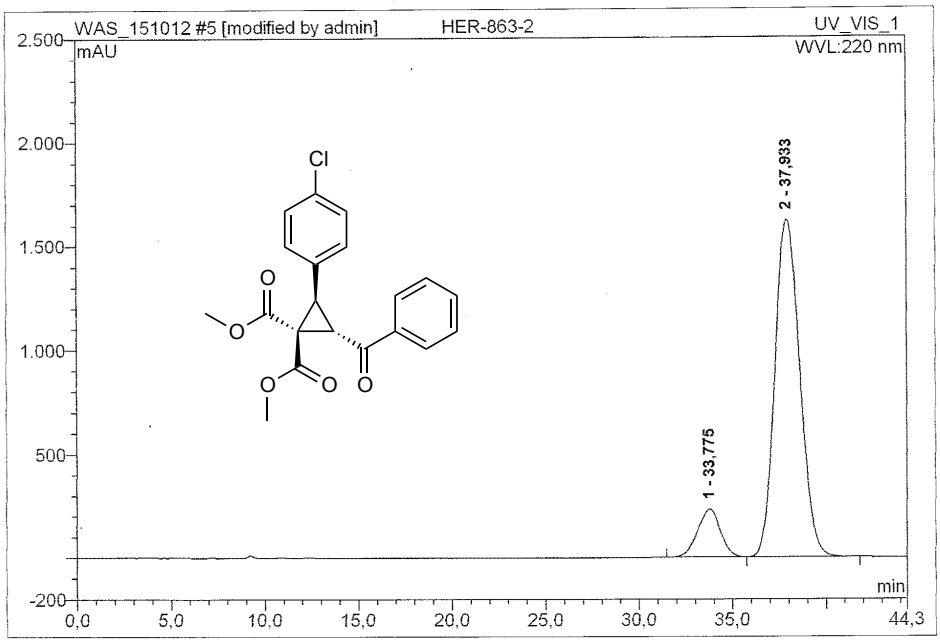
55 Vol% H2O
45 Vol% Acetonitril

Rueckl/Integration

Chromeleon (c) Dionex 1996-2006
Version 6.80 SR10 Build 2818 (166959)

5 HER-863-2

Sample Name:	**HER-863-2**	*Injection Volume:*	**10,0**
Vial Number:	**RA1**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**n.a.**
Control Program:	**OD_R_60_50zu50**	*Bandwidth:*	**n.a.**
Quantif. Method:	**default**	*Dilution Factor:*	**1,0000**
Recording Time:	**15.10.2012 15:06**	*Sample Weight:*	**1,0000**
Run Time (min):	**44,34**	*Flow ml/min:*	**0,70**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	33,78	n.a.	231,197	327,902	11,58	n.a.	Ru
2	37,93	n.a.	1619,946	2504,042	88,42	n.a.	BMB
Total:			1851,143	2831,944	100,00	0,000	

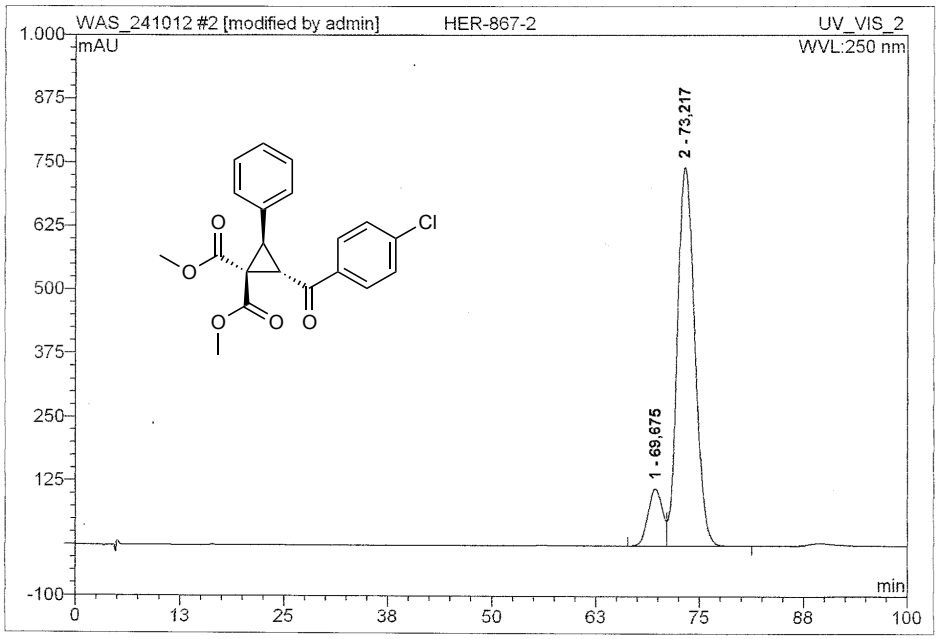
45 Vol% H2O
55 Vol% Acetonitril

50
50

Rueckl/Integration

2 HER-867-2

Sample Name:	**HER-867-2**	*Injection Volume:*	**10,0**
Vial Number:	**RA2**	*Channel:*	**UV_VIS_2**
Sample Type:	**unknown**	*Wavelength:*	**n.a.**
Control Program:	**100min_55zu45**	*Bandwidth:*	**n.a.**
Quantif. Method:	**default**	*Dilution Factor:*	**1,0000**
Recording Time:	**24.10.2012 11:19**	*Sample Weight:*	**1,0000**
Run Time (min):	**100,00**	*Flow ml/min:*	**0,70**

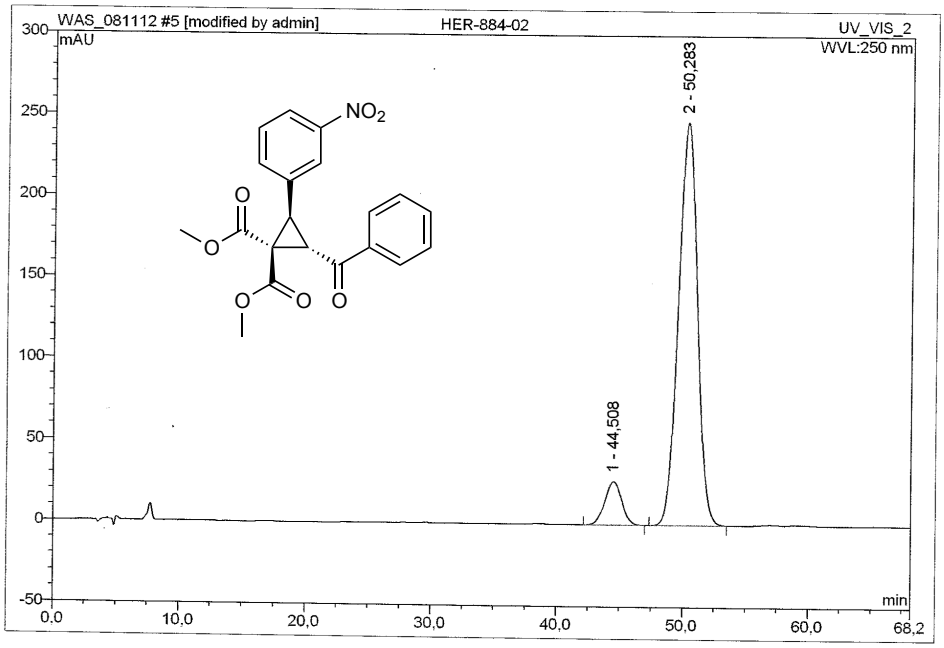


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	69,68	n.a.	112,245	225,876	11,18	n.a.	BM *
2	73,22	n.a.	740,740	1795,105	88,82	n.a.	MB*
Total:			852,985	2020,981	100,00	0,000	

55 Vol% H2O
45 Vol% Acetonitril

5 HER-884-02

Sample Name:	**HER-884-02**	*Injection Volume:*	**10,0**
Vial Number:	**RA1**	*Channel:*	**UV_VIS_2**
Sample Type:	**unknown**	*Wavelength:*	**n.a.**
Control Program:	**100min_55zu45**	*Bandwidth:*	**n.a.**
Quantif. Method:	**default**	*Dilution Factor:*	**1,0000**
Recording Time:	**8.11.2012 14:03**	*Sample Weight:*	**1,0000**
Run Time (min):	**68,17**	*Flow ml/min:*	**0,70**

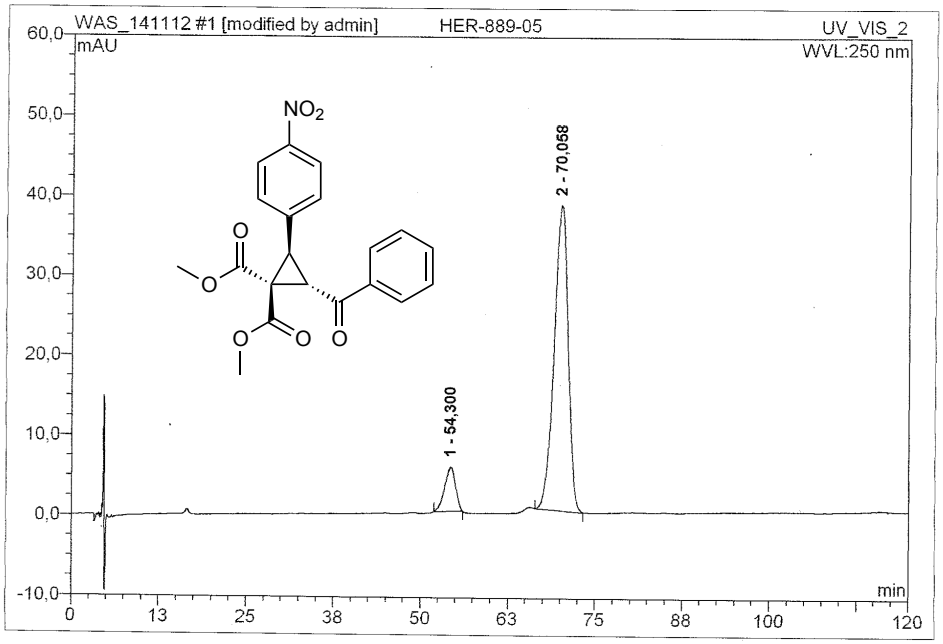


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	44,51	n.a.	26,727	42,069	8,89	n.a.	BMB
2	50,28	n.a.	246,803	431,181	91,11	n.a.	BMB
Total:			273,529	473,251	100,00	0,000	

55 Vol% H2O
45 Vol% Acetonitril

1 HER-889-05

Sample Name:	**HER-889-05**	*Injection Volume:*	**10,0**
Vial Number:	**RA1**	*Channel:*	**UV_VIS_2**
Sample Type:	**unknown**	*Wavelength:*	**n.a.**
Control Program:	**120min_55zu45**	*Bandwidth:*	**n.a.**
Quantif. Method:	**default**	*Dilution Factor:*	**1,0000**
Recording Time:	**14.11.2012 9:37**	*Sample Weight:*	**1,0000**
Run Time (min):	**120,00**	*Flow ml/min:*	**0,70**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	54,30	n.a.	5,510	9,930	10,13	n.a.	BMB*
2	70,06	n.a.	38,117	88,056	89,87	n.a.	BMB
Total:			43,627	97,986	100,00	0,000	

55 Vol% H2O
45 Vol% Acetonitril