

Diversity-oriented synthesis of chromenopyrrolidines from azomethine ylides and 2-hydroxybenzylideneindenediones *via* base controlled regio-divergent (3+2) cycloaddition.

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Table of Contents:

1.	General information	S2-S2
2.	Optimization of reaction conditions for 3aa and 4aa	S3-S3
3.	Determination of rotamers	S4-S5
4.	Derivatization of adducts 3 (<i>rac-9</i>)	S6-S6
5.	Derivatization of 4ha	S7-S7
6.	Plausible reaction mechanism	S7-S7
7.	Control experiments	S8-S8
8.	Chiral bases screened for the reaction between 1a and 2a	S9-S9
9.	Optimization of reaction conditions for the generation of chiral adducts 9	S9-S10
10.	Derivatization of chiral adducts 9	S10-S10
11.	Experimental procedures	
	Typical procedure for the preparation of iminodiester 1	S11-S11
	Typical procedure for the synthesis of 2-arylidene indanediones 2	S11-S11
	Typical procedure for the generation of 3 (<i>rac-9</i>)	S12-S12
	Typical procedure for the generation of 4	S12-S12
	Typical procedure for the hemiaminalization of 3 (<i>rac-9</i>)/ 9	S12-S13
	Typical procedure for the generation of chiral adducts 9	S13-S13
12.	Analytical data for all new compounds	S13-S57
13.	References	S58-S58
14.	X-ray structures of 3aa , 4aa and 9ae	S58-S61
15.	NMR Spectra of all new compounds	S62-S190
16.	HPLC chromatograms of adducts 9	S191-S204

1. General Information

All solvents and reagents were used as purchased from commercial suppliers without further purification. Starting materials and catalysts which were not commercially available were synthesized by the previously reported methods. Analytical thin layer chromatography (TLC) was performed on precoated alumina-backed silica gel plates (Merck 60 F254, 0.2 mm thickness) which were developed using UV fluorescence and iodine. Flash-chromatography was performed on silica gel (Merck Kieselgel 60Å 230-400 mesh). Melting points were measured on a hotstage melting point apparatus and are uncorrected. IR spectra were recorded on a Perkin Elmer 500 spectrometer and only selected peaks are shown. ¹H-NMR spectra were recorded on a Bruker Avance 400 MHz spectrometer, while ¹³C-NMR spectra were recorded on a 100 MHz instrument. Chemical shifts are reported in δ ppm referenced to an internal TMS standard for ¹H-NMR and chloroform-d ($\delta = 77.0$ ppm) for ¹³C-NMR. In some cases, while the chloroform-d couldn't dissolve the analyte well, the acetone-d₆ was applied instead. The chemical shifts refer to an internal acetone-d₆ standard for ¹H-NMR ($\delta = 2.05$ ppm) and for ¹³C-NMR ($\delta = 29.8$ ppm). HRMS spectra were recorded on JEOL SX-102A. For transferring liquids on a microliter scale, a micropipette (VITLAB C121) of 2-20 μ L capacity was used. The X-ray diffraction measurements were carried out at 298 K on a KAPPA APEX II CCD area detector system equipped with a graphite monochromator and a Mo-K α fine-focus sealed tube ($k = 0.71073$ Å). Optical rotations were measured in CH₂Cl₂ on a JASCO co. DIP-1000 digital polarimeter with a 50 mm cell (c given in g/100 mL). The known iminodiesters **1a**, **1b**, **1c**, **1d** and **1f** were synthesized following the procedure reported earlier by Xu et al.¹ and the iminodiesters **1e** and **1g** were synthesized following the procedure reported by our group.² Similarly, the known 2-hydroxybenzylidene indanediones **2a** and **2e** were synthesized following the procedure reported by our group.³ The iminodiester **1h** and the other 2-hydroxybenzylidene indandione substrates **2b**, **2c**, **2d**, **2f**, **2g** and **2h** were synthesized following the procedure mentioned in this supporting information.

2. Optimization of reaction conditions for the generation of **3aa** and **4aa**

Table 1. Optimization of the reaction conditions^a

Entry	Base	Solvent	T [°C]	t [h]	3aa/4aa Yield ^b [%]
1	DMAP	CHCl ₃	30	12	65/trace
2	DMAP	CH ₂ Cl ₂	30	18	66/trace
3	DMAP	toluene	30	12	61/trace
4	DMAP	THF	30	12	trace/trace
5	DMAP	EtOAc	30	12	30/trace
6	DMAP	CHCl ₃	40	9	65/trace
7	DMAP	CHCl ₃	50	6	66/trace
8 ^c	DMAP	CHCl ₃	30	18	71/trace
9	TMG	CHCl ₃	30	12	20/60
10	TMG	CH ₂ Cl ₂	30	12	18/59
11	TMG	toluene	30	12	20/58
12	TMG	EtOAc	30	12	11/56
13	TMG	CH ₃ CN	30	12	11/35
14	TMG	THF	30	12	10/57
15	TMG	THF	40	3	trace/64
16	TMG	THF	50	2	trace/67
17 ^d	TMG	THF	50	4	trace/82

^a Unless otherwise specified, all the reactions were carried out with **1a** (0.1 mmol), **2a** (1.0 equiv.), 4Å MS (100 mg) and base (20 mol%) in the given solvent (0.5 mL). ^b Determined by ¹H NMR analysis of crude reaction mixture using Ph₃CH as internal standard. ^c 4Å MS (30 mg) were used. ^d Reaction was carried out with **1a** (0.2 mmol), **2a** (1.2 equiv.), 4Å MS (200 mg), TMG (20 mol%) in THF (0.5 mL).

After establishing DMAP and TMG as suitable bases to preferentially result in **3aa** and **4aa** respectively, the effect of other parameters such as solvent, temperature, additives, and molar ratio of substrates were examined. Eventually, it was found that (i) solvents such as DCM and CHCl₃ that resulted in good solubility of **1** and **2** provided better yields of **3aa** as compared to THF and EtOAc (Table 1, entries 1, 2 Vs 4, 5) (ii) slight warming to 50 °C enhanced the reaction rate, although yield did not improve (entries 6 & 7) (iii) addition of molecular sieves improved the yield of **3aa** by effective trapping of generated EtOH (entry 8) (iv) optimal solvent for the formation of **4aa** is THF (entries 14-17) and (v) reaction temperature and molar ratio of **1** and **2** has significant effect on the yield of **4aa**. Based on our observations, it was deduced that the optimal conditions to carry out the reaction for selective synthesis of **3aa** and **4aa** were as listed in entries 8 and 17 respectively.

3. Determination of rotamers

The existence of adducts **3** as a mixture of rotamers was confirmed by the rapid exchange of CH₃ protons of ethyl group of **3aa** in different deuterated solvents as shown below.

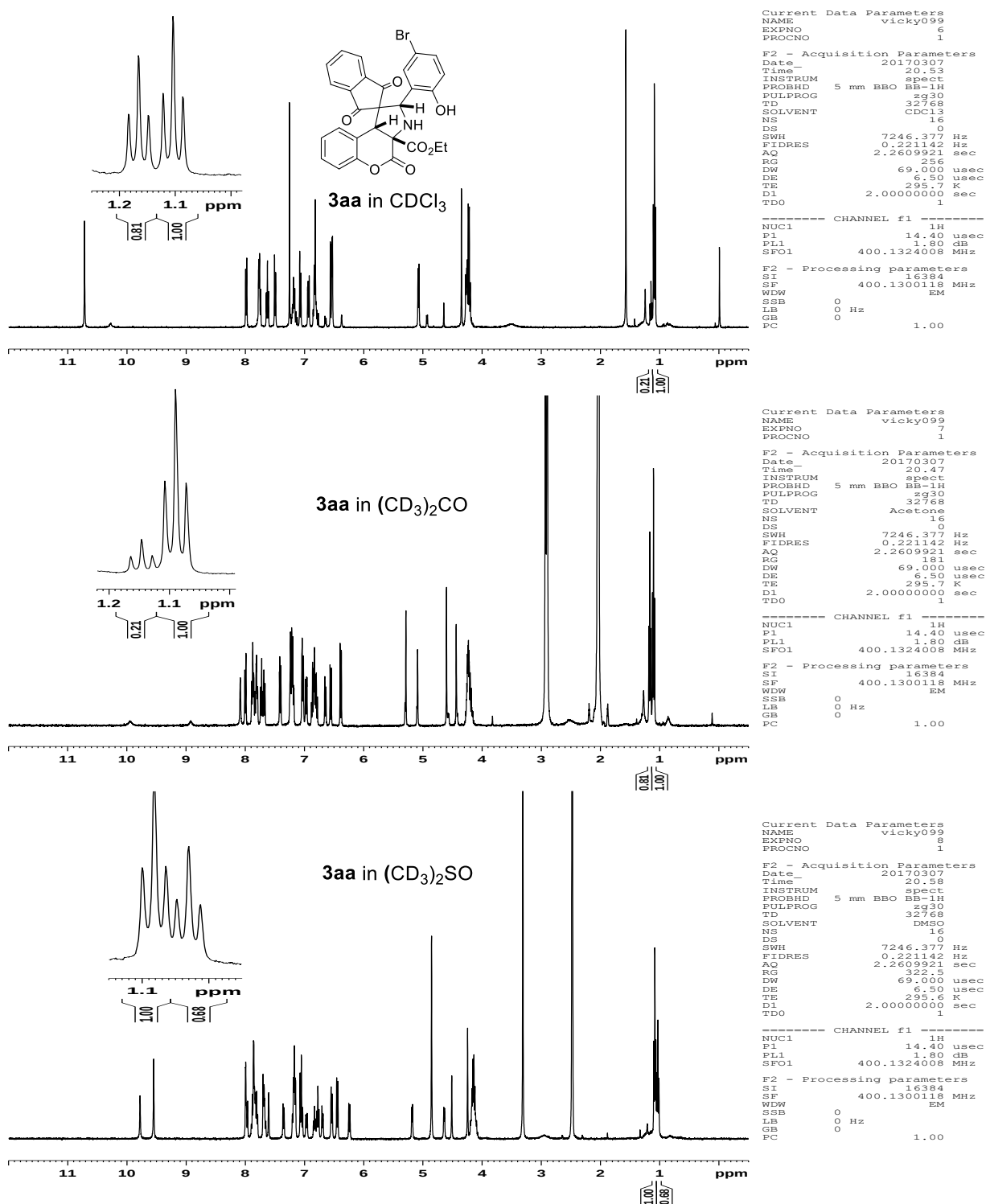


Figure 1. Comparison of ¹H NMR spectra of **3aa** in different deuterated solvents

In case of **3ha**, mixture of three rotamers could be seen. For clear understanding please refer to the ^{13}C NMR spectrum of **3ha** (page S100).

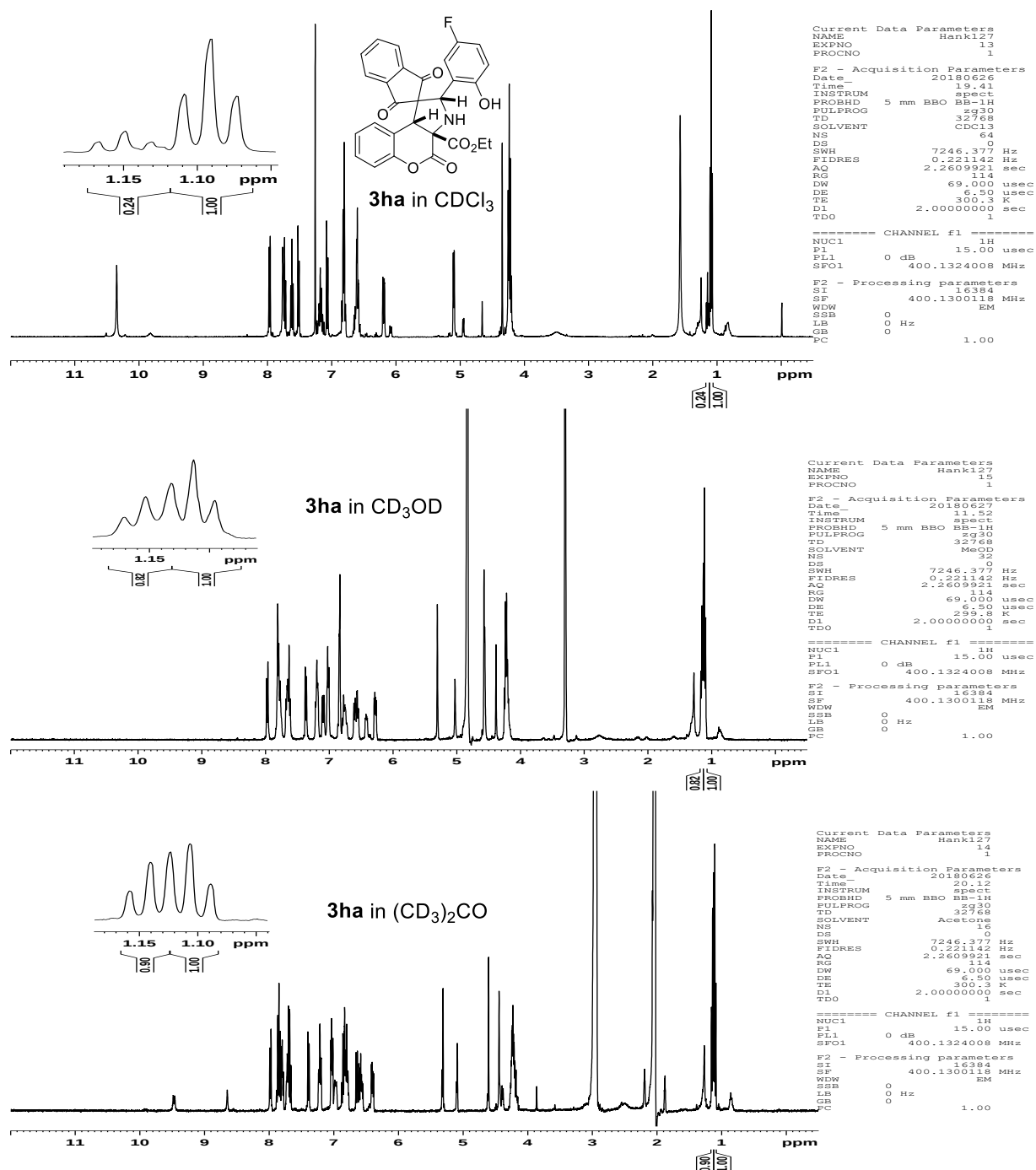
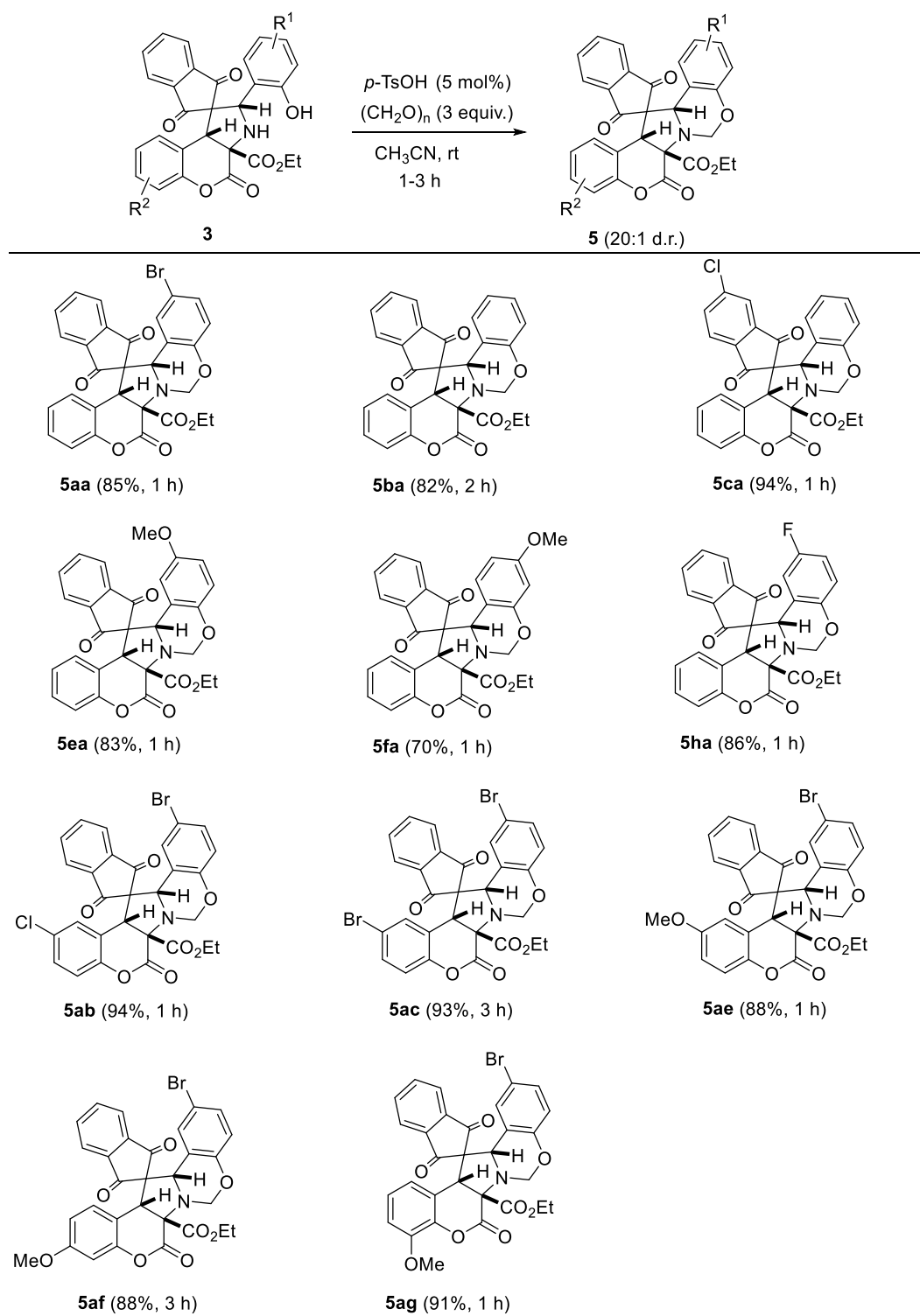


Figure 2. Comparison of ^1H NMR spectra of **3ha** in different deuterated solvents

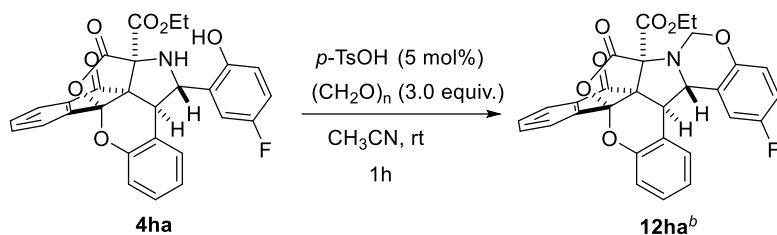
4. Derivatization of adducts **3**



Scheme 1. Hemiaminalization of adducts^a

^a **3** (0.1 mmol), PTSA (5 mol%) and paraformaldehyde (3.0 equiv.) in CH_3CN (0.5 mL). Reported yields are for the isolated product.

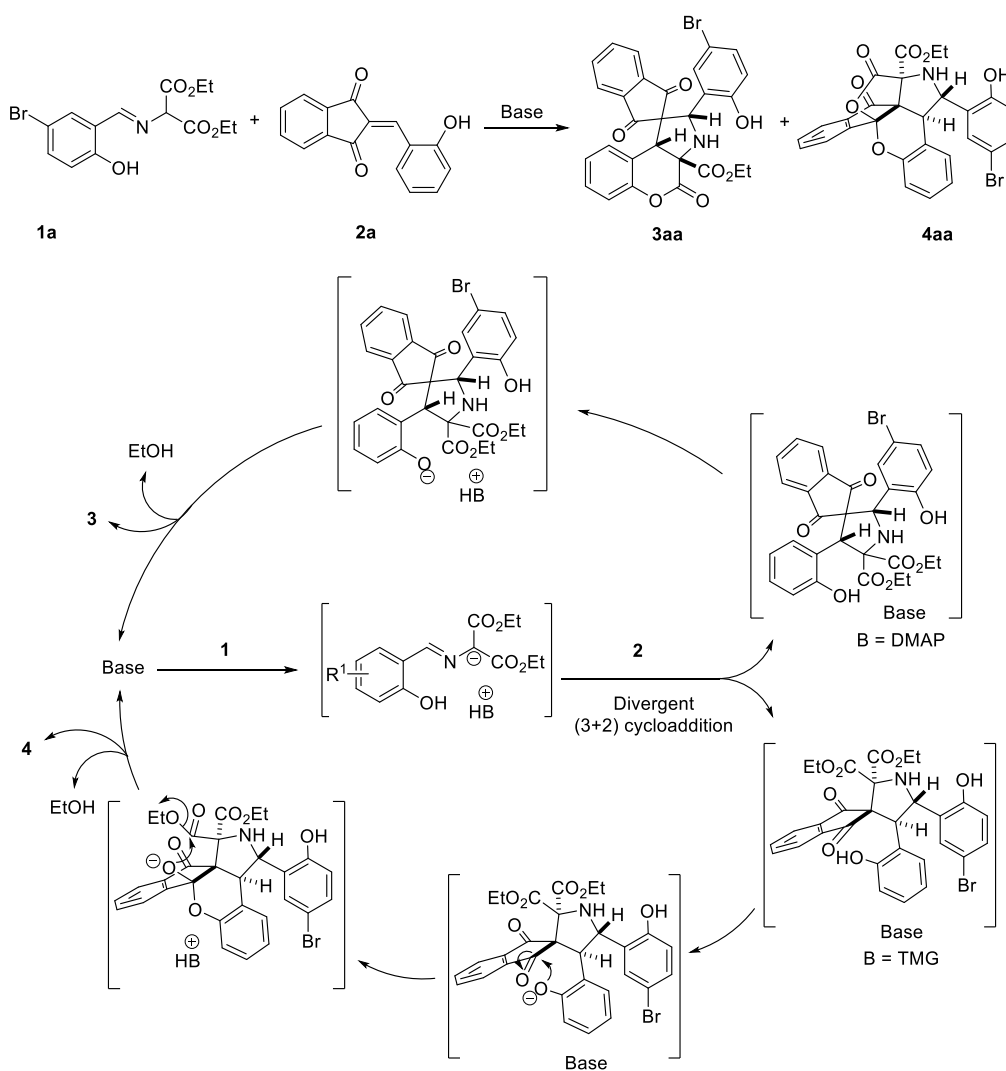
5. Derivatization of 4ha



Scheme 2. Hemiaminalization of 4ha^a

^a Reaction was performed with **4ha** (0.05 mmol) in CH₃CN (0.25 mL). ^b **12ha** was contaminated with very minor amount of some impurity that could not be separated.

6. Plausible reaction mechanism



Scheme 3. Plausible mechanism for the formation of **3** and **4**

7. Control experiments

To understand the fate of the azomethine ylide in the presence of bases DMAP and TMG, two separate experiments were conducted. Accordingly, **1a** (0.1 mmol) and DMAP (1.0 equiv.) were mixed in CDCl_3 (0.5 mL) and the NMR was recorded immediately. The NMR spectrum revealed the broadening of signals of the phenolic proton (H_c) as well as the proton flanked by the diester (H_a) while the imine proton (H_b) was unchanged. Similar experiment was conducted with TMG (1 equiv) and the NMR spectrum surprisingly revealed the diminishing of the intensity of the imine proton (H_b) along with the proton flanked by the diester (H_a) while the phenolic proton (H_c) could not be observed. These observations revealed that the configuration of the azomethine ylide is indeed different in the presence of DMAP and TMG.

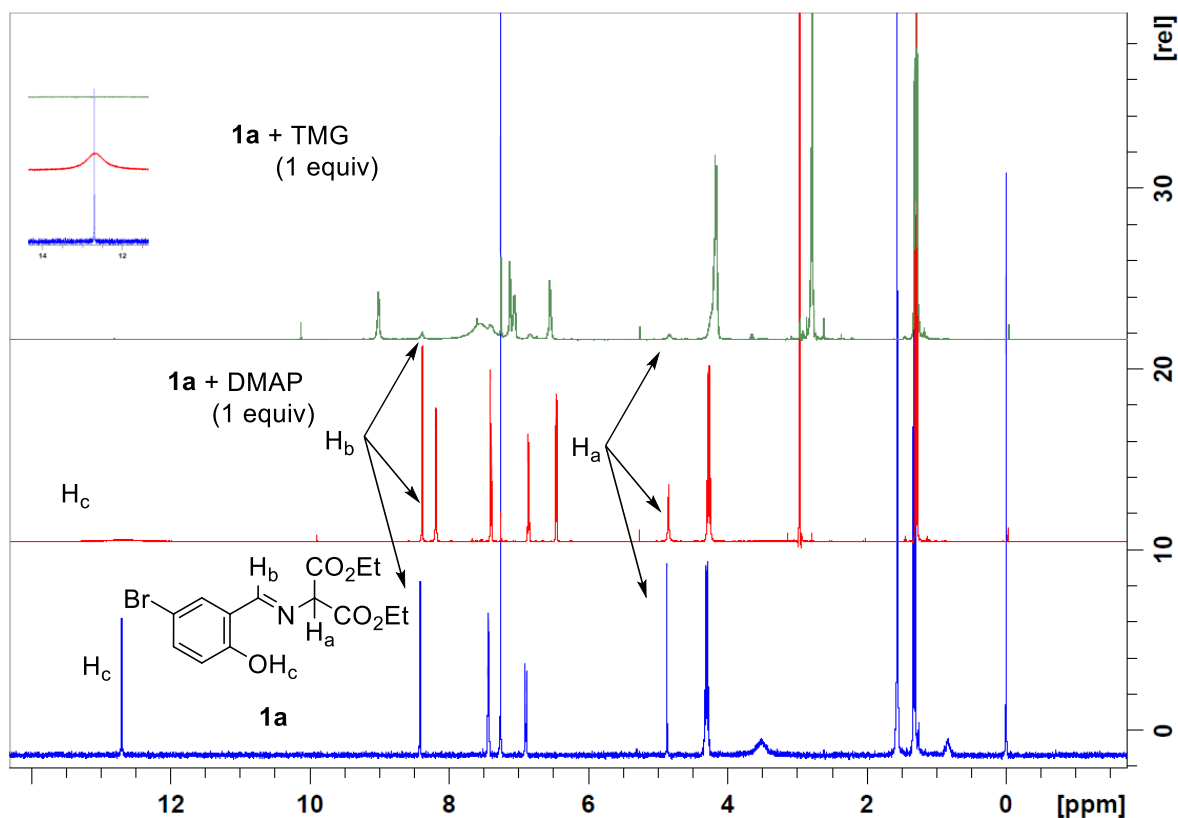


Figure 3. NMR behavior of azomethine ylide in the presence of DMAP and TMG

8. Chiral bases screened for the reaction between 1a and 2a

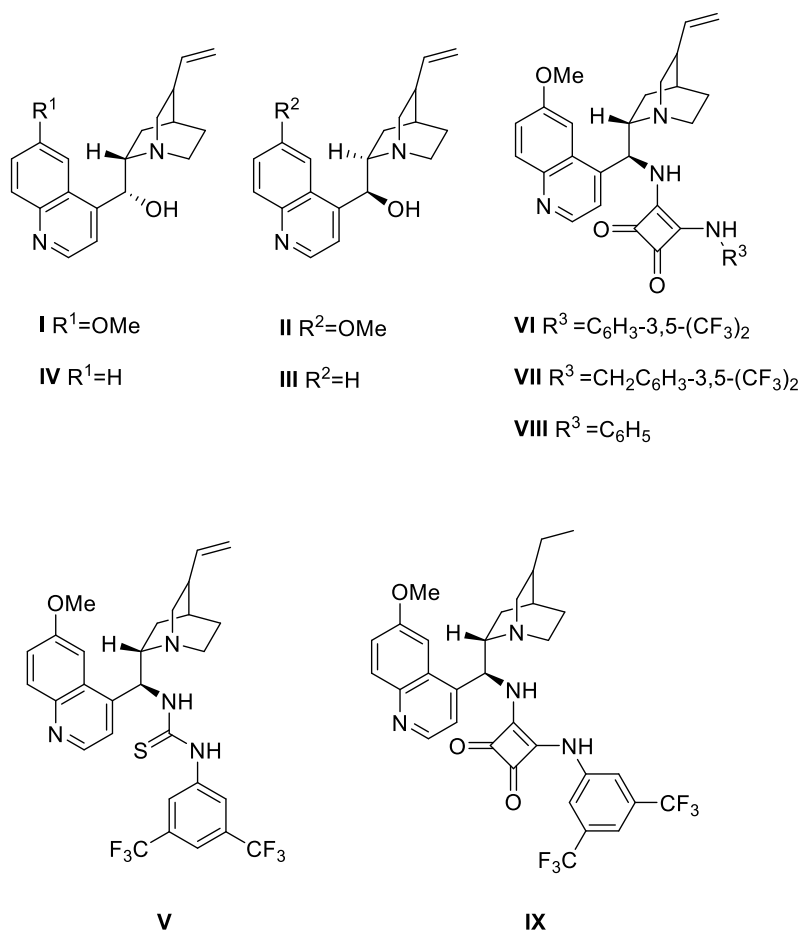
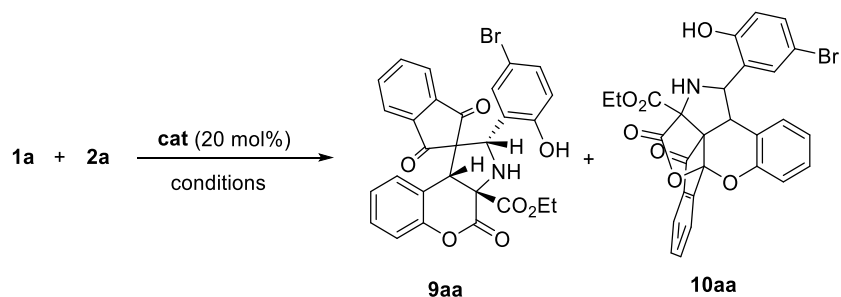


Figure 4. Catalysts screened for the cascade reaction

9. Optimization of reaction conditions for the generation of chiral adducts 9

Table 2. Optimization of reaction conditions^a

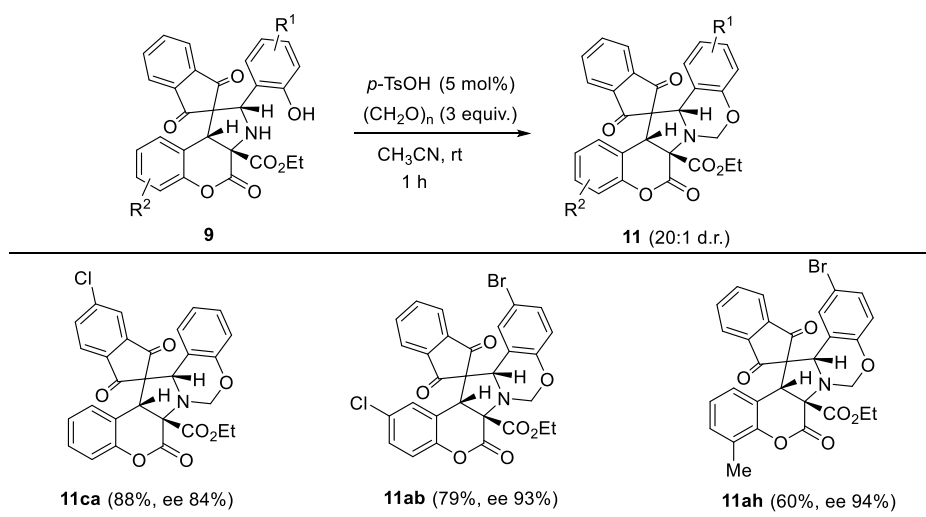


Entry	Cat.	Solvent	T [°C]	<i>t</i> ^b [h]	Yield ^c [%] 9aa/10aa	9aa ee ^d [%]
1	I	CHCl ₃	30	42	43/32	39(<i>ent</i>)
2	II	CHCl ₃	30	35	42/30	29
3	III	CHCl ₃	30	42	48/37	19(<i>ent</i>)

4	IV	CHCl ₃	30	24	36/34	17
5	V	CHCl ₃	30	36	35/0	17(<i>ent</i>)
6	VI	CHCl ₃	30	12	71/0	73
7	VII	CHCl ₃	30	12	43/0	47
8	VIII	CHCl ₃	30	24	50/0	54
9	IX	CHCl ₃	30	8	72/0	76
10	IX	THF	0	48	12/0	n.d. ^e
11	IX	EtOAc	0	48	29/0	88
12	IX	CH ₃ CN	0	48	trace/0	n.d. ^e
13	IX	toluene	0	48	29/0	80
14	IX	PhCl	0	48	39/0	87
15	IX	CH ₂ Cl ₂	0	48	35/0	84
16	IX	DCE	0	48	12/0	n.d. ^e
17	IX	CHCl ₃	0	48	67/0	85
18	IX	CHCl ₃	-5	216	50/0	87
19 ^f	IX	CHCl ₃	-5	216	70/0	88
20 ^{f,g}	IX	CHCl ₃	-5	144	68/0	89
21 ^{g,h}	IX	CHCl ₃	-5	144	76/0	89

^a Unless otherwise specified, all reactions were carried out with **1a** (0.1 mmol), **2a** (1.0 equiv.) and 0.5 mL solvent was used. ^b Indicates the time after which no considerable improvement in the yield of **9aa/10aa** was observed. ^c Determined by ¹H NMR analysis of crude reaction mixture using Ph₃CH as internal standard. ^d Determined by HPLC analysis on chiral stationary phase. ^e n.d. = not determined. ^f Equivalents of **2a**: **1a** is 1: 1.2. ^g Reaction was carried out with **2a** (0.2 mmol) and **1a** (1.2 equiv.) in 0.5 mL CHCl₃. ^h 4Å MS (50 mg) were used.

10. Derivatization of adducts **9**

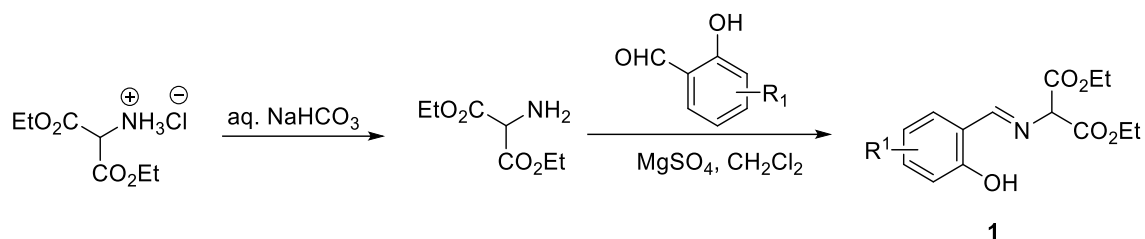


Scheme 4. Hemiaminalization of adducts **9**^{a,b}

^a **9** (0.1 mmol), PTSA (5 mol%) and paraformaldehyde (3.0 equiv.) in CH₃CN (0.5 mL). ^b Reported yields are for the isolated product.

11. Experimental Procedures

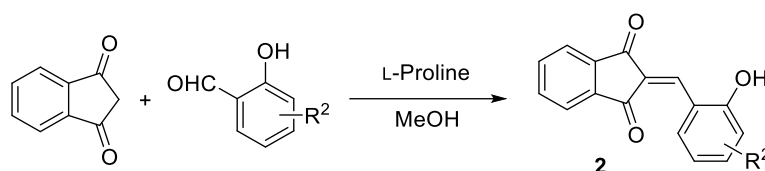
Typical procedure (TP-A) for the preparation of iminodiester 1.



To a stirred solution of diethyl aminomalonate hydrochloride (3.0 g, 14.2 mmol) and H₂O (30 mL) in a round-bottomed flask was added NaHCO₃ (1.1 equiv.) and stirred for 15 min. Thereafter, the reaction mixture was extracted with EtOAc (3 x 50 mL) and the combined organic layers were dried over MgSO₄, filtered and concentrated under vacuum to afford crude diethyl aminomalonate (2.302 g, 13.1 mmol) which was used without further purification.

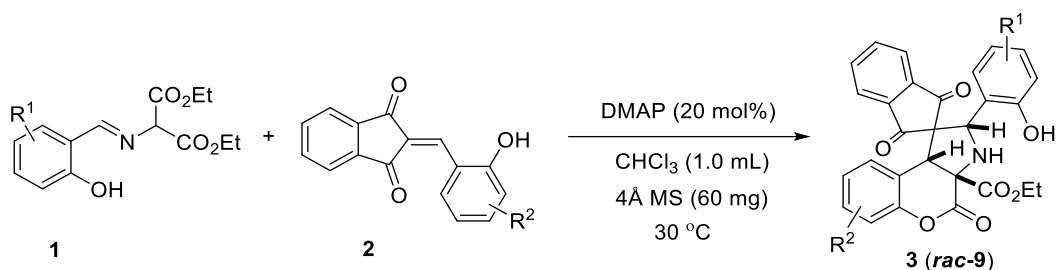
Substituted salicylaldehyde (1.0 equiv.) was added to a solution of diethyl aminomalonate (2.302 g, 13.1 mmol) and MgSO₄ (5.0 equiv) in CH₂Cl₂ (30 mL) and the resulting mixture was stirred for 48 h. Afterwards, MgSO₄ was removed by filtration and the filtrate was concentrated under reduced pressure to obtain crude **1**. If the product was solid, it was recrystallized from DCM/Hex to afford pure **1**. In case of liquids, the crude product was directly used without further purification.

Typical procedure (TP-B) for the preparation of 2-hydroxybenzylidene indandiones 2.



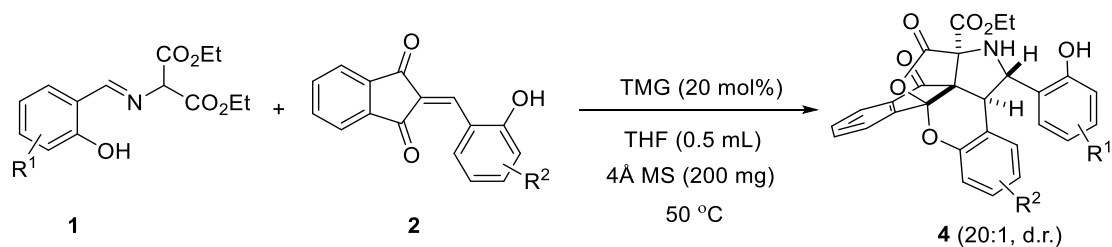
A 50-mL Schlenk flask, equipped with a magnetic stirring bar, was sequentially charged with a solution of 1,3-indanedione (1.5 g, 10 mmol), L-proline (0.3 equiv.) and salicylaldehyde (1.1 equiv) in methanol (20 mL). The reaction mixture was stirred for 12 h at room temperature. Thereafter, the resulting mixture was filtered under vacuum and the residue was washed with methanol and ethyl ether for several times to yield the corresponding products **2** as yellow solid.

Typical procedure (TP-C) for the generation of Chromeno[3,4-*b*]pyrrolidines 3 (*rac*-9).



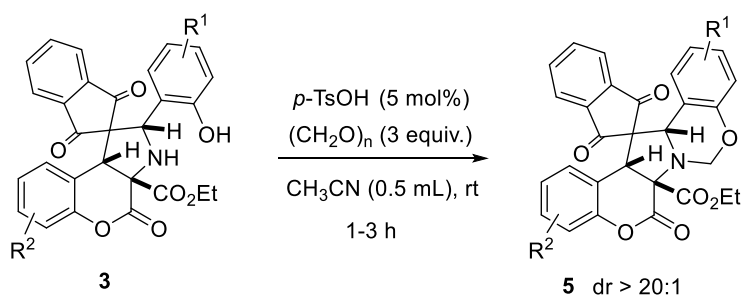
1 (0.2 mmol), **2** (1.0 equiv.), 4Å MS (60 mg), CHCl₃ (1.0 mL) and DMAP (20 mol %) were sequentially charged in a screw-capped glass vial equipped with a magnetic stirring bar and the resulting mixture was stirred at 30 °C. After the completion of reaction as indicated by TLC, solvent was removed *in vacuo* and the residue was subjected to flash column chromatography on silica gel to obtain the corresponding adducts **3**.

Typical procedure (TP-D) for the generation of Chromeno[3,4-*c*]pyrrolidines 4.



A screw-capped glass vial equipped with a magnetic stirring bar was sequentially charged with **2** (0.2 mmol), **1** (1.2 equiv.), 4Å MS (200 mg), THF (0.5 mL) and TMG (20 mol %) and the resulting mixture was stirred at 50 °C. After the completion of reaction as indicated by TLC, solvent was removed *in vacuo* and the residue was subjected to flash column chromatography on silica gel to obtain the adducts **4**.

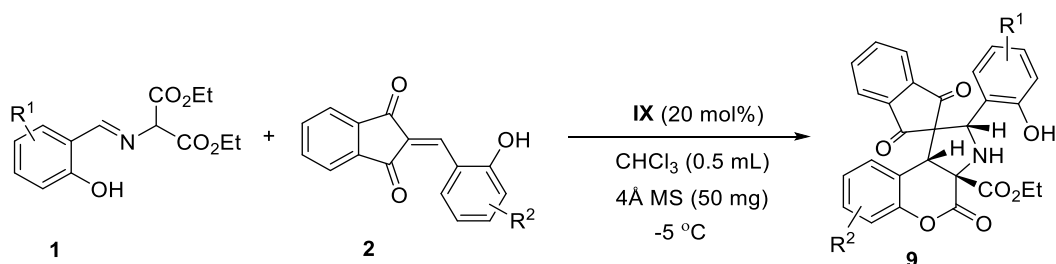
Typical procedure (TP-E) for the hemiaminalization of Chromeno[3,4-*b*]pyrrolidines 3.



A screw-capped glass vial equipped with a magnetic stirring bar was sequentially charged with **3** (0.1 mmol), paraformaldehyde (9.2 mg, 3 equiv.), MeCN (0.5 mL) and PTSA (5 mol %) and

the resulting mixture was stirred at 30 °C until the reaction was complete. Thereafter the mixture was diluted with saturated aqueous NaHCO₃ and extracted with DCM. Evaporation of the solvent *in vacuo* gave a residue which upon flash column chromatography on silica gel afforded the corresponding oxazinane adducts **5**.

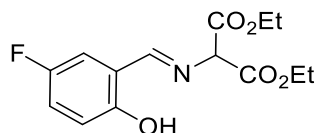
Typical procedure (TP-F) for the generation of chiral Chromeno[3,4-*b*]pyrrolidines **9**.



A screw-capped glass vial equipped with a magnetic stirring bar was sequentially charged with **2** (0.2 mmol), **1** (1.2 equiv.), 4Å MS (50 mg), CHCl₃ (0.5 mL) and **IX** (20 mol %) and the resulting mixture was stirred at -5 °C for appropriate time. After the completion of reaction, solvent was removed *in vacuo* and the residue was subjected to flash column chromatography on silica gel to obtain the chiral adducts **9**.

12. Analytical data for all new compounds

(*E*)-Diethyl 2-((5-fluoro-2-hydroxybenzylidene)amino)malonate (**1h**).



Following the TP-A, **1h** was obtained from 5-fluoro-2-hydroxybenzaldehyde (1.57 g, 1.0 equiv.) as a yellow oil (1.276 g, 38.2%).

R_f = 0.325 (EA:Hex = 1:6).

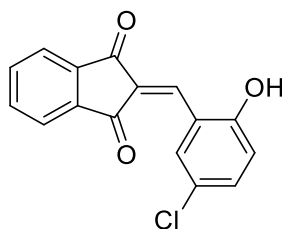
¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 12.47 (s, 1H), 8.43 (s, 1H), 7.09 (td, J = 8.0, 3.0 Hz, 1H), 7.02 (dd, J = 8.0, 3.0 Hz, 1H), 6.94 (dd, J = 8.0, 4.4 Hz, 1H), 4.88 (s, 1H), 4.30 (q, J = 7.1 Hz, 4H), 1.32 (t, J = 7.1 Hz, 6H).

¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 168.7 (d, J = 2.5 Hz), 165.9, 157.2 (d, J = 1.2 Hz), 155.3 (d, J = 237.3 Hz), 120.6 (d, J = 23.3 Hz), 118.5 (d, J = 7.4 Hz), 118.1 (d, J = 7.2 Hz), 117.1 (d, J = 23.3 Hz), 72.5, 62.5, 14.0.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3363, 3275, 2949, 2920, 2362, 2343, 1736, 1643, 1489, 1467, 1375, 1204.

HRMS (ESI) for $C_{14}H_{16}FNO_5$, $[M+H]^+$ (298.1085) found: 298.1092.

2-(5-Chloro-2-hydroxybenzylidene)-1*H*-indene-1,3(2*H*)-dione (2b).



Following the **TP-B**, **2b** was obtained from 2-hydroxy-5-chlorobenzaldehyde (1.76 g, 1.1 equiv.) as a yellow solid (2.41 g, 85%).

R_f = 0.150 (DCM:Hex = 3:1).

mp.: 250.1-251.1 °C.

1H NMR (400 MHz, $(CD_3)_2CO$, 25 °C) δ /ppm: 9.88 (s, 1H), 9.15 (d, J = 2.7 Hz, 1H), 8.38 (s, 1H), 8.09-7.93 (m, 4H), 7.47 (dd, J = 8.8, 2.7 Hz, 1H), 7.09 (d, J = 8.8 Hz, 1H).

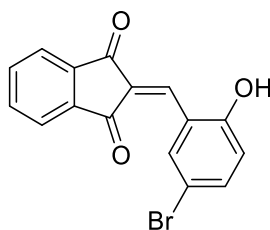
^{13}C NMR (100 MHz, $(CD_3)_2CO$, 25 °C) δ /ppm: 190.2, 189.9, 158.9, 143.4, 141.0, 139.0, 136.6, 136.5, 135.6, 133.3, 129.8, 124.8, 124.0, 123.8, 122.6, 118.4.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3160, 3098, 1722, 1681, 1594, 1489, 821, 734, 610.

HRMS (ESI) for $C_{16}H_8^{35}ClO_3$, $[M-H]^-$ (283.0167) found: 283.0160.

HRMS (ESI) for $C_{16}H_8^{37}ClO_3$, $[M-H]^-$ (285.0138) found: 285.0134.

2-(5-Bromo-2-hydroxybenzylidene)-1*H*-indene-1,3(2*H*)-dione (2c).



Following the TP-B, **2c** was obtained from 2-hydroxy-5-bromobenzaldehyde (2.26 g, 1.1 equiv.) as a yellow solid (2.70 g, 82%).

R_f = 0.125 (DCM:Hex = 3:1).

mp.: 250.2-251.2 °C.

1H NMR (400 MHz, $(CD_3)_2CO$, 25 °C) δ /ppm: 9.91 (s, 1H), 9.28 (d, J = 2.4 Hz, 1H), 8.37 (s, 1H), 8.06-7.92 (m, 4H), 7.59 (dd, J = 8.8, 2.4 Hz, 1H), 7.04 (d, J = 8.8 Hz, 1H).

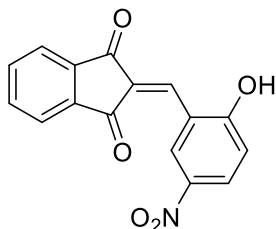
^{13}C NMR (100 MHz, $(CD_3)_2CO$, 25 °C) δ /ppm: 190.2, 189.9, 159.5, 143.4, 141.0, 138.9, 138.4, 136.6, 136.5, 136.3, 129.8, 124.0, 123.8, 123.2, 118.8, 111.8.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3468, 3179, 1722, 1681, 1583, 1484, 812, 734, 636.

HRMS (ESI) for $C_{16}H_{10}^{79}BrO_3$, $[M+H]^+$ (328.9808) found: 328.9812.

HRMS (ESI) for $C_{16}H_{10}^{81}BrO_3$, $[M+H]^+$ (330.9787) found: 330.9797.

2-(2-Hydroxy-5-nitrobenzylidene)-1*H*-indene-1,3(2*H*)-dione (2d).



Following the TP-B, **2d** was obtained from 2-hydroxy-3-methoxybenzaldehyde (1.86 g, 1.1 equiv.) as a green solid (2.24 g, 76%)

R_f = 0.375 (DCM:Hex:EA = 1:5:1).

mp.: 133.4-134.9 °C.

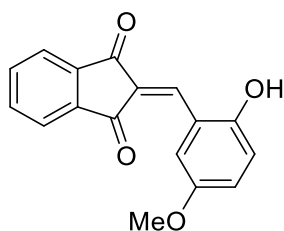
1H NMR (400 MHz, $(CD_3)_2CO$, 25 °C) δ /ppm: 12.42 (s, 1H), 9.91 (d, J = 2.8 Hz, 1H), 8.32 (dd, J = 9.1, 2.8 Hz, 1H), 8.18 (s, 1H), 8.08-7.95 (m, 4H), 7.16 (d, J = 9.2 Hz, 1H).

^{13}C NMR (100 MHz, $(CD_3)_2CO$, 50 °C) δ /ppm: 188.9, 188.5, 164.6, 141.7, 139.5, 139.1, 136.9, 135.8, 135.7, 129.6, 129.4, 128.8, 123.0, 122.9, 119.4, 116.5.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3318, 3059, 2918, 2850, 2362, 2338, 1656, 1603, 1586, 1455, 1317, 1302, 1250, 1230.

HRMS (ESI) for $C_{16}H_8NO_5$, $[M-H]^-$ (294.0408) found: 294.0404.

2-(2-Hydroxy-5-methoxybenzylidene)-1*H*-indene-1,3(2*H*)-dione (2e).



Following the TP-B, **2e** was obtained from 2-hydroxy-5-methoxybenzaldehyde (1.71 g, 1.1 equiv.) as a yellow solid (2.02 g, 72%).

R_f = 0.075 (DCM:Hex = 3:1).

mp.: 220.1-221.1 °C.

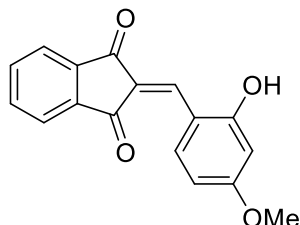
1H NMR (400 MHz, $(CD_3)_2CO$, 25 °C) δ /ppm: 9.27 (s, 1H), 8.86 (d, J = 3.1 Hz, 1H), 8.52 (s, 1H), 8.08-7.92 (m, 4H), 7.10 (dd, J = 8.9, 3.1 Hz, 1H), 7.00 (d, J = 8.9 Hz, 1H), 3.89 (s, 3H).

^{13}C NMR (100 MHz, $(CD_3)_2CO$, 25 °C) δ /ppm: 190.7, 190.2, 155.2, 153.5, 143.4, 141.1, 140.8, 136.3, 136.2, 128.4, 124.9, 123.8, 123.6, 121.6, 117.8, 116.6, 56.1.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3299, 3125, 2959, 1719, 1671, 1581, 1498, 1168, 816, 734.

HRMS (ESI) for C₁₇H₁₁O₄, [M-H]⁻ (279.0663) found: 279.0657.

2-(2-Hydroxy-4-methoxybenzylidene)-1H-indene-1,3(2H)-dione (2f).



Following the TP-B, **2f** was obtained from 2-hydroxy-4-methoxybenzaldehyde (1.71 g, 1.1 equiv.) as a yellow solid (2.13 g, 76%).

R_f = 0.125 (DCM:Hex = 3:1).

mp.: 240.4-241.4 °C.

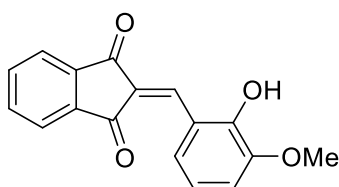
¹H NMR (400 MHz, (CD₃)₂CO, 25 °C) δ /ppm: 9.93 (s, 1H), 9.27 (d, J = 9.0 Hz, 1H), 8.48 (s, 1H), 8.02-7.86 (m, 4H), 6.65 (dd, J = 9.0, 2.4 Hz, 1H), 6.60 (d, J = 2.4 Hz, 1H), 3.90 (s, 3H).

¹³C NMR (100 MHz, (CD₃)₂CO, 25 °C) δ /ppm: 191.1, 190.3, 167.5, 143.1, 140.9, 140.7, 136.9, 135.9, 135.7, 125.3, 123.4, 123.3, 115.5, 107.8, 101.3, 56.0.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3258, 3087, 2826, 1708, 1667, 1594, 1488, 1075, 809, 735.

HRMS (ESI) for C₁₇H₁₃O₄, [M+H]⁺ (281.0808) found: 281.0815.

2-(2-Hydroxy-3-methoxybenzylidene)-1H-indene-1,3(2H)-dione (2g).



Following the TP-B, **2g** was obtained from 2-hydroxy-3-methoxybenzaldehyde (1.71 g, 1.1 equiv.) as a yellow solid (2.19 g, 78%).

R_f = 0.300 (DCM:Hex = 3:1).

mp.: 233.8-234.8 °C.

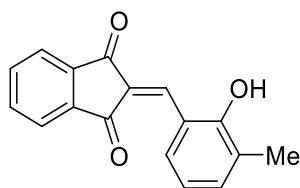
¹H NMR (400 MHz, (CD₃)₂CO, 25 °C) δ /ppm: 8.81 (s, 1H), 8.66 (d, J = 8.1 Hz, 1H), 8.55 (s, 1H), 8.08-7.90 (m, 4H), 7.22 (d, J = 8.1 Hz, 1H), 6.95 (t, J = 8.1 Hz, 1H), 3.92 (s, 3H).

¹³C NMR (100 MHz, (CD₃)₂CO, 25 °C) δ /ppm: 190.6, 189.7, 150.5, 148.4, 143.4, 140.9, 140.4, 136.4, 136.2, 128.9, 125.8, 123.8, 123.7, 121.1, 119.7, 117.2, 56.7.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3374, 3100, 2941, 1717, 1673, 1601, 1222, 832, 747.

HRMS (ESI) for C₁₇H₁₃O₄, [M+H]⁺ (281.0808) found: 281.0815.

2-(2-Hydroxy-3-methylbenzylidene)-1H-indene-1,3(2H)-dione (2h).



Following the TP-B, **2h** was obtained from 2-hydroxy-3-methylbenzaldehyde (1.52 g, 1.1 equiv.) as a yellow solid (1.85 g, 70%).

$R_f = 0.350$ (DCM:Hex = 3:1).

mp.: 211-212 °C.

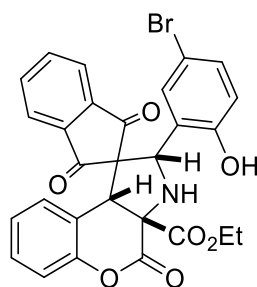
^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 9.80 (s, 1H), 8.05-8.02 (m, 3H), 7.85 (m, 2H), 7.59 (d, $J = 7.9$ Hz, 1H), 7.39 (d, $J = 7.2$ Hz, 1H), 6.93 (t, $J = 7.6$ Hz, 1H), 2.36 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 193.9, 189.4, 158.0, 144.7, 142.1, 139.9, 137.2, 136.1, 135.9, 135.3, 129.2, 125.4, 123.7, 123.5, 122.3, 120.5, 16.7.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3354, 1711, 1672, 1582, 1466, 1150, 992, 736.

HRMS (ESI) for $\text{C}_{17}\text{H}_{13}\text{O}_3$, $[\text{M}+\text{H}]^+$ (265.0859) found: 265.0866.

Ethyl-2-(5-bromo-2-hydroxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetrahydro-4H-spiro [chromeno[3,4-*b*]pyrrole-1,2'-indene]-3a(9bH)-carboxylate (3aa).



Following the TP-C, **3aa** was obtained from **1a** (71.6 mg, 0.2 mmol) and **2a** (50.0 mg, 1.0 equiv.) as a pale yellow solid (77.6 mg, 69%).

$R_f = 0.200$ (DCM:Hex = 3:1).

mp.: 118.5-119.5 °C.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 10.71 (s, 1H), 10.26 (s, 1H'), 7.99 (d, $J = 7.7$ Hz, 1H), 7.81-7.73 (m, 3H'+1H), 7.63 (t, $J = 7.5$ Hz, 1H), 7.51 (d, $J = 7.7$ Hz, 1H), 7.25-7.16 (m, 2H'+1H), 7.14 (d, $J = 8.2$ Hz, 1H'), 7.08 (d, $J = 8.2$ Hz, 1H), 6.94 (dd, $J = 8.8, 2.0$ Hz, 1H), 6.88-6.80 (m, 1H'+2H), 6.78 (d, $J = 8.8$ Hz, 1H'), 6.65 (d, $J = 7.5$ Hz, 1H'), 6.57 (d, $J = 8.8$ Hz, 1H), 6.54 (d, $J = 1.4$ Hz, 1H), 6.39 (s, 1H'), 5.08 (d, $J = 6.9$ Hz, 1H), 4.94

(d, $J = 6.2$ Hz, 1H'), 4.66 (s, 1H'), 4.36 (s, 1H) , 4.24 (q, $J = 7.2$ Hz, 4H'+3H), 1.16 (t, $J = 7.0$ Hz, 3H'), 1.10 (t, $J = 7.0$ Hz, 3H).

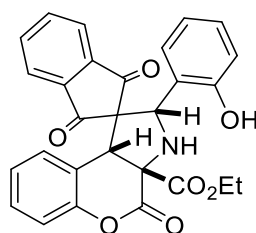
^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 197.7, 198.7 (C'), 196.9, 196.0 (C'), 168.7, 167.1 (C'), 164.6, 165.9 (C'), 156.6, 156.9 (C'), 151.5 (C'), 150.9, 143.1 (C'), 142.5, 141.3 (C'), 142.3, 136.6, 136.2 (C'), 136.1, 132.8 (C'), 132.5, 130.1, 130.3 (C'), 130.0 (C'), 129.8, 127.8 (C'), 127.2, 124.8 (C'), 124.5, 123.8 (C'), 123.7 (C'), 123.4, 123.2, 121.9 (C'), 120.3, 119.9 (C'), 118.3, 117.4 (C'), 117.6, 116.4 (C'), 115.7, 110.7 (C'), 110.1, 71.0, 69.7 (C'), 68.6, 68.2 (C'), 67.7, 67.5 (C'), 64.1, 63.6 (C'), 50.1 (C'), 49.3, 13.7 (C'), 13.6.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3317, 2922, 1771, 1742, 1708, 1591, 1178, 1108, 761.

HRMS (ESI) for $\text{C}_{28}\text{H}_{21}^{79}\text{BrNO}_7$, $[\text{M}+\text{H}]^+$ (562.0496) found: 562.0504.

HRMS (ESI) for $\text{C}_{28}\text{H}_{21}^{81}\text{BrNO}_7$, $[\text{M}+\text{H}]^+$ (564.0475) found: 564.0488.

Ethyl-2-(2-hydroxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetrahydro-4*H*-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3a(9*bH*)-carboxylate (3*ba*).



Following the TP-C, **3ba** was obtained from **1b** (55.9 mg, 0.2 mmol) and **2a** (50.0 mg, 1.0 equiv.) as a pale yellow solid (60.0 mg, 62%).

$R_f = 0.325$ (DCM:Hex:EA = 1:3:1).

mp.: 110.0-111.0 °C.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 10.55 (s, 1H), 10.23 (s, 1H'), 7.94 (d, $J = 7.6$ Hz, 1H), 7.79-7.73 (m, 3H'), 7.70 (t, $J = 7.6$ Hz, 1H'+1H), 7.59 (t, $J = 7.6$ Hz, 1H), 7.47 (d, $J = 7.6$ Hz, 1H), 7.26-7.14 (m, 2H'+1H), 7.11 (d, $J = 8.3$ Hz, 1H'), 7.06 (d, $J = 8.2$ Hz, 1H), 6.91-6.79 (m, 2H'+3H), 6.69-6.62 (m, 1H'+1H), 6.48 (d, $J = 7.5$ Hz, 1H), 6.44 (t, $J = 7.5$ Hz, 1H'), 6.32 (t, $J = 7.5$ Hz, 1H), 6.27 (d, $J = 7.5$ Hz, 1H'), 5.18 (d, $J = 5.8$ Hz, 1H), 5.01 (d, $J = 5.9$ Hz, 1H'), 4.71 (s, 1H'), 4.38 (s, 1H), 4.23 (q, $J = 7.1$ Hz, 3H'+3H), 1.16 (t, $J = 7.1$ Hz, 3H'), 1.10 (t, $J = 7.1$ Hz, 3H).

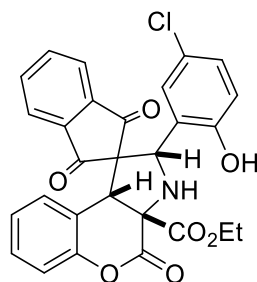
^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 199.0 (C'), 198.1, 197.2, 196.2 (C'), 168.9, 167.1 (C'), 166.1 (C'), 164.8, 157.6 (C'), 157.2, 151.5 (C'), 150.8, 143.2 (C'), 142.53, 142.47, 141.5 (C'), 136.4, 135.9 (C'), 135.8, 130.2 (C'), 130.0 (C'), 129.9, 129.6, 127.7 (C'), 127.5 (C'), 127.4, 127.1, 124.7 (C'), 124.4, 123.6 (C'), 123.5 (C'), 123.3, 123.0, 119.5 (C'),

118.9 (C'), 118.6, 118.3, 118.0 (C'), 117.5, 117.3 (C'), 116.7 (C'), 116.3, 115.9, 71.0, 69.8 (C'), 69.3, 68.5 (C'), 68.2 (C'), 67.6, 63.9, 63.5 (C'), 49.9 (C'), 49.4, 13.7 (C'), 13.6.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3318, 3068, 2985, 1774, 1742, 1708, 1592, 1492, 1179, 1126.

HRMS (ESI) for C₂₈H₂₂NO₇, [M+H]⁺ (484.1391) found: 484.1401.

Ethyl-2-(5-chloro-2-hydroxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetrahydro-2H-spiro [chromeno[3,4-*b*]pyrrole-1,2'-indene]-3a(9*bH*)-carboxylate (3ca**).**



Following the TP-C, **3ca** was obtained from **1c** (62.7 mg, 0.2 mmol) and **2a** (50.0 mg, 1.0 equiv.) as a pale yellow solid (70.5 mg, 70%).

R_f = 0.200 (DCM:Hex = 3:1).

mp.: 219.1-220.1 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 10.66 (s, 1H), 10.14 (s, 1H'), 7.99 (d, *J* = 7.7 Hz, 1H), 7.84-7.73 (m, 4H'+1H), 7.63 (td, *J* = 8.0, 0.9 Hz, 1H), 7.51 (d, *J* = 7.7 Hz, 1H), 7.26-7.16 (m, 1H'+1H), 7.13 (dd, *J* = 8.2, 0.9 Hz, 1H'), 7.07 (d, *J* = 8.0 Hz, 1H), 7.04 (d, *J* = 2.6 Hz, 1H'), 6.88-6.78 (m, 2H'+3H), 6.65 (dd, *J* = 7.5, 1.7 Hz, 1H'), 6.60 (d, *J* = 8.8 Hz, 1H), 6.43 (d, *J* = 2.6 Hz, 1H), 6.32 (d, *J* = 1.7 Hz, 1H'), 5.10 (d, *J* = 6.5 Hz, 1H), 4.96 (d, *J* = 6.2 Hz, 1H'), 4.65 (s, 1H'), 4.36 (s, 1H), 4.29 (d, *J* = 6.8 Hz, 1H), 4.23 (q, *J* = 7.1 Hz, 3H'+2H), 1.16 (t, *J* = 7.1 Hz, 3H'), 1.10 (t, *J* = 7.2 Hz, 3H).

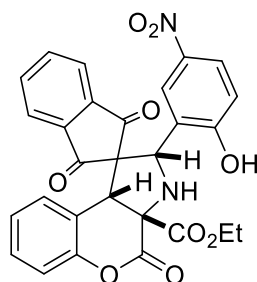
¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 198.9 (C'), 197.8, 196.9, 196.1 (C'), 168.7, 167.2 (C'), 166.0 (C'), 164.7, 156.3 (C'), 156.0, 151.5 (C'), 150.9, 143.1 (C'), 142.5, 142.3, 141.4 (C'), 136.7, 136.23 (C'), 136.17, 130.4 (C'), 130.1, 129.9 (C'), 129.6, 127.8 (C'), 127.2, 127.1 (C'), 126.9, 124.8 (C'), 124.5, 123.8 (C'), 123.7 (C'), 123.5, 123.2, 123.1, 121.6 (C'), 119.9, 119.3 (C'), 117.8, 117.6, 117.4 (C'), 116.4 (C'), 115.7, 70.9, 69.7 (C'), 68.6, 68.1 (C'), 67.7, 67.4 (C'), 64.1, 63.7 (C'), 50.2 (C'), 49.5, 13.72 (C'), 13.68.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3324, 2926, 1770, 1742, 1708, 1592, 1488, 1177, 1108, 762, 703.

HRMS (ESI) for C₂₈H₂₁⁷⁹ClNO₇, [M+H]⁺ (518.1001) found: 518.1009.

HRMS (ESI) for C₂₈H₂₁⁸¹ClNO₇, [M+H]⁺ (520.0972) found: 520.0994.

Ethyl-2-(2-hydroxy-5-nitrophenyl)-1',3',4-trioxo-1',2,3,3'-tetrahydro-4*H*-spiro [chromeno[3,4-*b*]pyrrole-1,2'-indene]-3a(9*bH*)-carboxylate (3da).



Following the TP-C, **3da** was obtained from **1d** (61.9 mg, 0.2 mmol) and **2a** (50.0 mg, 1.0 equiv.) as a pale yellow solid (61.3 mg, 58%).

R_f = 0.3.75 (DCM:Hex:EA = 1:3:1).

mp.: 227.2-228.1 °C.

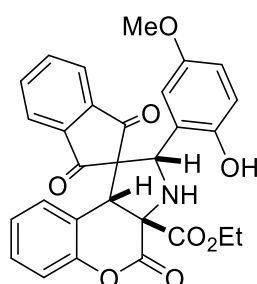
^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 11.93 (s, 1H), 8.01 (d, J = 7.6 Hz, 1H'+1H), 7.85-7.70 (m, 5H'+2H), 7.62 (t, J = 7.4 Hz, 1H), 7.48 (d, J = 7.6 Hz, 1H), 7.40 (s, 1H), 7.25-7.13 (m, 1H'+1H), 7.09 (d, J = 8.2 Hz, 1H), 6.99 (d, J = 9.2 Hz, 1H'), 6.94-6.79 (m, 2H'+2H), 6.67 (d, J = 9.1 Hz, 1H), 6.62 (d, J = 6.9 Hz, 1H'), 5.26 (s, 1H), 5.12 (s, 1H'), 4.61 (s, 1H'), 4.39 (s, 1H), 4.25 (q, J = 7.1 Hz, 2H'+2H), 3.45 (s, 1H), 1.10 (t, J = 7.1 Hz, 3H'+3H).

^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 198.4 (C'), 197.2, 197.0, 168.3, 167.1 (C'), 164.4, 163.8, 151.4 (C'), 150.8, 142.7 (C'), 142.4, 142.0, 141.4 (C'), 139.8 (C'), 139.2, 136.9, 136.7 (C'), 136.6, 130.6 (C'), 130.3, 127.8 (C'), 127.2, 126.2 (C'), 125.6, 124.9 (C'), 124.7, 124.1 (C'), 123.7, 123.6, 123.5, 119.1, 118.6 (C'), 117.7, 117.6 (C'), 116.9, 115.8 (C'), 115.2, 70.9, 69.7 (C'), 68.00, 67.99, 67.0 (C'), 64.4, 63.9 (C'), 50.8 (C'), 49.7, 13.7.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3319, 2924, 1786, 1743, 1707, 1592, 1485, 1252, 1174.

HRMS (ESI) for $\text{C}_{28}\text{H}_{21}\text{N}_2\text{O}_9$, $[\text{M}+\text{H}]^+$ (529.1242) found: 529.1249.

Ethyl-2-(2-hydroxy-5-methoxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetrahydro-4*H*-spiro [chromeno[3,4-*b*]pyrrole-1,2'-indene]-3a(9*bH*)-carboxylate (3ea).



Following the TP-C, **3ea** was obtained from **1e** (61.9 mg, 0.2 mmol) and **2a** (50.0 mg, 1.0 equiv.) as a pale yellow solid (59.6 mg, 59%).

R_f = 0.125 (DCM:Hex = 3:1).

mp.: 126.5-127.3 °C.

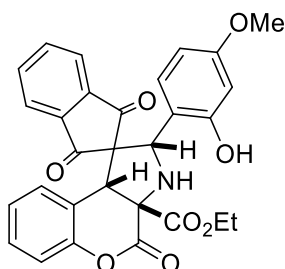
¹H NMR (400 MHz, CDCl₃, 25 °C) (mixture of rotamers) δ /ppm: 10.06 (s, 1H), 9.58 (s, 1H'), 7.95 (d, J = 7.7 Hz, 1H), 7.78-7.74 (m, 4H'), 7.71 (t, J = 7.5 Hz, 1H), 7.61 (t, J = 7.3 Hz, 1H), 7.50 (d, J = 7.7 Hz, 1H), 7.26-7.10 (m, 2H'+1H), 7.07 (d, J = 8.2 Hz, 1H), 6.87-6.77 (m, 2H'+2H), 6.67 (dd, J = 8.5, 2.4 Hz, 2H'), 6.59 (d, J = 8.9 Hz, 1H), 6.46 (dd, J = 8.9, 3.0 Hz, 1H), 6.01 (d, J = 3.0 Hz, 1H), 5.89 (d, J = 2.4 Hz, 1H'), 5.12 (d, J = 4.8 Hz, 1H), 4.95 (d, J = 5.9 Hz, 1H'), 4.70 (s, 1H'), 4.38 (s, 1H), 4.23 (q, J = 7.1 Hz, 3H'+3H), 3.42 (s, 3H), 3.37 (s, 3H'), 1.16 (t, J = 7.1 Hz, 3H'), 1.10 (t, J = 7.1 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 199.1 (C'), 198.0, 196.9, 196.0 (C'), 168.8, 167.2 (C'), 166.2 (C'), 164.8, 155.7 (C'), 152.0 (C'), 151.7, 151.4 (C'), 150.8, 150.7, 144.6 (C'), 143.2 (C'), 142.5, 142.4, 141.2 (C'), 136.4, 135.8, 130.1 (C'), 129.8, 127.7 (C'), 127.1, 124.6 (C'), 124.4, 123.5 (C'), 123.4 (C'), 123.3, 122.9, 120.5 (C'), 119.0, 118.3 (C'), 118.2 (C'), 117.4, 117.2 (C'), 116.6 (C'), 116.5, 116.2, 115.8, 115.7 (C'), 115.3 (C'), 112.3 (C'), 111.6, 70.8, 69.7 (C'), 69.0, 68.1 (C'), 67.6, 63.8, 63.3 (C'), 55.5, 55.4 (C'), 49.7 (C'), 49.3, 13.60 (C'), 13.56.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3317, 2929, 2855, 1774, 1742, 1708, 1591, 1492, 1252, 1161.

HRMS (ESI) for C₂₉H₂₄NO₈, [M+H]⁺ (514.1496) found: 514.1503.

Ethyl-2-(2-hydroxy-4-methoxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetrahydro-4H-spiro [chromeno[3,4-*b*]pyrrole-1,2'-indene]-3a(9b*H*)-carboxylate (3fa**).**



Following the TP-C, **3fa** was obtained from **1f** (76.5 mg, 0.2 mmol) and **2b** (50.0 mg, 1.0 equiv.) as a pale yellow solid (72.9 mg, 71%).

R_f = 0.150 (DCM:Hex = 3:1).

mp.: 123.9-124.1 °C.

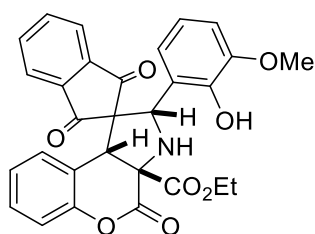
¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 10.63 (s, 1H), 10.44 (s, 1H'),

7.93 (d, $J = 7.6$ Hz, 1H), 7.78-7.66 (m, 3H'+1H), 7.61 (t, $J = 7.6$ Hz, 1H), 7.53 (d, $J = 7.6$ Hz, 1H), 7.23 (d, $J = 8.1$ Hz, 1H'), 7.21-7.12 (m, 2H'+1H), 7.07 (d, $J = 8.2$ Hz, 1H), 6.89-6.77 (m, 1H'+2H), 6.67 (d, $J = 7.4$ Hz, 1H'), 6.44(d, $J = 1.9$ Hz, 1H'), 6.37 (d, $J = 8.6$ Hz, 1H), 6.21 (d, $J = 1.9$ Hz, 1H), 6.09 (d, $J = 8.3$ Hz, 1H'), 5.98 (dd, $J = 8.3, 1.9$ Hz, 1H'), 5.91 (dd, $J = 8.3, 1.9$ Hz, 1H), 5.14 (d, $J = 6.3$ Hz, 1H), 4.96 (d, $J = 5.8$ Hz, 1H'), 4.72 (s, 1H'), 4.35 (s, 1H) , 4.30-4.15 (m, 3H'+3H), 3.71 (s, 3H'), 3.58 (s, 3H), 1.16 (t, $J = 7.1$ Hz, 3H'), 1.10 (t, $J = 7.1$ Hz, 3H).
 ^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 199.1 (C'), 198.3, 197.5, 196.3 (C'), 169.0, 167.1 (C'), 166.2 (C'), 164.9, 161.2 (C'), 160.7, 159.2 (C'), 158.6, 151.6 (C'), 150.9, 143.3 (C'), 142.61, 142.58, 141.6 (C'), 136.44, 136.37 (C'), 135.9 (C'), 135.8, 130.2 (C'), 129.9, 128.35 (C'), 128.27, 127.8 (C'), 127.1, 124.8 (C'), 124.4, 123.7 (C'), 123.6 (C'), 123.4, 123.1, 117.6, 117.3 (C'), 116.9 (C'), 116.0, 111.4 (C'), 108.5, 106.0, 105.8 (C'), 102.6 (C'), 102.5, 71.0, 69.7 (C'), 69.2, 68.7 (C'), 68.3 (C'), 67.5, 64.0, 63.5 (C'), 55.1 (C'), 55.0, 49.7 (C'), 49.5, 13.73 (C'), 13.69.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3324, 2925, 2854, 1775, 1742, 1708, 1591, 1501, 1251, 1180.

HRMS (ESI) for $\text{C}_{29}\text{H}_{24}\text{NO}_8$, $[\text{M}+\text{H}]^+$ (514.1496) found: 514.1506.

Ethyl-2-(2-hydroxy-3-methoxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetrahydro-4H-spiro [chromeno[3,4-*b*]pyrrole-1,2'-indene]-3a(9b*H*)-carboxylate (3ga).



Following the TP-C, **3ga** was obtained from **1g** (76.5 mg, 0.2 mmol) and **2b** (50.0 mg, 1.0 equiv.) as a pale yellow solid (81.1 mg, 79%).

$R_f = 0.138$ (DCM:Hex:EA = 1:3:1).

mp.: 142.5-143.5 °C.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 7.92 (d, $J = 7.6$ Hz, 1H), 7.79-7.69 (m, 1H+2H'), 7.67 (t, $J = 7.5$ Hz, 1H), 7.54 (t, $J = 7.5$ Hz, 1H), 7.42 (d, $J = 7.6$ Hz, 1H), 7.22-7.11 (m, 4H'), 7.08 (t, $J = 7.7$ Hz, 1H+1H'), 6.89-6.74 (m, 3H'+2H), 6.68-6.62 (m, 1H'+1H), 6.55-6.47 (m, 2H), 5.35 (s, 1H), 5.14 (s, 1H'), 4.58 (s, 1H'), 4.54 (s, 1H) , 4.23 (q, $J = 7.1$ Hz, 2H'+2H), 3.80 (s, 3H'), 3.67 (s, 3H), 1.16 (t, $J = 7.1$ Hz, 3H'), 1.13 (t, $J = 7.2$ Hz, 3H).

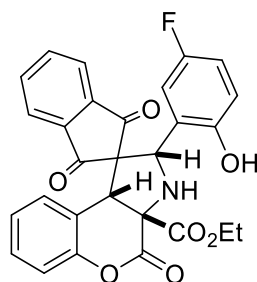
^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 199.8 (C'), 197.7, 196.8 (C'), 197.2, 169.4, 168.0 (C'), 166.8 (C'), 165.6, 151.4 (C'), 150.9, 147.0, 146.5 (C'), 144.9, 143.4

(C'), 143.2 (C'), 142.7, 141.2 (C'), 142.1, 135.7, 135.5 (C'), 135.4, 129.8 (C'), 129.6, 127.8 (C'), 127.4, 124.4 (C'), 124.3, 123.9 (C'), 123.3 (C'), 123.0 (C'), 122.9, 122.8, 120.1 (C'), 119.6, 119.3 (C'), 119.0, 118.8, 117.4, 117.2 (C'), 116.9 (C'), 116.4, 111.0, 110.5 (C'), 70.9, 69.8 (C'), 67.9, 66.0, 63.6 (C'), 63.5, 63.0 (C'), 56.0, 55.9 (C'), 50.1 (C'), 49.8, 13.8 (C'), 13.7.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3324, 2925, 2854, 1775, 1742, 1708, 1591, 1501, 1251, 1180.

HRMS (ESI) for C₂₉H₂₄NO₈, [M+H]⁺ (514.1496) found: 514.1503.

Ethyl-2-(5-fluoro-2-hydroxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetrahydro-4*H*-spiro [chromeno[3,4-*b*]pyrrole-1,2'-indene]-3a(9*bH*)-carboxylate (3*ha*).



Following the TP-C, **3ha** was obtained from **1h** (59.5 mg, 0.2 mmol) and **2b** (50.0 mg, 1.0 equiv.) as a pale yellow solid (46.1 mg, 46%).

R_f = 0.350 (DCM:Hex:EA = 1:5:1).

mp.: 164.8-165.3 °C.

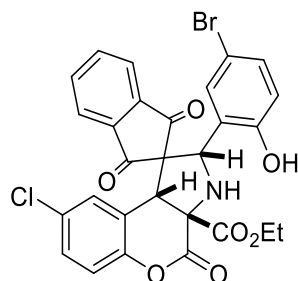
¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of 3 rotamers) 7.98 (d, J = 7.6 Hz, 1H''+1H), 7.84-7.71 (m, 4H'+1H), 7.63 (t, J = 7.4 Hz, 1H''+1H), 7.52 (d, J = 7.6 Hz, 1H''+1H), 7.26-7.16 (m, 1H+1H'+1H''), 7.13 (d, J = 8.2 Hz, 1H'), 7.07 (d, J = 8.2 Hz, 1H+1H''), 6.93-6.77 (m, 2H+2H'+2H''), 6.68-6.51 (m, 2H+2H'+2H''), 6.21 (dd, J = 2.5, 8.4 Hz, 1H+1H''), 6.13 (d, J = 8.3 Hz, 1H'), 5.11 (s, 1H), 5.08 (s, 1H''), 4.96 (s, 1H'), 4.66 (s, 1H'), 4.36 (s, 1H), 4.32 (s, 1H''), 4.23 (q, J = 7.1 Hz, 2H+2H'+2H''), 1.19-1.02 (m, 3H''+3H'+3H).

¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of 3 rotamers) 198.9, 197.8, 197.4, 196.8, 196.6, 196.1, 168.8, 168.6, 167.2, 166.0, 164.7, 164.4, 157.4, 156.8, 156.3, 154.4, 154.0, 153.5 (d, J = 1.6 Hz), 153.3 (d, J = 1.6 Hz), 153.2 (d, J = 2.4 Hz), 151.5, 150.9, 143.1, 142.5, 142.4, 142.4, 142.3, 141.3, 136.7, 136.5, 136.3, 136.1, 136.1, 130.3, 130.0, 127.8, 127.2, 124.7, 124.5, 123.7 (d, J = 14.6 Hz), 123.3 (d, J = 21.5 Hz), 119.4 (d, J = 7.6 Hz), 119.1 (d, J = 8.5 Hz), 118.8 (d, J = 7.7 Hz), 117.6, 117.4, 117.0, 117.0, 116.6, 116.4, 116.4, 115.7, 113.8 (d, J = 23.7 Hz), 113.3 (d, J = 23.5 Hz), 77.3, 77.0, 76.7, 70.8, 69.7, 68.8, 68.6, 68.0, 67.7, 67.4, 67.2, 64.2, 64.0, 63.6, 50.3, 49.6, 49.1, 13.7, 13.6. (major and minor rotamers could not be identified)

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3318, 3072.6, 2986, 1774, 1742, 1708, 1591, 1497, 1176.

HRMS (ESI) for $C_{28}H_{21}FNO_7$, $[M+H]^+$ (502.1297) found: 502.1302.

Ethyl-2-(5-bromo-2-hydroxyphenyl)-8-chloro-1',3',4-trioxo-1',2,3,3'-tetrahydro-4H-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3a(9b*H*)-carboxylate (3ab).



Following the TP-C, **3ab** was obtained from **1a** (71.6 mg, 0.2 mmol) and **2b** (57.0 mg, 1.0 equiv.) as a white solid (89.5 mg, 75%).

R_f = 0.438 (Hex:DCM:EA = 3:1:1).

mp.: 277.1-278.1 °C.

1H NMR (400 MHz, $CDCl_3$, 25 °C) δ /ppm: (mixture of rotamers) 10.59 (s, 1H), 9.97 (s, 1H'), 8.02 (d, J = 7.6 Hz, 1H), 7.86-7.76 (m, 1H+4H'), 7.67 (t, J = 7.6 Hz, 1H), 7.52 (d, J = 7.6 Hz, 1H), 7.17 (dd, J = 8.8, 2.4 Hz, 1H+2H'), 7.10 (d, J = 8.9 Hz, 1H'), 7.04 (d, J = 8.8 Hz, 1H), 6.94 (dd, J = 8.8, 2.0 Hz, 1H), 6.83 (d, J = 2.1 Hz, 1H), 6.76 (d, J = 8.9 Hz, 1H'), 6.67 (d, J = 2.3 Hz, 1H'), 6.58-6.49 (m, 2H), 6.43 (s, 1H'), 5.05 (d, J = 7.0 Hz, 1H), 4.88 (d, J = 5.9 Hz, 1H'), 4.62 (s, 1H'), 4.33-4.19 (m, 4H+3H'), 1.20 (t, J = 7.1 Hz, 3H'), 1.14 (t, J = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ /ppm: (mixture of rotamers) 198.4 (C'), 197.2, 196.7, 195.6 (C'), 168.3, 166.8 (C'), 165.5 (C'), 164.2, 156.5 (C'), 156.4, 150.1 (C'), 149.5, 143.1 (C'), 142.4, 142.1, 141.2 (C'), 136.8, 136.4, 132.8 (C'), 132.5, 130.4 (C'), 130.3, 130.2 (C'), 129.9, 129.6, 127.5 (C'), 127.0, 123.9 (C'), 123.8 (C'), 123.5, 123.4, 121.8 (C'), 120.3, 119.7 (C'), 119.0, 118.7 (C'), 118.3 (C'), 118.1, 117.5, 110.8 (C'), 110.2, 71.0, 69.4 (C'), 68.7, 68.2 (C'), 67.6 (C'), 67.4, 64.3, 63.8 (C'), 49.0 (C'), 48.5, 13.74 (C'), 13.69.

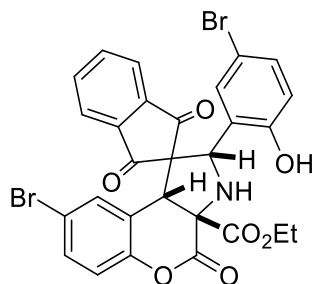
IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3316, 2985, 1778, 1742, 1707, 1591, 1484, 1253, 1174.

HRMS (ESI) for $C_{28}H_{20}^{79}Br^{35}ClNO_7$, $[M+H]^+$ (596.0106) found: 596.0114.

HRMS (ESI) for $C_{28}H_{20}^{81}Br^{35}ClNO_7$, $[M+H]^+$ (598.0086) found: 598.0095.

HRMS (ESI) for $C_{28}H_{20}^{81}Br^{37}ClNO_7$, $[M+H]^+$ (600.0057) found: 600.0122.

Ethyl-8-bromo-2-(5-bromo-2-hydroxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetrahydro-4H-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3a(9b*H*)-carboxylate (3ac).



Following the TP-C, **3ac** was obtained from **1a** (71.6 mg, 0.2 mmol) and **2c** (65.9 mg, 1.0 equiv.) as a white solid (92.3 mg, 72%).

R_f = 0.450 (Hex:DCM:EA = 3:1:1).

mp.: 285.7-286.7 °C.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 10.59 (s, 1H), 10.02 (s, 1H'), 8.02 (d, J = 7.5 Hz, 1H), 7.88-7.74 (m, 1H+4H'), 7.68 (t, J = 7.5 Hz, 1H), 7.53 (d, J = 7.5 Hz, 1H), 7.36 (dd, J = 8.6, 2.0 Hz, 1H'), 7.32 (dd, J = 8.6, 2.1 Hz, 1H), 7.18 (dd, J = 8.6, 2.0 Hz, 1H'), 7.04 (d, J = 8.8 Hz, 1H'), 7.00-6.96 (m, 2H), 6.94 (dd, J = 8.9, 2.2 Hz, 1H), 6.82 (d, J = 2.2 Hz, 1H'), 6.76 (d, J = 8.8 Hz, 1H'), 6.55 (d, J = 8.9 Hz, 1H), 6.52 (d, J = 2.2 Hz, 1H), 6.38 (s, 1H'), 5.05 (d, J = 6.9 Hz, 1H), 4.88 (d, J = 6.2 Hz, 1H'), 4.62 (s, 1H'), 4.34-4.18 (m, 4H+3H'), 1.21 (t, J = 7.1 Hz, 3H'), 1.14 (t, J = 7.0 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 198.4 (C'), 197.2, 196.7, 195.5(C'), 168.3, 166.8 (C'), 165.4 (C'), 164.1, 156.6 (C'), 156.4, 150.7 (C'), 150.1, 143.1 (C'), 142.5, 142.2, 141.2 (C'), 136.9 (C'), 136.8, 136.4, 133.4 (C'), 133.2, 132.9 (C'), 132.6, 130.4 (C'), 130.1 (C'), 129.90, 129.87, 124.0 (C'), 123.8 (C'), 123.5, 123.4, 121.5 (C'), 120.3, 119.8 (C'), 119.4, 119.1 (C'), 118.8(C'), 118.05, 117.98, 117.3 (C'), 116.9, 110.7 (C'), 110.2, 71.1, 69.5 (C'), 68.8, 68.2 (C'), 67.7 (C'), 67.4, 64.3, 63.8 (C'), 48.9(C'), 48.5, 13.75 (C'), 13.70.

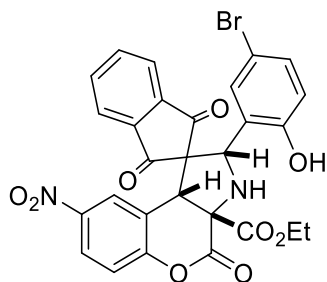
IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3315, 2981, 1777, 1742, 1707, 1591, 1482, 1252, 1174.

HRMS (ESI) for $\text{C}_{28}\text{H}_{20}^{79}\text{Br}_2\text{NO}_7$, $[\text{M}+\text{H}]^+$ (639.9601) found: 639.9609.

HRMS (ESI) for $\text{C}_{28}\text{H}_{20}^{79}\text{Br}^{81}\text{BrNO}_7$, $[\text{M}+\text{H}]^+$ (641.9581) found: 641.9592.

HRMS (ESI) for $\text{C}_{28}\text{H}_{20}^{81}\text{Br}_2\text{NO}_7$, $[\text{M}+\text{H}]^+$ (643.9561) found: 643.9577.

Ethyl-2-(5-bromo-2-hydroxyphenyl)-8-nitro-1',3',4-trioxo-1',2,3,3'-tetrahydro-4H-spiro [chromeno[3,4-*b*]pyrrole-1,2'-indene]-3a(9*bH*)-carboxylate (3ad**).**



Following the TP-C, **3ad** was obtained from **1a** (71.6 mg, 0.2 mmol) and **2d** (65.9 mg, 1.0 equiv.) as a white solid (87.5 mg, 72%).

R_f = 0.225 (DCM:Hex:EA = 1:3:1).

mp.: 230.9-231.4 °C.

^1H NMR (400 MHz, $(\text{CD}_3)_2\text{CO}$, 25 °C) δ /ppm: (mixture of rotamers) 9.75 (s, 1H), 9.18 (s, 1H'), 8.18-8.08 (m, 1H+2H'), 8.00 (d, J = 7.7 Hz, 1H), 7.93-7.79 (m, 2H+2H'), 7.75 (d, J = 2.7 Hz, 1H), 7.73-7.67 (m, 1H+1H'), 7.58 (d, J = 2.6 Hz, 1H'), 7.53-7.47 (m, 1H), 7.42-7.29 (m, 3H'+1H), 7.20 (dd, J = 8.5, 2.5 Hz, 1H'), 6.97 (dd, J = 8.6, 2.5 Hz, 1H), 6.57 (d, J = 8.5 Hz, 1H'), 6.36 (d, J = 8.6 Hz, 1H), 5.40-5.33 (m, 1H), 5.15-5.07 (m, 1H'), 4.84 (s, 1H), 4.60 (s, 1H'), 4.56-4.45 (m, 1H), 4.32-4.16 (m, 2H+2H'), 1.18 (t, J = 7.1 Hz, 3H'), 1.12 (t, J = 7.1 Hz, 3H).

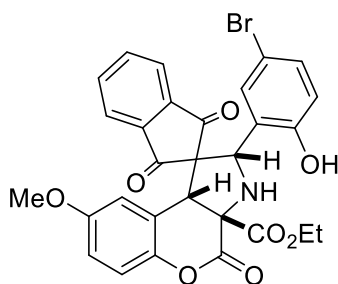
^{13}C NMR (100 MHz, $(\text{CD}_3)_2\text{CO}$, 25 °C) δ /ppm: (mixture of rotamers) 200.8, 198.4, 197.4, 197.0, 169.7, 169.7, 168.9, 166.3, 165.6, 156.9, 156.5, 155.4, 154.1, 144.5, 144.1, 142.2, 141.7, 137.4, 137.3, 137.2, 136.9, 132.3, 132.2, 131.9, 131.7, 129.8, 126.5, 126.3, 125.1, 124.8, 124.3, 124.0, 123.8, 123.7, 123.2, 119.7, 119.1, 117.7, 117.0, 111.9, 111.3, 71.4, 70.3, 70.2, 68.5, 68.5, 68.3, 64.8, 64.7, 64.3, 63.9, 62.8, 62.7, 50.6, 50.1, 14.1, 13.9. (major and minor rotamers could not be identified)

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3319, 3086, 2926, 2362, 2344, 1786, 1742, 1709, 1592, 1484, 1252, 1170.

HRMS (ESI) for $\text{C}_{28}\text{H}_{20}^{79}\text{BrN}_2\text{O}_9$, $[\text{M}+\text{H}]^+$ (607.0347) found: 607.0352.

HRMS (ESI) for $\text{C}_{28}\text{H}_{20}^{81}\text{BrN}_2\text{O}_9$, $[\text{M}+\text{H}]^+$ (609.0326) found: 609.0335.

Ethyl-2-(5-bromo-2-hydroxyphenyl)-8-methoxy-1',3',4-trioxo-1',2,3,3'-tetrahydro-4H-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3a(9*bH*)-carboxylate (3ae**).**



Following the TP-C, **3ae** was obtained from **1a** (71.6 mg, 0.2 mmol) and **2e** (56.1 mg, 1.0 equiv.) as a pale yellow solid (85.3 mg, 72%).

R_f = 0.150 (DCM:Hex = 3:1).

mp.: 269.3-270.3 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 10.73 (s, 1H), 10.24 (s, 1H'), 8.00 (d, J = 7.7 Hz, 1H), 7.77 (t, J = 7.8 Hz, 4H'+1H), 7.65 (t, J = 7.5 Hz, 1H), 7.52 (d, J = 7.7 Hz, 1H), 7.18 (dd, J = 8.6, 2.4 Hz, 1H'), 7.07 (d, J = 9.1 Hz, 1H'), 7.01 (d, J = 9.1 Hz, 1H), 7.00 (d, J = 9.1 Hz, 1H), 6.94 (dd, J = 8.8, 2.3 Hz, 1H), 6.80-6.74 (m, 2H'), 6.72 (dd, J = 9.0, 2.9 Hz, 1H), 6.55 (d, J = 8.8 Hz, 1H) 6.54 (d, J = 2.3 Hz, 1H), 6.40 (s, 1H'), 6.29 (d, J = 2.9 Hz, 1H), 6.13 (d, J = 2.9 Hz, 1H'), 5.07 (d, J = 6.9 Hz, 1H), 4.92 (d, J = 6.3 Hz, 1H'), 4.60 (s, 1H'), 4.31 (s, 1H), 4.30-4.16 (m, 3H'+2H), 3.55 (s, 3H), 3.50 (s, 3H'), 1.19 (t, J = 7.1 Hz, 3H'), 1.13 (t, J = 7.1 Hz, 3H).

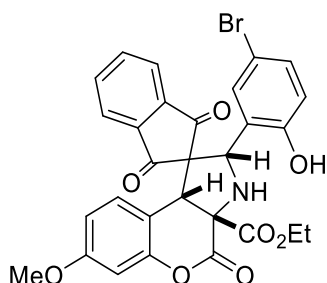
¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 198.5 (C'), 197.7, 196.7, 196.0 (C'), 168.8, 167.2 (C'), 166.0 (C'), 164.8, 156.8 (C'), 156.6, 156.1 (C'), 155.9, 145.4 (C'), 144.8, 143.0 (C'), 142.5, 142.4, 141.4 (C'), 136.7, 136.6 (C'), 136.23 (C'), 136.15, 132.8 (C'), 132.5, 130.0 (C'), 129.8, 123.9 (C'), 123.6, (C') 123.5, 123.2, 121.9 (C'), 120.3, 119.8 (C'), 118.5, 118.3, 117.1 (C'), 116.3, 115.6 (C'), 115.5, 112.5 (C'), 111.7, 110.7 (C'), 110.1, 71.0, 69.6 (C'), 68.6, 68.1 (C'), 67.5, 64.1, 63.6 (C'), 55.5, 50.2 (C'), 49.5, 13.75 (C'), 13.71.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3318, 2986, 2938, 2835, 1769, 1741, 1708, 1592, 1499, 1250, 1181.

HRMS (ESI) for C₂₉H₂₃⁷⁹BrNO₈, [M+H]⁺ (592.0602) found: 592.0607.

HRMS (ESI) for C₂₉H₂₃⁸¹BrNO₈, [M+H]⁺ (594.0581) found: 594.0591.

Ethyl-2-(5-bromo-2-hydroxyphenyl)-7-methoxy-1',3',4-trioxo-1',2,3,3'-tetrahydro-4H-spiro[chromeno[3,4-b]pyrrole-1,2'-indene]-3a(9bH)-carboxylate (3af).



Following the TP-C, **3af** was obtained from **1a** (71.6 mg, 0.2 mmol) and **2f** (56.1 mg, 1.0 equiv.) as a pale yellow solid (72.3 mg, 61%).

R_f = 0.175 (DCM:Hex = 4:1).

mp.: 132.7-133.0 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 10.74 (s, 1H), 10.26 (s, 1H'), 7.98 (d, J = 7.6 Hz, 1H), 7.84-7.72 (m, 4H'+1H), 7.65 (td, J = 7.6, 0.6 Hz, 1H), 7.52 (d, J = 7.6 Hz, 1H), 7.18 (dd, J = 8.7, 1.2 Hz, 1H'), 6.94 (dd, J = 8.8, 2.4 Hz, 1H), 6.78 (d, J = 8.7 Hz, 1H'), 6.70 (d, J = 8.8 Hz, 1H), 6.67 (d, J = 2.4 Hz, 1H'), 6.60 (d, J = 2.5 Hz, 1H), 6.58-6.51 (m, 1H'+2H), 6.43 (s, 1H'), 6.41-6.34 (m, 1H+1H'), 5.07 (d, J = 6.7 Hz, 1H), 4.94 (d, J = 6.3 Hz, 1H'), 4.60 (s, 1H'), 4.31 (s, 1H), 4.24 (q, J = 7.1 Hz, 3H+3H'), 3.73 (s, 3H'), 3.71 (s, 3H), 1.19 (t, J = 7.1 Hz, 3H'), 1.13 (t, J = 7.1 Hz, 3H).

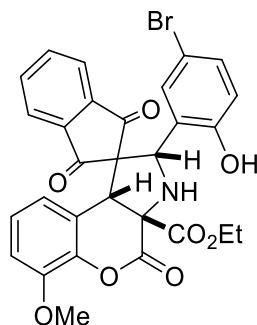
¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 199.1 (C'), 197.9, 197.2, 196.2 (C'), 168.8, 167.3 (C'), 166.1 (C'), 161.0 (C'), 164.8, 160.8, 156.9 (C'), 156.6, 152.4 (C'), 151.8, 143.1 (C'), 142.6, 142.4, 141.4 (C'), 136.6, 136.2 (C'), 136.1, 132.8 (C'), 132.4, 129.9 (C'), 129.8, 128.5 (C'), 127.9, 123.9 (C'), 123.7 (C'), 123.5, 123.2, 122.2 (C'), 120.3, 119.8 (C'), 118.5, 111.3, 110.7 (C'), 110.1, 107.9 (C'), 107.4, 102.5 (C'), 102.4, 70.9, 69.8 (C'), 68.5, 68.1 (C'), 67.7, 67.3 (C'), 64.1, 63.7 (C'), 55.5 (C'), 55.4, 50.1 (C'), 49.2, 13.8.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3318, 2936, 1771, 1708, 1627, 1590, 1484, 1162, 1109, 738, 630.

HRMS (ESI) for C₂₉H₂₃⁷⁹BrNO₈, [M+H]⁺ (592.0602) found: 592.0610.

HRMS (ESI) for C₂₉H₂₃⁸¹BrNO₈, [M+H]⁺ (594.0581) found: 594.0594.

Ethyl-2-(5-bromo-2-hydroxyphenyl)-6-methoxy-1',3',4-trioxo-1',2,3,3'-tetrahydro-4H-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3a(9*bH*)-carboxylate (3ag).



Following the TP-C, **3ag** was obtained from **1a** (71.6 mg, 0.2 mmol) and **2g** (56.1 mg, 1.0 equiv.) as a pale yellow solid (56.9 mg, 48%).

R_f = 0.225 (Hex:DCM:EA = 3:1:1).

mp.: 243.8-244.8 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 10.67 (s, 1H), 10.28 (s, 1H'), 7.99 (d, J = 7.6 Hz, 1H), 7.77 (t, J = 7.6 Hz, 1H+4H'), 7.64 (t, J = 7.6 Hz, 1H), 7.52 (d, J = 7.6 Hz, 1H), 7.18 (d, J = 8.6 Hz, 1H'), 6.94 (dd, J = 8.8, 2.3 Hz, 1H), 6.84-6.74 (m, 2H+3H'), 6.58-6.52 (m, 2H), 6.42-6.35 (m, 1H+1H'), 6.21 (d, J = 7.4 Hz, 1H'), 5.07 (d, J = 7.1 Hz, 1H), 4.94

(d, $J = 6.2$ Hz, 1H'), 4.66 (s, 1H'), 4.36 (s, 1H), 4.32-4.17 (m, 3H+3H'), 3.89 (s, 3H'), 3.85 (s, 3H), 1.18 (t, $J = 7.0$ Hz, 3H'), 1.13 (t, $J = 7.1$ Hz, 3H).

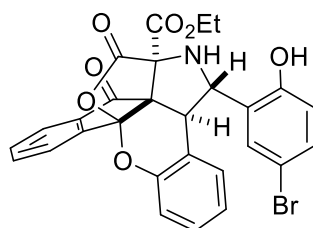
^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 198.6 (C'), 197.7, 196.8, 196.1 (C'), 168.7, 167.1 (C'), 165.4 (C'), 164.1, 156.5 (C'), 156.4, 147.7, 147.6 (C'), 143.0 (C'), 142.5, 142.2, 141.3 (C'), 140.8 (C'), 140.3, 136.5, 136.4 (C'), 135.8 (C'), 136.1, 132.6 (C'), 132.3, 130.0 (C'), 129.8, 127.1 (C'), 124.7 (C'), 124.4, 123.7 (C'), 123.4, 123.5 (C'), 123.1, 122.2 (C'), 120.2, 119.5 (C'), 118.8 (C'), 118.4, 118.2, 117.2 (C'), 116.5, 112.8 (C'), 112.6, 110.7 (C'), 110.0, 70.9, 69.5 (C'), 68.4, 68.0 (C'), 67.4, 67.0 (C'), 64.0, 63.5 (C'), 56.1, 50.1 (C'), 49.4, 13.7.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3317, 2981, 2937, 1769, 1742, 1707, 1589, 1485, 1254, 1180.

HRMS (ESI) for $\text{C}_{29}\text{H}_{23}^{79}\text{BrNO}_8$, $[\text{M}+\text{H}]^+$ (592.0602) found: 592.0610.

HRMS (ESI) for $\text{C}_{29}\text{H}_{23}^{81}\text{BrNO}_8$, $[\text{M}+\text{H}]^+$ (594.0581) found: 594.0594.

Ethyl-3-(5-bromo-2-hydroxyphenyl)-13,15-dioxo-3,3a-dihydro-13H-8a,1-(epoxymethano)indeno[1',2':2,3]chromeno[3,4-c]pyrrole-1(2H)-carboxylate (4aa).



Following the TP-D, **4aa** was obtained from **1a** (72.0 mg, 0.2 mmol) and **2a** (60.0 mg, 1.2 equiv.) as a pale yellow solid (88.9 mg, 79%).

$R_f = 0.375$ (Hex:DCM = 1:1).

mp.: 155.4-156.4 °C.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 10.2 (s, 1H), 8.04 (d, $J = 7.6$ Hz, 1H), 7.85 (t, $J = 7.6$ Hz, 1H), 7.70 (d, $J = 7.6$ Hz, 1H), 7.61 (t, $J = 7.6$ Hz, 1H), 7.30 (dd, $J = 8.7, 1.4$ Hz, 1H), 7.17 (d, $J = 7.8$ Hz, 1H), 6.97 (d, $J = 8.1$ Hz, 1H), 6.93-6.81 (m, 2H), 6.56-6.43 (m, 2H), 4.39 (d, $J = 8.1$ Hz, 1H), 4.24-4.07 (m, 2H), 3.97 (d, $J = 10.2$ Hz, 1H), 3.89 (s, 1H), 1.08 (t, $J = 7.1$ Hz, 3H).

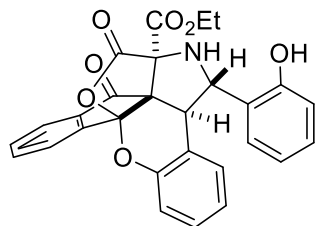
^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 196.0, 171.9, 165.7, 156.3, 149.6, 146.3, 137.4, 136.6, 132.6, 132.4, 131.6, 130.1, 129.5, 125.0, 124.2, 123.4, 122.4, 120.9, 119.8, 118.6, 110.8, 110.1, 75.4, 68.1, 67.4, 63.5, 50.6, 13.6.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3314, 2923, 2358, 1802, 1763, 1731, 1603, 1482, 1278, 1196.

HRMS (ESI) for $\text{C}_{28}\text{H}_{21}^{79}\text{BrNO}_7$, $[\text{M}+\text{H}]^+$ (562.0496) found: 562.0501.

HRMS (ESI) for $\text{C}_{28}\text{H}_{21}^{81}\text{BrNO}_7$, $[\text{M}+\text{H}]^+$ (564.0475) found: 564.0501.

Ethyl-3-(2-hydroxyphenyl)-13,15-dioxo-3,3a-dihydro-13*H*-8a,1-(epoxymethano)indeno [1',2':2,3]chromeno[3,4-*c*]pyrrole-1(2*H*)-carboxylate (4ba).



Following the TP-D, **4ba** was obtained from **1b** (55.9 mg, 0.2 mmol) and **2a** (60.0 mg, 1.2 equiv.) as a pale yellow solid (62.9 mg, 65 %).

R_f = 0.463 (EA:Hex:DCM = 1:3:1).

mp.: 253.0-254.0 °C.

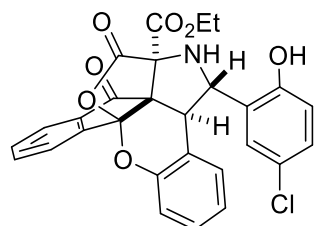
¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 8.03 (d, J = 7.8 Hz, 1H), 7.84 (t, J = 7.4 Hz, 1H), 7.70 (d, J = 7.7 Hz, 1H), 7.60 (t, J = 7.4 Hz, 1H), 7.21 (t, J = 7.7 Hz, 1H), 7.13 (t, J = 7.7 Hz, 1H), 6.99 (d, J = 8.1 Hz, 1H), 6.96 (d, J = 8.2 Hz, 1H), 6.79 (t, J = 7.3 Hz, 1H), 6.63 (t, J = 7.3 Hz, 1H), 6.46 (d, J = 7.4 Hz, 1H), 6.35 (d, J = 7.4 Hz, 1H), 4.46 (d, J = 10.3 Hz, 1H), 4.28-4.08 (m, 2H), 3.99 (d, J = 10.3 Hz, 1H), 1.10 (t, J = 7.1 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 196.0, 172.1, 165.8, 157.1, 149.7, 146.3, 137.2, 136.6, 132.3, 129.9, 129.8, 129.6, 129.2, 125.0, 124.0, 123.4, 121.1, 120.1, 119.1, 118.4, 118.0, 110.1, 75.3, 68.0, 67.8, 63.4, 50.4, 13.5.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3314, 3080, 2927, 2985, 2857, 1798, 1765, 1729, 1601, 1590, 1487, 1486.

HRMS (ESI) for C₂₈H₂₂NO₇, [M+H]⁺ (484.1391) found: 484.1393.

Ethyl-3-(5-chloro-2-hydroxyphenyl)-13,15-dioxo-3,3a-dihydro-13*H*-8a,1-(epoxymethano)indeno[1',2':2,3]chromeno[3,4-*c*]pyrrole-1(2*H*)-carboxylate (4ca).



Following the TP-D, **4ca** was obtained from **1c** (62.7 mg, 0.2 mmol) and **2a** (60.0 mg, 1.2 equiv.) as a pale yellow solid (73.8 mg, 71%).

R_f = 0.463 (EA:Hex:DCM = 1:3:1)

mp.: 237.3-238.0 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 10.19 (s, 1H), 8.03 (d, J = 7.7 Hz, 1H), 7.84 (t, J = 7.4 Hz, 1H), 7.70 (d, J = 7.7 Hz, 1H), 7.60 (t, J = 7.4 Hz, 1H), 7.22-7.11 (m, 2H), 6.95 (t, J

= 10.1 Hz, 2H), 6.85 (t, J = 7.4 Hz, 1H), 6.51 (d, J = 7.3 Hz, 1H), 6.34 (s, 1H), 4.42 (d, J = 10.2 Hz, 1H), 4.26-4.07 (m, 2H), 3.96 (d, J = 10.2 Hz, 1H), 3.80 (s, 1H), 1.08 (t, J = 7.1 Hz, 3H).

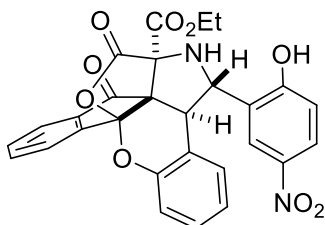
^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 195.8, 171.8, 165.7, 155.8, 149.6, 146.2, 137.3, 136.6, 132.4, 130.1, 129.7, 129.4, 128.6, 125.0, 124.2, 123.6, 123.4, 121.7, 120.8, 119.4, 118.6, 110.1, 75.2, 68.0, 67.3, 63.5, 50.4, 13.5.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3313, 3068, 2986, 2360, 1803, 1764, 1730, 1603, 1485, 1463.

HRMS (ESI) for $\text{C}_{28}\text{H}_{21}^{35}\text{ClNO}_7$, $[\text{M}+\text{H}]^+$ (518.1001) found: 518.1006.

HRMS (ESI) for $\text{C}_{28}\text{H}_{21}^{37}\text{ClNO}_7$, $[\text{M}+\text{H}]^+$ (520.0972) found: 520.1000.

Ethyl-3-(2-hydroxy-5-nitrophenyl)-13,15-dioxo-3,3a-dihydro-13H-8a,1-(epoxymethano)indeno[1',2':2,3]chromeno[3,4-c]pyrrole-1(2H)-carboxylate (4da).



Following the TP-D, **4da** was obtained from **1d** (64.9 mg, 0.2 mmol) and **2a** (60.0 mg, 1.2 equiv.) as a pale yellow solid (52.9 mg, 50%).

R_f = 0.388 (EA:Hex:DCM = 1:3:1)

mp.: 253.2-254.7 °C.

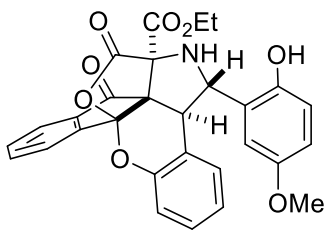
^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 11.38 (s, 1H), 8.15 (dd, J = 8.0, 2.7 Hz, 1H), 8.05 (d, J = 7.8 Hz, 1H), 7.87 (t, J = 7.5 Hz, 1H), 7.71 (d, J = 7.7 Hz, 1H), 7.63 (t, J = 7.5 Hz, 1H), 7.33 (d, J = 2.6 Hz, 1H), 7.19 (t, J = 7.5 Hz, 1H), 7.08 (d, J = 9.0 Hz, 1H), 7.00 (d, J = 8.0 Hz, 1H), 6.84 (t, J = 7.5 Hz, 1H), 6.45 (d, J = 7.0 Hz, 1H), 4.61 (d, J = 10.3 Hz, 1H), 4.28-4.10 (m, 2H), 3.92 (s, 1H), 3.90 (s, 1H), 1.09 (t, J = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 195.5, 171.5, 165.5, 163.4, 149.6, 146.1, 139.9, 137.5, 136.5, 132.5, 130.6, 129.1, 126.0, 125.1, 125.1, 124.5, 123.5, 120.6, 120.3, 118.9, 118.6, 110.1, 75.0, 68.0, 67.2, 63.7, 50.7, 13.5.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3314, 2927, 2853, 1802, 1763, 1733, 1587, 1520, 1486, 1461.

HRMS (ESI) for $\text{C}_{28}\text{H}_{21}\text{N}_2\text{O}_9$, $[\text{M}+\text{H}]^+$ (529.1242) found: 529.1248.

Ethyl-3-(2-hydroxy-5-methoxyphenyl)-13,15-dioxo-3,3a-dihydro-13H-8a,1-(epoxymethano)indeno[1',2':2,3]chromeno[3,4-c]pyrrole-1(2H)-carboxylate (4ea).



Following the TP-D, **4ea** was obtained from **1e** (63.1 mg, 0.2 mmol) and **2a** (60.0 mg, 1.2 equiv.) as a pale yellow solid (27.2 mg, 26%).

R_f = 0.450 (EA:Hex:DCM = 1:3:1)

mp.: 186.8-187.8 °C.

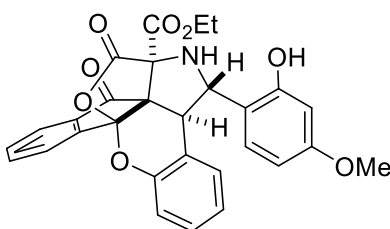
^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 10.17 (s, 1H), 8.04 (d, J = 7.8 Hz, 1H), 7.85 (t, J = 7.5 Hz, 1H), 7.71 (d, J = 7.7 Hz, 1H), 7.60 (t, J = 7.5 Hz, 1H), 7.14 (t, J = 7.7 Hz, 1H), 6.95 (d, J = 8.0 Hz, 1H), 6.81 (t, J = 7.4 Hz, 1H), 6.56 (d, J = 2.2 Hz, 1H), 6.51 (d, J = 7.4 Hz, 1H), 6.25 (d, J = 8.4 Hz, 1H), 6.20 (dd, J = 8.0, 2.3 Hz, 1H), 4.42 (d, J = 10.2 Hz, 1H), 4.27-4.10 (m, 2H), 3.94 (d, J = 10.2 Hz, 1H), 3.78 (s, 3H), 3.53 (s, 1H), 1.10 (t, J = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 196.1, 172.2, 165.8, 161.2, 158.4, 149.7, 146.4, 137.3, 136.7, 132.3, 129.9, 129.8, 129.7, 125.0, 124.0, 123.4, 121.2, 118.4, 112.3, 110.1, 105.5, 103.1, 75.1, 68.0, 67.5, 63.4, 55.2, 50.5, 13.6.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3309, 2920, 2851, 1800, 1760, 1728, 1603, 1588, 1487, 1464.

HRMS (ESI) for $\text{C}_{29}\text{H}_{24}\text{NO}_8$, $[\text{M}+\text{H}]^+$ (514.1496) found: 514.1498.

Ethyl-3-(2-hydroxy-4-methoxyphenyl)-13,15-dioxo-3,3a-dihydro-13H-8a,1-(epoxymethano)indeno[1',2':2,3]chromeno[3,4-c]pyrrole-1(2H)-carboxylate (4fa**).**



Following the TP-D, **4fa** was obtained from **1f** (63.1 mg, 0.2 mmol) and **2a** (60.0 mg, 1.2 equiv.) as a pale yellow solid (10.5 mg, 10%).

R_f = 0.375 (EA:Hex:DCM = 1:3:1)

mp.: 261.8-262.3 °C.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 8.03 (d, J = 7.8 Hz, 1H), 7.84 (t, J = 7.5 Hz, 1H), 7.70 (d, J = 7.7 Hz, 1H), 7.60 (t, J = 7.5 Hz, 1H), 7.13 (t, J = 7.7 Hz, 1H), 6.95 (d, J = 8.0 Hz, 1H), 6.81 (t, J = 7.5 Hz, 1H), 6.56 (s, 1H), 6.51 (d, J = 7.4 Hz, 1H), 6.25 (d, J = 8.4 Hz, 1H), 6.19 (d, J = 8.4 Hz, 1H), 4.41 (d, J = 10.2 Hz, 1H), 4.27-4.08 (m, 2H), 3.94 (d, J = 10.2 Hz,

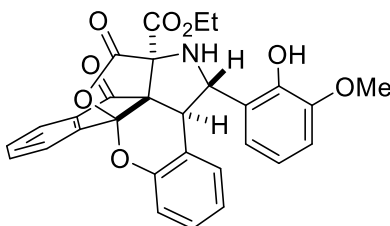
1H), 3.77 (s, 3H), 1.10 (t, $J = 7.1$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 196.0, 172.2, 165.8, 161.2, 158.4, 149.7, 146.4, 137.2, 136.7, 132.3, 129.9, 129.8, 129.7, 125.0, 124.0, 123.4, 121.3, 118.4, 112.3, 110.1, 105.5, 103.1, 75.1, 68.0, 67.5, 63.4, 55.2, 50.5, 13.6.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3295, 3081, 2991, 1802, 1766, 1730, 1626, 1589, 1511, 1487, 1459.

HRMS (ESI) for $\text{C}_{29}\text{H}_{24}\text{NO}_8$, $[\text{M}+\text{H}]^+$ (514.1496) found: 514.1496.

Ethyl-3-(2-hydroxy-3-methoxyphenyl)-13,15-dioxo-3,3a-dihydro-13H-8a,1-(epoxymethano)indeno[1',2':2,3]chromeno[3,4-c]pyrrole-1(2H)-carboxylate (4ga).



Following the TP-D, **4ga** was obtained from **1g** (63.1 mg, 0.2 mmol) and **2a** (60.0 mg, 1.2 equiv.) as a pale yellow solid (49.2 mg, 47%).

$R_f = 0.275$ (EA:Hex:DCM = 1:3:1)

mp.: 228.7-229.5 °C.

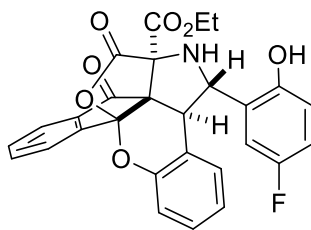
^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 8.10 (s, 1H), 8.04 (d, $J = 7.8$ Hz, 1H), 7.83 (t, $J = 7.5$ Hz, 1H), 7.70 (d, $J = 7.7$ Hz, 1H), 7.59 (t, $J = 7.5$ Hz, 1H), 7.10 (t, $J = 7.7$ Hz, 1H), 6.94 (d, $J = 8.0$ Hz, 1H), 6.85 (d, $J = 8.0$ Hz, 1H), 6.76 (t, $J = 7.4$ Hz, 1H), 6.67 (t, $J = 7.9$ Hz, 1H), 6.50 (d, $J = 7.4$ Hz, 1H), 6.30 (d, $J = 7.6$ Hz, 1H), 4.43 (dd, $J = 8.2, 8.0$ Hz, 1H), 4.30-4.05 (m, 4H), 3.92 (s, 3H), 1.06 (t, $J = 7.1$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 197.1, 172.0, 165.5, 149.7, 147.7, 146.6, 145.3, 137.0, 136.7, 132.1, 129.4, 129.3, 124.9, 123.7, 123.2, 121.7, 121.6, 120.9, 119.2, 118.2, 111.4, 109.9, 69.0, 67.5, 63.3, 56.1, 52.0, 13.5.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3448, 3311, 3062, 2982, 2940, 2843, 1805, 1760, 1728, 1603, 1588, 1486.

HRMS (ESI) for $\text{C}_{29}\text{H}_{24}\text{NO}_8$, $[\text{M}+\text{H}]^+$ (514.1496) found: 514.1501.

Ethyl-3-(5-fluoro-2-hydroxyphenyl)-13,15-dioxo-3,3a-dihydro-13H-8a,1-(epoxymethano)indeno[1',2':2,3]chromeno[3,4-c]pyrrole-1(2H)-carboxylate (4ha).



Following the TP-D, **4ha** was obtained from **1h** (61.3 mg, 0.2 mmol) and **2a** (60.0 mg, 1.2 equiv.) as a pale yellow solid (54.8 mg, 53%).

R_f = 0.500 (EA:Hex:DCM = 1:4:1)

mp.: 265.9-266.4 °C.

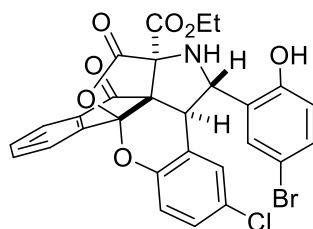
¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 9.88 (s, 1H), 9.74 (s, 1H'), 8.04 (d, J = 7.8 Hz, 1H+1H'), 7.85 (t, J = 7.4 Hz, 1H+1H'), 7.71 (d, J = 7.6 Hz, 1H+1H'), 7.61 (t, J = 7.5 Hz, 1H+1H'), 7.16 (t, J = 7.7 Hz, 1H+1H'), 7.03-6.91 (m, 3H+1H'), 6.84 (t, J = 7.4 Hz, 1H+1H'), 6.51 (d, J = 7.4 Hz, 1H+1H'), 6.33 (d, J = 2.0 Hz, 1H'), 6.30-6.24 (m, 1H'), 6.19-6.14 (m, 1H'), 6.10 (d, J = 7.4 Hz, 1H), 4.41 (d, J = 10.2 Hz, 1H+1H'), 4.28-4.10 (m, 2H+2H'), 3.99 (d, J = 10.2 Hz, 1H), 3.95 (s, 1H'), 3.69 (s, 1H+1H'), 1.17 (t, J = 7.1 Hz, 3H'), 1.10 (t, J = 7.1 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 195.8, 172.0, 165.7, 155.6 (d, J = 237.7 Hz), 153.1 (d, J = 2.0 Hz), 149.7, 146.3, 137.3, 136.7, 132.6 (C'), 132.4, 130.2 (C'), 130.1, 129.5, 125.2 (C'), 125.0, 124.6 (C'), 124.2, 124.3, 123.5, 121.0 (d, J = 6.8 Hz), 120.9, 118.9 (d, J = 7.6 Hz), 118.6, 116.3 (d, J = 22.7 Hz), 115.3 (d, J = 23.6 Hz), 110.1, 75.2, 68.0, 67.5, 63.6 (C'), 63.5, 50.4 (C'), 50.3, 13.5.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3319, 3099, 2919, 2848, 2360, 2334, 1798, 1761, 1731, 1603, 1485, 1470.

HRMS (ESI) for C₂₈H₂₀FNNaO₇, [M+Na]⁺ (524.1116) found: 524.1123.

Ethyl-3-(5-bromo-2-hydroxyphenyl)-5-chloro-13,15-dioxo-3,3a-dihydro-13H-8a,1-(epoxymethano)indeno[1',2':2,3]chromeno[3,4-c]pyrrole-1(2H)-carboxylate (4ab).



Following the TP-E, **4ab** was obtained from **1a** (72.0 mg, 0.2 mmol) and **2b** (68.4 mg, 1.2 equiv.) as a pale yellow solid (90.0 mg, 75%).

R_f = 0.488 (EA:Hex:DCM = 1:3:1)

mp.: 241.0-241.8 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 8.02 (d, J = 7.7 Hz, 1H), 7.87 (t, J = 7.5 Hz, 1H), 7.73 (d, J = 7.7 Hz, 1H), 7.64 (t, J = 7.5 Hz, 1H), 7.34 (dd, J = 10.0, 2.2 Hz, 1H), 7.14 (dd, J = 8.0, 2.4 Hz, 1H), 6.91 (dd, J = 8.0, 5.4 Hz, 2H), 6.52 (dd, J = 6.0, 2.3 Hz, 2H), 4.39 (d, J = 10.3 Hz, 1H), 4.27-4.08 (m, 2H), 3.91 (d, J = 10.2 Hz, 1H), 1.09 (t, J = 7.1 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 195.3, 171.6, 165.5, 156.2, 148.2, 145.9, 137.5, 136.5, 132.9, 132.6, 131.4, 130.2, 129.3, 129.1, 124.9, 123.6, 122.6, 121.8, 120.05, 119.98, 111.0, 110.1, 75.1, 67.7, 67.1, 63.6, 50.2, 13.5.

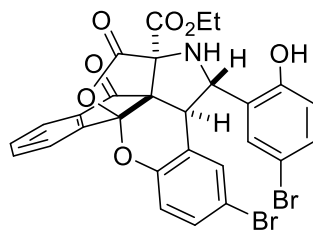
IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3310, 2927, 2857, 1801, 1764, 1731, 1603, 1481.

HRMS (ESI) for C₂₈H₂₀⁷⁹Br ³⁵Cl NO₇, [M+H]⁺ (596.0106) found: 596.0112.

HRMS (ESI) for C₂₈H₂₀⁷⁹Br ³⁷Cl NO₇, [M+H]⁺ (598.0077) found: 598.0081.

HRMS (ESI) for C₂₈H₂₀⁸¹Br ³⁷Cl NO₇, [M+H]⁺ (600.0056) found: 600.0086.

Ethyl-5-bromo-3-(5-bromo-2-hydroxyphenyl)-13,15-dioxo-3,3a-dihydro-13*H*-8a,1-(epoxymethano)indeno[1',2':2,3]chromeno[3,4-*c*]pyrrole-1(2*H*)-carboxylate (4ac).



Following the TP-D, **4ac** was obtained from **1a** (72.0 mg, 0.2 mmol) and **2c** (79.0 mg, 1.2 equiv.) as a pale yellow solid (101.8 mg, 79%).

R_f = 0.475 (EA:Hex:DCM = 1:3:1)

mp.: 255.0-255.8 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 10.08 (s, 1H), 8.01 (d, J = 7.7 Hz, 1H), 7.86 (t, J = 7.4 Hz, 1H), 7.72 (d, J = 7.6 Hz, 1H), 7.63 (t, J = 7.4 Hz, 1H), 7.34 (d, J = 8.3 Hz, 1H), 7.28 (d, J = 9.3 Hz, 1H), 6.90 (d, J = 8.7 Hz, 1H), 6.86 (d, J = 8.6 Hz, 1H), 6.64 (s, 1H), 6.53 (s, 1H), 4.39 (d, J = 10.0 Hz, 1H), 4.26-4.07 (m, 2H), 3.89 (d, J = 10.2 Hz, 1H), 3.83 (s, 1H), 1.08 (t, J = 7.1 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 195.3, 171.5, 165.5, 156.2, 148.7, 145.9, 137.5, 136.5, 133.2, 132.9, 132.6, 132.0, 131.4, 124.9, 123.6, 123.0, 121.9, 120.3, 120.0, 116.6, 110.9, 110.0, 75.1, 67.7, 67.1, 63.6, 50.1, 13.5.

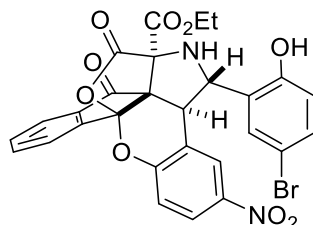
IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3315, 3075, 2984, 2927, 2853, 1803, 1764, 1730, 1603, 1578, 1478, 1420.

HRMS (ESI) for C₂₈H₂₀⁷⁹Br₂NO₇, [M+H]⁺ (639.9601) found: 639.9601.

HRMS (ESI) for C₂₈H₂₀⁷⁹Br⁸¹BrNO₇, [M+H]⁺ (641.9581) found: 641.9578.

HRMS (ESI) for $C_{28}H_{20}^{81}Br_2NO_7$, $[M+H]^+$ (643.9560) found: 643.9583.

Ethyl-3-(5-bromo-2-hydroxyphenyl)-5-nitro-13,15-dioxo-3,3a-dihydro-13H-8a,1-(epoxymethano)indeno[1',2':2,3]chromeno[3,4-c]pyrrole-1(2H)-carboxylate (4ad).



Following the TP-D, **4ad** was obtained from **1a** (72.0 mg, 0.2 mmol) and **2d** (70.9 mg, 1.2 equiv.) as a pale yellow solid (68.4 mg, 56%).

R_f = 0.325 (EA:Hex:DCM = 1:3:1)

mp.: 269.2-270.0 °C.

1H NMR (400 MHz, $CDCl_3$, 25 °C) δ /ppm: 9.76 (s, 1H), 8.10 (dd, J = 8.0, 2.1 Hz, 1H), 8.05 (d, J = 7.8 Hz, 1H), 7.91 (t, J = 7.5 Hz, 1H), 7.75 (d, J = 7.7 Hz, 1H), 7.67 (t, J = 7.4 Hz, 1H), 7.44 (s, 1H), 7.36 (d, J = 8.7 Hz, 1H), 7.14 (d, J = 8.9 Hz, 1H), 6.93 (d, J = 8.7 Hz, 1H), 6.50 (s, 1H), 4.35 (dd, J = 8.0, 4.4 Hz, 1H), 4.27-4.11 (m, 2H), 4.09 (d, J = 10.2 Hz, 1H), 3.81 (d, J = 4.2 Hz, 1H), 1.09 (t, J = 7.1 Hz, 3H).

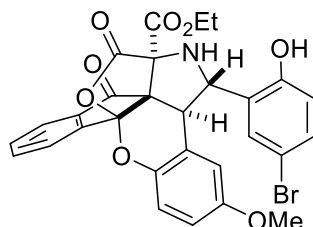
^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ /ppm: 194.8, 171.2, 165.1, 156.0, 154.4, 145.6, 143.6, 137.7, 136.2, 133.4, 132.9, 131.2, 126.1, 125.1, 124.9, 123.8, 121.8, 121.3, 120.3, 119.4, 111.3, 110.0, 75.3, 67.1, 67.1, 63.8, 49.6, 13.5.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3460, 3323, 2932, 2359, 1806, 1763, 1733, 1625, 1603, 1529, 1482.

HRMS (ESI) for $C_{28}H_{18}^{79}BrN_2O_9$, $[M-H]^-$ (605.0201) found: 605.0196.

HRMS (ESI) for $C_{28}H_{18}^{81}BrN_2O_9$, $[M-H]^-$ (607.0181) found: 607.0179.

Ethyl-3-(5-bromo-2-hydroxyphenyl)-5-methoxy-13,15-dioxo-3,3a-dihydro-13H-8a,1-(epoxymethano)indeno[1',2':2,3]chromeno[3,4-c]pyrrole-1(2H)-carboxylate (4ae).



Following the TP-E, **4ae** was obtained from **1a** (72.0 mg, 0.2 mmol) and **2e** (67.3 mg, 1.2 equiv.) as a pale yellow solid (83.4 mg, 70%).

R_f = 0.475 (EA:Hex:DCM = 1:3:1).

mp.: 165.4-166.2 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 8.02 (d, J = 7.7 Hz, 1H), 7.85 (t, J = 7.4 Hz, 1H), 7.70 (d, J = 7.6 Hz, 1H), 7.60 (t, J = 7.4 Hz, 1H), 7.31 (d, J = 8.1 Hz, 1H), 6.95-6.82 (m, 2H), 6.68 (dd, J = 10.0, 2.2 Hz, 1H), 6.53 (s, 1H), 6.01 (d, J = 2.0 Hz, 1H), 4.44 (d, J = 10.2 Hz, 1H), 4.26-4.07 (m, 2H), 3.88 (d, J = 10.2 Hz, 1H), 3.53 (s, 3H), 1.08 (t, J = 7.1 Hz, 3H).

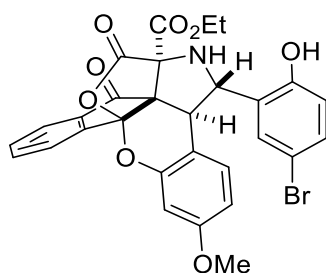
¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 195.8, 171.8, 165.7, 156.3, 155.8, 146.3, 143.1, 137.3, 136.6, 132.6, 132.4, 131.5, 125.0, 123.4, 122.3, 121.7, 119.9, 119.5, 116.2, 113.6, 110.6, 110.2, 75.1, 67.8, 67.1, 63.5, 55.6, 51.0, 13.5.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3455, 3313, 2985, 2936, 2835, 1802, 1764, 1731, 1624, 1601, 1590, 1509, 1481.

HRMS (ESI) for C₂₉H₂₃⁷⁹BrNO₈, [M+H]⁺ (592.0602) found: 592.0604.

HRMS (ESI) for C₂₉H₂₃⁸¹BrNO₈, [M+H]⁺ (594.0581) found: 594.0579.

Ethyl-3-(5-bromo-2-hydroxyphenyl)-6-methoxy-13,15-dioxo-3,3a-dihydro-13H-8a,1-(epoxymethano)indeno[1',2':2,3]chromeno[3,4-c]pyrrole-1(2H)-carboxylate (4af).



Following the TP-D, **4af** was obtained from **1a** (72.0 mg, 0.2 mmol) and **2f** (67.3 mg, 1.2 equiv.) as a pale yellow solid (90.5 mg, 76%).

R_f = 0.465 (EA:Hex:DCM = 1:3:1)

mp.: 234.5-235.5 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 8.03 (d, J = 7.7 Hz, 1H), 7.86 (t, J = 7.3 Hz, 1H), 7.71 (d, J = 7.7 Hz, 1H), 7.62 (t, J = 7.4 Hz, 1H), 7.31 (d, J = 8.6 Hz, 1H), 6.88 (d, J = 8.6 Hz, 1H), 6.52 (d, J = 7.9 Hz, 2H), 6.41 (s, 2H), 4.34 (d, J = 10.2 Hz, 1H), 4.26-4.08 (m, 2H), 3.91 (d, J = 10.2 Hz, 1H), 3.70 (s, 3H), 1.09 (t, J = 7.1 Hz, 3H).

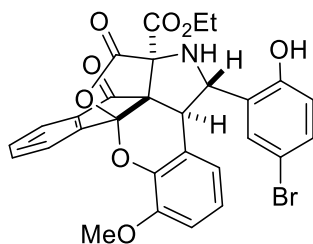
¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 195.9, 171.9, 165.7, 161.0, 156.3, 150.5, 146.2, 137.3, 136.6, 132.6, 132.4, 131.5, 129.9, 125.0, 123.4, 122.3, 119.9, 112.4, 110.8, 110.0, 109.7, 104.7, 75.0, 67.9, 67.3, 63.5, 55.4, 49.9, 13.5.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3310, 3095, 2957, 1802, 1764, 1731, 1624, 1601, 1590, 1509, 1481.

HRMS (ESI) for C₂₉H₂₃⁷⁹Br NO₈, [M+H]⁺ (592.0602) found: 592.0600.

HRMS (ESI) for C₂₉H₂₃⁸¹Br NO₈, [M+H]⁺ (594.0581) found: 594.0587.

Ethyl-3-(5-bromo-2-hydroxyphenyl)-7-methoxy-13,15-dioxo-3,3a-dihydro-13*H*-8a,1-(epoxymethano)indeno[1',2':2,3]chromeno[3,4-*c*]pyrrole-1(2*H*)-carboxylate (4ag**).**



Following the TP-D, **4ag** was obtained from **1a** (72.0 mg, 0.2 mmol) and **2g** (67.3 mg, 1.2 equiv.) as a pale yellow solid (82.2 mg, 69%).

R_f = 0.450 (EA:Hex:DCM = 1:3:1)

mp.: 253.5-254.1 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 10.30 (s, 1H), 8.12 (d, J = 7.8 Hz, 1H), 7.83 (t, J = 7.5 Hz, 1H), 7.69 (d, J = 7.6 Hz, 1H), 7.60 (t, J = 7.5 Hz, 1H), 7.30 (dd, J = 8.0, 2.2 Hz, 1H), 6.88 (d, J = 8.7 Hz, 1H), 6.80 (t, J = 7.9 Hz, 1H), 6.74 (d, J = 8.0 Hz, 1H), 6.51 (d, J = 2.2 Hz, 1H), 6.12 (d, J = 7.3 Hz, 1H), 4.49 (dd, J = 10.0, 2.2 Hz, 1H), 4.29-4.10 (m, 2H), 3.92 (d, J = 10.2 Hz, 1H), 3.79 (s, 3H), 3.78 (s, 1H), 1.10 (t, J = 7.1 Hz, 3H).

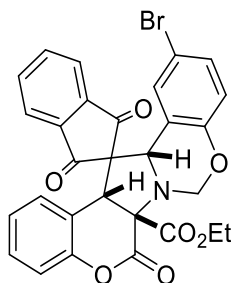
¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 195.8, 171.9, 165.9, 156.4, 149.6, 146.2, 138.4, 137.2, 136.8, 132.6, 132.4, 131.5, 125.4, 124.3, 123.3, 122.7, 122.5, 120.8, 119.9, 112.7, 110.7, 110.5, 74.9, 68.5, 67.1, 63.5, 55.9, 50.9, 13.6.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3316, 3062, 2917, 2850, 1802, 1765, 1731, 1684, 1602, 1590, 1482.

HRMS (ESI) for C₂₉H₂₃⁷⁹Br NO₈, [M+H]⁺ (592.0602) found: 592.0601.

HRMS (ESI) for C₂₉H₂₃⁸¹Br NO₈, [M+H]⁺ (594.0581) found: 594.0584.

Ethyl-12-bromo-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*,13*bH*-spiro[benzo[*e*]chromeno[4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6a(14a*H*)-carboxylate (5aa**).**



Following the TP-E, **5aa** was obtained from **3aa** (56.2 mg, 0.1 mmol) as a pale yellow solid (48.8 mg, 85%).

R_f = 0.375 (DCM:Hex = 3:1).

mp.: 174.8-175.3 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 8.10 (d, J = 7.7 Hz, 1H), 7.84 (t, J = 7.5 Hz, 1H), 7.70 (t, J = 7.5 Hz, 1H), 7.45 (d, J = 7.7 Hz, 1H), 7.14 (td, J = 6.8, 1.8 Hz, 1H), 7.01 (d, J = 8.0 Hz, 2H), 6.88-6.73 (m, 3H), 6.19 (d, J = 1.8 Hz, 1H), 5.81 (d, J = 10.6 Hz, 1H), 5.37 (s, 1H), 5.04 (d, J = 10.6 Hz, 1H), 4.53 (s, 1H), 4.28-4.08 (m, 2H), 1.08 (t, J = 7.1 Hz, 3H).

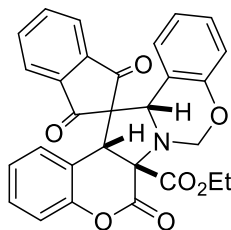
¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 198.4, 196.5, 168.9, 163.7, 155.4, 150.9, 143.0, 142.4, 136.7, 136.2, 131.3, 130.0, 128.0, 127.2, 124.4, 123.42, 123.37, 121.4, 120.5, 117.5, 115.7, 112.2, 76.4, 68.5, 68.3, 63.8, 63.2, 51.2, 13.7.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 2922, 2854, 1775, 1743, 1708, 1592, 1484, 1157, 1124, 761, 614.

HRMS (ESI) for C₂₉H₂₁⁷⁹NO₇, [M+H]⁺ (574.0496) found: 574.0504.

HRMS (ESI) for C₂₉H₂₁⁸¹NO₇, [M+H]⁺ (576.0475) found: 576.0490.

Ethyl-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*,13*bH*-spiro[benzo[*e*]chromeno[4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6*a*(14*aH*)-carboxylate (5*ba*).



Following the TP-E, **5ba** was obtained from **3ba** (48.4 mg, 0.1 mmol) as a pale yellow solid (40.6 mg, 82%).

R_f = 0.250 (DCM:Hex = 3:1).

mp.: 238.9-239.5 °C.

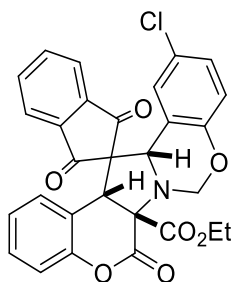
¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 8.06 (d, J = 7.6 Hz, 1H), 7.79 (td, J = 7.4, 0.7 Hz, 1H), 7.63 (td, J = 7.4, 0.7 Hz, 1H), 7.39 (dd, J = 7.1, 0.7 Hz, 1H), 7.12 (td, J = 7.8, 1.6 Hz, 1H), 7.00 (d, J = 8.2 Hz, 1H), 6.96-6.84 (m, 2H), 6.84-6.74 (m, 2H), 6.33 (td, J = 6.9, 1.2 Hz, 1H), 6.13 (d, J = 7.8 Hz, 1H), 5.81 (dd, J = 10.5, 1.2 Hz, 1H), 5.5 (s, 1H), 5.08 (d, J = 10.5 Hz, 1H), 4.54 (s, 1H), 4.26-4.10 (m, 2H), 1.09 (t, J = 7.1 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 198.8, 196.6, 169.2, 163.8, 156.3, 150.9, 143.1, 142.5, 136.4, 135.9, 129.9, 128.4, 127.2, 125.0, 124.2, 123.3, 123.2, 120.1, 119.6, 118.8, 117.5, 115.8, 76.3, 68.6, 68.3, 64.3, 63.1, 51.6, 13.8.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 2984, 2920, 1774, 1743, 1708, 1592, 1491, 1159, 1114, 759.

HRMS (ESI) for C₂₉H₂₂NO₇, [M+H]⁺ (496.1391) found: 496.1400.

Ethyl-12-chloro-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*,13*bH*-spiro[benzo[*e*]chromeno[4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6*a*(14*aH*)-carboxylate (5*ca*).



Following the TP-E, **5ca** was obtained from **3ca** (51.7 mg, 0.1 mmol) as a yellow solid (49.8 mg, 94%).

R_f = 0.375 (EA:Hex = 1:3).

mp.: 102.8-103.6 °C.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 8.09 (d, J = 7.7 Hz, 1H), 7.83 (t, J = 7.5 Hz, 1H), 7.69 (t, J = 7.5 Hz, 1H), 7.44 (d, J = 7.7 Hz, 1H), 7.14 (td, J = 7.6, 1.7 Hz, 1H), 7.01 (d, J = 8.2 Hz, 1H), 6.89 (dd, J = 8.8, 2.0 Hz, 1H), 6.86-6.74 (m, 3H), 6.06 (d, J = 2.0 Hz, 1H), 5.81 (d, J = 10.5 Hz, 1H), 5.38 (s, 1H), 5.05 (d, J = 10.5 Hz, 1H), 4.53 (s, 1H), 4.27-4.09 (m, 2H), 1.09 (t, J = 7.1 Hz, 3H).

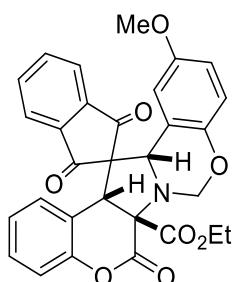
^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 198.4, 196.4, 168.9, 163.6, 154.9, 150.9, 143.0, 142.4, 136.7, 136.1, 130.0, 128.5, 127.2, 125.0, 124.9, 124.3, 123.4, 123.3, 120.9, 120.2, 117.5, 115.6, 76.5, 68.5, 68.2, 63.8, 63.2, 51.4, 13.7.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 2922, 1774, 1743, 1708, 1592, 1488, 1159, 1116, 830, 761, 738.

HRMS (ESI) for $\text{C}_{29}\text{H}_{21}^{35}\text{ClNO}_7$, $[\text{M}+\text{H}]^+$ (530.1001) found: 530.1009.

HRMS (ESI) for $\text{C}_{29}\text{H}_{21}^{37}\text{ClNO}_7$, $[\text{M}+\text{H}]^+$ (532.0972) found: 532.0991.

Ethyl-12-methoxy-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*,13*bH*-spiro[benzo[*e*]chromeno [4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6a(14a*H*)-carboxylate (5ea**).**



Following the TP-E, **5ea** was obtained from **3ea** (51.4 mg, 0.1 mmol) as a pale yellow solid (43.6 mg, 83%).

R_f = 0.450 (DCM:Hex = 3:1).

mp.: 108.4-109.3 °C.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 8.06 (d, J = 7.6 Hz, 1H), 7.79 (t, J = 7.5 Hz, 1H),

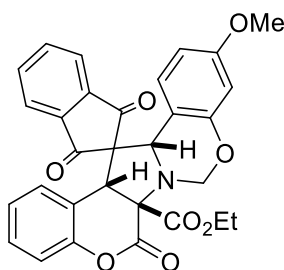
7.66 (t, $J = 7.5$ Hz, 1H), 7.45 (d, $J = 7.6$ Hz, 1H), 7.13 (t, $J = 7.7$ Hz, 1H), 7.02 (d, $J = 8.2$ Hz, 1H), 6.86-6.74 (m, 3H), 6.52 (dd, $J = 9.0, 2.6$ Hz, 1H), 5.78 (d, $J = 10.5$ Hz, 1H), 5.60 (d, $J = 2.6$ Hz, 1H), 5.42 (s, 1H), 5.03 (d, $J = 10.5$ Hz, 1H), 4.54 (s, 1H), 4.25-4.10 (m, 2H), 3.10 (s, 3H), 1.09 (t, $J = 7.1$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 198.9, 196.4, 169.2, 163.7, 152.9, 143.3, 142.6, 136.4, 135.7, 129.9, 127.2, 124.2, 123.5, 123.0, 119.7, 119.6, 117.5, 115.8, 115.7, 108.9, 77.3, 77.0, 76.7, 68.6, 68.3, 64.6, 63.1, 55.1, 51.5, 13.7.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3436, 2926, 2855, 1775, 1743, 1708, 1592, 1500, 1245, 1164.

HRMS (ESI) for $\text{C}_{30}\text{H}_{24}\text{NO}_8$, $[\text{M}+\text{H}]^+$ (526.1496) found: 526.1506.

Ethyl-11-methoxy-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*,13*bH*-spiro[benzo[*e*]chromeno [4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6*a*(14*aH*)-carboxylate (5fa).



Following the TP-E, **5fa** was obtained from **3fa** (51.4 mg, 0.1 mmol) as a pale yellow solid (36.8 mg, 70% yield).

$R_f = 0.375$ (DCM:Hex = 3:1).

mp.: 150.6-151.7 °C.

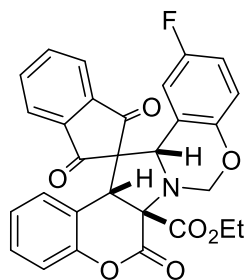
^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 8.06 (d, $J = 7.7$ Hz, 1H), 7.81 (t, $J = 7.5$ Hz, 1H), 7.65 (t, $J = 7.5$ Hz, 1H), 7.41 (d, $J = 7.7$ Hz, 1H), 7.12 (t, $J = 7.6$ Hz, 1H), 7.00 (d, $J = 8.2$ Hz, 1H), 6.84-6.74 (m, 2H), 6.42 (d, $J = 2.3$ Hz, 1H), 6.02 (d, $J = 8.6$ Hz, 1H), 5.92 (dd, $J = 8.6, 2.3$ Hz, 1H), 5.80 (d, $J = 10.4$ Hz, 1H), 5.41 (s, 1H), 5.06 (d, $J = 10.4$ Hz, 1H), 4.53 (s, 1H), 4.26-4.11 (m, 2H), 3.61 (s, 3H), 1.09 (t, $J = 7.1$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 198.8, 196.7, 169.1, 163.7, 159.4, 157.3, 150.8, 143.0, 142.4, 136.4, 135.8, 129.7, 127.1, 125.6, 124.1, 123.2, 123.1, 117.3, 115.8, 111.5, 107.8, 102.8, 76.3, 68.5, 68.3, 64.1, 63.0, 55.0, 51.3, 13.7.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3429, 2920, 2853, 1772, 1742, 1708, 1592, 1494, 1249, 1158.

HRMS (ESI) for $\text{C}_{30}\text{H}_{24}\text{NO}_8$, $[\text{M}+\text{H}]^+$ (526.1496) found: 526.1501.

Ethyl-12-fluoro-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*,13*bH*-spiro [benzo[*e*]chromeno [4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6*a*(14*aH*)-carboxylate (5ha).



Following the TP-E, **5ha** was obtained from **3ha** (50.1 mg, 0.1 mmol) as a white solid (44.2 mg, 86%).

R_f = 0.325 (DCM:Hex EA=1:3:1).

mp.: 168.8-173.8 °C.

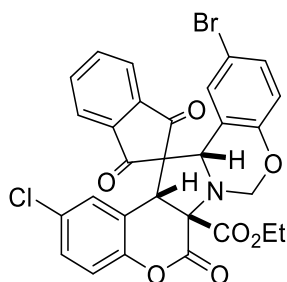
^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 8.07 (d, J = 7.6 Hz, 1H), 7.82 (t, J = 7.5 Hz, 1H), 7.67 (t, J = 7.5 Hz, 1H), 7.45 (d, J = 7.7 Hz, 1H), 7.13 (td, J = 7.3, 1.8 Hz, 1H), 7.01 (d, J = 8.2 Hz, 2H), 6.88-6.73 (m, 3H), 6.66 (td, J = 8.5, 2.5 Hz, 1H), 5.84 (dd, J = 8.5, 2.6 Hz, 1H), 5.80 (d, J = 10.6 Hz, 1H), 5.41 (s, 1H), 5.04 (d, J = 10.5 Hz, 1H), 4.51 (s, 1H), 4.29-4.09 (m, 2H), 1.09 (t, J = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 198.5, 196.4, 169.0, 163.6, 157.4, 155.0, 152.4 (d, J = 1.8 Hz), 150.9, 142.7 (d, J = 61.5 Hz), 136.6, 136.1, 128.6 (d, J = 277.5 Hz), 124.3, 123.5 (d, J = 6.1 Hz), 120.5, 120.4, 120.1, 120.0, 117.5, 115.6, 115.3, 111.1, 110.9, 68.6, 68.1, 63.8, 63.2, 51.7, 13.7.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3468.0, 2953, 2922, 2853, 1794, 1763, 1735, 1492, 1463, 1279, 1211, 1022

HRMS (ESI) for $\text{C}_{29}\text{H}_{21}\text{FNO}_7$, $[\text{M}+\text{H}]^+$ (514.1297) found: 514.1301.

Ethyl-12-bromo-2-chloro-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*,13*bH*-spiro[benzo[*e*]chromeno[4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6*a*(14*aH*)-carboxylate (5ab**).**



Following the TP-D, **5ab** was obtained from **3ab** (59.8 mg, 0.1 mmol) as a white solid (57.2 mg, 94%).

R_f = 0.313 (Hex:EA = 4:1).

mp.: 169.5-170.5 °C.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 8.14 (d, J = 7.5 Hz, 1H), 7.89 (t, J = 7.5 Hz, 1H),

7.75 (t, $J = 7.5$ Hz, 1H), 7.47 (d, $J = 7.5$ Hz, 1H), 7.12 (dd, $J = 8.8, 2.4$ Hz, 1H), 7.02 (dd, $J = 8.9, 2.2$ Hz, 1H), 6.97 (d, $J = 8.8$ Hz, 1H), 6.84 (d, $J = 2.4$ Hz, 1H), 6.76 (d, $J = 8.8$ Hz, 1H), 6.18 (s, 1H), 5.80 (d, $J = 10.6$ Hz, 1H), 5.34 (s, 1H), 5.01 (d, $J = 10.6$ Hz, 1H), 4.50 (s, 1H), 4.27-4.13 (m, 2H), 1.13 (t, $J = 7.1$ Hz, 3H).

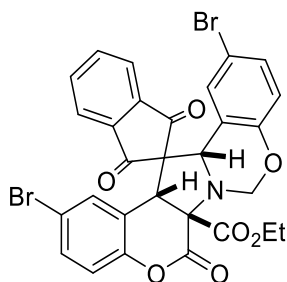
^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 197.8, 196.3, 168.6, 163.1, 155.4, 149.5, 142.9, 142.4, 137.0, 136.5, 131.4, 130.2, 129.4, 128.2, 127.0, 123.6, 123.5, 121.0, 120.5, 118.9, 117.5, 112.2, 76.3, 68.3, 68.2, 64.0, 63.4, 50.5, 13.8.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 2982, 2922, 1778, 1742, 1708, 1591, 1484, 1215, 1154.

HRMS (ESI) for $\text{C}_{29}\text{H}_{20}^{79}\text{Br}^{35}\text{ClNO}_7$, $[\text{M}+\text{H}]^+$ (608.0106) found: 608.0114.

HRMS (ESI) for $\text{C}_{29}\text{H}_{20}^{81}\text{Br}^{35}\text{ClNO}_7$, $[\text{M}+\text{H}]^+$ (610.0086) found: 610.0098.

Ethyl-2,12-dibromo-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*,13*bH*-spiro[benzo[*e*]chromeno [4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6*aH*)-carboxylate (5ac).



Following the TP-E, **5ac** was obtained from **3ac** (64.2 mg, 0.1 mmol) as a white solid (60.8 mg, 93%).

$R_f = 0.300$ (Hex:EA = 4:1).

mp.: 128.2-129.2 °C.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 8.14 (d, $J = 7.6$ Hz, 1H), 7.89 (t, $J = 7.6$ Hz, 1H), 7.75 (t, $J = 7.6$ Hz, 1H), 7.47 (d, $J = 7.6$ Hz, 1H), 7.26 (d, $J = 8.7$ Hz, 1H), 7.06-6.97 (m, 2H), 6.91 (d, $J = 8.9$ Hz, 1H), 6.75 (d, $J = 8.8$ Hz, 1H), 6.18 (s, 1H), 5.80 (d, $J = 10.6$ Hz, 1H), 5.34 (s, 1H), 5.01 (d, $J = 10.6$ Hz, 1H), 4.49 (s, 1H), 4.27-4.14 (m, 2H), 1.13 (t, $J = 7.1$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 197.8, 196.2, 168.5, 163.0, 155.4, 150.0, 142.9, 142.4, 136.9, 136.5, 133.1, 131.4, 129.9, 128.1, 123.5, 123.4, 121.0, 120.5, 119.2, 117.9, 116.7, 112.2, 76.3, 68.3, 68.2, 63.9, 63.4, 50.4, 13.8.

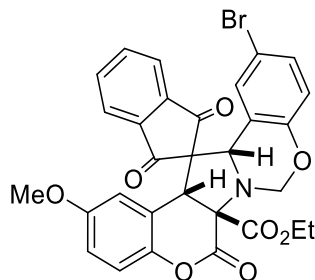
IR (KBr) $\tilde{\nu}$ (cm^{-1}): 2982, 2921, 1778, 1743, 1707, 1591, 1483, 1214, 1154.

HRMS (ESI) for $\text{C}_{29}\text{H}_{20}^{79}\text{Br}_2\text{NO}_7$, $[\text{M}+\text{H}]^+$ (651.9601) found: 651.9609.

HRMS (ESI) for $\text{C}_{29}\text{H}_{20}^{79}\text{Br}^{81}\text{BrNO}_7$, $[\text{M}+\text{H}]^+$ (653.9581) found: 653.9592.

HRMS (ESI) for $\text{C}_{29}\text{H}_{20}^{81}\text{Br}_2\text{NO}_7$, $[\text{M}+\text{H}]^+$ (655.9561) found: 655.9577.

Ethyl-12-bromo-2-methoxy-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*,13*bH*-spiro[benzo[e]chromeno[4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6*a*(14*aH*)-carboxylate (5ae**).**



Following the TP-E, **5ae** was obtained from **3ae** (59.2 mg, 0.1 mmol) as a pale yellow solid (53.2 mg, 88%).

R_f = 0.100 (DCM:Hex = 3:1).

mp.: 126.7-127.1 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 8.10 (d, J = 7.6 Hz, 1H), 7.85 (t, J = 7.5 Hz, 1H), 7.72 (t, J = 7.5 Hz, 1H), 7.47 (d, J = 7.6 Hz, 1H), 7.02 (dd, J = 8.9, 1.8 Hz, 1H), 6.95 (d, J = 8.9 Hz, 1H), 6.76 (d, J = 8.9 Hz, 1H), 6.67 (dd, J = 8.9, 2.8 Hz, 1H), 6.31 (d, J = 2.8 Hz, 1H), 6.19 (s, 1H), 5.82 (d, J = 10.5 Hz, 1H), 5.35 (s, 1H), 5.03 (d, J = 10.5 Hz, 1H), 4.49 (s, 1H), 4.26-4.12 (m, 2H), 3.54 (s, 3H), 1.12 (t, J = 7.1 Hz, 3H).

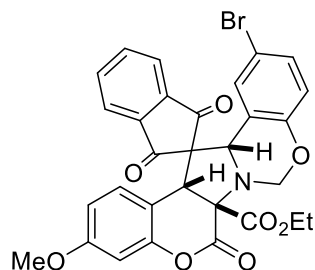
¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 198.4, 196.2, 169.0, 163.8, 155.8, 155.4, 144.8, 143.1, 142.5, 136.8, 136.2, 131.3, 128.0, 123.5, 123.3, 121.4, 120.5, 118.4, 116.3, 115.7, 112.1, 111.5, 76.4, 68.3, 68.2, 63.8, 63.2, 55.5, 51.4, 13.8.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3424, 2928, 2854, 1770, 1742, 1707, 1593, 1500, 1250, 1164.

HRMS (ESI) for C₃₀H₂₃⁷⁹BrNO₈, [M+H]⁺ (604.0602) found: 604.0608.

HRMS (ESI) for C₃₀H₂₃⁸¹BrNO₈, [M+H]⁺ (606.0581) found: 606.0593.

Ethyl-12-bromo-3-methoxy-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*,13*bH*-spiro[benzo[e]chromeno[4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6*a*(14*aH*)-carboxylate (5af**).**



Following the TP-D, **5af** was obtained from **3af** (59.2 mg, 0.1 mmol) as a yellow solid (53.2 mg, 88%).

R_f = 0.250 (DCM:Hex = 3:1).

mp.: 199.0-200.0 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 8.09 (d, J = 7.6 Hz, 1H), 7.83 (td, J = 7.4, 1.0 Hz, 1H), 7.71 (td, J = 7.4, 1.0 Hz, 1H), 7.47 (d, J = 7.6 Hz, 1H), 7.01 (dd, J = 8.8, 2.5 Hz, 1H), 6.76 (d, J = 8.8 Hz, 1H), 6.71 (d, J = 8.6 Hz, 1H), 6.55 (d, J = 2.4 Hz, 1H), 6.35 (dd, J = 8.6, 2.5 Hz, 1H), 6.18 (d, J = 2.4 Hz, 1H), 5.79 (d, J = 10.5 Hz, 1H), 5.35 (s, 1H), 5.03 (d, J = 10.5 Hz, 1H), 4.48 (s, 1H), 4.28-4.11 (m, 2H), 3.68 (s, 3H), 1.12 (t, J = 7.1 Hz, 3H).

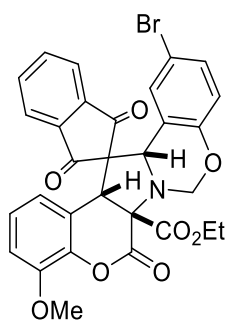
¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 198.5, 196.7, 169.0, 163.8, 160.7, 155.4, 151.8, 143.1, 142.5, 136.7, 136.1, 131.3, 128.0, 127.9, 123.4, 123.3, 121.5, 120.5, 112.1, 111.2, 107.4, 102.3, 76.4, 68.5, 68.2, 63.7, 63.2, 55.4, 51.0, 13.8.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 2925, 1775, 1743, 1708, 1627, 1591, 1484, 1163, 1129, 737, 616.

HRMS (ESI) for C₃₀H₂₃⁷⁹BrNO₈, [M+H]⁺ (604.0608) found: 604.0609.

HRMS (ESI) for C₃₀H₂₃⁸¹BrNO₈, [M+H]⁺ (606.0581) found: 606.0593.

Ethyl-12-bromo-4-methoxy-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*,13*bH*-spiro[benzo[e]chromeno[4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6a(14*aH*)-carboxylate (5ag**).**



Following the TP-E, **5ag** was obtained from **3ag** (59.4 mg, 0.1 mmol) as a pale yellow solid (55.0 mg, 91%).

R_f = 0.088 (Hex:EA = 4:1).

mp.: 254.2-255.2 °C.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 8.10 (d, J = 7.6 Hz, 1H), 7.86 (t, J = 7.6 Hz, 1H), 7.72 (t, J = 7.6 Hz, 1H), 7.45 (d, J = 7.6 Hz, 1H), 7.0 (dd, J = 8.8, 2.1 Hz, 1H), 6.76-6.68 (m, 3H), 6.40 (dd, J = 7.1, 1.7 Hz, 1H), 6.19 (d, J = 2.1 Hz, 1H), 5.82 (d, J = 10.5 Hz, 1H), 5.35 (s, 1H), 5.02 (d, J = 10.5 Hz, 1H), 4.56 (s, 1H), 4.29-4.10 (m, 2H), 3.78 (s, 3H), 1.12 (t, J = 7.1 Hz, 3H).

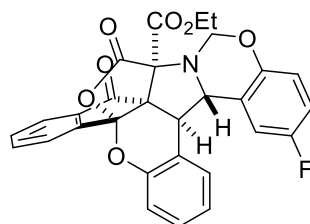
¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 198.3, 196.3, 168.8, 162.8, 155.2, 147.5, 142.9, 142.3, 140.2, 136.7, 136.1, 131.1, 127.9, 124.2, 123.32, 123.26, 121.3, 120.4, 118.2, 116.4, 112.4, 112.0, 76.3, 68.1, 68.0, 63.6, 63.1, 55.9, 51.2, 13.7.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 2981, 2937, 1770, 1742, 1707, 1590, 1486, 1213.

HRMS (ESI) for $C_{30}H_{23}^{79}BrNO_8$, $[M+H]^+$ (604.0608) found: 604.0609.

HRMS (ESI) for $C_{30}H_{23}^{81}BrNO_8$, $[M+H]^+$ (606.0581) found: 606.0594.

Ethyl-3-fluoro-14,19-dioxo-4b,4c-dihydro-14*H*,15*H*,17*H*-9a,15-(epoxymethano)benzo[*e*]indeno[1'',2'':2',3']chromeno[4',3':3,4]pyrrolo[1,2-*c*][1,3]oxazine-15-carboxylate (12ha).



Following the TP-E, **12ha** was obtained from **4ha** (25.1 mg, 0.05 mmol) as a yellow solid (19.3 mg, 75%);

R_f = 0.500 (EA:Hex = 1:3).

mp.: 171.0-172.0 °C.

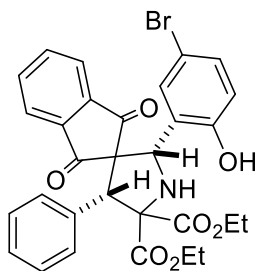
¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 8.02 (d, J = 7.7 Hz, 1H), 7.84 (t, J = 7.3 Hz, 1H), 7.67 (d, J = 7.8 Hz, 1H), 7.59 (t, J = 7.5 Hz, 1H), 7.28-7.20 (m, 2H), 7.08 (t, J = 7.5 Hz, 1H), 7.00 (d, J = 8.0 Hz, 1H), 6.94-6.86 (m, 2H), 6.54 (d, J = 9.2 Hz, 1H), 5.37 (d, J = 8.5 Hz, 1H), 4.85 (d, J = 8.5 Hz, 1H), 4.57 (d, J = 9.5 Hz, 1H), 4.25-4.15 (m, 1H), 4.12-4.01 (m, 1H), 3.82 (d, J = 9.5 Hz, 1H), 1.01 (t, J = 7.1 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 196.1, 169.6, 164.5, 156.7 (d, J = 239.1 Hz), 150.4, 149.5 (d, J = 2.1 Hz), 146.2, 137.2, 136.9, 131.3 (d, J = 189.9 Hz), 130.0, 125.0, 124.7, 123.2, 122.7, 119.3, 119.0 (d, J = 8.0 Hz), 115.6, 115.3, 111.5, 111.3, 110.5, 76.5, 75.3, 70.3, 63.0, 60.7 (d, J = 1.2 Hz), 50.3, 13.4.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3626, 3530, 2954, 2923, 2852, 1774, 1743, 1708, 1592, 1495, 1459, 1347.

HRMS (ESI) for $C_{29}H_{21}FNO_7$, $[M+H]^+$ (514.1297) found: 514.1307.

Diethyl-2'-(5-bromo-2-hydroxyphenyl)-1,3-dioxo-4'-phenyl-1,3-dihydrospiro[indene-2,3'-pyrrolidine]-5',5'-dicarboxylate (7a).



Following the TP-C, **7a** was obtained from **1a** (71.6 mg, 0.2 mmol) and **6** (46.9 mg, 1.0 equiv.) as a pale yellow solid (69.9 mg, 59%).

$R_f = 0.325$ (DCM:Hex:EA = 1:3:1).

mp.: 159.0-160.5 °C.

^1H NMR (400 MHz, CDCl_3 , 25 °C) (mixture of rotamers) δ /ppm: 7.88-7.78 (m, 1H+1H'), 7.74 (d, $J = 3.0$ Hz, 3H), 7.67-7.63 (m, 3H'), 7.31 (t, $J = 2.9$ Hz, 3H+1H'), 7.18-7.11 (m, 2H+2H'), 7.11-7.06 (m, 2H'), 7.01 (dd, $J = 8.8, 2.2$ Hz, 1H), 6.69 (d, $J = 2.0$ Hz, 1H'+1H), 6.64 (d, $J = 8.8$ Hz, 1H), 6.55 (d, $J = 2.2$ Hz, 1H'), 5.64 (s, 1H), 4.91 (s, 1H'), 4.85 (s, 1H'), 4.66 (s, 1H), 4.58-4.48 (m, 1H), 4.44-4.33 (m, 1H+1H'), 4.33-4.28 (m, 1H'), 4.22-4.14 (m, 1H'), 3.98-3.89 (m, 1H'), 3.87-3.67 (m, 1H'+2H), 1.46 (t, $J = 7.1$ Hz, 3H), 1.31 (t, $J = 7.1$ Hz, 3H'), 1.01 (t, $J = 7.1$ Hz, 3H'), 0.66 (t, $J = 7.1$ Hz, 3H).

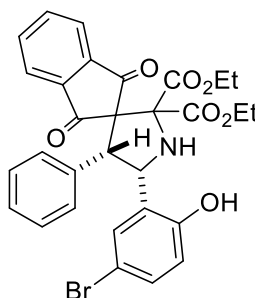
^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 198.9 (C'), 198.1, 197.7 (C'), 195.9, 170.3 (C'), 168.9, 168.7, 167.8 (C'), 157.1, 156.6 (C'), 147.0, 142.8 (C'), 142.5, 141.7 (C'), 140.9, 136.3 (C'), 136.0, 135.4 (C'), 135.6, 135.2 (C'), 135.0, 134.3 (C'), 134.1, 132.3 (C'), 132.1, 130.5 (C'), 130.0, 129.9 (C'), 129.7, 128.8 (C'), 128.3, 128.1, 124.0, 123.4 (C'), 123.4, 123.2 (C'), 120.4 (C'), 119.9, 110.2, 110.2 (C'), 78.3, 69.3 (C'), 68.3, 67.2 (C'), 66.5, 63.3, 63.2 (C'), 63.1, 62.5 (C'), 62.4 (C'), 62.4, 58.2 (C'), 57.2, 14.0 (C'), 14.0, 13.5 (C'), 13.1

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3304, 2982, 2362, 2338, 1727, 1709, 1597, 1485, 1243, 1169.

HRMS (ESI) for $\text{C}_{30}\text{H}_{27}^{79}\text{BrNO}_7$, $[\text{M}+\text{H}]^+$ (592.0965) found: 592.0977.

HRMS (ESI) for $\text{C}_{30}\text{H}_{27}^{81}\text{BrNO}_7$, $[\text{M}+\text{H}]^+$ (594.0945) found: 594.0962.

Diethyl-5'-(5-bromo-2-hydroxyphenyl)-1,3-dioxo-4'-phenyl-1,3-dihydrospiro[indene-2,3'-pyrrolidine]-2',2'-dicarboxylate (8a**).**



Following the TP-D, **8a** was obtained from **1a** (72.0 mg, 0.2 mmol) and **6** (56.2 mg, 1.2 equiv.) as a pale yellow solid (70.9 mg, 60%).

R_f = 0.500 (EA:Hex:DCM = 1:4:1)

mp.: 174.3-175.2 °C.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 7.84-7.77 (m, 1H), 7.76-7.70 (m, 1H), 7.70-7.64 (m, 2H), 7.13 (dd, J = 8.0, 2.2 Hz, 1H), 7.02-6.91 (m, 6H), 6.74 (d, J = 8.7 Hz, 1H), 5.56 (d, J = 11.4 Hz, 1H), 4.48-4.33 (m, 2H), 4.29 (d, J = 11.4 Hz, 1H), 4.05 (q, J = 7.1 Hz, 2H), 1.34 (t, J = 7.2 Hz, 3H), 0.90 (t, J = 7.1 Hz, 3H).

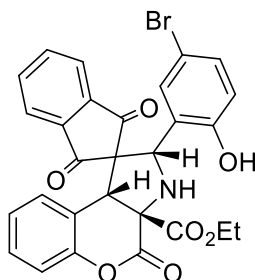
^{13}C NMR (100 MHz, CDCl_3 , 25 °C) δ /ppm: 199.7, 196.5, 169.5, 168.3, 156.5, 141.9, 141.7, 135.8, 135.6, 132.4, 131.9, 131.1, 128.9, 128.4, 128.2, 123.3, 123.2, 122.7, 119.5, 110.3, 74.7, 67.4, 63.7, 63.2, 62.8, 57.6, 13.8, 13.2.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3327, 2954, 2919, 2851, 2361, 2338, 1736, 1708, 1467.

HRMS (ESI) for $\text{C}_{30}\text{H}_{27}^{79}\text{BrNO}_7$, $[\text{M}+\text{H}]^+$ (592.0965) found: 592.0972.

HRMS (ESI) for $\text{C}_{30}\text{H}_{27}^{81}\text{BrNO}_7$, $[\text{M}+\text{H}]^+$ (594.0945) found: 594.0956.

Ethyl (2*S*,3*aR*,9*bR*)-2-(5-bromo-2-hydroxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetra hydro-4*H*-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3*a*(9*bH*)-carboxylate (9aa**).**



Following the TP-F, **9aa** was obtained from **2a** (50.0 mg, 0.2 mmol) and **1a** (86.0 mg, 1.2 equiv.) as a pale yellow solid (85.1 mg, 76%).

R_f = 0.200 (DCM:Hex = 3:1).

mp.: 96.3-97.3 °C.

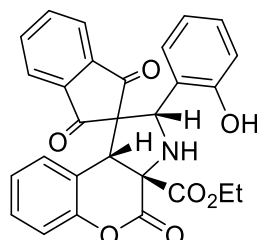
$[\alpha]_{\text{D}}^{24}$ = 347.0° (c = 0.50, CH_2Cl_2).

HPLC: 89% ee, Chiralpak IA column, n -hexane/EtOH = 80:20, flow rate = 0.70 mL/min, λ = 245 nm, t_{minor} = 23.61 min, t_{major} = 38.14 min.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 10.70 (s, 1H), 10.24 (s, 1H'), 7.99 (d, J = 7.5 Hz, 1H), 7.84-7.73 (m, 3H'+1H), 7.63 (t, J = 7.5 Hz, 1H), 7.50 (d, J = 7.5 Hz, 1H), 7.25-7.16 (m, 2H'+1H), 7.14 (d, J = 8.2 Hz, 1H'), 7.08 (d, J = 8.2 Hz, 1H), 6.94 (d, J = 8.8 Hz, 1H), 6.89-6.80 (m, 1H'+2H), 6.78 (d, J = 8.7 Hz, 1H'), 6.65 (d, J = 7.7 Hz, 1H'), 6.60-

6.51 (m, 2H), 6.40 (s, 1H'), 5.08 (d, $J = 6.8$ Hz, 1H), 4.94 (d, $J = 6.2$ Hz, 1H'), 4.66 (s, 1H'), 4.36 (s, 1H), 4.32-4.26 (m, 1H'+1H), 4.23 (q, $J = 7.1$ Hz, 2H+3H'), 1.15 (t, $J = 7.1$ Hz, 3H'), 1.10 (t, $J = 7.1$ Hz, 3H).

Ethyl (2*S*,3*aR*,9*bR*)-2-(2-hydroxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetra hydro-4*H*-spiro [chromeno[3,4-*b*]pyrrole-1,2'-indene]-3*a*(9*bH*)-carboxylate (9*ba*).



Following the TP-F, **9ba** was obtained from **2a** (50.0 mg, 0.2 mmol) and **1b** (67.0 mg, 1.2 equiv.) as a pale yellow solid (73.9 mg, 76%).

$R_f = 0.325$ (DCM:Hex:EA = 3:1:0.1).

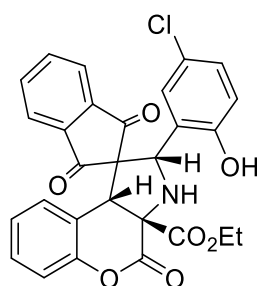
mp.: 100.1-101.1 °C.

$[\alpha]_D^{22} = 38.4^\circ$ ($c = 0.50$, CH₂Cl₂).

HPLC: 68% ee, Chiralpak IA column, *n*-hexane/EtOH = 80:20, flow rate = 0.70 mL/min, $\lambda = 245$ nm, $t_{\text{minor}} = 27.94$ min, $t_{\text{major}} = 50.71$ min.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 10.53 (s, 1H), 10.22 (s, 1H'), 7.94 (d, $J = 7.6$ Hz, 1H), 7.79-7.73 (m, 3H'), 7.70 (td, $J = 7.6, 1.1$ Hz, 1H'+1H), 7.59 (td, $J = 7.6, 1.1$ Hz, 1H), 7.48 (d, $J = 7.6$ Hz, 1H), 7.24 (td, $J = 7.6, 1.6$ Hz, 1H'), 7.21-7.15 (m, 1H), 7.14 (d, $J = 1.6$ Hz, 1H'), 7.11-7.05 (m, 1H'+1H), 6.90-6.80 (m, 2H'+3H), 6.68 (d, $J = 1.5$ Hz, 1H'), 6.65 (dd, $J = 8.2, 1.0$ Hz, 1H), 6.48 (dd, $J = 8.0, 1.5$ Hz, 1H), 6.44 (td, $J = 7.5, 1.1$ Hz, 1H'), 6.32 (td, $J = 7.5, 1.2$ Hz, 1H), 6.28 (dd, $J = 7.5, 1.2$ Hz, 1H'), 5.18 (d, $J = 5.8$ Hz, 1H), 5.01 (d, $J = 5.9$ Hz, 1H'), 4.71 (s, 1H'), 4.39 (s, 1H), 4.23 (q, $J = 7.1$ Hz, 3H'+3H), 1.16 (t, $J = 7.1$ Hz, 3H'), 1.10 (t, $J = 7.1$ Hz, 3H).

Ethyl (2*S*,3*aR*,9*bR*)-2-(5-chloro-2-hydroxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetra hydro-4*H*-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3*a*(9*bH*)-carboxylate (9*ca*).



Following the TP-F, **9ca** was obtained from **2a** (50.0 mg, 0.2 mmol) and **1c** (75.3 mg, 1.2 equiv.) as a pale yellow solid (54.8 mg, 46%).

$R_f = 0.200$ (DCM:Hex = 3:1).

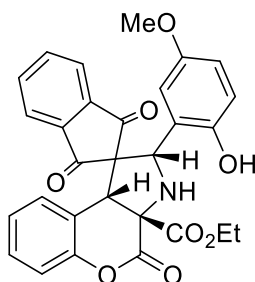
mp.: 92.8-93.8 °C.

$[\alpha]_D^{22} = 129.7^\circ$ ($c = 0.50$, CH_2Cl_2).

HPLC: 87% ee, Chiralpak IA column, n -hexane/EtOH = 80:20, flow rate = 0.70 mL/min, $\lambda = 245$ nm, $t_{\text{minor}} = 24.01$ min, $t_{\text{major}} = 39.06$ min.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 10.66 (s, 1H), 10.15 (s, 1H'), 7.99 (d, $J = 7.6$ Hz, 1H), 7.84-7.72 (m, 4H'+1H), 7.63 (t, $J = 7.6$ Hz, 1H), 7.51 (d, $J = 7.6$ Hz, 1H), 7.26-7.16 (m, 1H'+1H), 7.13 (d, $J = 8.1$ Hz, 1H'), 7.07 (d, $J = 8.1$ Hz, 1H), 7.04 (d, $J = 2.3$ Hz, 1H'), 6.87-6.79 (m, 2H'+3H), 6.65 (d, $J = 7.7$ Hz, 1H'), 6.60 (d, $J = 8.8$ Hz, 1H), 6.43 (d, $J = 2.2$ Hz, 1H), 6.31 (s, 1H'), 5.10 (d, $J = 6.6$ Hz, 1H), 4.95 (d, $J = 6.2$ Hz, 1H'), 4.65 (s, 1H'), 4.36 (s, 1H), 4.29 (d, $J = 7.1$ Hz, 1H), 4.23 (q, $J = 7.1$ Hz, 3H'+2H), 1.16 (t, $J = 7.1$ Hz, 3H'), 1.10 (t, $J = 7.1$ Hz, 3H).

Ethyl (2*S*,3*aR*,9*bR*)-2-(2-hydroxy-5-methoxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetra hydro-4*H*-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3*a*(9*bH*)-carboxylate (9ea**).**



Following the TP-F, **9ea** was obtained from **2a** (50.0 mg, 0.2 mmol) and **1e** (79.8 mg, 1.2 equiv.) as a pale yellow solid (88.1 mg, 86%).

$R_f = 0.125$ (DCM:Hex = 3:1).

mp.: 114.2-115.2 °C.

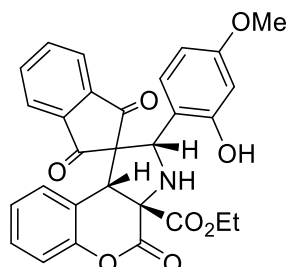
$[\alpha]_D^{22} = 76.7^\circ$ ($c = 0.50$, CH_2Cl_2).

HPLC: 82% ee, Chiralpak IA column, n -hexane/EtOH = 80:20, flow rate = 0.70 mL/min, $\lambda = 245$ nm, $t_{\text{minor}} = 43.20$ min, $t_{\text{major}} = 62.84$ min.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 7.95 (d, $J = 7.6$ Hz, 1H), 7.79-7.74 (m, 4H'), 7.71 (t, $J = 7.6$ Hz, 1H), 7.60 (t, $J = 7.6$ Hz, 1H), 7.51 (d, $J = 7.6$ Hz, 1H), 7.23 (d, $J = 8.1$ Hz, 1H'), 7.21-7.12 (m, 1H'+1H), 7.07 (d, $J = 8.2$ Hz, 1H), 6.87-6.79 (m, 2H+2H'), 6.71-6.65 (m, 2H'), 6.60 (d, $J = 8.9$ Hz, 1H), 6.47 (dd, $J = 8.9, 2.8$ Hz, 1H), 6.00 (d,

$J = 2.8$ Hz, 1H), 5.85 (s, 1H'), 5.12 (s, 1H), 4.95 (s, 1H'), 4.70 (s, 1H'), 4.37 (s, 1H), 4.23 (q, $J = 7.1$ Hz, 2H+2H'), 3.42 (s, 3H), 3.37 (s, 3H'), 1.16 (t, $J = 7.1$ Hz, 3H'), 1.11 (t, $J = 7.1$ Hz, 3H).

Ethyl (2*S*,3*aR*,9*bR*)-2-(2-hydroxy-4-methoxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetra hydro-4*H*-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3*a*(9*bH*)-carboxylate (9*fa*).



Following the TP-F, **9fa** was obtained from **2a** (50.0 mg, 0.2 mmol) and **1f** (76.5 mg, 1.2 equiv.) as a pale yellow solid (72.8 mg, 71%).

$R_f = 0.150$ (DCM:Hex = 3:1).

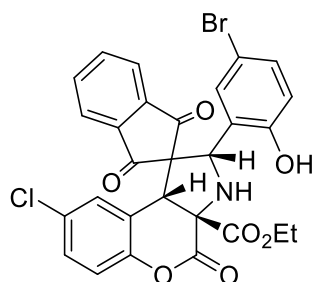
mp.: 119.4-120.4 °C.

$[\alpha]_D^{23} = 26.6^\circ$ (c = 0.50, CH₂Cl₂).

HPLC: 75% ee, Chiralpak IA column, *n*-hexane/EtOH = 80:20, flow rate = 0.70 mL/min, $\lambda = 245$ nm, $t_{\text{minor}} = 41.90$ min, $t_{\text{major}} = 89.65$ min.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 10.65 (s, 1H), 10.46 (s, 1H'), 7.94 (d, $J = 7.6$ Hz, 1H), 7.79-7.73 (m, 3H'), 7.70 (t, $J = 7.6$ Hz, 1H), 7.61 (t, $J = 7.6$ Hz, 1H), 7.53 (d, $J = 7.6$ Hz, 1H), 7.23 (d, $J = 8.4$ Hz, 1H'), 7.21-7.12 (m, 1H'+1H), 7.07 (d, $J = 8.2$ Hz, 1H), 6.91-6.78 (m, 1H'+2H), 6.71-6.64 (m, 2H'), 6.44 (d, $J = 2.5$ Hz, 1H'), 6.37 (d, $J = 8.6$ Hz, 1H), 6.21 (d, $J = 2.5$ Hz, 1H), 6.09 (d, $J = 8.4$ Hz, 1H'), 5.98 (dd, $J = 8.5, 2.5$ Hz, 1H'), 5.90 (dd, $J = 8.5, 2.5$ Hz, 1H), 5.14 (d, $J = 5.0$ Hz, 1H), 4.96 (d, $J = 5.7$ Hz, 1H'), 4.72 (s, 1H'), 4.36 (s, 1H), 4.23 (q, $J = 7.1$ Hz, 3H'+3H), 3.71 (s, 3H'), 3.58 (s, 3H), 1.17 (t, $J = 7.1$ Hz, 3H'), 1.10 (t, $J = 7.1$ Hz, 3H).

Ethyl(2*S*,3*aR*,9*bR*)-2-(5-bromo-2-hydroxyphenyl)-8-chloro-1',3',4-trioxo-1',2,3, 3'-tetrahydro-4*H*-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3*a*(9*bH*)-carboxylate (9*ab*).



Following the TP-F, **9ab** was obtained from **2b** (57.0 mg, 0.2 mmol) and **1a** (86.0 mg, 1.2

equiv.) as a white solid (79.7 mg, 67%).

$R_f = 0.438$ (Hex:DCM:EA = 3:1:1).

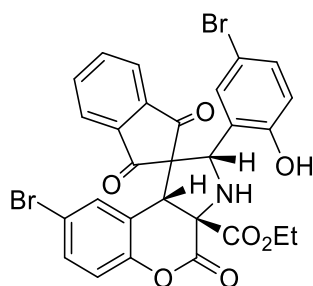
mp.: 110.7-111.7 °C.

$[\alpha]_D^{23} = 62.5^\circ$ (c = 0.50, CH₂Cl₂).

HPLC: 91% ee, Chiralpak IA column, *n*-hexane/EtOH = 80:20, flow rate = 0.70 mL/min, $\lambda = 245$ nm, $t_{\text{minor}} = 26.14$ min, $t_{\text{major}} = 31.91$ min.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 10.59 (s, 1H), 10.01 (s, 1H'), 8.02 (d, $J = 7.6$ Hz, 1H), 7.87-7.75 (m, 1H+4H'), 7.67 (t, $J = 7.6$ Hz, 1H), 7.52 (d, $J = 7.6$ Hz, 1H), 7.23 (dd, $J = 8.8, 2.4$ Hz, 1H'), 7.17 (dd, $J = 8.8, 2.4$ Hz, 1H+1H'), 7.09 (d, $J = 8.8$ Hz, 1H'), 7.04 (d, $J = 8.8$ Hz, 1H), 6.95 (dd, $J = 8.8, 2.3$ Hz, 1H), 6.83 (d, $J = 2.3$ Hz, 1H), 6.76 (d, $J = 8.8$ Hz, 1H'), 6.67 (d, $J = 2.2$ Hz, 1H'), 6.54 (d, $J = 8.8$ Hz, 1H), 6.52 (d, $J = 2.4$ Hz, 1H), 6.41 (s, 1H'), 5.05 (d, $J = 6.7$ Hz, 1H), 4.89 (d, $J = 6.1$ Hz, 1H'), 4.62 (s, 1H'), 4.36-4.17 (m, 4H+3H'), 1.19 (t, $J = 7.1$ Hz, 3H'), 1.14 (t, $J = 7.1$ Hz, 3H).

Ethyl (2*S*,3*aR*,9*bR*)-8-bromo-2-(5-bromo-2-hydroxyphenyl)-1',3',4-trioxo-1',2,3,3'-tetrahydro-4*H*-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3*a*(9*bH*)-carboxylate (9ac)



Following the TP-F, **9ac** was obtained from **2c** (65.9 mg, 0.2 mmol) and **1a** (86.0 mg, 1.2 equiv.) as a white solid (97.4 mg, 76%).

$R_f = 0.450$ (Hex:DCM:EA = 3:1:1).

mp.: 109.5-110.5 °C.

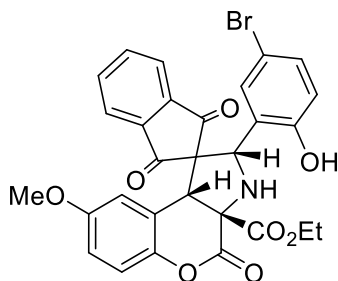
$[\alpha]_D^{23} = 37.8^\circ$ (c = 0.50, CH₂Cl₂).

HPLC: 89% ee, Chiralpak IA column, *n*-hexane/EtOH = 80:20, flow rate = 0.70 mL/min, $\lambda = 245$ nm, $t_{\text{minor}} = 26.71$ min, $t_{\text{major}} = 35.00$ min.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 8.02 (d, $J = 7.6$ Hz, 1H), 7.89-7.74 (m, 1H+4H'), 7.67 (t, $J = 7.6$ Hz, 1H), 7.53 (d, $J = 7.6$ Hz, 1H), 7.36 (dd, $J = 8.7, 2.0$ Hz, 1H'), 7.32 (dd, $J = 8.7, 2.0$ Hz, 1H), 7.18 (dd, $J = 8.7, 2.2$ Hz, 1H'), 7.04 (d, $J = 8.8$ Hz, 1H'), 7.02-6.90 (m, 3H), 6.81 (d, $J = 1.7$ Hz, 1H'), 6.76 (d, $J = 8.7$ Hz, 1H'), 6.55 (d, $J = 8.8$ Hz, 1H), 6.52 (d, $J = 2.0$ Hz, 1H), 6.38 (s, 1H'), 5.05 (s, 1H), 4.88 (s, 1H'), 4.62 (s, 1H'), 4.34-4.17

(m, 3H+2H'), 1.21 (t, $J = 7.1$ Hz, 3H'), 1.14 (t, $J = 7.1$ Hz, 3H).

Ethyl (2*S*,3*aR*,9*bR*)-2-(5-bromo-2-hydroxyphenyl)-8-methoxy-1',3',4-trioxo-1',2,3,3'-tetrahydro-4*H*-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3*a*(9*bH*)-carboxylate (9ae).



Following the TP-F, **9ae** was obtained from **2e** (56.1 mg, 0.2 mmol) and **1a** (86.0 mg, 1.2 equiv.) as a pale yellow solid (100.2 mg, 84%).

$R_f = 0.150$ (DCM:Hex = 3:1).

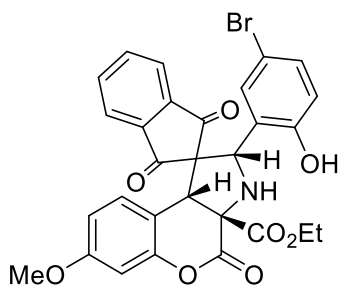
mp.: 235.5-236.5 °C.

$[\alpha]_D^{23} = 16.1^\circ$ (c = 0.50, CH₂Cl₂).

HPLC: 96% ee, Chiralpak IB column, *n*-hexane/EtOH = 85:15, flow rate = 1.0 mL/min, $\lambda = 245$ nm, $t_{\text{minor}} = 37.96$ min, $t_{\text{major}} = 41.87$ min.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 8.0 (d, $J = 7.5$ Hz, 1H), 7.88-7.74 (m, 4H'+1H), 7.65 (t, $J = 7.5$ Hz, 1H), 7.52 (d, $J = 7.5$ Hz, 1H), 7.18 (dd, $J = 8.7, 2.2$ Hz, 1H'), 7.07 (d, $J = 9.0$ Hz, 1H'), 7.01 (d, $J = 9.0$ Hz, 1H), 6.94 (dd, $J = 8.8, 2.2$ Hz, 1H), 6.81-6.69 (m, 2H'+1H), 6.56, (d, $J = 9.0$ Hz, 1H), 6.54 (d, $J = 2.1$ Hz, 1H), 6.40 (s, 1H'), 6.30 (d, $J = 2.7$ Hz, 1H), 6.13 (d, $J = 2.7$ Hz, 1H'), 5.07 (s, 1H), 4.92 (s, 1H'), 4.61 (s, 1H'), 4.31 (s, 1H), 4.24 (q, $J = 6.8$ Hz, 2H'+2H), 3.55 (s, 3H), 3.50 (s, 3H'), 1.19 (t, $J = 7.2$ Hz, 3H'), 1.13 (t, $J = 7.2$ Hz, 3H).

Ethyl (2*S*,3*aR*,9*bR*)-2-(5-bromo-2-hydroxyphenyl)-7-methoxy-1',3',4-trioxo-1',2,3,3'-tetrahydro-4*H*-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3*a*(9*bH*)-carboxylate (9af).



Following the TP-F, **9af** was obtained from **2f** (56.1 mg, 0.2 mmol) and **1a** (86.0 mg, 1.2 equiv.) as a pale yellow solid (70.2 mg, 59%).

$R_f = 0.175$ (DCM:Hex = 4:1).

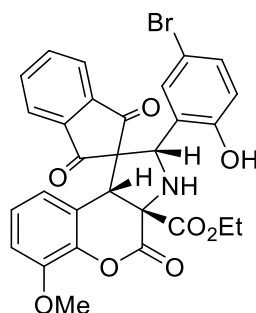
mp.: 131.1-132.1 °C.

$[\alpha]_D^{23} = 111.3^\circ$ ($c = 0.50$, CH_2Cl_2).

HPLC: 69% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 80:20, flow rate = 0.70 mL/min, $\lambda = 245$ nm, $t_{\text{minor}} = 32.37$ min, $t_{\text{major}} = 44.34$ min.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 10.73 (s, 1H), 10.29 (s, 1H'), 7.99 (d, $J = 7.6$ Hz, 1H), 7.83-7.73 (m, 4H'+1H), 7.64 (t, $J = 7.6$ Hz, 1H), 7.53 (d, $J = 7.6$ Hz, 1H), 7.18 (d, $J = 8.4$ Hz, 1H'), 6.94 (dd, $J = 8.8, 2.0$ Hz, 1H), 6.78 (d, $J = 8.7$ Hz, 1H'), 6.70 (d, $J = 8.5$ Hz, 1H), 6.67 (d, $J = 2.0$ Hz, 1H'), 6.64-6.50 (m, 1H'+3H), 6.42-6.34 (m, 1H+2H'), 5.07 (d, $J = 6.9$ Hz, 1H), 4.94 (d, $J = 5.0$ Hz, 1H'), 4.60 (s, 1H'), 4.30 (s, 1H), 4.24 (q, $J = 7.1$ Hz, 3H+3H'), 3.73 (s, 3H'), 3.71 (s, 3H), 1.19 (t, $J = 7.1$ Hz, 3H'), 1.13 (t, $J = 7.1$ Hz, 3H).

Ethyl (2*S*,3*aR*,9*bR*)-2-(5-bromo-2-hydroxyphenyl)-6-methoxy-1',3',4-trioxo-1',2,3,3'-tetrahydro-4*H*-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3*a*(9*bH*)-carboxylate (9ag).



Following the TP-F, **9ag** was obtained from **2g** (56.1 mg, 0.2 mmol) and **1a** (86.0 mg, 1.2 equiv.) as a pale yellow solid (90.0 mg, 76%).

$R_f = 0.225$ (Hex:DCM:EA = 3:1:1).

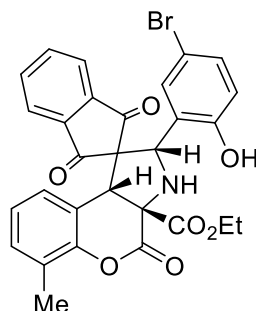
mp.: 129.4-130.4 °C.

$[\alpha]_D^{23} = -115.2^\circ$ ($c = 0.50$, CH_2Cl_2).

HPLC: 58% ee, Chiralpak IA column, *n*-hexane/EtOH = 80:20, flow rate = 0.70 mL/min, $\lambda = 245$ nm, $t_{\text{minor}} = 32.68$ min, $t_{\text{major}} = 51.94$ min.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: (mixture of rotamers) 10.67 (s, 1H), 10.29 (s, 1H'), 7.99 (d, $J = 7.6$ Hz, 1H), 7.76 (t, $J = 7.54$ Hz, 1H+4H'), 7.63 (t, $J = 7.6$ Hz, 1H), 7.52 (d, $J = 7.6$ Hz, 1H), 7.18 (d, $J = 8.1$ Hz, 1H'), 6.94 (dd, $J = 8.8, 2.3$ Hz, 1H), 6.82-6.73 (m, 2H+3H'), 6.60-6.52 (m, 2H), 6.41-6.34 (m, 1H+1H'), 6.21 (d, $J = 8.2$ Hz, 1H'), 5.06 (d, $J = 6.9$ Hz, 1H), 4.94 (d, $J = 6.4$ Hz, 1H'), 4.66 (s, 1H'), 4.36 (s, 1H), 4.31-4.20 (m, 3H+3H'), 3.90 (s, 3H'), 3.85 (s, 3H), 1.18 (t, $J = 7.1$ Hz, 3H'), 1.13 (t, $J = 7.1$ Hz, 3H).

Ethyl (2*S*,3*aR*,9*bR*)-2-(5-bromo-2-hydroxyphenyl)-6-methyl-1',3',4-trioxo-1',2,3,3'-tetrahydro-4*H*-spiro[chromeno[3,4-*b*]pyrrole-1,2'-indene]-3*a*(9*bH*)-carboxylate (9*ah*).



Following the TP-F, **9ah** was obtained from **2h** (52.9 mg, 0.2 mmol) and **1a** (86.0 mg, 1.2 equiv.) as a pale yellow solid (39.2 mg, 34%).

R_f = 0.413 (Hex:DCM:EA = 3:1:1).

mp.: 95.0-96.0 °C.

$[\alpha]_D^{23}$ = 174.6° (c = 0.50, CH₂Cl₂).

HPLC: 95% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 85:15, flow rate = 0.70 mL/min, λ = 245 nm, t_{minor} = 29.80 min, t_{major} = 35.08 min.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 10.77 (s, 1H), 10.22 (s, 1H'), 7.99 (d, J = 7.6 Hz, 1H), 7.85-7.71 (m, 1H+4H'), 7.63 (t, J = 7.6 Hz, 1H), 7.49 (d, J = 7.6 Hz, 1H), 7.17 (d, J = 8.6 Hz, 1H'), 7.06 (d, J = 7.6 Hz, 1H'), 7.03 (d, J = 7.4 Hz, 1H), 6.93 (dd, J = 8.7, 1.9 Hz, 1H), 6.82-6.68 (m, 1H+2H'), 6.65 (d, J = 7.6 Hz, 1H), 6.61-6.40 (m, 2H+2H'), 5.10 (d, J = 6.6 Hz, 1H), 4.95 (d, J = 6.1 Hz, 1H'), 4.63 (s, 1H'), 4.37 (s, 1H), 4.35-4.11 (m, 3H+3H'), 2.36 (s, 3H'), 2.31 (s, 3H), 1.16 (t, J = 7.1 Hz, 3H'), 1.10 (t, J = 7.1 Hz, 3H).

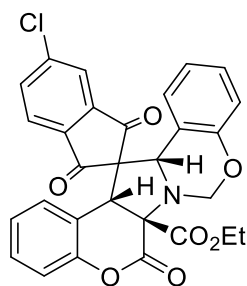
¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: (mixture of rotamers) 198.8, 197.8, 196.9, 196.1, 168.7, 167.2, 166.0, 164.8, 156.6, 156.5, 149.7, 149.0, 143.0, 142.5, 142.2, 141.3, 136.5, 136.1, 132.6, 132.3, 131.7, 131.4, 129.9, 129.7, 126.8, 126.7, 125.2, 124.6, 123.9, 123.7, 123.5, 123.3, 123.1, 122.2, 120.2, 119.6, 118.5, 115.9, 115.2, 110.6, 110.0, 71.0, 69.6, 68.4, 68.1, 67.6, 67.1, 63.9, 63.5, 50.3, 49.4, 15.9, 15.8, 13.62, 13.58.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 3316, 2926, 1767, 1742, 1707, 1592, 1482, 1255, 1183.

HRMS (ESI) for C₂₉H₂₃⁷⁹BrNO₇, [M+H]⁺ (576.0652) found: 576.0657.

HRMS (ESI) for C₂₉H₂₃⁸¹BrNO₇, [M+H]⁺ (578.0632) found: 578.0641

Ethyl (6*aR*,13*bS*,14*aR*)-5'-chloro-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*,13*bH*-spiro[benzo[e]chromeno[4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6*a*(14*aH*)-carboxylate (11*ca*).



Following the TP-D, **11ca** was obtained from **9ca** (51.7 mg, 0.1 mmol) as a yellow solid (46.6 mg, 88%); $R_f = 0.375$ (EA:Hex = 1:3).

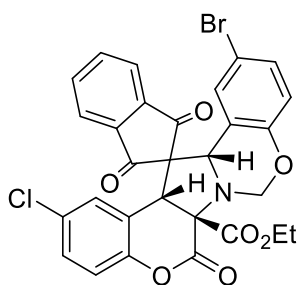
mp.: 211.4-212.4 °C.

$[\alpha]_D^{24} = 204.5^\circ$ ($c = 0.50$, CH_2Cl_2).

HPLC: 84% ee, Chiralpak IA column, n -hexane/EtOH = 80:20, flow rate = 1.0 mL/min, $\lambda = 248$ nm, $t_{\text{minor}} = 13.53$ min, $t_{\text{major}} = 20.74$ min.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 8.09 (d, $J = 7.6$ Hz, 1H), 7.84 (t, $J = 7.6$ Hz, 1H), 7.70 (t, $J = 7.6$ Hz, 1H), 7.45 (d, $J = 7.6$ Hz, 1H), 7.14 (td, $J = 7.5, 1.9$ Hz, 1H), 7.01 (d, $J = 8.2$ Hz, 1H), 6.89 (dd, $J = 8.8, 2.0$ Hz, 1H), 6.86-6.76 (m, 3H), 6.06 (d, $J = 2.2$ Hz, 1H), 5.81 (d, $J = 10.6$ Hz, 1H), 5.38 (s, 1H), 5.05 (d, $J = 10.5$ Hz, 1H), 4.53 (s, 1H), 4.26-4.10 (m, 2H), 1.09 (t, $J = 7.1$ Hz, 3H).

Ethyl (6a*R*,13b*S*,14a*R*)-12-bromo-2-chloro-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*, 13b*H*-spiro[benzo[*e*]chromeno[4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6a(14a*H*)-carboxylate (11ab**).**



Following the TP-D, **11ab** was obtained from **9ab** (59.8 mg, 0.1 mmol) as a white solid (48.1 mg, 79%).

$R_f = 0.313$ (Hex:EA = 4:1).

mp.: 118.3-119.3 °C.

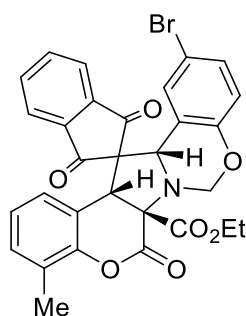
$[\alpha]_D^{25} = 80.2^\circ$ ($c = 0.50$, CH_2Cl_2).

HPLC: 93% ee, Chiralpak IA column, n -hexane/EtOH = 90:10, flow rate = 1.0 mL/min, $\lambda = 248$ nm, $t_{\text{minor}} = 27.86$ min, $t_{\text{major}} = 35.69$ min.

^1H NMR (400 MHz, CDCl_3 , 25 °C) δ /ppm: 8.14 (d, $J = 7.6$ Hz, 1H), 7.89 (t, $J = 7.6$ Hz, 1H),

7.75 (t, $J = 7.6$ Hz, 1H), 7.47 (d, $J = 7.6$ Hz, 1H), 7.12 (dd, $J = 8.8, 2.2$ Hz, 1H), 7.02 (dd, $J = 8.8, 2.1$ Hz, 1H), 6.97 (d, $J = 8.8$ Hz, 1H), 6.84 (d, $J = 2.2$ Hz, 1H), 6.76 (d, $J = 8.8$ Hz, 1H), 6.18 (s, 1H), 5.80 (d, $J = 10.6$ Hz, 1H), 5.34 (s, 1H), 5.01 (d, $J = 10.6$ Hz, 1H), 4.49 (s, 1H), 4.27-4.16 (m, 2H), 1.13 (t, $J = 7.1$ Hz, 3H).

Ethyl (6a*R*,13b*S*,14a*R*)-12-bromo-4-methyl-1',3',6-trioxo-1',3'-dihydro-6*H*,8*H*, 13b*H*-spiro[benzo[*e*]chromeno[4',3':4,5]pyrrolo[1,2-*c*][1,3]oxazine-14,2'-indene]-6a(14a*H*)-carboxylate (11a*h*).



Following the TP-D, **11a*h*** was obtained from **9a*h*** (57.8 mg, 0.1 mmol) as a pale yellow solid (35.3 mg, 60%).

$R_f = 0.263$ (Hex:EA = 4:1).

mp.: 201.6-202.6 °C.

$[\alpha]_D^{24} = 462.6^\circ$ ($c = 0.50$, CH₂Cl₂).

HPLC: 94% ee, Chiralpak IA column, *n*-hexane/EtOH = 90:10, flow rate = 1.0 mL/min, $\lambda = 248$ nm, $t_{\text{minor}} = 24.54$ min, $t_{\text{major}} = 33.35$ min.

¹H NMR (400 MHz, CDCl₃, 25 °C) δ /ppm: 8.09 (d, $J = 7.6$ Hz, 1H), 7.83 (t, $J = 7.6$ Hz, 1H), 7.70 (t, $J = 7.6$ Hz, 1H), 7.45 (d, $J = 7.6$ Hz, 1H), 7.01 (dd, $J = 8.8, 2.1$ Hz, 1H), 6.97 (d, $J = 7.0$ Hz, 1H), 6.75 (d, $J = 8.8$ Hz, 1H), 6.72-6.63 (m, 2H), 6.19 (d, $J = 1.8$ Hz, 1H), 5.83 (d, $J = 10.5$ Hz, 1H), 5.36 (s, 1H), 5.03 (d, $J = 10.5$ Hz, 1H), 4.53 (s, 1H), 4.25-4.11 (m, 2H), 2.27 (s, 3H), 1.10 (t, $J = 7.1$ Hz, 3H).

¹³C NMR (100 MHz, CDCl₃, 25 °C) δ /ppm: 198.4, 196.5, 169.0, 163.6, 155.5, 149.1, 143.0, 142.5, 136.7, 136.1, 131.4, 131.2, 128.0, 126.8, 124.7, 123.8, 123.4, 123.3, 121.5, 120.5, 115.3, 112.1, 76.5, 68.5, 68.4, 63.8, 63.1, 51.3, 16.0, 13.7.

IR (KBr) $\tilde{\nu}$ (cm⁻¹): 2922, 1769, 1743, 1708, 1592, 1471, 1242, 1212, 1159.

HRMS (ESI) for C₃₀H₂₂⁷⁹BrNO₇, [M+H]⁺ (588.0652) found: 588.0659.

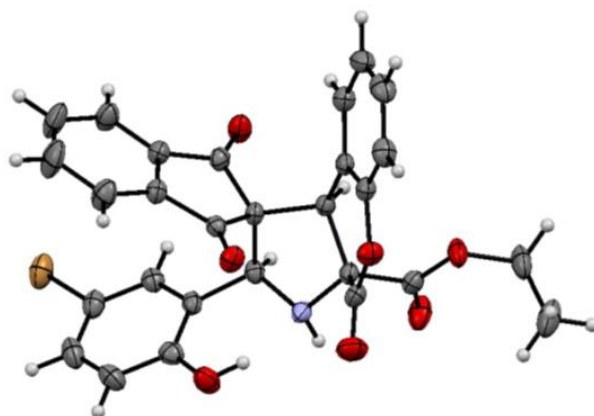
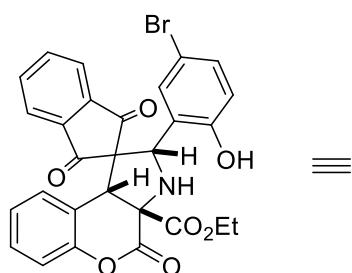
HRMS (ESI) for C₃₀H₂₂⁸¹BrNO₇, [M+H]⁺ (590.0632) found: 590.0643.

13. References

- (1) L. Tian, G.-Q. Xu, Y.-H. Li, Y.-M. Liang, P.-F. Xu, *Chem. Commun.* 2014, **50**, 2428.
- (2) S.-M. Yang, Y.-L. Tsai, G. M. Reddy, L. Möehlmann, W. Lin, *J. Org. Chem.* 2017, **82**, 9182
- (3) G.-H. Chang, C.-Y. Wang, G. M. Reddy, Y.-L. Tsai, W. Lin, *J. Org. Chem.* 2016, **81**, 10071.

14. X-ray crystallographic data for selected compounds

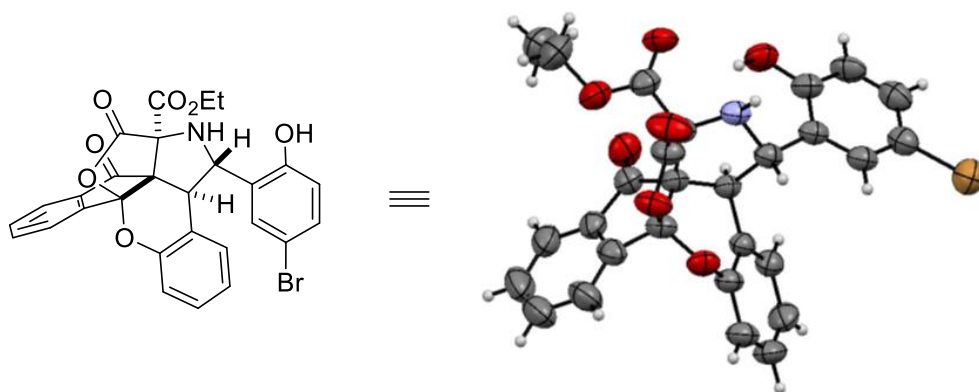
a) **3aa** (CCDC no. 1841899): The thermal ellipsoid drawn at 30% probability level.



Empirical formula	C ₂₈ H ₂₀ Br N O ₇	
Formula weight	562.36	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 9.0255(3) Å	α = 80.012(2)°.
	b = 10.5951(3) Å	β = 77.0370(10)°.
	c = 13.5639(4) Å	γ = 72.6250(10)°.
Volume	1198.40(6) Å ³	
Z	2	
Density (calculated)	1.558 Mg/m ³	
Absorption coefficient	1.766 mm ⁻¹	
F(000)	572	
Crystal size	0.28 x 0.25 x 0.06 mm ³	
Theta range for data collection	1.55 to 25.09°.	

Index ranges	-10<=h<=10, -12<=k<=12, -16<=l<=15
Reflections collected	10500
Independent reflections	4238 [R(int) = 0.0230]
Completeness to theta = 25.09°	99.2 %
Absorption correction	multi-scan
Max. and min. transmission	0.9014 and 0.6376
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4238 / 0 / 334
Goodness-of-fit on F ²	1.082
Final R indices [I>2sigma(I)]	R1 = 0.0366, wR2 = 0.1112
R indices (all data)	R1 = 0.0470, wR2 = 0.1261
Largest diff. peak and hole	0.445 and -0.463 e.Å ⁻³

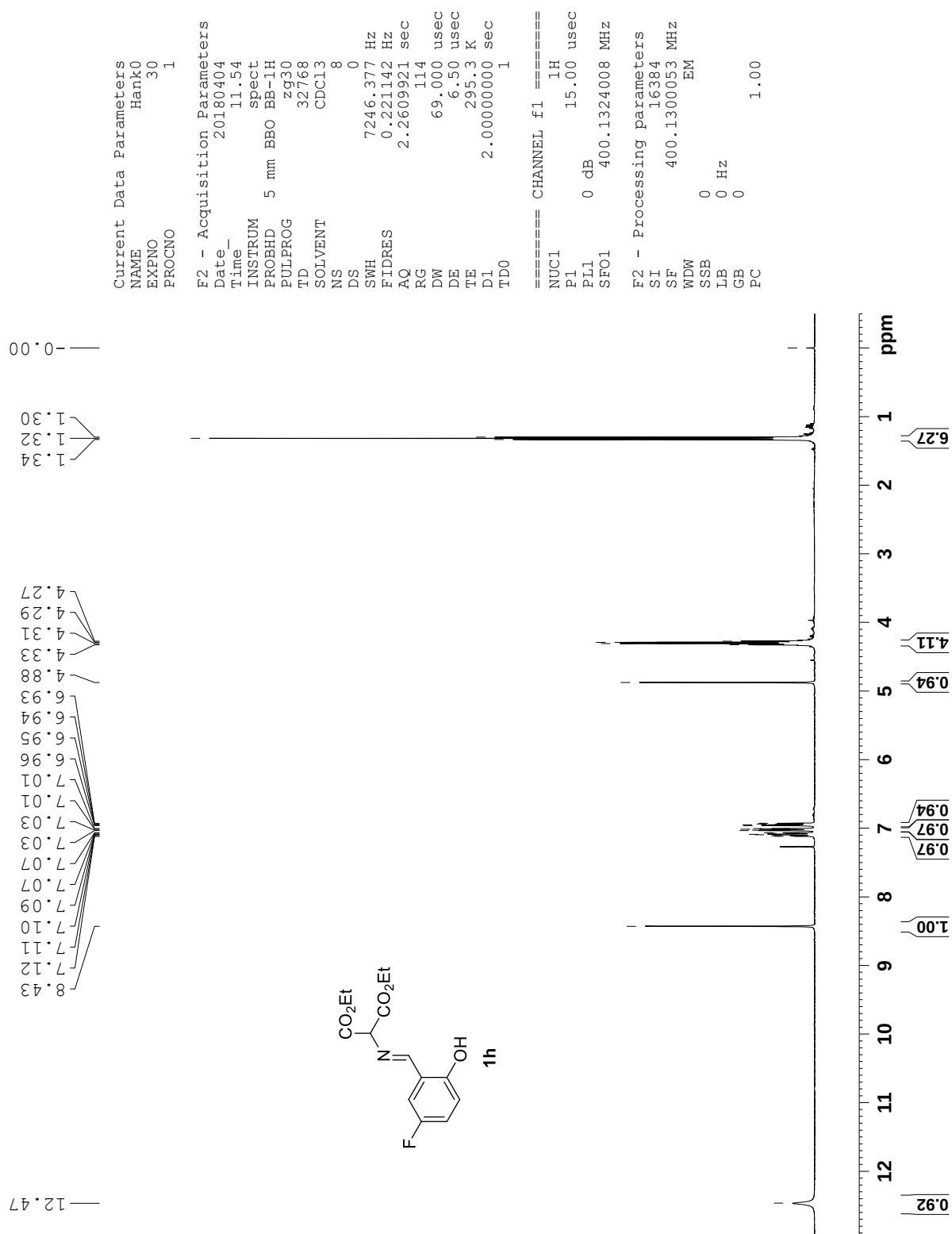
b) **4aa** (CCDC no. 1551249): The thermal ellipsoid drawn at 30% probability level.

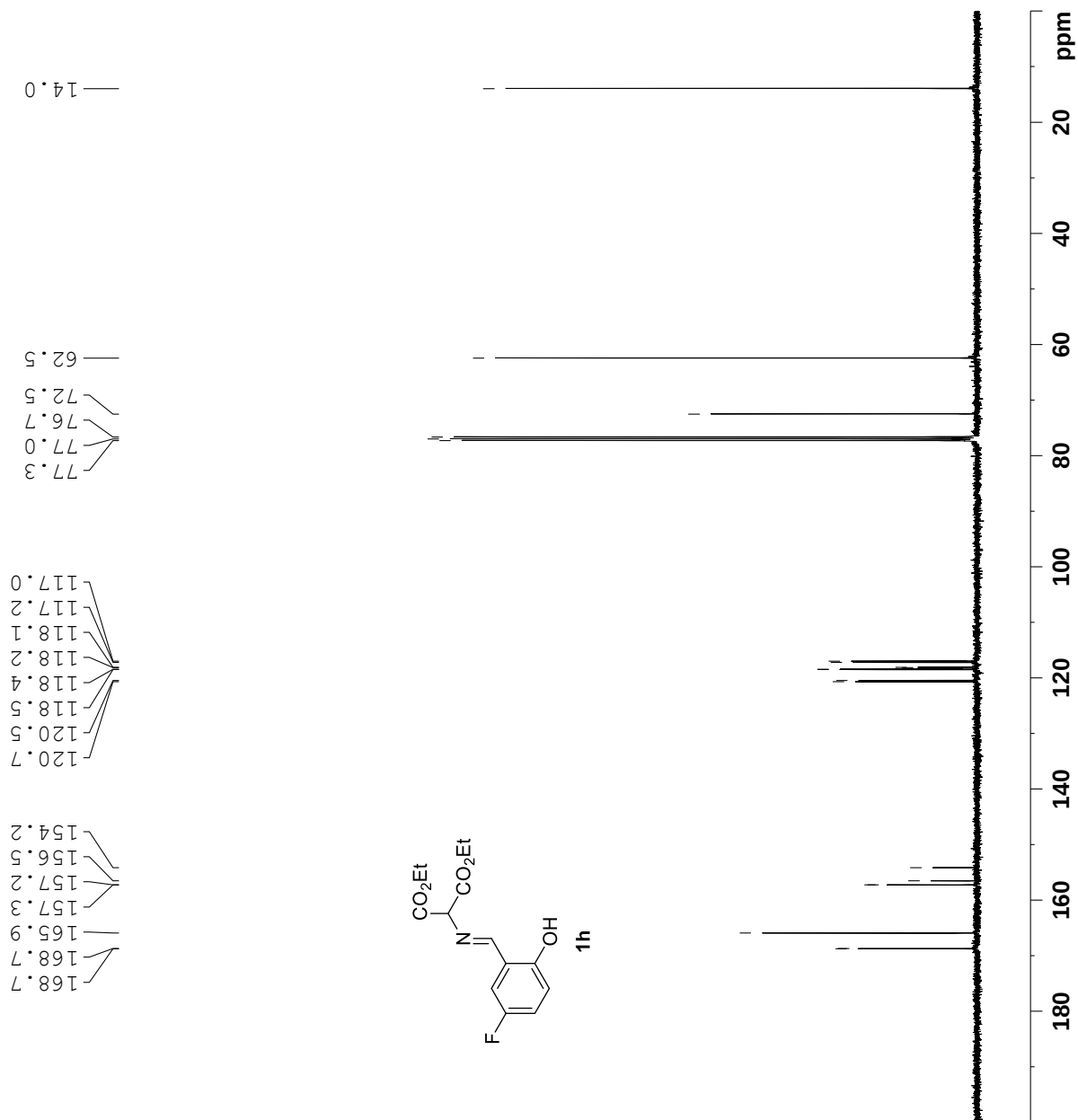


Empirical formula	C ₂₈ H ₂₀ Br N O ₇
Formula weight	562.36
Temperature	295(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P -1
Unit cell dimensions	$a = 9.4700(5) \text{ Å}$ $b = 10.6706(6) \text{ Å}$ $c = 13.3664(8) \text{ Å}$
Volume	1213.24(12) Å ³
Z	2
Density (calculated)	1.539 Mg/m ³
Absorption coefficient	1.744 mm ⁻¹

Z	2
Density (calculated)	1.500 Mg/m ³
Absorption coefficient	1.620 mm ⁻¹
F(000)	604
Crystal size	0.20 x 0.07 x 0.03 mm ³
Theta range for data collection	2.24 to 25.07°.
Index ranges	-11<=h<=11, -14<=k<=14, -15<=l<=15
Reflections collected	25308
Independent reflections	4630 [R(int) = 0.0518]
Completeness to theta = 25.07°	99.5 %
Absorption correction	multi-scan
Max. and min. transmission	0.9530 and 0.7376
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4630 / 1 / 349
Goodness-of-fit on F ²	1.024
Final R indices [I>2sigma(I)]	R1 = 0.0526, wR2 = 0.1166
R indices (all data)	R1 = 0.0780, wR2 = 0.1285
Largest diff. peak and hole	0.620 and -0.853 e.Å ⁻³

15. ¹H NMR and ¹³C NMR spectra for all new compounds





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Current Data Parameters
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PROCNO    1

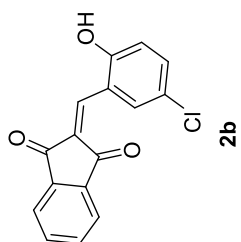
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SFO1       100.6233325 MHz

===== CHANNEL f2 =====
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PCPD2      90.00 usec
PL2        0 dB
PL12       15.00 dB
PL13       20.00 dB
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F2 - Processing parameters
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7.02
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7.47
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7.45
7.10
7.08



2.05

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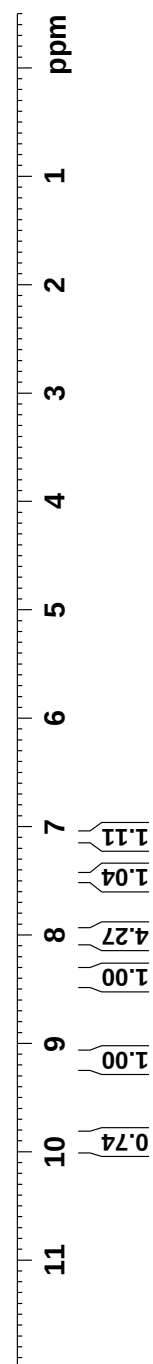
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DW         69.000 usec
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TE         297.1 K
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PL1        1.80 dB
SFO1       400.1324008 MHz

F2 - Processing parameters
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Current Data Parameters
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 PROCNO 1

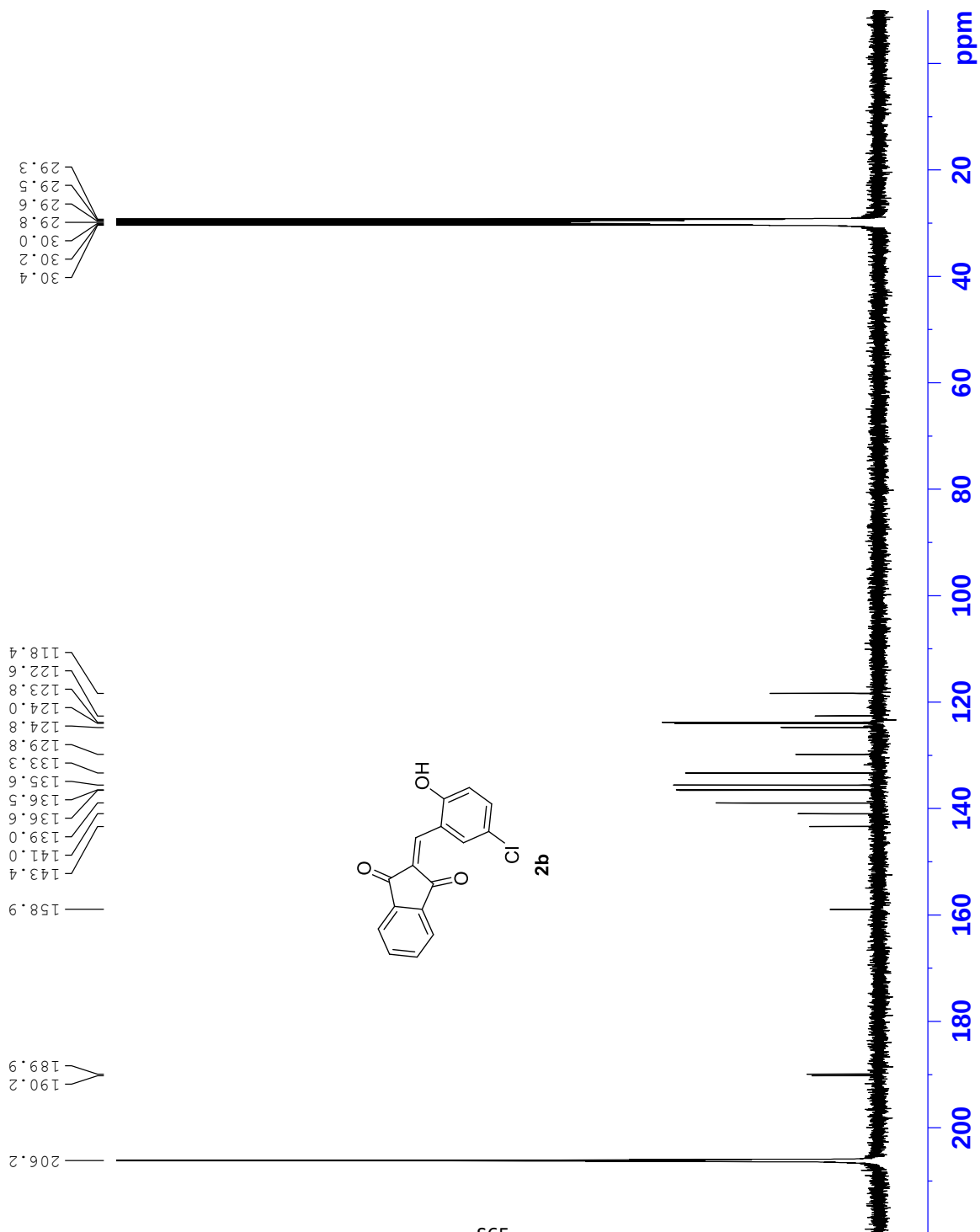
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 AQ 0.6815744 sec
 RG 8192
 DW 20.800 usec
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 TE 295.4 K
 D1 2.00000000 sec
 D11 0.03000000 sec
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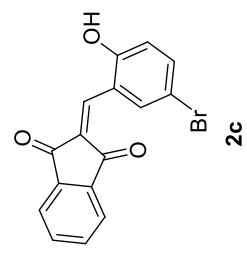
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 PL13 20.00 dB
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F2 - Processing parameters
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 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00



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7.58
7.58
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2.05



2c

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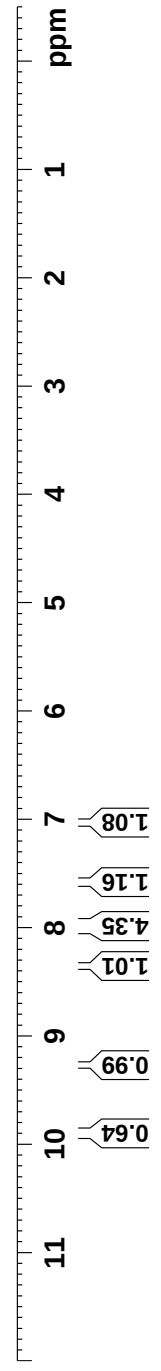
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DS         0
SWH        7246.377 Hz
FIDRES     0.221142 Hz
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RG         456.1
DW         69.000 usec
DE         6.50 usec
TE         297.1 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.40 usec
PL1        1.80 dB
SFO1       400.1324008 MHz

F2 - Processing parameters
SI         16384
SF         400.1300068 MHz
WDW        EM
SSB        0
LB         0 Hz
GB         0
PC         1.00

```



```

Current Data Parameters
NAME          liu050
EXPNO         3
PROCNO        1

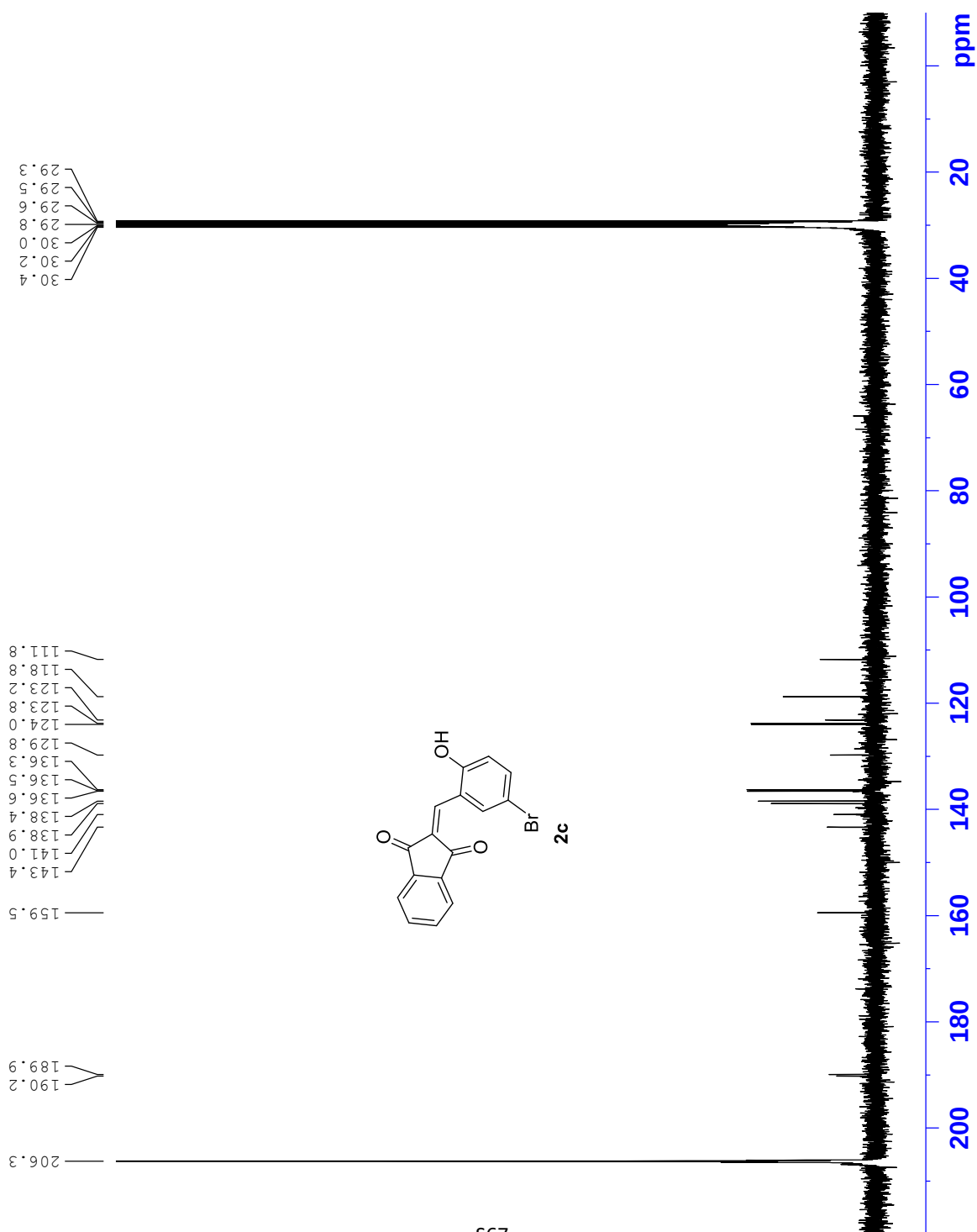
F2 - Acquisition Parameters
Date_         20170518
Time_         12.20
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            32768
SOLVENT       Acetone
NS            1509
DS            0
SWH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG            11585.2
DE            20.800 usec
TE            294.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.00 usec
PL1           7.00 dB
SFO1          100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG[2]     waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           1.80 dB
PL12          17.00 dB
PL13          20.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI            32768
SF            100.6126795 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.00

```

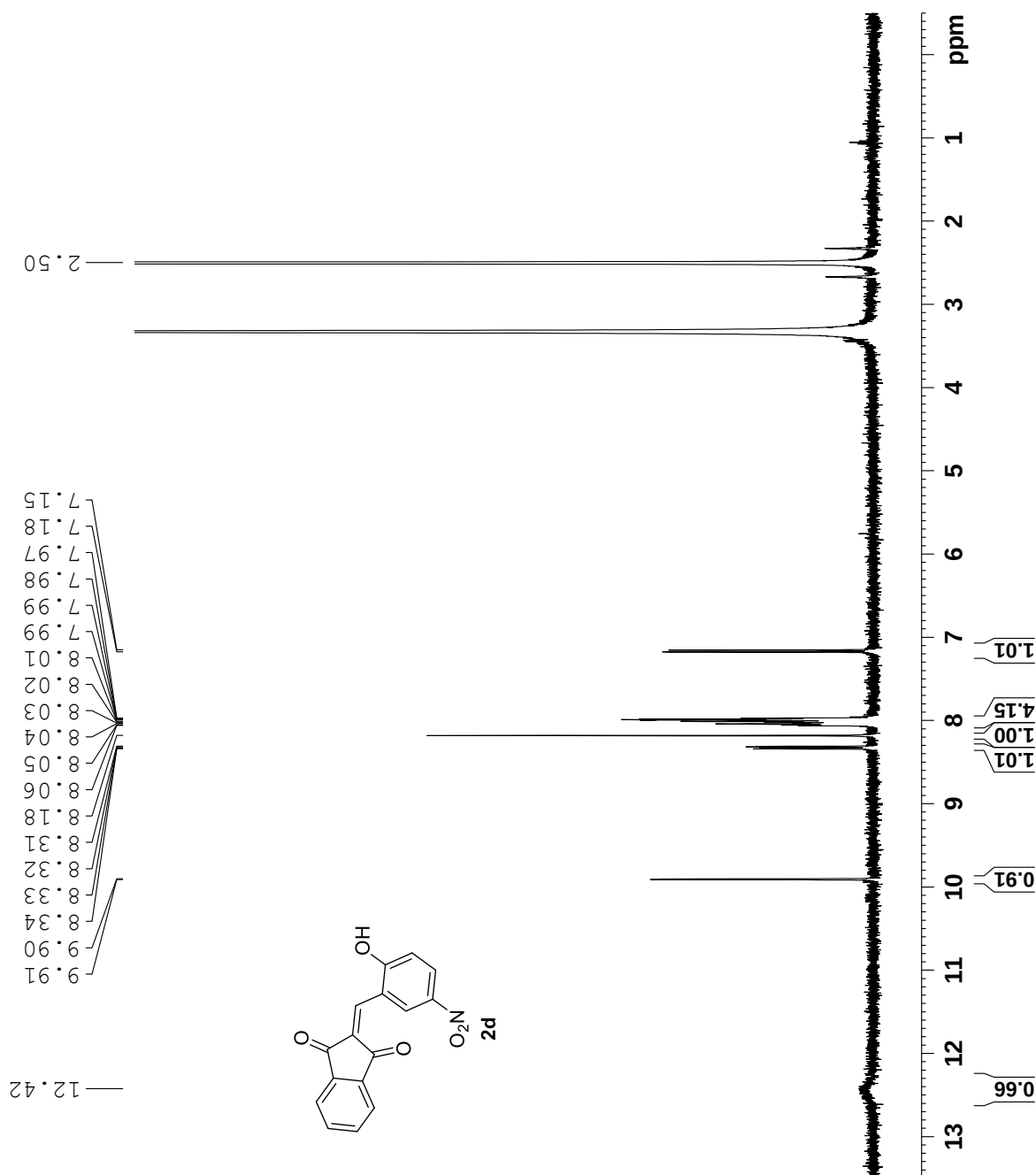


Current Data Parameters
NAME B06
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180618
Time_ 11.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 198.09
DW 69.333 usec
DE 10.50 usec
TE 297.9 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1324008 MHz
NUC1 ¹H
P1 12.90 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1300033 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



Current Data Parameters
NAME 12
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters

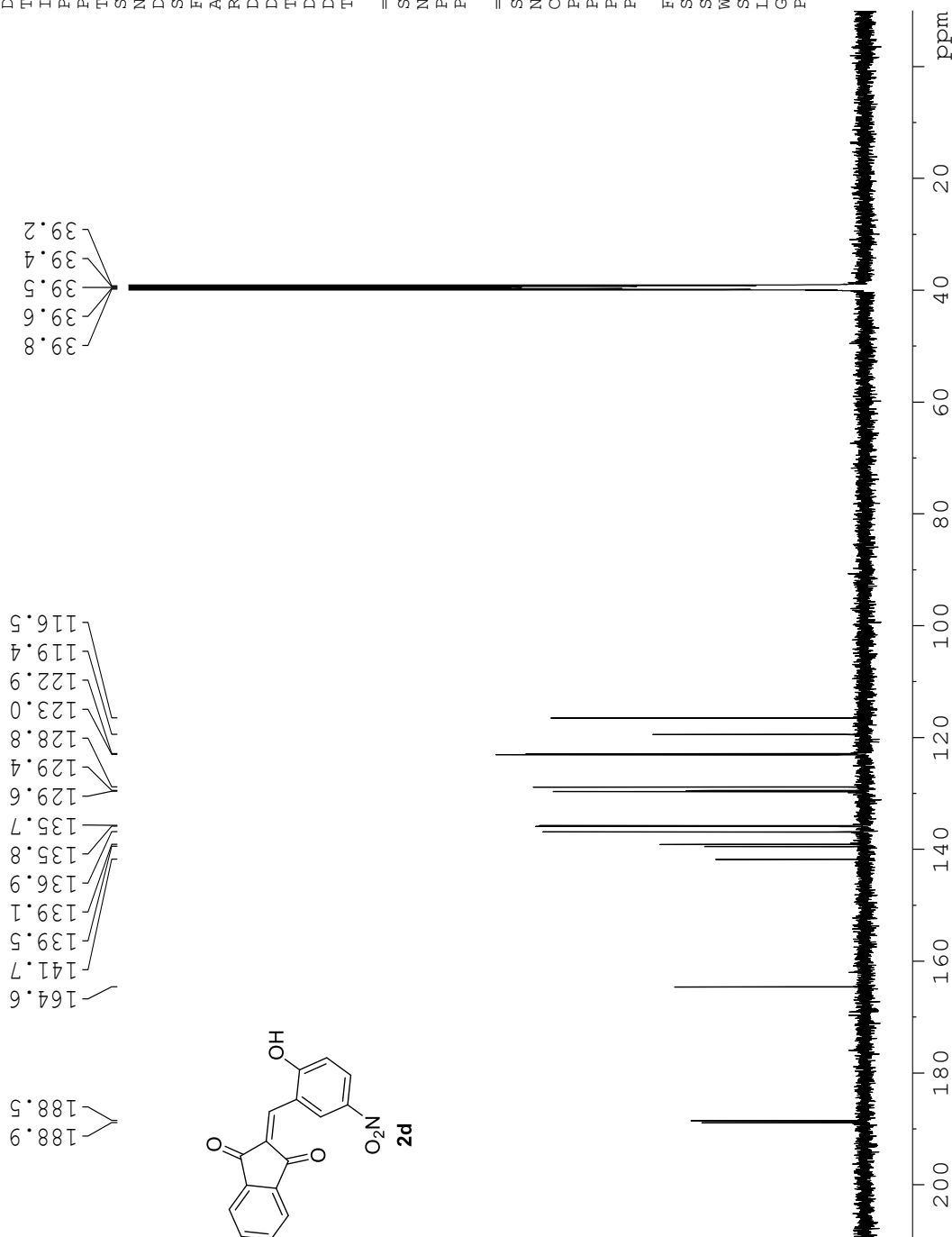
Date_ 20180611
Time 12.07
INSTRUM spect
PROBHD 5 mm PATBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1200
DS 0
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 2050
DW 13.867 usec
DE 6.50 usec
TE 323.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 150.9287115 MHz
NUC1 13C
P1 10.80 usec
PLW1 50.0000000 W

===== CHANNEL f2 =====
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2] waltz64
PCPD2 70.00 usec
PLW2 30.00000000 W
PLW12 1.03470004 W
PLW13 0.50700003 W

F2 - Processing parameters

SI 32768
SF 150.9129726 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



```

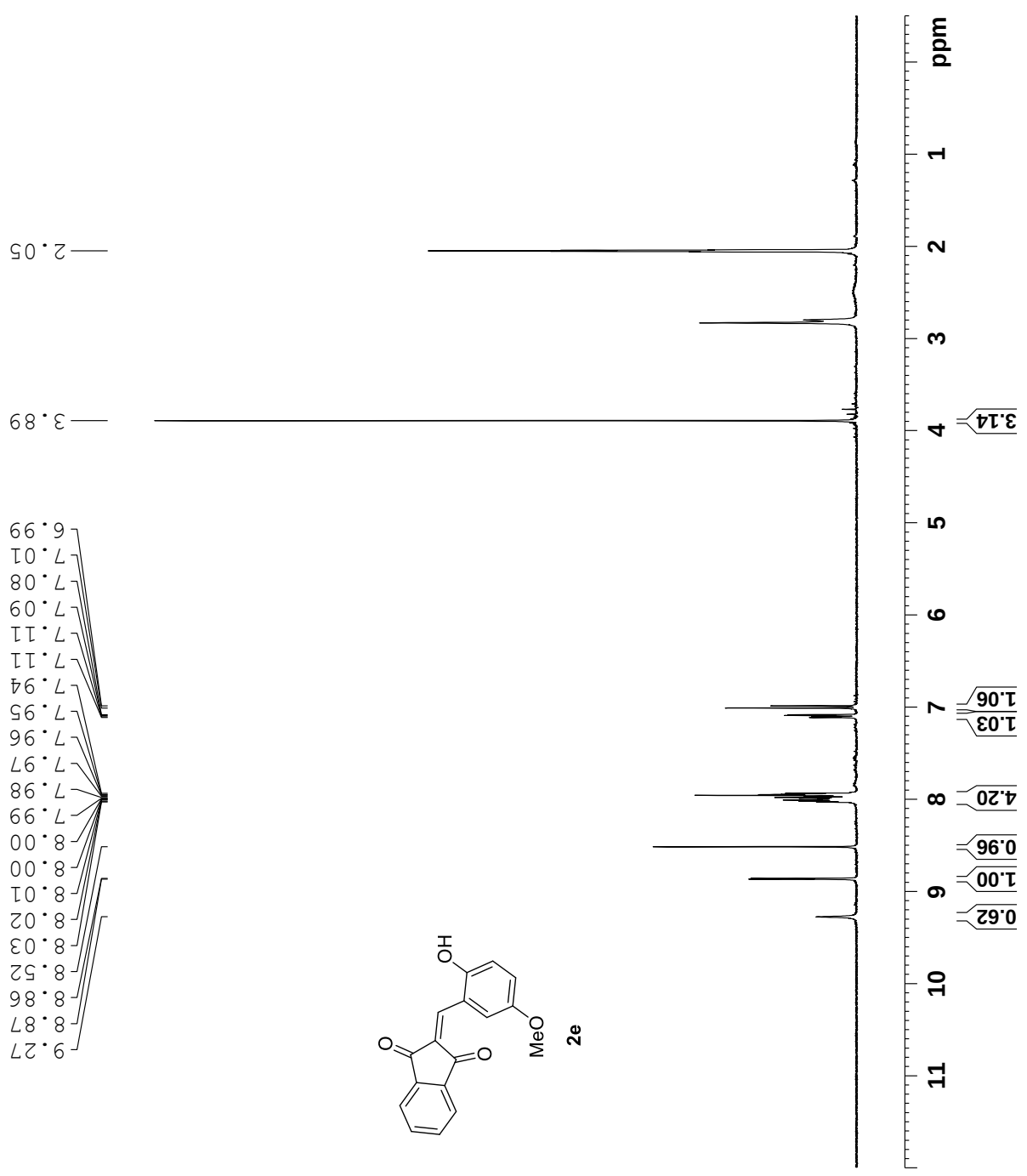
Current Data Parameters
NAME      liu048
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20170511
Time      15.17
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD         32768
SOLVENT   Acetone
NS         16
DS         0
SWH        7246.377 Hz
FIDRES     0.221142 Hz
AQ         2.2609921 sec
RG         456.1
DW         69.000 usec
DE         6.50 usec
TE         297.1 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.40 usec
PL1        1.80 dB
SFO1       400.1324008 MHz

F2 - Processing parameters
SI          16384
SF          400.1300069 MHz
WDW         EM
SSB         0
LB          0 Hz
GB          0
PC          1.00

```



Current Data Parameters
NAME liu048
EXPNO 3
PROCNO 1

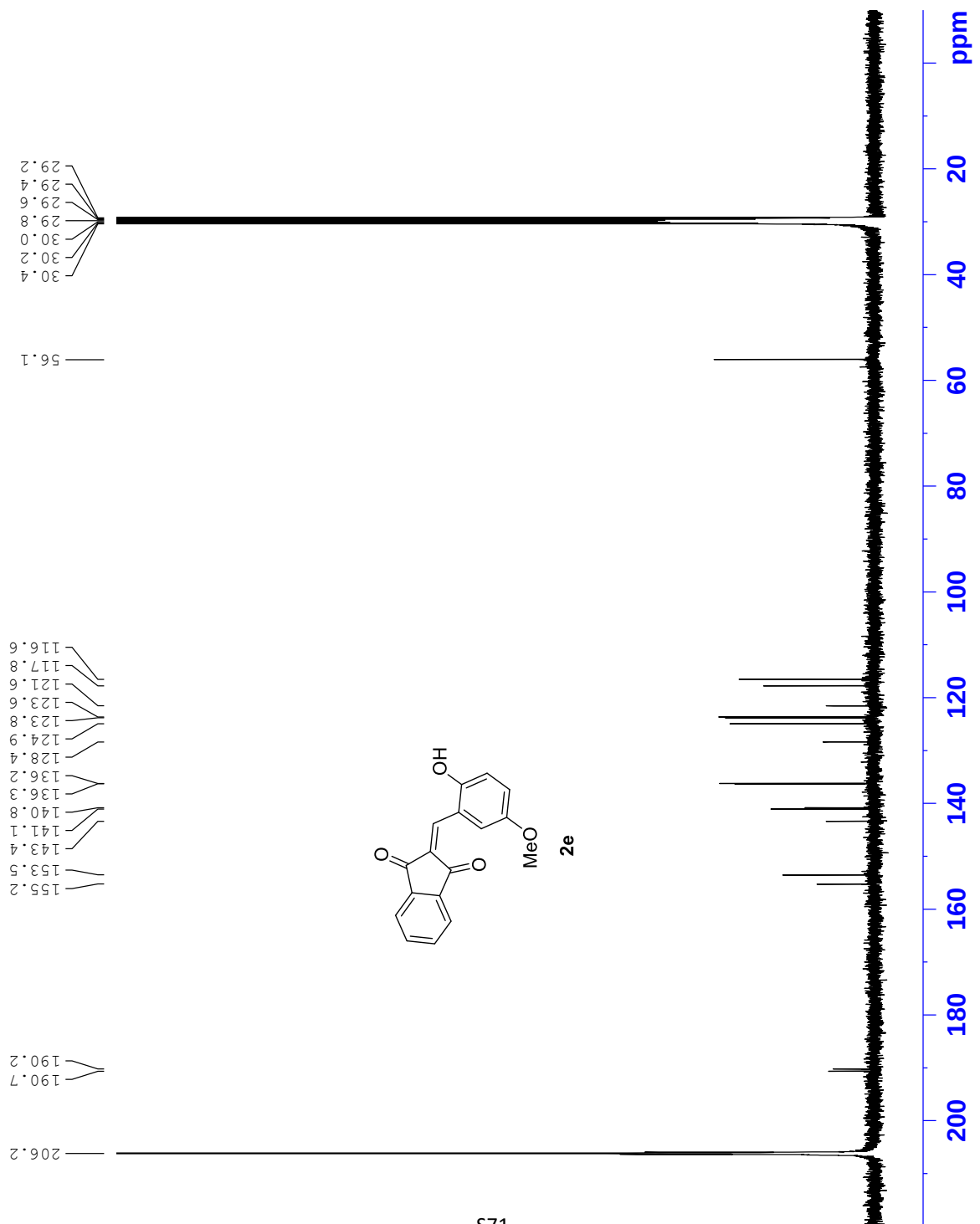
F2 - Acquisition Parameters

Date_ 20170528
Time_ 10.17
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT Acetone
NS 11248
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 9195.2
DW 20.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6126785 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



```

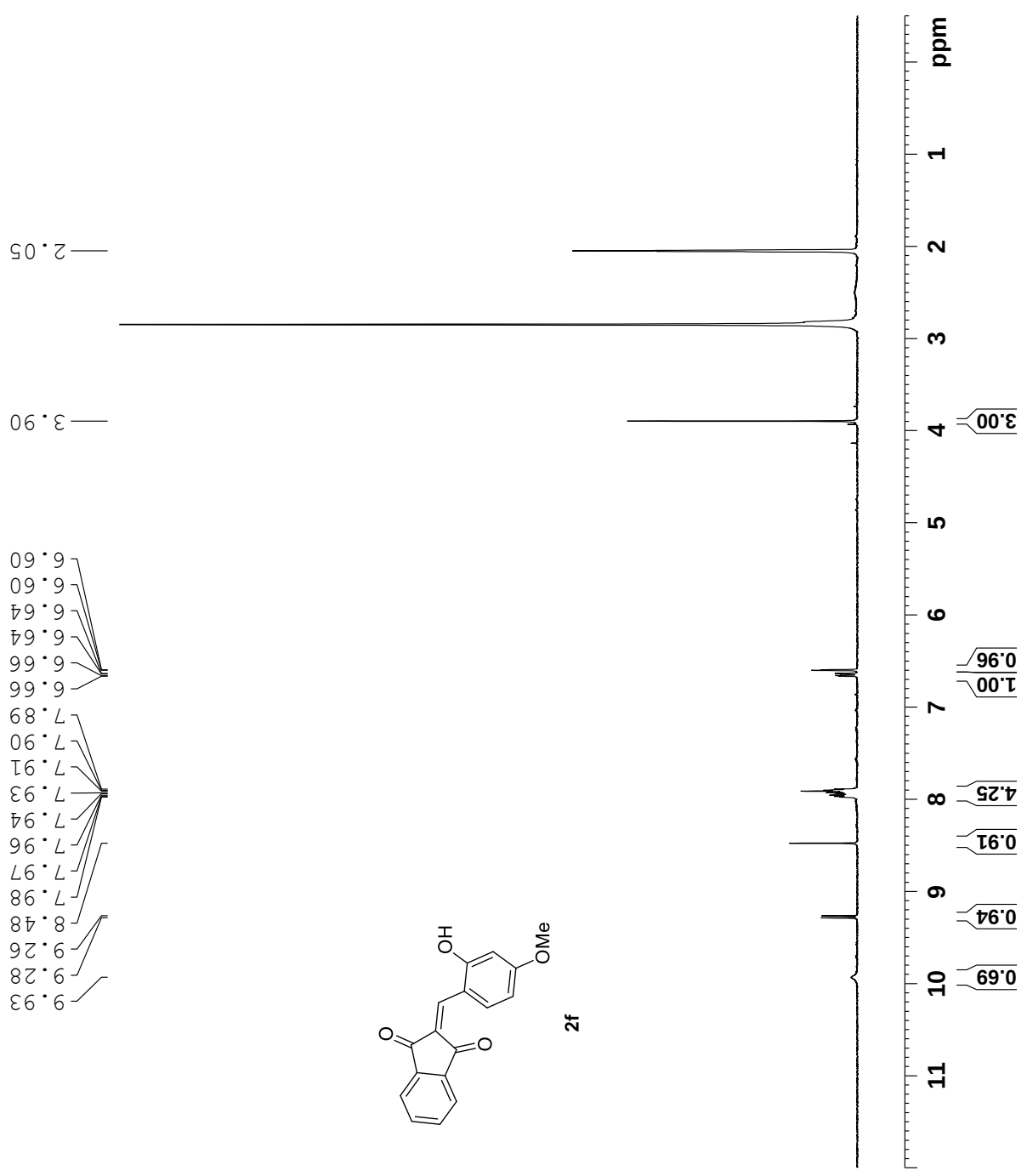
Current Data Parameters
NAME      liu047
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20170511
Time      17.12
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD         32768
SOLVENT   Acetone
NS         16
DS         0
SWH        7246.377 Hz
FIDRES     0.221142 Hz
AQ         2.260921 sec
RG         362
DW         69.000 usec
DE         6.50 usec
TE         297.1 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.40 usec
PL1        1.80 dB
SFO1       400.1324008 MHz

F2 - Processing parameters
SI         16384
SF         400.1300069 MHz
WDW        EM
SSB        0
LB         0 Hz
GB         0
PC         1.00

```



Current Data Parameters
NAME liu047
EXPNO 3
PROCNO 1

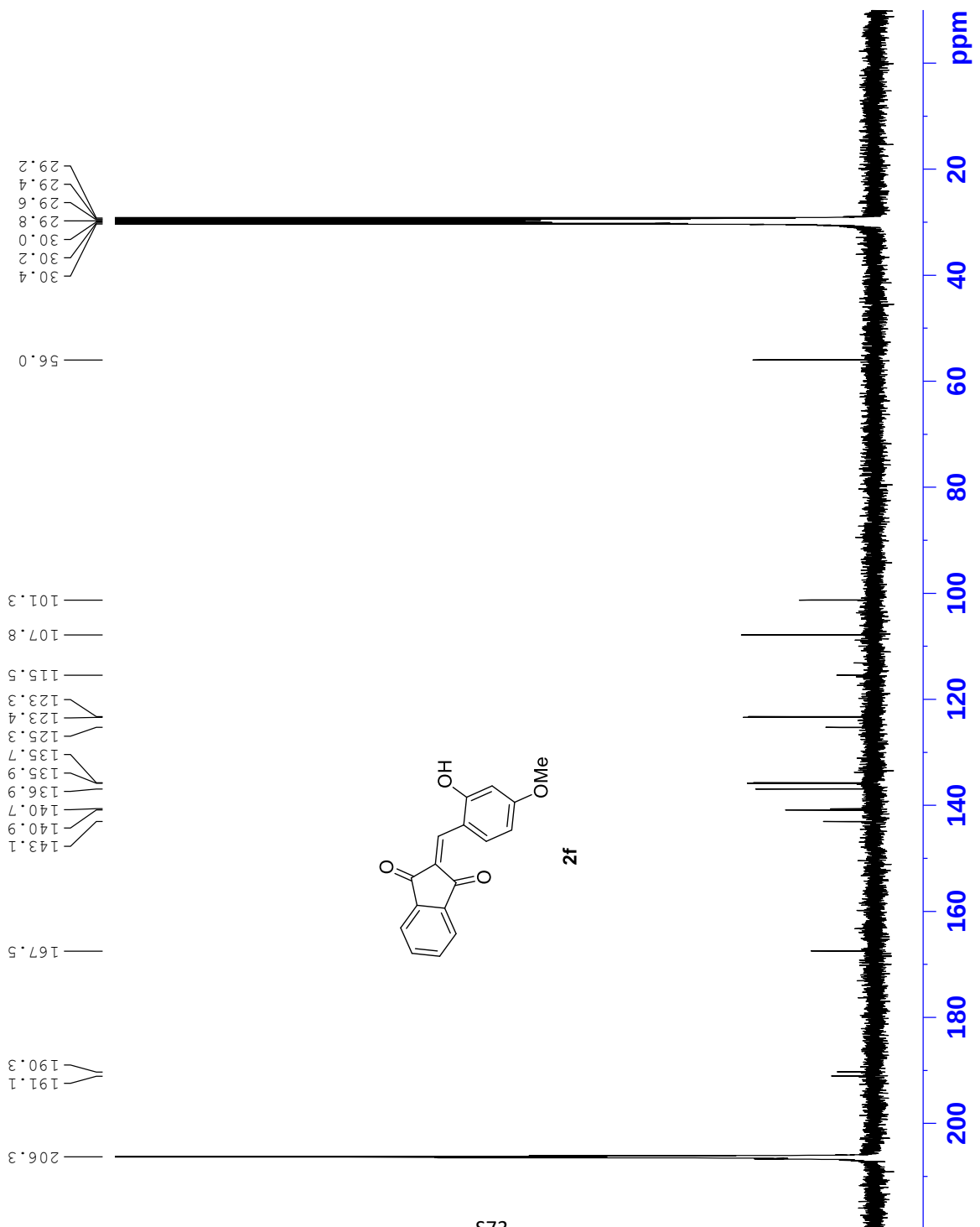
F2 - Acquisition Parameters

Date_ 20170528
Time_ 23.11
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT Acetone
NS 15173
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 11585.2
DW 20.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

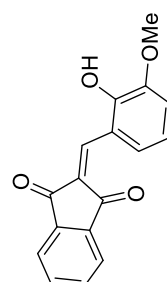
F2 - Processing parameters
SI 32768
SF 100.6126842 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



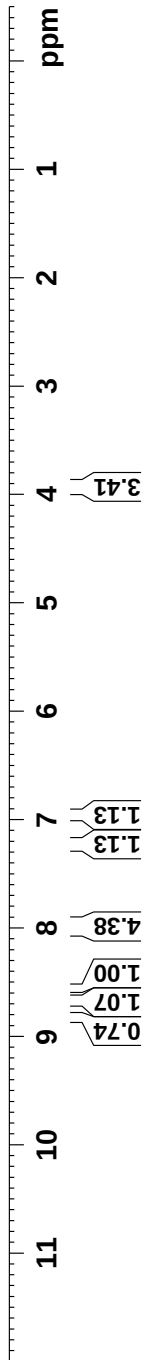
8.81
8.80
8.67
8.65
8.55
8.02
8.01
8.00
7.98
7.97
7.96
7.95
7.94
7.23
7.21
7.97
6.95
6.93

3.92

2.05



2g

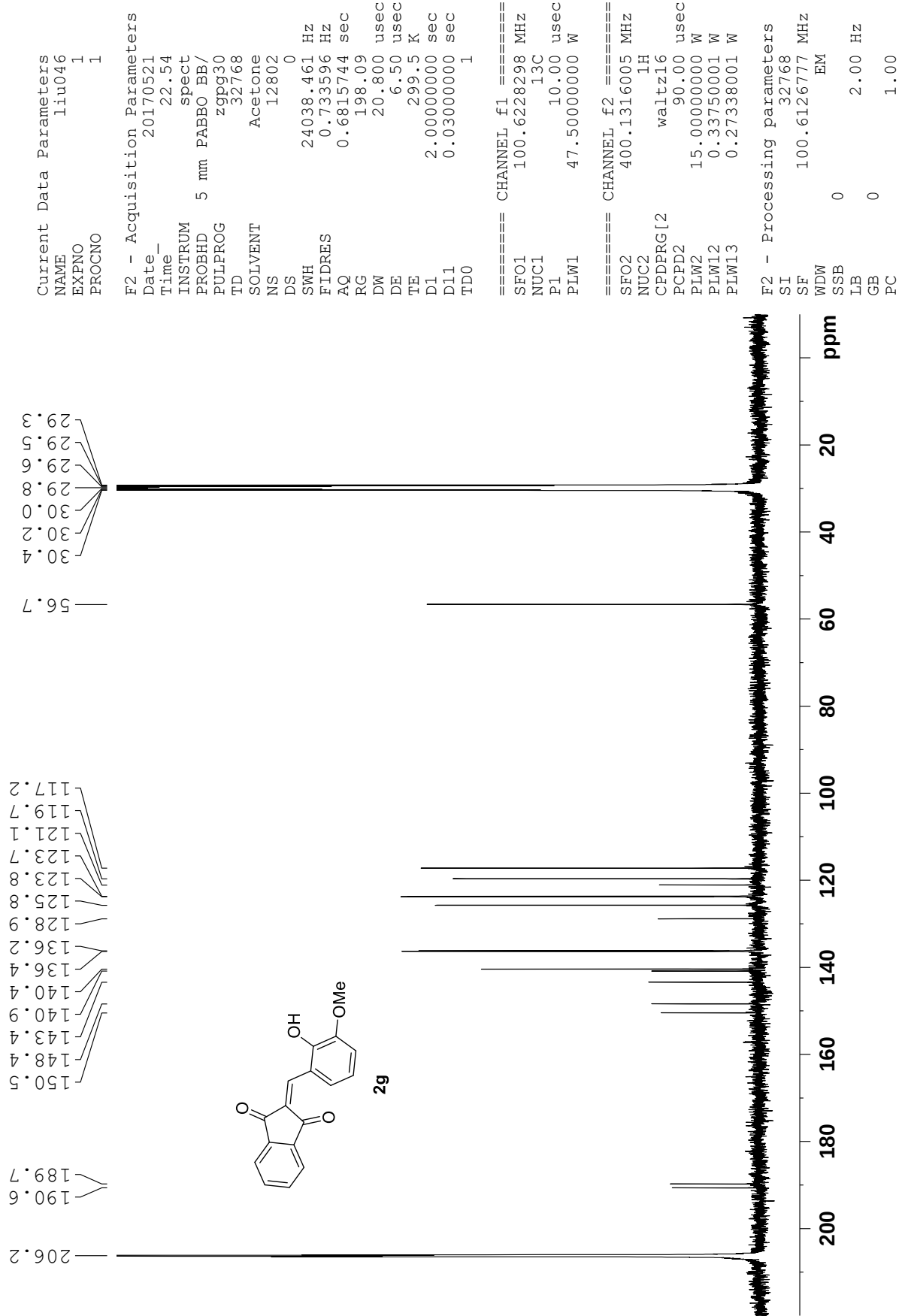


Current Data Parameters
NAME liu046
EXPNO 1
PROCNO 1

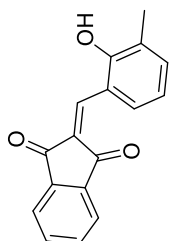
F2 - Acquisition Parameters
Date_ 20170511
Time_ 15.24
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT Acetone
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2609921 sec
RG 456.1
DW 69.000 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.40 usec
PL1 1.80 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300068 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



9.80
9.805
9.803
9.802
9.89
9.87
9.85
9.83
9.82
9.78
9.76
9.75
9.73
9.69
9.63
9.91



2h

0.00

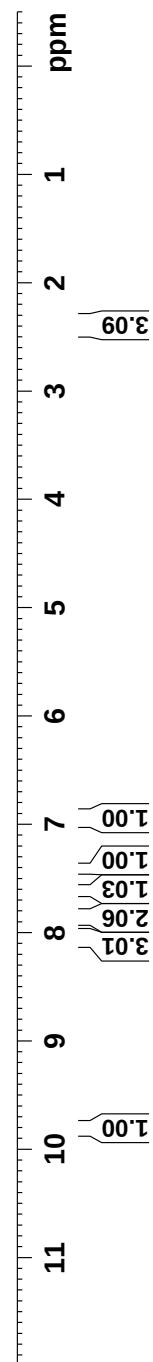
2.36

Current Data Parameters
NAME vicky157
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170520
Time_ 16.11
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.260921 sec
RG 362
DW 69.000 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.40 usec
PL1 1.80 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300086 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



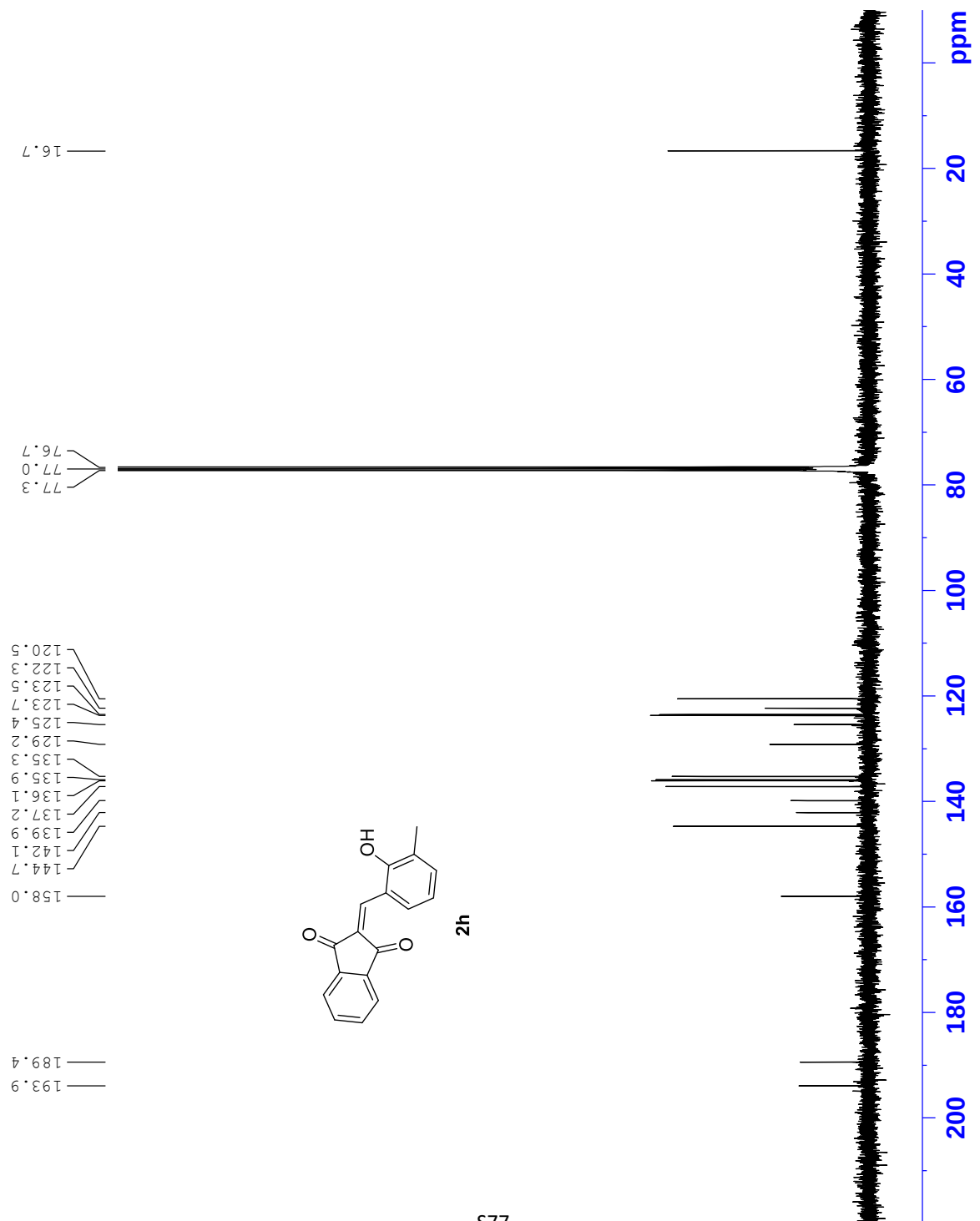
Current Data Parameters
 NAME vicky157
 EXPNO 8
 PROCNO 1

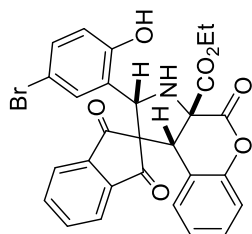
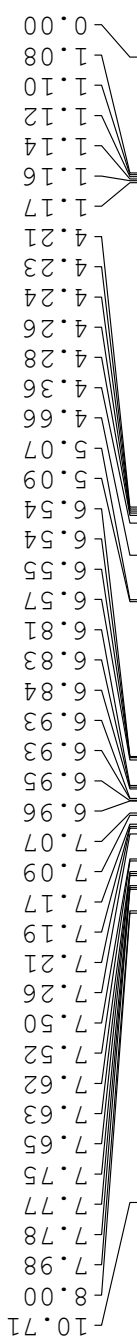
F2 - Acquisition Parameters
 Date_ 20170529
 Time_ 11.49
 INSTRUM spect
 PROBD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 4377
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6815744 sec
 RG 9195.2
 DW 20.800 usec
 DE 6.50 usec
 TE 298.7 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PL1 7.00 dB
 SFO1 100.6233325 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 1.80 dB
 PL12 17.00 dB
 PL13 20.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127713 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00





3aa (mixture of rotamers)

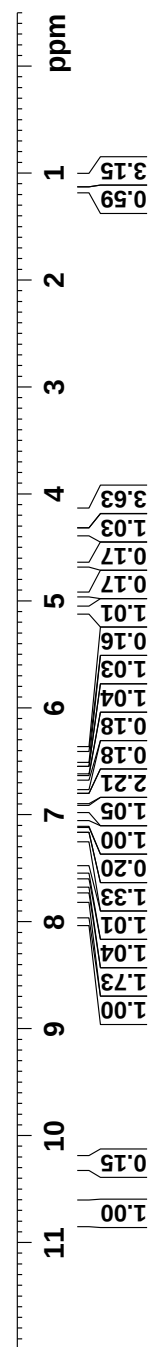
```

Current Data Parameters
NAME      3aa
EXPNO     3
PROCNO    1

F2 - Acquisition Parameters
Date_     20170429
Time      15.02
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD        32768
SOLVENT   CDCl3
NS         16
DS         0
SWH        7246.377 Hz
FIDRES     0.221142 Hz
AQ         2.260921 sec
RG         322.5
DE         69.000 usec
TE         298.6 K
D1         2.0000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.40 usec
PL1        1.80 dB
SFO1       400.1324008 MHz

F2 - Processing parameters
SI         16384
SF         400.1300082 MHz
WDW        EM
SSB        0
LB         0 Hz
GB         0
PC         1.00
  
```



Current Data Parameters
NAME check
EXPNO 19
PROCNO 1

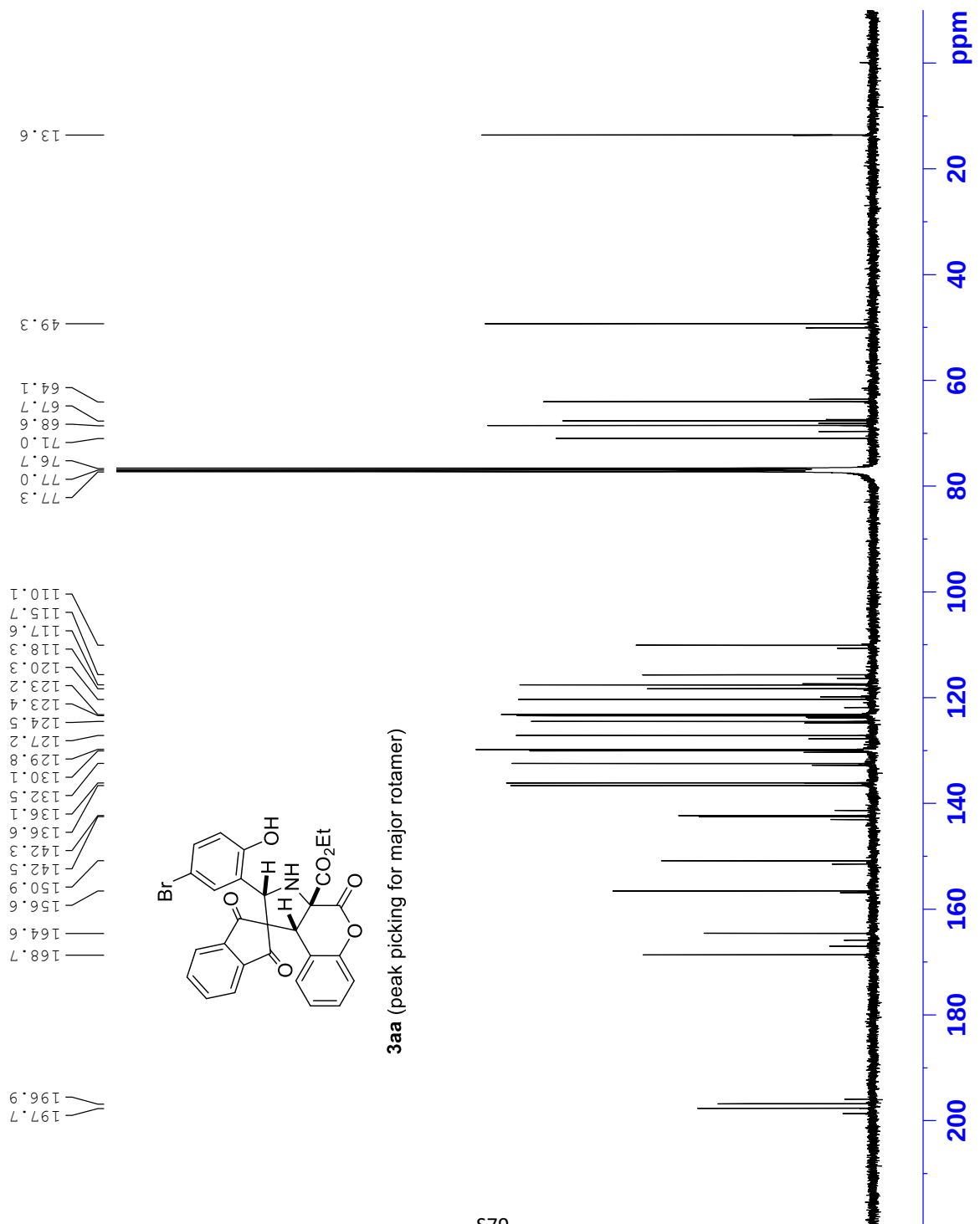
F2 - Acquisition Parameters

Date_ 20170529
Time_ 16.54
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 21287
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 7298.2
DW 20.800 usec
DE 6.50 usec
TE 299.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127715 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



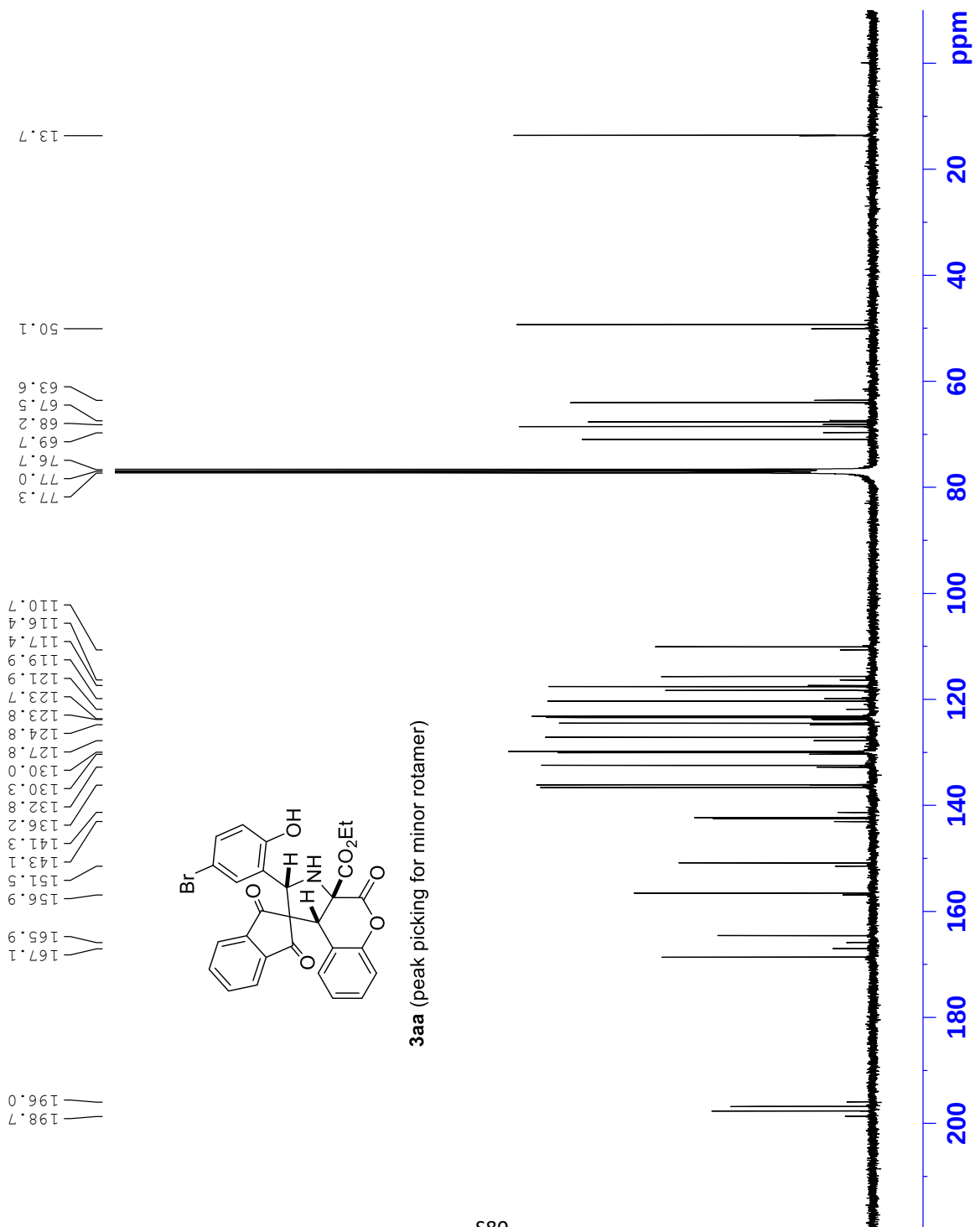
Current Data Parameters
NAME check
EXPNO 19
PROCNO 1

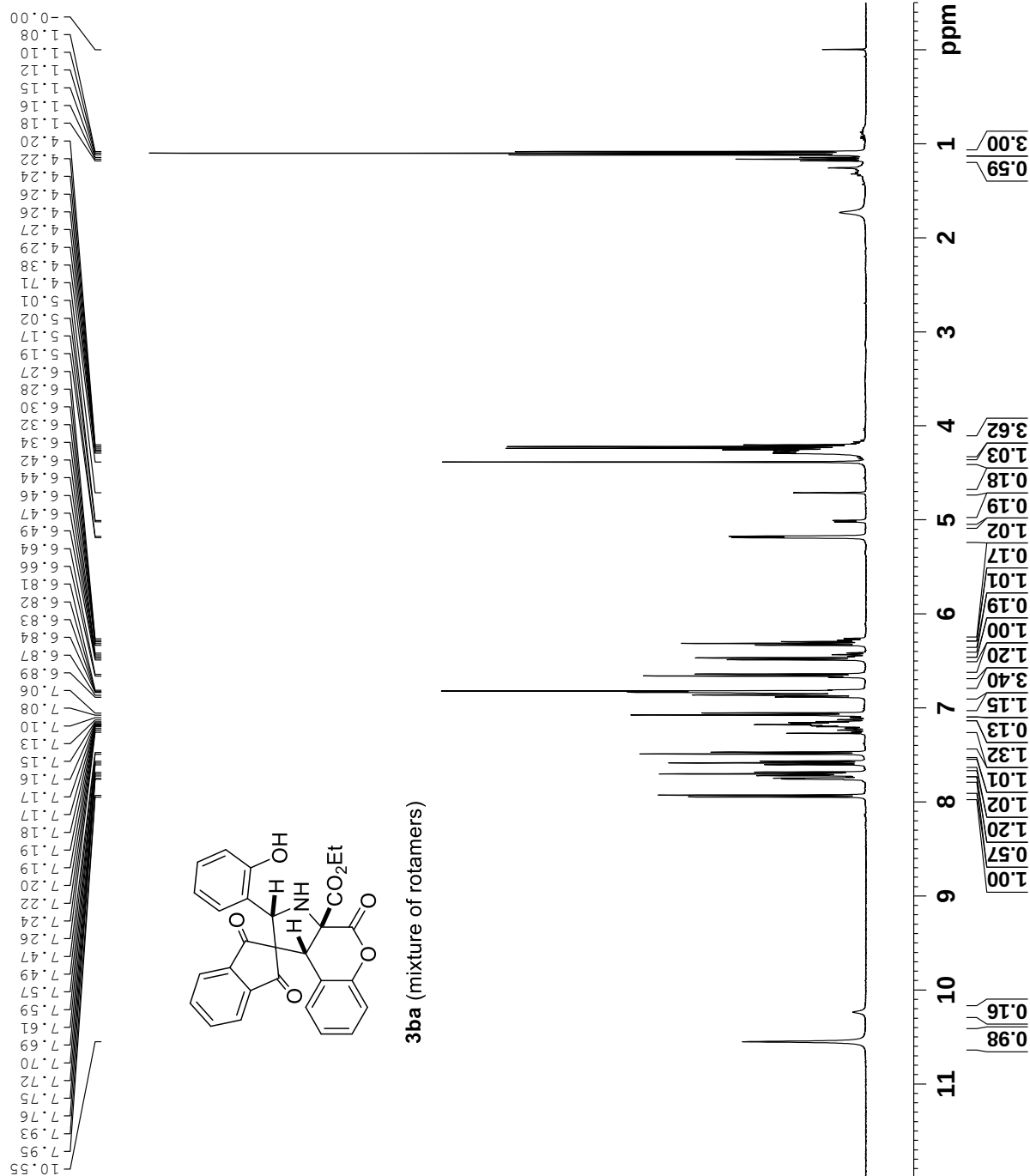
F2 - Acquisition Parameters
Date_ 20170529
Time_ 16.54
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 21287
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 7298.2
DW 20.800 usec
DE 6.50 usec
TE 299.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.623325 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127715 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00





```

Current Data Parameters
NAME      Liu75
EXPNO     2
PROCNO    1

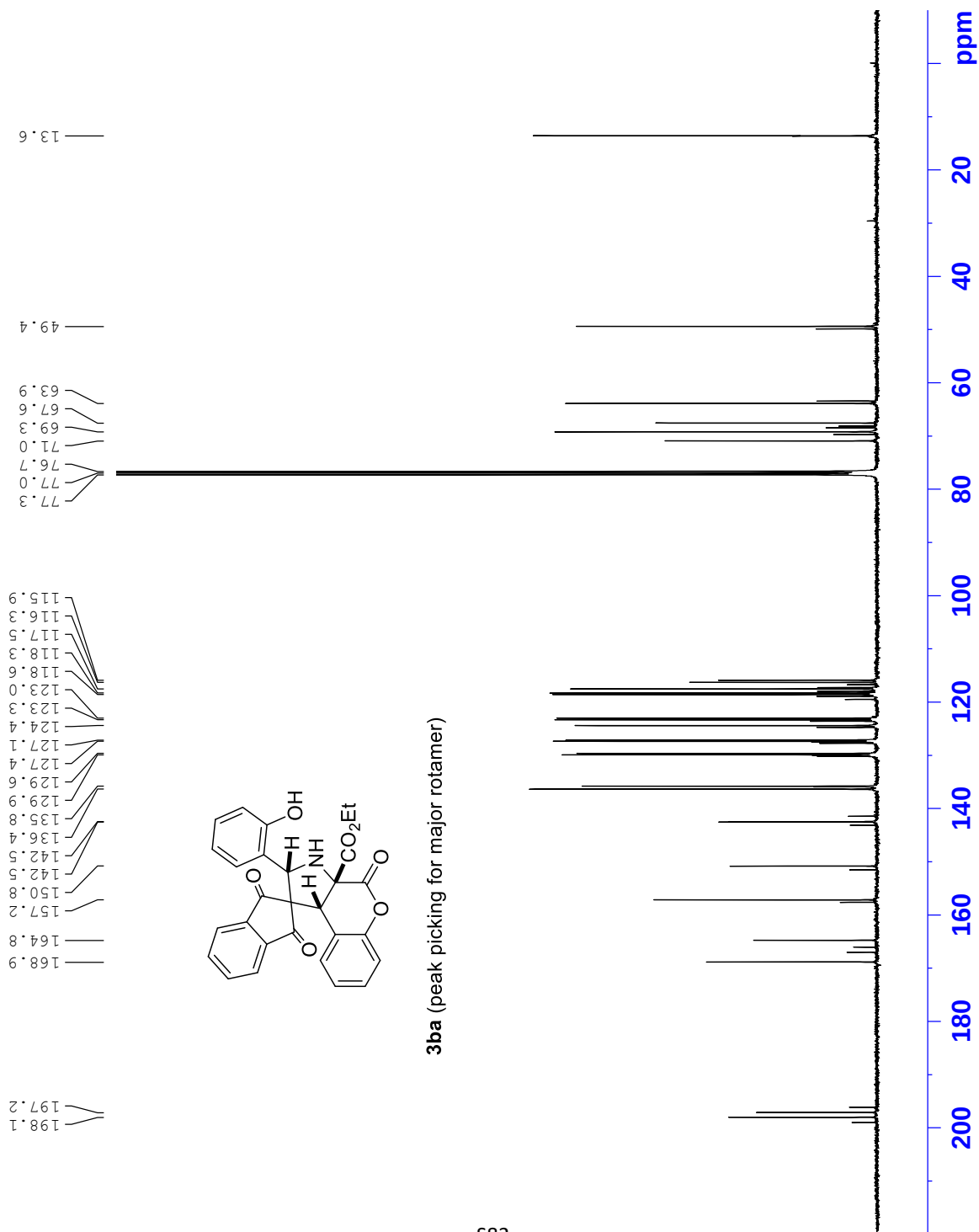
F2 - Acquisition Parameters
Date_     20170410
Time      22.14
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         32768
SOLVENT    CDCl3
NS         5800
DS         0
SWH        24038.461 Hz
FIDRES     0.733596 Hz
AQ         0.6815744 sec
RG         198.09
DW         20.800 usec
DE         6.50 usec
TE         298.3 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      100.6228298 MHz
NUC1       13C
P1         10.00 usec
PLW1       47.50000000 W

===== CHANNEL f2 =====
SFO2      400.1316005 MHz
NUC2       1H
CPDPRG[2   waltz16
PCPD2      90.00 usec
PLW2       15.00000000 W
PLW12      0.33750001 W
PLW13      0.27338001 W

F2 - Processing parameters
SI         32768
SF         100.6127743 MHz
WDW         EM
SSB         0
LB         2.00 Hz
GB         0
PC         1.00

```



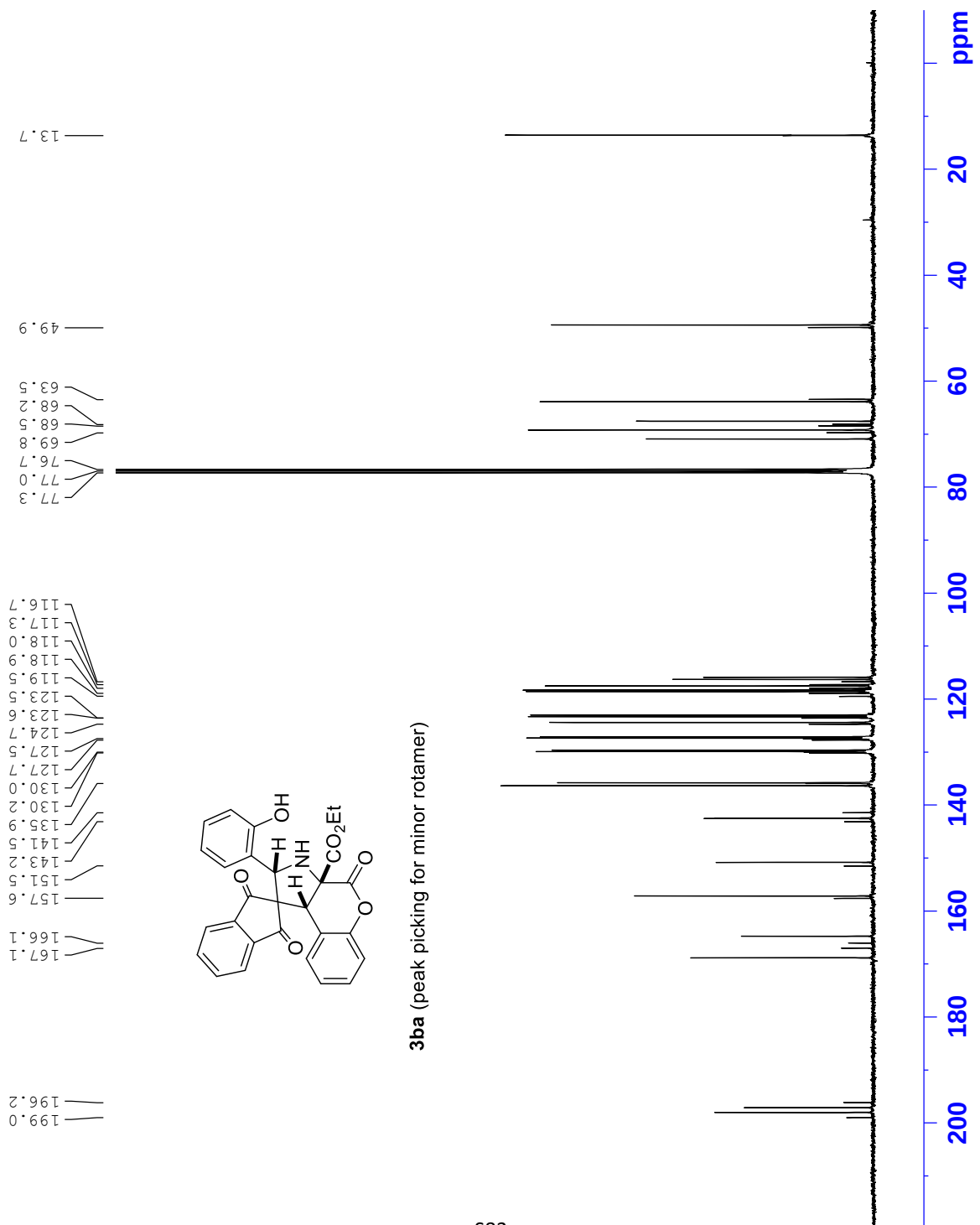
Current Data Parameters
NAME Liu75
EXPNO 2
PROCNO 1

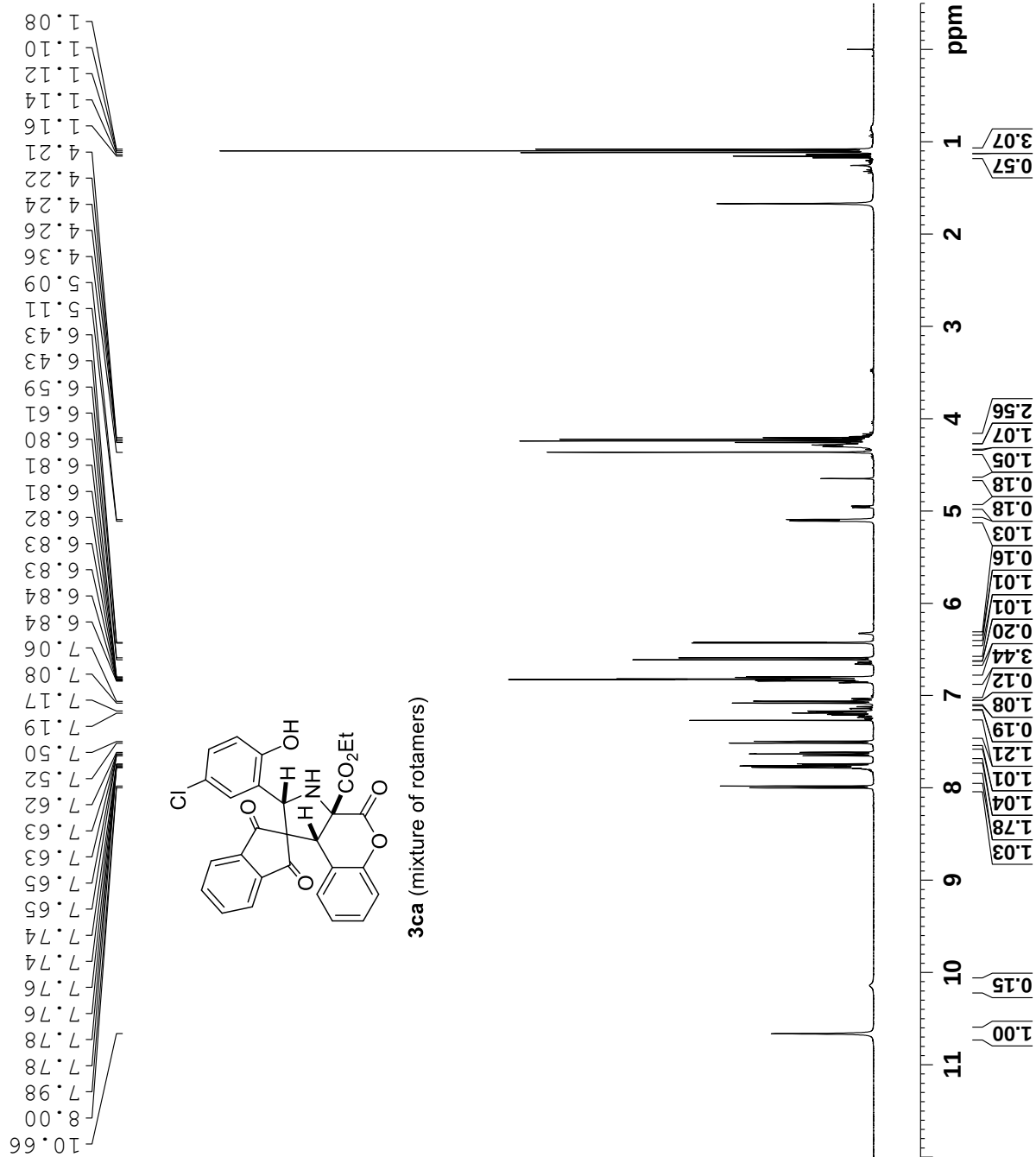
F2 - Acquisition Parameters
Date_ 20170410
Time_ 22.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 5800
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

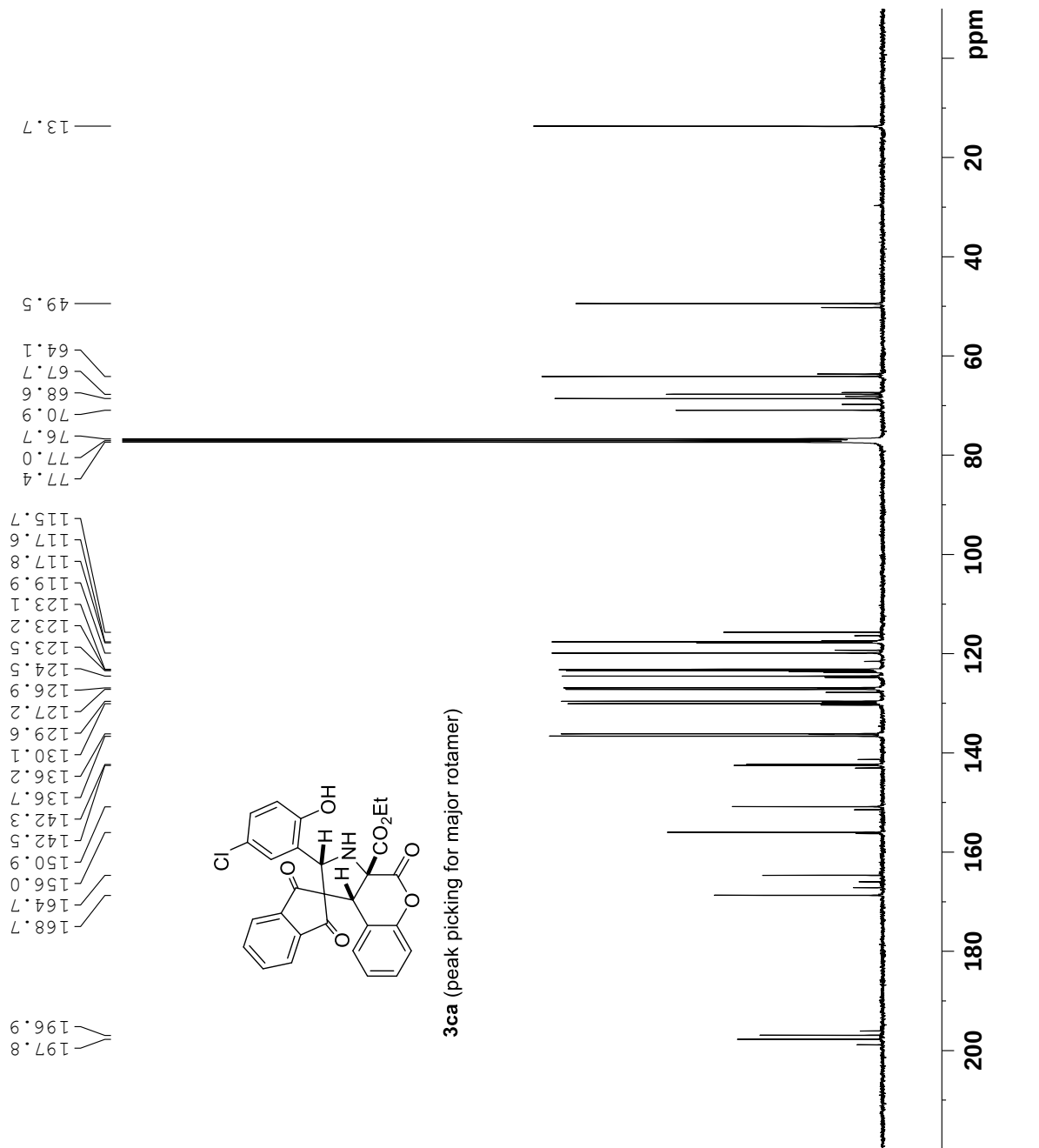
===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127743 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00







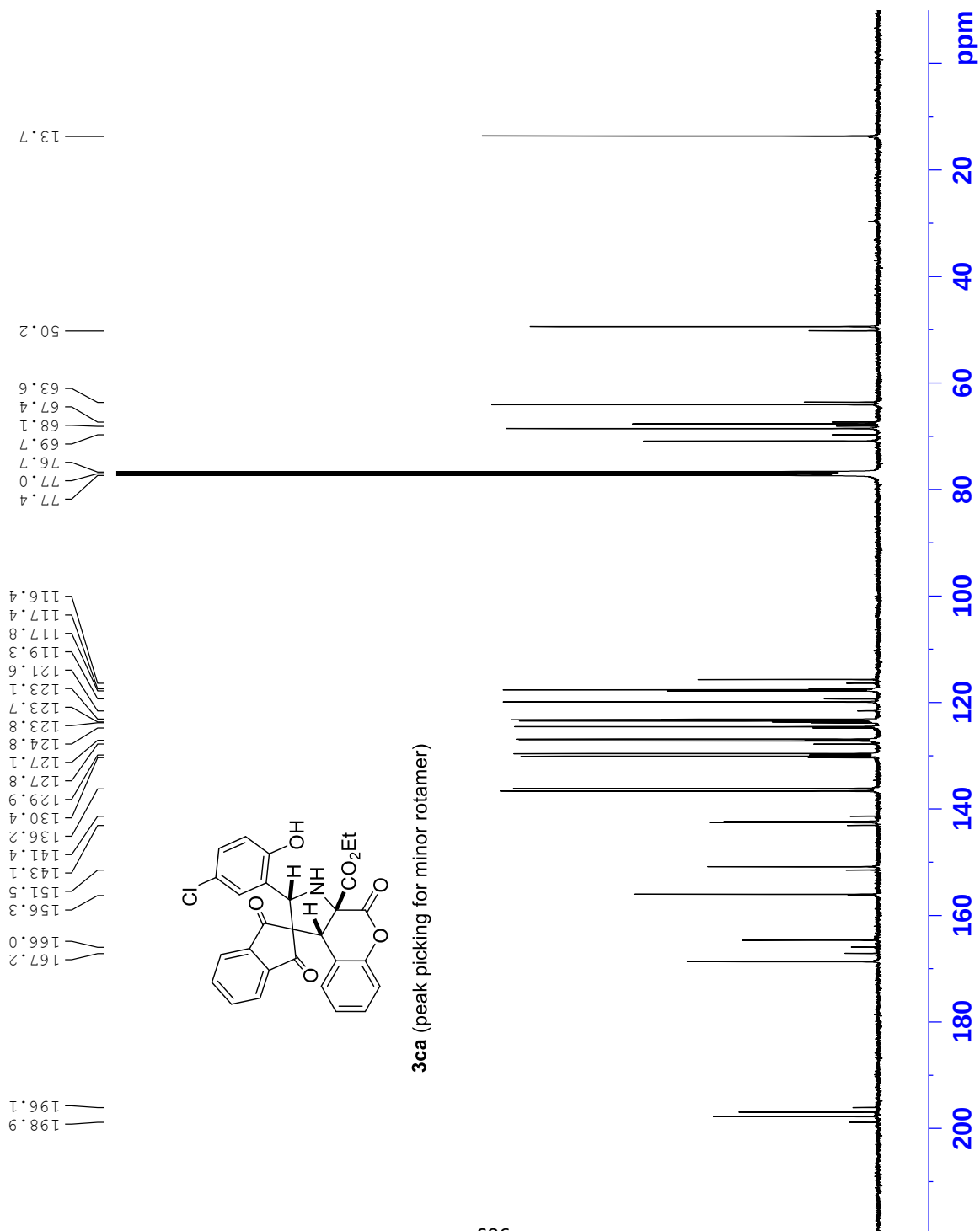
Current Data Parameters
NAME Liu74
EXPNO 2
PROCNO 1

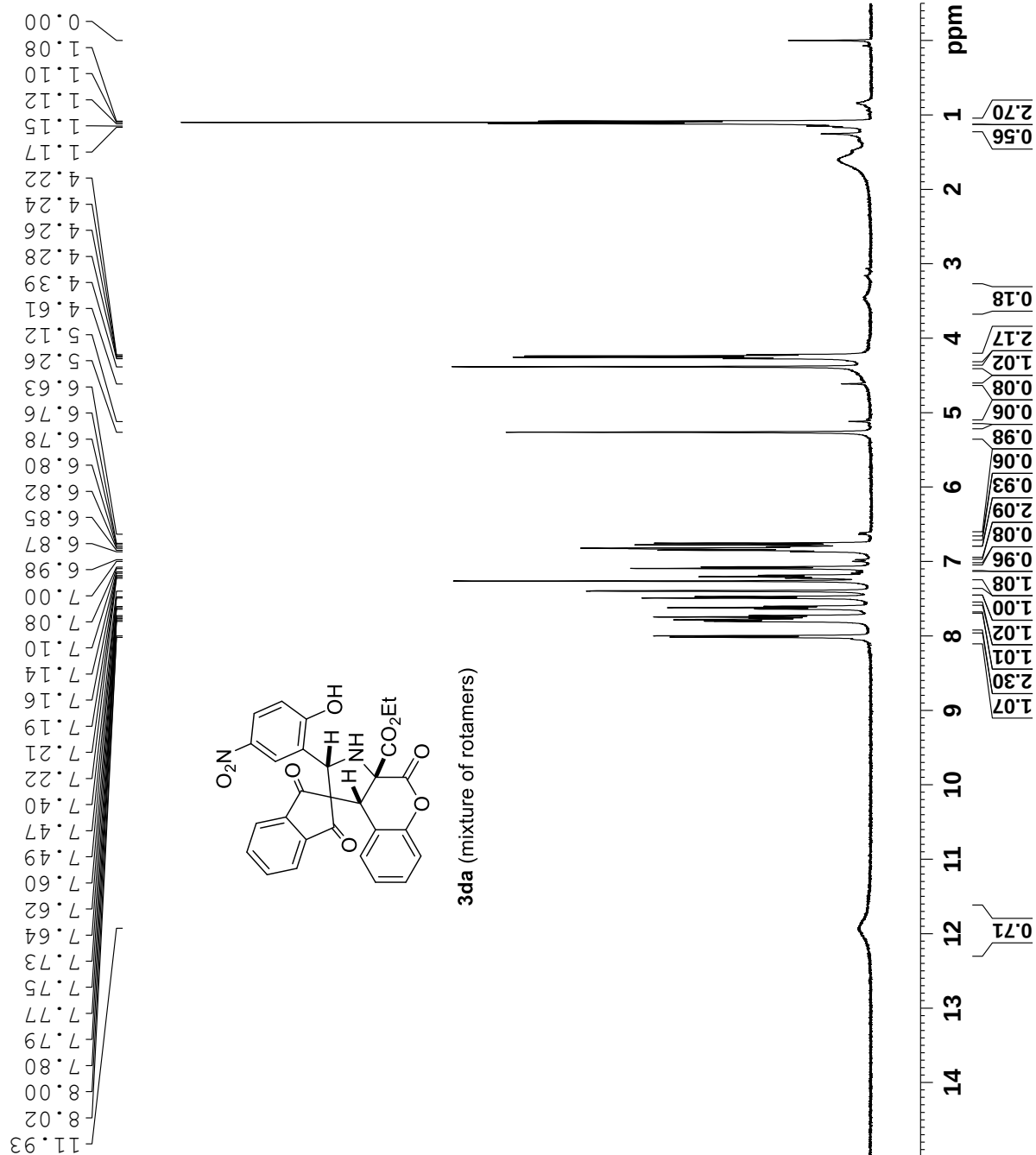
F2 - Acquisition Parameters
Date_ 20170411
Time_ 2.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 9666
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00





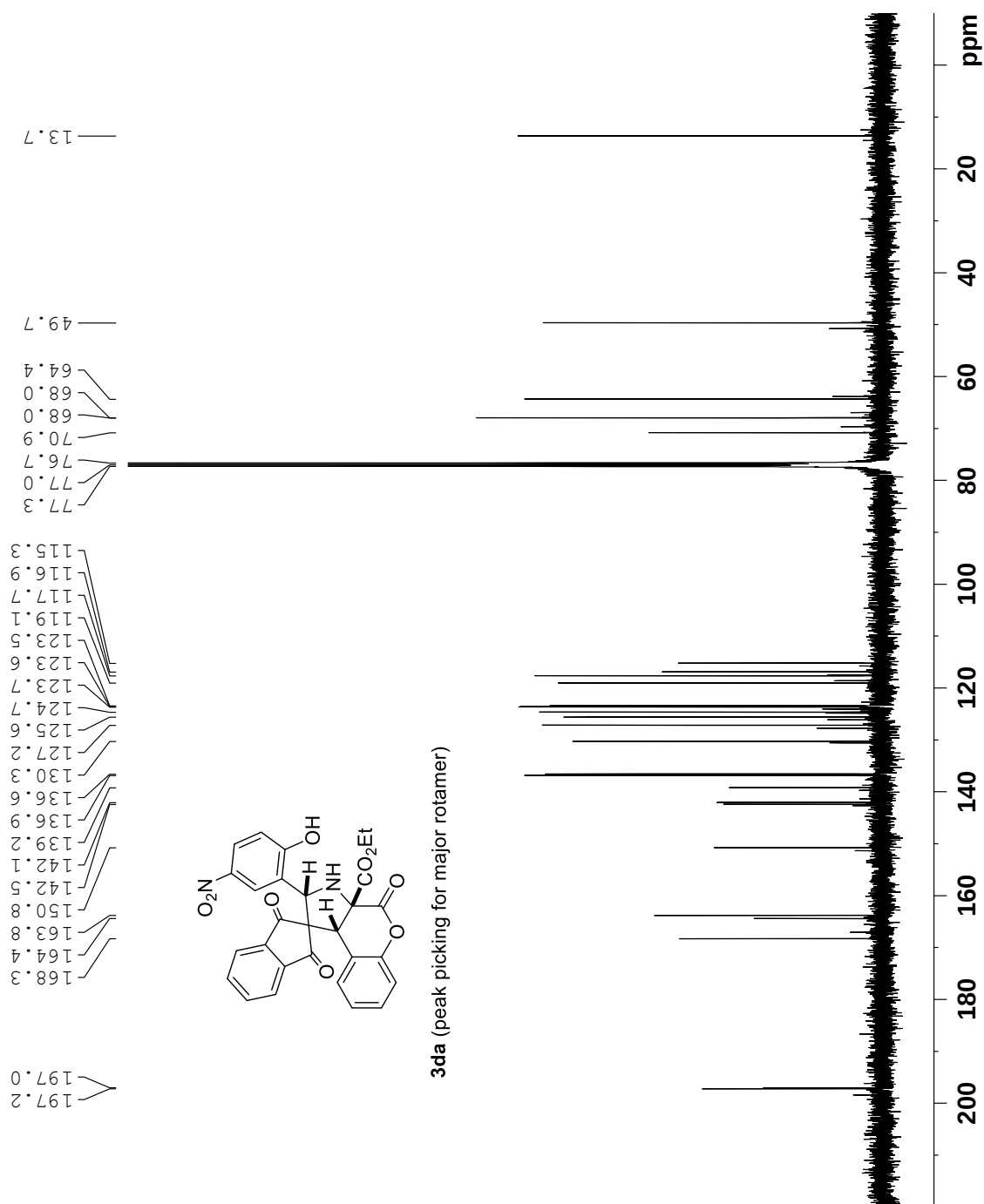
Current Data Parameters
NAME B274
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180501
Time_ 8.16
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4941
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 8192
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 10.45 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

==== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0 dB
PL12 15.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



Current Data Parameters
NAME B274
EXPNO 6
PROCNO 1

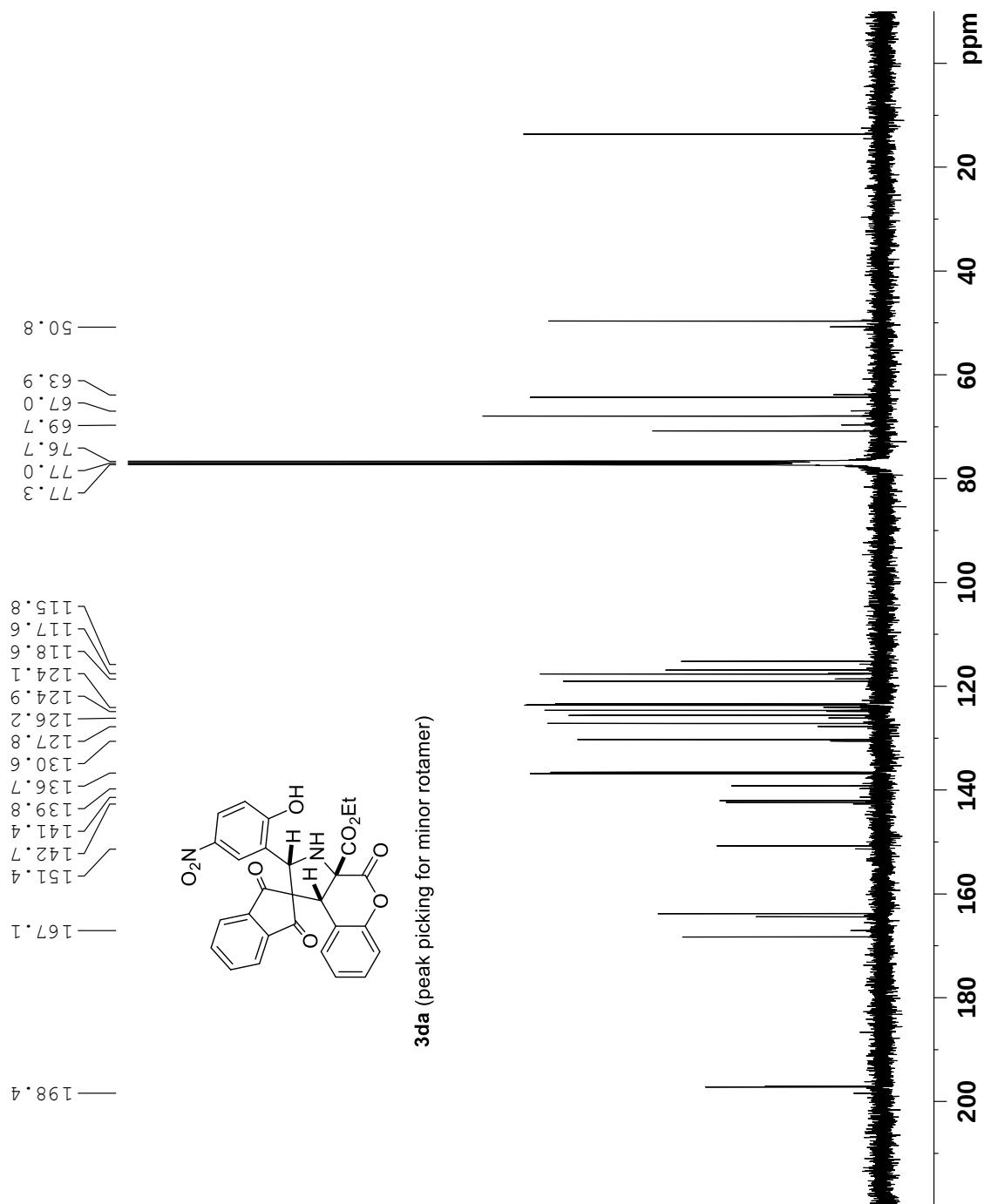
F2 - Acquisition Parameters

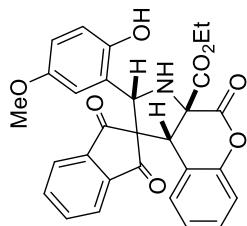
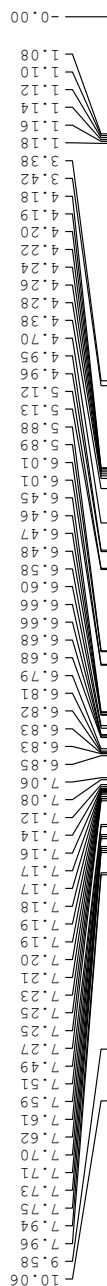
Date_ 20180501
Time 8.16
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 4941
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 8192
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.45 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

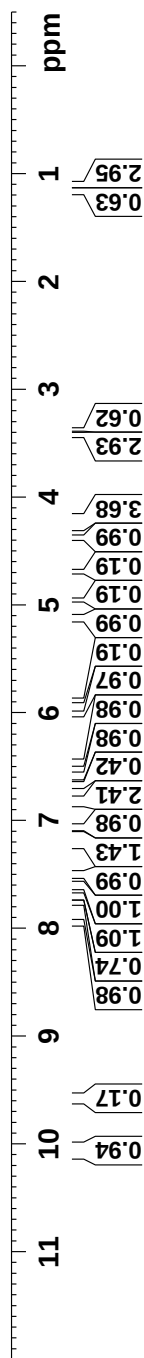
===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0 dB
PL12 15.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00





3ea (mixture of rotamers)



Current Data Parameters
NAME Bonyl10
EXNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170515
Time_ 17.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 78.51
DW 69.333 usec
DE 10.50 usec
TE 298.7 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1300053 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

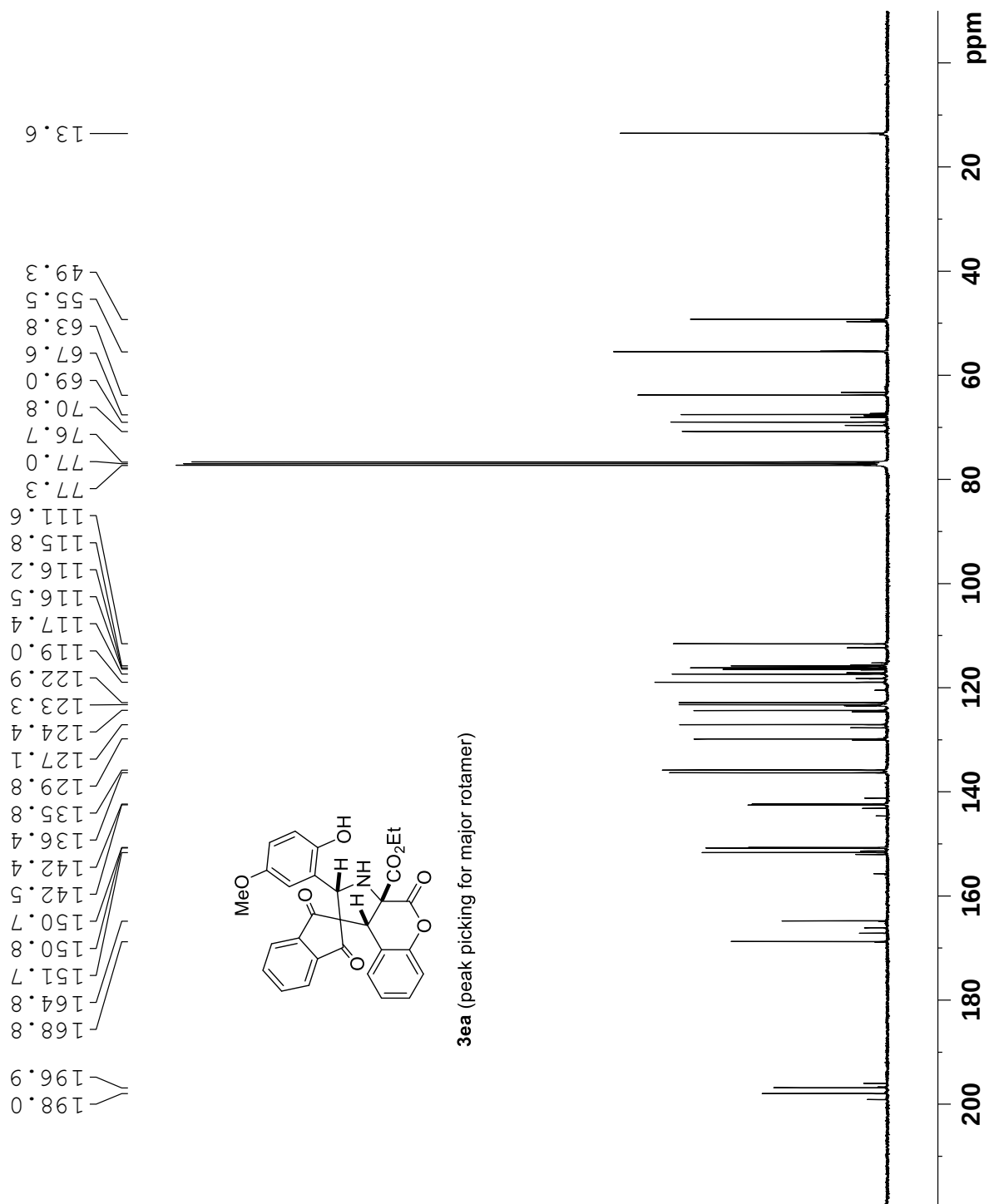
Current Data Parameters
NAME B110
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170411
Time 2.55
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 5718
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 32768
DW 20.800 usec
DE 6.50 usec
TE 295.8 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.623325 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127815 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



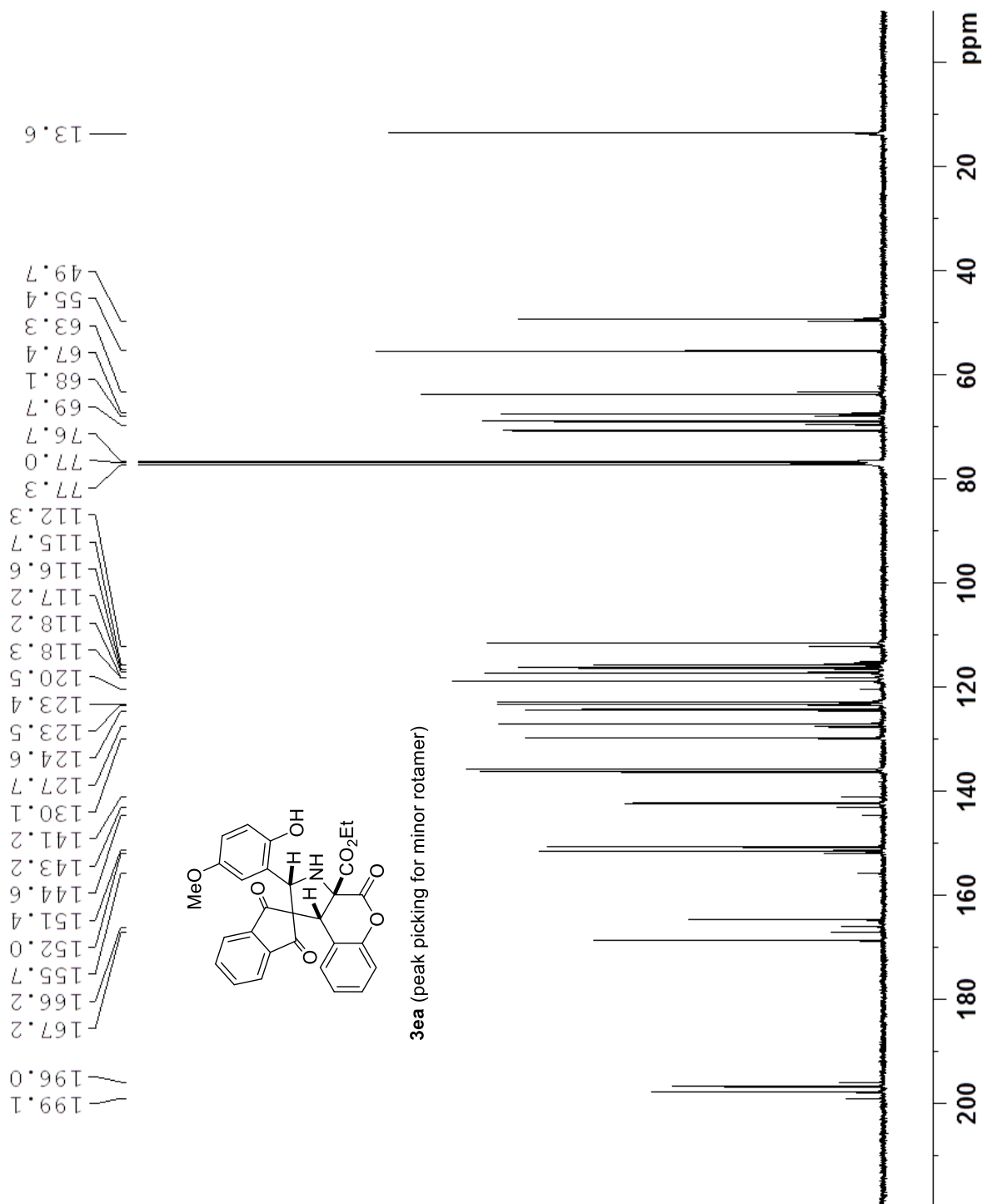
Current Data Parameters
 NAME B110
 EXPNO 5
 PROCNO 1

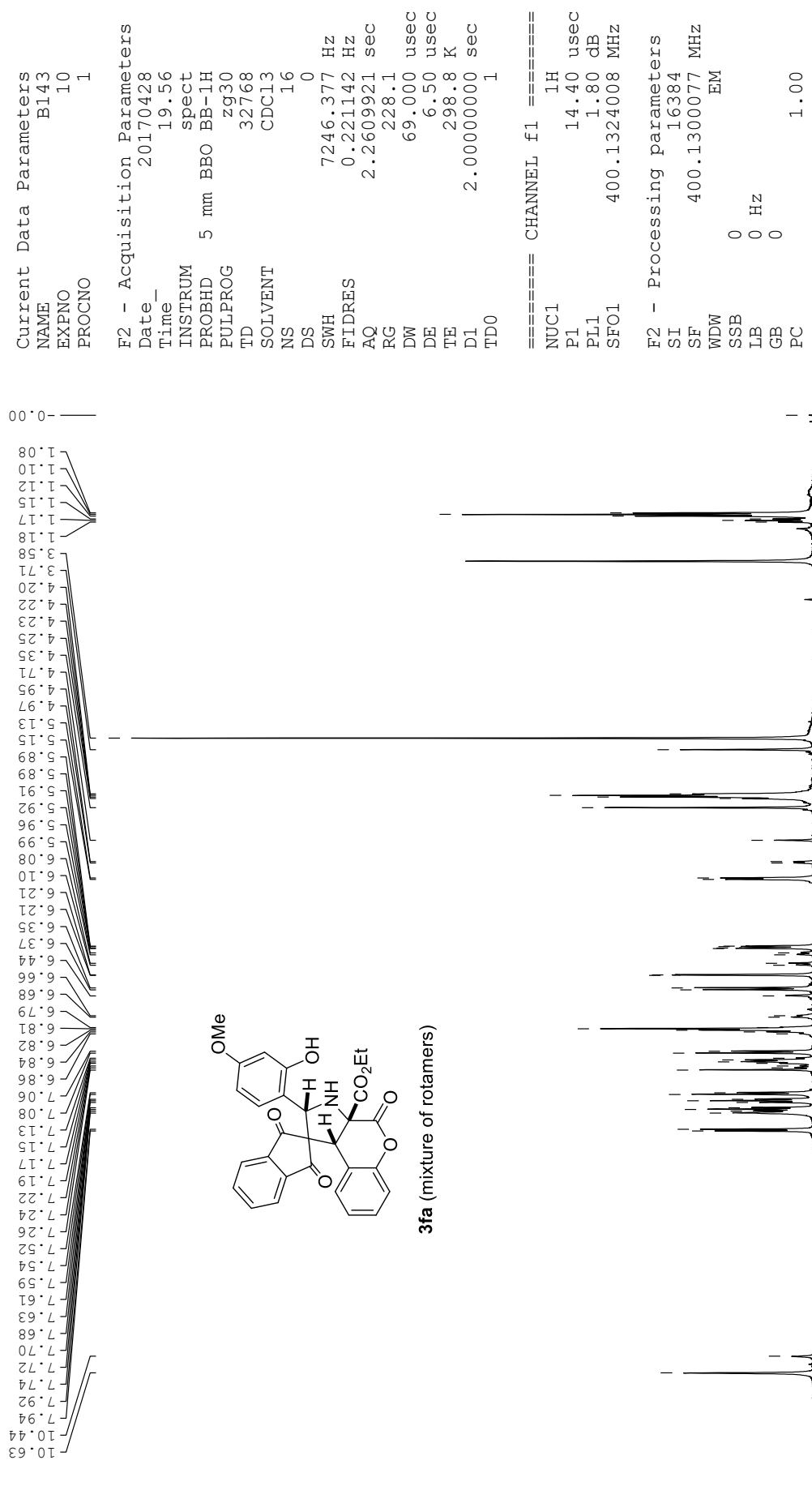
F2 - Acquisition Parameters
 Date_ 20170411
 Time_ 2.55
 INSTRUM spect
 PROBD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 5718
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6815744 sec
 RG 32768
 DW 20.800 usec
 DE 6.50 usec
 TE 295.8 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PL1 7.00 dB
 SFO1 100.623325 MHz

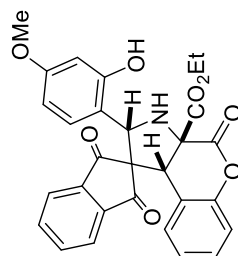
==== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 1.80 dB
 PL12 17.00 dB
 PL13 20.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127815 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

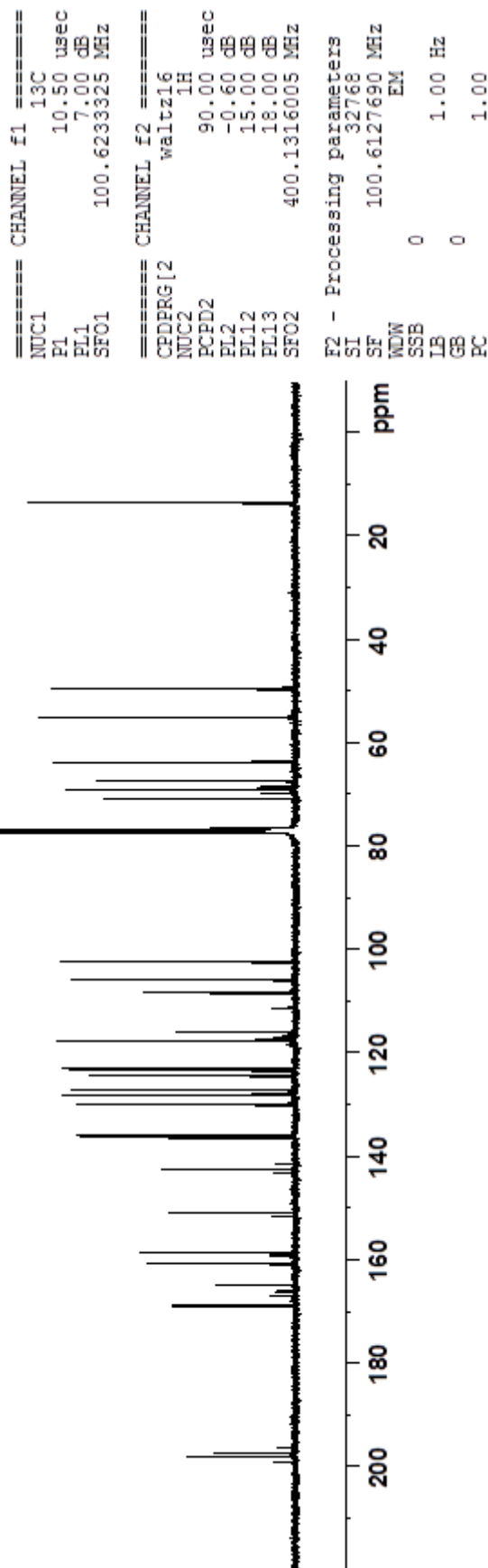


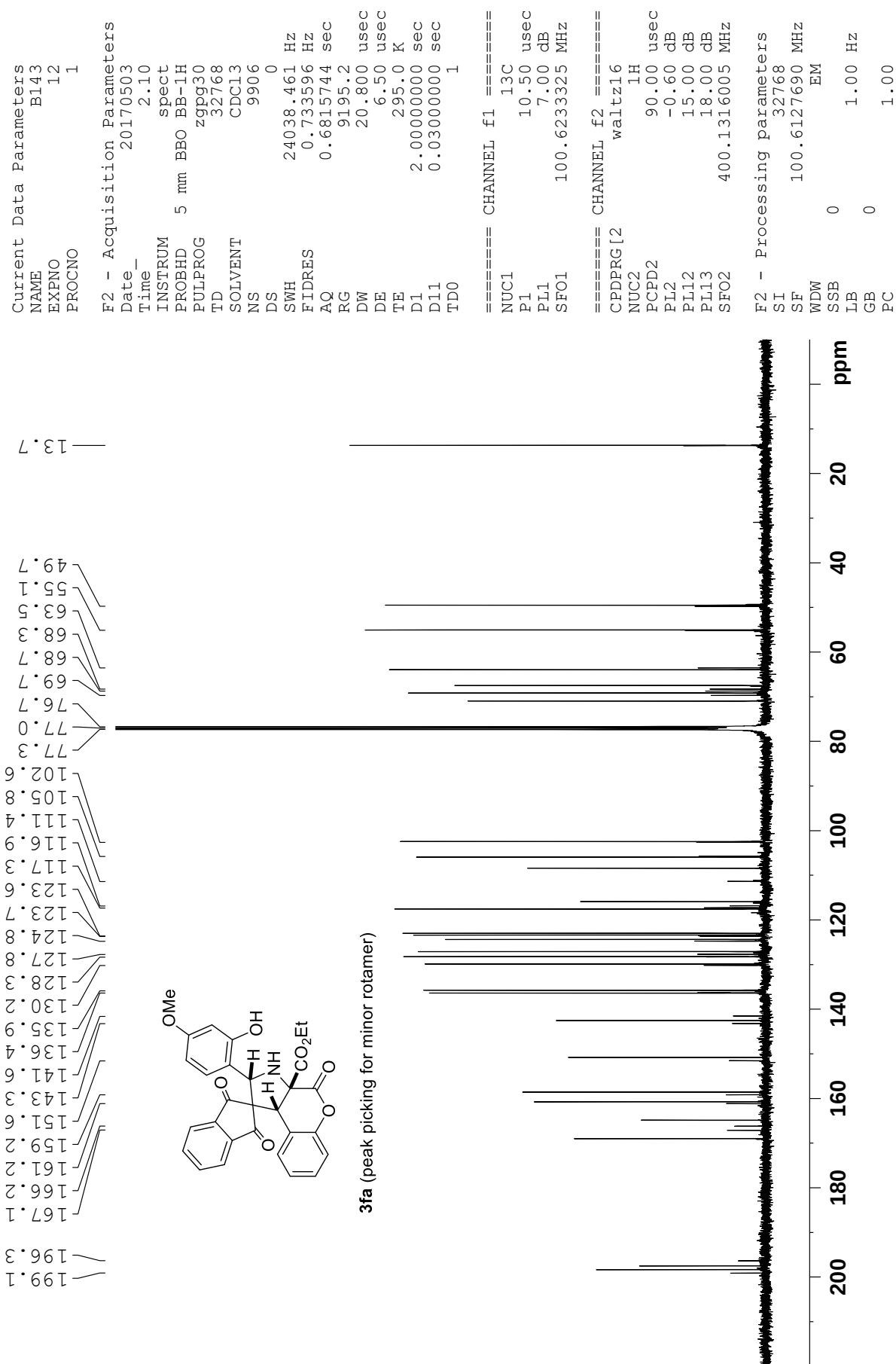


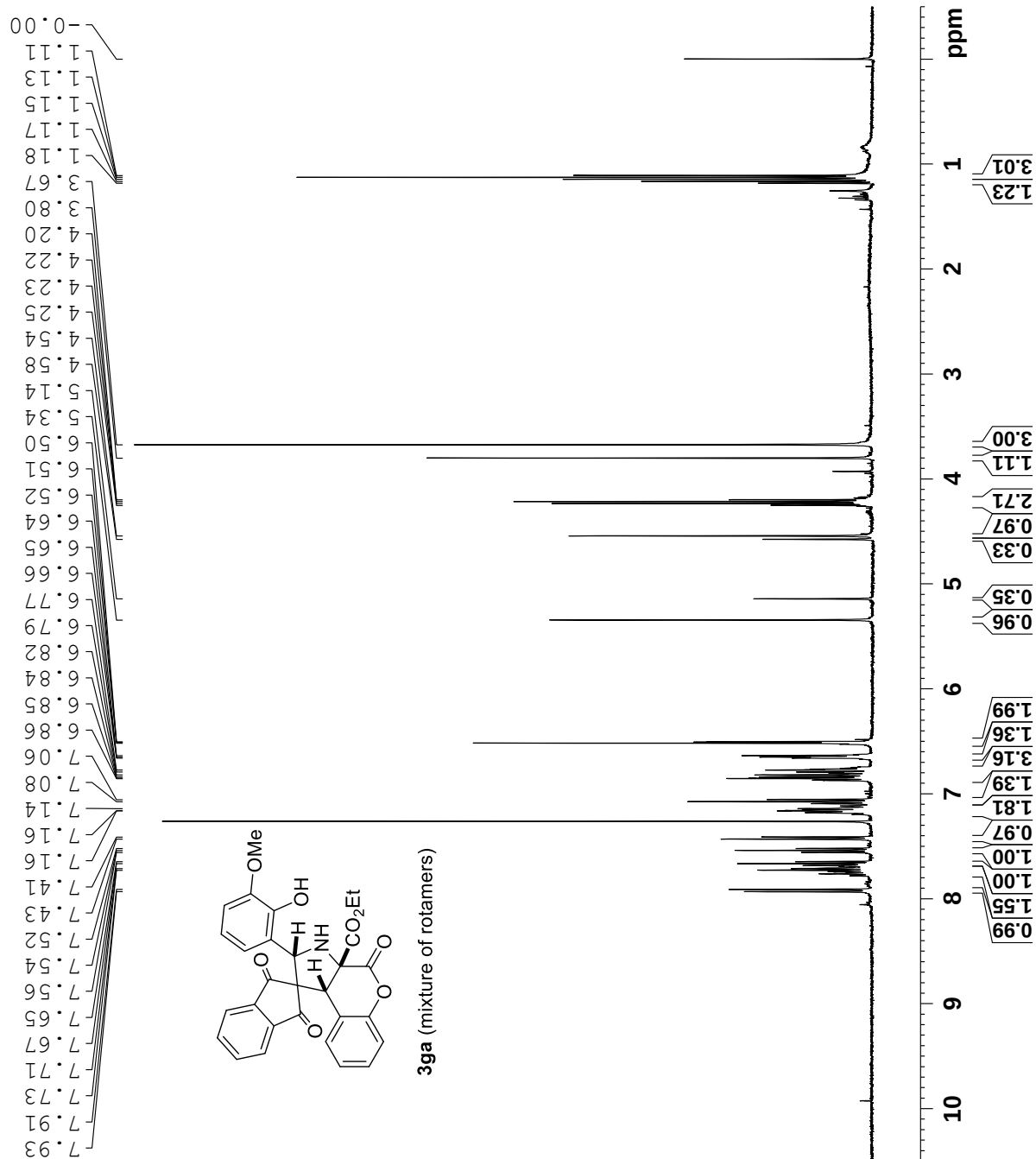
198.3
197.5
169.0
164.9
160.7
158.6
150.9
142.6
142.6
142.6
136.4
135.8
129.9
128.3
127.1
124.4
123.4
123.1
117.6
116.0
108.5
106.0
102.5
71.0
69.2
67.5
64.0
55.0
49.5
13.7



3fa (peak picking for major rotamer)







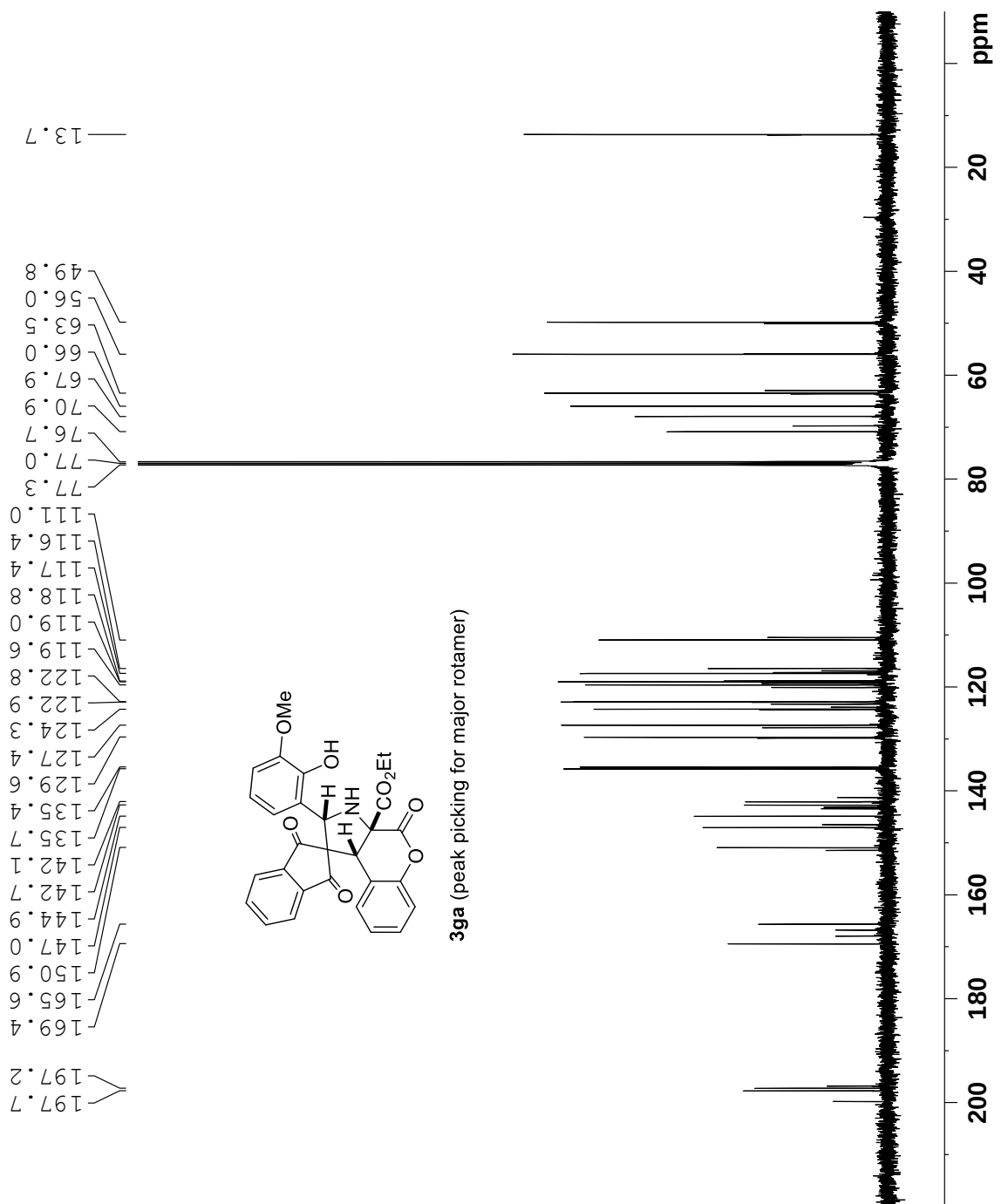
Current Data Parameters
 NAME B315
 EXPNO 9
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180506
 Time_ 10.34
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 1903
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6815744 sec
 RG 2298.8
 DW 20.800 usec
 DE 6.50 usec
 TE 673.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.45 usec
 PL1 7.00 dB
 SF01 100.6233325 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0 dB
 PL12 15.00 dB
 PL13 20.00 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127747 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00



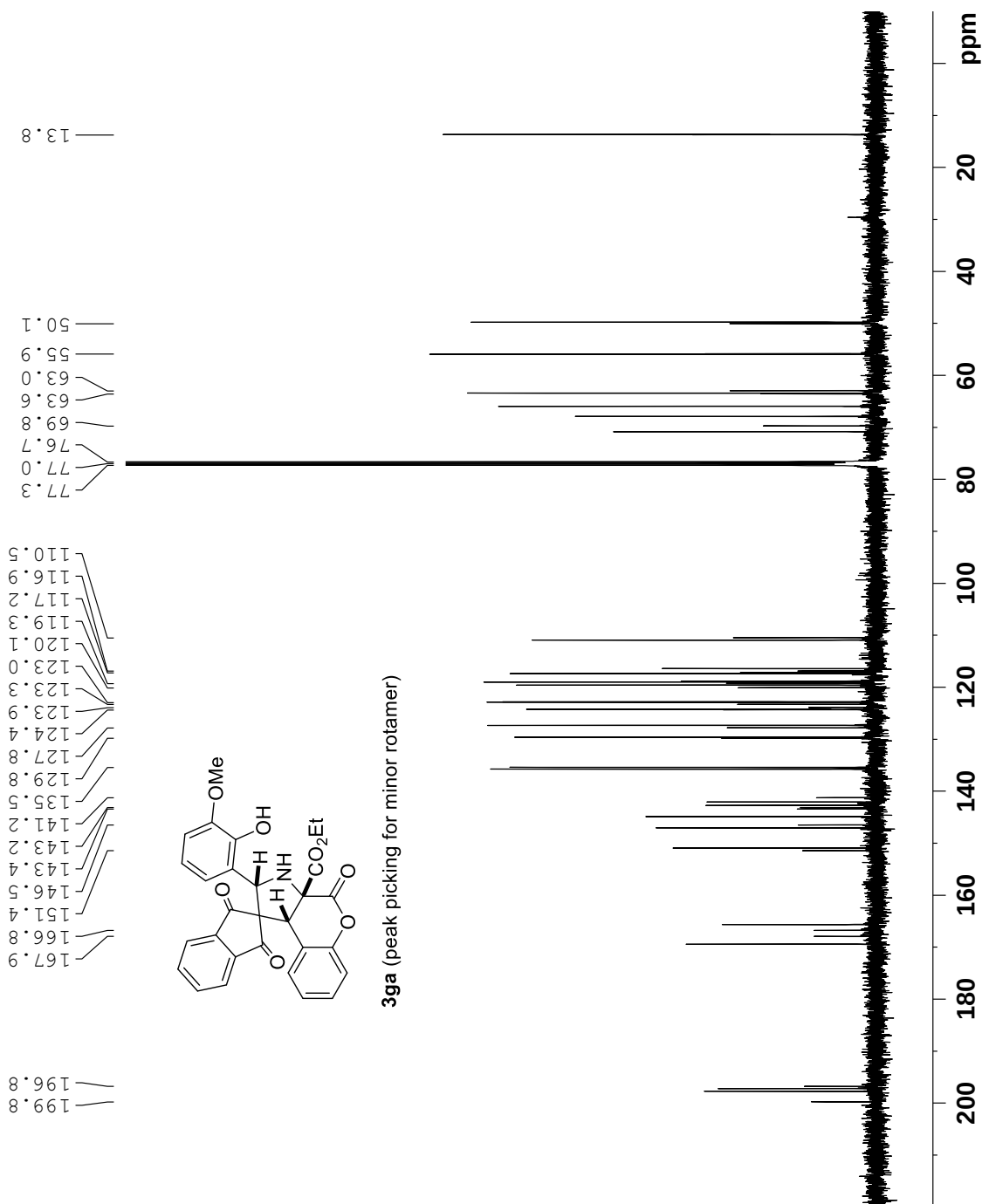
Current Data Parameters
NAME B315
EXPNO 9
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180506
Time_ 10.34
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 1903
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 2298.8
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 ¹³C
P1 10.45 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 ¹H
PCPD2 90.00 usec
PL2 0 dB
PL12 15.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127725 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



Current Data Parameters
NAME Hank127
EXPNO 10
PROCNO 1

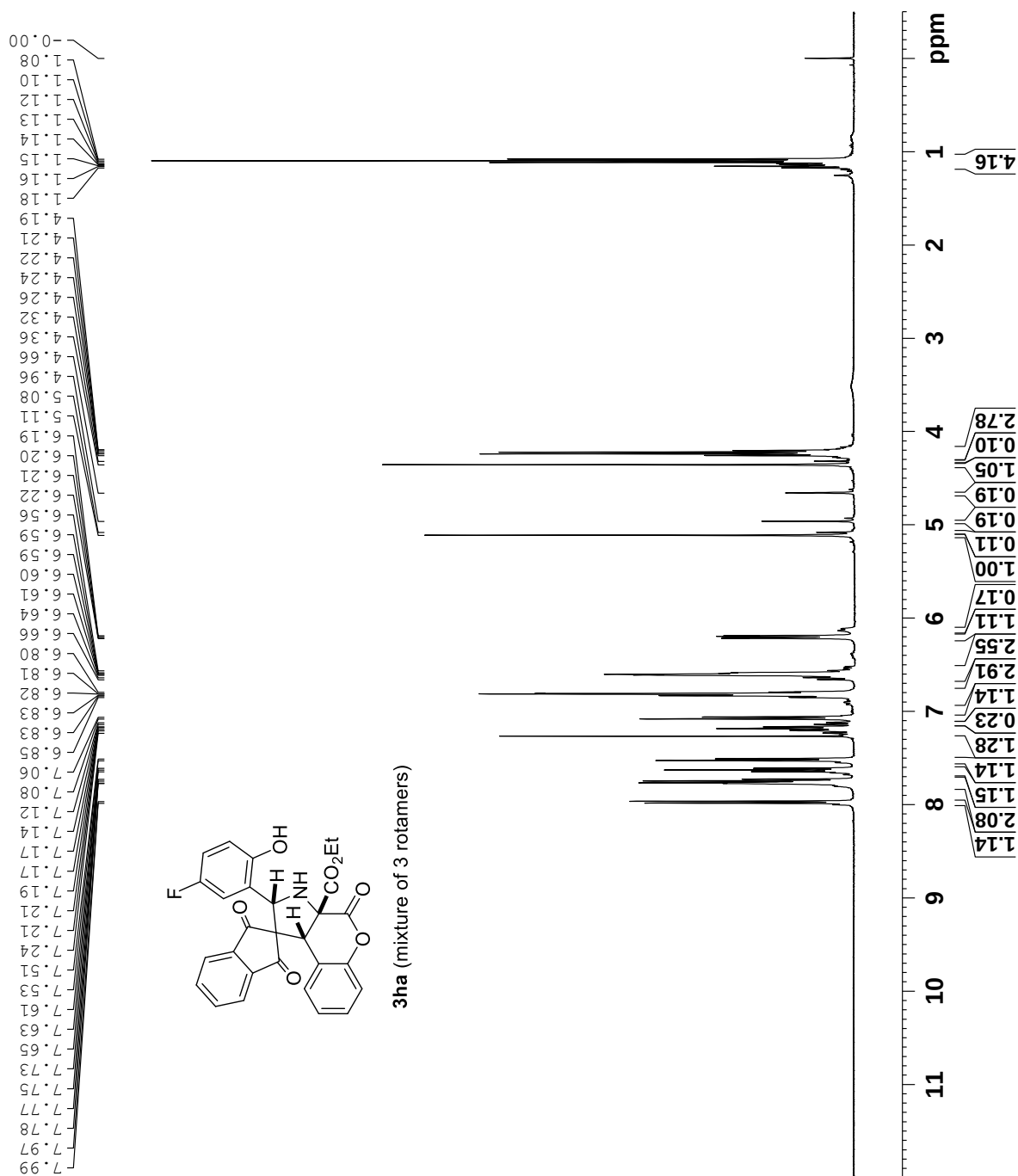
F2 - Acquisition Parameters

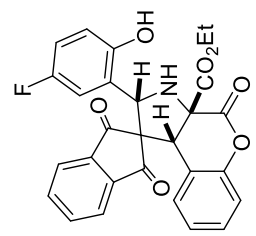
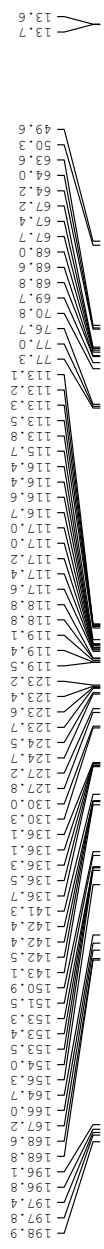
Date_ 20180501
Time_ 17.58
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.260921 sec
RG 114
DW 69.000 usec
DE 6.50 usec
TE 673.2 K
D1 2.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 0 dB
SFO1 400.1324008 MHz

F2 - Processing parameters

SI 16384
SF 400.1300060 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00





3ha (mixture of 3 rotamers)

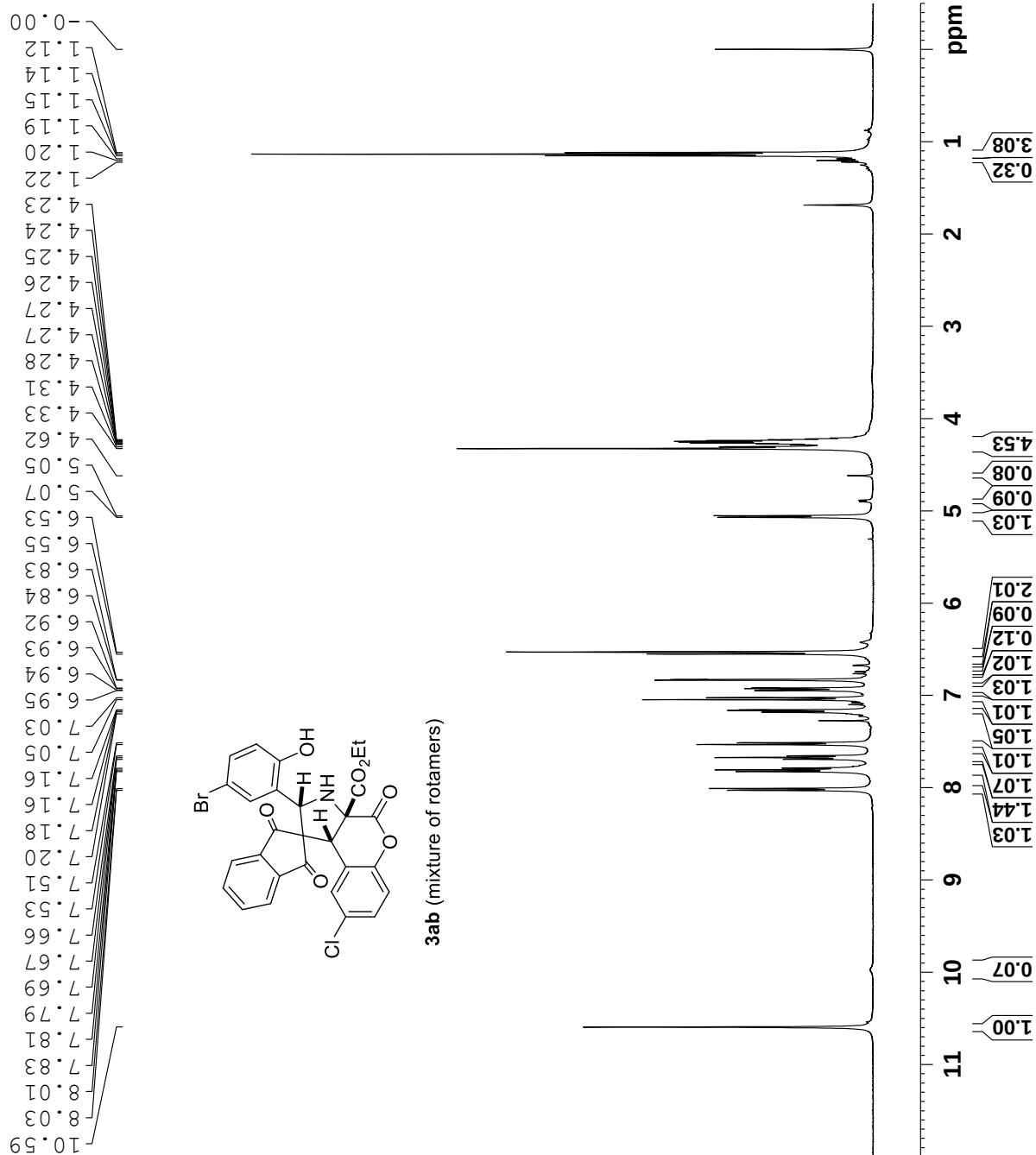
Current Data Parameters
 NAME Hank127
 EXPNO 8
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180328
 Time 22.40
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl₃
 NS 12516
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6815744 sec
 RG 5792.6
 DW 20.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.45 usec
 PL1 7.00 dB
 SFO1 100.6233325 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0 dB
 PL12 15.00 dB
 PL13 20.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127701 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00



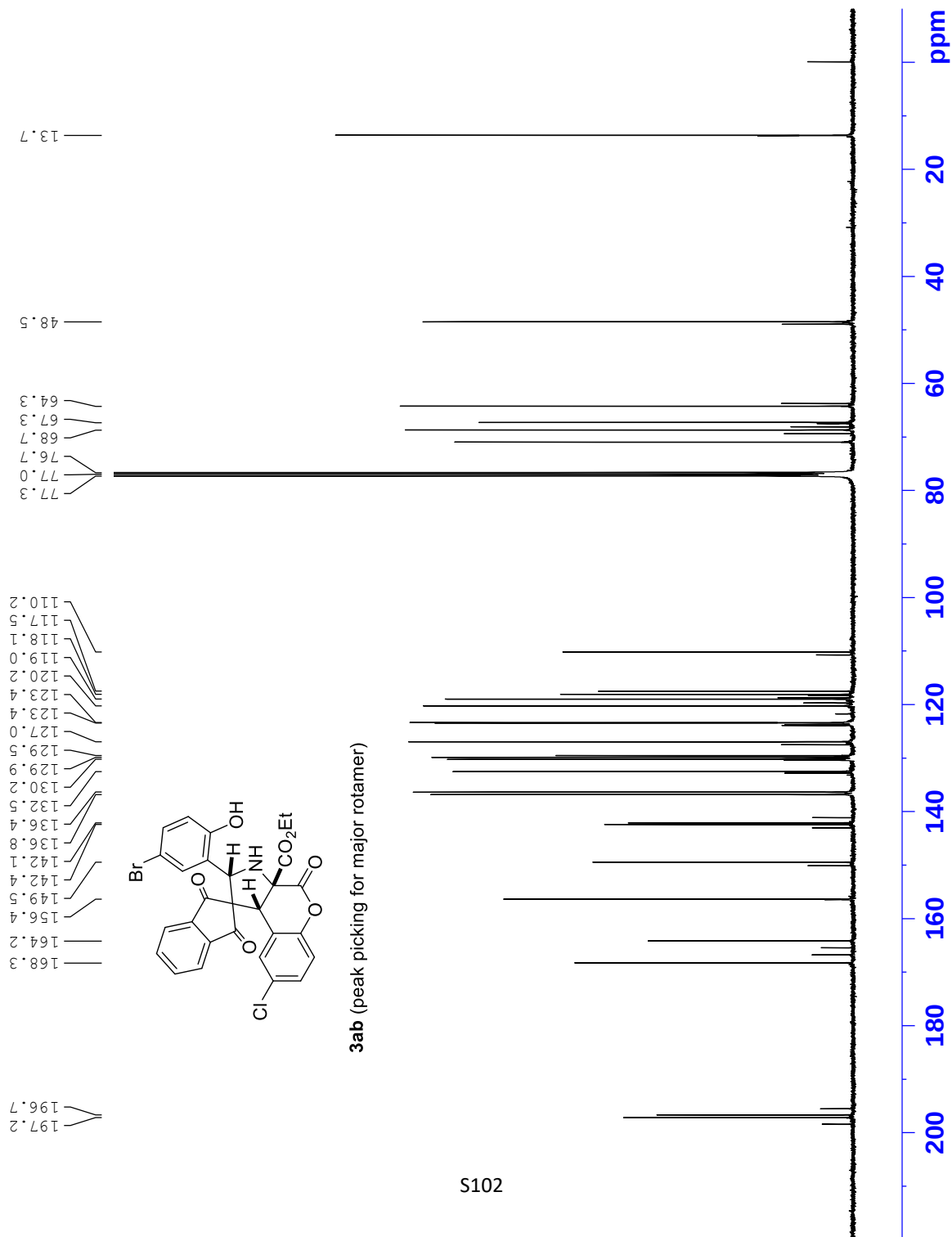
Current Data Parameters
NAME liul45
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170304
Time 22.18
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 25262
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 5792.6
DW 20.800 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

==== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127742 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



Current Data Parameters
 NAME liul145
 EXPNO 4
 PROCNO 1

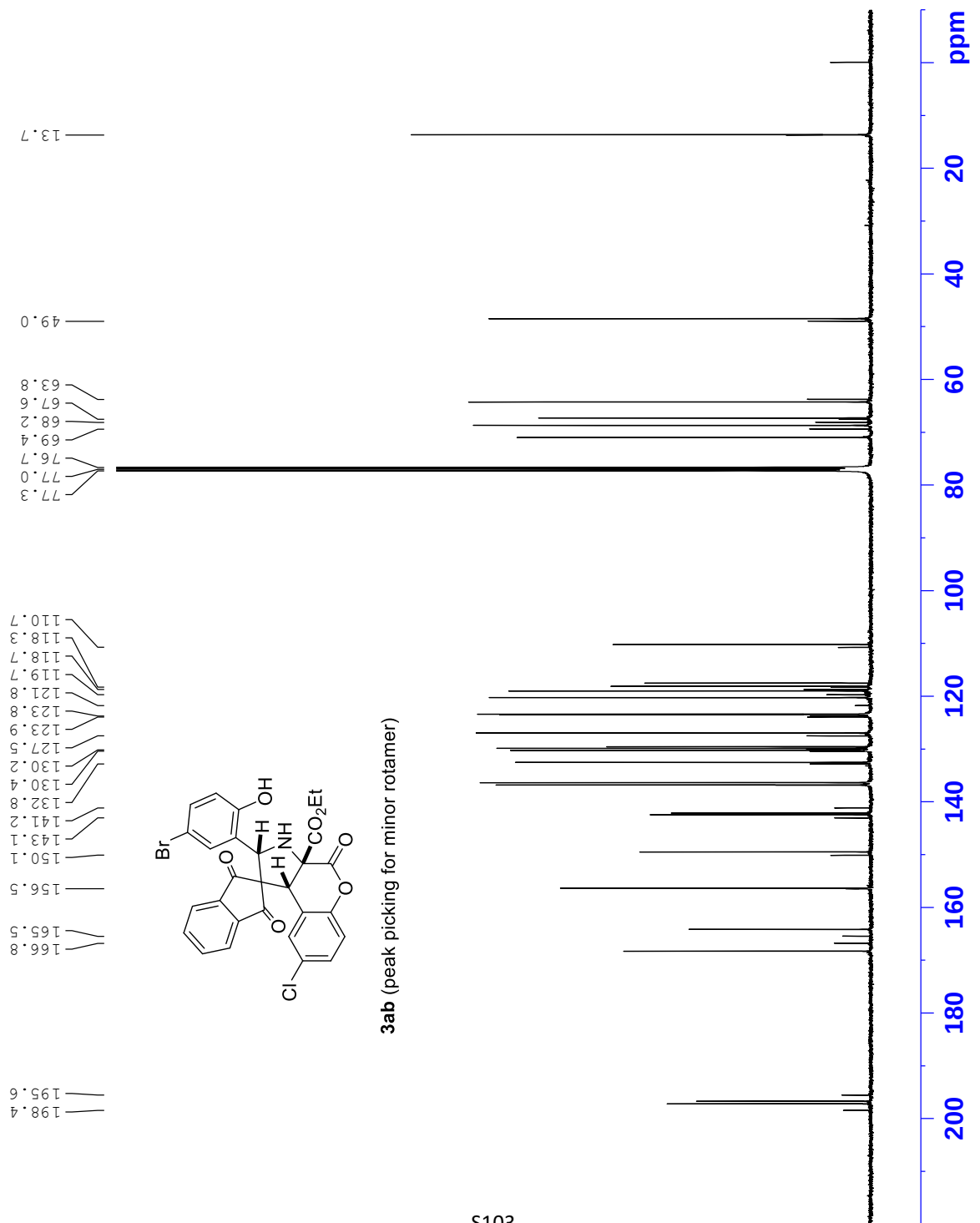
F2 - Acquisition Parameters

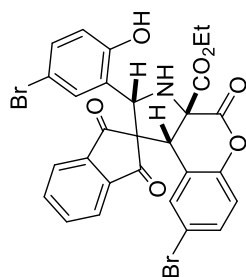
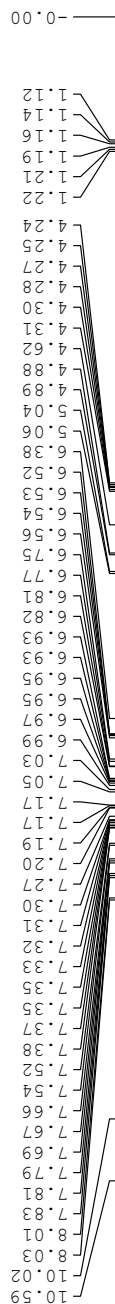
Date_ 20170304
 Time_ 22.18
 INSTRUM spect
 PROBD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDC13
 NS 25262
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6815744 sec
 RG 5792.6
 DW 20.800 usec
 DE 6.50 usec
 TE 295.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PL1 7.00 dB
 SFO1 100.6233325 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 1.80 dB
 PL12 17.00 dB
 PL13 20.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127727 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00





3ac (mixture of rotamers)

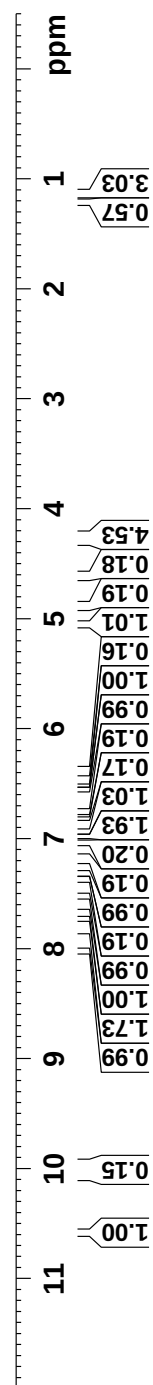
Current Data Parameters
NAME 3ac
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters

Date_ 20170306
Time 22.04
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2609921 sec
RG 161.3
DW 69.000 usec
DE 6.50 usec
TE 295.8 K
D1 2.0000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 14.40 usec
PL1 1.80 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300057 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



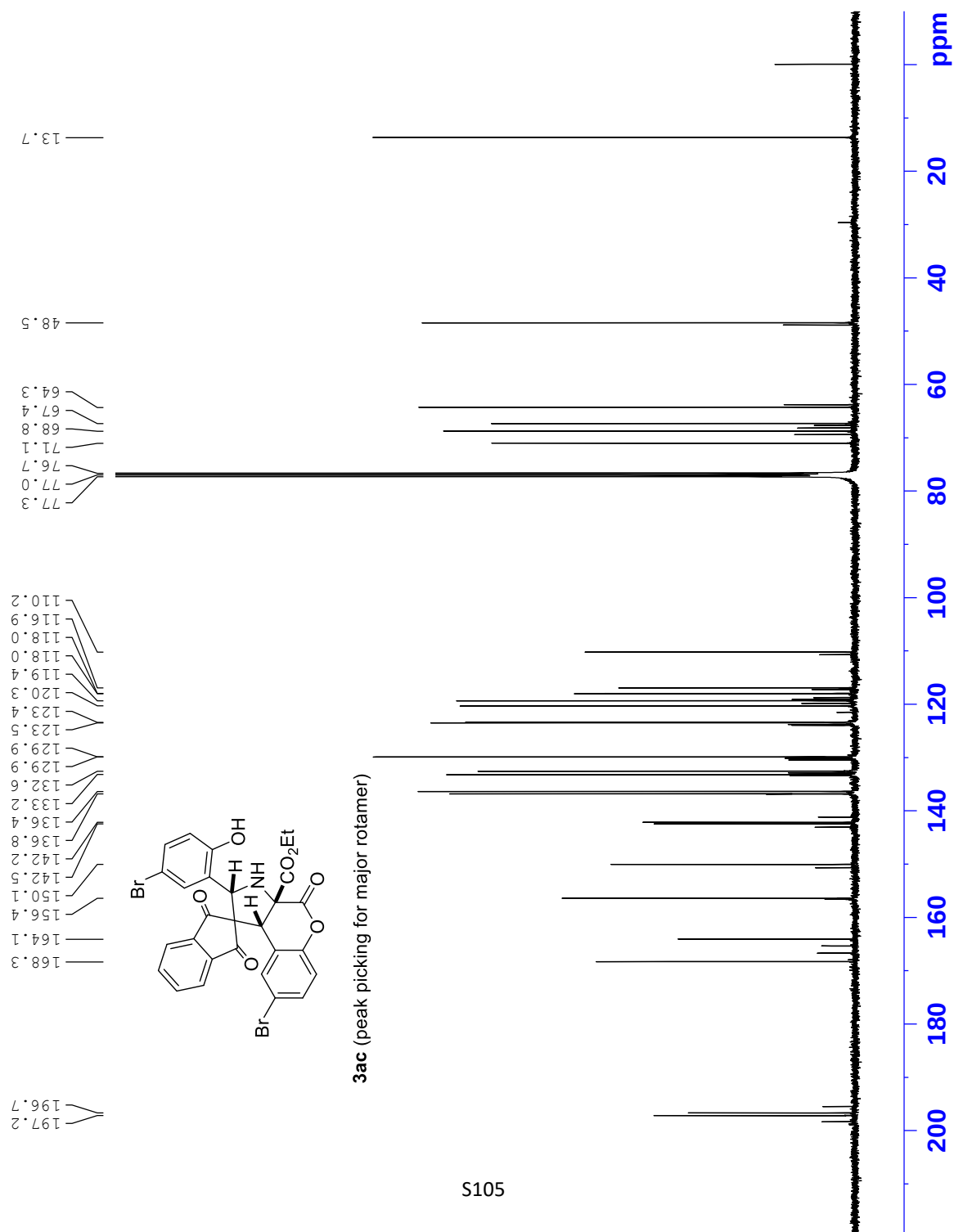
Current Data Parameters
NAME liul46
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170306
Time 22.06
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 14161
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 5792.6
DE 20.800 usec
TE 295.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.60 dB
PL12 15.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127720 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



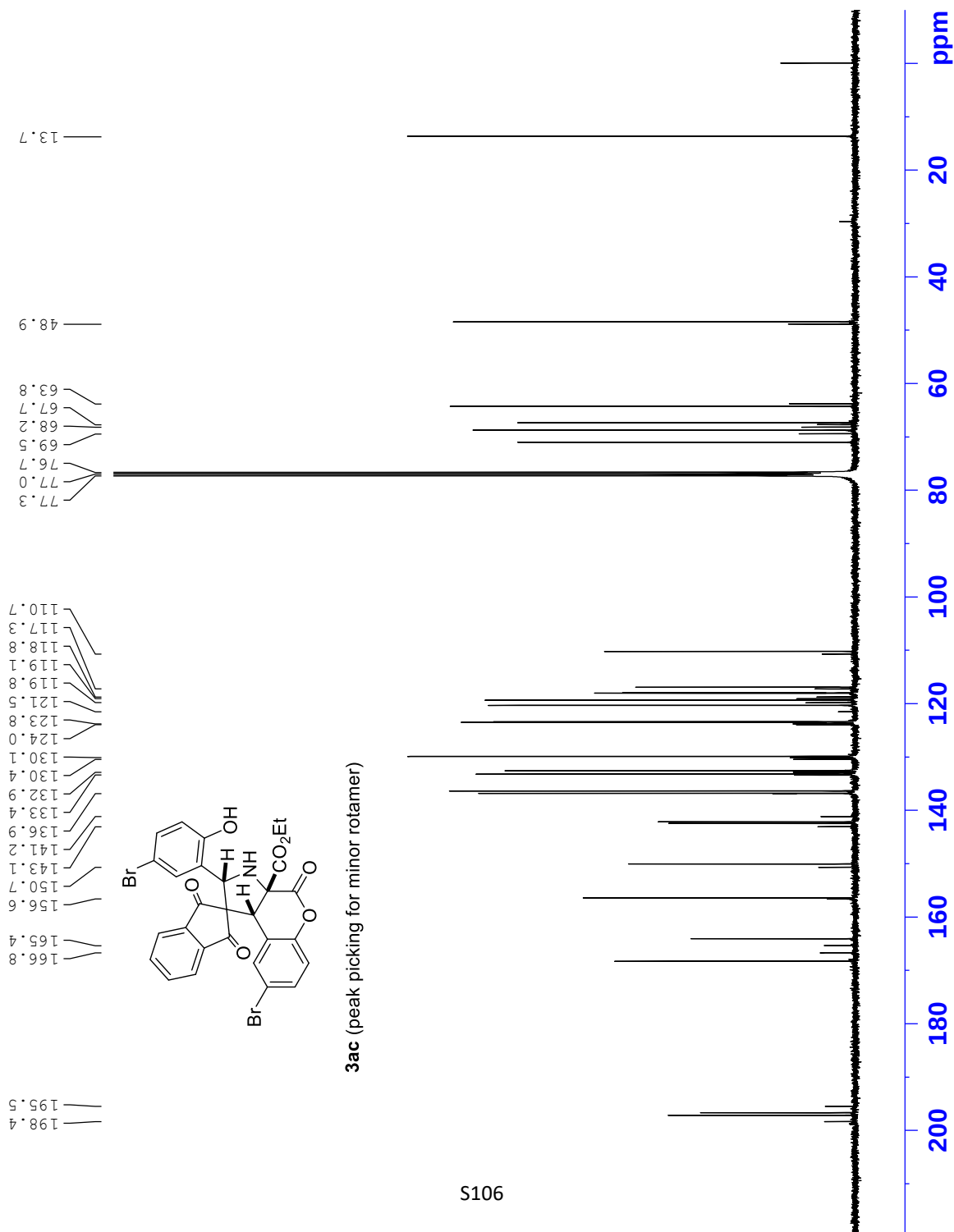
Current Data Parameters
NAME liul46
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170306
Time 22.06
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 14161
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 5792.6
DW 20.800 usec
DE 6.50 usec
TE 295.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.60 dB
PL12 15.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127720 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

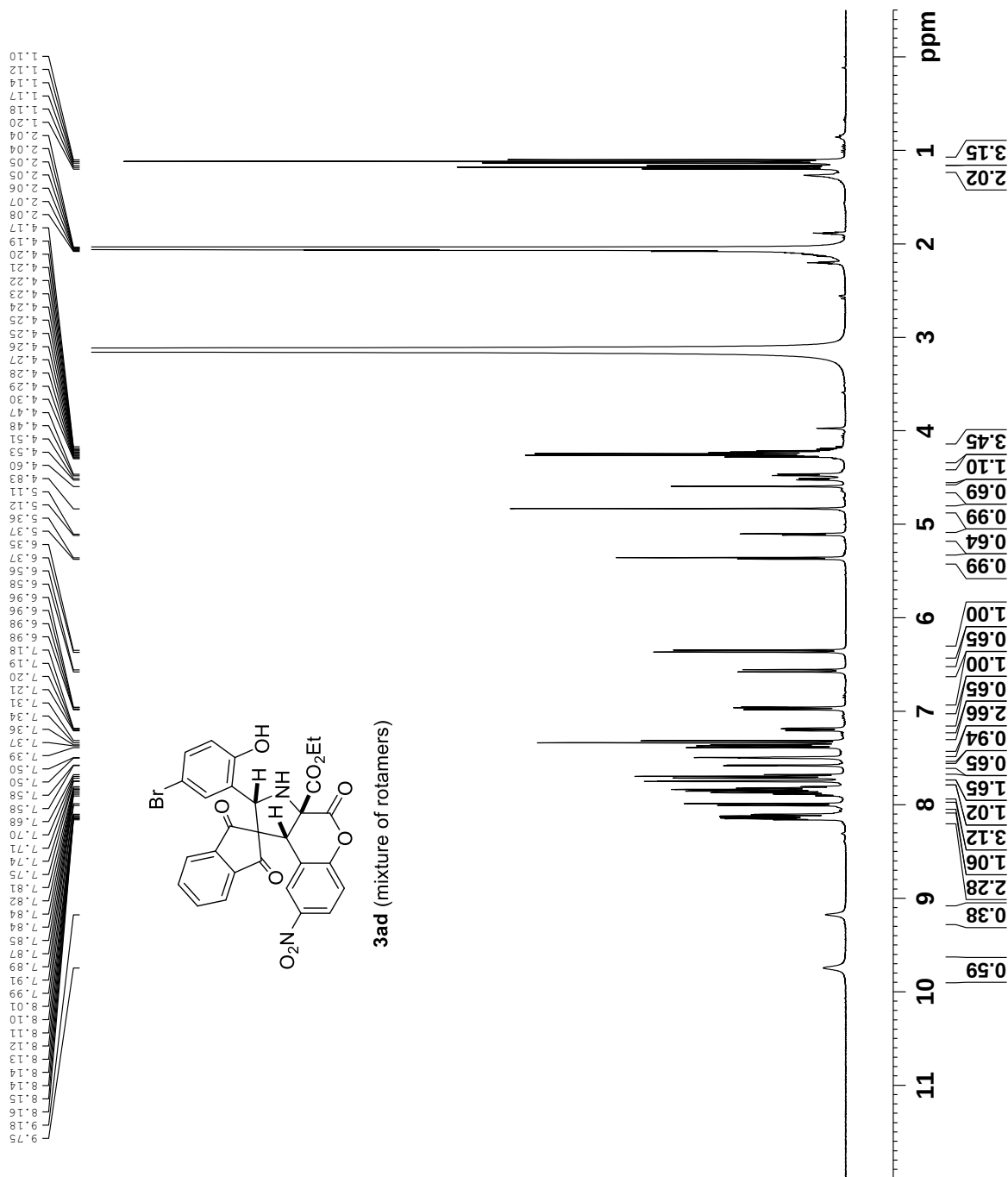


Current Data Parameters
NAME Bony271
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180501
Time_ 22.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT Acetone
NS 32
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 51.8
DW 69.333 usec
DE 10.50 usec
TE 298.1 K
D1 2.0000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.0000000 W

F2 - Processing parameters
SI 16384
SF 400.1300074 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



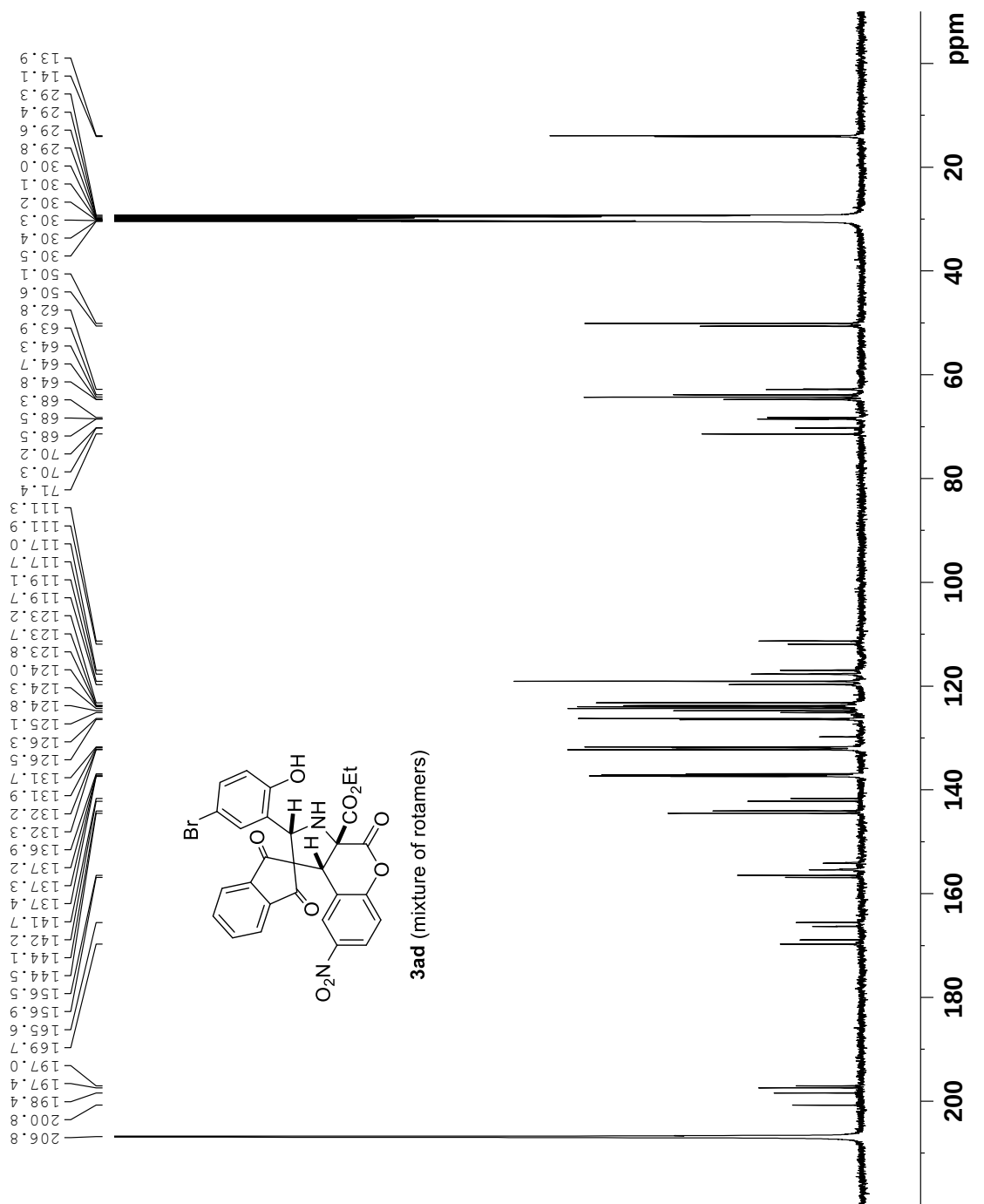
Current Data Parameters
NAME Bony271
EXPNO 2
PROCNO 1

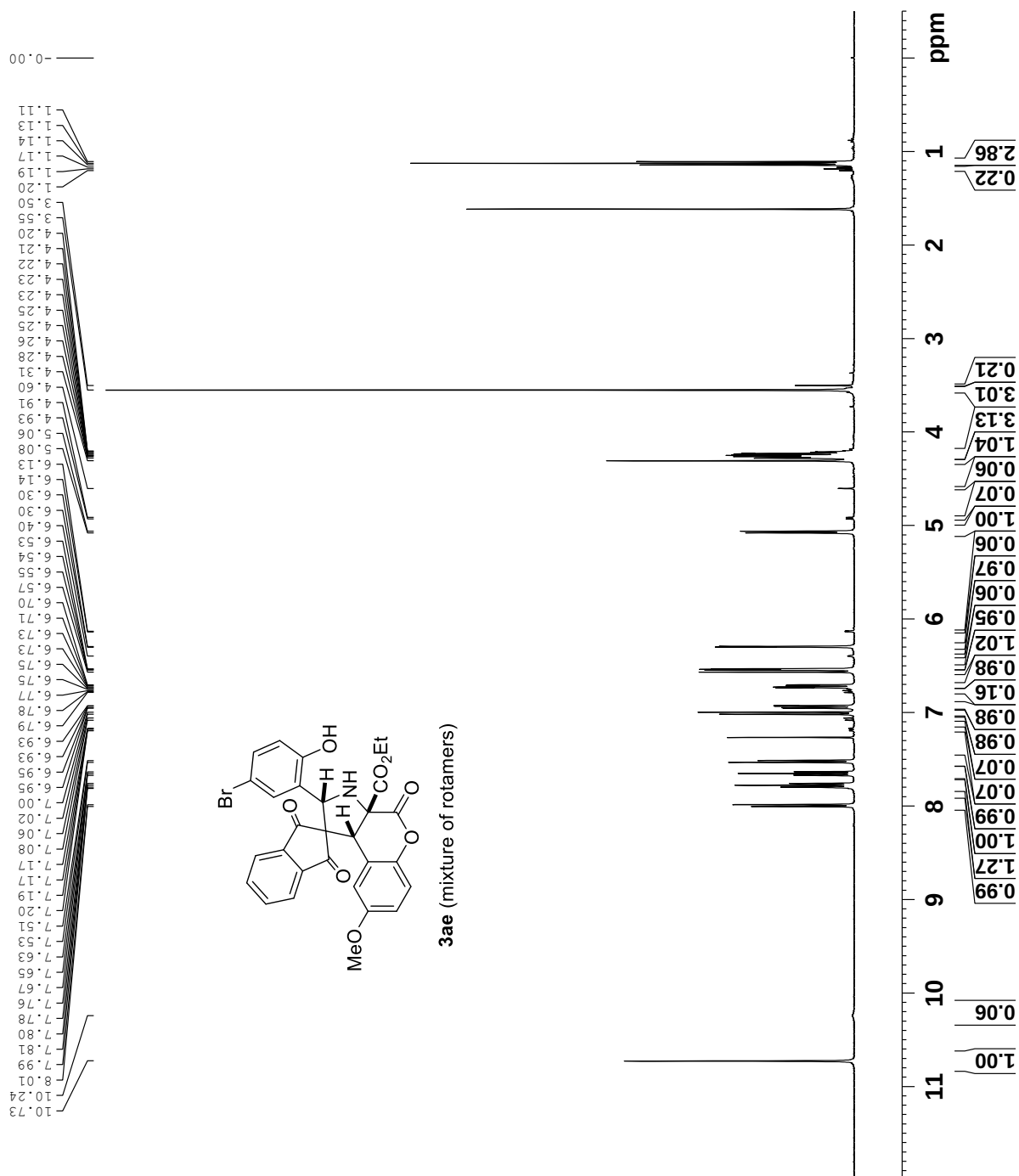
F2 - Acquisition Parameters
Date_ 20180501
Time_ 22.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT Acetone
NS 12829
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

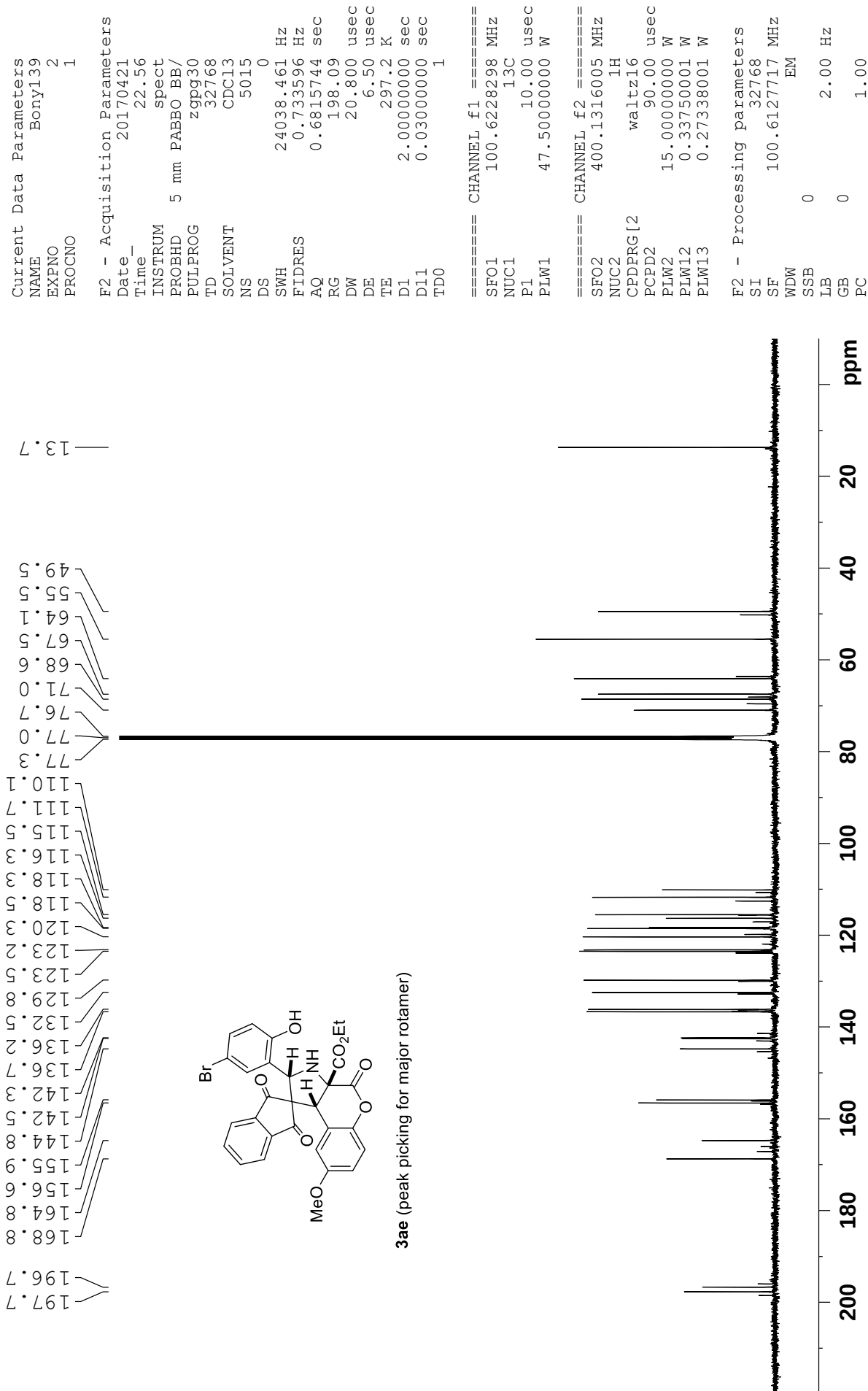
===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6126843 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00







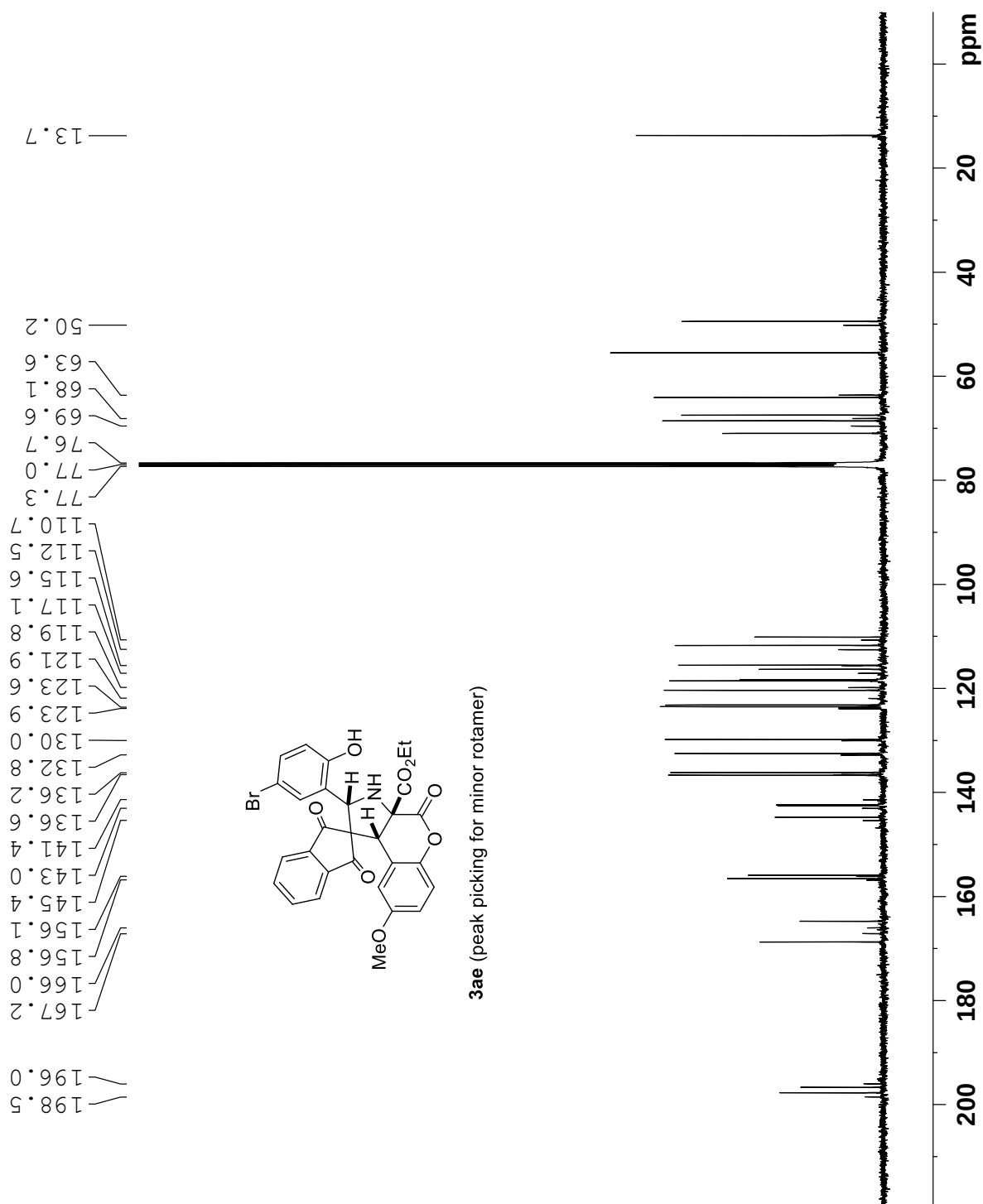
Current Data Parameters
NAME Bonyl39
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170421
Time 22.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 5015
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

==== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG12 waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127717 MHz
WDW EM
SSB 0
LB 0 2.00 Hz
GB 0
PC 1.00

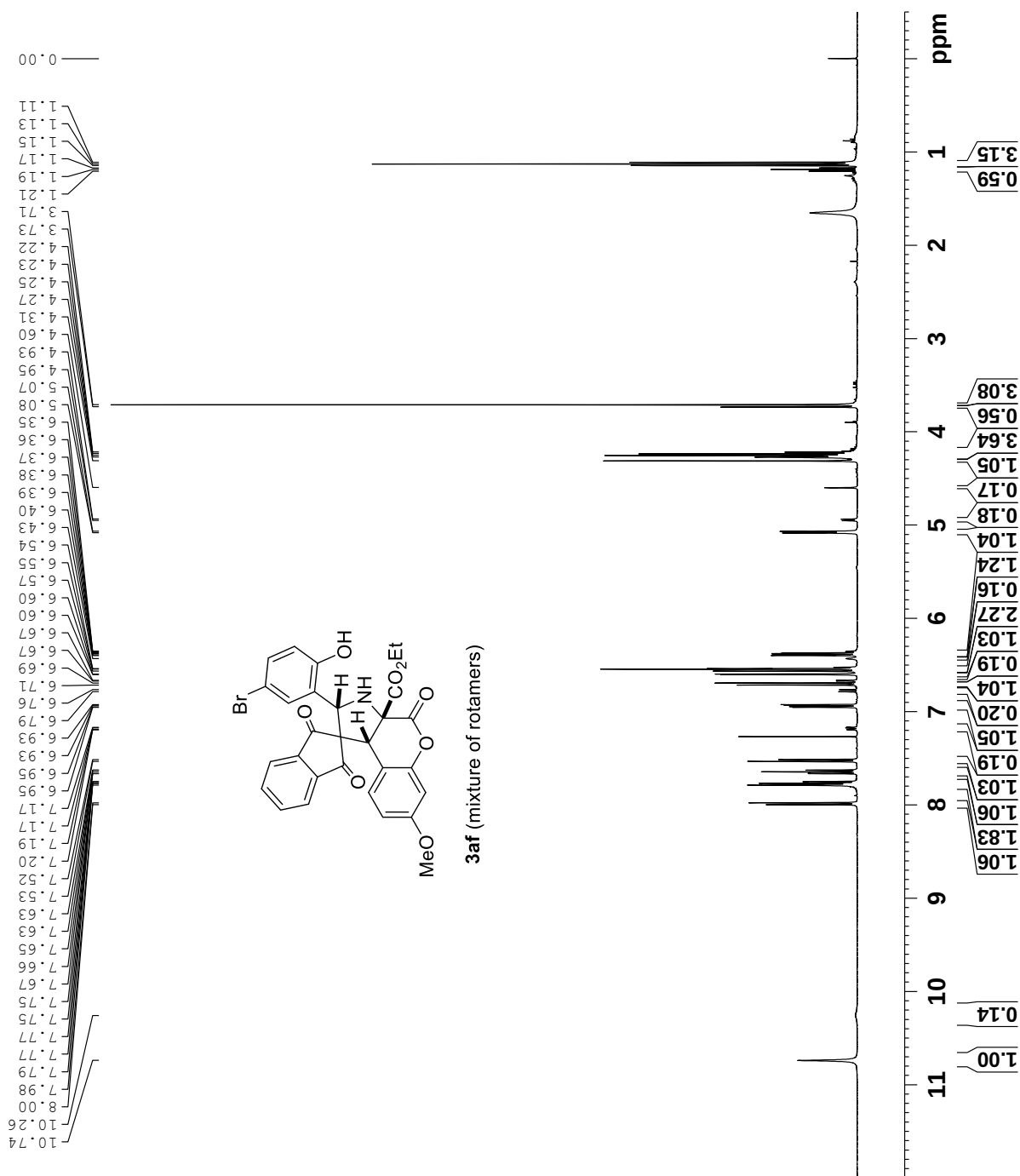


Current Data Parameters
NAME vicky174
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170503
Time 0.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 99.72
DW 69.333 usec
DE 10.50 usec
TE 297.0 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1300062 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



Current Data Parameters
NAME vicky174
EXPNO 4
PROCNO 1

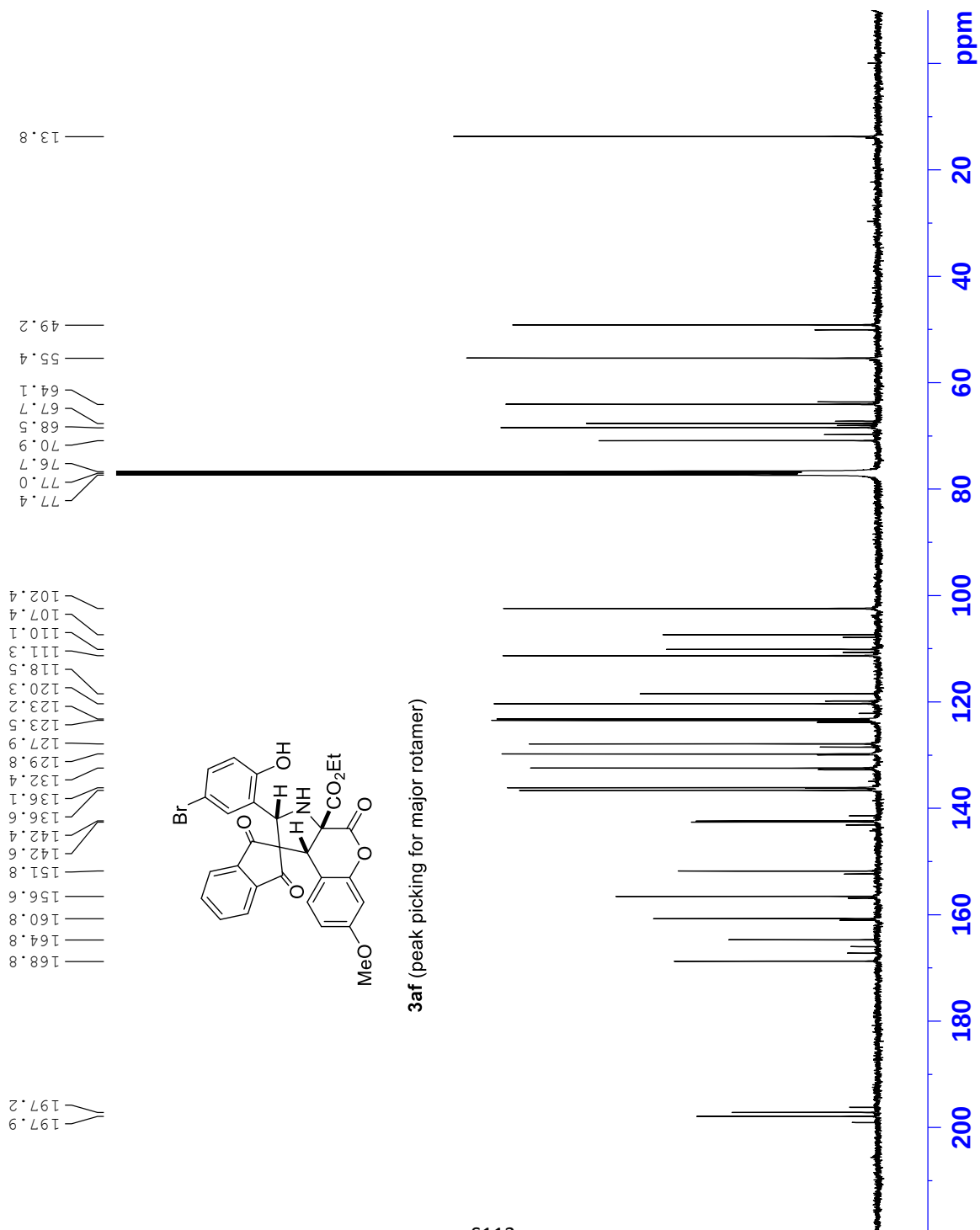
F2 - Acquisition Parameters

Date_ 20170503
Time_ 0.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 11807
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



Current Data Parameters
NAME vicky174
EXPNO 4
PROCNO 1

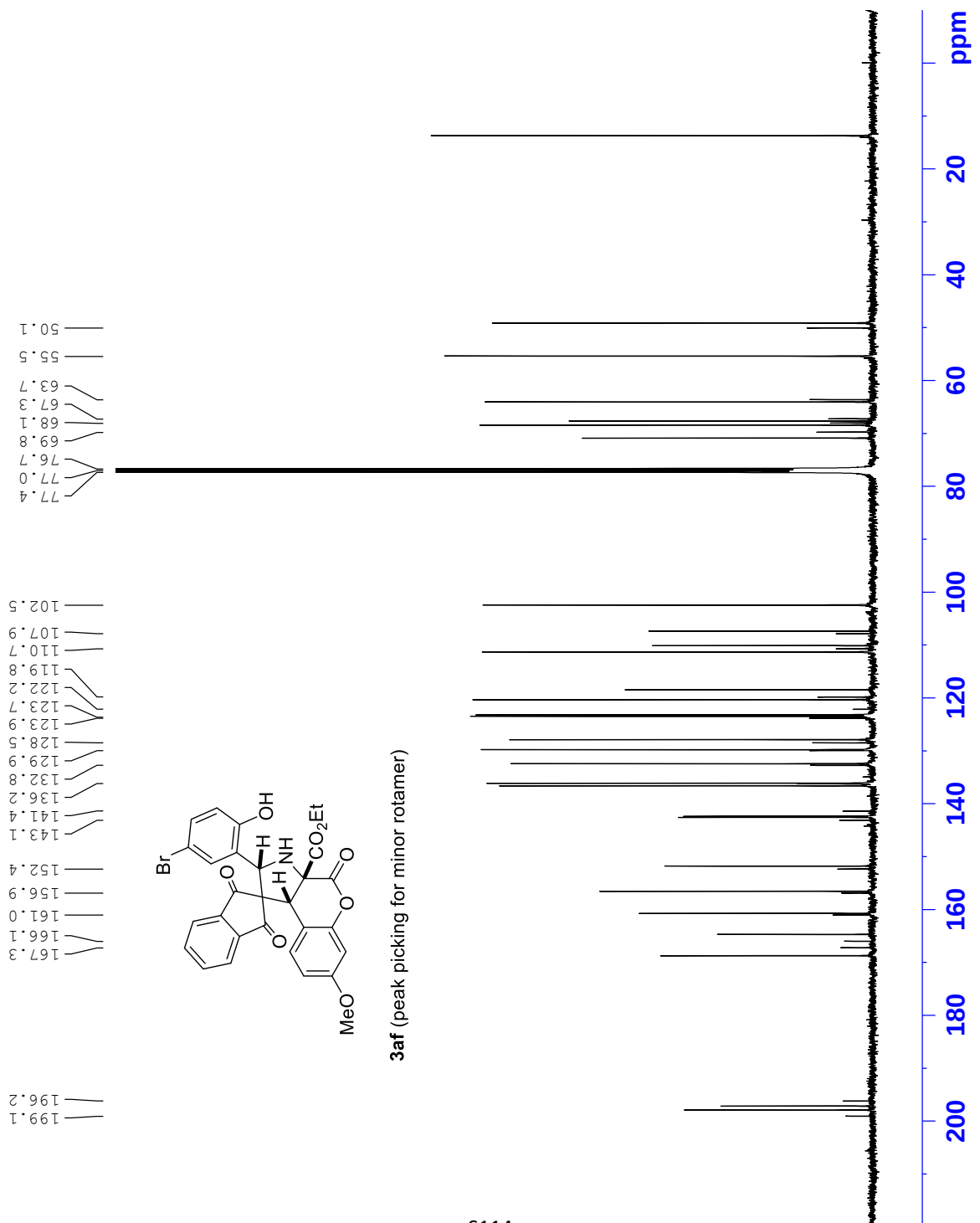
F2 - Acquisition Parameters

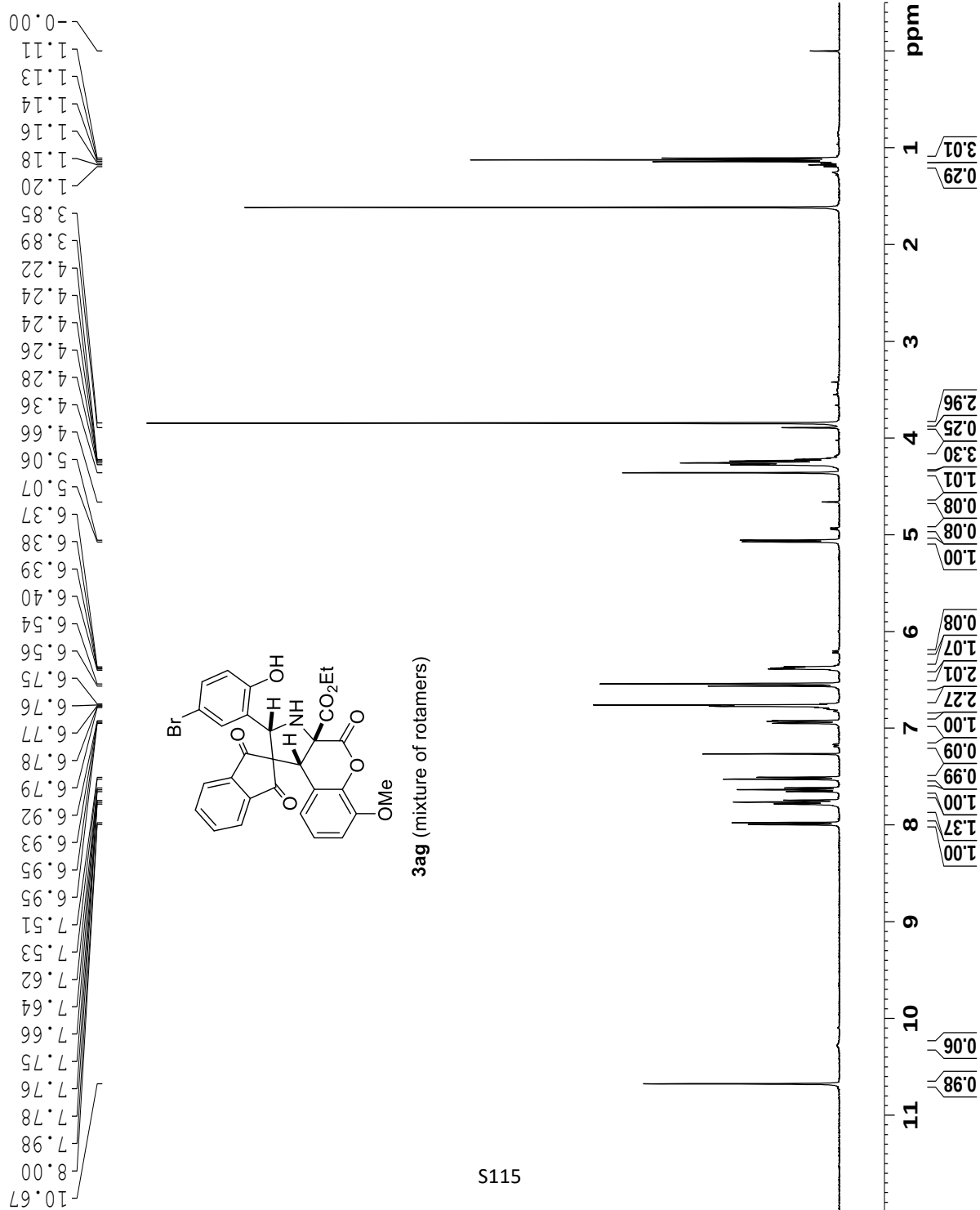
Date_ 20170503
Time_ 0.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 11807
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00





Current Data Parameters
NAME 3OMe
EXPNO 2
PROCNO 1

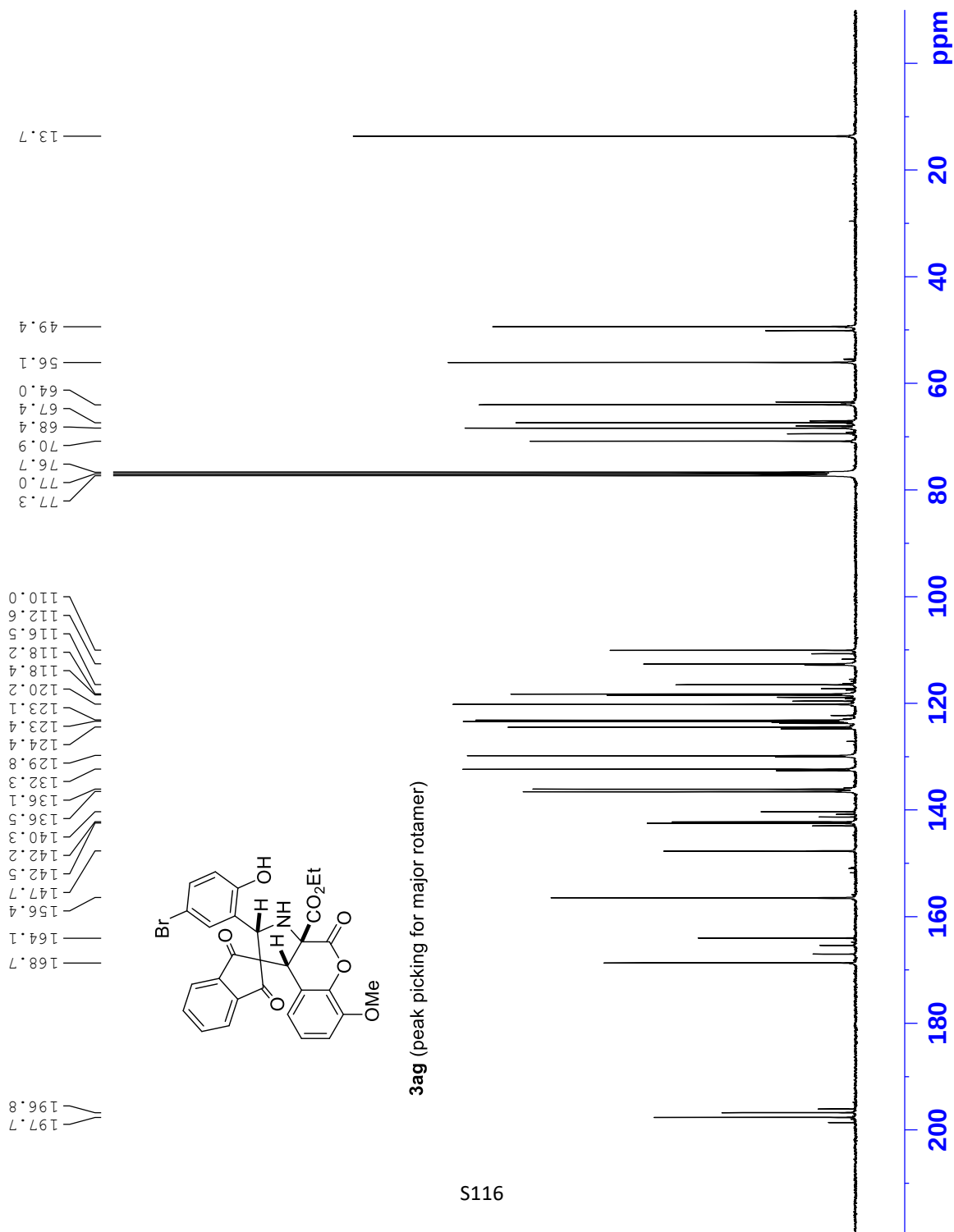
F2 - Acquisition Parameters

Date_ 20170416
Time 21.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16774
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127765 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



Current Data Parameters
 NAME 3OMe
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

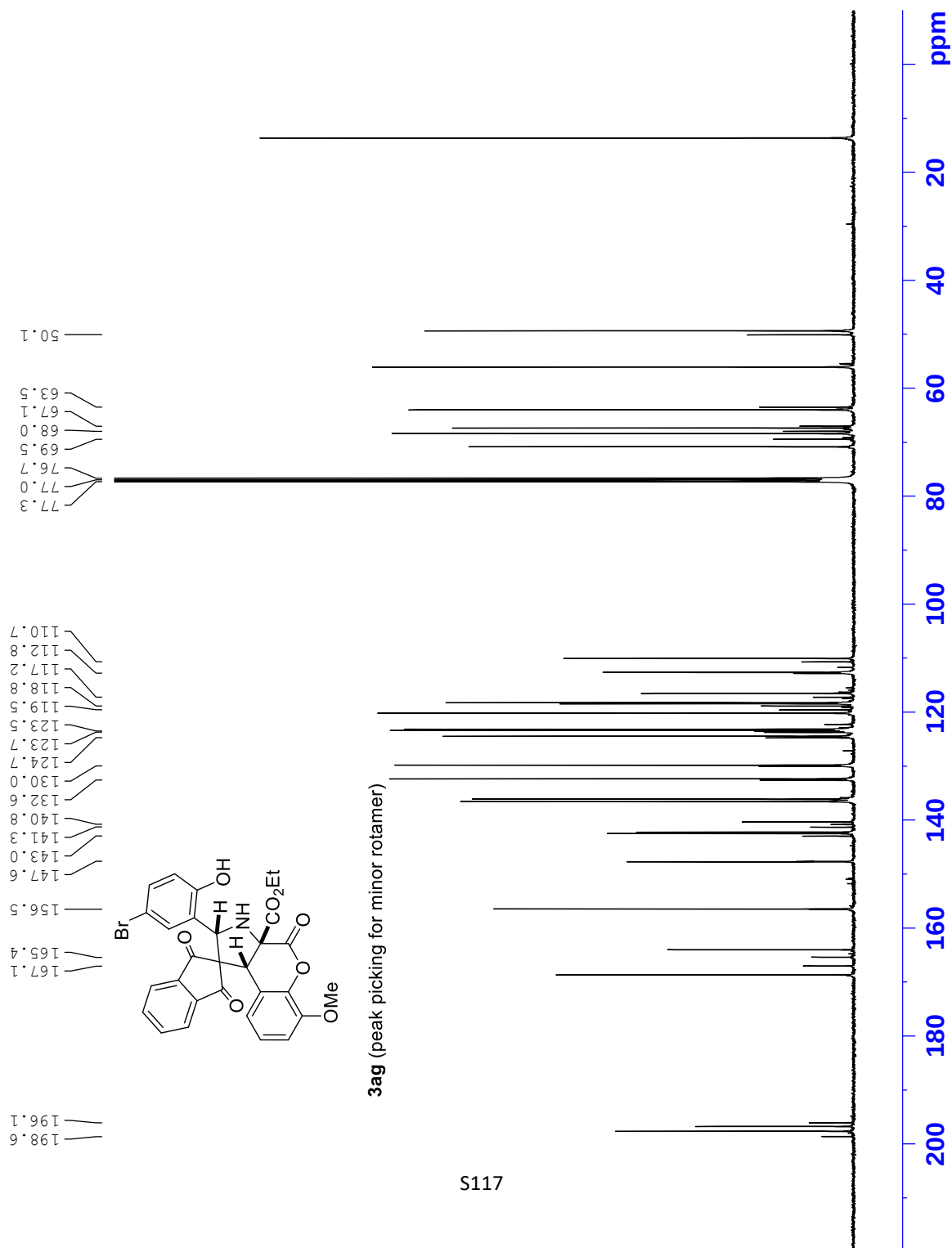
Date_ 20170416
 Time_ 21.19
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 16774
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6815744 sec
 RG 198.09
 DW 20.800 usec
 DE 6.50 usec
 TE 299.3 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

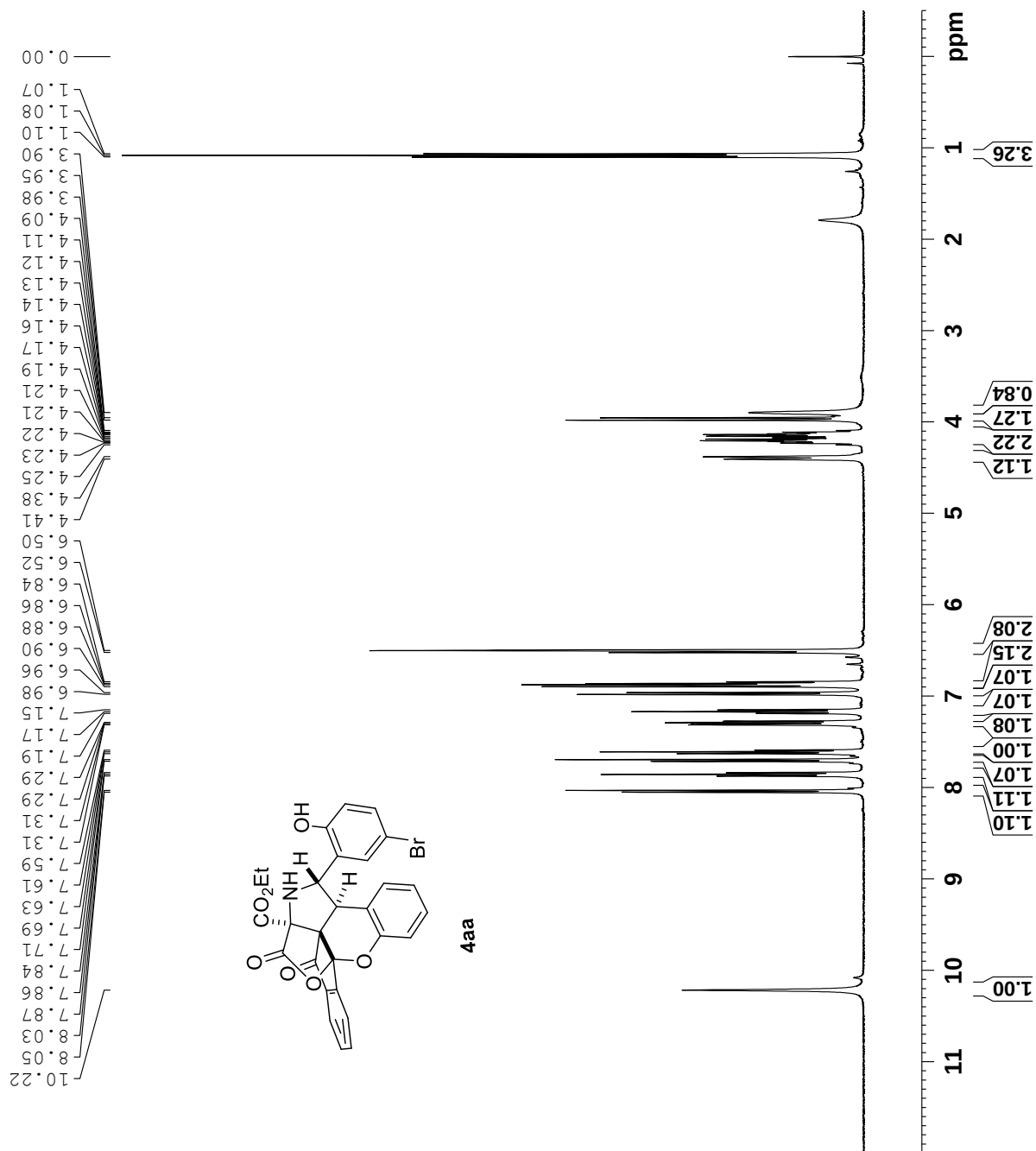
===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 47.50000000 W

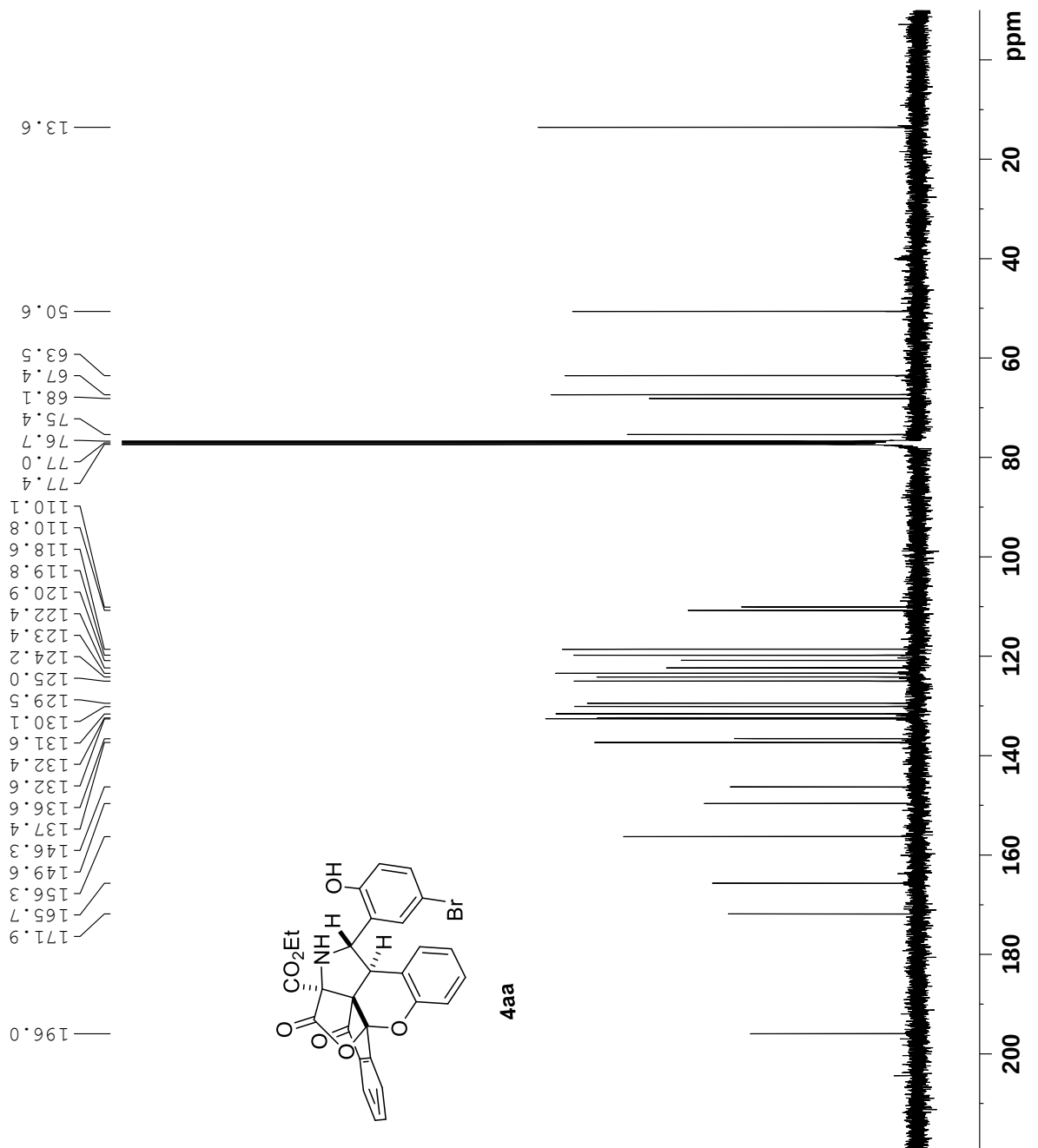
===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 15.00000000 W
 PLW12 0.33750001 W
 PLW13 0.27338001 W

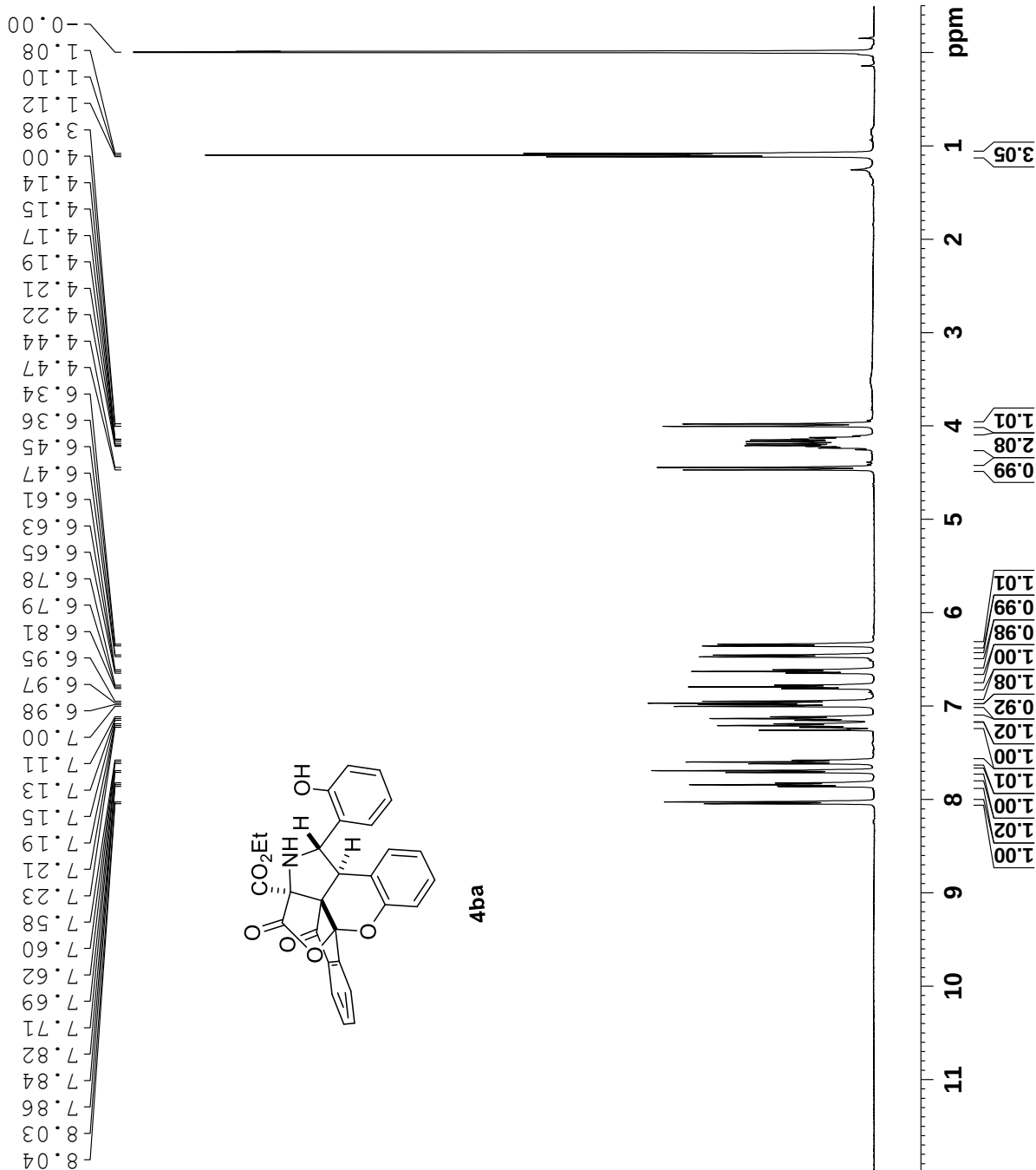
F2 - Processing parameters

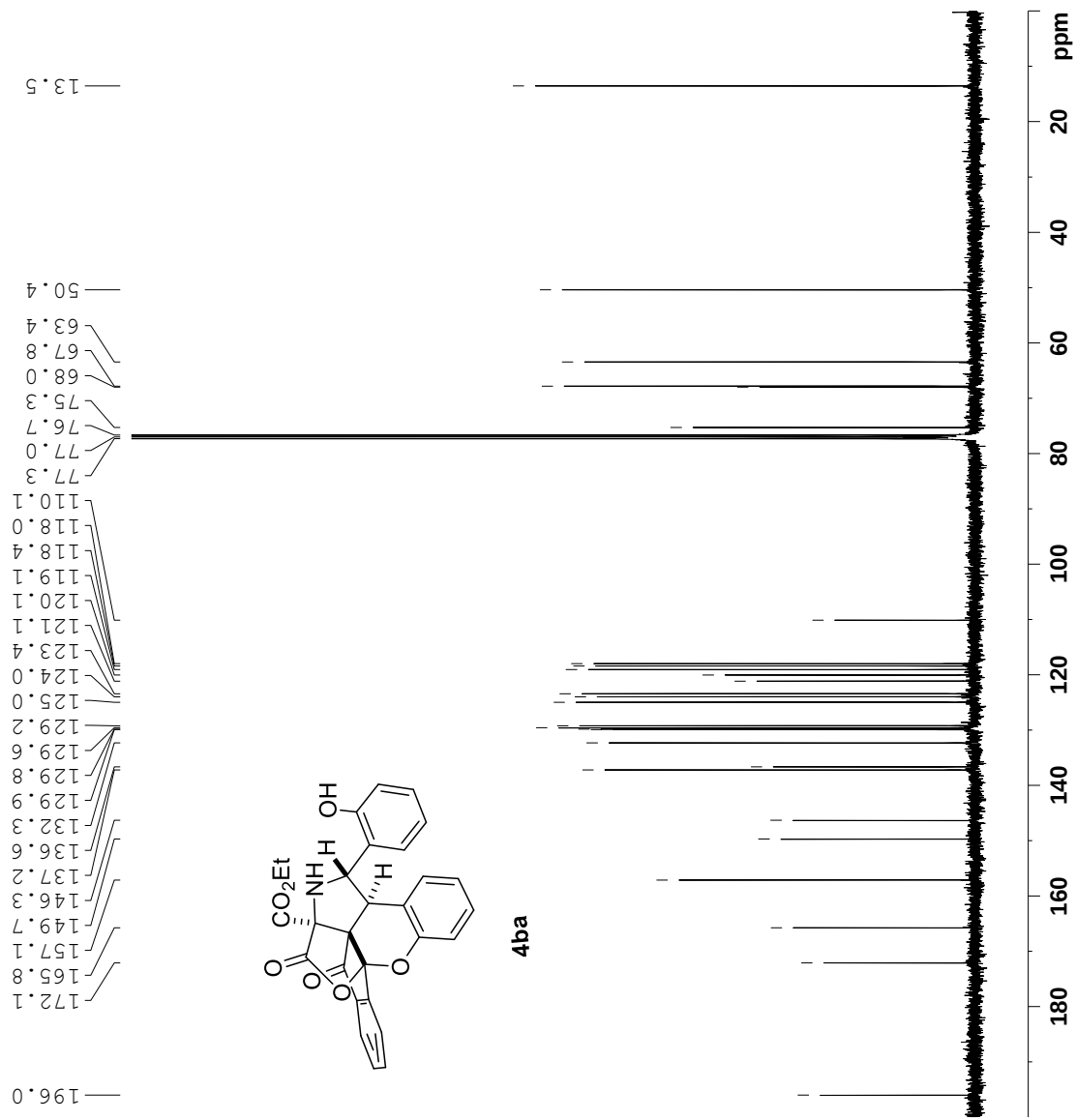
SI 32768
 SF 100.6127769 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.00











Current Data Parameters
 NAME hank097
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171021
 Time_ 17.14
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 1330
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6815744 sec
 RG 5792.6
 DW 20.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 10.45 usec
 PL1 7.00 dB
 SFO1 100.6233325 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0 dB
 PLI2 15.00 dB
 PLI3 20.00 dB
 SFO2 400.1316005 MHz

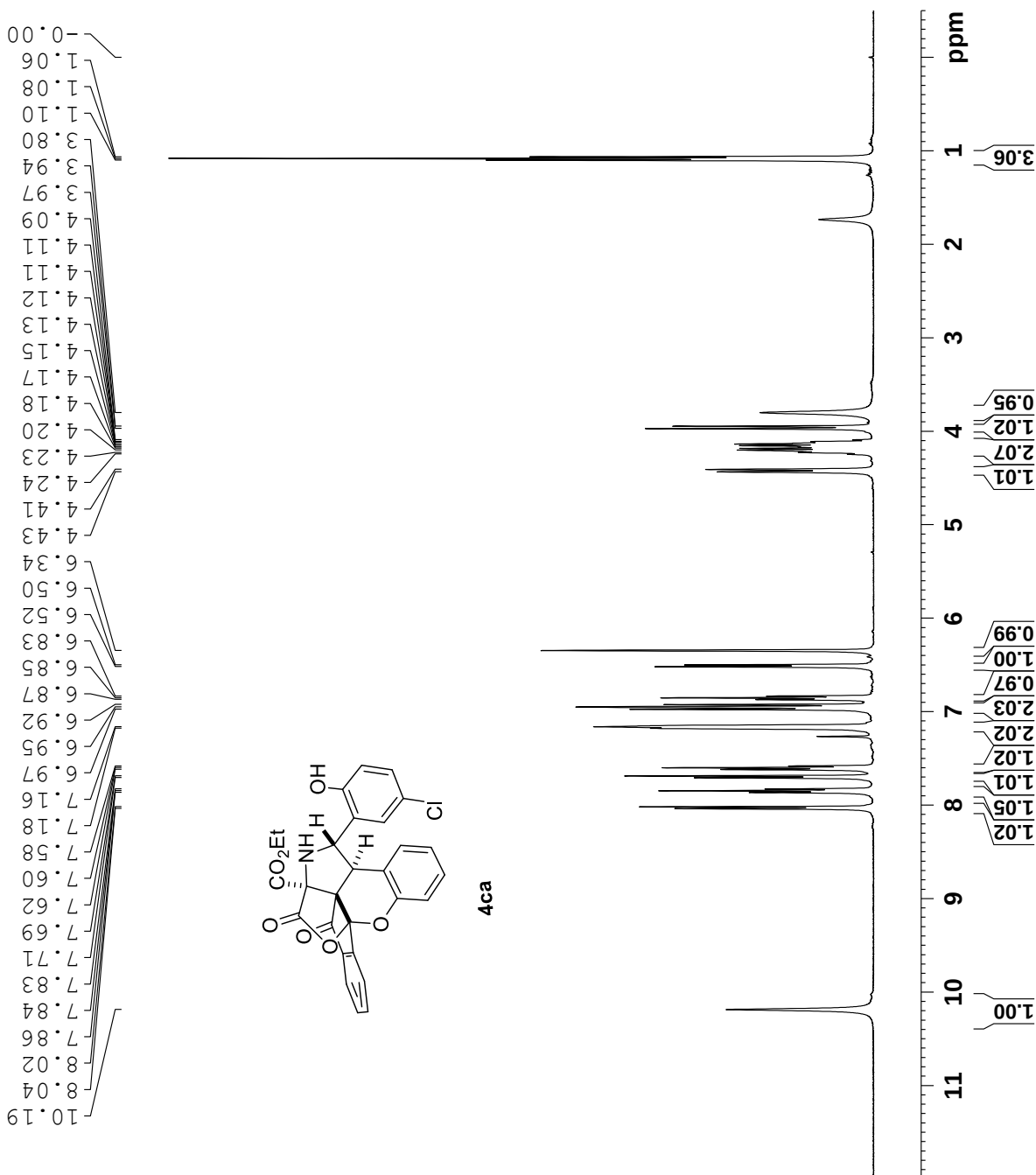
F2 - Processing parameters
 SI 32768
 SF 100.6127727 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME hank102
 EXPNO 7
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171117
 Time_ 21.22
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 7246.377 Hz
 FIDRES 0.221142 Hz
 AQ 2.260921 sec
 RG 71.8
 DW 69.000 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 0 dB
 SFO1 400.132408 MHz

F2 - Processing parameters
 SI 16384
 SF 400.130080 MHz
 WDW EM
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00



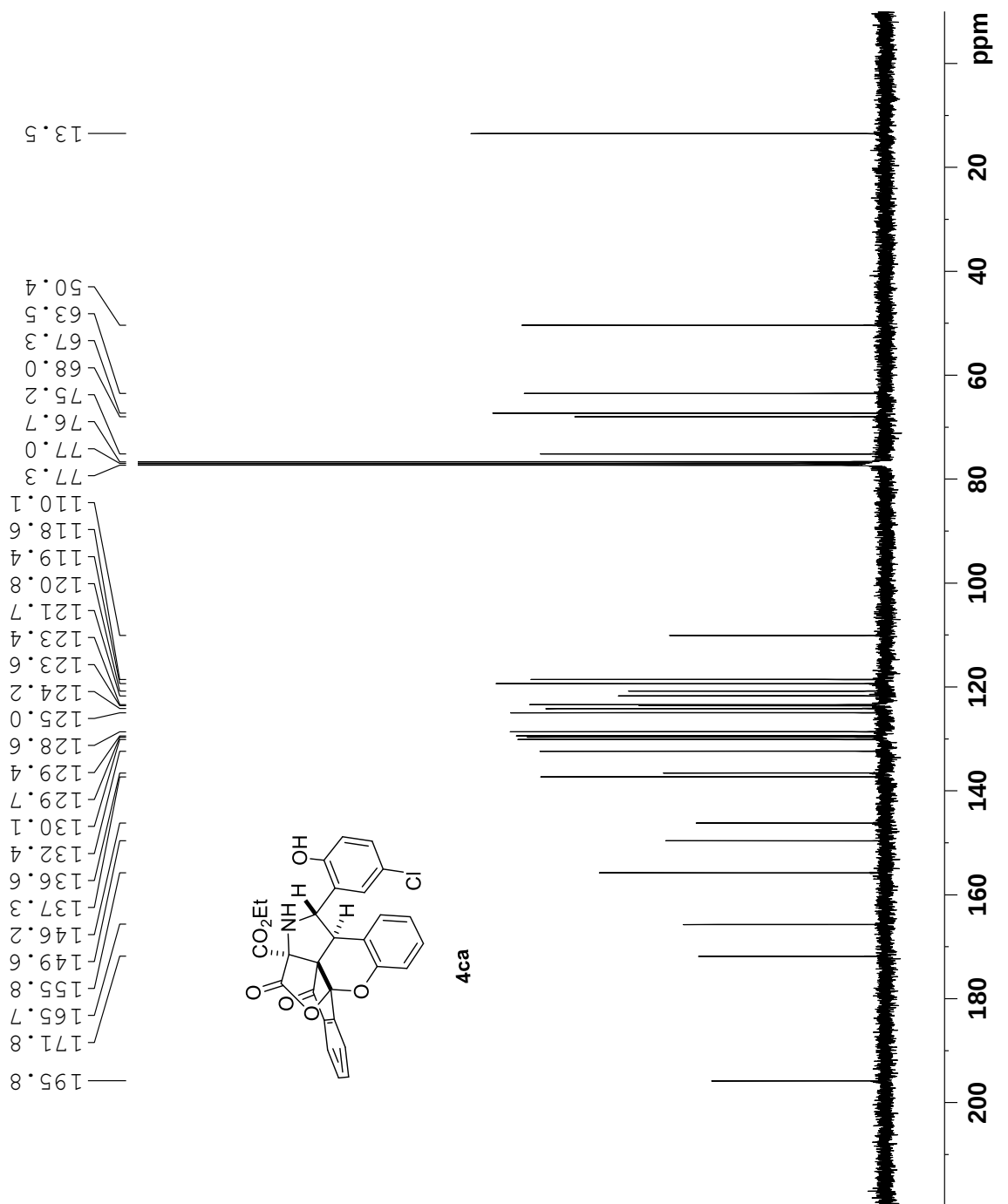
Current Data Parameters
NAME hank102
EXPNO 8
PROCNO 1

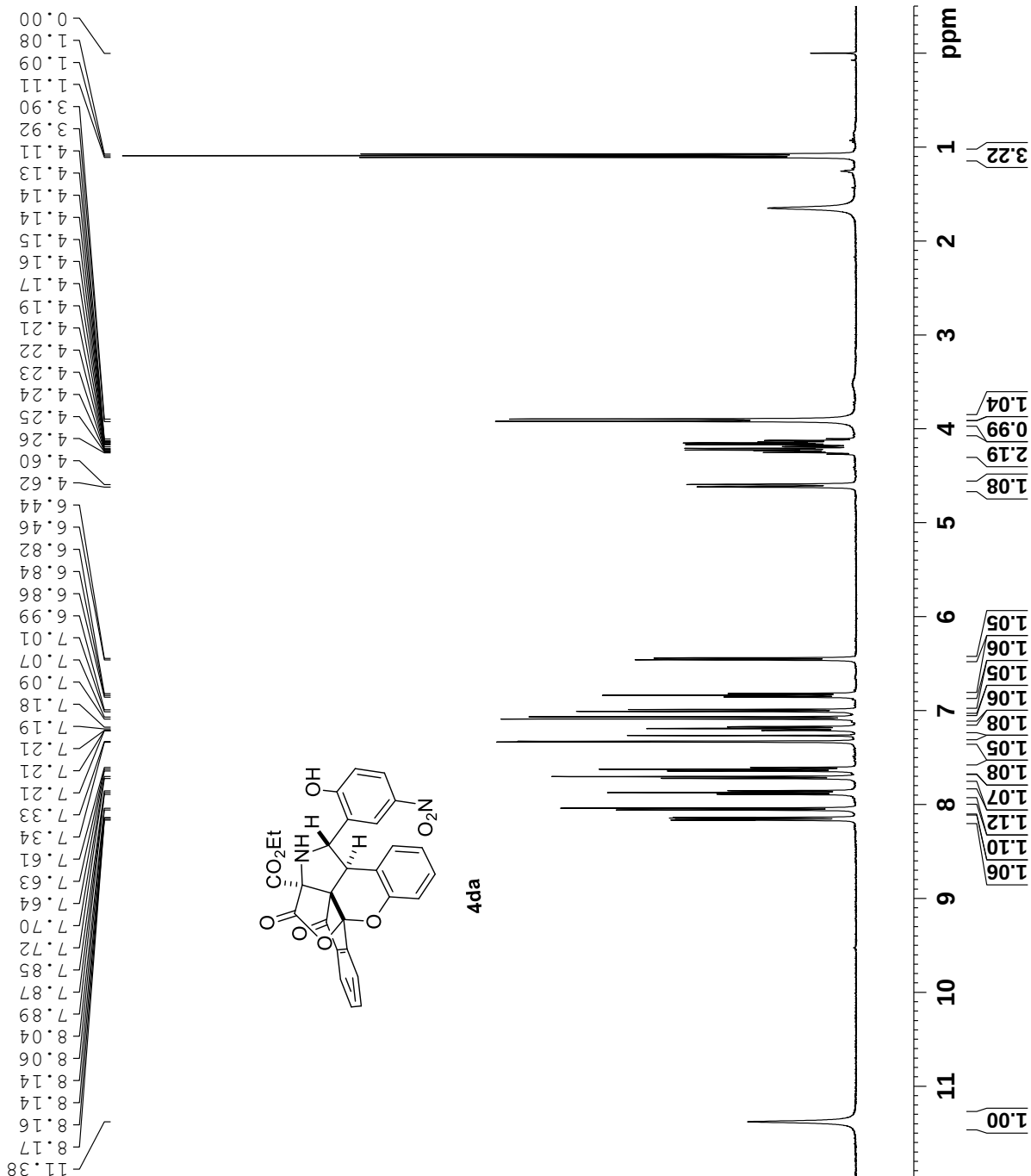
F2 - Acquisition Parameters
Date_ 20171117
Time_ 21.23
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1151
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 4096
DW 20.800 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PL1 7.00 dB
SFO1 100.623325 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.60 dB
PL12 15.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127749 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00





Current Data Parameters
 NAME hank103
 EXPNO 21
 PROCNO 1

F2 - Acquisition Parameters

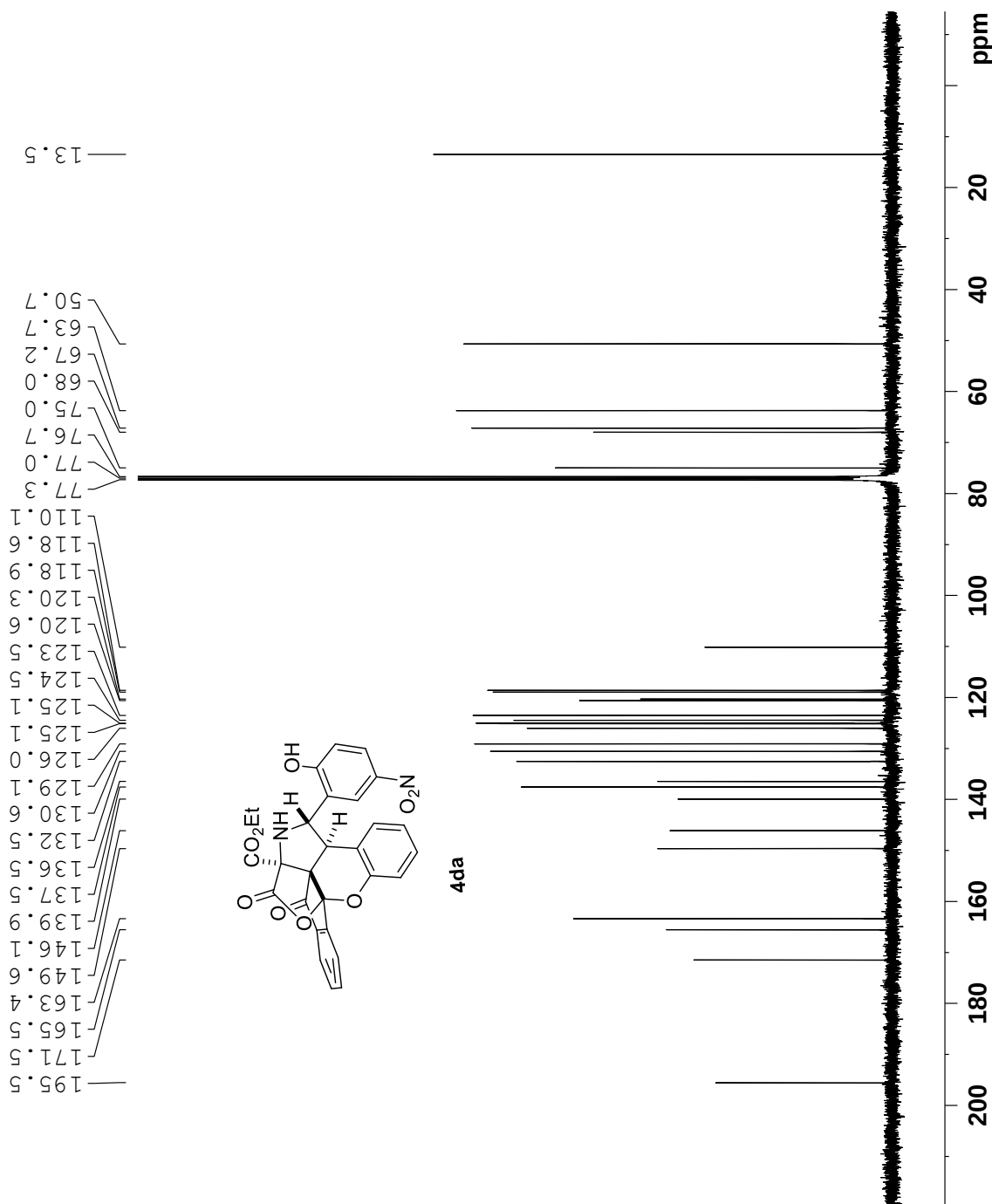
Date_ 20180222
 Time_ 11.59
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 2500
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6815744 sec
 RG 5792.6
 DW 20.800 usec
 DE 6.50 usec
 TE 295.6 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

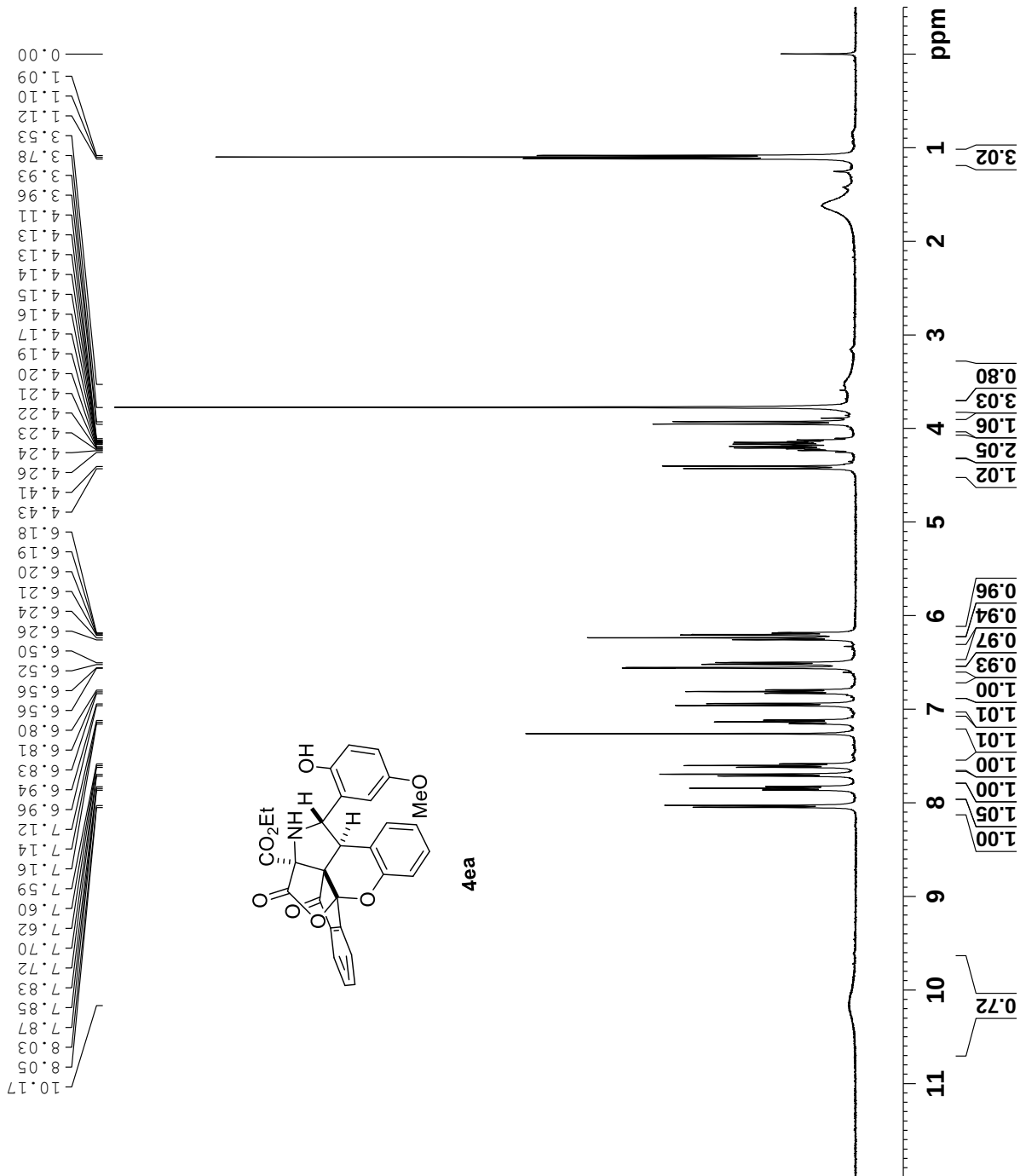
===== CHANNEL f1 =====
 NUC1 13C
 P1 10.45 usec
 PL1 7.00 dB
 SF01 100.6233325 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0 dB
 PL12 15.00 dB
 PL13 20.00 dB
 SF02 400.1316005 MHz

F2 - Processing parameters

SI 32768
 SF 100.6127736 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00





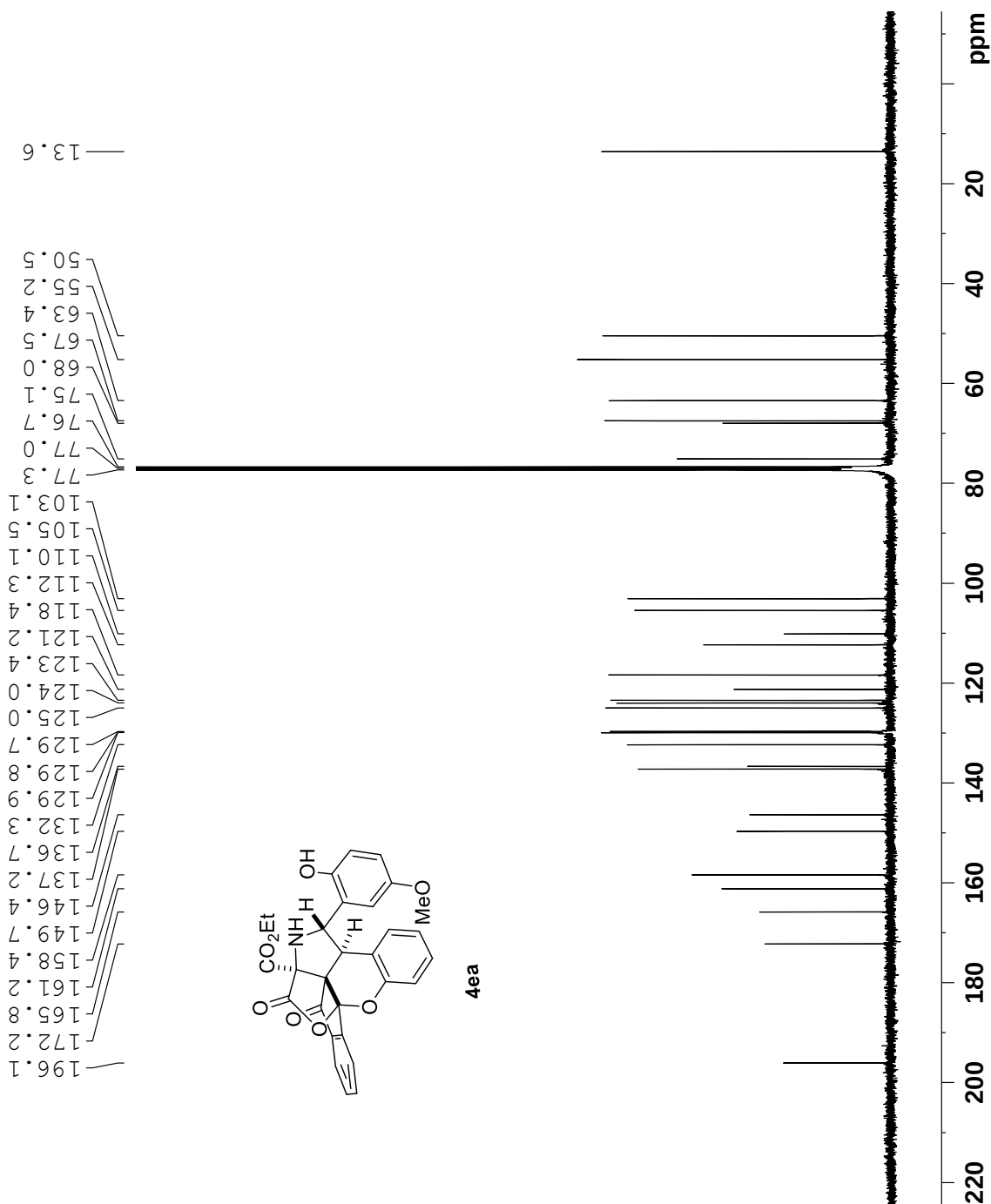
Current Data Parameters
NAME B301
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180221
Time_ 21:58
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 13599
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 5792.6
DE 20.800 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.45 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0 dB
PL12 15.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



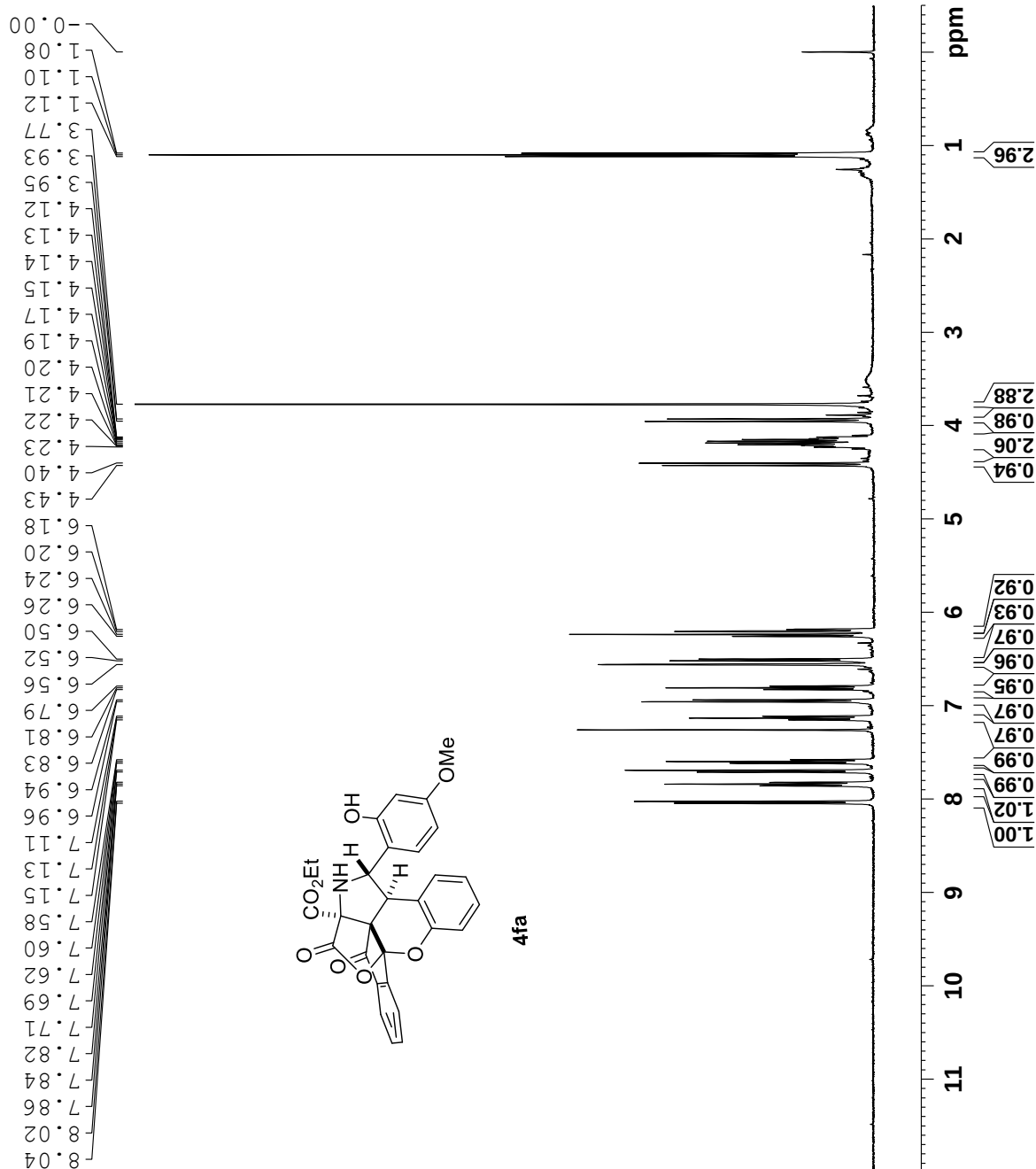
Current Data Parameters
 NAME B3
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180302
 Time 21.57

INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 7246.377 Hz
 FIDRES 0.221142 Hz
 AQ 2.260921 sec
 RG 181
 DW 69.000 usec
 DE 6.50 usec
 TE 299.9 K
 D1 2.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 0 dB
 SFO1 400.1324008 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300101 MHz
 WDW EM
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00



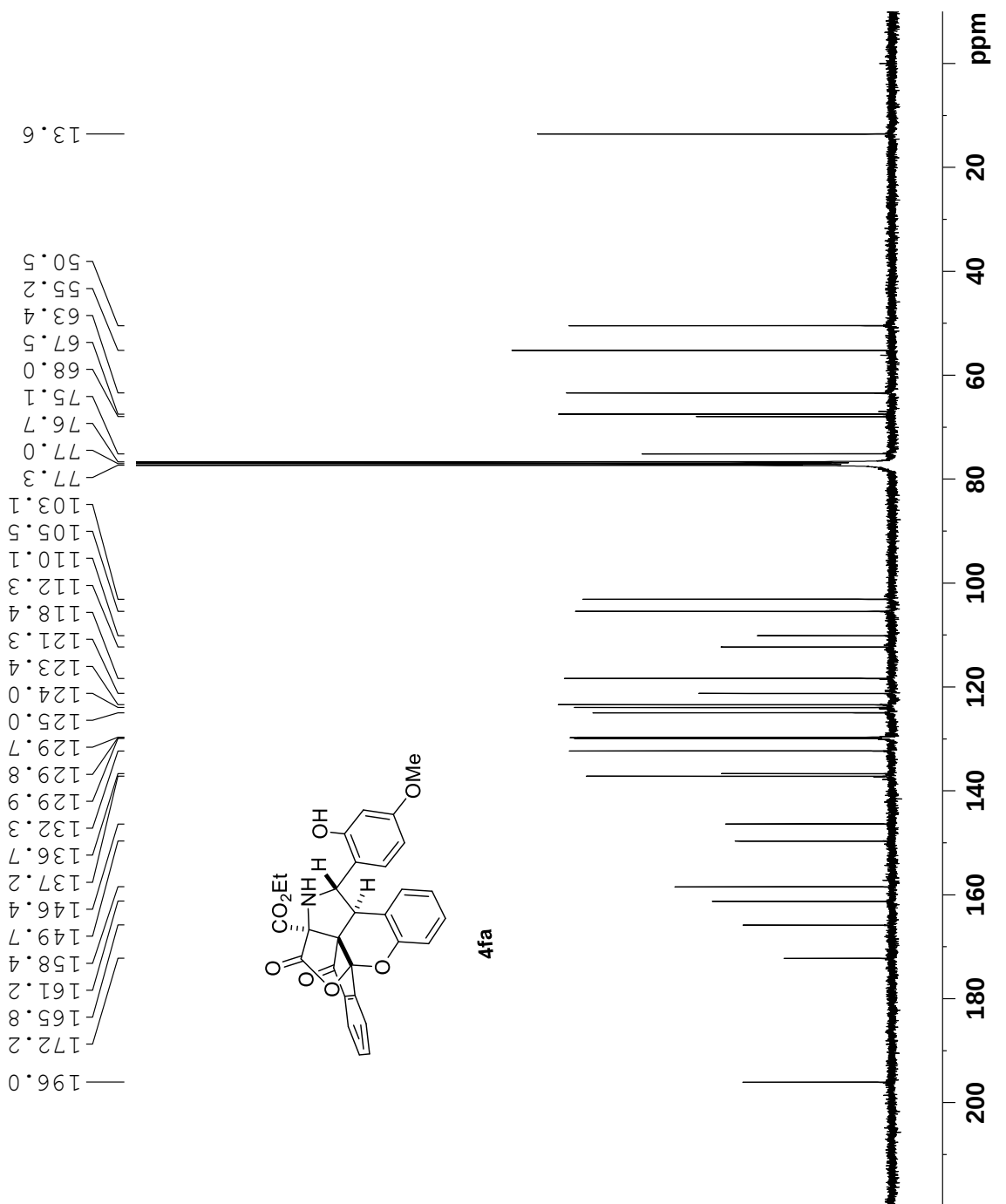
Current Data Parameters
 NAME B3
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180302
 Time_ 21:58
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDC13
 NS 15315
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6815744 sec
 RG 5792.6
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.45 usec
 PL1 7.00 dB
 SFO1 100.6233325 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0 dB
 PL12 15.00 dB
 PL13 20.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00



Current Data Parameters
NAME Bony302
EXPNO 4
PROCNO 1

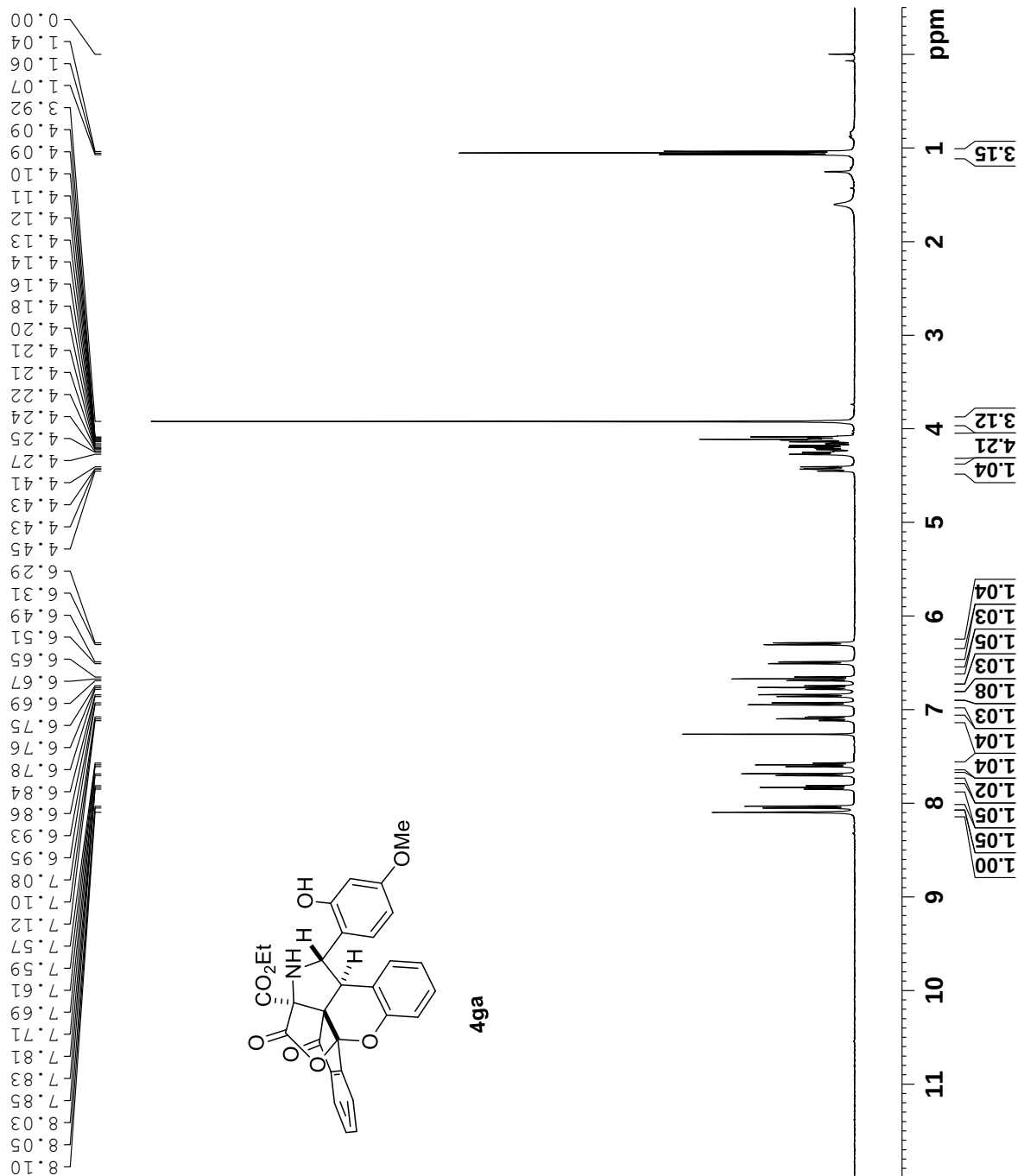
F2 - Acquisition Parameters

Date_ 20180224
Time 16.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 198.09
DW 69.333 usec
DE 10.50 usec
TE 297.4 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.00000000 W

F2 - Processing parameters

SI 16384
SF 400.1300084 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



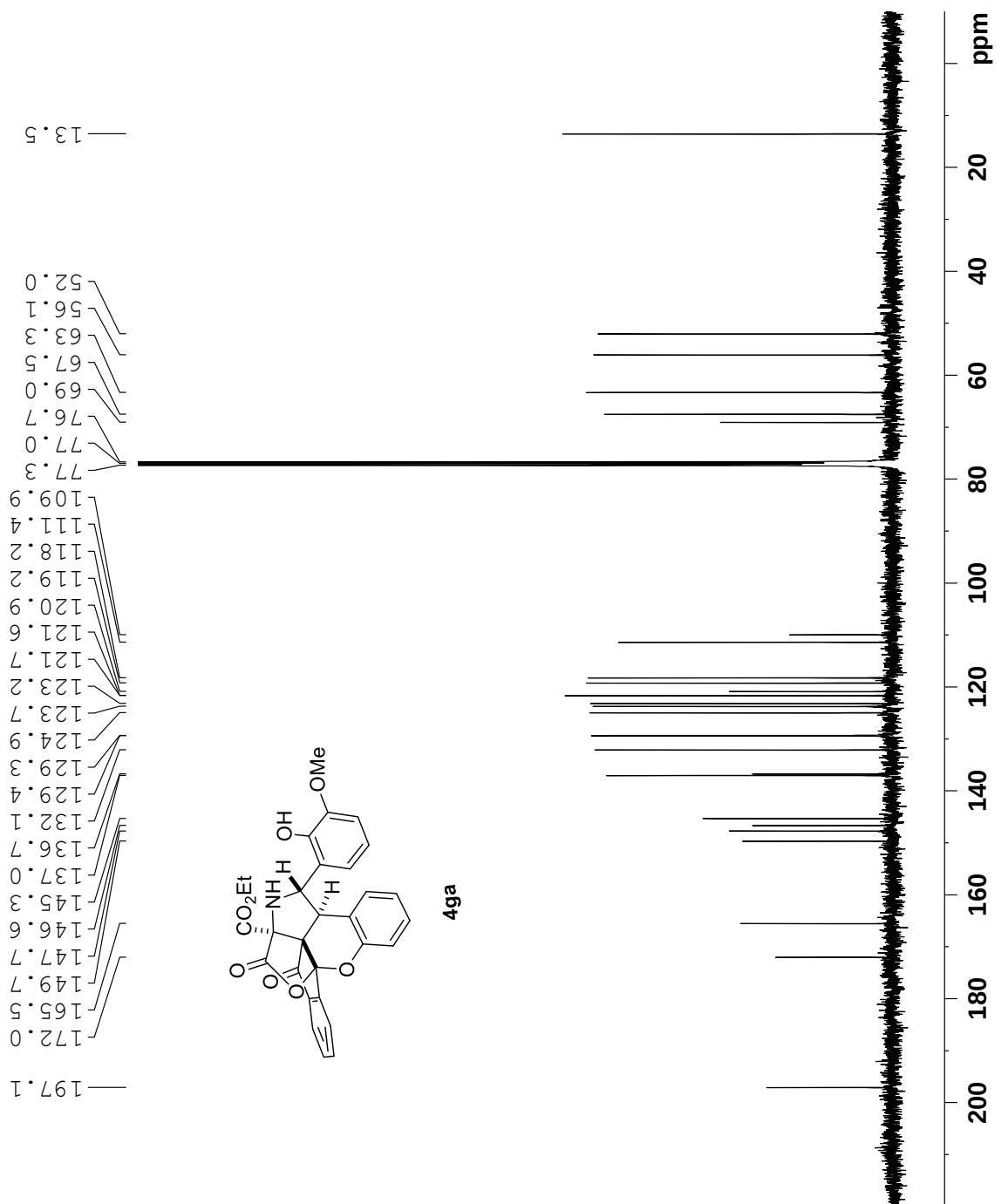
Current Data Parameters
NAME Bony302
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180224
Time_ 16.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1600
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

==== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127726 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

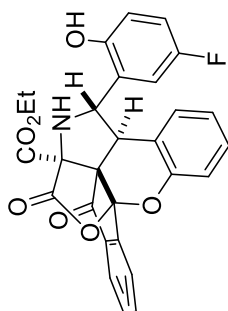


Current Data Parameters
NAME hank123
EXPNO 27
PROCNO 1

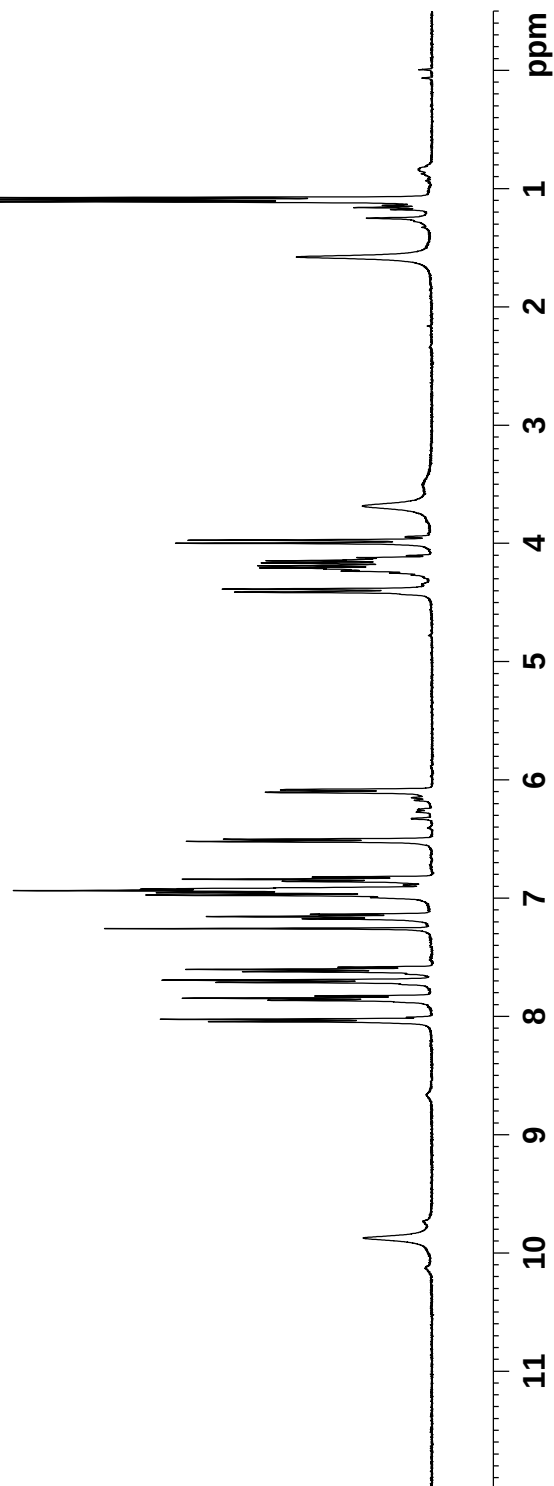
F2 - Acquisition Parameters
Date_ 20180518
Time_ 0.12
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.260921 sec
RG 161.3
DW 69.000 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 0 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300118 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



4ha (mixture of rotamers)



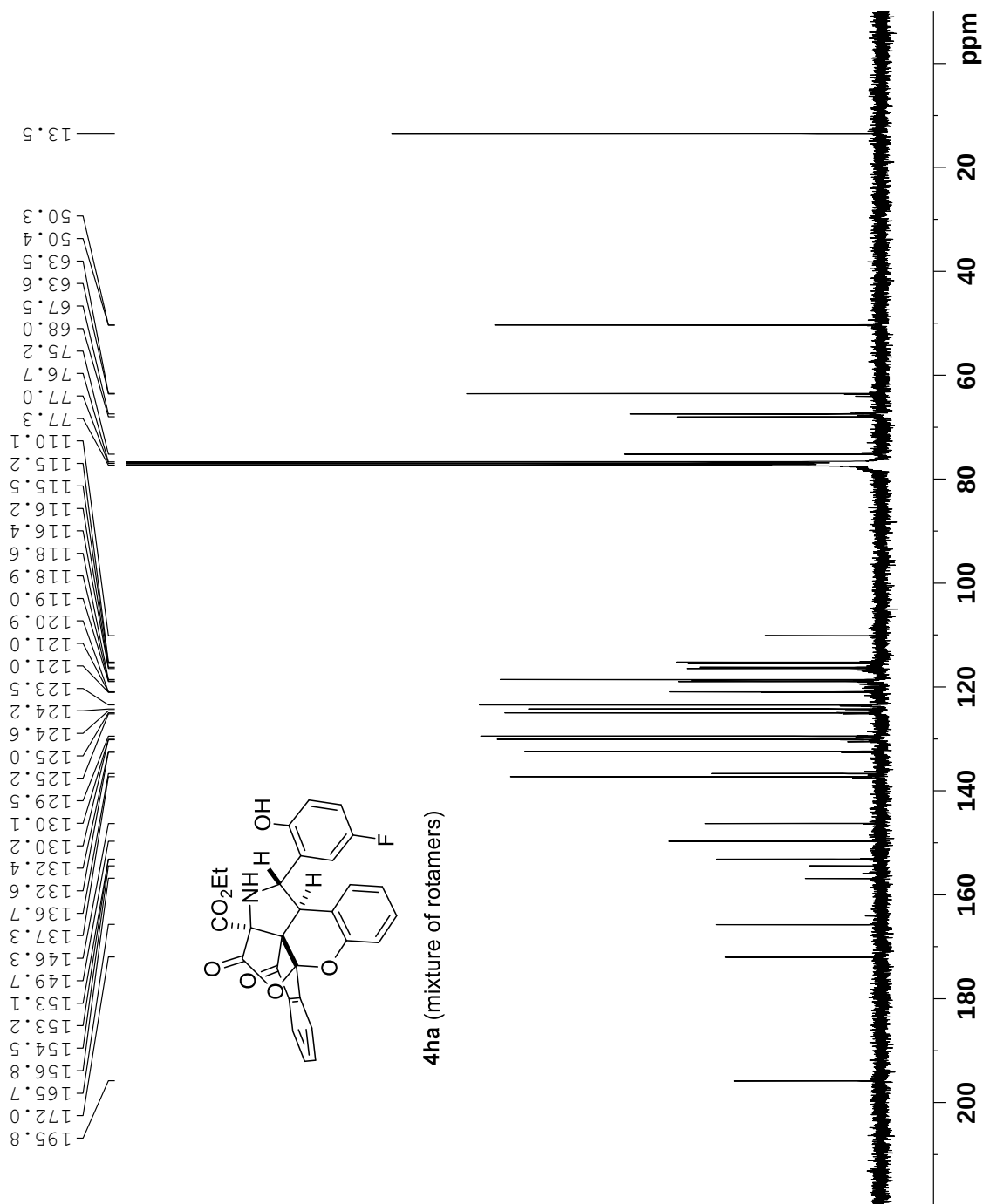
Current Data Parameters
 NAME hank123
 EXPNO 28
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180518
 Time_ 0.14
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 9096
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6815744 sec
 RG 5792.6
 DW 20.800 usec
 DE 6.50 usec
 TE 673.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.45 usec
 PL1 7.00 dB
 SFO1 100.623325 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0 dB
 PL12 15.00 dB
 PL13 20.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

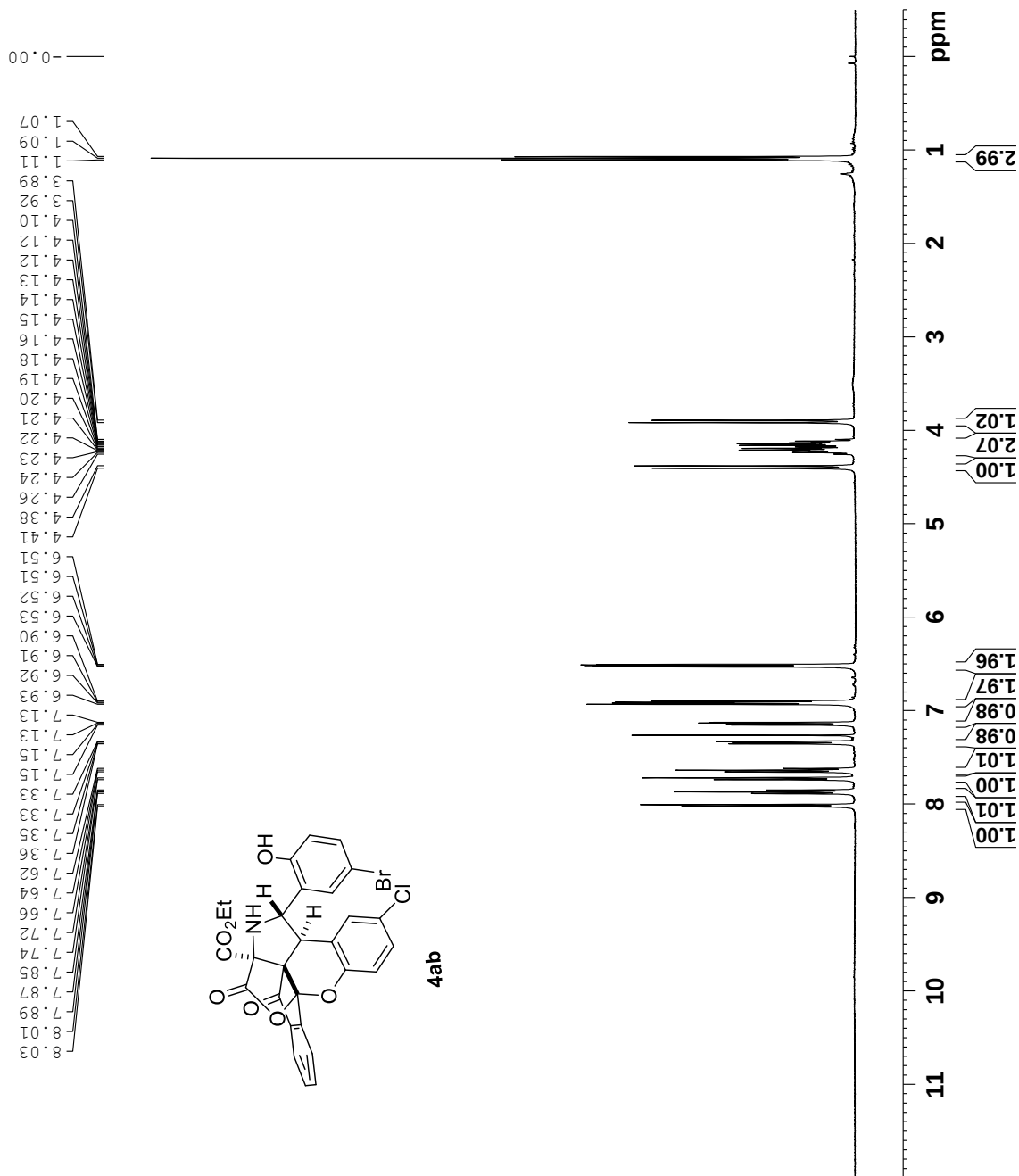


Current Data Parameters
 NAME Hank145
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180508
 Time 19.33
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDC13
 NS 16
 DS 0
 SWH 7246.377 Hz
 FIDRES 0.221142 Hz
 AQ 2.260921 sec
 RG 114
 DW 69.000 usec
 DE 6.50 usec
 TE 673.2 K
 D1 2.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 0 dB
 SFO1 400.1324008 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300080 MHz
 WDW EM
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00

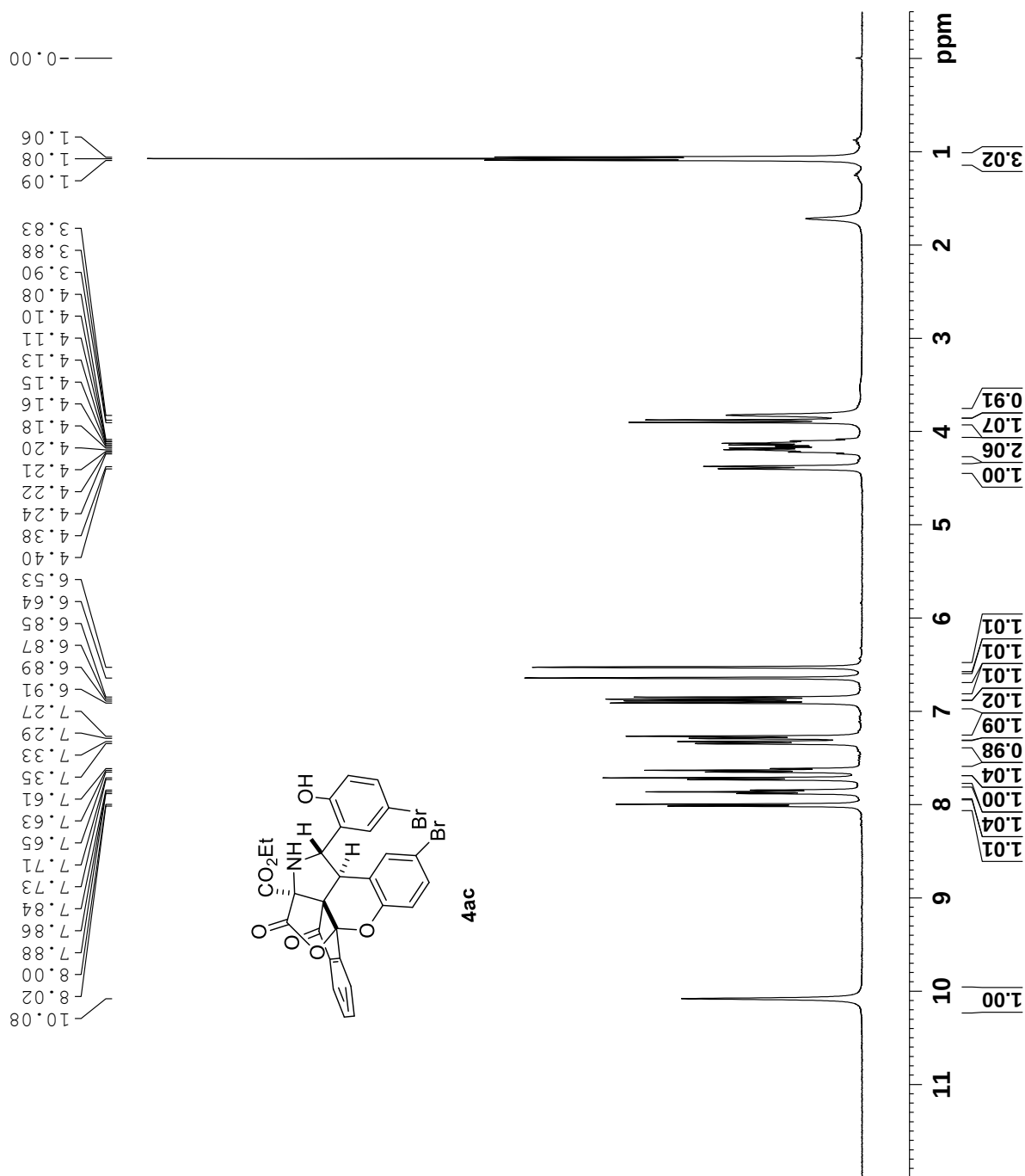


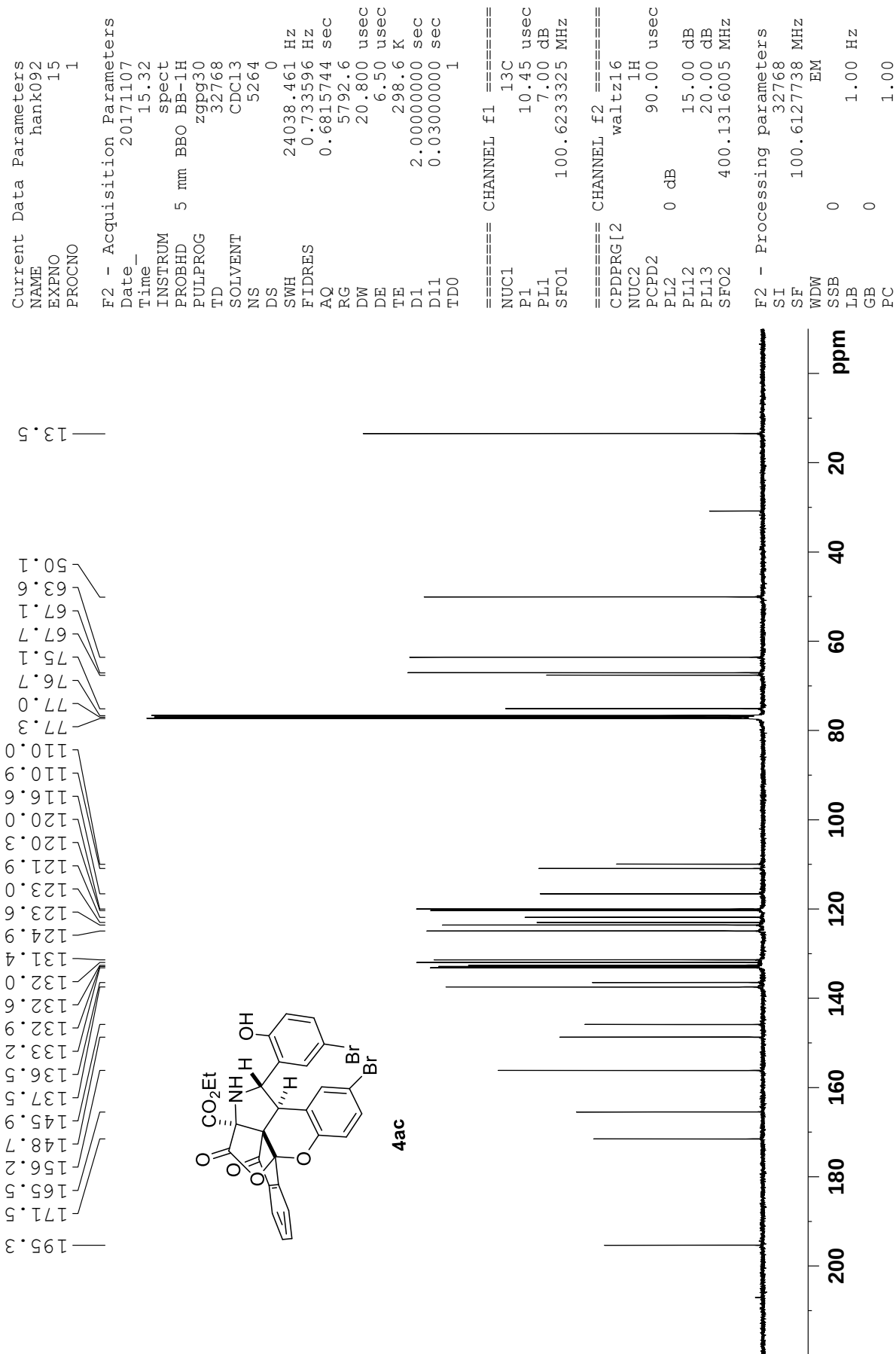
Current Data Parameters
NAME hank092
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171107
Time_ 15.30
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.260921 sec
RG 114
DW 69.000 usec
DE 6.50 usec
TE 298.5 K
D1 2.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 0 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300075 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00





Current Data Parameters
NAME Hank111
EXPNO 33
PROCNO 1

F2 - Acquisition Parameters

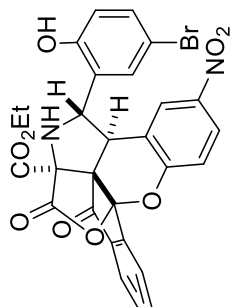
Date_ 20180224
Time_ 12.05
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.260921 sec
RG 181
DW 69.000 usec
DE 6.50 usec
TE 295.2 K
D1 2.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 0 dB
SFO1 400.1324008 MHz

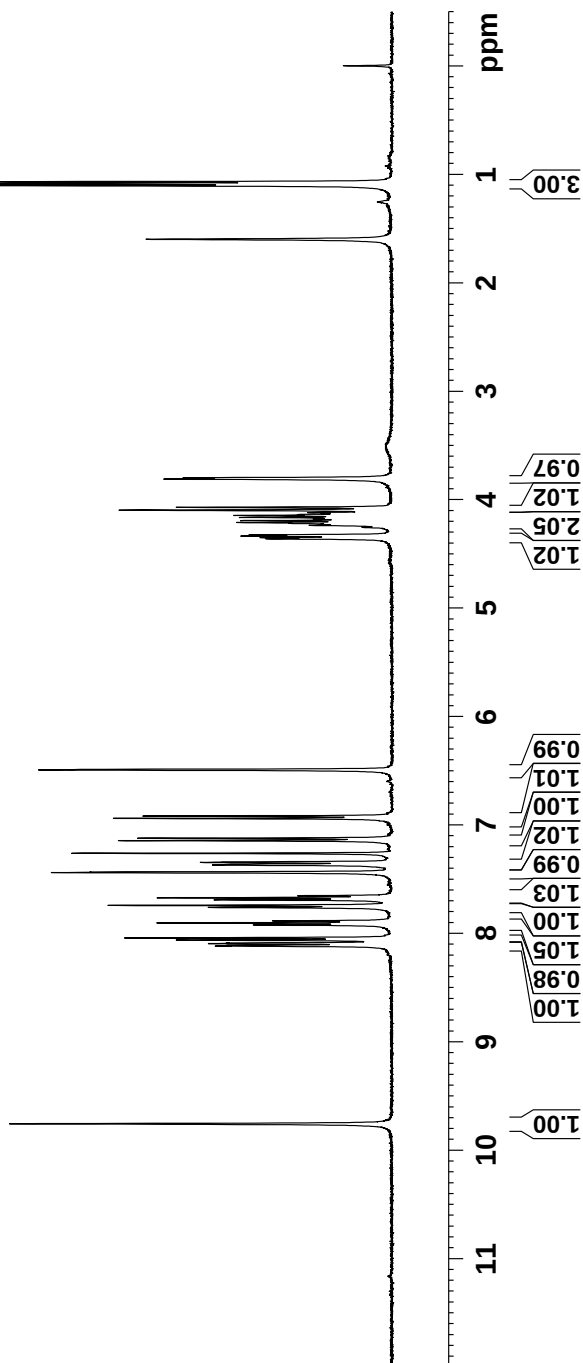
F2 - Processing parameters

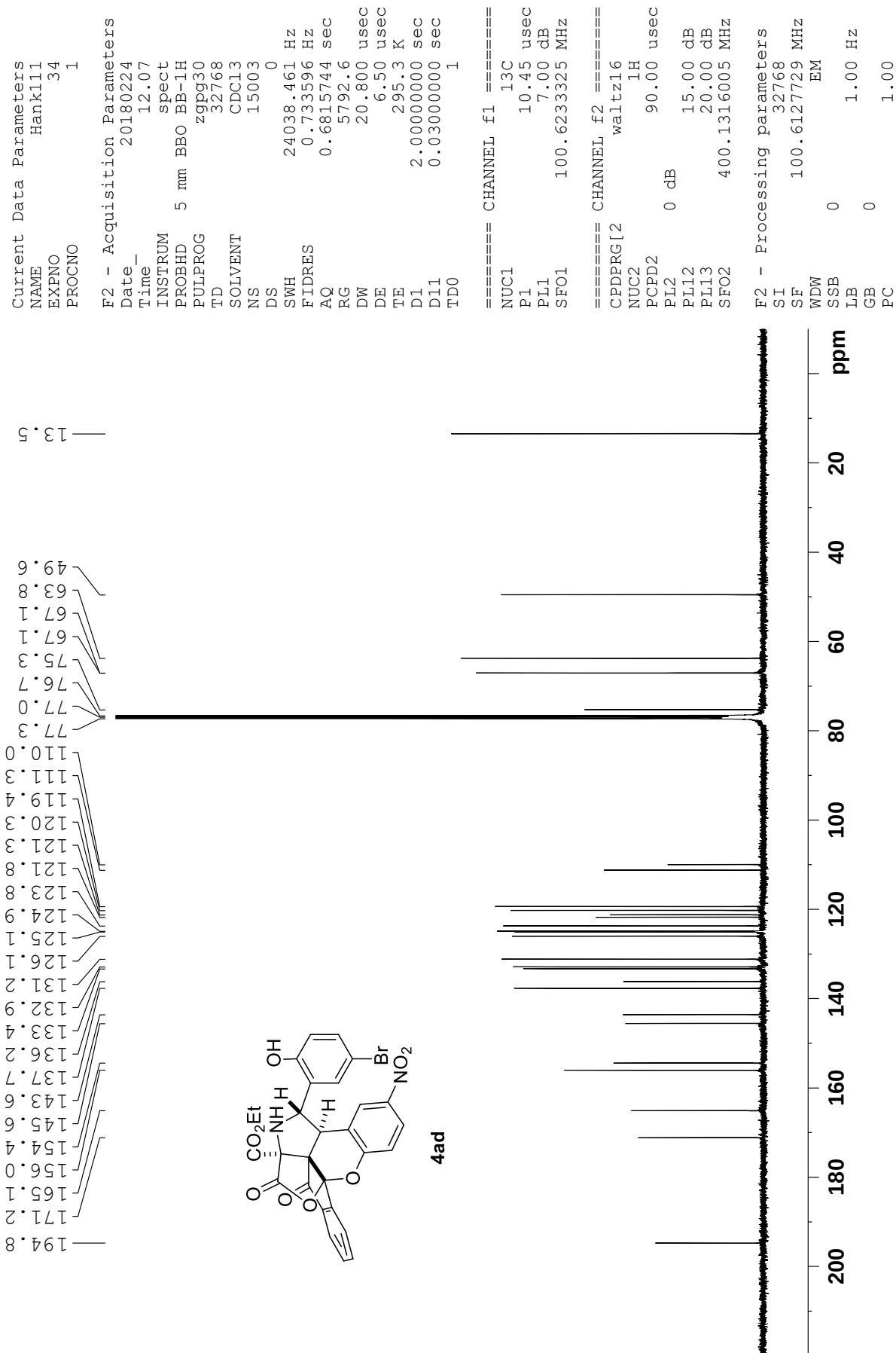
SI 16384
SF 400.1300077 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

9.76
8.12
8.11
8.10
8.09
8.06
8.04
7.92
7.91
7.89
7.76
7.74
7.69
7.67
7.65
7.44
7.37
7.35
7.15
7.13
6.94
6.92
6.50
4.36
4.35
4.34
4.33
4.26
4.24
4.23
4.22
4.21
4.20
4.18
4.18
4.17
4.15
4.14
4.13
4.12
4.10
4.07
3.81
3.80
1.11
1.09
1.07
0.00



4ad





Current Data Parameters
 NAME hank091
 EXPNO 6
 PROCNO 1

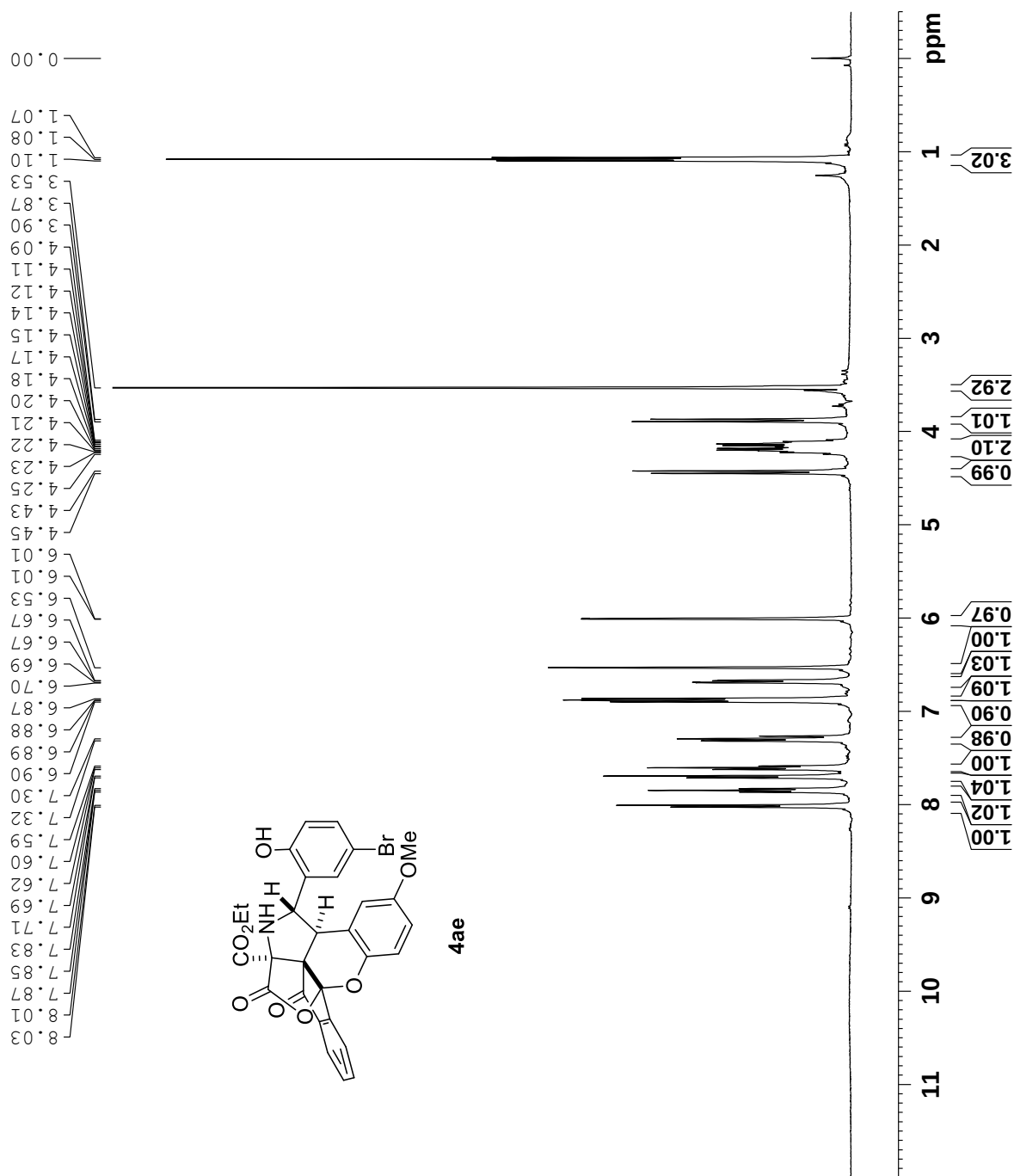
F2 - Acquisition Parameters

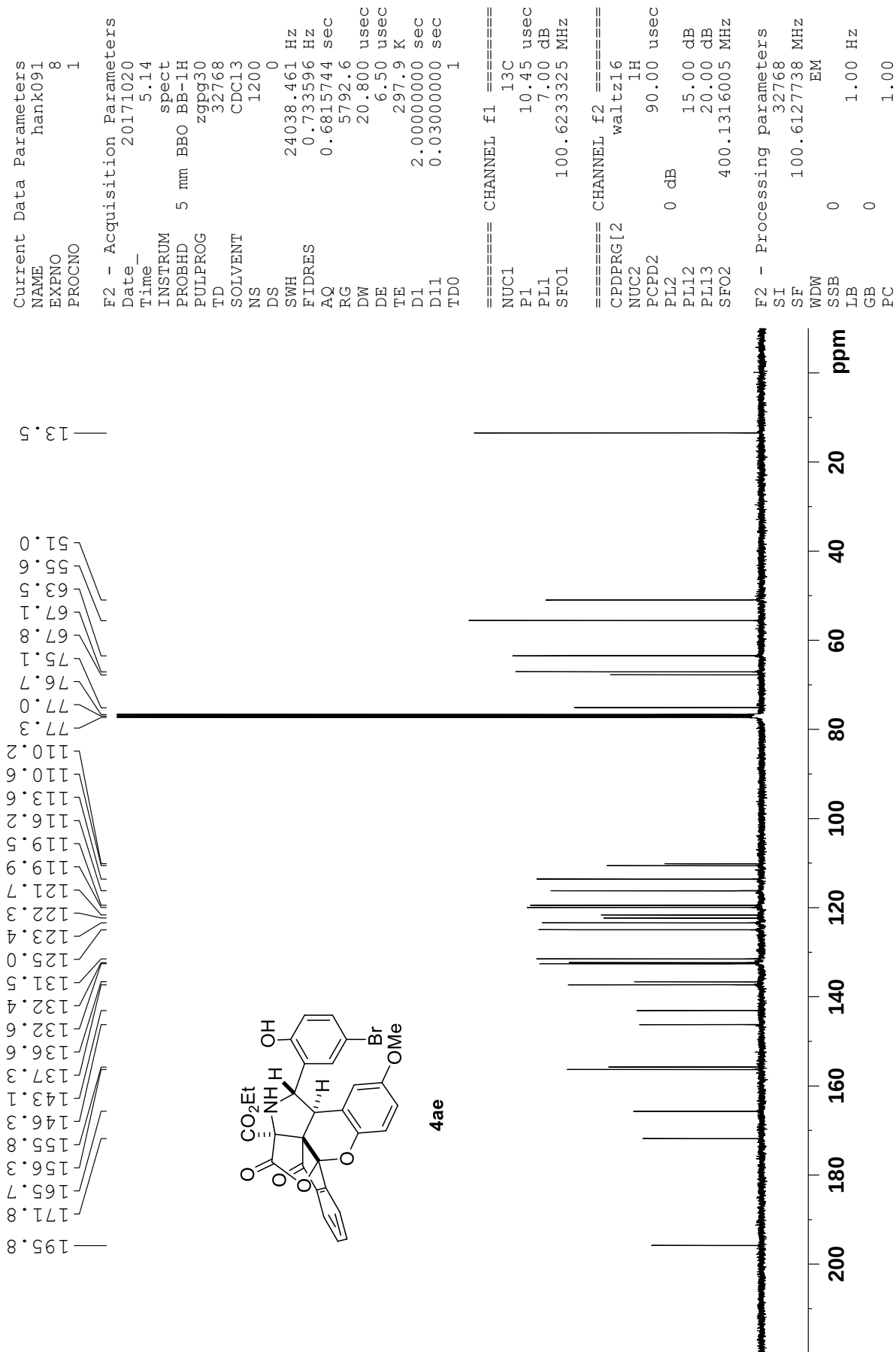
Date_ 20171019
 Time_ 20.27
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 1
 DS 0
 SWH 7246.377 Hz
 FIDRES 0.221142 Hz
 AQ 2.260921 sec
 RG 114
 DW 69.000 usec
 DE 6.50 usec
 TE 297.6 K
 D1 2.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 0 dB
 SFO1 400.1324008 MHz

F2 - Processing parameters

SI 16384
 SF 400.1300069 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.00





Current Data Parameters
 NAME Hank144
 EXPNO 3
 PROCNO 1

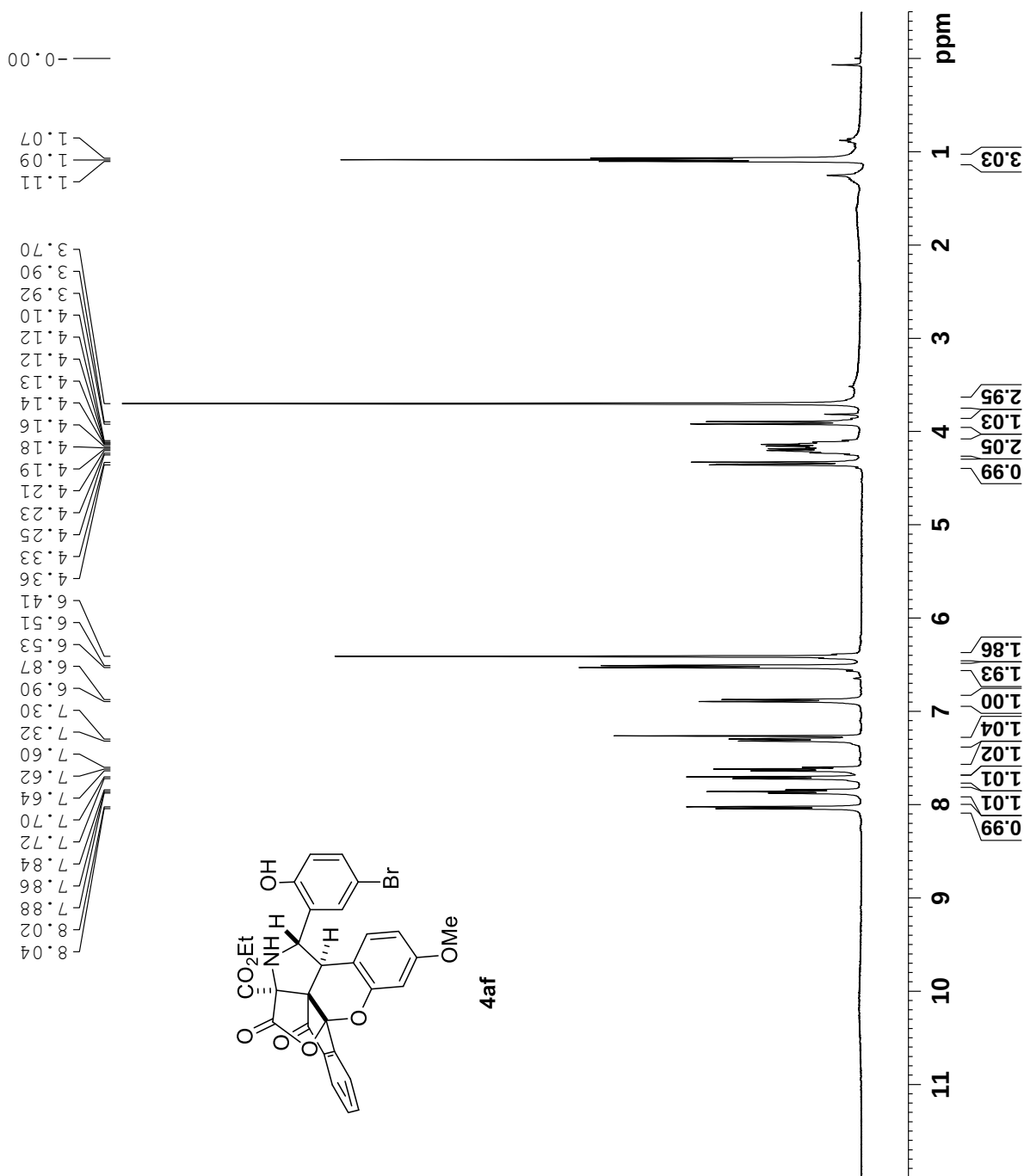
F2 - Acquisition Parameters

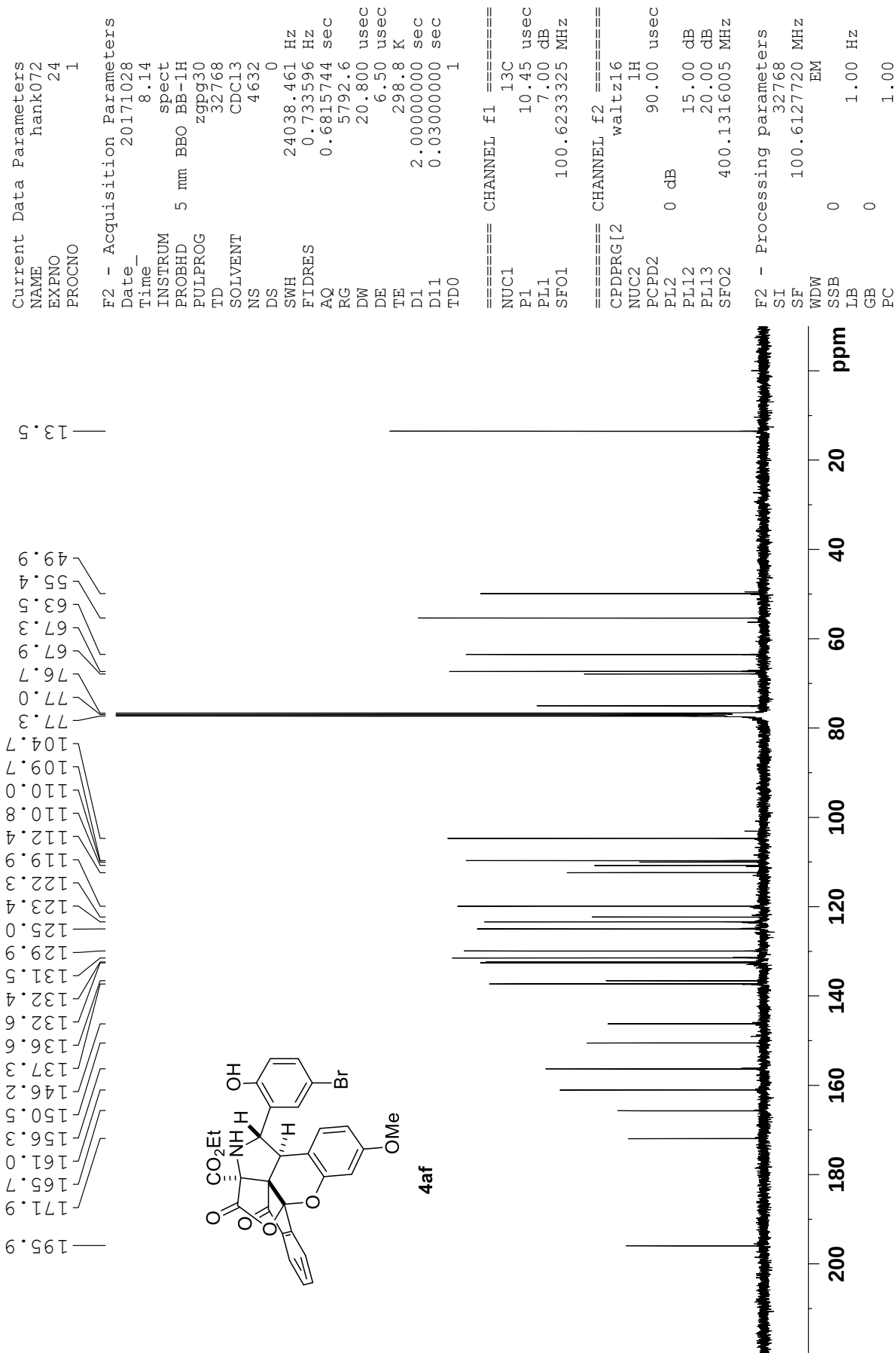
Date_ 20180508
 Time_ 17.34
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 7246.377 Hz
 FIDRES 0.221142 Hz
 AQ 2.260921 sec
 RG 114
 DW 69.000 usec
 DE 6.50 usec
 TE 673.2 K
 D1 2.0000000 sec
 TD0 1

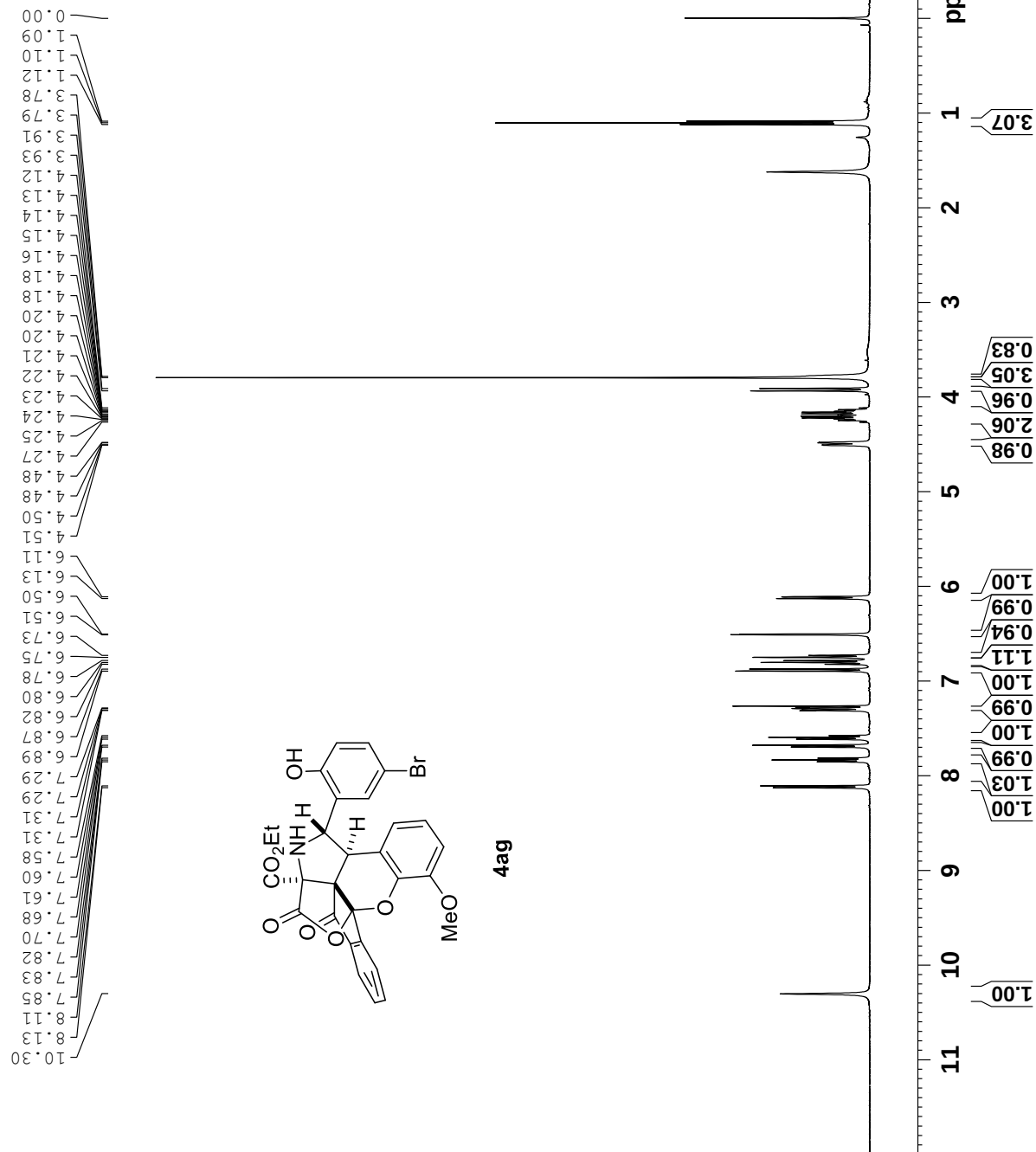
===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 0 dB
 SFO1 400.1324008 MHz

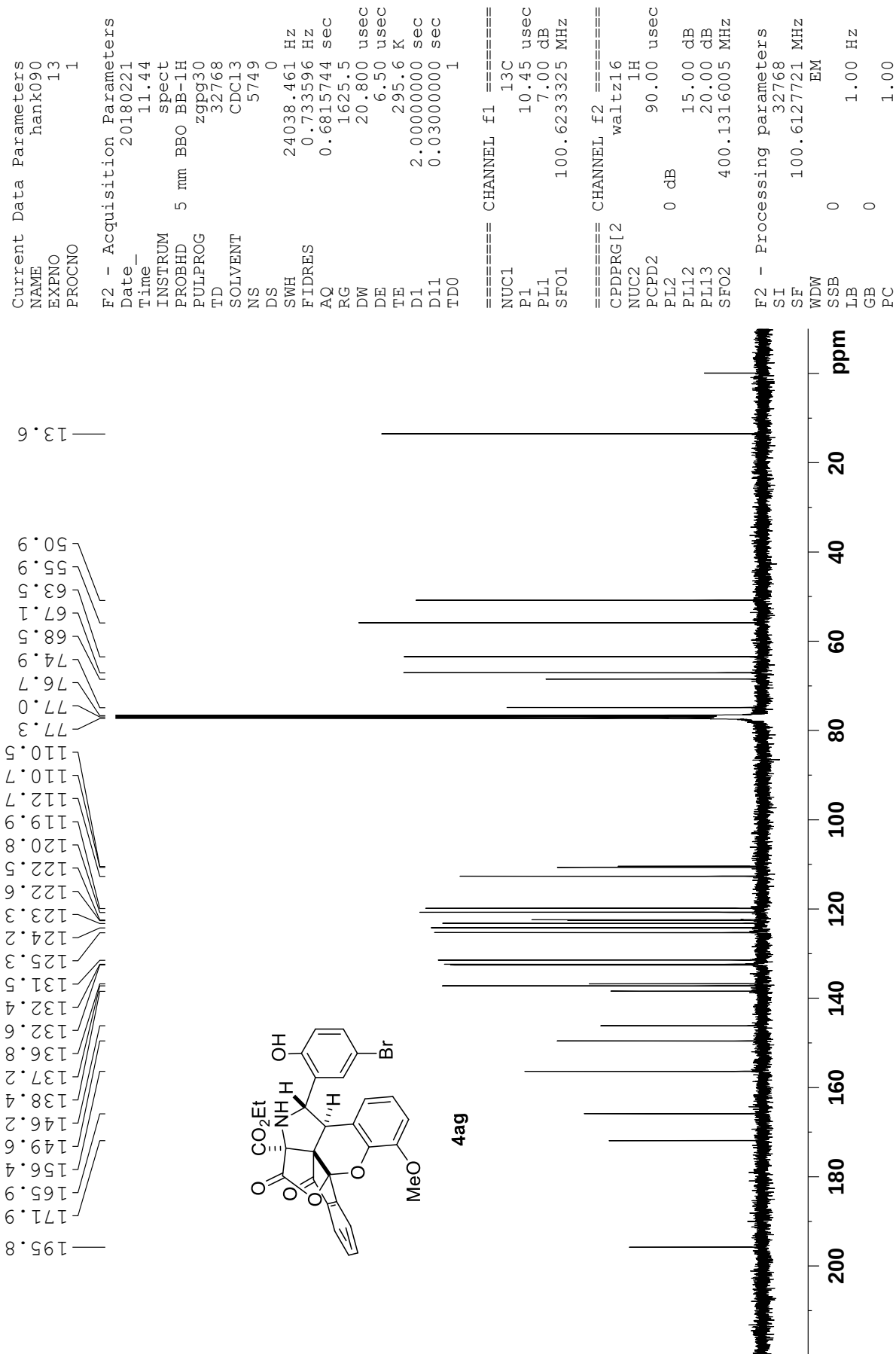
F2 - Processing parameters

SI 16384
 SF 400.1300079 MHz
 WDW EM
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00

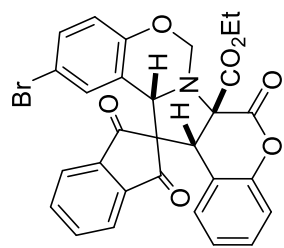




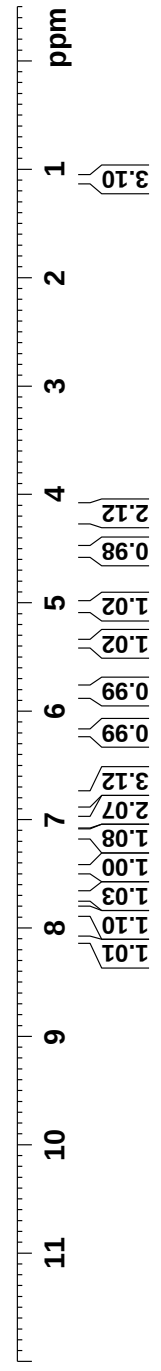




8.11
8.09
8.06
7.86
7.84
7.82
7.72
7.70
7.68
7.46
7.44
7.16
7.14
7.12
7.03
7.01
6.84
6.83
6.80
6.78
6.75
6.20
6.19
5.83
5.80
5.37
5.06
5.03
4.53
4.23
4.22
4.21
4.20
4.18
4.18
4.16
4.15
4.14
4.13
1.11
1.09
1.07
0.00



5aa



Current Data Parameters
NAME vicky188
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170429
Time_ 15.07
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.260921 sec
RG 256
DW 69.000 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 14.40 usec
PL1 1.80 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300076 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

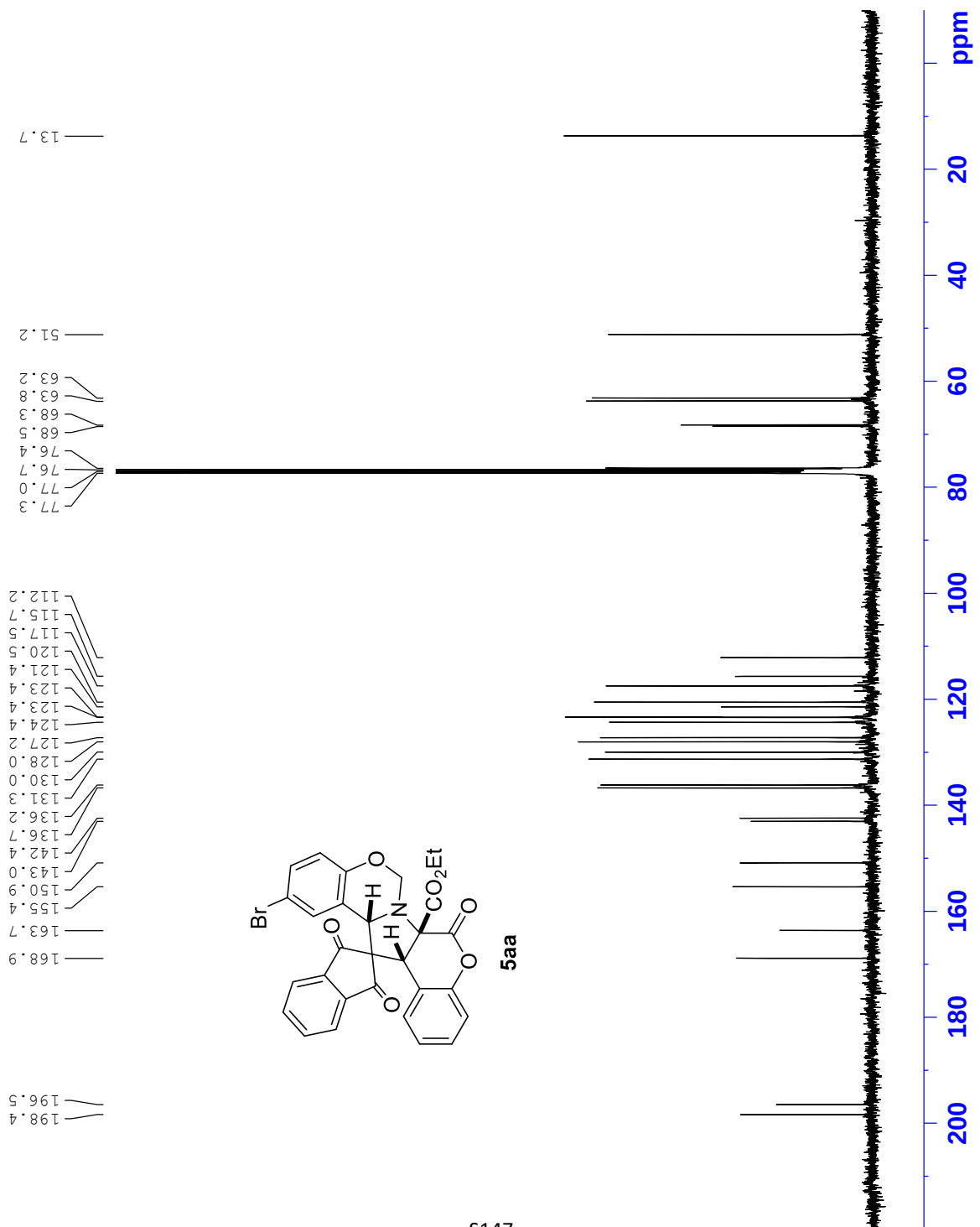
Current Data Parameters
NAME vicky188
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170502
Time_ 22.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2738
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

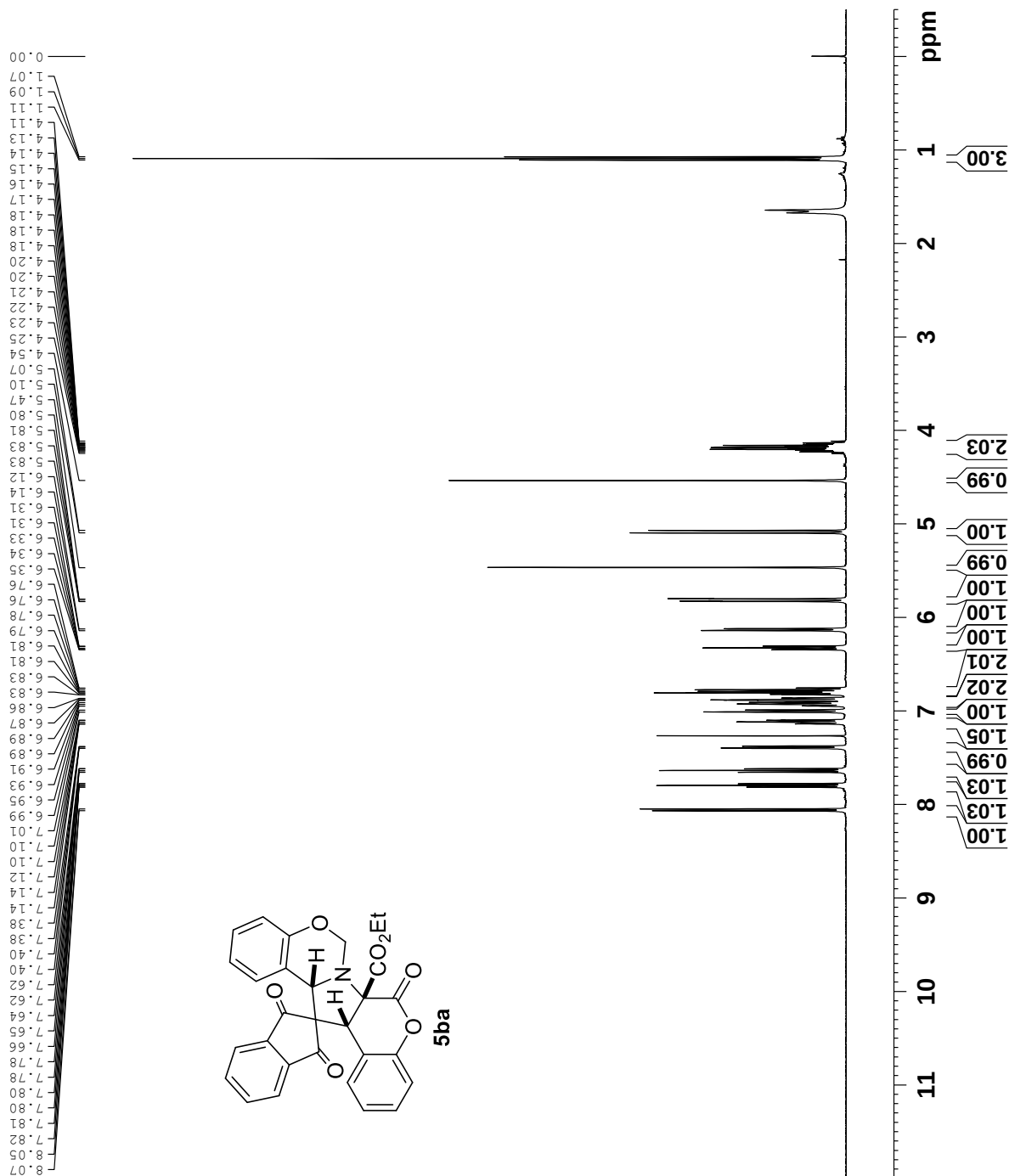


Current Data Parameters
NAME vicky180
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170422
Time_ 2.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 99.72
DW 69.333 usec
DE 10.50 usec
TE 297.1 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1300063 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



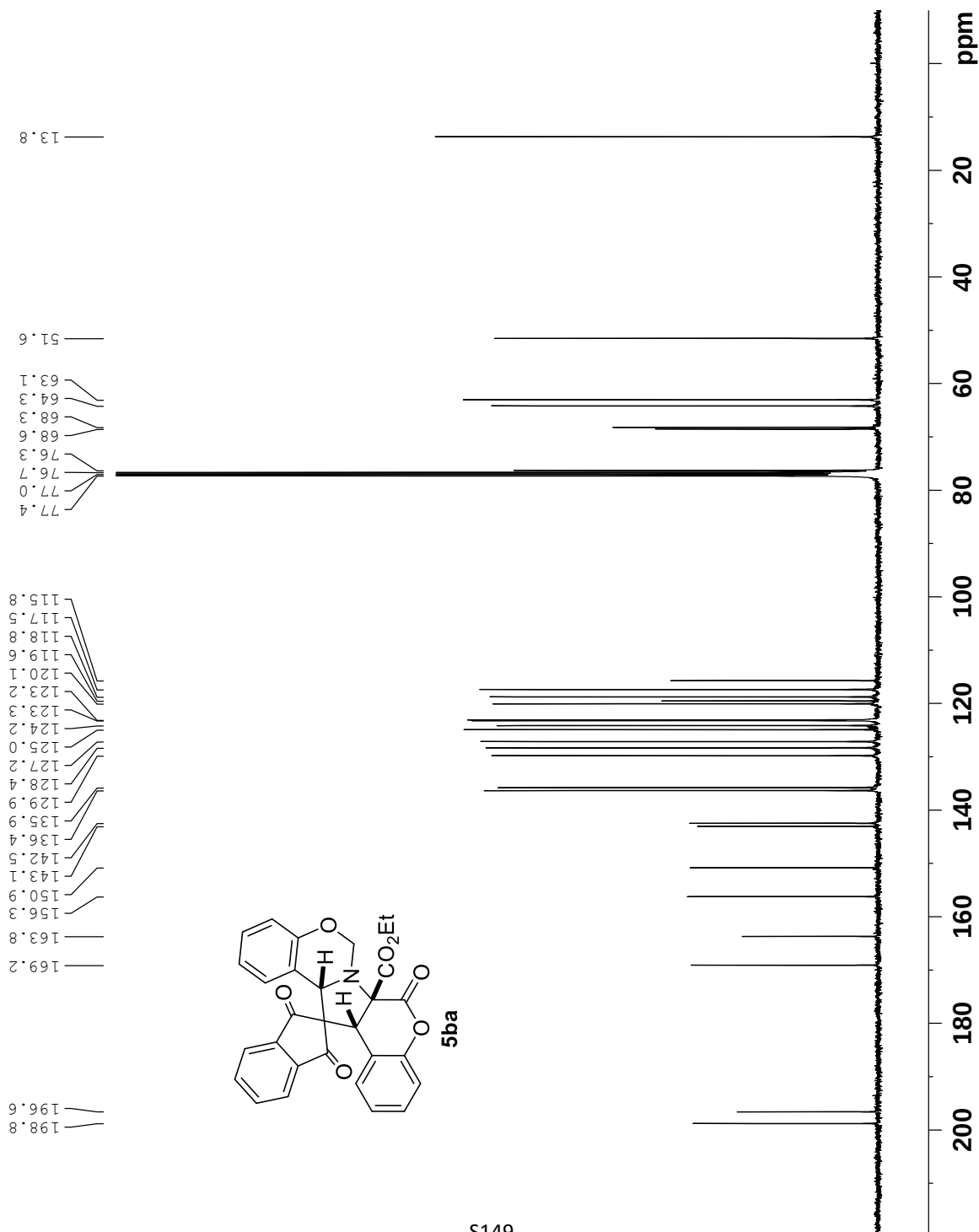
Current Data Parameters
NAME vicky180
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170422
Time_ 2.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 8858
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



5ca

F2 - Processing parameters	
SI	16384
SF	400.130074 MHz
WDW	EM
SSB	0
LB	0 Hz
GB	0
PC	1.00

Current Data Parameters
NAME vicky176
EXPNO 10
PROCNO 1

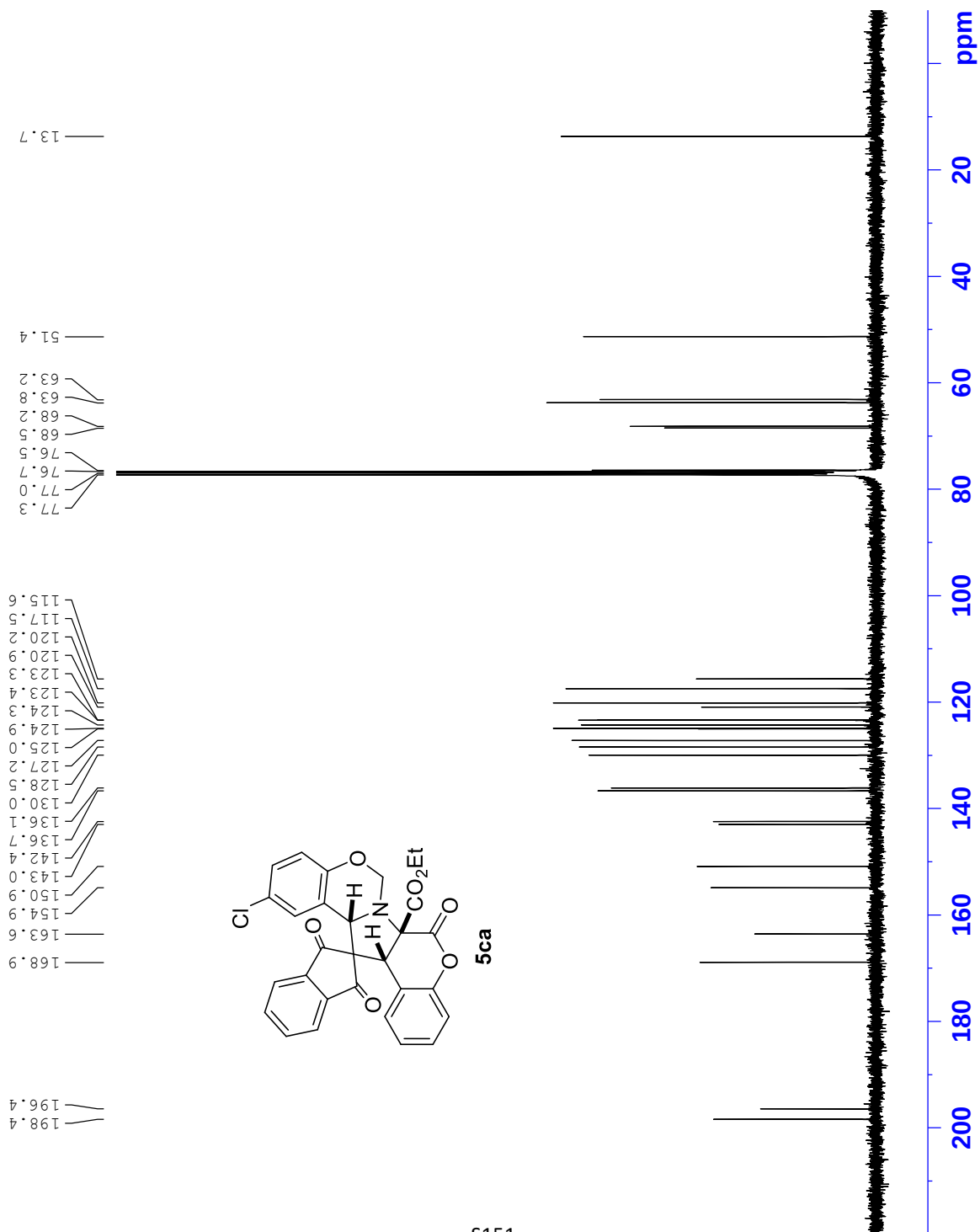
F2 - Acquisition Parameters

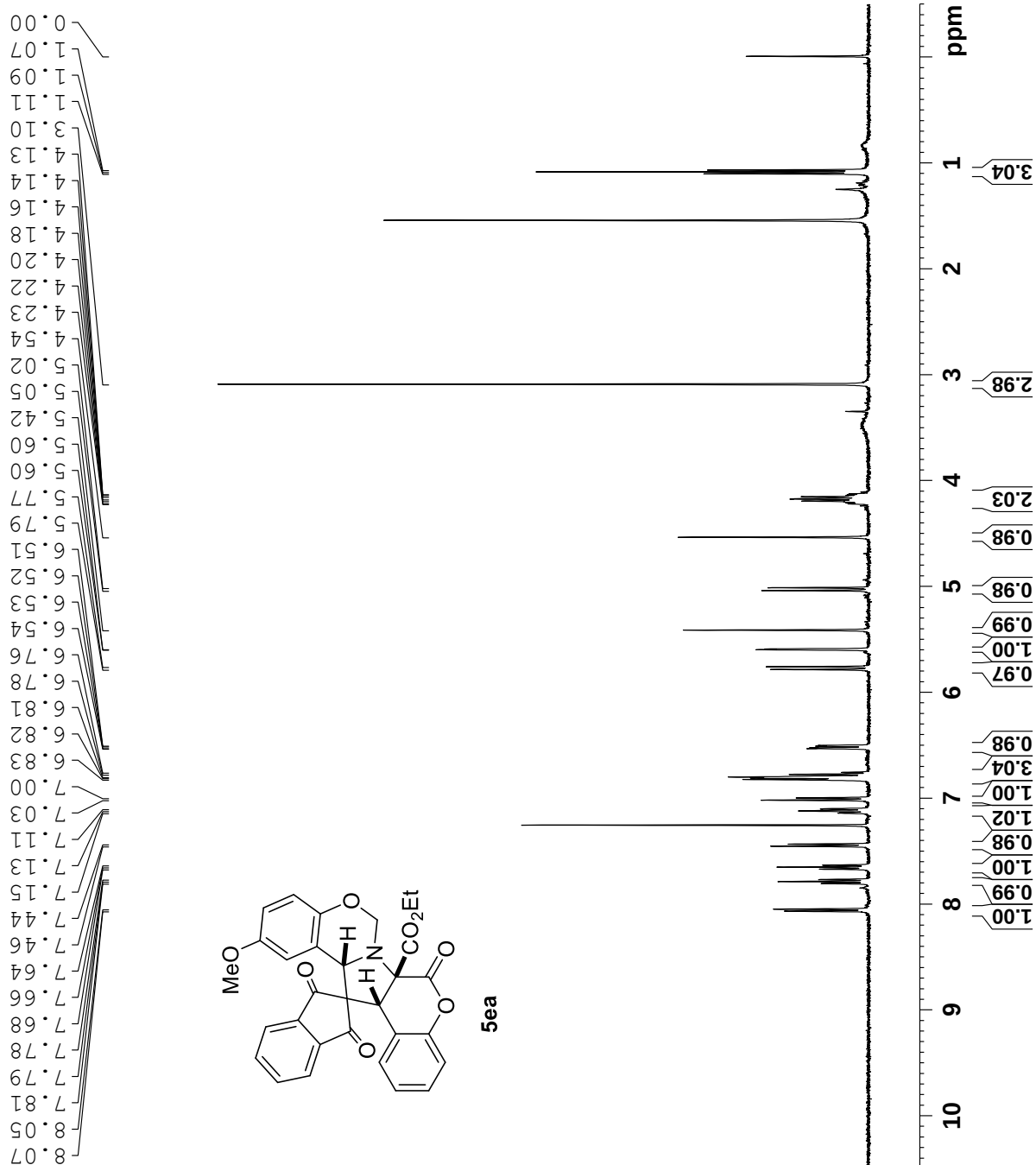
Date_ 20170426
Time_ 22.22
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 5181
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 6502
DW 20.800 usec
DE 6.50 usec
TE 298.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00





Current Data Parameters
NAME B141
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170516
Time_ 19.16
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.260921 sec
RG 574.7
DW 69.000 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.40 usec
PL1 1.80 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300118 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

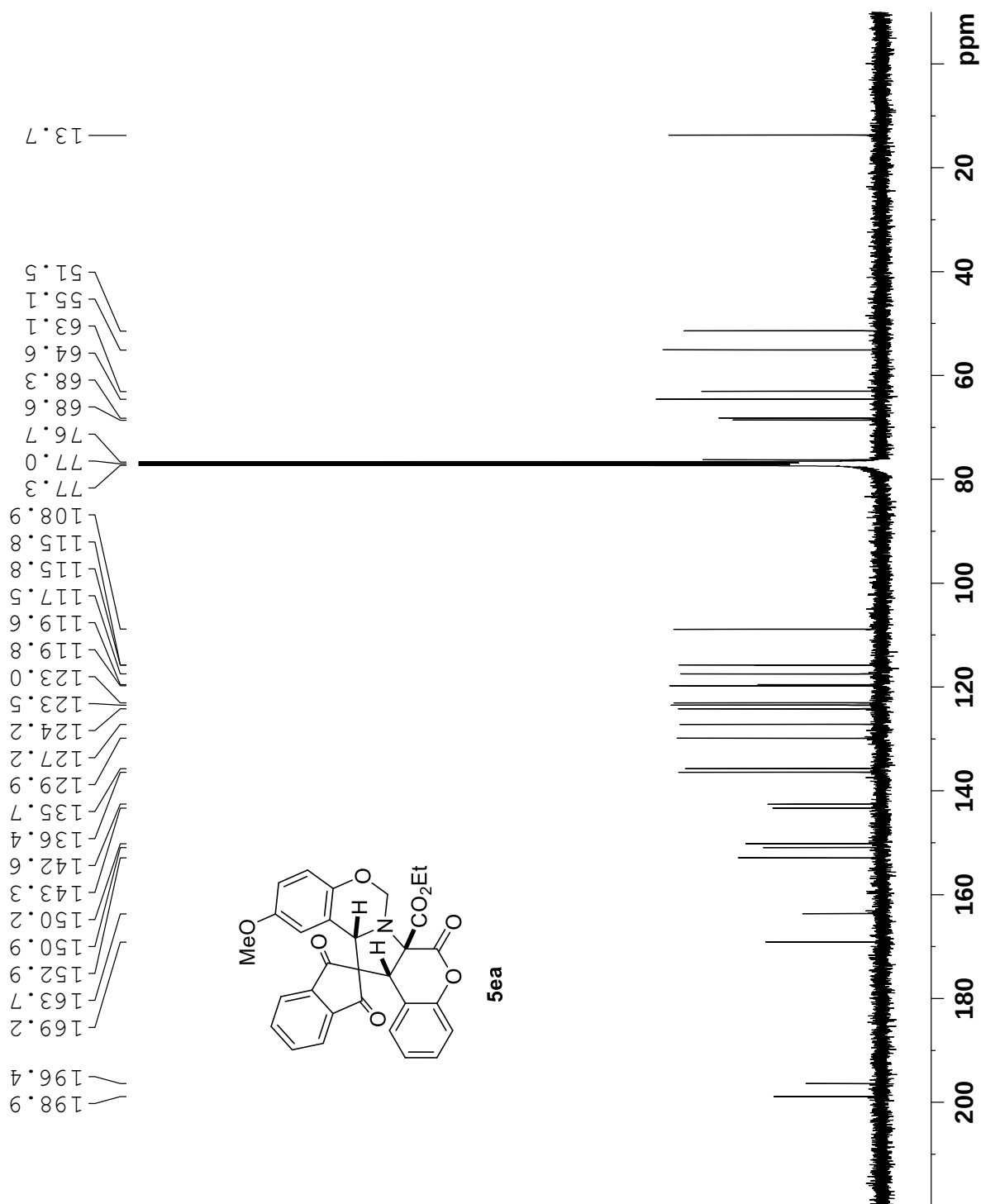
Current Data Parameters
NAME B141
EXPNO 20
PROCNO 1

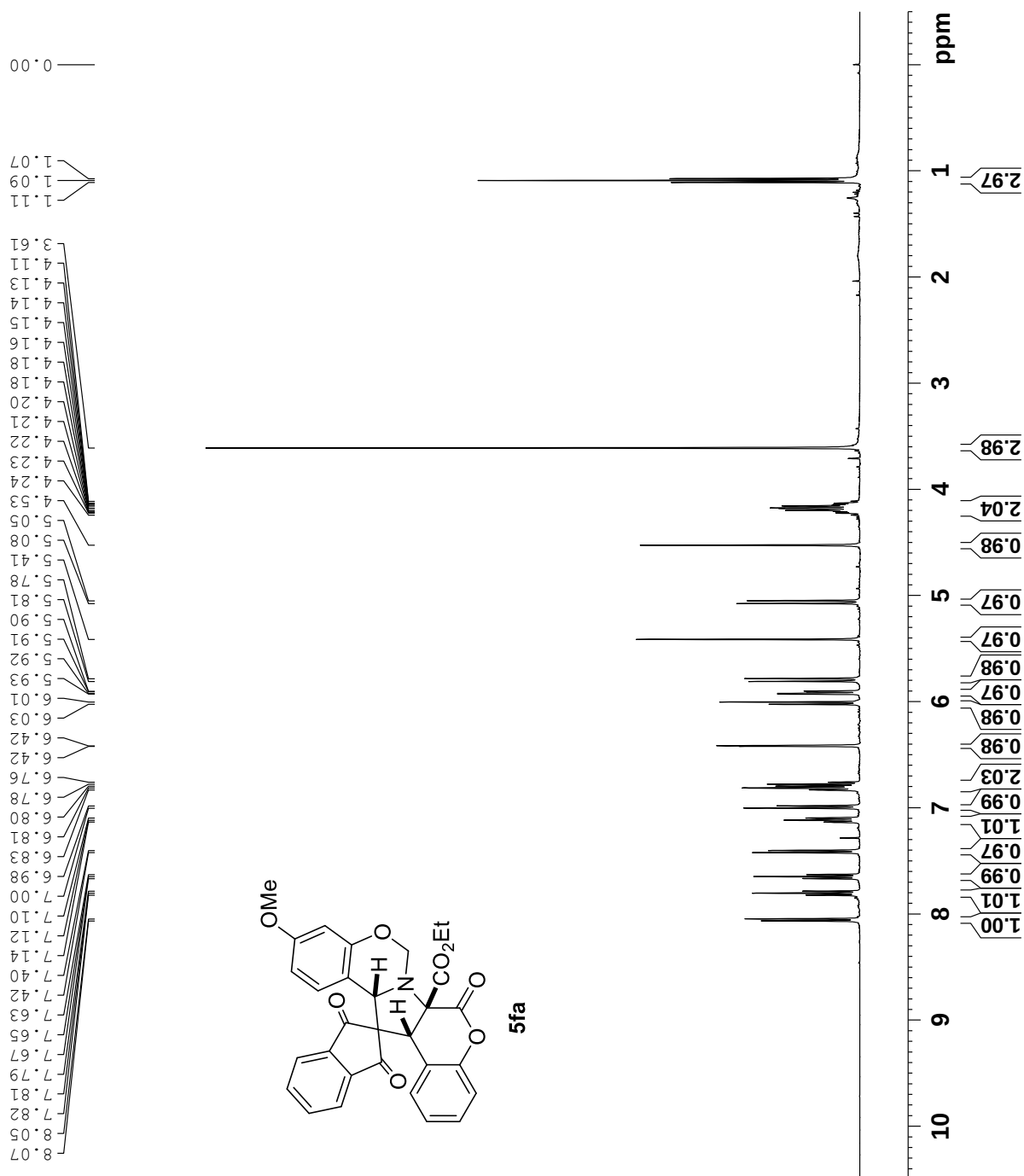
F2 - Acquisition Parameters
Date_ 20170516
Time 22.50
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 10754
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 16384
DW 20.800 usec
DE 6.50 usec
TE 299.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

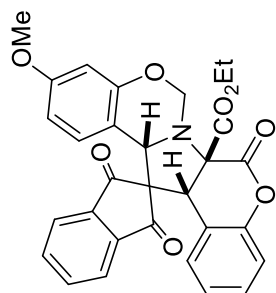
==== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

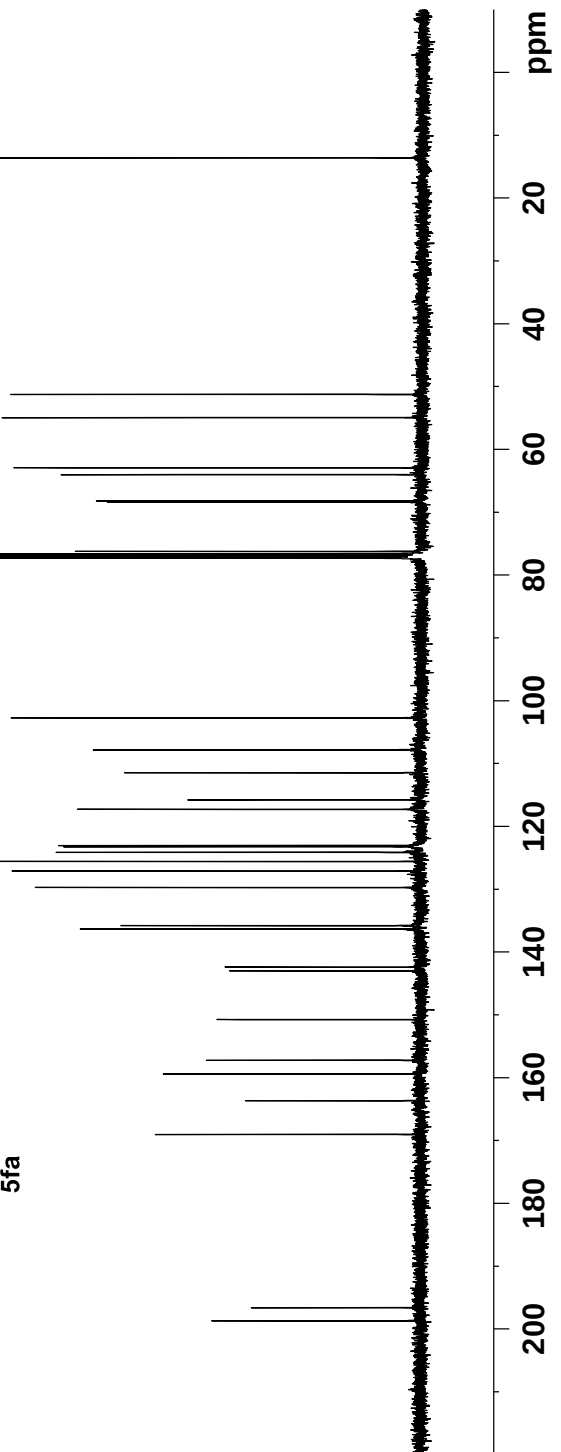




198.8
196.7
169.1
163.7
159.4
157.3
150.8
143.0
142.4
136.4
135.8
129.7
127.1
125.6
124.1
123.2
123.1
117.3
115.8
111.5
107.8
102.8
77.3
77.0
76.7
76.3
68.5
68.3
64.1
63.0
55.0
51.3
13.7



5fa



Current Data Parameters
NAME B146
EXPNO 21
PROCNO 1

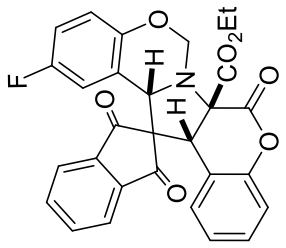
F2 - Acquisition Parameters
Date_ 20170527
Time 7.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 627
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 5792.6
DW 20.800 usec
DE 6.50 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

==== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127781 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

7.80
7.70
7.68
7.66
7.46
7.44
7.14
7.13
7.11
7.02
7.00
6.87
6.86
6.85
6.83
6.81
6.79
6.77
6.76
6.69
6.68
6.66
6.66
6.66
6.64
6.64
6.86
5.86
5.85
5.84
5.83
5.82
5.79
5.42
5.05
5.03
4.51
4.23
4.22
4.21
4.20
4.18
4.18
4.16
4.15
4.14
4.13
1.10
1.09
1.07



5ha

S156

10
9
8
7
6
5
4
3
2
1
ppm

1.00
1.07
1.02
0.99
1.03
1.02
3.06
0.97
1.96
1.00
0.99
0.97
2.10
3.10

Current Data Parameters
NAME B357
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180526
Time 11.54
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.260921 sec
RG 228.1
DW 69.000 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 0 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

198.5
196.4
169.0
163.6
157.4
155.0
152.4
152.4
150.9
143.0
142.4
136.6
136.1
130.0
127.2
124.3
123.4
123.3
120.5
120.4
120.1
120.0
117.5
115.6
115.3
111.1
110.9
77.3
77.0
76.7
68.6
68.1
63.8
63.2
51.7

Current Data Parameters
NAME B357
EXPNO 2
PROCNO 1

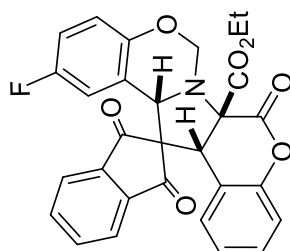
F2 - Acquisition Parameters

Date_ 20180509
Time_ 22.29
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 14044
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 5792.6
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

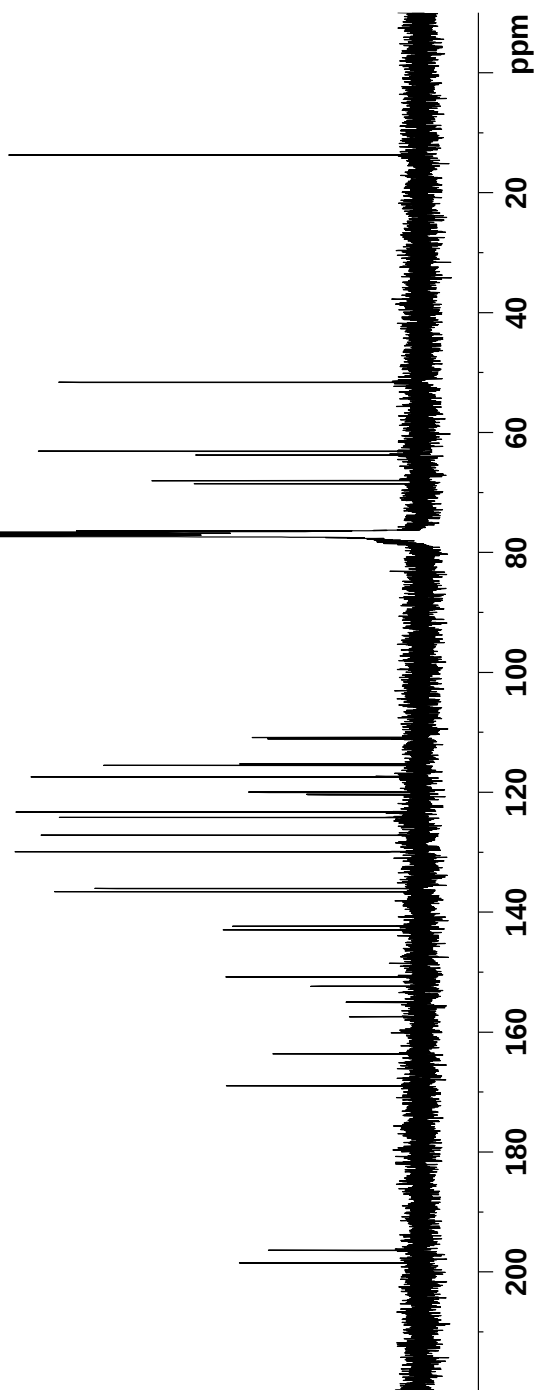
===== CHANNEL f1 =====
NUC1 13C
P1 10.45 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0 dB
PL12 15.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



5ha



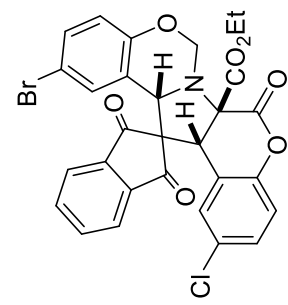
7.91 7.89 7.87 7.77 7.75 7.73 7.48 7.46 7.13 7.13 7.11 7.10 7.03 7.01 6.99 6.96 6.85 6.84 6.77 6.75 6.18 5.81 5.79 5.34 5.03 5.00 4.50 4.24 4.23 4.22 4.21 4.21 4.20 4.19 4.18 4.17 1.15 1.13 1.11

Current Data Parameters
 NAME 5Cl derivative
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170511
 Time_ 9.44
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDC13
 NS 16
 DS 0
 SWH 7246.377 Hz
 FIDRES 0.221142 Hz
 AQ 2.2609921 sec
 RG 181
 DW 69.000 usec
 DE 6.50 usec
 TE 296.0 K
 D1 2.0000000 sec
 TD0 1

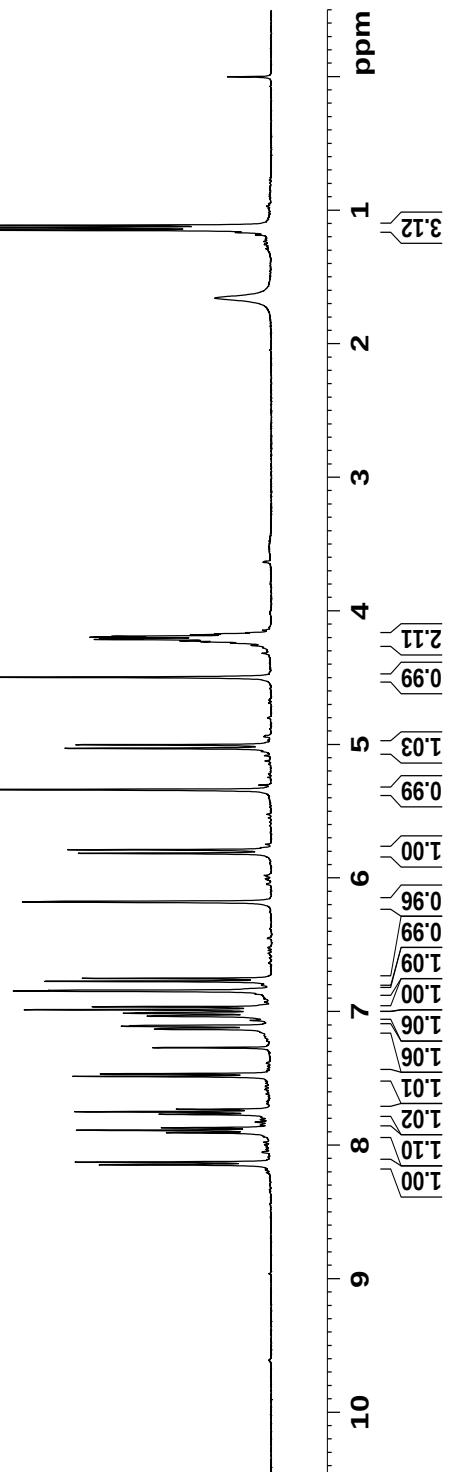
===== CHANNEL f1 =====
 NUC1 1H
 P1 14.40 usec
 PL1 1.80 dB
 SFO1 400.1324008 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300058 MHz
 WDW EM
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00



5ab

S158



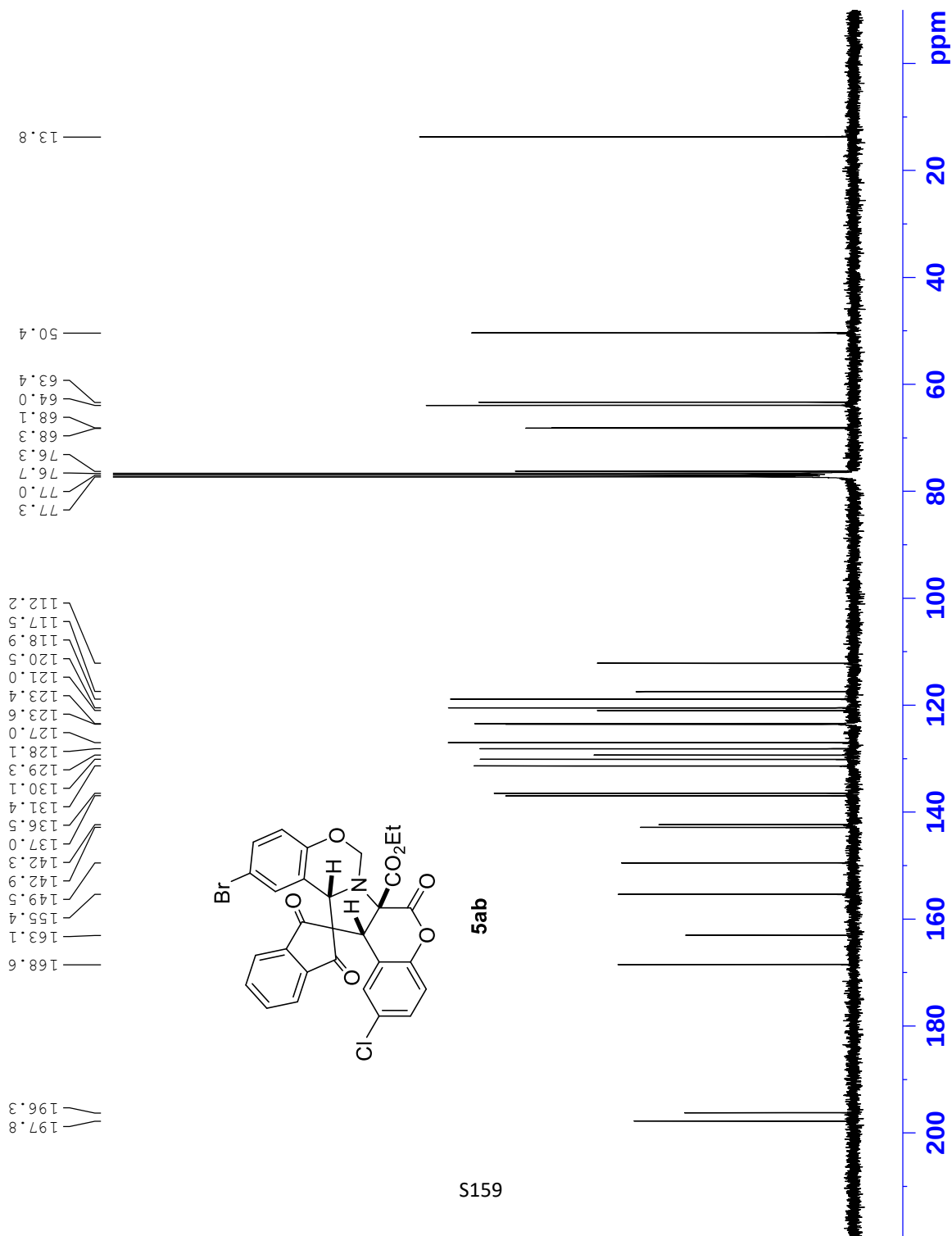
Current Data Parameters
NAME 5Cl derivative
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170423
Time 17.57
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2663
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 8192
DW 20.800 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

==== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

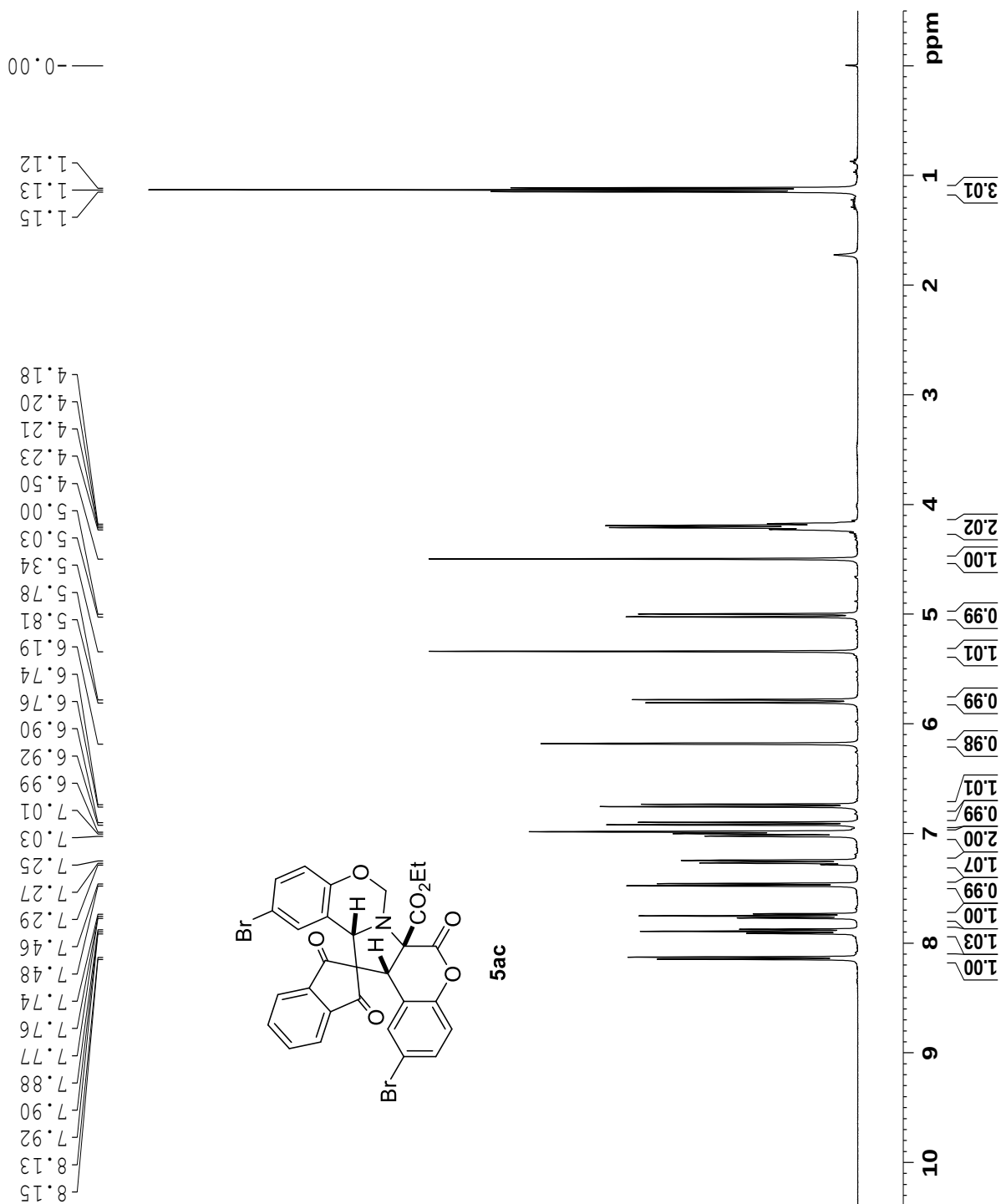
F2 - Processing parameters
SI 32768
SF 100.6127730 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



F2 - Acquisition Parameters	
Date_	20170425
Time_	22.54
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDC13
NS	16
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2609921 sec
RG	57
DW	69.000 usec
DE	6.50 usec
TE	298.3 K
D1	2.00000000 sec
TD0	1

```
===== CHANNEL f1 =====
NUC1      1H
P1        14.40 usec
PL1       1.80 dB
SF01      400.1324008 MHz

F2 - Processing parameters
SI        16384
SF        400.1299981 MHz
EM
SSB       0
LB       0 Hz
GB       0
PC       1.00
```



Current Data Parameters
NAME lilul57
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters

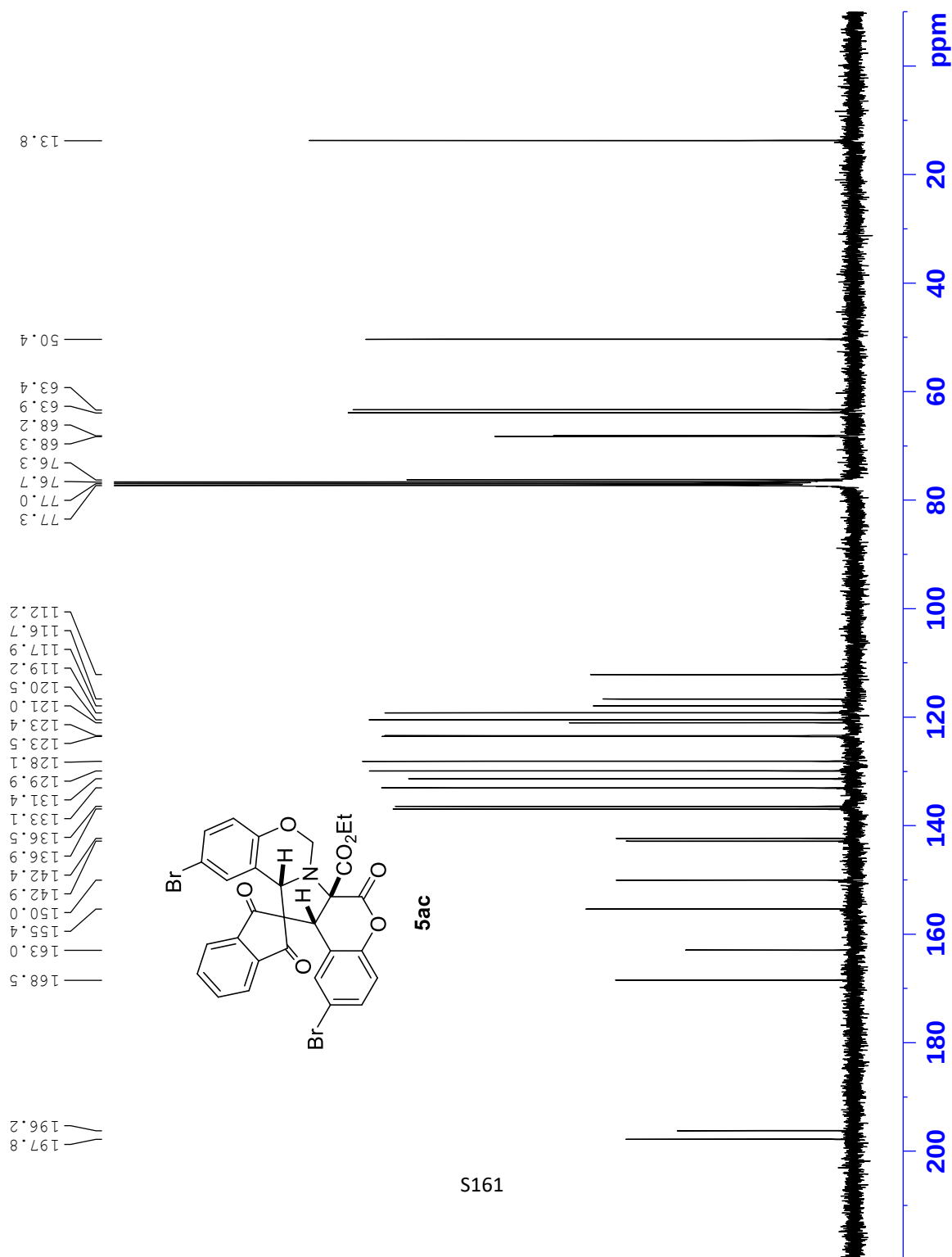
Date_ 20170325
Time 22.15
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1740
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 9195.2
DW 20.800 usec
DE 6.50 usec
TE 298.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

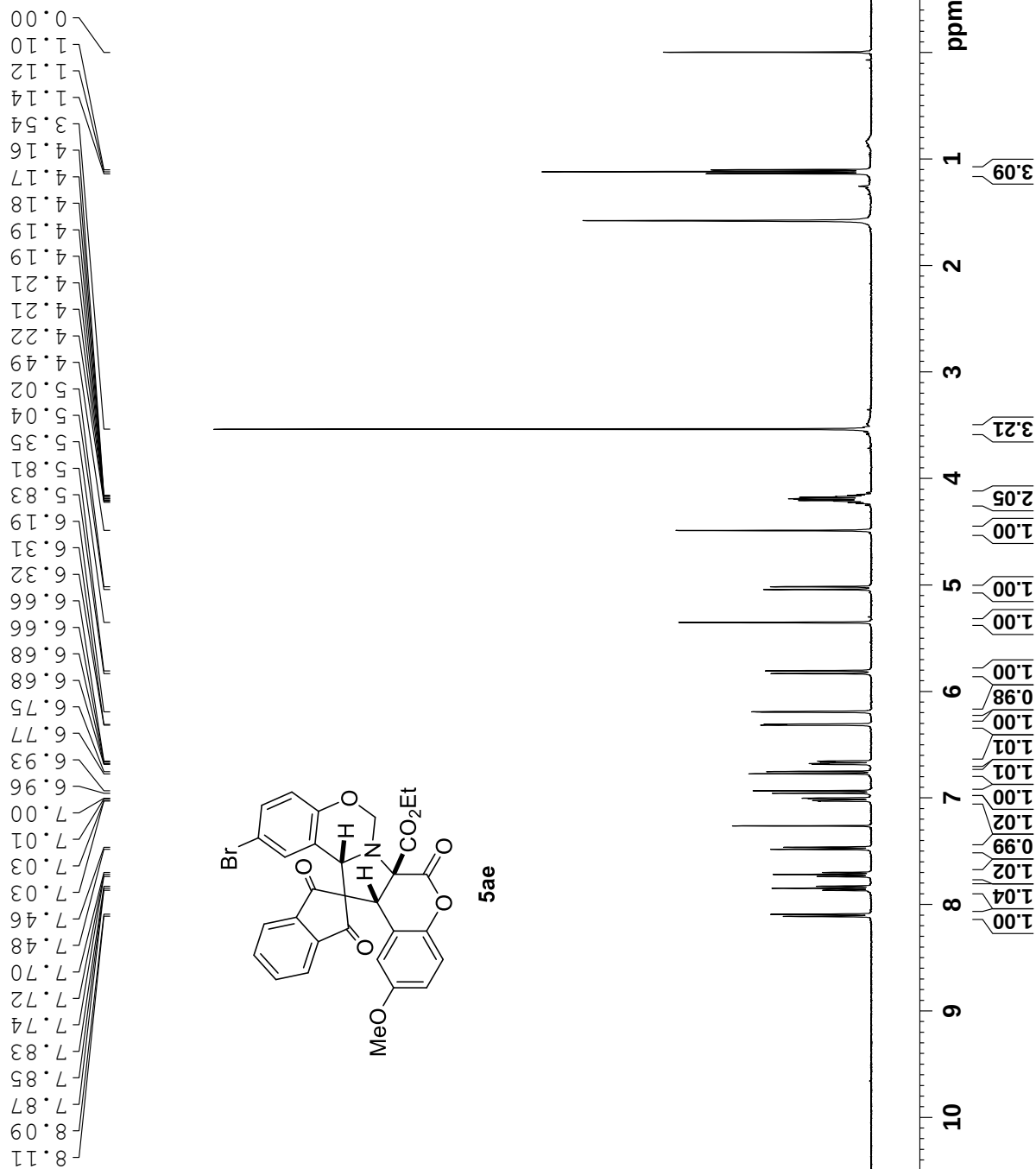
==== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

==== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.60 dB
PL12 15.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters

SI 32768
SF 100.6127734 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00





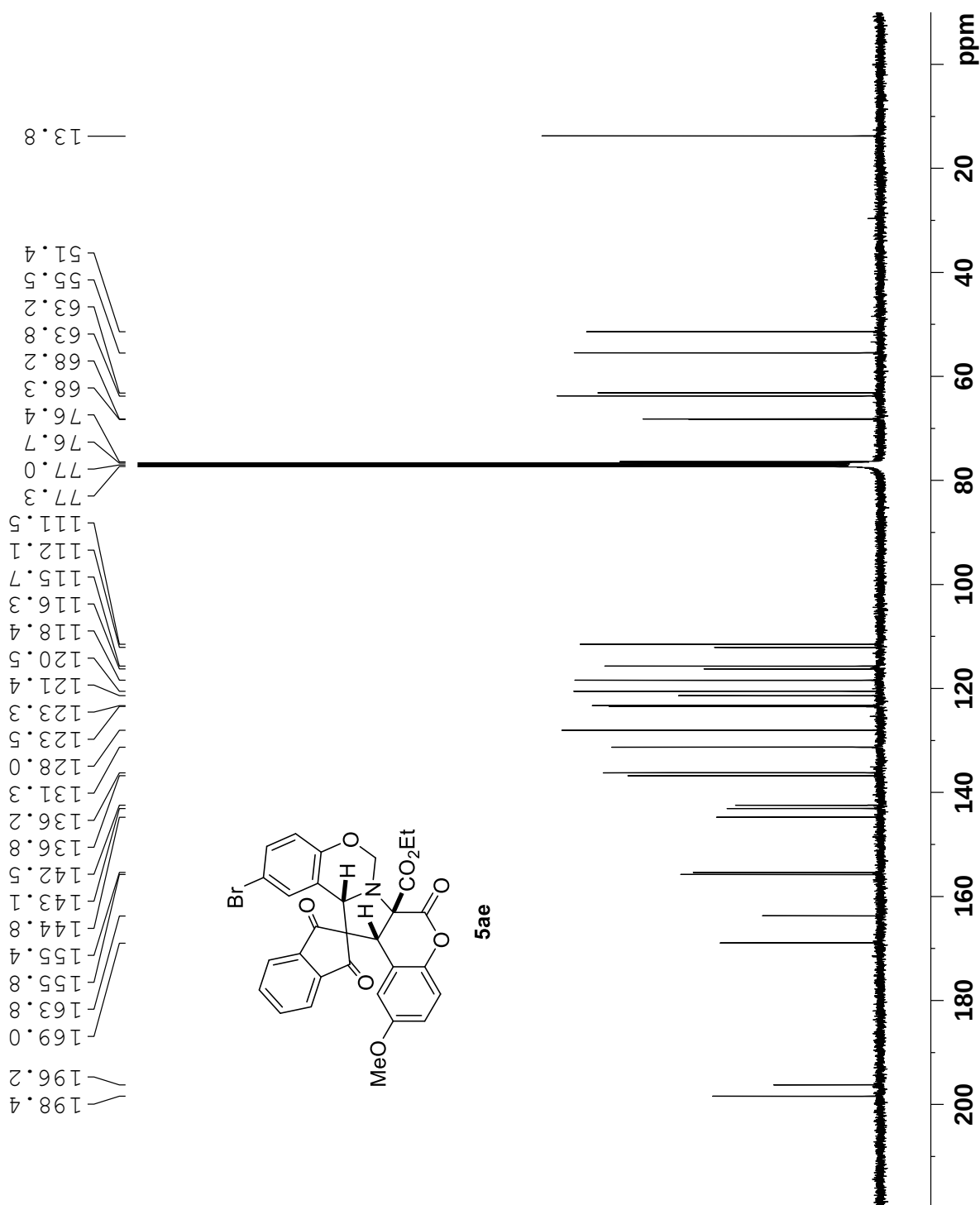
Current Data Parameters
NAME B144
EXPNO 7
PROCNO 1

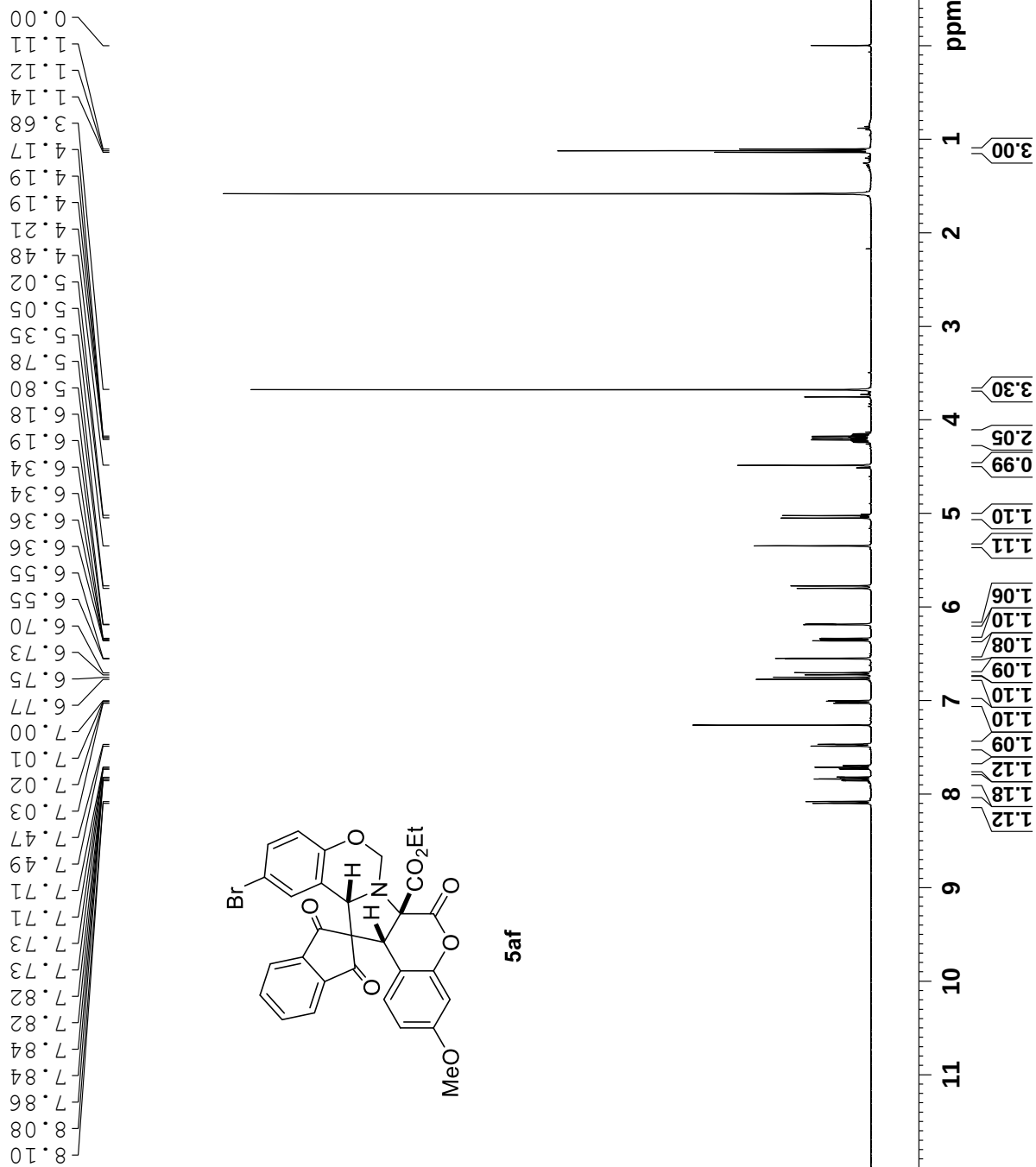
F2 - Acquisition Parameters
Date_ 20170502
Time_ 21.54
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 5407
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 9195.2
DW 20.800 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

==== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.60 dB
PL12 15.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00





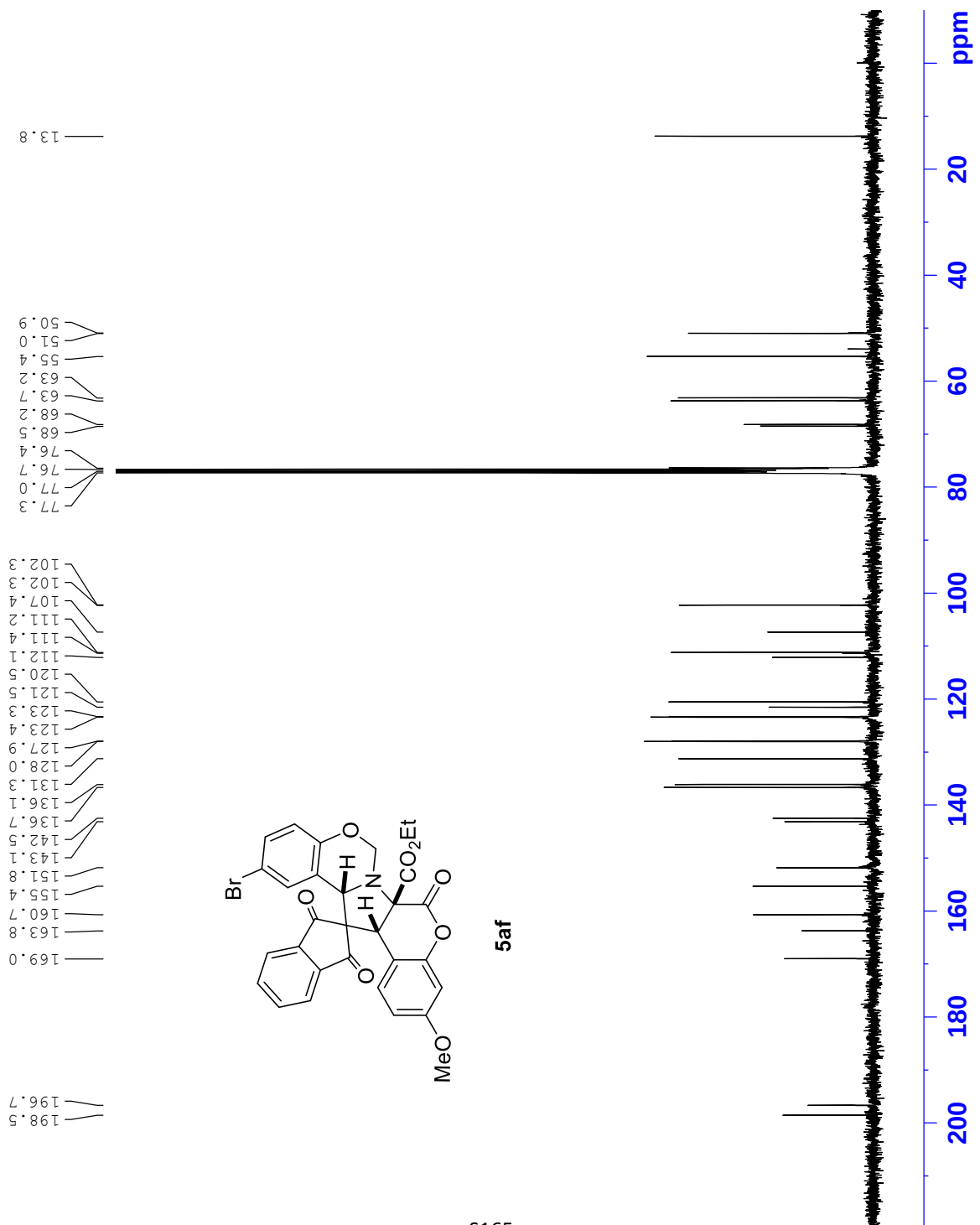
Current Data Parameters
NAME vicky189
EXPNO 2
PROCNO 1

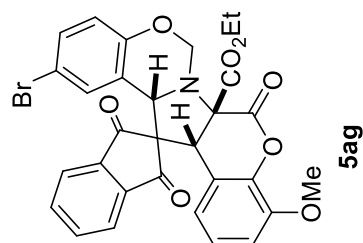
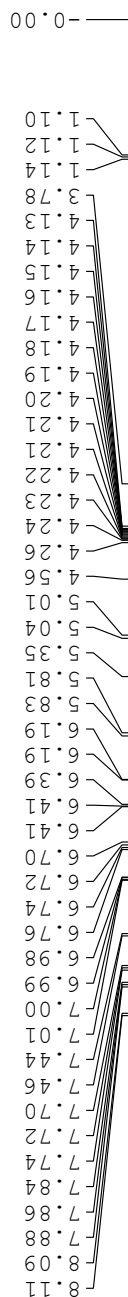
F2 - Acquisition Parameters
Date_ 20170511
Time_ 3.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6390
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00





Current Data Parameters
 NAME 3OMe-derivative
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170424
 Time_ 11:58
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 7246.377 Hz
 FIDRES 0.221142 Hz
 AQ 2.260921 sec
 RG 57
 DW 69.000 usec
 DE 6.50 usec
 TE 295.3 K
 D1 2.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.40 usec
 PL1 1.80 dB
 SF01 400.1324008 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1299978 MHz
 WDW EM
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME 3OMe-derivative
 EXPNO 3
 PROCNO 1

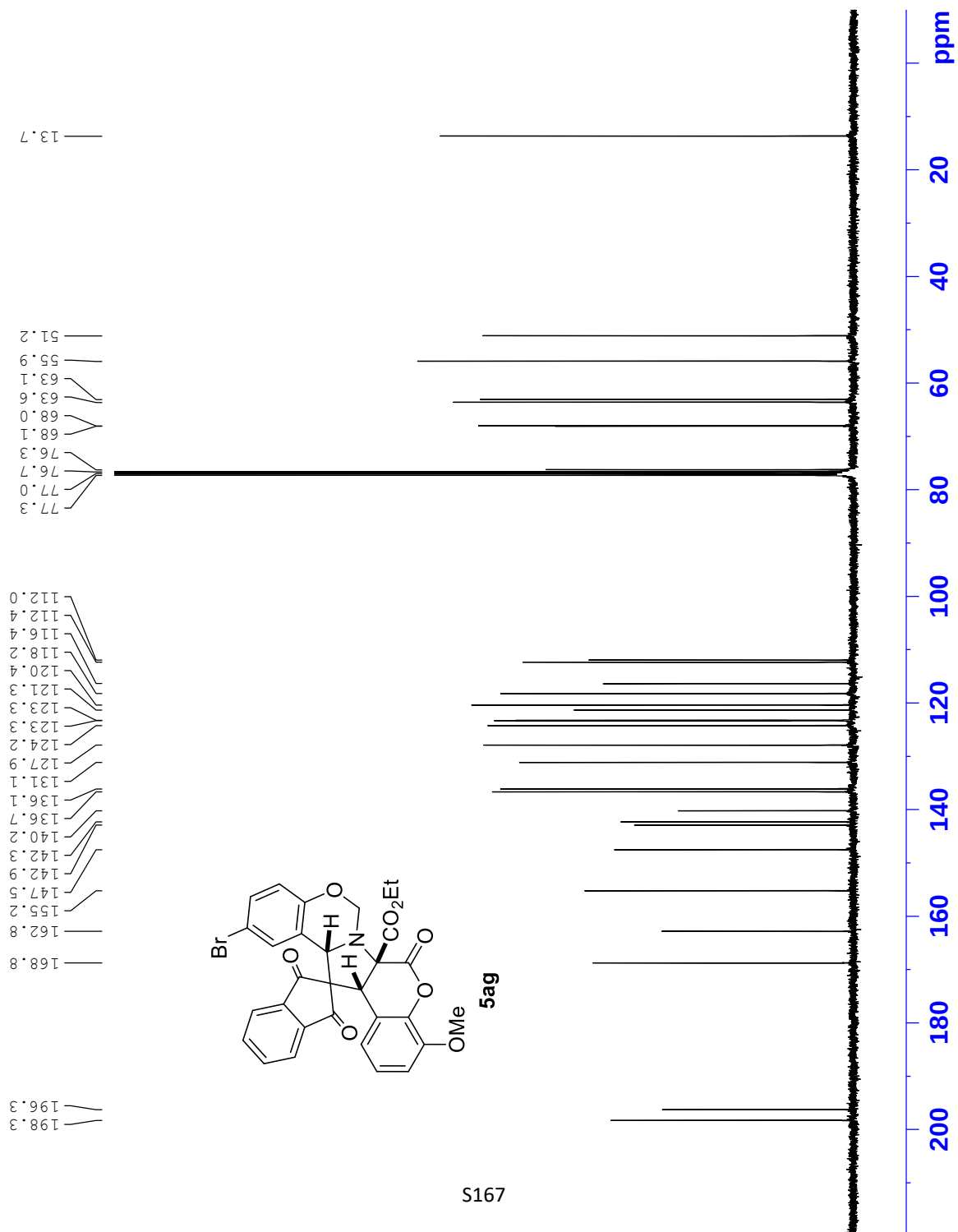
F2 - Acquisition Parameters
 Date_ 20170424
 Time 12.01

INSTRUM spect
 PROBD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 1389
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6815744 sec
 RG 5792.6
 DW 20.800 usec
 DE 6.50 usec
 TE 295.3 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PL1 7.00 dB
 SFO1 100.6233325 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 1.80 dB
 PL12 17.00 dB
 PL13 20.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127791 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00



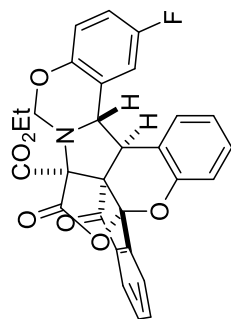
8.03
8.01
7.86
7.84
7.82
7.68
7.66
7.61
7.59
7.57
7.27
7.25
7.23
7.10
7.08
7.06
7.01
6.99
6.90
6.88
6.56
6.53
5.38
5.36
4.86
4.84
4.58
4.56
4.24
4.22
4.21
4.20
4.19
4.18
4.17
4.16
4.10
4.09
4.08
4.07
4.06
4.05
4.04
4.02
3.83
3.80
1.03
1.01
0.99

Current Data Parameters
NAME Hank153
EXPNO 10
PROCNO 1

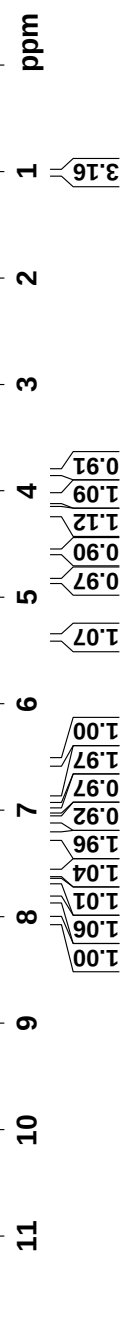
F2 - Acquisition Parameters
Date_ 20180527
Time 4.49
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2609921 sec
RG 57
DW 69.000 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 0 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300065 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



12ha and inseparable impurity



Current Data Parameters
NAME Hank153
EXPNO 11
PROCNO 1

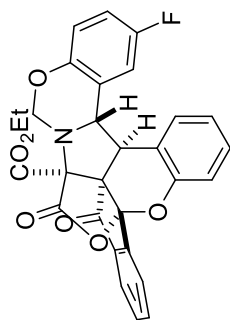
F2 - Acquisition Parameters
Date_ 20180527
Time 4.52
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6293
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 5792.6
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.45 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

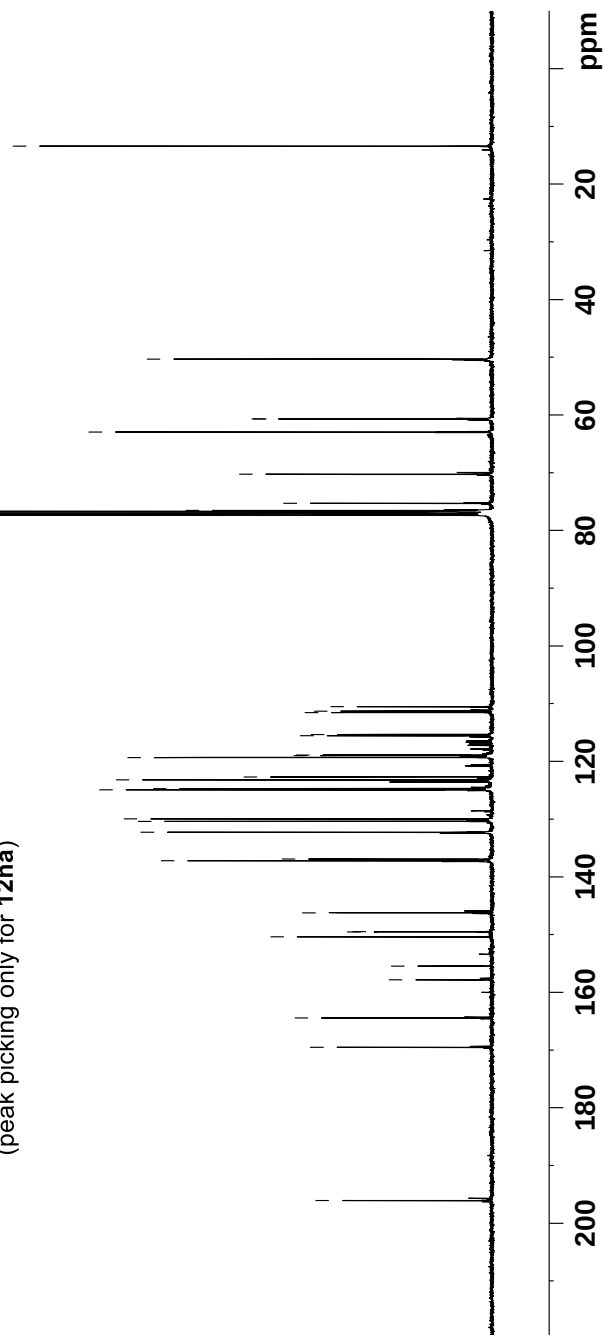
===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0 dB
PL12 15.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127740 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

196.1
169.6
164.5
157.9
155.5
150.4
149.5
149.5
146.2
137.2
136.9
132.3
130.4
130.0
125.0
124.7
123.2
122.7
119.3
119.0
118.9
115.6
115.3
111.5
111.3
110.5
77.3
77.0
76.7
76.5
75.3
70.3
63.0
60.7
60.7
50.3
13.4



12ha and inseparable impurity
(peak picking only for **12ha**)



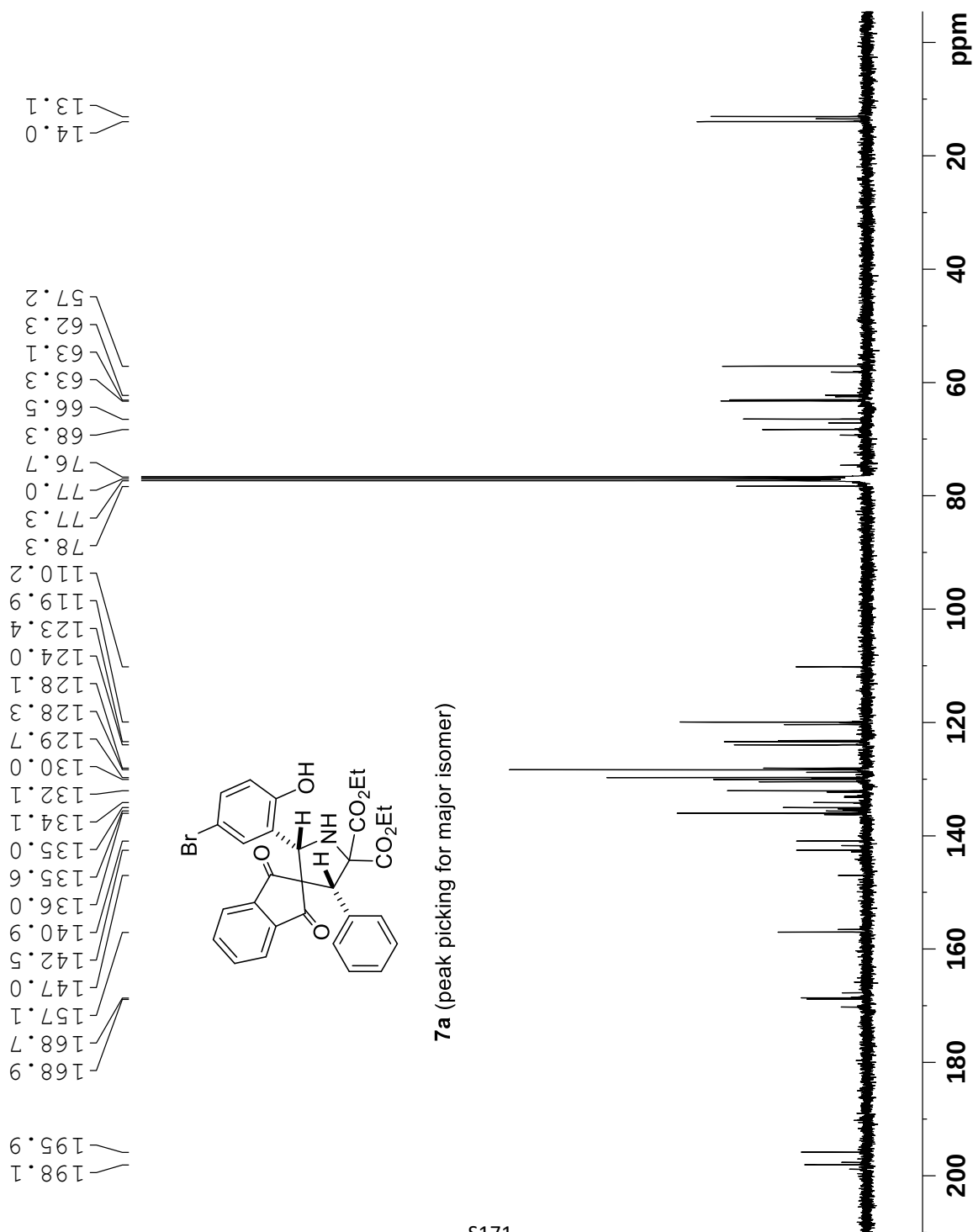
Current Data Parameters
 NAME B350
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180502
 Time_ 8.32
 INSTRUM spect
 PROBD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 1486
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6816244 sec
 RG 2298.8
 DW 20.800 usec
 DE 6.50 usec
 TE 673.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.45 usec
 PL1 7.00 dB
 SFO1 100.623325 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0 dB
 PL12 15.00 dB
 PL13 20.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00



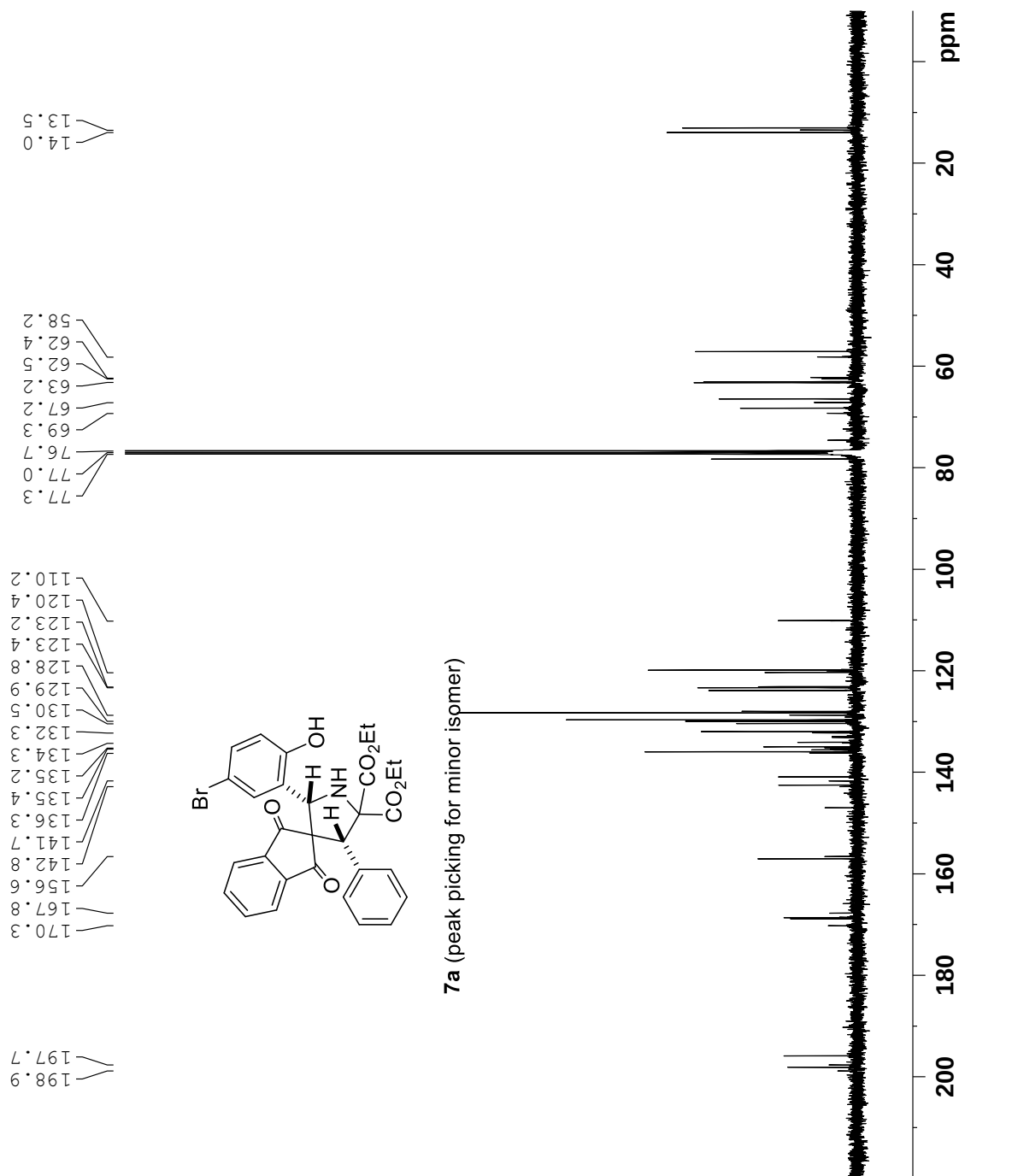
Current Data Parameters
 NAME B350
 EXPNO 10
 PROCNO 1

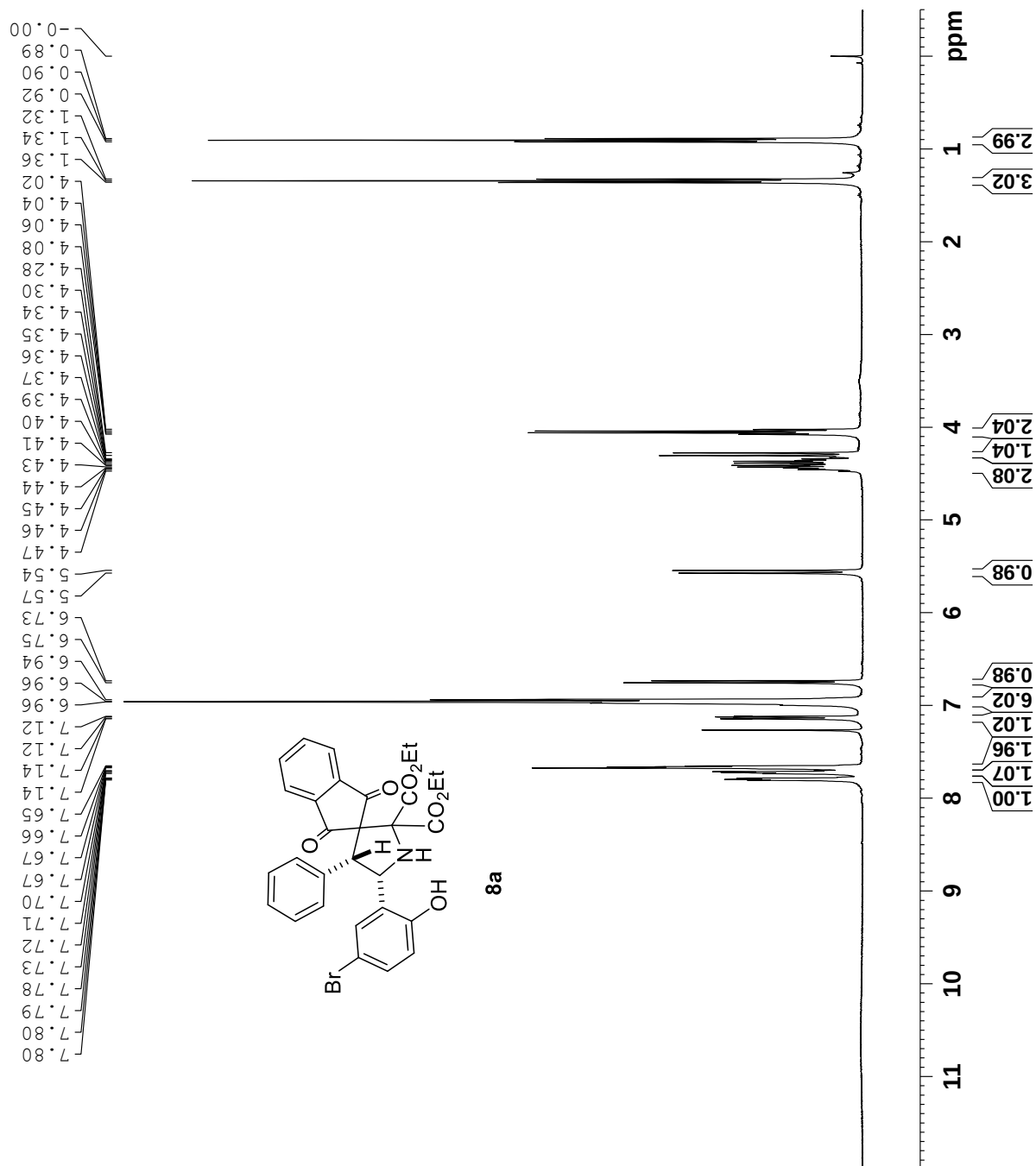
F2 - Acquisition Parameters
 Date_ 20180502
 Time_ 8.32
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 1486
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 0.6815744 sec
 RG 2298.8
 DW 20.800 usec
 DE 6.50 usec
 TE 673.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.45 usec
 PL1 7.00 dB
 SFO1 100.6233325 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0 dB
 PL12 15.00 dB
 PL13 20.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127689 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00





```

Current Data Parameters
NAME      Hank140
EXPNO     3
PROCNO    1

F2 - Acquisition Parameters
Date_     20180430
Time      12.02
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD        32768
SOLVENT   CDCl3
NS        16
DS        0
SWH       7246.377 Hz
FIDRES    0.221142 Hz
AQ        2.260921 sec
RG        114
DW        69.000 usec
DE        6.50 usec
TE        673.2 K
D1        2.0000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      1H
P1        15.00 usec
PL1       0 dB
SFO1      400.1324008 MHz

F2 - Processing parameters
SI        16384
SF        400.1300073 MHz
WDW       EM
SSB       0
LB        0 Hz
GB        0
PC        1.00

```

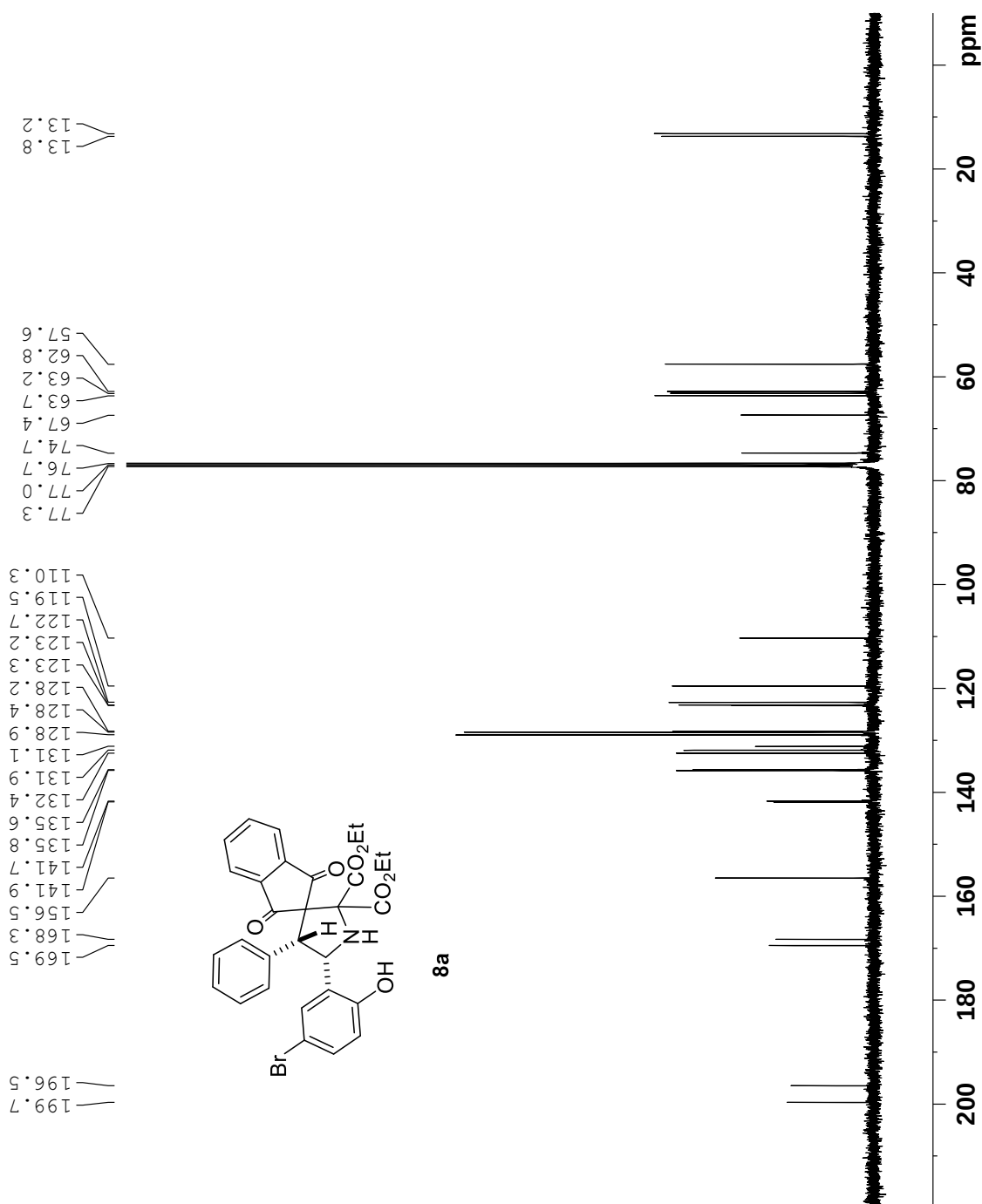
Current Data Parameters
NAME Hank140
EXPNO 4
PROCNO 1

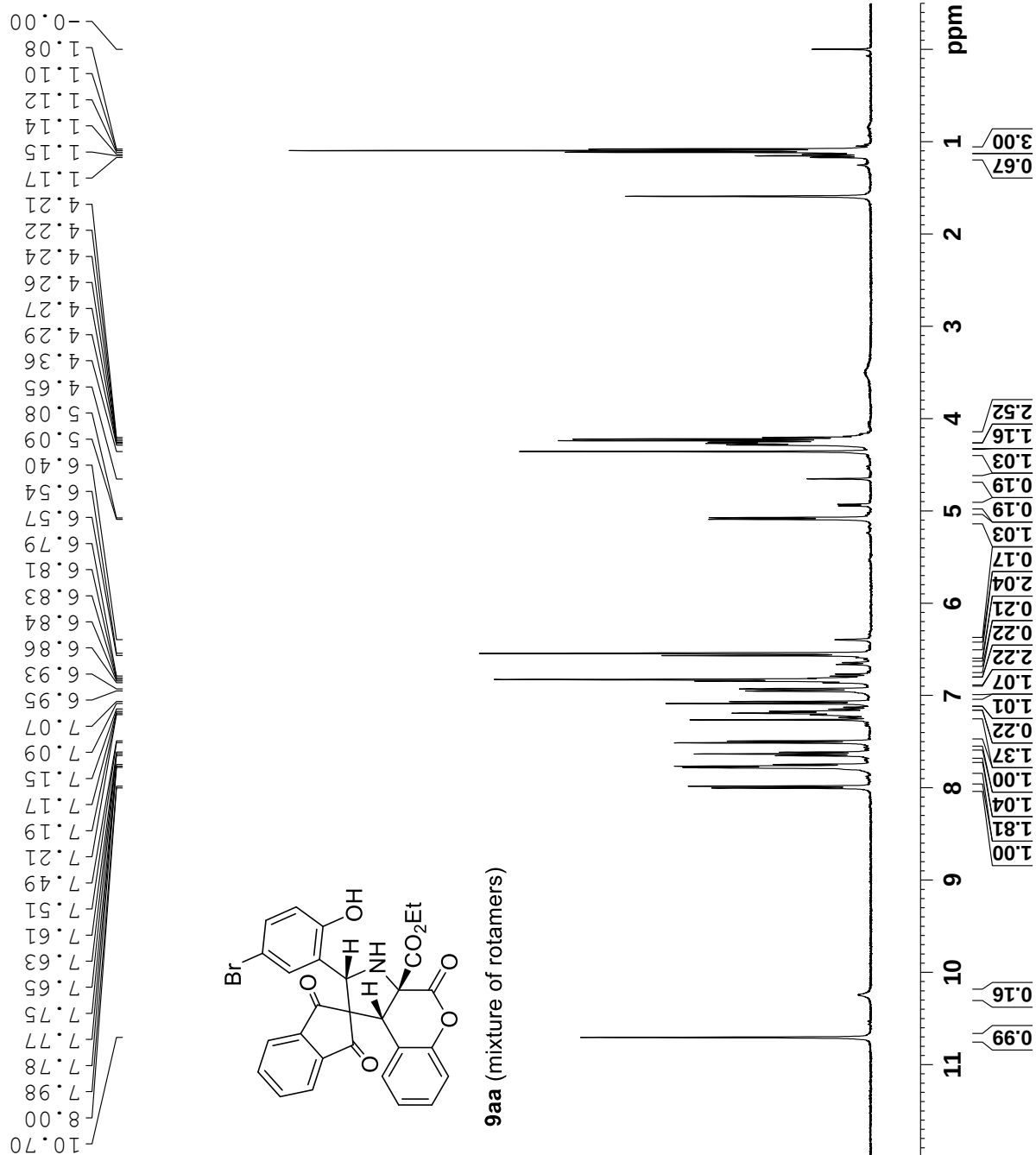
F2 - Acquisition Parameters
Date_ 20180430
Time_ 12.04
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 1257
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 5792.6
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

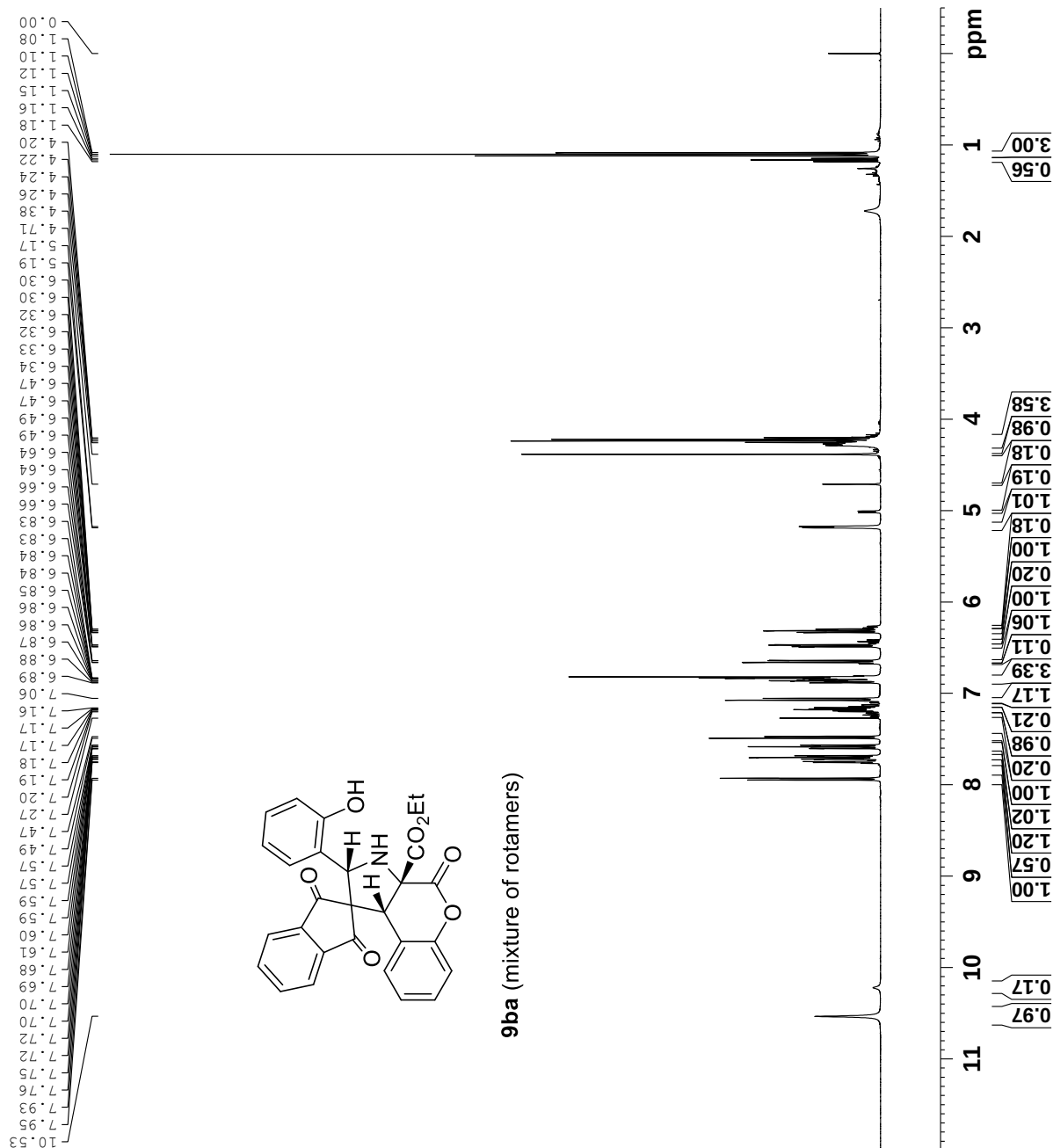
===== CHANNEL f1 =====
NUC1 13C
P1 10.45 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

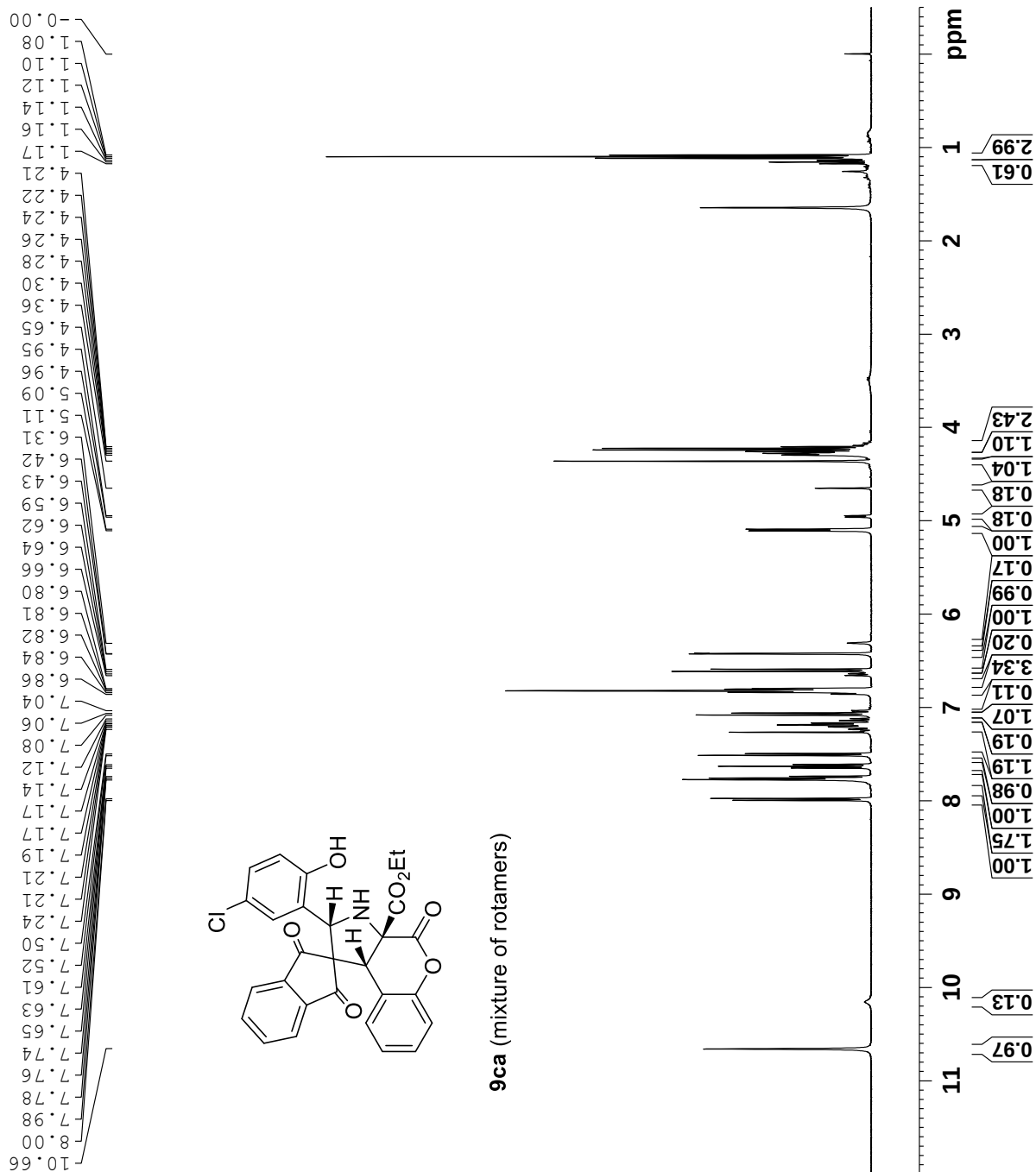
===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0 dB
PL12 15.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

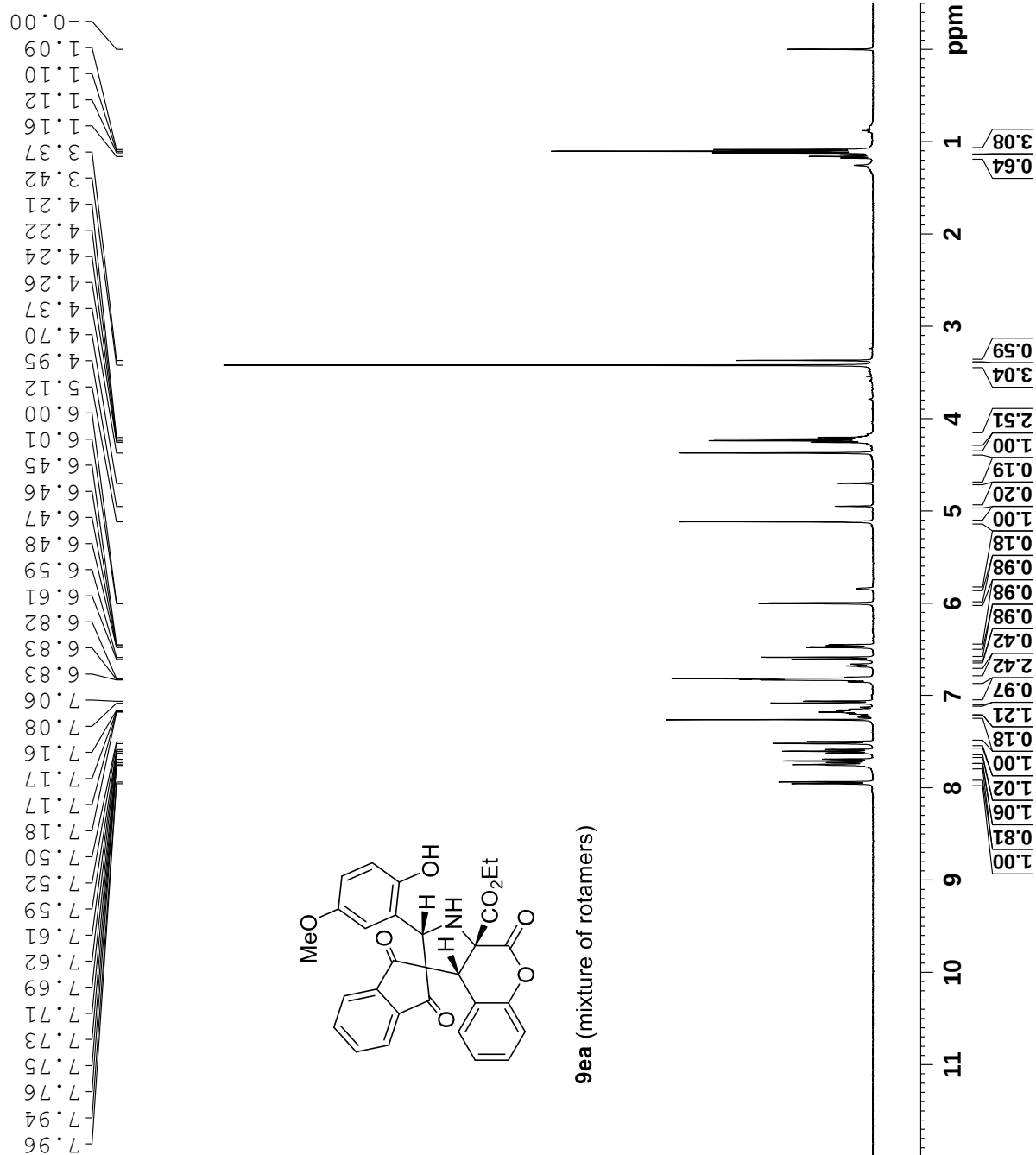
F2 - Processing parameters
SI 32768
SF 100.6127716 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

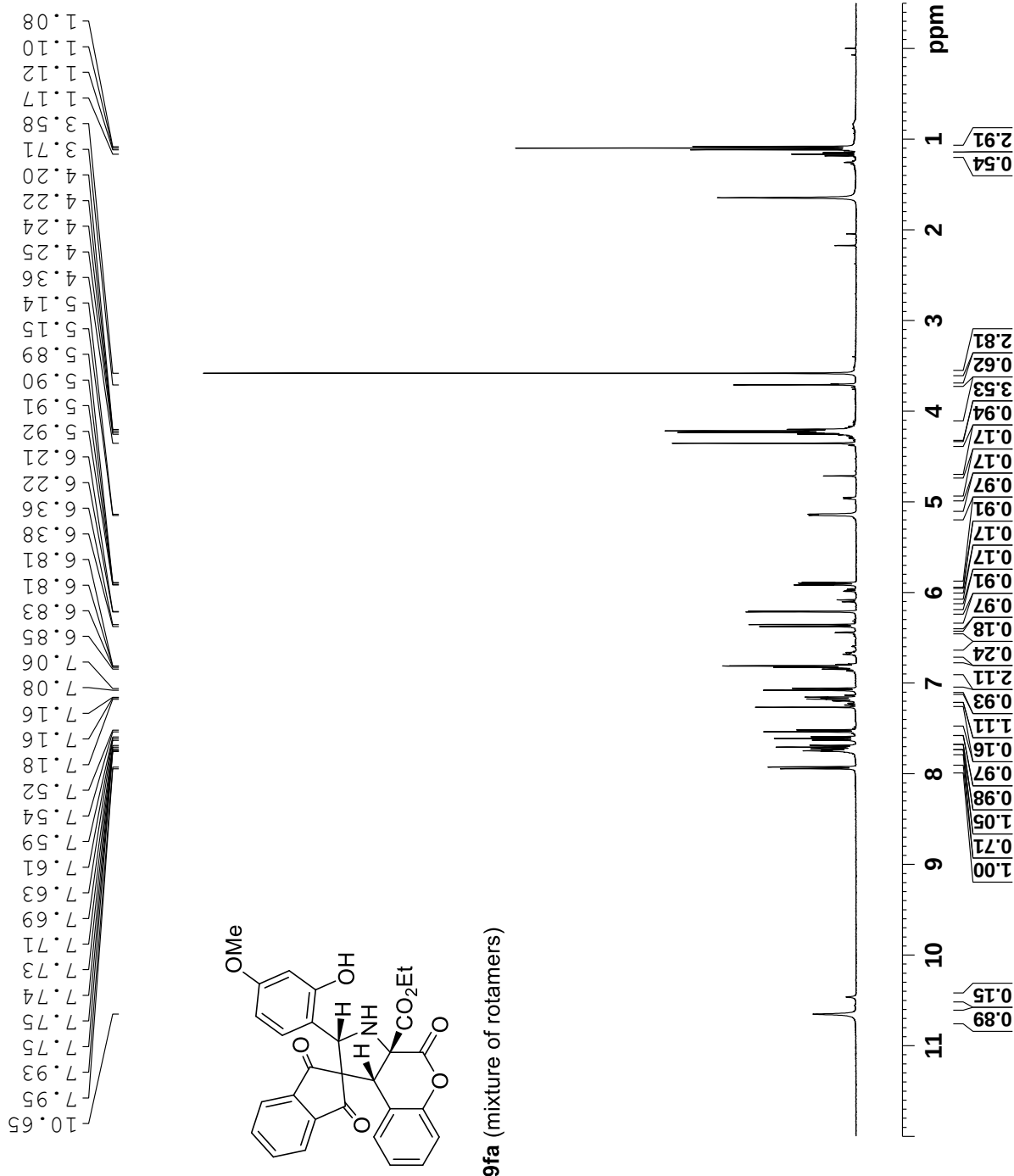


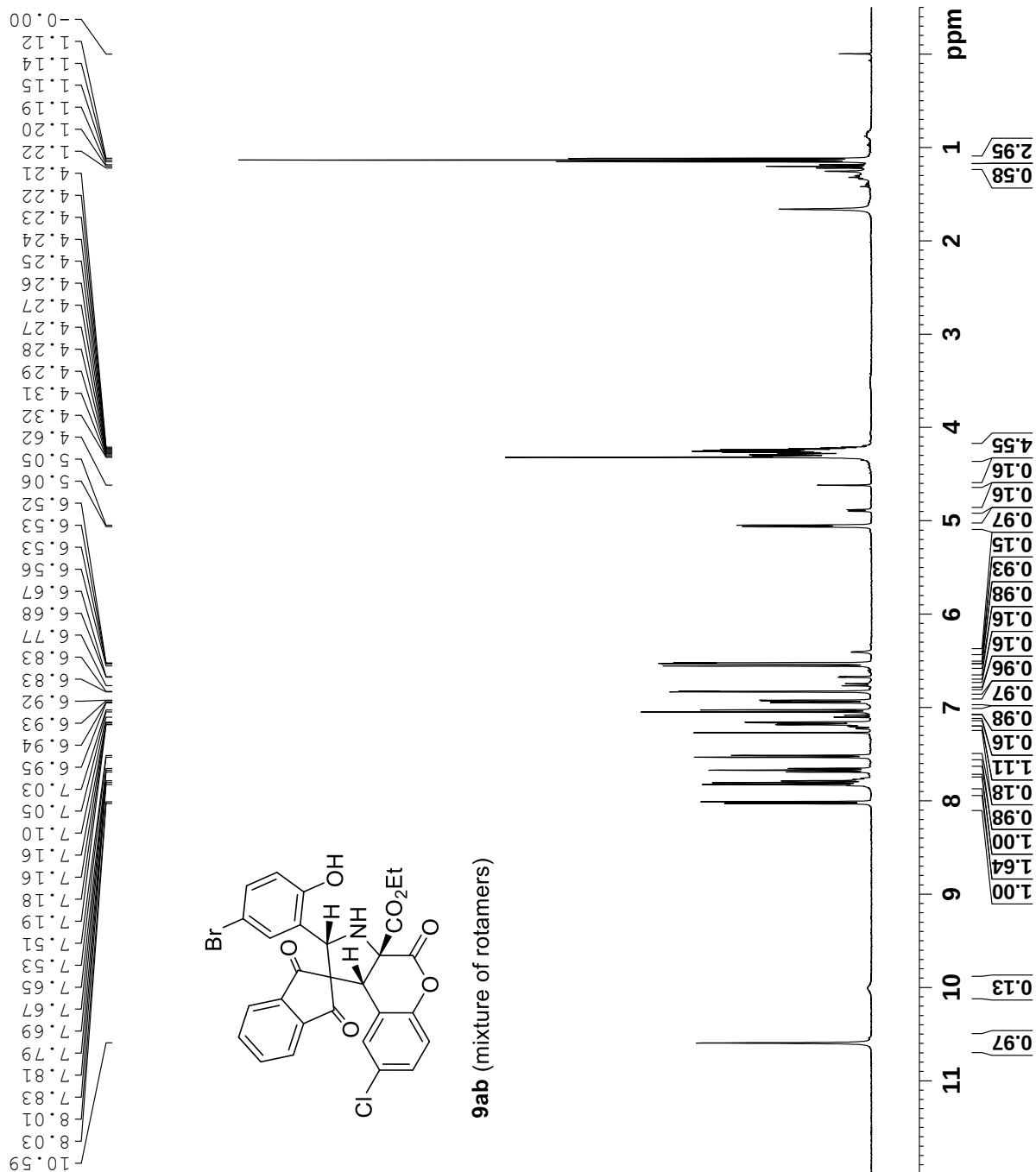


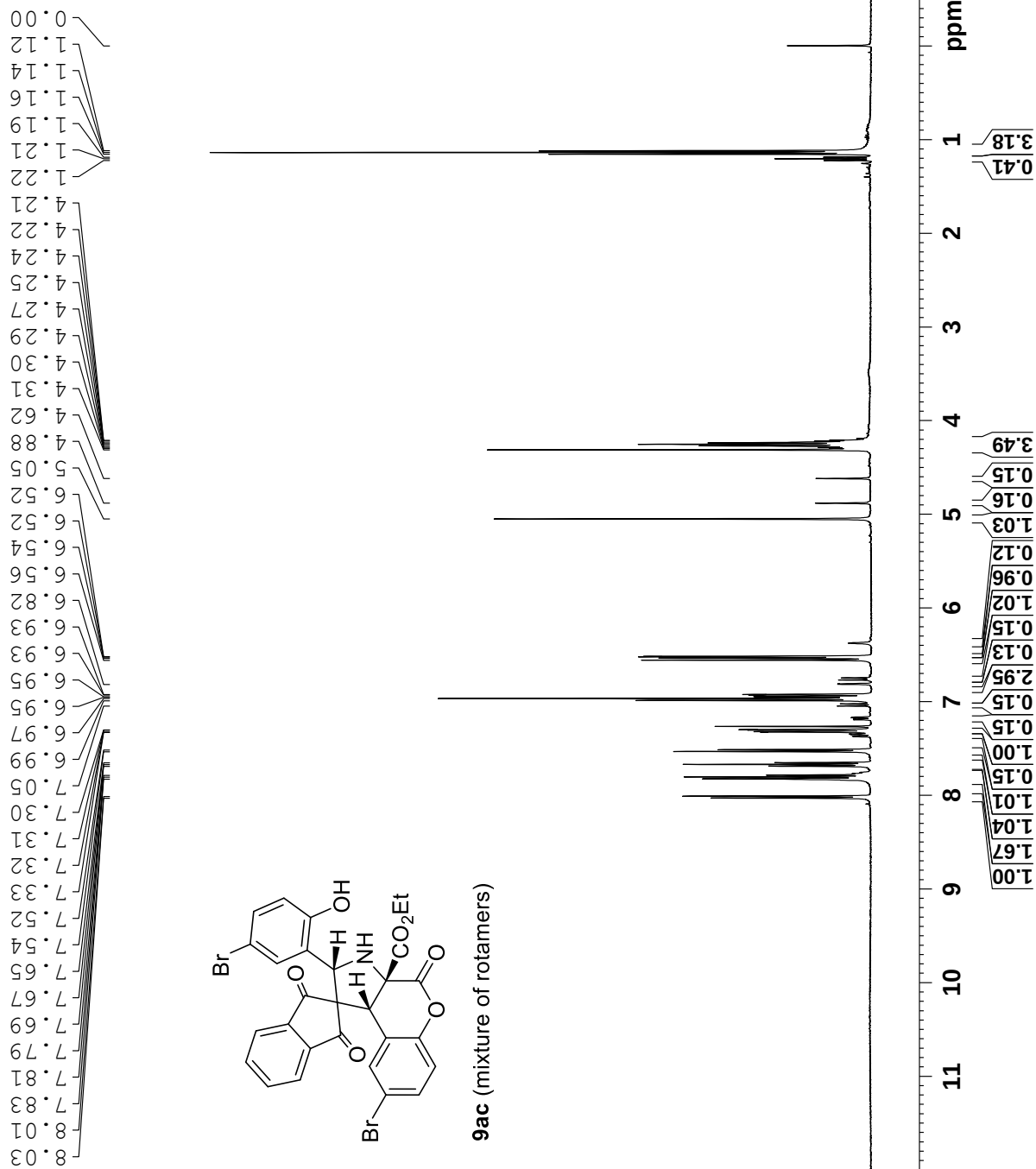


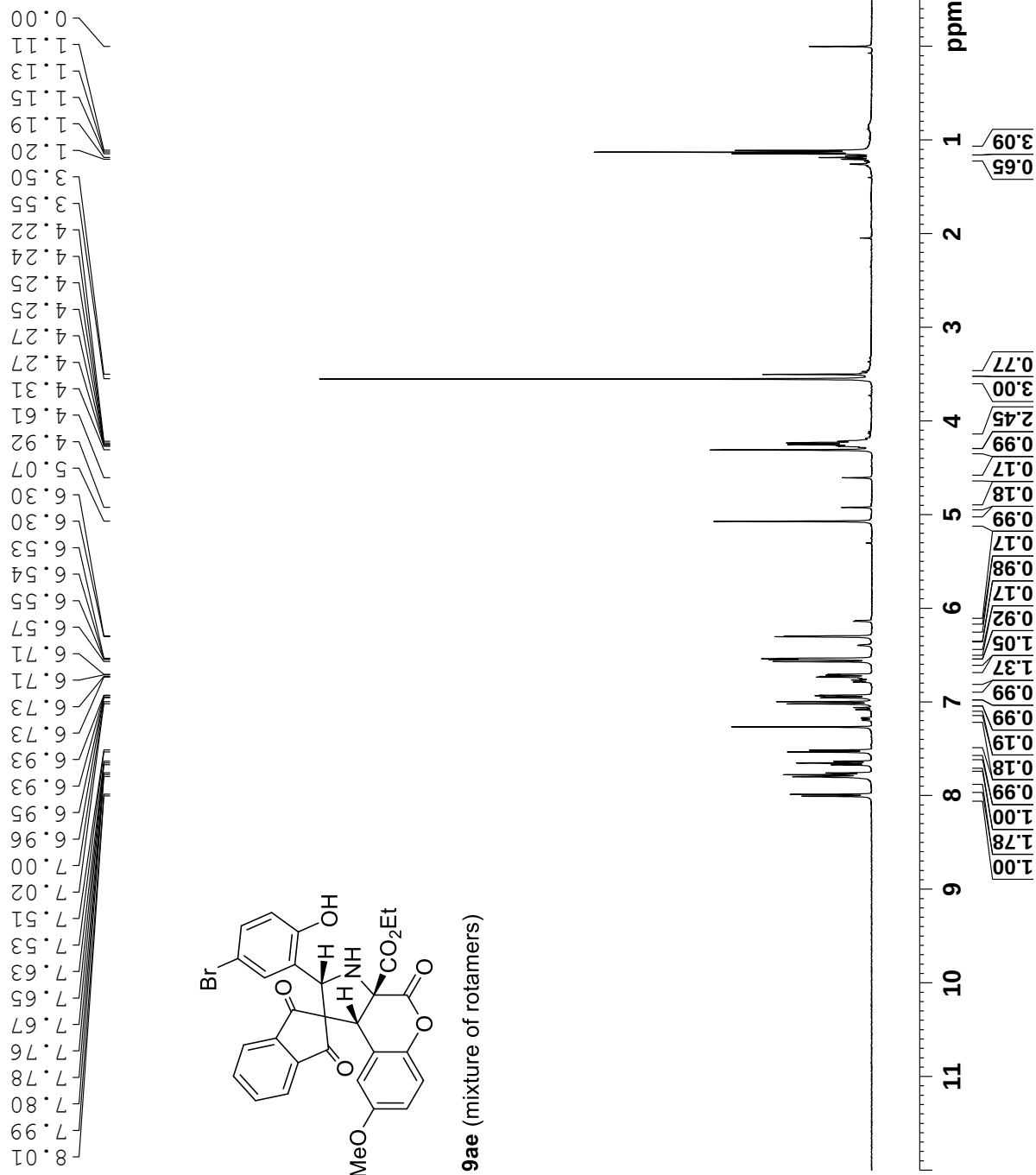


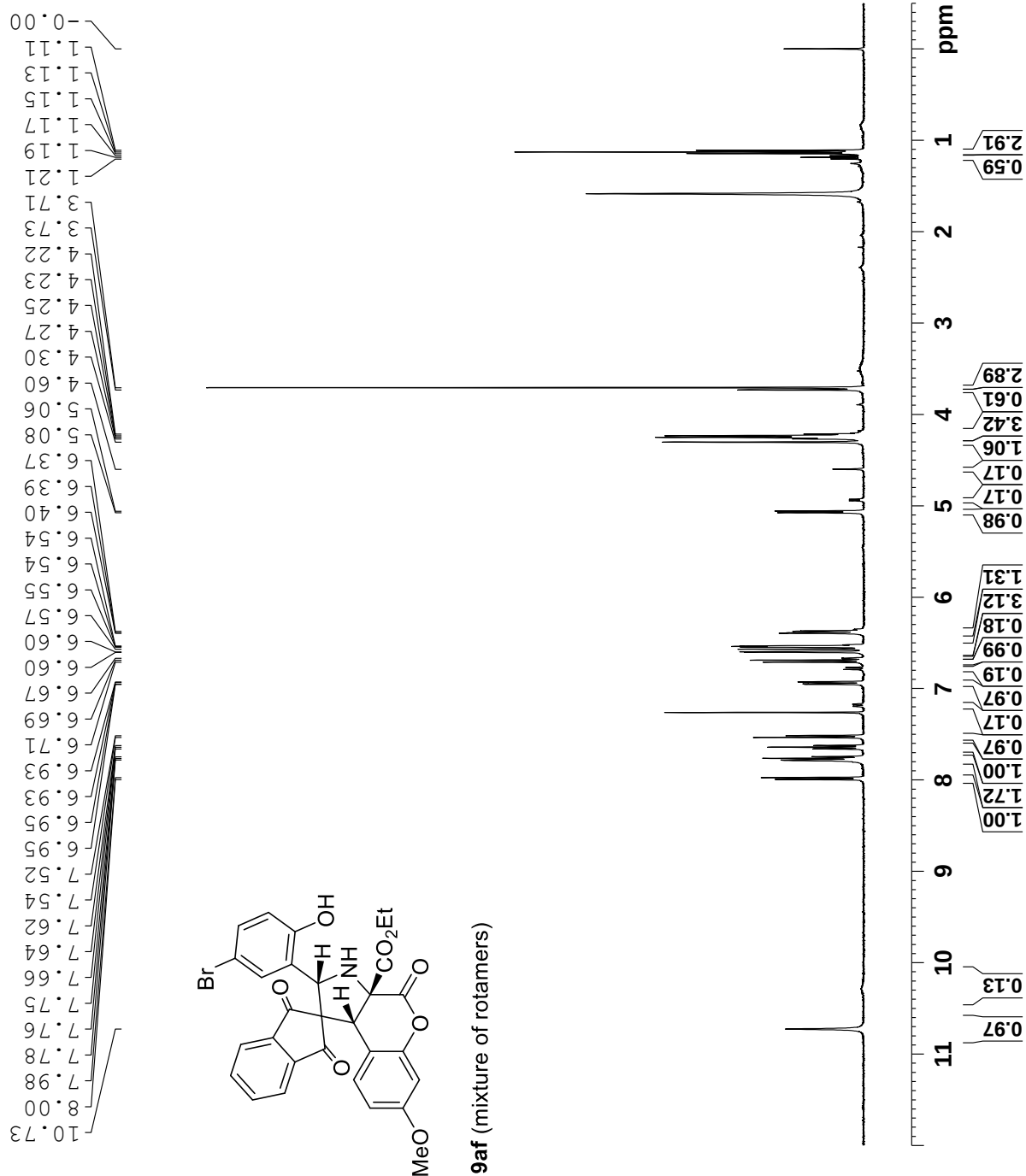


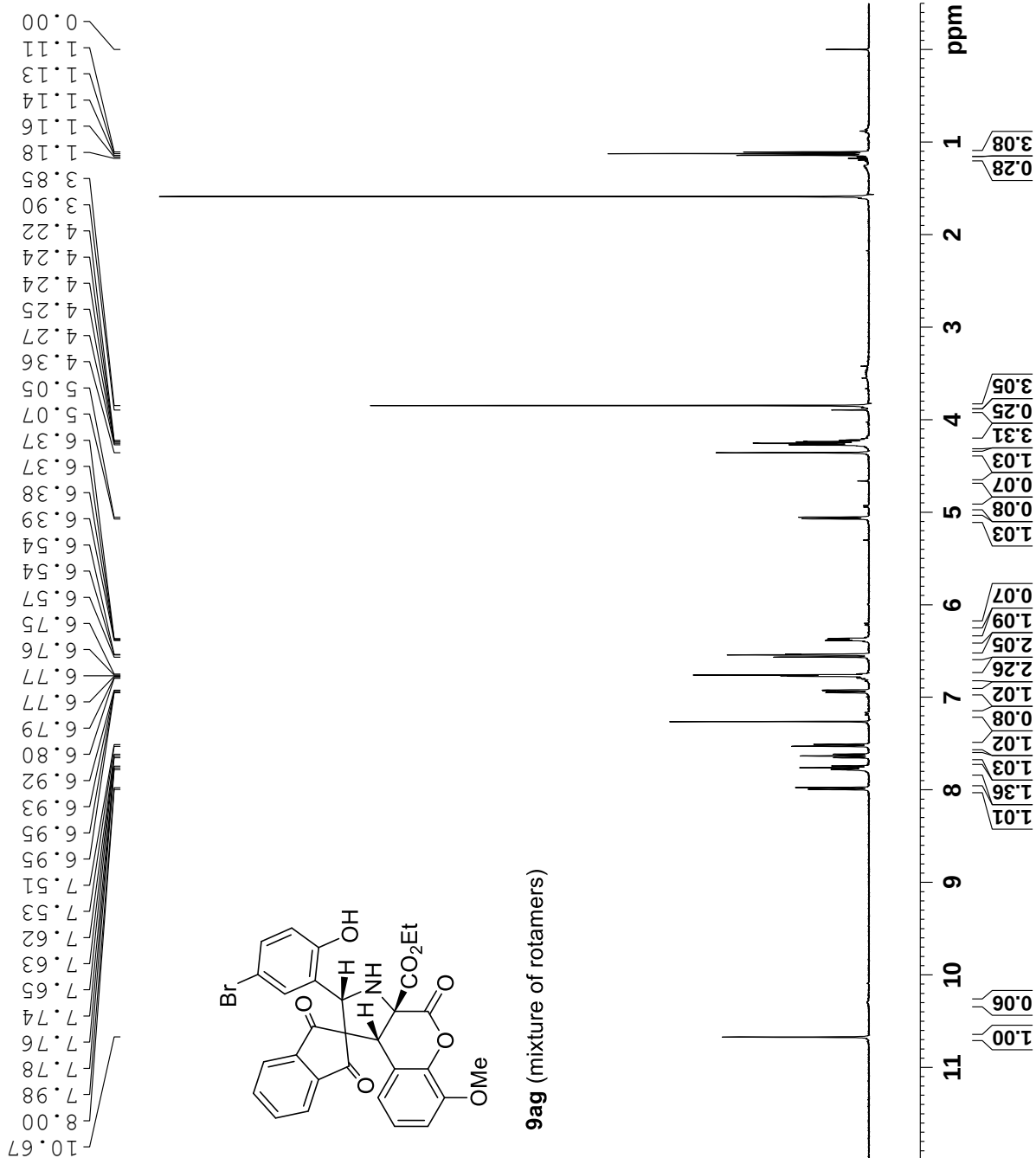




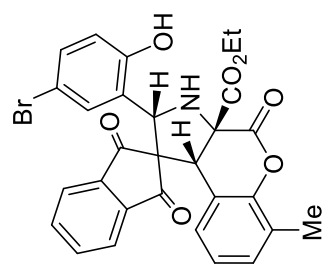








10.77
10.22
8.00
7.98
7.79
7.77
7.75
7.65
7.63
7.61
7.50
7.48
7.18
7.18
7.16
7.07
7.06
7.04
7.02
6.94
6.94
6.92
6.92
6.77
6.75
6.73
6.71
6.69
6.66
6.64
6.55
6.53
6.48
6.46
5.10
5.09
4.95
4.94
4.63
4.37
4.32
4.30
4.28
4.25
4.23
4.22
4.20
2.36
2.31
1.18
1.16
1.14
1.12
1.10
1.08
0.00



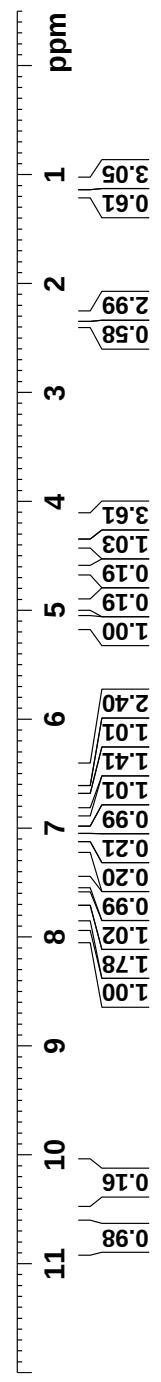
9ah (mixture of rotamers)

Current Data Parameters
NAME 3Me
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170414
Time_ 21.14
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.260921 sec
RG 114
DW 69.000 usec
DE 6.50 usec
TE 295.2 K
D1 2.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.40 usec
PL1 1.80 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300010 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



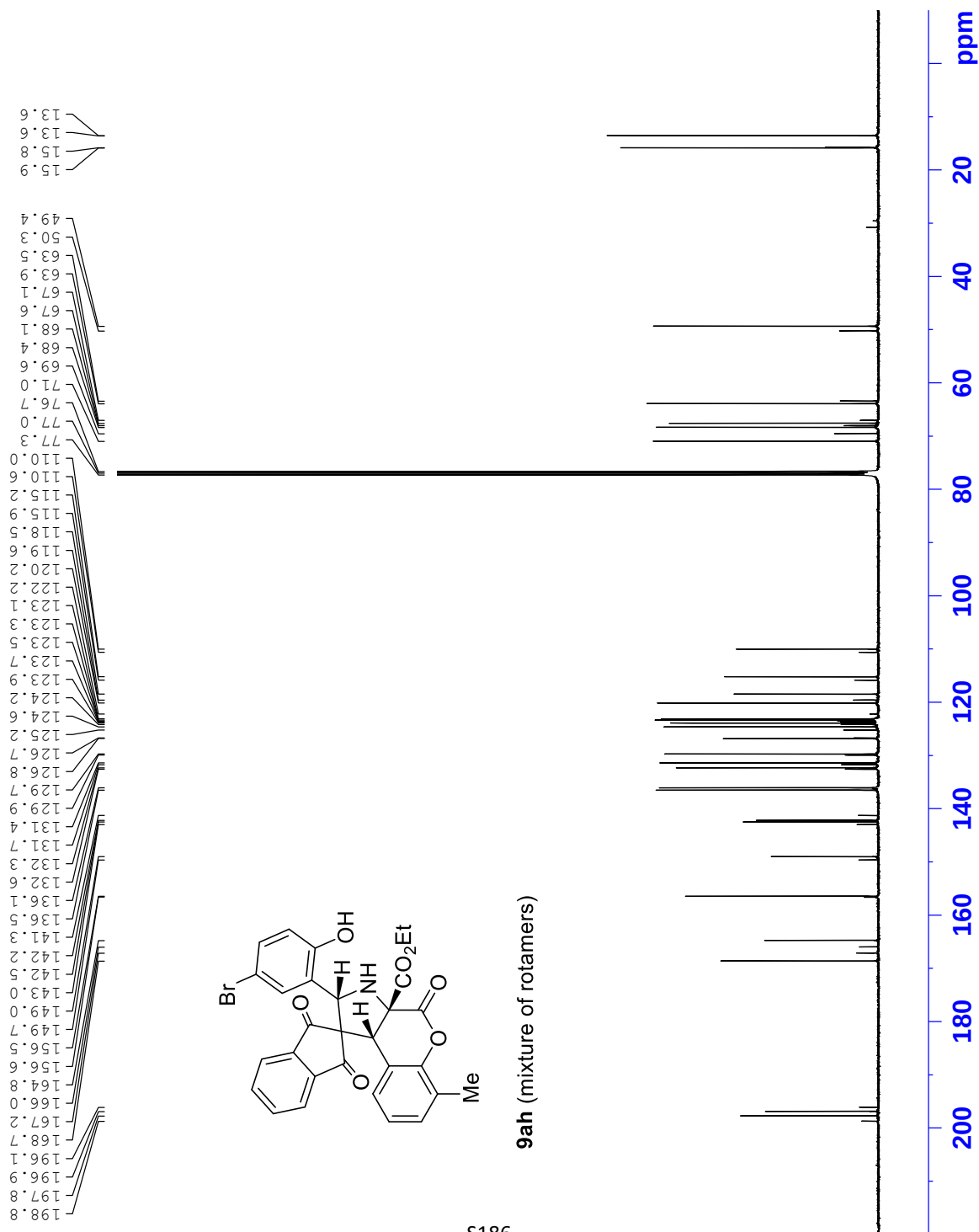
Current Data Parameters
NAME 3Me
EXPNO 3
PROCNO 1

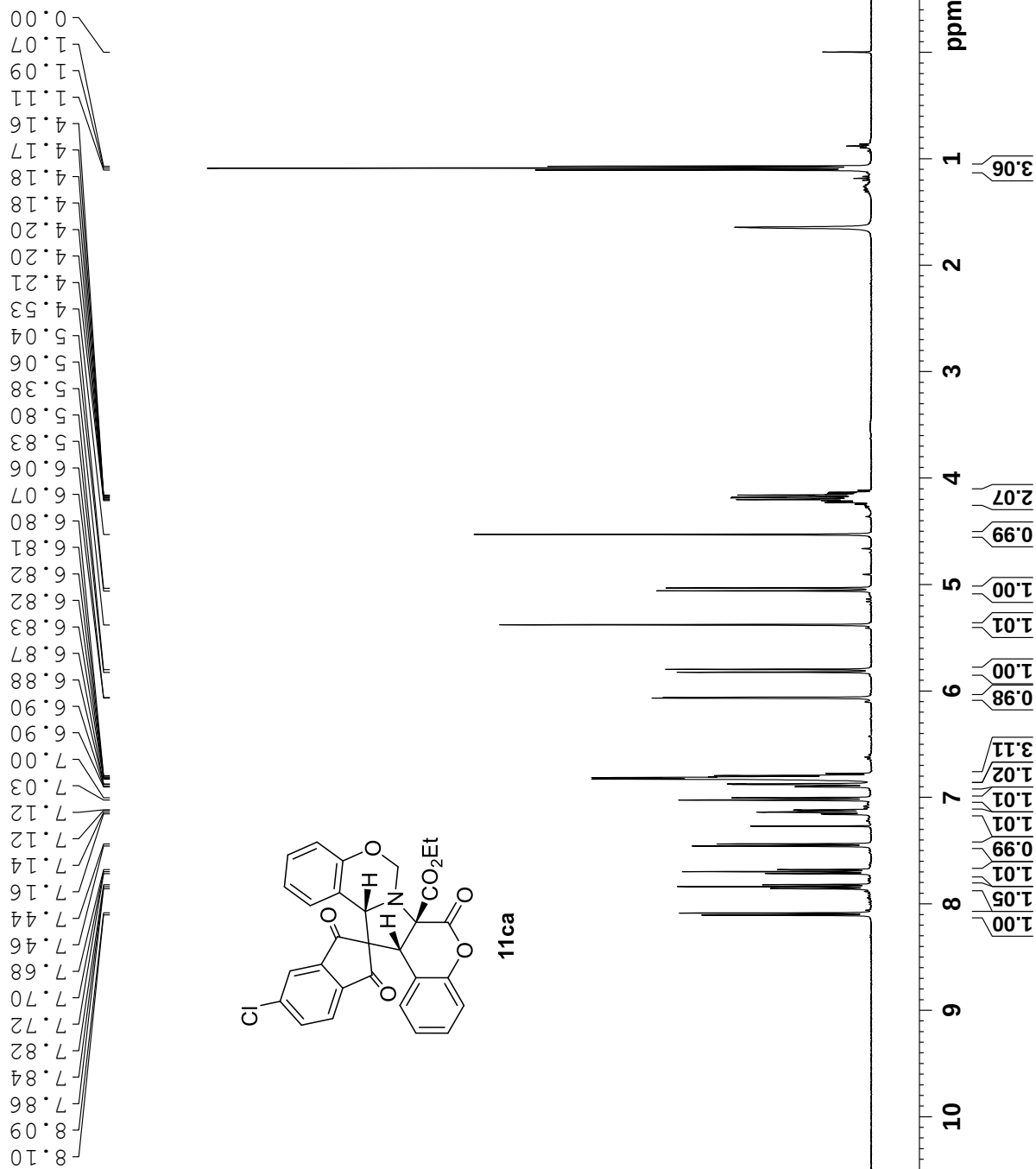
F2 - Acquisition Parameters
Date_ 20170414
Time_ 22.06
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 15008
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 5792.6
DW 20.800 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

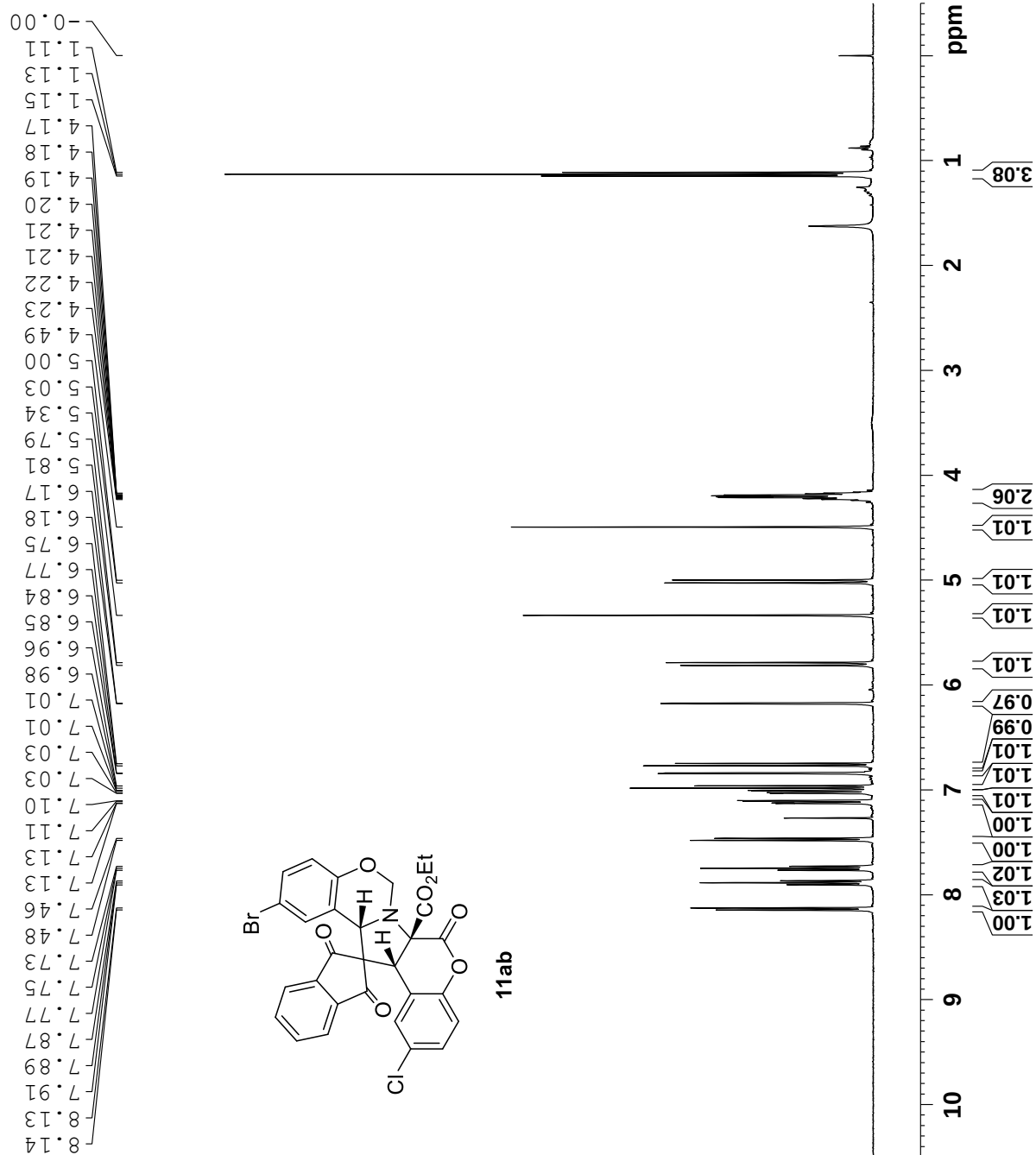
===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127774 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00







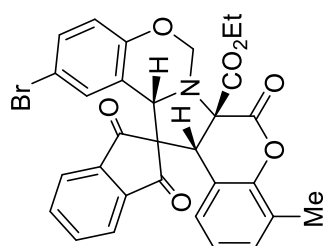
Current Data Parameters	
NAME	Liu160
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

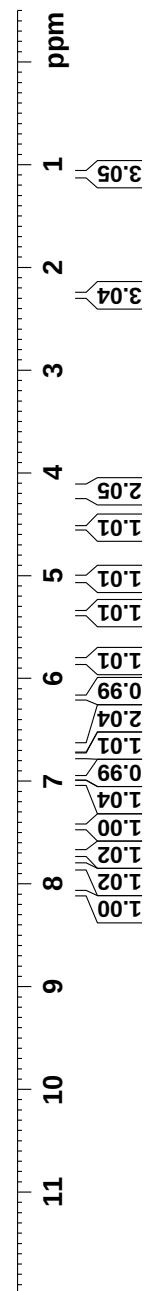
Date_	20170410
Time_	18.08
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2609921 sec
RG	161.3
WDW	69.000 usec
DE	6.50 usec
TE	296.2 K
DD1	2.00000000 sec
TD0	1

```
===== CHANNEL f1 =====
NUC1      1H
P1        14.40 usec
PL1       1.80 dB
SFO1      400.1324008 MHz
```

F2 - Processing parameters	
SSI	16384
SF	400.130061 MHz
WDW	EM
SSB	0
LB	0 Hz
GB	0
PC	1.00



11ah



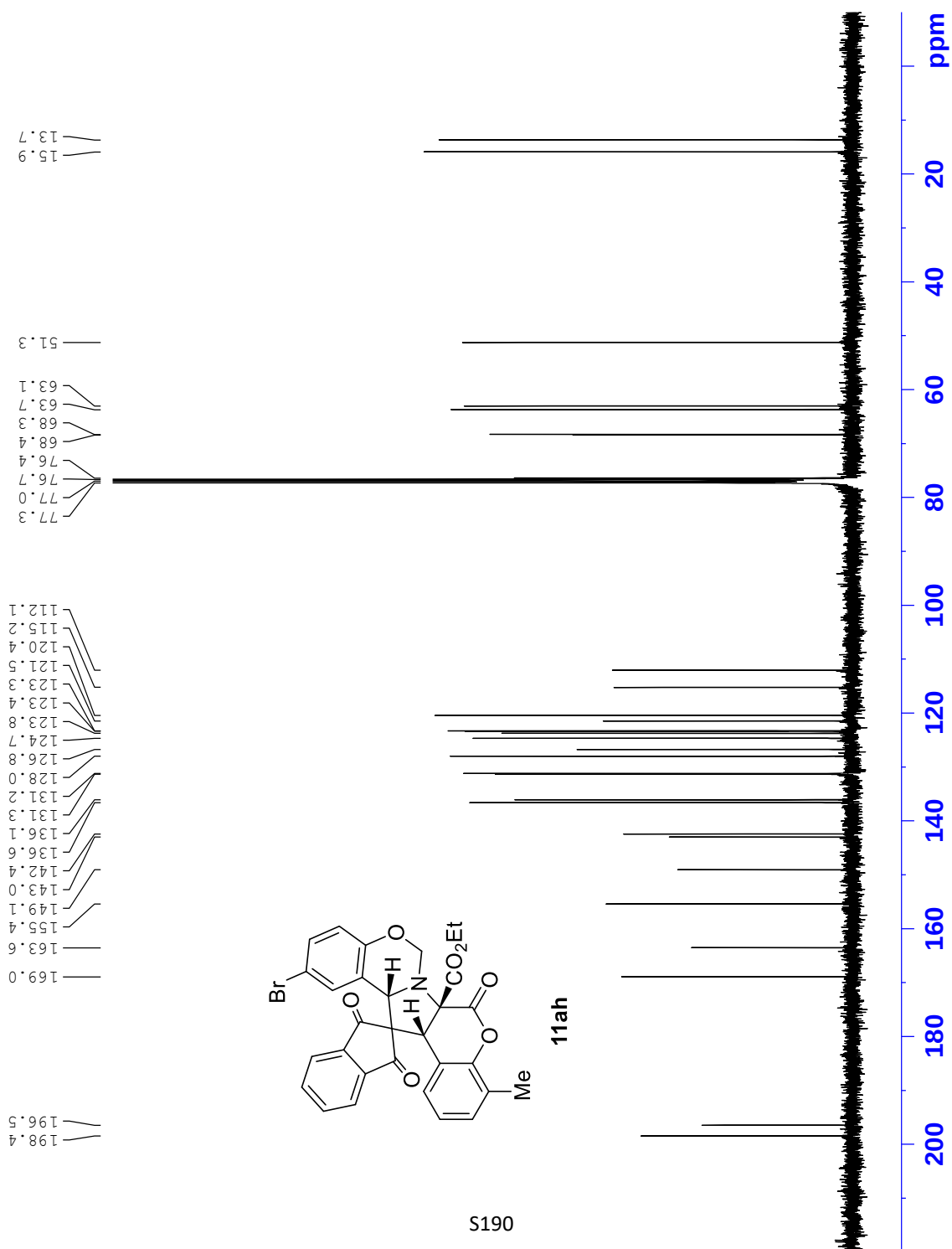
Current Data Parameters
NAME Liul60
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170410
Time 18.11
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2608
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 8192
DW 20.800 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

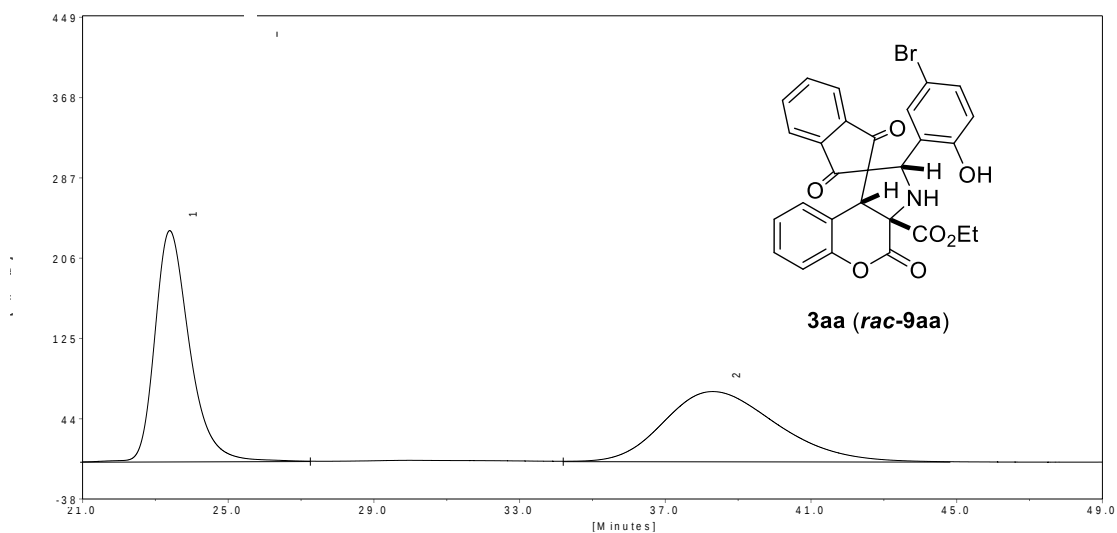
F2 - Processing parameters
SI 32768
SF 100.6127727 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



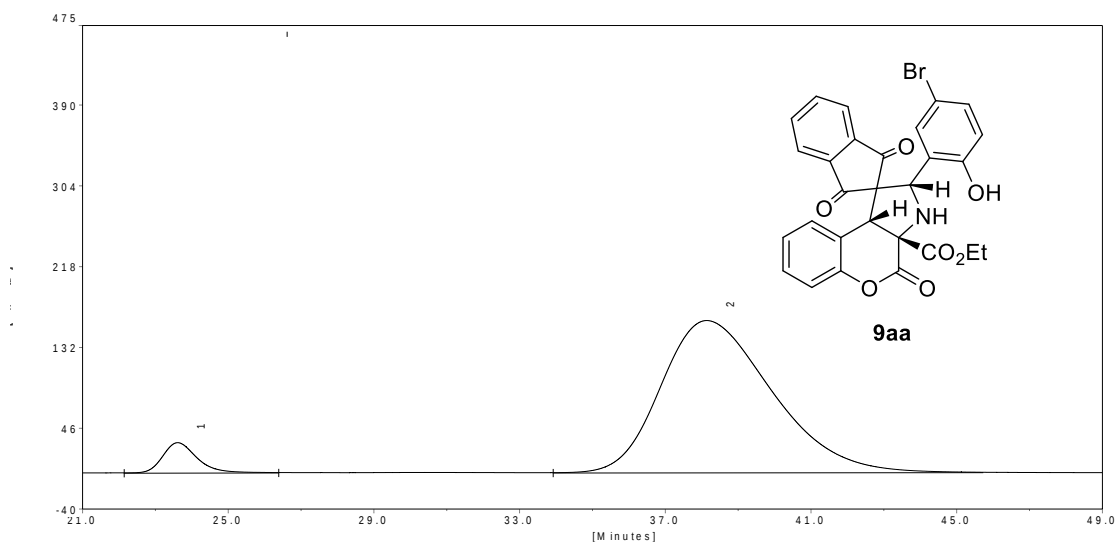
16. HPLC chromatograms of adducts 9

HPLC chromatogram for **9aa**

Column: CHIRALPAK IA	Flow rate: 0.7 ml/min
Solvent: Hex:EtOH = 80: 20	Detector: UV 245 nm



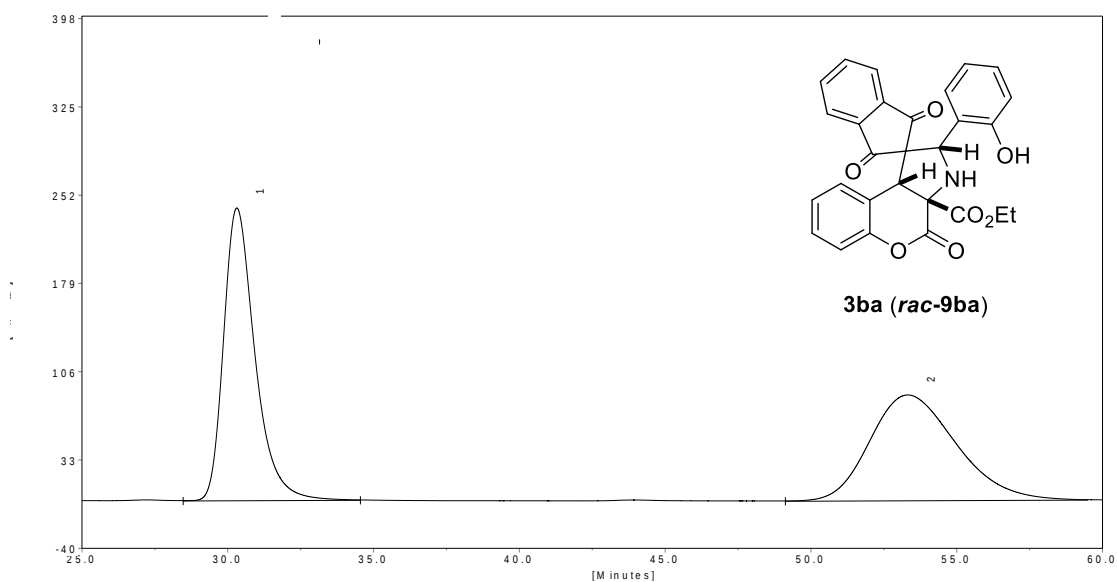
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
23.39	233.04	15351.91	50.3951
38.31	70.49	15111.22	49.6049



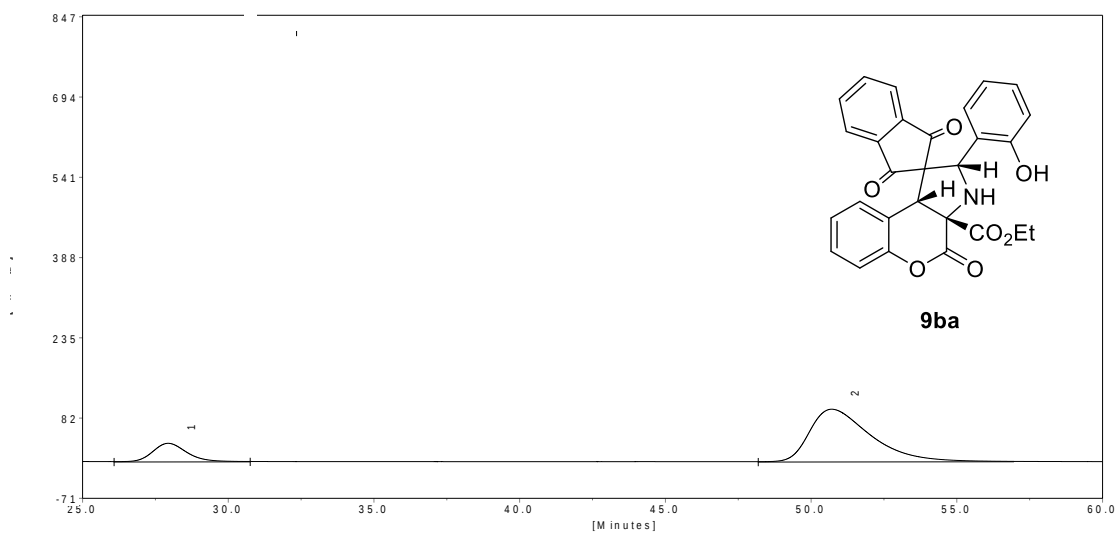
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
23.61	32.01	2061.08	5.6761
38.14	161.74	34250.14	94.3239

HPLC chromatogram for **9ba**

Column: CHIRALPAK IA	Flow rate: 0.7 ml/min
Solvent: Hex:EtOH = 80: 20	Detector: UV 245 nm



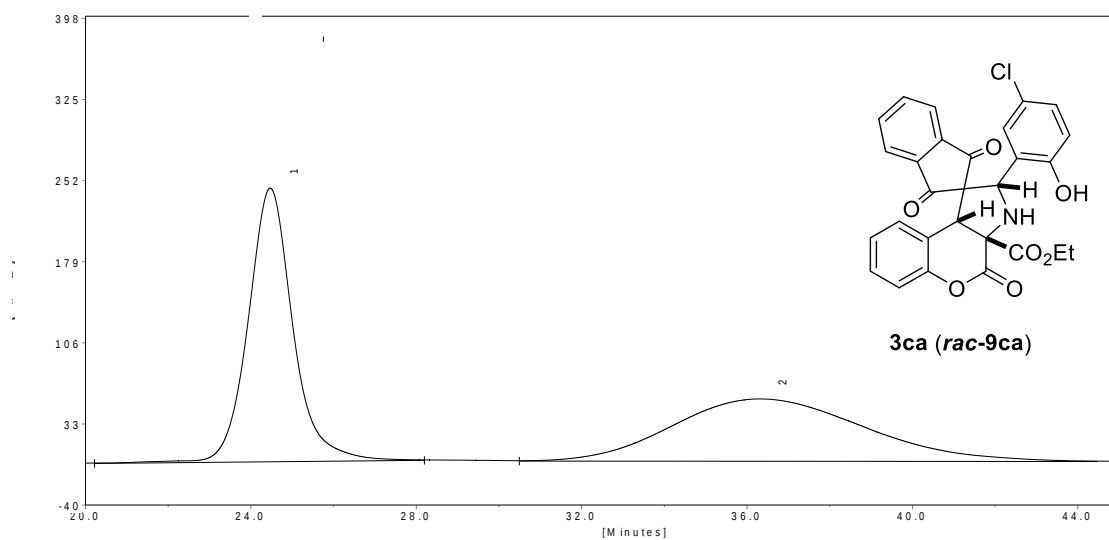
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
30.31	241.83	18045.84	50.2331
53.32	87.20	17878.35	49.7669



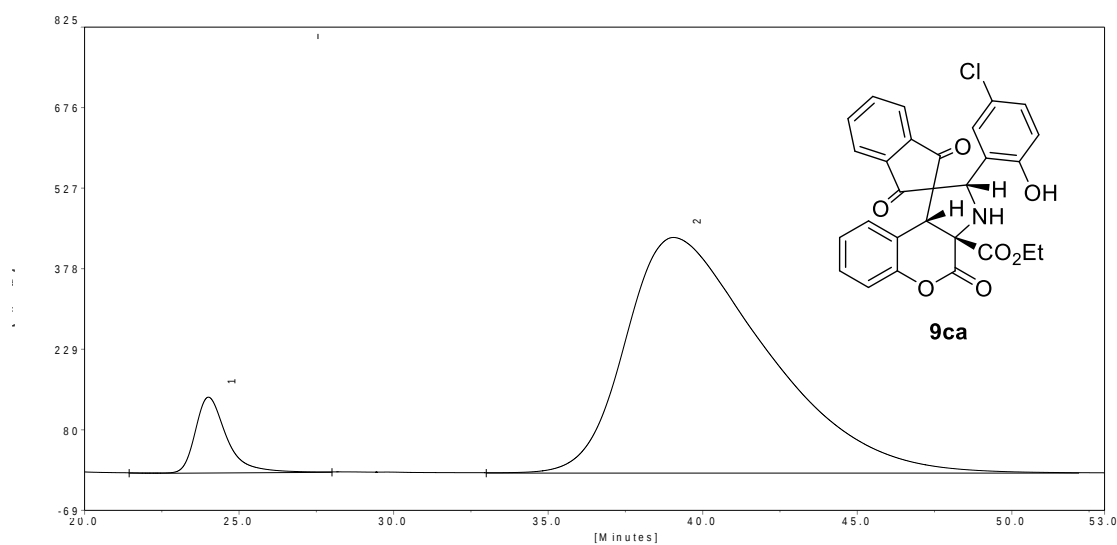
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
27.94	34.79	2765.17	16.0978
50.71	99.85	14412.13	83.9022

HPLC chromatogram for 9ca

Column: CHIRALPAK IA	Flow rate: 0.7 ml/min
Solvent: Hex:EtOH = 80: 20	Detector: UV 245 nm



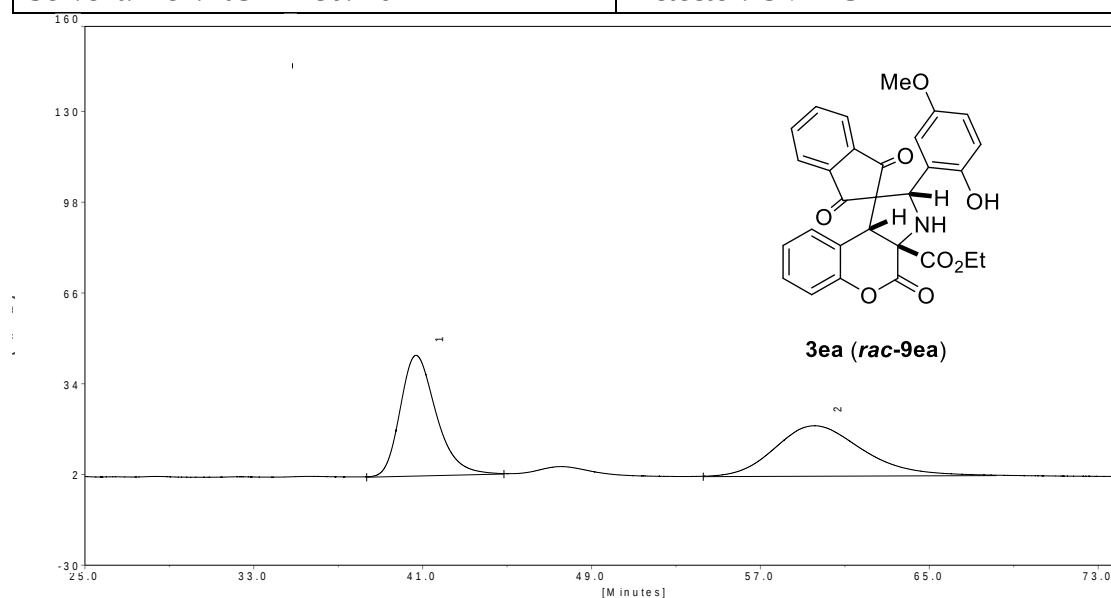
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
24.47	246.06	18397.71	50.8580
36.29	55.74	17776.93	49.1420



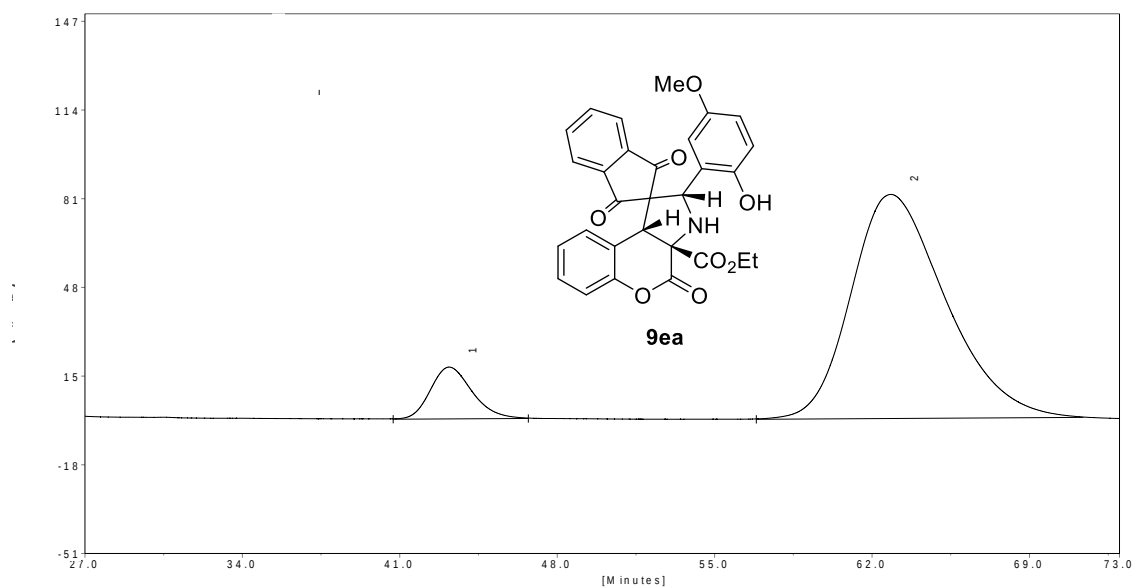
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
24.01	139.20	9597.82	6.5248
39.06	435.05	137499.40	93.4752

HPLC chromatogram for 9ea

Column: CHIRALPAK IA	Flow rate: 0.7 ml/min
Solvent: Hex:EtOH = 80: 20	Detector: UV 245 nm



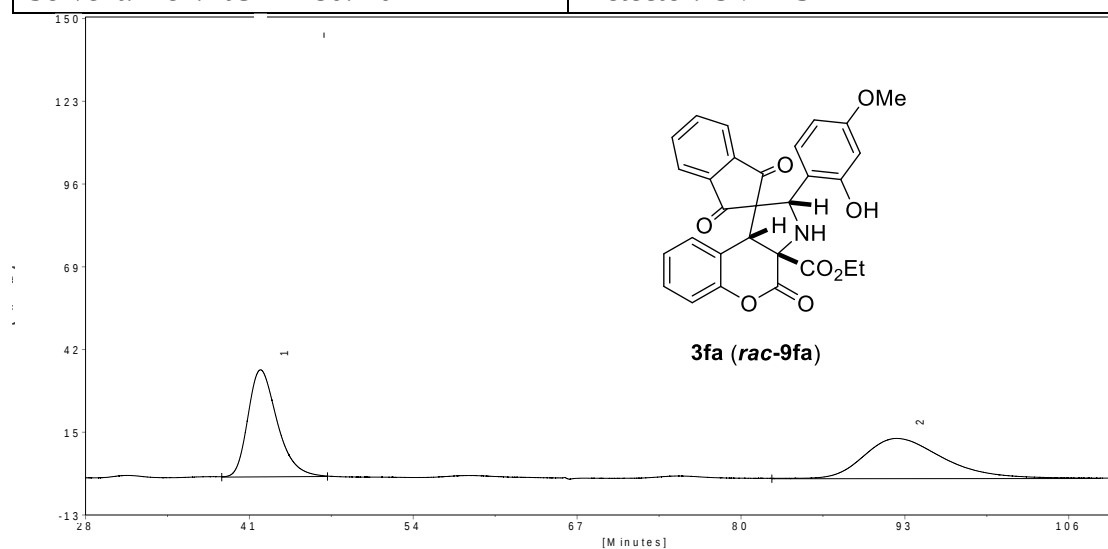
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
40.69	42.43	4999.48	49.6542
59.55	17.66	5069.12	50.3458



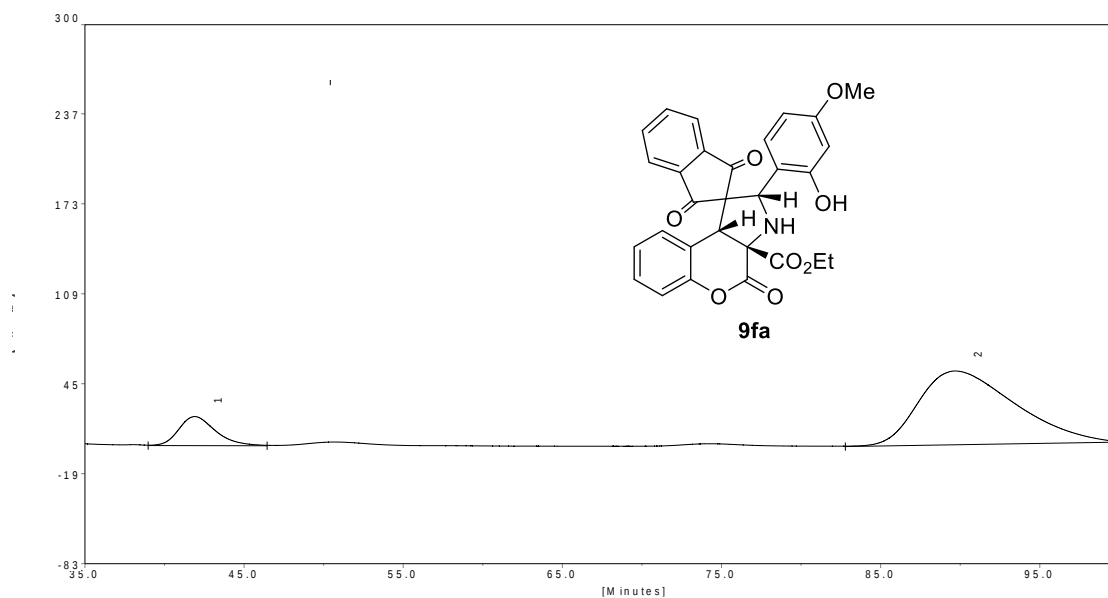
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
43.20	19.14	2431.42	9.1001
62.84	83.23	24287.13	90.8999

HPLC chromatogram for 9fa

Column: CHIRALPAK IA	Flow rate: 0.7 ml/min
Solvent: Hex:EtOH = 80: 20	Detector: UV 245 nm



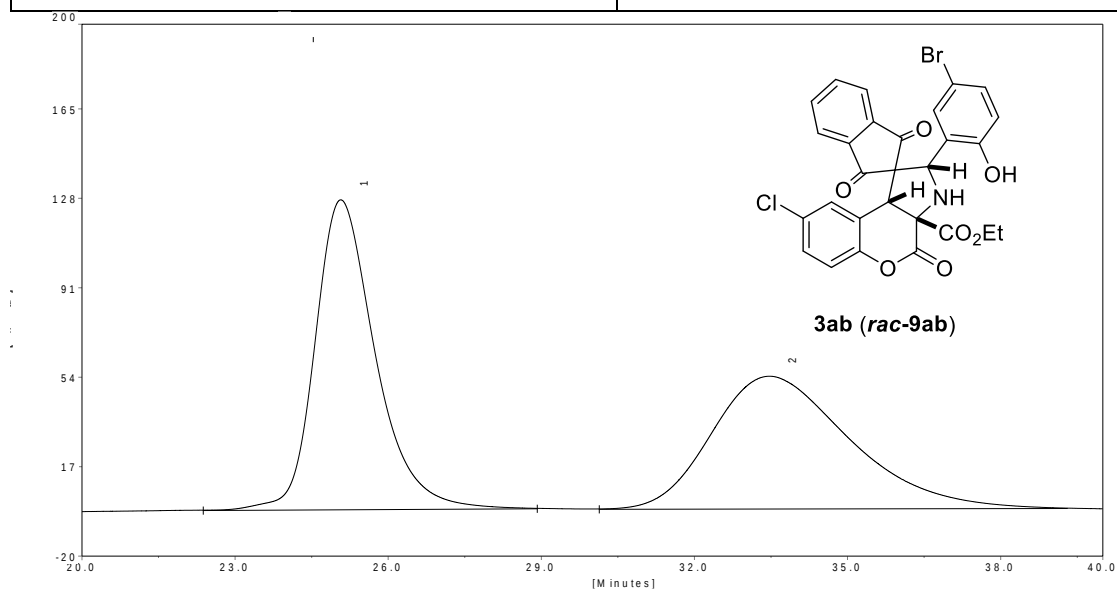
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
41.90	34.81	5568.92	49.8442
92.33	12.99	5603.73	50.1558



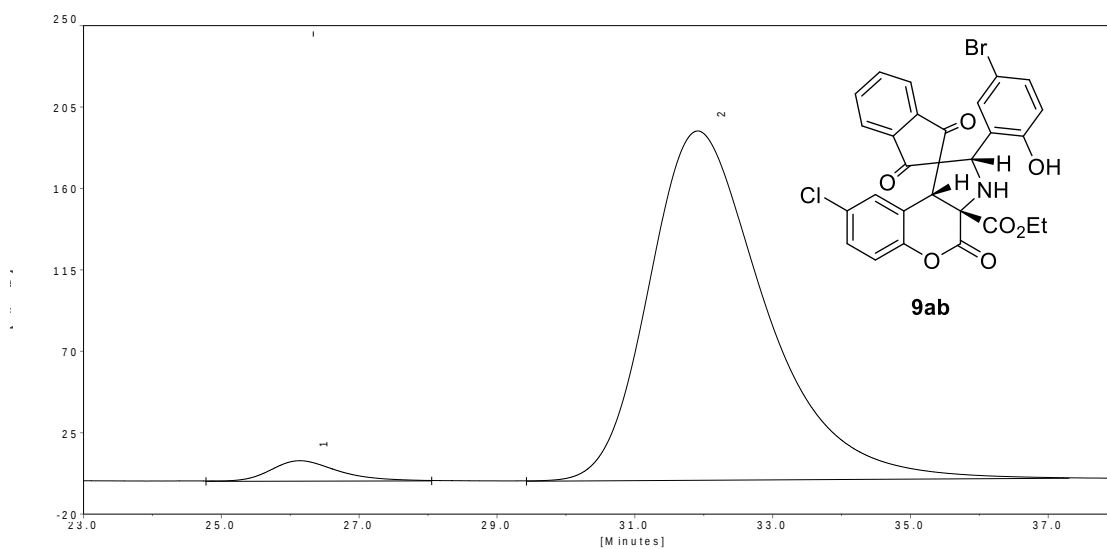
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
41.90	20.40	3191.36	12.7666
89.65	52.20	21806.46	87.2334

HPLC chromatogram for 9ab

Column: CHIRALPAK IA	Flow rate: 0.7 ml/min
Solvent: Hex:EtOH = 80:20	Detector: UV 245 nm



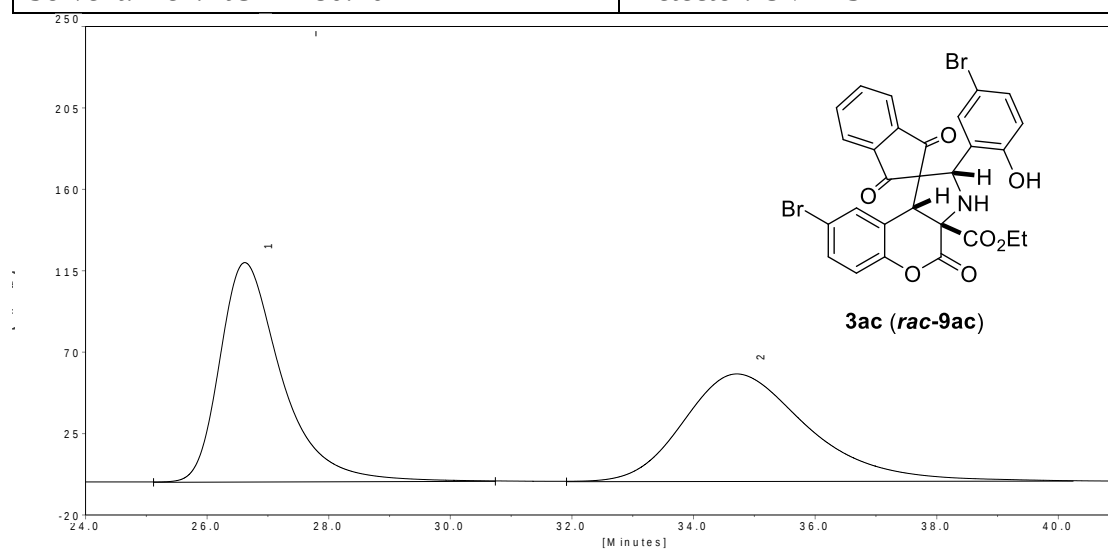
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
25.07	128.05	10485.75	50.5154
33.47	54.77	10271.78	49.4846



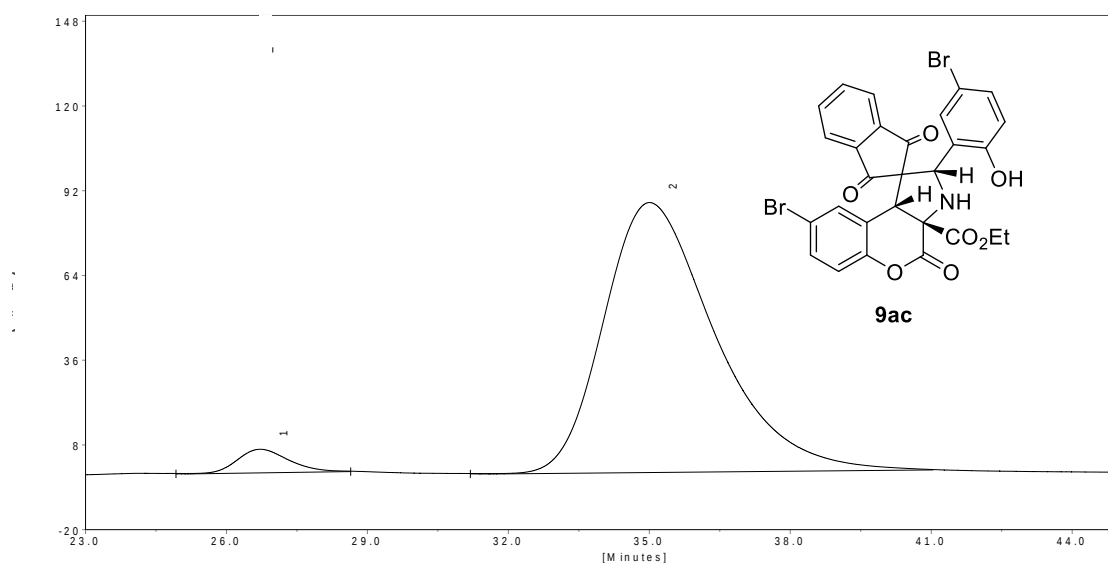
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
26.14	11.07	726.80	3.1104
31.91	192.86	22640.39	96.8896

HPLC chromatogram for **9ac**

Column: CHIRALPAK IA	Flow rate: 0.7 ml/min
Solvent: Hex:EtOH = 80:20	Detector: UV 245 nm



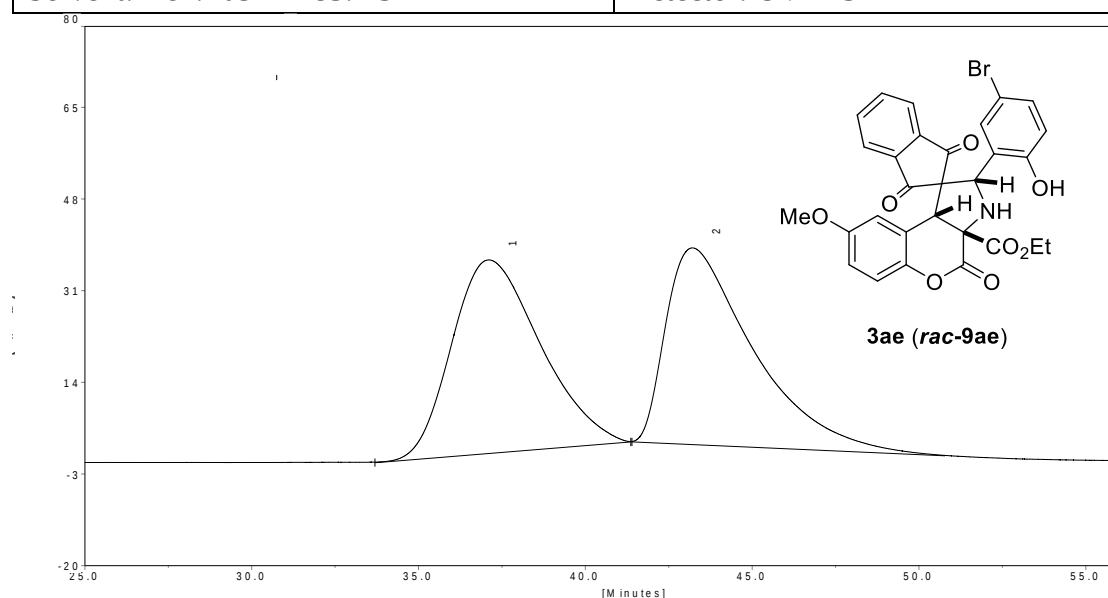
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
26.62	121.08	8525.40	50.3847
34.71	59.25	8395.20	49.6153



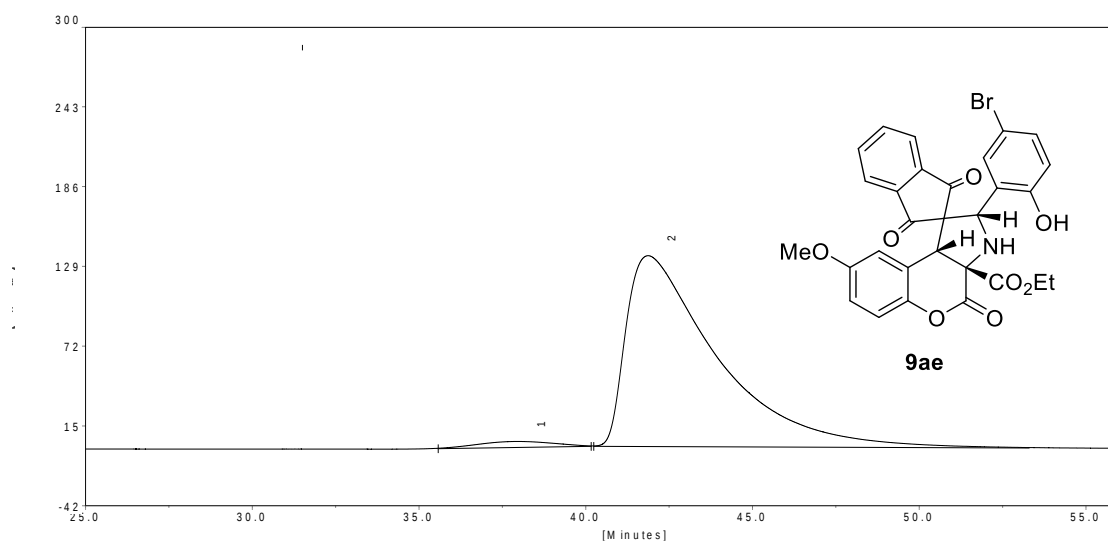
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
26.71	7.62	559.62	3.6857
35.00	89.02	14624.01	96.3143

HPLC chromatogram for 9ae

Column: CHIRALPAK IB	Flow rate: 1.0 ml/min
Solvent: Hex:EtOH = 85: 15	Detector: UV 245 nm



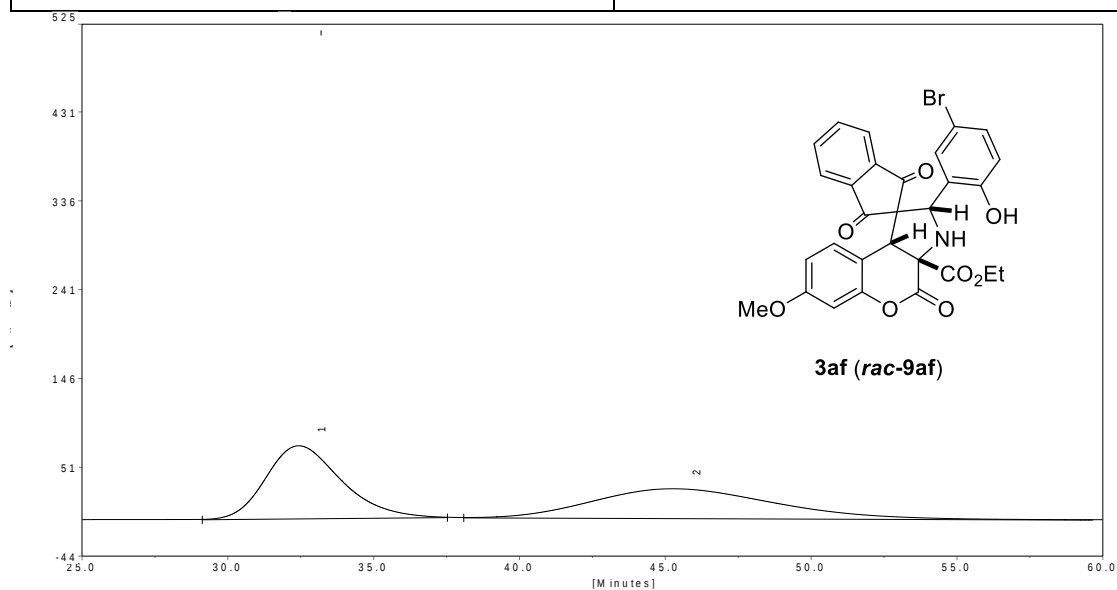
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
37.11	35.79	6578.72	49.8725
43.22	36.40	6612.34	50.1275



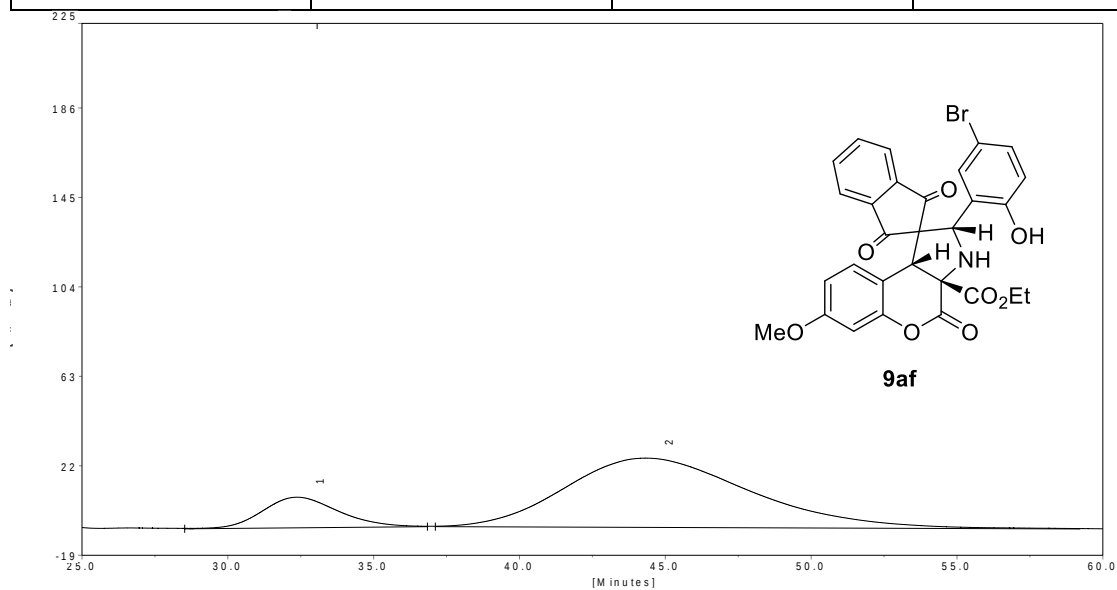
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
37.96	3.99	603.02	2.2956
41.87	136.02	25665.39	97.7044

HPLC chromatogram for **9af**

Column: CHIRALPAK IB	Flow rate: 0.7 ml/min
Solvent: Hex:IPA = 80: 20	Detector: UV 245 nm



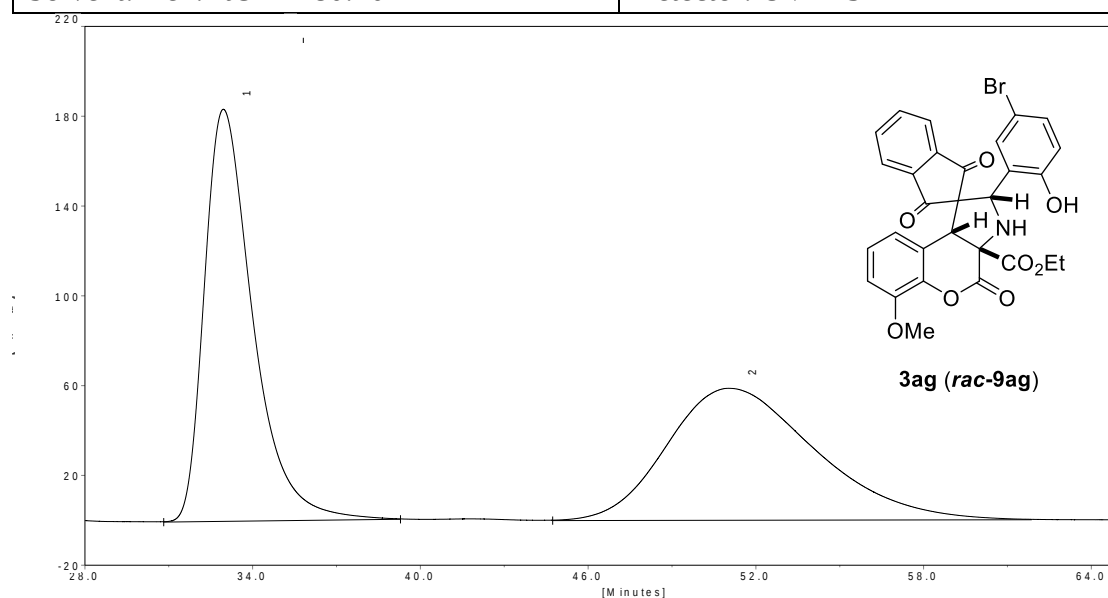
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
32.43	77.82	13921.01	50.8612
45.29	31.50	13449.61	49.1388



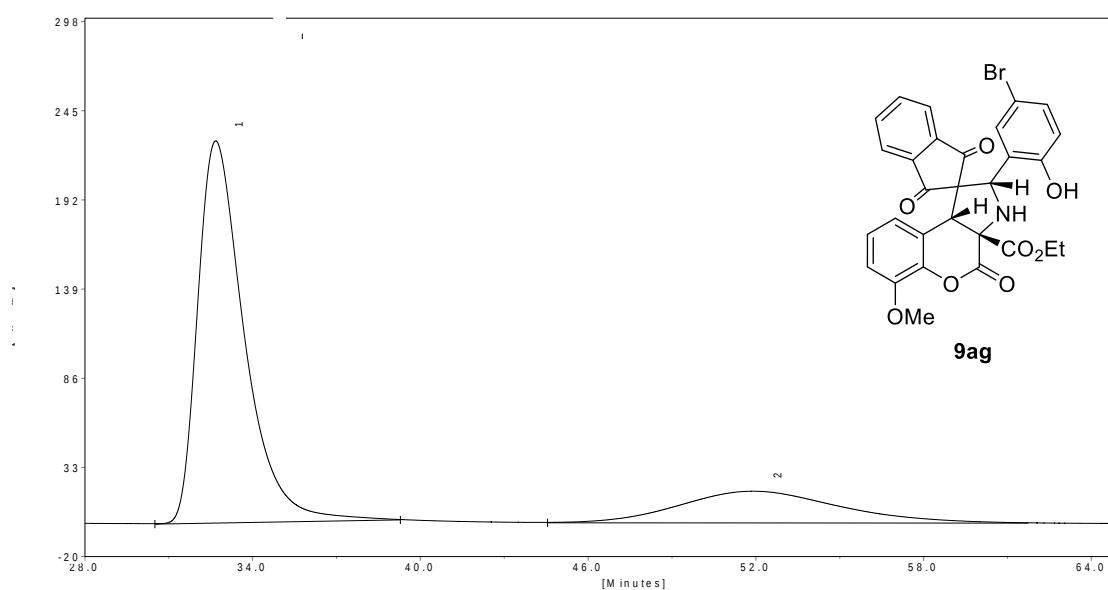
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
32.37	13.84	2445.19	15.0386
44.34	31.55	13814.23	84.9614

HPLC chromatogram for 9ag

Column: CHIRALPAK IA	Flow rate: 0.7 ml/min
Solvent: Hex:EtOH = 80:20	Detector: UV 245 nm



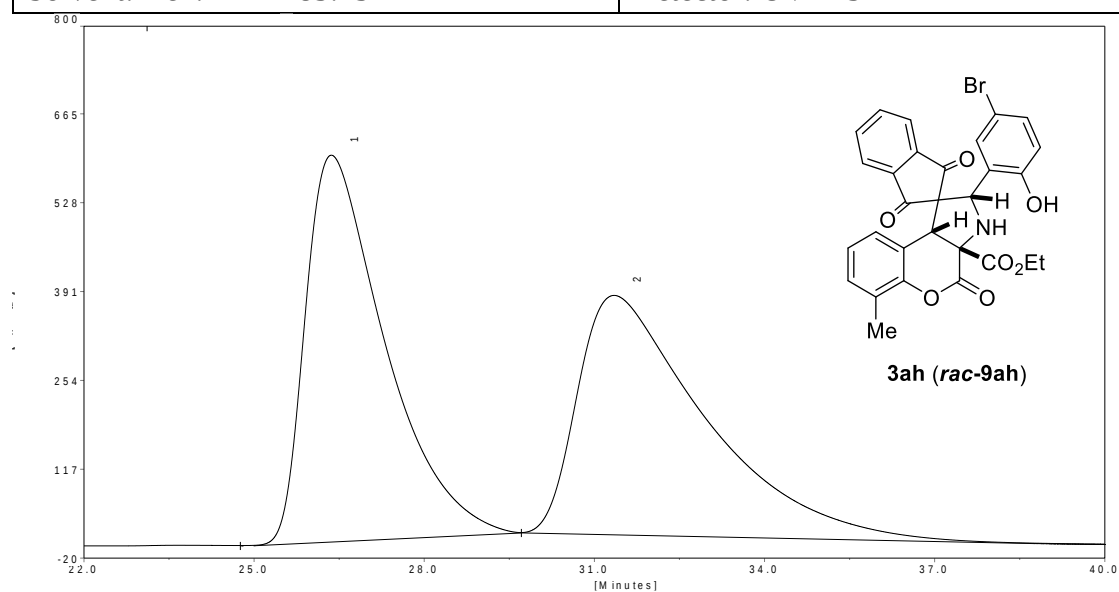
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
32.95	183.41	22037.57	50.5947
51.03	58.59	21519.52	49.4053



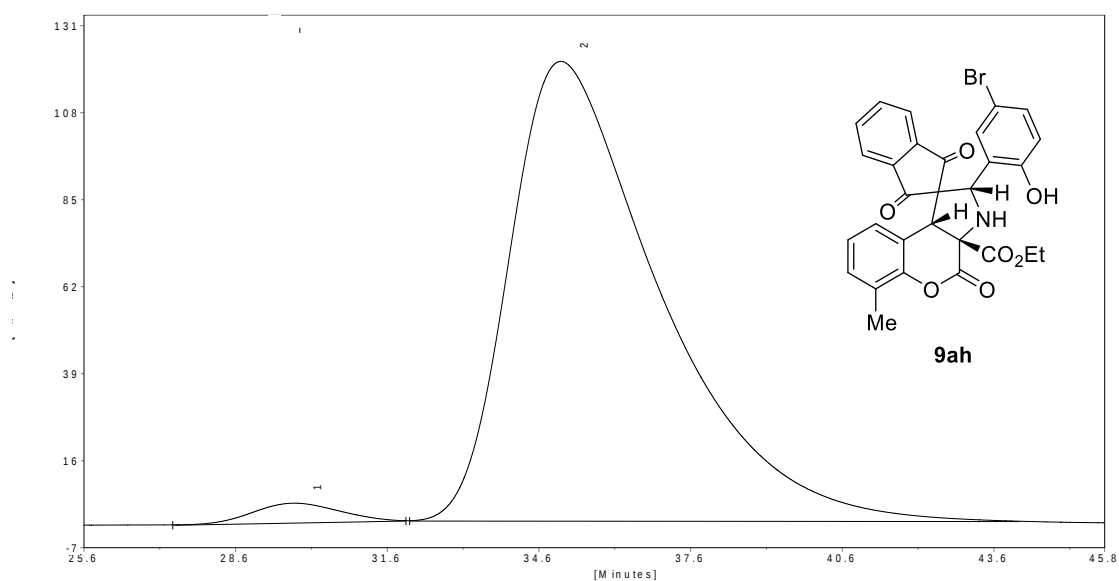
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
32.68	226.95	26215.93	78.2696
51.94	18.61	7278.45	21.7304

HPLC chromatogram for 9ah

Column: CHIRALPAK IB	Flow rate: 0.7 ml/min
Solvent: Hex: IPA = 85:15	Detector: UV 245 nm



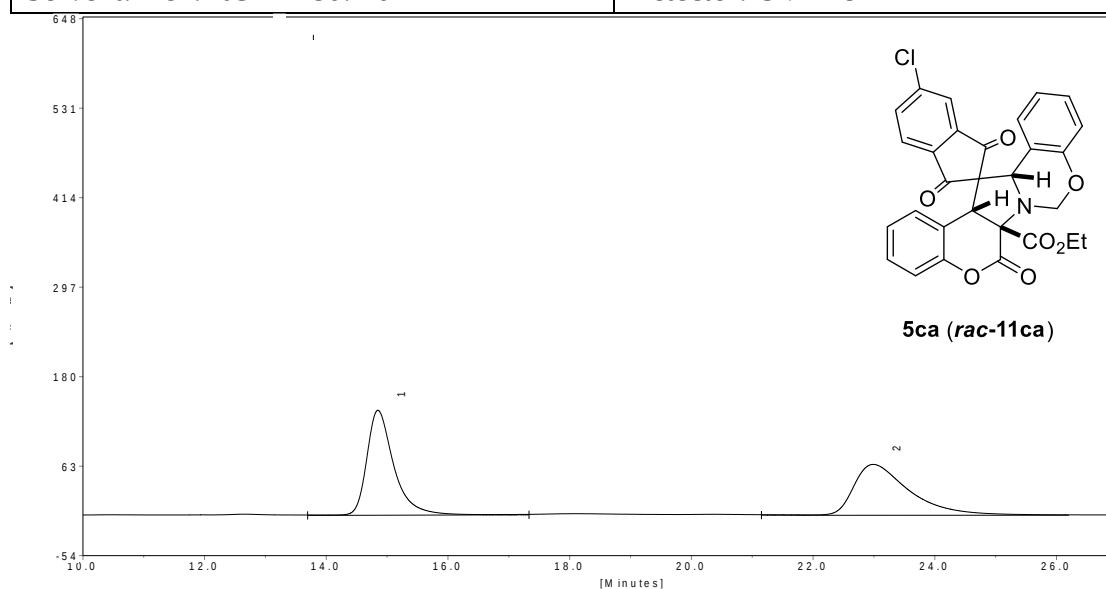
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
26.36	595.26	57840.16	50.2760
31.35	368.36	57205.00	49.7240



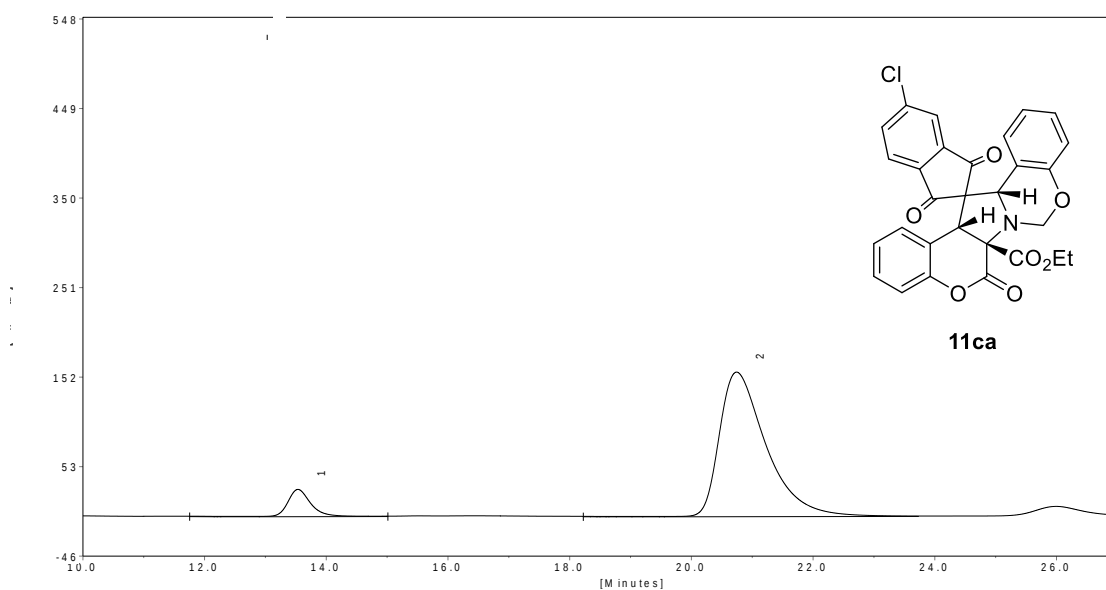
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
29.80	5.15	599.97	2.3274
35.08	121.47	25178.17	97.6726

HPLC chromatogram for **11ca**

Column: CHIRALPAK IA	Flow rate: 1.0 ml/min
Solvent: Hex:EtOH = 80: 20	Detector: UV 248 nm



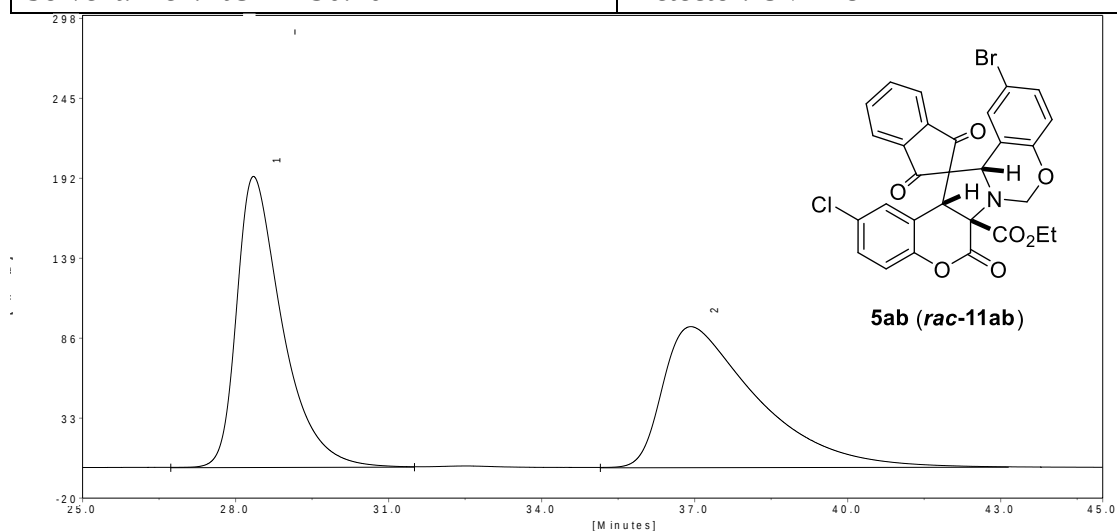
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
14.85	136.67	4132.89	50.3024
22.99	65.83	4083.19	49.6976



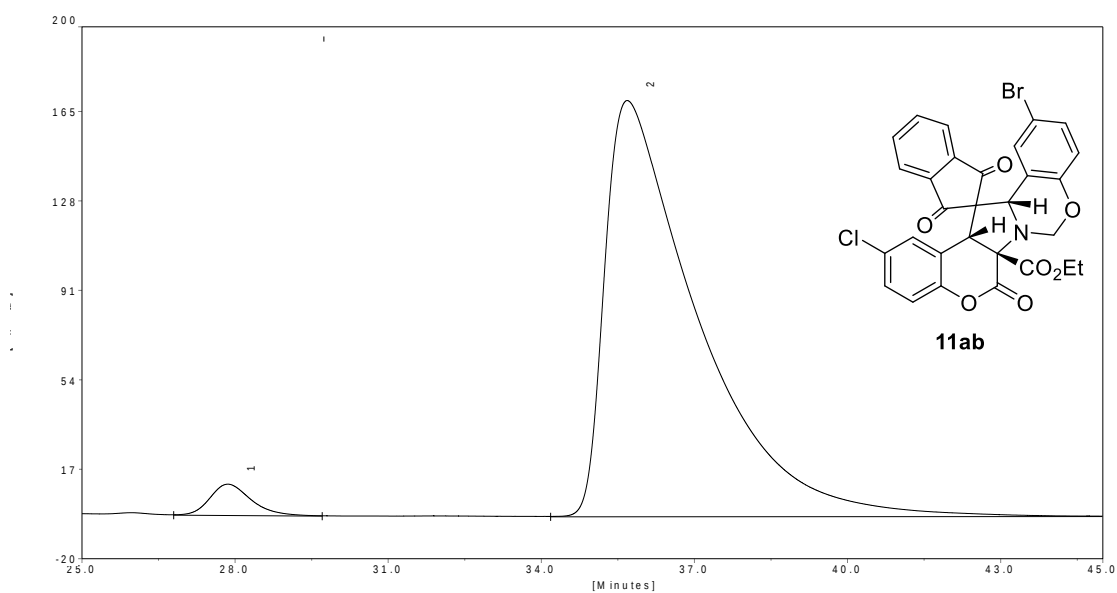
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
13.53	29.63	729.85	7.9769
20.74	159.38	8419.72	92.0231

HPLC chromatogram for **11ab**

Column: CHIRALPAK IA	Flow rate: 1.0 ml/min
Solvent: Hex:EtOH = 90:10	Detector: UV 248 nm



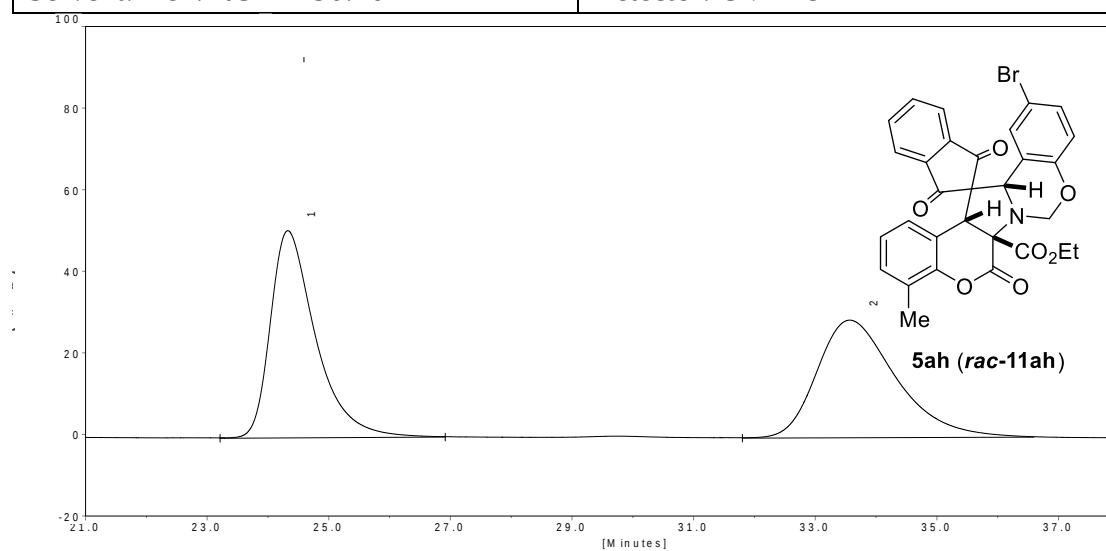
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
28.35	192.73	11925.96	49.8796
36.93	93.26	11983.53	50.1204



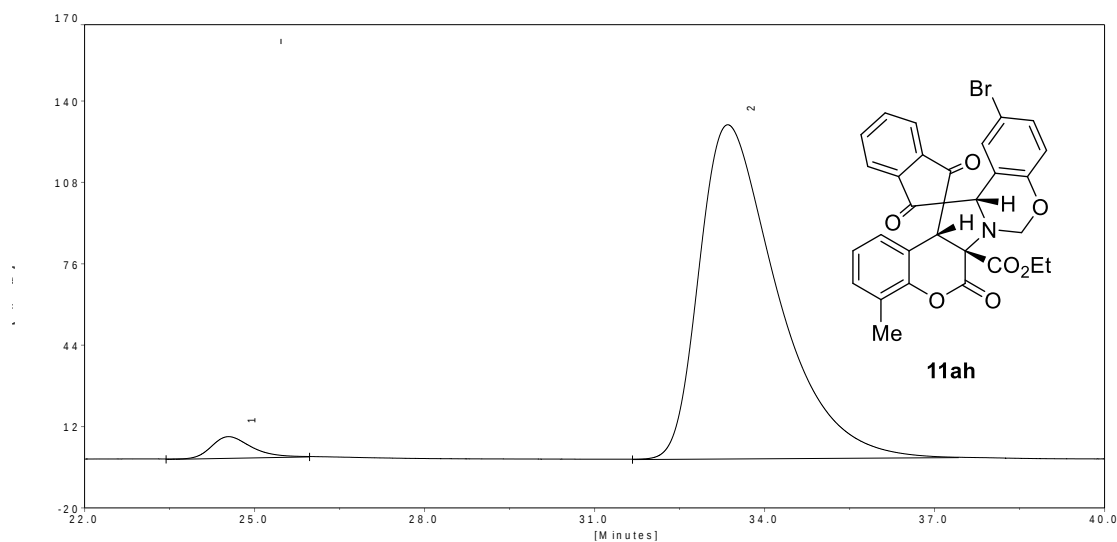
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
27.86	12.82	708.76	3.1473
35.69	171.99	21810.75	96.8527

HPLC chromatogram for **11ah**

Column: CHIRALPAK IA	Flow rate: 1 ml/min
Solvent: Hex:EtOH = 90:10	Detector: UV 248 nm



Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
24.33	50.70	2606.81	50.2428
33.57	28.74	2581.61	49.7572



Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
24.54	8.33	396.40	3.0742
33.35	131.27	12497.89	96.9258