Supporting Information

An Unprecedented Base-Promoted Domino Reaction of Methyleneindolinones and N-Tosyloxycarbamates for the Construction of Bispirooxindoles and Spiroaziridine Oxindoles

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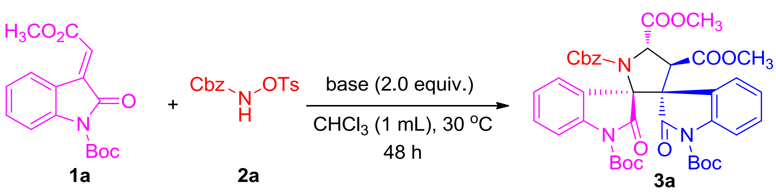
1. General methods

Commercial grade solvent was dried and purified by standard procedures as specified in Purification of Laboratory Chemicals, 4th Ed (Armarego, W. L. F.; Perrin, D. D. Butterworth Heinemann: 1997). NMR spectra were recorded with tetramethylsilane as the internal standard. ^{1}H NMR spectra were recorded at 300 MHz, and ^{13}C NMR spectra were recorded at 75 MHz (Bruker Avance). ^{1}H NMR chemical shifts (δ) are reported in ppm relative to tetramethylsilane (TMS) with the solvent signal as the internal standard ($CDCl_3$ at 7.26 ppm, $(CD_3)_2SO$ at 2.50 ppm). ^{13}C NMR chemical shifts are reported in ppm from tetramethylsilane (TMS) with the solvent resonance as the internal standard ($CDCl_3$ at 77.00 ppm, $(CD_3)_2SO$ at 39.52 ppm). Data are given as: s (singlet), d (doublet), t (triplet), q (quartet), dd (double of doublet), br (broad) or m (multiplets), coupling constants (Hz) and integration. Flash column chromatography was carried out using silica gel eluting with ethyl acetate and petroleum ether. Highresolution mass spectra were obtained with the Q-TOF-Premier mass spectrometer. Reactions were monitored by TLC and visualized with ultraviolet light. IR spectra were recorded on a ThermoFisherNicolet Avatar 360 FTIR spectrometer on a KBr beamsplitter.

2. Condition optimization for the synthesis of compound 3a

In order to develop an efficient approach for the synthesis of bispirooxindole **3a**, the easily available methyleneindolinone **1a** was chosen to react with N-tosyloxycarbamate **2a** for the condition optimization. We first screened a series of bases. As shown in Table S1, Na_2CO_3 was proved to be the best inorganic base (entry 2) and DABCO·$6H_2O$ was the best organic base (entry 6), respectively. After a combination of Na_2CO_3 and DABCO·$6H_2O$ (1:1), better result was obtained (entry 11) and bispirooxindole **3a** was obtained in 85% yield. Also the loading of DABCO·$6H_2O$ could be reduced to 0.2 equiv, giving **3a** in 85% yield (entry 12). Thus, the combined base (Na_2CO_3 and DABCO·$6H_2O$ (1:1)) was recommended for further optimization.

Table S1: The optimization of bases [a]

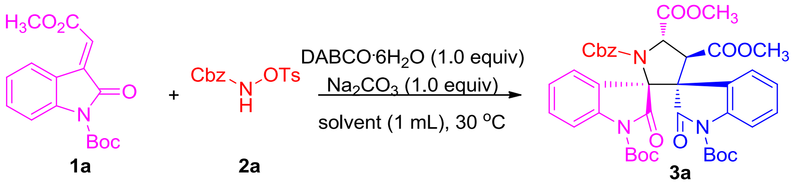


entry	base	time (h)	yield [b] (%)
1	$NaHCO_3$	48	41
2	Na_2CO_3	48	48
3	K_2CO_3	48	35
4	Cs_2CO_3	48	47
5	NaOAc	48	25
6	DABCO·$6H_2O$	48	67
7	NEt_3	48	49
8	pyridine	48	27
9	DMAP	48	47
10	DBU	48	14
11	DABCO·$6H_2O$ (1.0 eq) + Na_2CO_3 (1.0 eq)	48	85
12	DABCO·$6H_2O$ (0.2 eq) + Na_2CO_3 (1.0 eq)	48	83

[a] Unless otherwise specified, the reaction was conducted on a scale of 0.12 mmol with 1.0 equiv. of **2a** and 2.1 equiv. of **1a**. All reactions afforded **3a** as a single diastereomer. [b] Isolated yield.

Then, the reaction media was investigated and the typical results were shown in Table S2. The reaction proceeded smoothly in aprotic solvents such as halogenated hydrocarbons, ethers, aromatic solvents, and esters (entries 1-10). Also the reaction could take place in protic solvent, and **3a** could be generated in EtOH in 38% yield (entry 11). Among the solvents screened, THF turned out to be the best in terms of the yield of **3a** (entry 3).

Table S2: Screening of solvents [a]



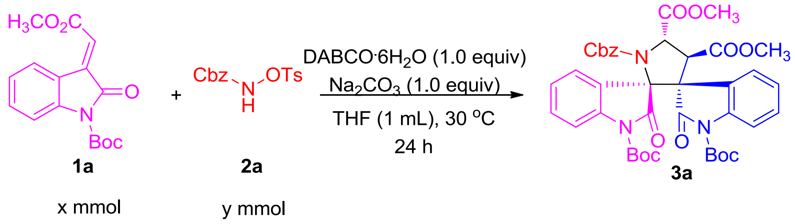
entry	solvent	time (h)	yield [b] (%)
1	$CHCl_3$	48	85
2	CH_2Cl_2	48	80
3	THF	24	85

4	MTBE	48	83
5	toluene	48	85
6	CH_3CN	8	67
7	EtOAc	29	71
8	DMC	29	60
9	DEC	29	66
10	*n*-Butyl acetate	29	67
11	EtOH	24	38

[a] Unless otherwise specified，the reaction was conducted with 0.12 mmol **2a**，2.1 equiv. **1a**, 2.0 equiv. base in 1 mL $CHCl_3$ at 30 °C. All reactions afforded **3a** as a single diastereomer. [b] Isolated yield. EA= ethyl acetate, DMC= dimethyl carbonate, DEC=diethyl carbonate.

In order to further optimize the reaction condition, we investigated the effect of the substrate ratios. The substrate ratios had an obvious influence on the reaction efficiency and the results were summarized in Table S3. When the molar ratio of **1a** to **2a** was varied from 2.1:1 to 3.5:1, the yield increased from 85% to 94% (entries 1-7). And no further increase occurred when the molar ratio of **1a** to **2a** was improved to 4.5:1 (entries 7 vs 9-10). By contrast, when excessive **2a** was used, the yields decreased sharply and product **3a** was obtained in only 39% yield when using 1.5 equiv of **2a** and the spirocyclic aziridine **4a** was the major product (entry 11). Thus, a 3.5:1 ratio of **1a** /**2a** was optimal. Notably, when 1.0 equiv of DABCO·$6H_2O$ was used instead of the combined base, the comparable result was obtained (entries 7 vs 8). Consequently, the following conditions were recommended: 0.12 mmol **2a** and 3.5 equiv of **1a** with 1.0 equiv of DABCO·$6H_2O$ in 1.0 mL THF at 30 °C.

Table S3: Screening of substrate ratios [a]



entry	x	y	x/y	Yield [b]
1	0.252	0.12	2.1:1	85
2	0.264	0.12	2.2:1	79

3	0.288	0.12	2.4:1	84
4	0.300	0.12	2.5:1	89
5	0.360	0.12	3.0:1	93
6	0.420	0.12	3.5:1	94
7 [c]	0.420	0.12	3.5:1	92
8	0.480	0.12	4.0:1	90
9	0.540	0.12	4.5:1	87
10	0.240	0.144	1:1.2	71
11	0.240	0.18	1:1.5	39

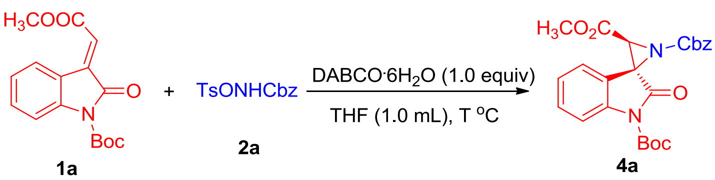
[a] Unless otherwise specified, the reaction was conducted on a 0.12 mmol scale. All reactions afforded **3a** as a single diastereomer. [b] Isolated yield. [c] Only 1.0 equiv of DABCO.6H_2O used instead of the combined base.

3. Condition optimization for the synthesis of compound 4a

During the screening of substrate ratios for the synthesis of bispirooxindole **3a**, we found that excess **2a** could result in the formation of spirocyclic aziridine **4a** which is a class of unique and versatile synthon. In order to develop a more general and robust approach for the synthesis of spirocyclic aziridines, we examined the condition parameters such as substrate ratios and reaction temperatures (Table S4). When using a 1:2.5 ratio of **1a**/**2a**, spirocyclic aziridine **4a** could be obtained in 60% yield (entry 1) and no further improvement took place by a 1:3 ratio of **1a**/**2a** (entry 2). Therefore, a 1:2.5 ratio of **1a**/**2a** was chosen for further investigation. Temperatures also affected the reaction. Lowering the reaction temperature resulted in lower yields and prolonged reaction time (entries 1 vs 3-4). By contrast, product **4a** could be obtained in 72% yield when the reaction was conducted at 50 ^{o}C (entry 5).

Based on the above screenings, the optimal condition was determined as the following: 0.15 mmol **1a** and 2.5 equiv of **2a** with 1.0 equiv of DABCO·6H_2O in 1.0 mL THF at 50 ^{o}C.

Table S4: Optimizations of the aziridination of methyleneindolinones [a].

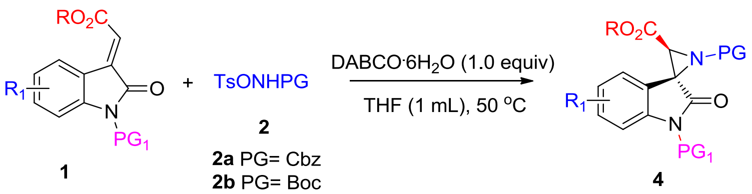


entry	**1a/2a**	T (^{o}C)	time	yield [b] (%)

1	1:2.5	30	45 min	60
2	1:3	30	45 min	56
3	1:2.5	-5	7 h	24
4	1:2.5	-30	12 h	21
5	1:2.5	50	10 min	72

[a] Unless otherwise specified, the reaction was conducted on a scale of 0.15 mmol. All cases afforded **4a** as a single diastereomer. [b] Isolated yield.

Table S5. Scope of substrates for synthesis of aziridines [a,b]



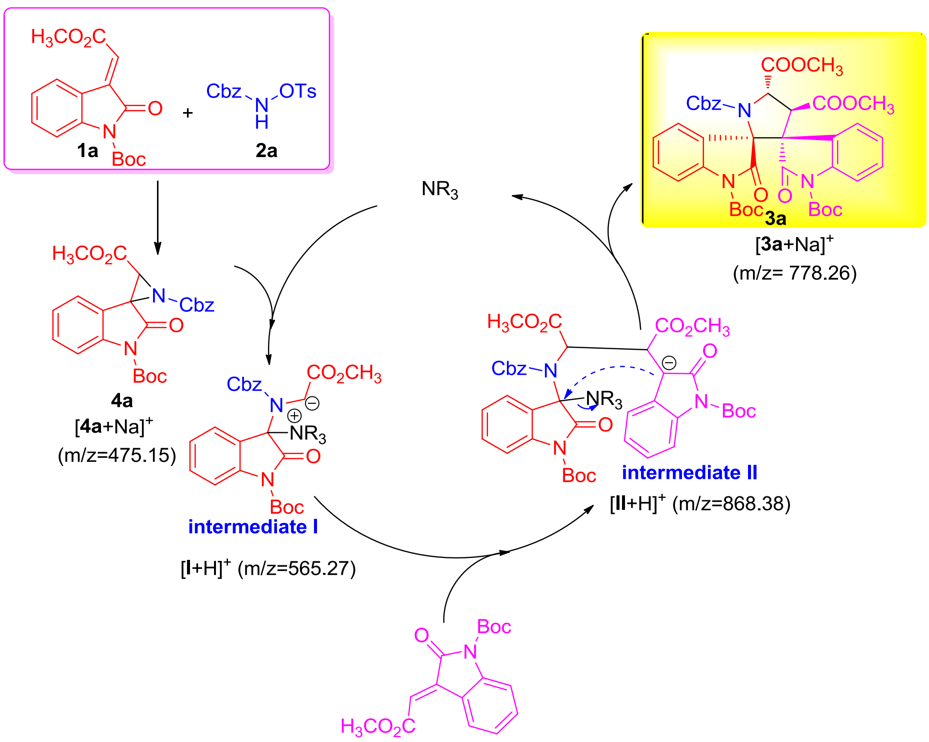
entry	**1**	R_1	PG_1	R	**2**	time	**4**/yield [b] (%)
1	**1a**	H	Boc	Me	**2a**	10 min	**4a**/72
2	**1a**	H	Boc	Me	**2b**	10 min	**4b**/55
3	**1b**	H	Boc	Et	**2a**	10 min	**4c**/77
4	**1b**	H	Boc	Et	**2b**	20 min	**4d**/58
5	**1c**	H	Boc	*t*-Bu	**2a**	3 h	**4e**/70
6	**1c**	H	Boc	*t*-Bu	**2b**	12 h	**4f**/59
7	**1d**	H	CO_2Et	Me	**2a**	5 min	**4g**/59
8	**1e**	H	Cbz	Me	**2a**	5 min	**4h**/72
9	**1g**	5-F	Boc	Me	**2a**	5 min	**4i**/81
10	**1g**	5-F	Boc	Me	**2b**	5 min	**4j**/91
11	**1h**	5-Cl	Boc	Me	**2a**	5 min	**4k**/80
12	**1h**	5-Cl	Boc	Me	**2b**	5 min	**4l**/79
13	**1i**	5-Br	Boc	Me	**2a**	5 min	**4m**/65
14	**1i**	5-Br	Boc	Me	**2b**	5 min	**4n**/63
15	**1j**	5-Me	Boc	Me	**2a**	10 min	**4o**/55
16	**1k**	7-Cl	Boc	Me	**2a**	5 min	**4p**/65

[a] Unless otherwise specified, the reaction was conducted on a scale of 0.15 mmol. [b] Isolated yield.

With the optimized condition in hand, the generality of this transformation was next investigated and the results were shown in Table S5. This domino reaction had a wide tolerance of methyleneindolinones and all the cases could afford the desired products (**4a-o**) in moderate to good yields (58-91%). Increasing the bulkiness of the ester group had minimal impact on the yields, albeit with longer reaction time (Table S5, entries 1-6). Then, the influence of the N-protecting groups, the positions or the electronic nature of the substitution patterns on the methyleneindolinones 1 was studied. And the results revealed that they had some effect on the yields (Table S5, entries 7-16), especially the electronic nature. Substrates with electron-donating substituents gave lower yields than those with electron-withdrawing ones (Table S5, entries 15 vs 9-14).

4. Mechanistic studies

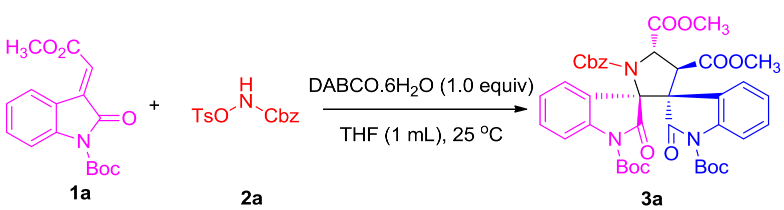
Based on the experimental data and the important roles of tertiary amines played in MBH reaction, a reaction mechanism has been proposed (Scheme S1). First, spirocyclic aziridine **4a** was obtained via a Michael/S_N2 sequence by reaction of compounds **1a** and **2a**. Then, ring-opening reaction of aziridine **4a** occurred after the attack by DABCO·$6H_2O$, and the corresponding zwitterionic intermediate **I** was obtained. Subsequently, intermediate **I** reacted with methyleneindolinone **1a** to form zwitterionic intermediate **II**. Finally, the annulation product **3a** was obtained through an intramolecular nucleophilic substitution and the tertiary amine was released.



Scheme S1: A plausible catalytic cycle for this domino reaction

To get support for the proposed catalytic cycle, both ESI-HRMS analysis and control experiments were conducted, and the results were discussed below.

First, the formation of the spirocyclic aziridine **4a** and zwitterionic intermediates **I** and **II** was supported by ESI-HRMS. Three typical spectrums obtained after blending **1a**, **2a** and DABCO·6H_2O for 2 minutes were shown in Scheme S2 and Figure S1. A characteristic signal at m/z 475.15 was observed which was consistent with spirocyclic aziridine **4a**, and [**I**+H]$^+$ at 565.27 as well as [**II**+H]$^+$ at 565.27 (A, Figure S1). However, after stirred for 25 hours, the signal at m/z 475.15 and 629.21 disappeared as expected and the signal of product **3a** at m/z 778.26 became stronger (B, Figure S1). These observations indicated that the reaction proceeded in a stepwise mechanism.



Scheme S2: The reaction for HRMS analysis

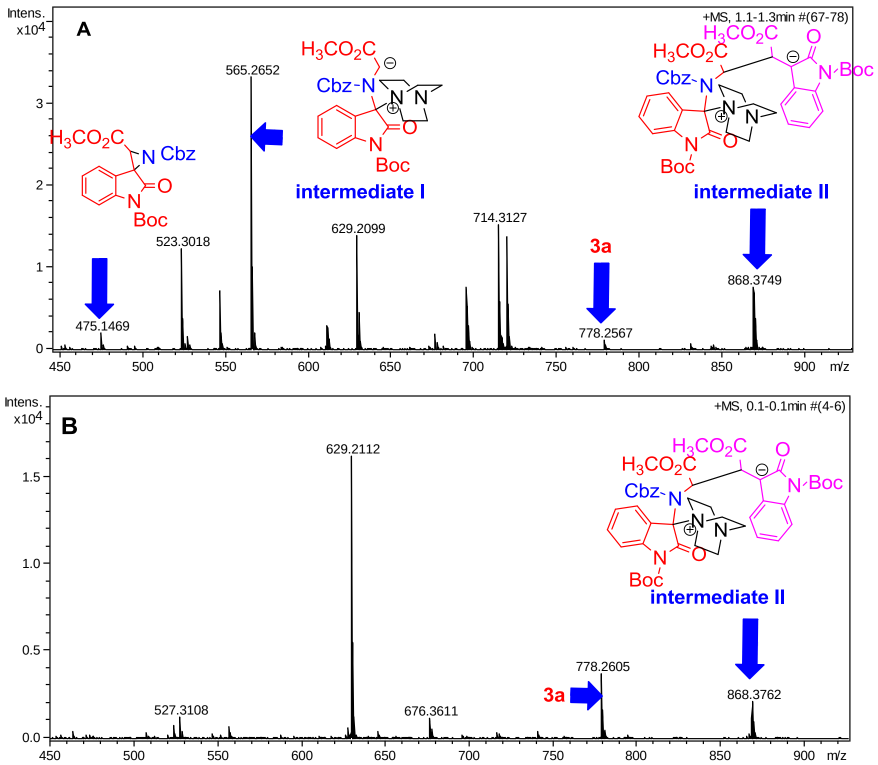


Figure S1: A) ESI-HRMS spectrum of the reaction of **1a**, **2a** and DABCO·6H_2O in 1.0 mL of THF after 2 minutes; B) ESI-HRMS spectrum of the reaction of **1a**, **2a** and DABCO·6H_2O in 1.0 mL of THF after 25 hours.

In addition, the intermediates **I** and **II** could also be detected by mixing **4a**, **1a** and DABCO·6H_2O (Figure S2). It is noteworthy that the tertiary amine played an important role in the reaction (Scheme S3). When the mixture of **4a** (0.1 mmol), **1a** (0.25 mmol) and DABCO·6H_2O (0.1 mmol) was stirred at 25 ^{o}C for 48 h, product **3a** was obtained in 97% yield. However, no reaction occurred without DABCO·6H_2O. Moreover, the reaction could be promoted by a catalytic loading of DABCO·6H_2O and **3a** was generated in 94% yield.

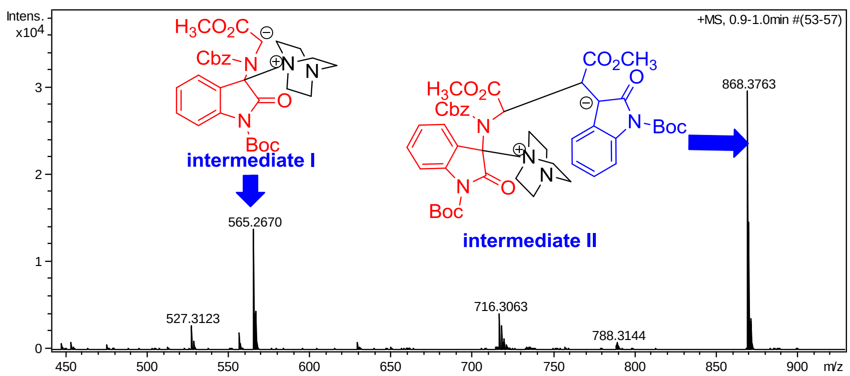
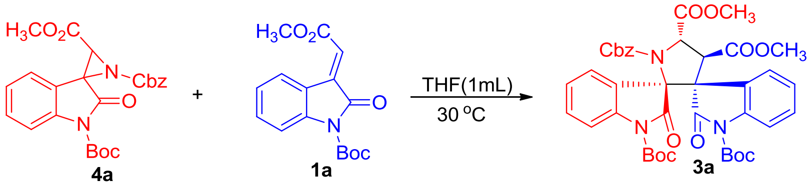
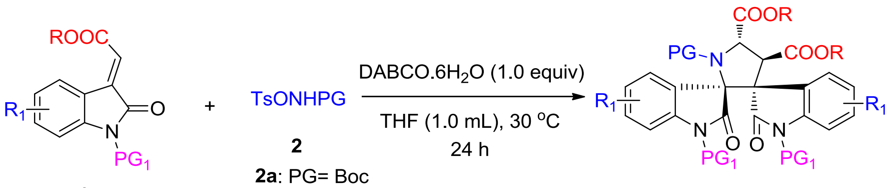


Figure S2: ESI-HRMS spectrum of the reaction of **1a**, **4a** and DABCO·6H_2O in 1.0 mL of THF after 2.5 h

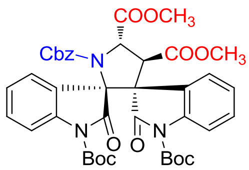


Scheme S3: Examination of the role played by the tertiary amine

5. Experimental data for bispirooxindoles 3

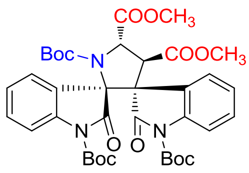


General procedure: To a 5.0 mL vial were successively added methyleneindolinones **1** (0.42 mmol), N-tosyloxycarbamates **2** (0.12 mmol), DABCO·6H$_2$O (26.4 mg, 0.12 mmol) and 1.0 mL THF. The resulting mixture was stirred for 24-48 h at 30 °C till almost full consumption of **2** by TLC analysis, and then the reaction mixture was directly subjected to flash column chromatography on silica gel (petroleum ether/ ethyl acetate) to afford the corresponding products **3**.



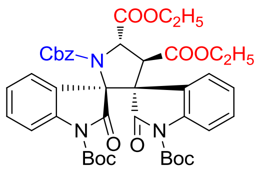
3a

Product **3a** was obtained in 92% yield as a white solid. ^{1}H NMR (300 MHz, CDCl$_3$), δ 7.83 (d, J = 5.4 Hz, 1H), 7.74 (d, J = 7.8 Hz, 1H), 7.55 (s, 1H), 7.31-7.10 (m, 9H), 6.68 (s, 1H), 5.48 (d, J = 5.8 Hz, 1H), 5.10-4.86 (m, 2H), 4.25 (d, J = 8.0 Hz, 1H), 3.75 (d, J = 104.4 Hz, 3H), 3.41 (s, 3H), 1.44 (s, 9H), 1.38 (s, 9H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 171.2, 170.5, 167.4, 152.6, 148.2, 139.1, 129.9, 128.3, 126.7, 126.2, 124.3, 123.9, 122.3, 114.5, 84.4, 83.7, 68.3, 62.0, 52.8, 52.4, 28.0, 27.7. IR (KBr) v 3435.9, 2980.9, 2954.3, 2918.6, 1805.7, 1779.4, 1731.5, 1608.1, 1481.0, 1468.8, 1346.4, 1294.3, 1250.6, 1153.6, 1097.8, 761.1 cm^{-1}. HRMS (ESI) Calcd. for C40H41N3NaO12 [M+Na]$^+$: 778.2582, Found: 778.2553.



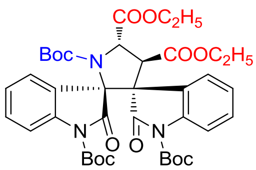
3b

Product **3b** was obtained in 96%[+] yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.92 (s, 1H), 7.76 (d, *J* = 8.3 Hz, 1H), 7.69 (s, 1H), 7.30 (t, *J* = 6.5 Hz, 3H), 7.23-7.16 (m, 2H), 5.37 (d, *J* = 8.8 Hz, 1H), 4.27 (d, *J* = 8.8 Hz, 1H), 3.92 (s, 3H), 3.38 (s, 3H), 1.44 (s, 9H), 1.40 (s, 11H), 1.04 (s, 7H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.6, 170.2, 167.4, 151.6, 148.4, 148.2, 139.0, 138.1, 129.7, 129.6, 129.4, 126.6, 126.2, 124.2, 123.8, 122.8, 114.5, 114.3, 114.0, 84.2, 83.7, 82.5, 61.6, 52.9, 52.3, 52.2, 52.1, 51.8, 28.0, 27.9, 27.8. IR (KBr) *v* 3434.6, 2978.1, 2917.9, 1779.2, 1752.9, 1731.0, 1714.8, 1370.1, 1349.7, 1294.0, 1249.2, 1152.4, 1099.3, 765.8. HRMS (ESI) Calcd. for C37H43N3NaO12 $[M+Na]^+$: 744.2739, Found: 744.2719.



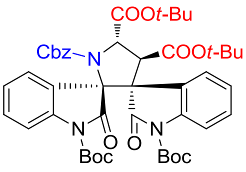
3c

Product **3c** was obtained in 91% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.94 (s, 1H), 7.68 (dd, J_1= 8.0 Hz, J_2= 7.4 Hz, 2H), 7.41-7.00 (m, 9H), 6.67 (s, 1H), 5.39 (d, *J*= 9.0 Hz, 1H), 5.07-4.80 (m, 2H), 4.38 (brs, 1H), 4.29 (d, *J*= 9.0 Hz, 1H), 4.10 (brs, 1H), 3.96-3.85 (m, 1H), 3.82-3.76 (m,1H), 1.46-1.12 (m, 21H), 0.74 (t, *J*= 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 169.8, 166.6, 152.4, 148.3, 148.2, 139.1, 137.9, 135.1, 130.1, 129.7, 128.2, 127.2, 126.8, 126.3, 124.2, 123.8, 122.8, 114.4, 84.2, 83.6, 68.1, 62.0, 61.5, 52.5, 51.7, 28.0, 27.9, 13.9, 13.2. IR (KBr) *v* 3439.2, 2981.5, 2920.0, 1779.4, 1732.1, 1720.9, 1346.6, 1293.6, 1249.4, 1153.4, 765.0, 752.9. HRMS (ESI) Calcd. for C42H45N3NaO12 $[M+Na]^+$: 806.2895, Found: 806.2897.



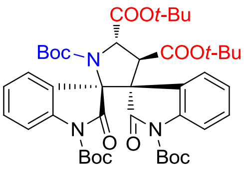
3d

Product **3d** was obtained in 98% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 8.03 (d, *J*= 6.2 Hz, 1H), 7.73 (dd, J_1= 8.0 Hz, J_2= 7.4 Hz, 2H), 7.38-7.13 (m, 5H), 5.31 (d, *J*= 9.3 Hz, 1H), 4.42-4.35 (m, 2H), 4.29 (d, *J*= 6.2 Hz, 1H), 3.94-3.89 (m, 1H), 3.80-3.74 (m, 1H), 1.44 (s, 9H), 1.39 (s, 15H), 1.03 (s, 6H), 0.72 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.0, 169.8, 166.6, 151.5, 148.4, 148.4, 139.0, 138.0, 129.6, 129.5, 126.8, 126.5, 124.1, 123.8, 123.2, 114.3, 114.0, 84.1, 83.6, 82.4, 61.9, 61.6, 61.4, 53.3, 52.0, 28.0, 27.7, 27.5, 14.0, 13.2. IR (KBr) *v* 3441.4, 2981.1, 2933.1, 1781.7, 1735.3, 1713.4, 1369.1, 1349.3, 1294.4, 1249.4, 754.5. HRMS (ESI) Calcd. for C39H47N3NaO12 $[M+Na]^+$: 872.3052, Found: 772.3039.



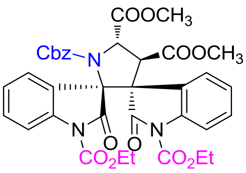
3e

Product **3e** was obtained in 80% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 8.04 (dd, J_1= J_2= 7.2 Hz, 1H), 7.82-.63 (m, 2H), 7.42 (d, *J*= 7.5 Hz, 1H), 7.33-7.05 (m, 8H), 6.67 (d, *J*= 6.7 Hz, 1H), 5.20 (d, *J*= 7.9 Hz, 1H), 5.18-4.23 (m, 2H), 4.21 (d, *J*= 9.6 Hz, 1H), 1.54-1.25 (m, 27H), 0.96 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.9, 169.6, 169.3, 165.1, 152.3, 148.6, 138.9, 138.1, 129.7, 129.6, 128.4, 128.1, 127.6, 127.1, 126.7, 126.5, 126.2, 124.1, 123.8, 123.5, 114.6, 114.3, 84.0, 83.4, 82.7, 68.2, 67.8, 62.5, 62.4, 61.6, 53.7, 52.6, 27.9, 27.7, 27.0, 26.7. IR (KBr) *v* 3438.2, 2980.1, 2930.2, 1781.2, 1732.6, 1719.7, 1369.1, 1352.4, 1295.4, 1250.1, 1153.8, 1099.0, 844.2, 751.9. HRMS (ESI) Calcd. for C46H53N3NaO12 $[M+Na]^+$: 862.3521, Found: 862.3498.



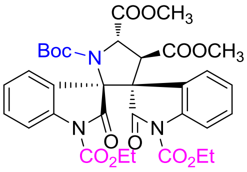
3f

Product **3f** was obtained in 74% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 8.04 (dd, J_1= 7.3 Hz, J_2= 6.9 Hz, 1H), 7.80-1.68 (m, 2H), 7.42 (t, *J*= 7.6 Hz, 1H), 7.33-7.14 (m, 4H), 5.12 (t, *J*= 13.6 Hz, 1H), 4.17 (dd, J_1= 10.2 Hz, J_2= 9.8 Hz, 1H), 1.56 (s, 9H), 1.38 (t, *J*= 10.7 Hz, 21H), 1.04 (s, 6H), 0.96 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.3, 169.7, 169.4, 165.2, 151.4, 148.6, 138.9, 137.7, 129.5, 129.4, 127.1, 126.8, 126.2, 125.9, 123.9, 123.8, 114.7, 114.1, 113.9, 84.0, 83.6, 82.6, 82.5, 62.3, 62.1, 61.7, 53.4, 52.9, 28.2, 27.9, 27.7, 27.5, 27.1. IR (KBr) *v* 3439.7, 2979.3, 2929.9, 1780.3, 1735.1, 1715.3, 1480.1, 1369.6, 1351.5, 1295.3, 1250.5, 1154.0, 1099.1, 844.7, 751.7. HRMS (ESI) Calcd. for C43H55N3NaO12 $[M+Na]^+$: 828.3678, Found: 828.3660.



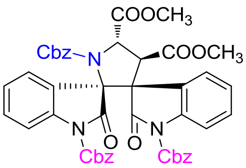
3g

Product **3g** was obtained in 94% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.86 (d, *J*= 6.4 Hz, 1H), 7.68 (dd, J_1= 8.2 Hz, J_2= 26.2 Hz, 2H), 7.43-7.10 (m, 9H), 6.68 (d, *J*= 6.3 Hz, 1H), 5.49 (d, *J*= 6.7 Hz, 1H), 5.09-4.83 (m, 2H), 4.35-4.13 (m, 5H), 3.95-3.61 (m, 3H), 3.43 (s, 3H), 1.36-1.21 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.1, 167.3, 152.7, 149.8, 138.9, 130.2, 128.2, 127.7, 127.0, 126.5, 124.6, 124.1, 121.7, 114.7, 68.4, 63.5, 63.0, 61.7, 58.4, 52.9, 52.5, 18.4, 14.1. IR (KBr) *v* 3440.5, 2955.3, 2922.8, 2851.2, 1778.6, 1737.7, 1371.3, 1342.7, 1289.2, 1236.6, 1174.4, 756.4, 699.0. HRMS (ESI) Calcd. for C36H33N3NaO12 $[M+Na]^+$: 722.1956, Found: 722.1935.



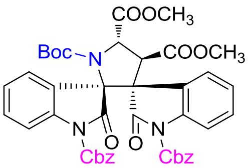
3h

Product **3h** was obtained in 98% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.96 (s, 1H), 7.73 (dd, J_1= 8.1 Hz, J_2= 8.0 Hz, 2H), 7.33-7.14 (m, 5H), 5.36 (d, *J*= 8.6 Hz, 1H), 4.33-4.21 (m, 5H), 3.91 (s, 3H), 3.37 (s, 3H), 1.29-1.20 (m, 9H), 1.01 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.4, 170.1, 167.2, 151.5, 150.0, 149.9, 138.6, 137.9, 130.0, 129.9, 126.8, 126.5, 125.8, 124.5, 124.1, 122.2, 115.1, 114.6, 114.3, 82.7, 63.4, 63.3, 63.0, 61.3, 58.3, 52.9, 52.4, 52.3, 27.4, 18.3, 14.0. IR (KBr) *v* 3445.9, 2979.3, 2924.1, 1805.9, 1781.6, 1737.4, 1708.9, 1370.5, 1345.4, 1291.4, 1237.8, 1170.9, 767.3. HRMS (ESI) Calcd. for C33H35N3NaO12 $[M+Na]^+$: 688.2113, Found: 688.2093.



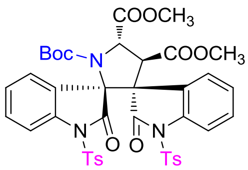
3i

Product **3i** was obtained in 93% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.84 (s, 1H), 7.68 (d, *J*= 8.1 Hz, 1H), 7.36-6.98 (m, 20 H), 6.69-6.63 (m, 1H), 5.50 (d, *J*= 9.1 Hz, 1H), 5.53-4.79 (m, 6H), 4.26 (d, *J*= 7.8 Hz, 1H), 3.98-3.59 (m, 3H), 3.40 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.1, 167.2, 152.6, 149.7, 138.6, 134.8, 134.7, 130.3, 128.7, 128.5, 128.4, 128.3, 128.2, 128.0, 127.8, 127.7, 126.9, 126.4, 124.7, 124.2, 121.6, 114.7, 68.5, 68.4, 68.2, 68.1, 61.8, 52.9, 52.7, 52.6, 52.5. IR (KBr) *v* 3436.8, 3033.6, 2954.2, 1779.1, 1741.0, 1382.0, 1343.6, 1289.2, 1225.6, 1171.4, 754.4, 697.8 cm^{-1}. HRMS (ESI) Calcd. for C46H37N3NaO12 $[M+Na]^+$: 846.2269, Found: 846.2256.



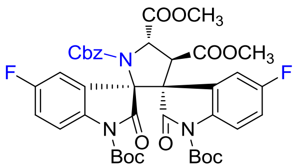
3j

Product **3j** was obtained in 97% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.96 (s, 1H), 7.69 (t, *J*= 8.9 Hz, 2H), 7.35-7.07 (m, 15H), 5.39 (d, *J*= 8.6 Hz, 1H), 5.28 (t, *J*= 5.6 Hz, 2H), 5.20 (t, *J*= 7.2 Hz, 2H), 4.29 (d, *J*= 8.6 Hz, 1H), 3.92 (s, 3H), 3.34 (s, 3H), 1.29 (s, 2H), 0.98 (s, 7H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.5, 170.1, 167.2, 151.5, 149.9, 149.7 138.4, 137.7, 134.8, 134.7, 130.1, 129.9, 128.5, 128.4, 128.3, 128.2, 128.1, 127.7, 127.6, 126.9, 126.5, 124.5, 124.2, 122.2, 114.6, 114.3, 82.7, 68.4, 68.1, 61.4, 58.3, 53.4, 52.9, 52.6, 52.5, 52.4, 27.8 (d, *J*= 50.4 Hz, 1C). IR (KBr) *v* 3447.9, 2954.7, 2926.0, 1780.4, 1741.1, 1713.2, 1482.2, 1466.8, 1379.5, 1345.5, 1290.0, 1224.7, 1169.5, 1150.3, 1099.7, 754.3.697.0. HRMS (ESI) Calcd. for C43H39N3NaO12 $[M+Na]^+$: 812.2426, Found: 812.2424.



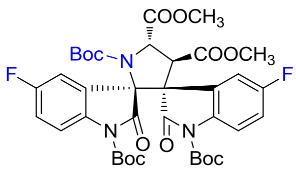
3k

Product **3k** was obtained in 93% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 8.03 (d, *J*= 7.4 Hz, 1H), 7.83-7.70 (m, 5H), 7.52 (d, *J*= 8.1 Hz, 1H), 7.36 (t, *J*= 7.7 Hz, 1H), 7.24-7.08 (m, 8H), 5.09 (d, *J*= 10.1 Hz, 1H), 4.13 (d, *J*= 10.2 Hz, 1H), 3.84 (s, 3H), 2.85 (d, *J*= 95.8 Hz, 3H), 2.46 (s, 3H), 2.38 (s, 3H), 1.23 (s, 3H), 0.95 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.6, 169.2, 169.1, 166.3, 145.7, 145.5, 137.7, 137.6, 134.5, 130.4, 129.9, 129.6, 129.3, 128.3, 128.2, 127.3, 127.1, 124.3, 124.1, 113.1, 112.8, 83.4, 72.4, 60.5, 53.2, 53.1, 51.8, 27.8, 22.7, 21.7. IR (KBr) *v* 3446.6, 2956.3, 2924.0, 1764.3, 1712.9, 1603.2, 1464.1, 1377.9, 1235.8, 1191.0, 1088.6, 812.7, 753.4, 688.9, 660.4 cm^{-1}. HRMS (ESI) Calcd. for C41H39N3NaO12S2 $[M+Na]^+$: 852.1867, Found: 852.1829.



3l

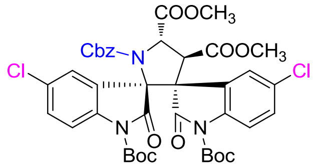
Product **3l** was obtained in 96% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.73 (s, 2H), 7.50 (dd, J_1= 2.6 Hz, J_2= 8.0 Hz, 1H), 7.33 (t, *J*= 4.8 Hz, 2H), 7.21-6.95 (m, 5H), 6.76 (s, 1H), 5.48 (d, *J*= 7.3 Hz, 1H), 5.17-4.81 (m, 2H), 4.12 (d, *J*= 6.7 Hz, 1H), 3.79 (d, *J*= 96.2 Hz, 3H), 3.54 (s, 3H), 1.25 (s, 18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.9, 170.7, 168.9, 167.3, 159.6 (d, *J*= 242.8 Hz, 1C), 159.0 (d, *J*= 242.7 Hz, 1C), 152.6, 148.1 (d, *J*= 11.6 Hz, 1C), 135.4, 134.6, 128.5, 128.4, 128.3, 128.0, 127.8, 123.0 (d, *J*= 8.3 Hz, 1C), 117.2, 117.0, 116.7, 116.4, 115.9, 115.8, 114.6 (d, *J*= 25.8 Hz, 1C), 113.8 (d, *J*= 25.8 Hz, 1C), 85.0, 84.3, 68.5, 52.9, 27.7. IR (KBr) *v* 3442.2, 2957.1, 2924.1, 2852.9, 1777.6, 1736.0, 1486.0, 1345.7, 1300.3, 1266.6, 1151.6, 1102.8, 820.6, 765.7, 698.3 cm^{-1}. HRMS (ESI) Calcd. for C40H39F2N3NaO12 $[M+Na]^+$: 814.2394, Found: 814.2389.



3m

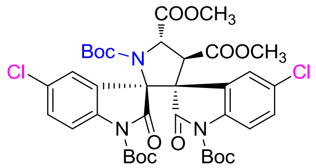
Product **3m** was obtained in 98% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.77-7.61 (m, 3H), 6.99 (q, *J*= 8.3 Hz, 3H), 5.37 (d, *J*= 6.5 Hz, 1H), 4.15 (d, *J*= 7.7 Hz, 1H), 3.92 (s, 3H), 3.48 (s, 3H), 1.46 (s, 9H), 1.43 (s, 9H), 1.24 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.3, 170.3, 167.3, 159.6 (d, *J*= 242.6 Hz, 1C), 159.0 (d, *J*= 242.7 Hz, 1C), 151.5, 148.2, 148.1, 135.2 (d, *J*= 2.4 Hz, 1C), 134.3 (d, *J*= 2.4 Hz, 1C), 116.8, 116.5, 116.2, 115.7 (d, *J*= 7.8 Hz, 1C), 115.4, 114.7, 114.3, 114.0, 113.7, 84.8, 84.4, 83.0, 61.7, 58.3, 53.0, 52.8, 51.6, 27.7.

IR (KBr) *v* 3441.1, 2981.2, 2923.9, 2851.5, 1777.4, 1737.6, 1486.1, 1371.0, 1347.7, 1300.4, 1269.0, 1251.0, 1152.9, 821.9 cm^{-1}. HRMS (ESI) Calcd. for C37H41F2N3NaO12 $[M+Na]^+$: 780.2551, Found: 780.2562.



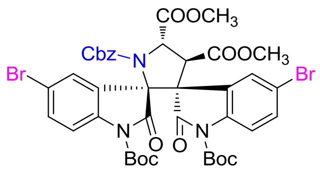
3n

Product **3n** was obtained in 92% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.74-7.70 (m, 3H), 7.35-7.13 (m, 7H), 6.77 (d, *J*= 7.0 Hz, 1H), 5.46 (d, *J*= 7.4 Hz, 1H), 5.15-4.82 (m,2H), 4.14 (d, *J*= 6.9 Hz, 1H), 3.95-3.61 (m, 3H), 3.53 (s, 3H), 1.48 (s, 9H), 1.43 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 170.3, 167.2, 152.6, 147.9, 137.8, 137.1, 130.6, 130.3, 130.2, 130.0, 129.5, 128.5, 128.4, 128.3, 127.8, 127.0, 126.3, 125.4, 123.1, 115.9, 115.8, 85.1, 84.9, 84.5, 68.6, 68.4, 52.9, 52.8, 27.8. IR (KBr) *v* 3440.7, 2982.2, 2924.1, 2851.8, 1780.8, 1756.2, 1730.4, 1476.1, 1336.9, 1295.3, 1250.1, 1155.2, 1111.5, 823.1, 700.2 cm^{-1}. HRMS (ESI) Calcd. for C40H39Cl2N3NaO12 $[M+Na]^+$: 846.1803, Found: 846.1804.



3o

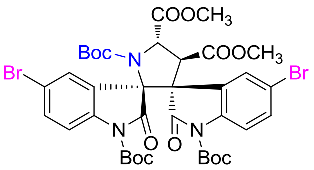
Product **3o** was obtained in 97% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.82 (s, 1H), 7.70 (d, *J*= 8.9 Hz, 1H), 7.66-7.64 (m, 1H), 7.29-7.22 (m, 3H), 5.37 (t, *J*= 2.6 Hz, 1H), 4.15 (d, *J*= 7.7 Hz, 1H), 3.92 (s, 3H), 3.48 (s, 3H), 1.47 (s, 9H), 1.44 (s, 9H), 1.24 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.1, 170.1,167.2, 151.6, 148.2, 148.0, 137.8, 136.9, 130.5, 130.0, 129.9, 129.8, 129.5, 126.3, 125.5, 123.6, 115.7, 85.0, 84.8, 83.0, 61.7, 59.8, 58.4, 53.0, 52.8, 52.6, 51.8, 48.9, 27.8. IR (KBr) *v* 3440.2, 2982.0, 2927.7, 2852.6, 1784.8, 1741.3, 1728.1, 1710.9, 1476.6, 1370.9, 1338.3, 1298.4, 1249.3, 1152.3, 1108.6, 821.2, 766.3 cm^{-1}. HRMS (ESI) Calcd. for C37H41Cl2N3NaO12 $[M+Na]^+$: 812.1960, Found: 812.1949.



3p

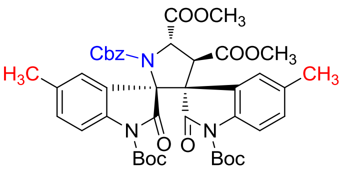
Product **3p** was obtained in 98% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.87 (d, *J*= 2.1 Hz, 1H), 7.68-7.62 (m, 2H), 7.47-7.33 (m, 6H), 7.22-7.16 (m, 1H), 6.74 (s, 1H), 5.45 (d, *J*= 6.0 Hz, 1H), 5.15-4.83 (m, 2H), 4.14 (d, *J*= 7.0 Hz, 1H), 3.95-3.61 (m, 3H), 3.52 (s, 3H), 1.49 (s, 9H), 1.44 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.9, 170.7, 167.1, 152.5, 147.9, 138.4, 137.6, 133.5, 133.1, 133.0, 129.8, 129.1, 128.5, 128.4, 128.2, 128.0, 127.8, 126.8, 126.7, 123.5, 117.5, 116.9, 116.2, 116.1, 85.2, 84.6, 68.6, 61.8, 52.9, 52.6, 27.7. IR (KBr) *v*

3442.4, 2924.1, 1852.5, 1781.7, 1756.6, 1724.7, 1472.2, 1335.5, 1249.9, 1155.2, 1112.8, 1020.0, 821.7, 701.4 cm^{-1}. HRMS (ESI) Calcd. for C40H39Br2N3NaO12 [M+Na]$^+$: 934.0793, Found: 934.0793.



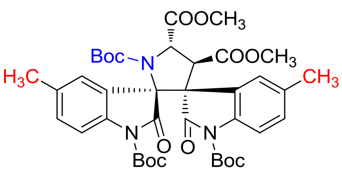
3q

Product **3q** was obtained in 98% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ7.94 (s, 1H), 7.67-7.58 (m, 2H), 7.43-7.39 (m, 2H), 7.35 (s., 1H), 5.36 (d, *J*= 4.4 Hz, 1H), 4.15 (d, *J*= 7.7 Hz, 1H), 3.93 (s, 3H), 3.48 (s, 3H), 1.47 (s, 9H), 1.44 (s, 9H), 1.24 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.1, 169.9, 169.1, 167.2, 151.6, 148.1, 148.0, 138.2, 137.4, 133.0, 132.7, 129.7, 129.2, 128.3, 127.4, 126.7, 117.4, 117.0, 116.4, 85.1, 84.6, 83.1, 61.7, 53.0, 52.6, 51.9, 27.8. IR (KBr) *ν* 3441.3, 2981.8, 2924.3, 2852.0, 1784.1, 1731.7, 1474.7, 1369.8, 1338.7, 1298.7, 1250.1, 1151.3, 1109.0, 835.9 cm^{-1}. HRMS (ESI) Calcd. for C37H41Br2N3NaO12 [M+Na]$^+$: 900.0949, Found: 900.0926.



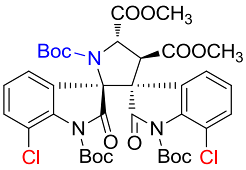
3r

Product **3r** was obtained in 86% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ7.58 (d, *J*= 8.6 Hz, 2H), 7.33 (s, 3H), 7.07 (d, *J*= 7.7 Hz, 4H), 6.66 (s, 1H), 5.47 (s, 1H), 5.08-4.77 (m, 2H), 4.23 (d, *J*= 8.1 Hz, 1H), 3.75 (d, *J*= 109.7 Hz, 3H), 3.42 (s, 3H), 2.37 (s, 3H), 2.33 (s, 3H), 1.44 (s, 9H), 1.39 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.2, 170.7, 170.2, 167.5, 152.7, 148.2, 136.7, 136.0, 135.2, 133.8, 133.5, 130.3, 128.2, 127.3, 126.7, 125.0, 122.1, 114.3, 114.2, 84.1, 83.5, 68.2, 61.8, 58.4, 52.7, 52.4, 51.5, 27.7, 21.3, 21.1. IR (KBr) *ν* 3439.6, 2923.5, 1777.4, 1754.4, 1728.7, 1719.2, 1492.3, 1341.7, 1159.7, 1114.8, 996.6, 765.3, 700.6 cm^{-1}. HRMS (ESI) Calcd. for C42H45N3NaO12 [M+Na]$^+$: 806.2895, Found: 806.2894.



3s

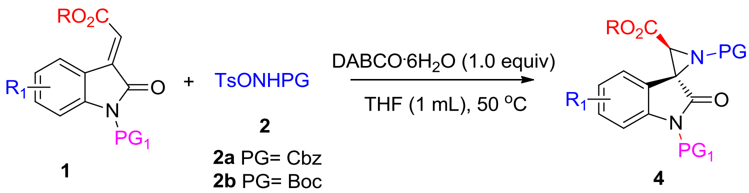
Product **3s** was obtained in 82% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ7.66-7.49 (m, 3H), 7.06 (d, *J*= 7.3 Hz, 3H), 5.37 (d, *J*= 8.6 Hz, 1H), 4.23 (d, *J*= 8.6 Hz, 1H), 3.91 (s, 3H), 3.38 (s, 3H), 2.37 (s, 3H), 2.33 (s, 3H), 1.43 (s, 9H), 1.40 (s, 9H), 1.02 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.5, 170.7, 170.4, 167.6, 151.8, 148.4, 48.2, 136.6, 135.8, 133.7, 133.4, 130.2, 130.0, 127.2, 126.7, 125.9, 122.4, 114.1, 113.8, 84.0, 83.5, 82.4, 61.7, 58.3, 52.8, 52.3, 51.8, 27.7, 21.2, 21.1. IR (KBr) *ν* 3447.4, 2980.1, 2928.5, 2854.1, 1778.9, 1734.6, 1716.8, 1491.7, 1370.1, 1341.4, 1249.7, 1157.0, 819.9 cm^{-1}. HRMS (ESI) Calcd. for C39H47N3NaO12 $[M+Na]^+$: 772.3052, Found: 772.3031.



3t

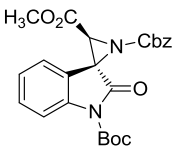
Product **3t** was obtained in 98% yield as a white solid. ^1H NMR (300 MHz, $CDCl_3$), δ8.01 (d, *J*= 7.4 Hz, 1H), 7.32-7.28 (m, 3H), 7.21-7.10 (m, 2H), 5.26 (d, *J*= 9.4 Hz, 1H), 4.25 (d, *J*= 9.5 Hz, 1H), 3.90 (s, 3H), 3.37 (s, 3H), 1.50 (s, 9H), 1.45 (s, 9H), 1.08 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.6, 170.2, 169.9, 166.7, 151.3, 147.2, 146.7, 136.4, 135.3, 131.5, 131.3, 129.2, 125.7, 125.6, 125.2, 125.0, 124.5, 118.6, 118.3, 85.6, 85.1, 83.4, 62.3, 60.8, 58.4, 53.2, 52.6, 27.6. IR (KBr) *v* 3441.9, 2983.2, 2924.8, 1803.9, 1782.7, 1754.5, 1729.8, 1711.7, 1370.6, 1347.9, 1250.8, 1149.6, 839.7, 734.6 cm^{-1}. HRMS (ESI) Calcd. for C37H41Cl2N3NaO12 $[M+Na]^+$: 812.1960, Found: 812.1938.

6. Experimental data for spirocyclic aziridines 4



General procedure: To a 5.0 mL vial were successively added methyleneindolinones **1** (0.15 mmol), N-tosyloxycarbamates **2** (0.375 mmol), DABCO·6H_2O (33.0 mg, 0.15 mmol) and 1.0 mL THF. The reaction was kept at 50 °C till almost full consumption of **1** by TLC analysis (5-60 min), and then the reaction mixture was directly subjected to flash column chromatography on silica gel (petroleum ether/ ethyl acetate) to afford the corresponding products **4**.

1-benzyl 1'-tert-butyl 3-methyl 2'-oxospiro[aziridine -2,3'-indoline] -1,1',3-tricarboxylate(4a)

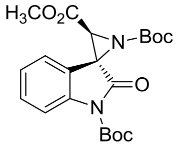


4a

Product **4a** was obtained in 72% yield as a white solid. ^1H NMR (300 MHz, $CDCl_3$), δ 7.94 (d, *J* = 8.3 Hz, 1H), 7.43-7.34 (m, 7H), 7.17 (t, *J* = 7.5 Hz, 1H), 5.19 (q, *J* = 12.0 Hz, 2H), 3.91 (s, 1H), 3.75 (s, 3H), 1.64 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.5, 164.7, 157.9, 148.4, 141.4, 134.7, 130.7, 128.7, 128.5, 128.4, 124.9, 123.8, 119.6, 115.4, 85.2, 69.3, 52.8, 49.4, 49.1, 28.0. IR (KBr) *v* 3435.5, 2924.0, 2851.4, 1775.3, 1741.4, 1324.9, 1293.6, 1252.1, 1216.6, 1169.9, 1099.5, 762.4, 730.7 cm^{-1}.

HRMS (ESI) Calcd. for C24H24N2NaO7 $[M+Na]^+$: 475.1476, Found: 475.1468.

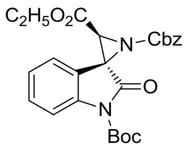
1,1'-di-tert-butyl 3-methyl 2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarboxylate (4b)



4b

Product **4b** was obtained in 55% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.93 (d, *J* = 8.0 Hz, 1H), 7.41 (t, *J* = 7.2 Hz, 2H), 7.17 (t, *J* = 7.8 Hz, 1H), 3.86 (s, 1H), 3.76 (s, 3H), 1.63 (s, 9H), 1.45 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.4, 165.2, 156.4, 148.5, 141.3, 130.4, 124.8, 123.8, 120.1, 115.2, 85.0, 83.7, 52.8, 49.3, 49.1, 28.0, 27.8. HRMS (ESI) Calcd. for C21H26N2NaO7 $[M+Na]^+$: 441.1632, Found: 441.1627.

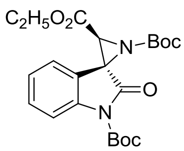
1-benzyl 1'-tert-butyl 3-ethyl 2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarboxylate (4c)



4c

Product **4c** was obtained in 77% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.93 (d, *J* = 8.1 Hz, 1H), 7.43-7.31 (m, 7H), 7.16 (t, *J* = 7.8 Hz, 1H), 5.18 (q, *J* = 12.0 Hz, 2H), 4.27-4.14 (m, 2H), 3.90 (s, 1H), 1.65 (s, 9H), 1.24 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.5, 164.2, 158.0, 148.4, 141.3, 134.7, 130.6, 128.7, 128.5, 128.4, 124.8, 123.9, 119.6, 115.4, 85.2, 69.3, 62.2, 49.5, 49.0, 28.0, 14.0. IR (KBr) *v* 3440.3, 2923.2, 2851.2, 1771.0, 1758.3, 1743.8, 1322.1, 1246.1, 1183.1, 1152.0, 1038.4, 779.2 cm^{-1}. HRMS (ESI) Calcd. for C25H26N2NaO7 $[M+Na]^+$: 489.1632, Found: 489.1623.

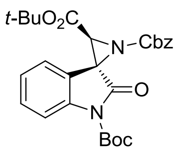
1,1'-di-tert-butyl 3-ethyl 2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarboxylate (4d)



4d

Product **4d** was obtained in 58% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.93 (d, *J* = 8.1 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.16 (t, *J* = 7.5 Hz, 1H), 4.27-4.14 (m, 2H), 3.84 (s, 1H), 1.63 (s, 9H), 1.44 (s, 9H), 1.24 (t, *J* = 4.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.4, 164.7, 156.5, 148.5, 141.3, 130.4, 124.7, 123.9, 120.2, 115.2, 85.0, 83.6, 62.1, 49.4, 49.0, 28.0, 27.8, 14.0. IR (KBr) *v* 3456.5, 2981.7, 2932.4, 1771.6, 1738.4, 1468.7, 1369.9, 1325.8, 1252.9, 1189.5, 1151.4, 1100.6, 1025.2, 771.8 cm^{-1}. HRMS (ESI) Calcd. for C22H28N2NaO7 $[M+Na]^+$: 455.1789, Found: 455.1783.

1-benzyl 1',3-di-tert-butyl 2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarboxylate (4e)

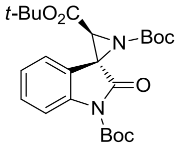


4e

Product **4e** was obtained in 70% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.93 (d, *J* = 8.1 Hz, 1H), 7.43-7.31 (m, 7H), 7.15 (t, *J* = 7.5 Hz, 1H), 5.18 (q, *J* = 12.0 Hz, 2H), 3.82 (s, 1H), 1.65 (s, 9H), 1.40 (s, 9H); ^{13}C NMR (75

MHz, $CDCl_3$) δ 167.7, 163.1, 158.2, 148.5, 141.2, 134.8, 130.5, 128.7, 128.4, 124.6, 123.9, 119.8, 115.3, 85.1, 83.6, 69.2, 50.1, 48.7, 28.0, 27.9. IR (KBr) *ν* 3438.7, 2982.8, 2922.8, 1795.8, 1766.1, 1742.8, 1326.0, 1254.5, 1150.5, 838.1, 806.7, 774.9 cm^{-1}. HRMS (ESI) Calcd. for C27H30N2NaO7 $[M+Na]^+$: 517.1945, Found: 517.1934.

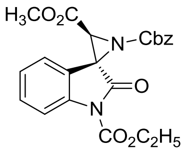
tri-tert-butyl 2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarboxylate (4f)



4f

Product **4f** was obtained in 59% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.92 (d, *J* = 8.2 Hz, 1H), 7.44-7.37 (m, 2H), 7.15 (t, *J* = 7.6 Hz, 1H), 3.76 (s, 1H), 1.63 (s, 9H), 1.44 (s, 9H), 1.40 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.6, 163.6, 156.7, 148.6, 141.2, 130.3, 124.6, 123.9, 120.3, 115.1, 84.9, 83.4, 83.3, 50.0, 48.8, 28.0, 27.9, 27.8. IR (KBr) *ν* 3440.1, 2980.9, 2930.7, 1798.2, 1774.5, 1740.1, 1370.6, 1326.1, 1292.5, 1254.0, 1149.0, 841.7, 808.3, 756.4 cm^{-1}. HRMS (ESI) Calcd. for C24H32N2NaO7 $[M+Na]^+$: 483.2102, Found: 483.2090.

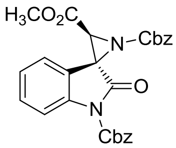
1-benzyl 1'-ethyl 3-methyl 2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarboxylate (4g)



4g

Product **4g** was obtained in 59% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 8.00 (d, *J* = 8.1 Hz, 1H), 7.43 (t, *J* = 7.8 Hz, 2H), 7.34 (s, 5H), 7.19 (t, *J* = 7.6 Hz, 1H), 5.19 (q, *J* = 12.0 Hz, 2H), 4.53-4.46 (m, 2H), 3.92 (s, 1H), 3.76 (s, 3H), 1.46 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.4, 164.6, 157.7, 150.1, 141.0, 134.7, 130.8, 128.7, 128.5, 128.4, 125.2, 124.0, 119.7, 115.5, 69.4, 63.9, 52.9, 49.5, 49.0, 14.2. IR (KBr) *ν* 3436.7, 2959.9, 2921.1, 2850.8, 1799.0, 1776.6, 1748.7, 1741.0, 1251.1, 1184.0, 1171.9, 1098.9, 1026.9, 799.3, 760.0 cm^{-1}. HRMS (ESI) Calcd. for C22H20N2NaO7 $[M+Na]^+$: 447.1163, Found: 447.1163.

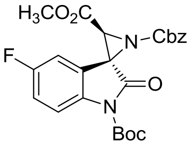
1,1'-dibenzyl 3-methyl 2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarboxylate (4h)



4h

Product **4h** was obtained in 72% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 8.00 (d, *J* = 8.4 Hz, 1H), 7.53-7.30 (m, 12 H), 7.19 (t, *J* = 7.6 Hz, 1H), 5.46 (q, *J* = 12.3 Hz, 2H), 5.19 (q, *J* = 12.0 Hz, 2H), 3.93 (s, 1H), 3.75 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.3, 164.6, 157.7, 150.0, 140.9, 134.6, 134.5, 130.8, 128.7, 128.6, 128.5, 128.4, 128.2, 125.3, 124.0, 119.7, 115.6, 69.4, 69.1, 52.9, 49.5, 49.0. IR (KBr) *ν* 3439.8, 2961.0, 2921.4, 2850.8, 1798.6, 1774.1, 1744.7, 1260.7, 1223.6, 1099.7, 1028.7, 803.0, 760.6, 698.8 cm^{-1}. HRMS (ESI) Calcd. for C27H22N2NaO7 $[M+Na]^+$: 509.1319, Found: 509.1316.

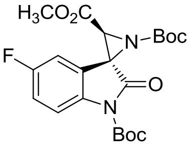
1-benzyl 1'-tert-butyl 3-methyl 5'-fluoro-2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarb-oxylate (4i)



4i

Product **4i** was obtained in 81% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.93 (dd, J_1 = J_2= 4.4 Hz, 1H), 7.34 (s, 5H), 7.18 (dd, J_1 = 2.7 Hz, J_2 = 2.6 Hz, 1H), 7.14-7.07 (m, 1H), 5.19 (t, J = 12.5 Hz, 2H), 3.91 (s, 1H), 3.78 (s, 3H), 1.64 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.1, 163.0 (d, J = 229.4 Hz, 1C), 158.2, 157.6, 148.3, 137.3, 134.6, 128.7, 128.6, 128.5, 121.5 (d, J = 9.3 Hz, 1C), 117.3 (d, J = 23.0 Hz, 1C), 116.9 (d, J = 7.8 Hz, 1C), 111.7 (d, J = 26.2 Hz, 1C), 85.4, 69.5, 53.0, 49.5, 48.8, 28.0. IR (KBr) ν 3438.2, 2954.3, 2923.7, 2850.9, 1777.7, 1740.9, 1484.7, 1283.2, 1256.4, 1169.4, 803.2, 730.3 cm^{-1}. HRMS (ESI) Calcd. for C24H23FN2NaO7 [M+Na]$^+$: 493.1382, Found: 493.1387.

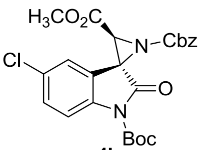
1,1'-di-tert-butyl 3-methyl 5'-fluoro-2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarb-oxylate (4j)



4j

Product **4j** was obtained in 91% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.91 (dd, J_1= J_2= 4.4 Hz, 1H), 7.18 (dd, J_1= J_2= 2.7 Hz, 1H), 7.13-7.07 (m, 1H), 3.86 (s, 1H), 3.78 (s, 3H), 1.62 (s, 9H), 1.45 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.1, 163.2 (d, J = 265.1 Hz, 1C), 158.2, 156.0, 148.1, 137.3 (d, J = 2.6 Hz, 1C), 122.0 (d, J = 9.1 Hz, 1C), 117.1 (d, J = 23.0 Hz, 1C), 116.7 (d, J = 7.8 Hz, 1C), 111.7 (d, J = 26.1 Hz, 1C), 85.2, 83.9, 52.9, 49.4, 48.9, 28.0, 27.8. IR (KBr) ν 3431.4, 2989.1, 2981.0, 2924.1, 1774.3, 1759.9, 1748.2, 1739.9, 1481.7, 1369.9, 1329.3, 1299.1, 1272.1, 1209.7, 1150.4, 841.1, 806.9 cm^{-1}. HRMS (ESI) Calcd. for C21H25FN2NaO7 [M+Na]$^+$: 459.1538, Found: 459.1525.

1-benzyl 1'-tert-butyl 3-methyl 5'-chloro-2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarb-oxylate (4k)

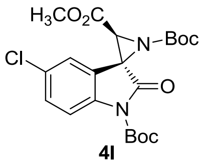


4k

Product **4k** was obtained in 80% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.90 (d, J = 8.7 Hz, 1H), 7.43-7.34 (m, 7H), 5.19 (q, J = 12.1 Hz, 2H), 3.90 (s, 1H), 3.78 (s, 3H), 1.63 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.9, 164.5, 157.5, 148.2, 139.8, 134.6, 130.7, 130.6, 128.7, 128.6, 128.5, 124.3, 121.4, 116.7, 85.6, 69.5, 53.0, 49.5, 48.6, 27.9. IR (KBr) ν 3438.3, 2981.3, 1926.6, 1778.9, 1744.6, 1457.1, 1328.0, 1297.7, 1255.3, 1178.1, 1153.1, 829.0, 696.5 cm^{-1}. HRMS (ESI) Calcd. for

C24H23ClN2NaO7 [M+Na]+: 509.1086, Found: 509.1078.

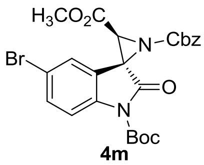
1,1'-di-tert-butyl 3-methyl 5'-chloro-2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarboxylate (4l)



4l

Product **4l** was obtained in 79% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.88 (d, *J* = 8.7 Hz, 1H), 7.42 (d, *J* = 2.0 Hz, 1H), 7.37 (dd, *J* = 2.2 Hz, 1H), 3.85 (s, 1H), 3.79 (s, 3H), 1.62 (s, 9H), 1.45 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.8, 164.9, 156.0, 148.3, 139.8, 130.5, 130.4, 124.3, 121.9, 116.5, 85.4, 84.0, 52.9, 49.4, 48.7, 28.0, 27.8. IR (KBr) *ν* 3433.5, 2977.4, 2929.3, 1775.4, 1760.2, 1742.8, 1371.3, 1326.0, 1253.8, 1153.8, 827.6, 808.4 cm^{-1}. HRMS (ESI) Calcd. for C21H25ClN2NaO7 [M+Na]+: 475.1242, Found: 475.1253.

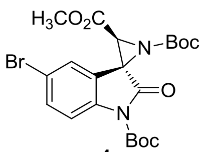
1-benzyl 1'-tert-butyl 3-methyl 5'-bromo-2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarb-oxylate (4m)



4m

Product **4m** was obtained in 65% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.69 (d, *J* = 8.6 Hz, 1H), 7.56-7.51 (m, 2H), 7.34 (s, 5H), 5.19 (q, *J* = 12.2 Hz, 2H), 3.90 (s, 1H), 3.79 (s, 3H), 1.63 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.7, 164.5, 157.5, 148.2, 140.4, 134.6, 133.6, 128.7, 128.6, 128.5, 127.1, 121.7, 118.0, 117.1, 85.6, 69.5, 53.0, 49.5, 48.5, 28.0. IR (KBr) *ν* 3438.3, 2982.0, 2927.3, 1764.2, 1753.1, 1730.1, 1263.5, 1173.9, 1151.0, 833.5, 800.5, 755.7, 701.8 cm^{-1}. HRMS (ESI) Calcd. for C24H23BrN2NaO7 [M+Na]+: 553.0581, Found: 553.0575.

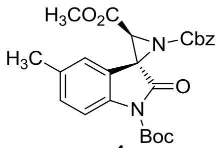
1,1'-di-tert-butyl 3-methyl 5'-bromo-2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarboxylate (4n)



4n

Product **4n** was obtained in 63% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.82 (d, *J* = 8.6 Hz, 1H), 7.56-7.51 (m, 2H), 3.85 (s, 1H), 3.79 (s, 3H), 1.62 (s, 9H), 1.44 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.7, 164.9, 156.0, 148.3, 140.3, 133.4, 127.1, 122.2, 118.0, 116.8, 85.4, 84.0, 52.9, 49.5, 48.6, 28.0, 27.4. IR (KBr) *ν* 3434.1, 2922.7, 2851.2, 1776.1, 1761.9, 1749.9, 1736.9, 1326.3, 1259.6, 1150.1, 826.5, 807.4 cm^{-1}. HRMS (ESI) Calcd. for C21H25BrN2NaO7 [M+Na]+: 519.0737, Found: 519.0731.

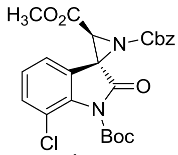
1-benzyl 1'-tert-butyl 3-methyl 5'-methyl-2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarb-oxylate (4o)



4o

Product **4o** was obtained in 55% yield as a white solid. [1]H NMR (300 MHz, $CDCl_3$), δ 7.80 (d, *J* = 8.8 Hz, 1H), 7.33 (s, 5H), 7.20 (d, *J* = 5.0 Hz, 2H), 5.18 (q, *J* = 12.0 Hz, 2H), 3.90 (s, 1H), 3.76 (s, 3H), 2.32 (s, 3H), 1.64 (s, 9H); [13]C NMR (75 MHz, $CDCl_3$) δ 167.6, 164.8, 158.0, 148.4, 139.0, 134.8, 131.2, 128.7, 128.5, 128.4, 124.3, 119.5, 115.2, 85.0, 69.3, 52.8, 49.4, 49.2, 28.0, 21.0 (one carbon missing). IR (KBr) *v* 3438.6, 2982.0, 2921.4, 1794.8, 1754.9, 1734.5, 1486.0, 1320.4, 1266.2, 1189.4, 1153.1, 1125.9, 827.5, 800.6, 753.7, 700.5 cm^{-1}. HRMS (ESI) Calcd. for C25H26N2NaO7 $[M+Na]^+$: 489.1632, Found: 489.1630.

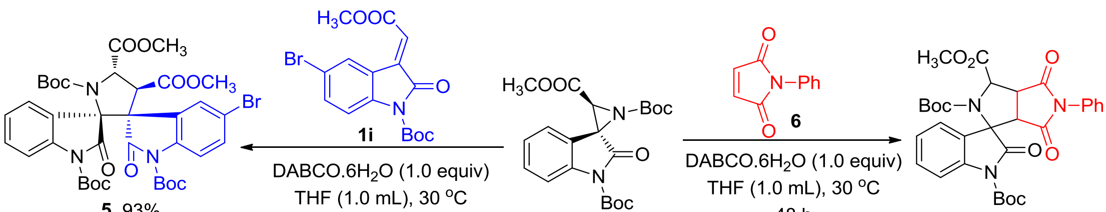
1-benzyl 1'-tert-butyl 3-methyl 7'-chloro-2'-oxospiro[aziridine-2,3'-indoline]-1,1',3-tricarboxylate (4p)



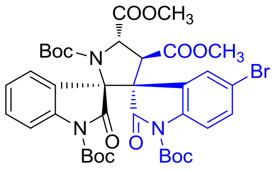
4p

Product **4p** was obtained in 65% yield as a white solid. [1]H NMR (300 MHz, $CDCl_3$), δ 7.40-7.31 (m, 7H), 7.10 (t, *J* = 7.8 Hz, 1H), 5.17 (q, *J* = 12.0 Hz, 2H), 3.92 (s, 1H), 3.77 (s, 3H), 1.63 (s, 9H); [13]C NMR (75 MHz, $CDCl_3$) δ 167.7, 164.6, 157.7, 146.9, 138.2, 134.5, 132.3, 128.7, 128.6, 128.5, 125.6, 122.9, 122.8, 119.1, 86.3, 69.5, 52.9, 49.2, 49.0, 27.6. IR (KBr) *v* 3435.6, 2957.5, 2920.2, 1758.2, 1250.5, IR (KBr) *v* 3435.6, 2957.5, 2920.2, 1758.2, 1250.5, 1227.7, 1172.7, 1139.0, 807.7, 784.1, 755.4, 701.0 cm^{-1}. HRMS (ESI) Calcd. for C24H23ClN2NaO7 $[M+Na]^+$: 509.1086, Found: 509.1091.

7. **Experimental data for compounds 5 and 7**

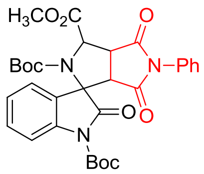


General procedure: To a 5.0 mL vial were successively added spiroaziridine oxindole **4b** (0.10 mmol), 5-Br-substituted methyleneindolinone **1i** or **N**-phenyl maleimide **6**, DABCO·$6H_2O$ (33.0 mg, 0.10 mmol) and 1.0 mL THF. The reaction was kept at 30 °C till almost full consumption of **4b** by TLC analysis (2 days), and then the reaction mixture was directly subjected to flash column chromatography on silica gel (petroleum ether/ ethyl acetate) to afford the corresponding products **5** or **7**.



5

Product **5** was obtained in 93% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.83 (s, 1H), 7.65-7.60 (m, 2H), 7.43-7.37 (m, 2H), 7.29 (d, J = 7.6 Hz, 1H), 7.23-7.16 (m, 1H), 5.37 (d, J = 8.0 Hz, 1H), 4.20 (d, J = 8.1 Hz, 1H), 3.92 (s, 3H), 3.48 (s, 3H), 1.45 (s, 9H), 1.43 (s, 9H), 1.24 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.3, 170.0, 169.3, 167.3, 151.6, 148.4, 148.0, 138.3, 138.2, 133.3, 132.8, 129.8, 126.2, 125.4, 124.3, 116.8, 116.0, 114.6, 114.2, 84.7, 82.7, 61.6, 59.8, 52.9, 52.5, 51.7, 48.7, 28.1. IR (KBr) v 3445.3, 2980.4, 2954.8, 2926.6, 2852.9, 1779.7, 1736.4, 1713.7, 1477.4, 1370.3, 1346.2, 1297.5, 1250.8, 1152.0, 1105.0, 70.1 cm^{-1}. HRMS (ESI) Calcd. for C37H42BrN3NaO12 $[M+Na]^+$: 822.1844, Found: 822.1848.



7

Product **7** was obtained in 95% yield as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$), δ 7.89-7.84 (m, 2H), 7.47-7.29 (m, 6H), 7.04 (d, J = 7.0 Hz, 1H), 5.19 (dd, J_1 = 9.7 Hz, J_2 = 44.6 Hz, 1H), 4.16-4.00 (m, 1H), 3.89 (d, J = 11.1 Hz, 3H), 3.70 (d, J = 8.4 Hz, 1H), 1.62 (d, J = 16.5 Hz, 9H), 1.07 (d, J = 9.5 Hz, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.1, 172.5, 172.0, 170.5, 148.6, 139.7, 130.3, 129.9, 129.1, 129.0, 128.9, 126.8, 125.9, 125.4, 124.6, 115.2, 84.9, 84.5, 62.3, 61.7, 53.0, 52.8, 48.0, 28.0. IR (KBr) v 3436.0, 2978.8, 2920.6, 2850.9, 1797.9, 1758.7, 1723.6, 1370.7, 1150.1, 754.5 cm^{-1}. HRMS (ESI) Calcd. for C31H33N3NaO9 $[M+Na]^+$: 614.2109, Found: 614.2110.

8. Crystal data for 3a and 4a

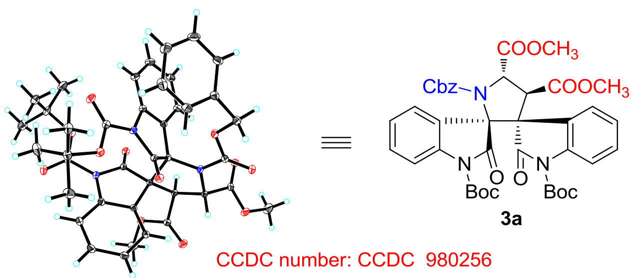


Table 1. Crystal data and structure refinement for compound **3a**.

Identification code	**3a**
Empirical formula	C40H41N3O12

Formula weight	755.76
Temperature	100(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P 21/c
Unit cell dimensions	a = 10.8958(12) A alpha = 90 deg.
	b = 17.3981(19) A beta = 91.143(2) deg.
	c = 19.868(2) A gamma = 90 deg.
Volume	3765.6(7) A^3
Z, Calculated density	4, 1.333 Mg/m^3
Absorption coefficient	0.099 mm^-1
F(000)	1592
Crystal size	0.44 x 0.35 x 0.35 mm
Theta range for data collection	1.56 to 30.10 deg.
Limiting indices	-15<=h<=13, -24<=k<=24, -28<=l<=26
Reflections collected / unique	38954 / 10572 [R(int) = 0.0365]
Completeness to theta = 30.10	95.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9661 and 0.9576
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	10572 / 0 / 504
Goodness-of-fit on F^2	1.041
Final R indices [I>2sigma(I)]	R1 = 0.0449, wR2 = 0.1174
R indices (all data)	R1 = 0.0597, wR2 = 0.1276
Largest diff. peak and hole	0.454 and -0.310 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for compound **3a**.

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Atom	x	y	z	U(eq)

O(1)	2217(1)	5932(1)	8564(1)	23(1)
O(2)	2284(1)	9436(1)	10126(1)	19(1)
C(39)	1416(1)	8719(1)	11372(1)	25(1)
C(38)	627(2)	8170(1)	11626(1)	31(1)
C(40)	1857(2)	4612(1)	8153(1)	35(1)
O(6)	3682(1)	5282(1)	9162(1)	28(1)
O(7)	2955(1)	7284(1)	8061(1)	19(1)
O(8)	3918(1)	8129(1)	10546(1)	18(1)
O(9)	1007(1)	6553(1)	10566(1)	23(1)
O(10)	2969(1)	6775(1)	10922(1)	21(1)
O(11)	3420(1)	10125(1)	8185(1)	22(1)
O(12)	5432(1)	10373(1)	8358(1)	26(1)
O(3)	5851(1)	7679(1)	7873(1)	23(1)
O(4)	6764(1)	8777(1)	8206(1)	27(1)
O(5)	3630(1)	10359(1)	9793(1)	20(1)
N(1)	3893(1)	6563(1)	8925(1)	17(1)
N(2)	3644(1)	9145(1)	9359(1)	16(1)
N(3)	2153(1)	7517(1)	10111(1)	15(1)
C(1)	257(2)	5647(1)	8134(1)	35(1)
C(2)	1311(1)	5287(1)	8531(1)	22(1)
C(3)	3270(1)	5851(1)	8895(1)	18(1)
C(4)	3566(1)	7239(1)	8566(1)	15(1)
C(5)	4183(1)	7913(1)	8946(1)	13(1)
C(6)	3192(1)	8361(1)	9380(1)	13(1)
C(7)	3224(1)	9710(1)	9772(1)	16(1)
C(8)	1822(1)	9893(1)	10683(1)	22(1)
C(9)	949(1)	9355(1)	11021(1)	20(1)
C(10)	-629(2)	8252(1)	11526(1)	36(1)
C(11)	928(1)	5086(1)	9238(1)	26(1)
C(12)	4910(1)	6721(1)	9372(1)	16(1)

C(13)	5149(1)	7511(1)	9372(1)	15(1)
C(14)	1872(1)	8252(1)	9158(1)	15(1)
C(15)	1209(1)	8612(1)	8647(1)	18(1)
C(16)	-33(1)	8438(1)	8566(1)	22(1)
C(17)	-587(1)	7915(1)	8995(1)	24(1)
C(18)	66(1)	7555(1)	9516(1)	21(1)
C(19)	1301(1)	7737(1)	9590(1)	15(1)
C(20)	3193(1)	8008(1)	10100(1)	14(1)
C(21)	1959(1)	6903(1)	10558(1)	17(1)
C(22)	3061(1)	6092(1)	11368(1)	21(1)
C(23)	4381(1)	6149(1)	11625(1)	29(1)
C(24)	2159(1)	6166(1)	11939(1)	22(1)
C(25)	2863(2)	5362(1)	10958(1)	29(1)
C(26)	4646(1)	9286(1)	8900(1)	14(1)
C(27)	4396(1)	9983(1)	8450(1)	16(1)
C(28)	5348(2)	11029(1)	7908(1)	37(1)
C(29)	4643(1)	8546(1)	8476(1)	14(1)
C(30)	5881(1)	8366(1)	8179(1)	17(1)
C(31)	6971(1)	7462(1)	7539(1)	32(1)
C(32)	-312(1)	9435(1)	10942(1)	26(1)
C(33)	-1101(1)	8880(1)	11196(1)	36(1)
C(34)	6115(1)	7811(1)	9751(1)	18(1)
C(35)	6846(1)	7307(1)	10132(1)	21(1)
C(36)	6584(1)	6526(1)	10136(1)	24(1)
C(37)	5611(1)	6216(1)	9758(1)	22(1)

Table 3. Bond lengths [A] and angles [deg] for compound **3a**.

O(1)-C(3)	1.3186(16)
O(1)-C(2)	1.4953(15)

O(2)-C(7)	1.3418(15)
O(2)-C(8)	1.4592(15)
C(39)-C(38)	1.388(2)
C(39)-C(9)	1.3978(19)
C(39)-H(39)	0.9500
C(38)-C(10)	1.386(2)
C(38)-H(38)	0.9500
C(40)-C(2)	1.522(2)
C(40)-H(40A)	0.9800
C(40)-H(40B)	0.9800
C(40)-H(40C)	0.9800
O(6)-C(3)	1.2056(16)
O(7)-C(4)	1.1963(15)
O(8)-C(20)	1.1937(15)
O(9)-C(21)	1.2039(16)
O(10)-C(21)	1.3237(15)
O(10)-C(22)	1.4849(15)
O(11)-C(27)	1.2023(16)
O(12)-C(27)	1.3331(15)
O(12)-C(28)	1.4515(16)
O(3)-C(30)	1.3415(15)
O(3)-C(31)	1.4498(16)
O(4)-C(30)	1.1996(16)
O(5)-C(7)	1.2123(15)
N(1)-C(3)	1.4130(15)
N(1)-C(4)	1.4173(15)
N(1)-C(12)	1.4320(16)
N(2)-C(7)	1.3654(15)
N(2)-C(6)	1.4514(14)
N(2)-C(26)	1.4582(15)

N(3)-C(21)	1.4069(15)
N(3)-C(20)	1.4196(15)
N(3)-C(19)	1.4299(16)
C(1)-C(2)	1.516(2)
C(1)-H(1A)	0.9800
C(1)-H(1B)	0.9800
C(1)-H(1C)	0.9800
C(2)-C(11)	1.5133(19)
C(4)-C(5)	1.5411(16)
C(5)-C(13)	1.5089(16)
C(5)-C(29)	1.5356(16)
C(5)-C(6)	1.5969(16)
C(6)-C(14)	1.5089(16)
C(6)-C(20)	1.5571(16)
C(8)-C(9)	1.5026(19)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-C(32)	1.3868(19)
C(10)-C(33)	1.370(3)
C(10)-H(10)	0.9500
C(11)-H(11A)	0.9800
C(11)-H(11B)	0.9800
C(11)-H(11C)	0.9800
C(12)-C(37)	1.3859(18)
C(12)-C(13)	1.3990(16)
C(13)-C(34)	1.3835(17)
C(14)-C(15)	1.3841(17)
C(14)-C(19)	1.3948(16)
C(15)-C(16)	1.3930(18)
C(15)-H(15)	0.9500

C(16)-C(17)	1.3913(19)
C(16)-H(16)	0.9500
C(17)-C(18)	1.3948(19)
C(17)-H(17)	0.9500
C(18)-C(19)	1.3875(17)
C(18)-H(18)	0.9500
C(22)-C(23)	1.520(2)
C(22)-C(24)	1.5212(18)
C(22)-C(25)	1.5228(19)
C(23)-H(23A)	0.9800
C(23)-H(23B)	0.9800
C(23)-H(23C)	0.9800
C(24)-H(24A)	0.9800
C(24)-H(24B)	0.9800
C(24)-H(24C)	0.9800
C(25)-H(25A)	0.9800
C(25)-H(25B)	0.9800
C(25)-H(25C)	0.9800
C(26)-C(27)	1.5276(16)
C(26)-C(29)	1.5377(16)
C(26)-H(26)	1.0000
C(28)-H(28A)	0.9800
C(28)-H(28B)	0.9800
C(28)-H(28C)	0.9800
C(29)-C(30)	1.5162(17)
C(29)-H(29)	1.0000
C(31)-H(31A)	0.9800
C(31)-H(31B)	0.9800
C(31)-H(31C)	0.9800
C(32)-C(33)	1.394(2)

C(32)-H(32)	0.9500
C(33)-H(33)	0.9500
C(34)-C(35)	1.3972(18)
C(34)-H(34)	0.9500
C(35)-C(36)	1.3880(19)
C(35)-H(35)	0.9500
C(36)-C(37)	1.396(2)
C(36)-H(36)	0.9500
C(37)-H(37)	0.9500
C(3)-O(1)-C(2)	120.53(10)
C(7)-O(2)-C(8)	118.87(10)
C(38)-C(39)-C(9)	120.29(13)
C(38)-C(39)-H(39)	119.9
C(9)-C(39)-H(39)	119.9
C(10)-C(38)-C(39)	119.61(14)
C(10)-C(38)-H(38)	120.2
C(39)-C(38)-H(38)	120.2
C(2)-C(40)-H(40A)	109.5
C(2)-C(40)-H(40B)	109.5
H(40A)-C(40)-H(40B)	109.5
C(2)-C(40)-H(40C)	109.5
H(40A)-C(40)-H(40C)	109.5
H(40B)-C(40)-H(40C)	109.5
C(21)-O(10)-C(22)	120.41(10)
C(27)-O(12)-C(28)	116.16(11)
C(30)-O(3)-C(31)	115.20(11)
C(3)-N(1)-C(4)	126.17(11)
C(3)-N(1)-C(12)	124.06(10)
C(4)-N(1)-C(12)	109.60(10)
C(7)-N(2)-C(6)	122.83(10)

C(7)-N(2)-C(26)	121.29(10)
C(6)-N(2)-C(26)	115.75(9)
C(21)-N(3)-C(20)	126.63(10)
C(21)-N(3)-C(19)	123.87(10)
C(20)-N(3)-C(19)	109.50(10)
C(2)-C(1)-H(1A)	109.5
C(2)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1B)	109.5
C(2)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5
O(1)-C(2)-C(11)	109.14(11)
O(1)-C(2)-C(1)	101.82(11)
C(11)-C(2)-C(1)	110.94(13)
O(1)-C(2)-C(40)	109.68(11)
C(11)-C(2)-C(40)	113.39(12)
C(1)-C(2)-C(40)	111.23(13)
O(6)-C(3)-O(1)	128.30(12)
O(6)-C(3)-N(1)	121.79(12)
O(1)-C(3)-N(1)	109.87(10)
O(7)-C(4)-N(1)	127.29(11)
O(7)-C(4)-C(5)	126.31(11)
N(1)-C(4)-C(5)	106.39(10)
C(13)-C(5)-C(29)	116.15(10)
C(13)-C(5)-C(4)	102.41(9)
C(29)-C(5)-C(4)	113.07(9)
C(13)-C(5)-C(6)	113.27(9)
C(29)-C(5)-C(6)	102.15(9)
C(4)-C(5)-C(6)	110.06(9)
N(2)-C(6)-C(14)	115.60(10)

N(2)-C(6)-C(20)	113.75(9)
C(14)-C(6)-C(20)	101.68(9)
N(2)-C(6)-C(5)	102.14(9)
C(14)-C(6)-C(5)	115.56(9)
C(20)-C(6)-C(5)	108.36(9)
O(5)-C(7)-O(2)	126.46(11)
O(5)-C(7)-N(2)	124.36(11)
O(2)-C(7)-N(2)	109.16(10)
O(2)-C(8)-C(9)	103.30(10)
O(2)-C(8)-H(8A)	111.1
C(9)-C(8)-H(8A)	111.1
O(2)-C(8)-H(8B)	111.1
C(9)-C(8)-H(8B)	111.1
H(8A)-C(8)-H(8B)	109.1
C(32)-C(9)-C(39)	119.11(13)
C(32)-C(9)-C(8)	121.44(13)
C(39)-C(9)-C(8)	119.19(12)
C(33)-C(10)-C(38)	120.70(15)
C(33)-C(10)-H(10)	119.6
C(38)-C(10)-H(10)	119.6
C(2)-C(11)-H(11A)	109.5
C(2)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
C(2)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
C(37)-C(12)-C(13)	121.44(12)
C(37)-C(12)-N(1)	129.21(11)
C(13)-C(12)-N(1)	109.34(10)
C(34)-C(13)-C(12)	120.68(11)

C(34)-C(13)-C(5)	130.19(11)
C(12)-C(13)-C(5)	109.06(10)
C(15)-C(14)-C(19)	120.63(11)
C(15)-C(14)-C(6)	129.68(11)
C(19)-C(14)-C(6)	109.55(10)
C(14)-C(15)-C(16)	118.52(12)
C(14)-C(15)-H(15)	120.7
C(16)-C(15)-H(15)	120.7
C(17)-C(16)-C(15)	120.28(12)
C(17)-C(16)-H(16)	119.9
C(15)-C(16)-H(16)	119.9
C(16)-C(17)-C(18)	121.82(12)
C(16)-C(17)-H(17)	119.1
C(18)-C(17)-H(17)	119.1
C(19)-C(18)-C(17)	117.06(12)
C(19)-C(18)-H(18)	121.5
C(17)-C(18)-H(18)	121.5
C(18)-C(19)-C(14)	121.68(11)
C(18)-C(19)-N(3)	128.97(11)
C(14)-C(19)-N(3)	109.12(10)
O(8)-C(20)-N(3)	127.65(11)
O(8)-C(20)-C(6)	126.92(11)
N(3)-C(20)-C(6)	105.44(9)
O(9)-C(21)-O(10)	127.96(12)
O(9)-C(21)-N(3)	122.22(12)
O(10)-C(21)-N(3)	109.78(10)
O(10)-C(22)-C(23)	101.57(10)
O(10)-C(22)-C(24)	109.93(10)
C(23)-C(22)-C(24)	111.39(11)
O(10)-C(22)-C(25)	109.95(10)

C(23)-C(22)-C(25)	110.95(12)
C(24)-C(22)-C(25)	112.51(12)
C(22)-C(23)-H(23A)	109.5
C(22)-C(23)-H(23B)	109.5
H(23A)-C(23)-H(23B)	109.5
C(22)-C(23)-H(23C)	109.5
H(23A)-C(23)-H(23C)	109.5
H(23B)-C(23)-H(23C)	109.5
C(22)-C(24)-H(24A)	109.5
C(22)-C(24)-H(24B)	109.5
H(24A)-C(24)-H(24B)	109.5
C(22)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5
C(22)-C(25)-H(25A)	109.5
C(22)-C(25)-H(25B)	109.5
H(25A)-C(25)-H(25B)	109.5
C(22)-C(25)-H(25C)	109.5
H(25A)-C(25)-H(25C)	109.5
H(25B)-C(25)-H(25C)	109.5
N(2)-C(26)-C(27)	111.89(10)
N(2)-C(26)-C(29)	102.03(9)
C(27)-C(26)-C(29)	110.20(9)
N(2)-C(26)-H(26)	110.8
C(27)-C(26)-H(26)	110.8
C(29)-C(26)-H(26)	110.8
O(11)-C(27)-O(12)	125.44(12)
O(11)-C(27)-C(26)	124.41(11)
O(12)-C(27)-C(26)	109.99(10)
O(12)-C(28)-H(28A)	109.5

O(12)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5
O(12)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
C(30)-C(29)-C(5)	113.13(10)
C(30)-C(29)-C(26)	113.19(10)
C(5)-C(29)-C(26)	105.35(9)
C(30)-C(29)-H(29)	108.3
C(5)-C(29)-H(29)	108.3
C(26)-C(29)-H(29)	108.3
O(4)-C(30)-O(3)	124.35(12)
O(4)-C(30)-C(29)	125.34(11)
O(3)-C(30)-C(29)	110.31(10)
O(3)-C(31)-H(31A)	109.5
O(3)-C(31)-H(31B)	109.5
H(31A)-C(31)-H(31B)	109.5
O(3)-C(31)-H(31C)	109.5
H(31A)-C(31)-H(31C)	109.5
H(31B)-C(31)-H(31C)	109.5
C(9)-C(32)-C(33)	120.35(14)
C(9)-C(32)-H(32)	119.8
C(33)-C(32)-H(32)	119.8
C(10)-C(33)-C(32)	119.90(15)
C(10)-C(33)-H(33)	120.1
C(32)-C(33)-H(33)	120.1
C(13)-C(34)-C(35)	118.52(12)
C(13)-C(34)-H(34)	120.7
C(35)-C(34)-H(34)	120.7
C(36)-C(35)-C(34)	120.17(12)

C(36)-C(35)-H(35)	119.9
C(34)-C(35)-H(35)	119.9
C(35)-C(36)-C(37)	121.90(12)
C(35)-C(36)-H(36)	119.1
C(37)-C(36)-H(36)	119.1
C(12)-C(37)-C(36)	117.26(12)
C(12)-C(37)-H(37)	121.4
C(36)-C(37)-H(37)	121.4

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for compound **3a**.

The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

Atom	U11	U22	U33	U23	U13	U12
O(1)	23(1)	19(1)	26(1)	4(1)	-3(1)	-7(1)
O(2)	20(1)	18(1)	20(1)	-4(1)	7(1)	-2(1)
C(39)	23(1)	30(1)	21(1)	0(1)	5(1)	5(1)
C(38)	40(1)	28(1)	27(1)	3(1)	14(1)	6(1)
C(40)	40(1)	32(1)	32(1)	-14(1)	5(1)	-11(1)
O(6)	30(1)	15(1)	38(1)	2(1)	-4(1)	-1(1)
O(7)	21(1)	19(1)	16(1)	-1(1)	0(1)	-2(1)
O(8)	18(1)	21(1)	15(1)	-2(1)	-1(1)	-2(1)
O(9)	23(1)	24(1)	23(1)	6(1)	-1(1)	-8(1)
O(10)	21(1)	24(1)	18(1)	8(1)	-2(1)	-2(1)
O(11)	21(1)	23(1)	22(1)	4(1)	-3(1)	1(1)
O(12)	22(1)	22(1)	34(1)	15(1)	-4(1)	-6(1)
O(3)	18(1)	23(1)	27(1)	-8(1)	6(1)	0(1)
O(4)	20(1)	27(1)	34(1)	-6(1)	9(1)	-6(1)

O(5)	23(1)	14(1)	21(1)	-2(1)	0(1)	-2(1)
N(1)	19(1)	13(1)	18(1)	0(1)	0(1)	-1(1)
N(2)	18(1)	12(1)	16(1)	-1(1)	4(1)	-3(1)
N(3)	14(1)	18(1)	13(1)	2(1)	0(1)	-3(1)
C(1)	32(1)	36(1)	37(1)	11(1)	-10(1)	-14(1)
C(2)	25(1)	20(1)	22(1)	0(1)	0(1)	-10(1)
C(3)	22(1)	16(1)	18(1)	-2(1)	3(1)	-2(1)
C(4)	16(1)	14(1)	15(1)	-1(1)	4(1)	-1(1)
C(5)	14(1)	12(1)	13(1)	0(1)	2(1)	-1(1)
C(6)	14(1)	12(1)	13(1)	0(1)	2(1)	-1(1)
C(7)	17(1)	16(1)	14(1)	0(1)	0(1)	1(1)
C(8)	23(1)	21(1)	21(1)	-7(1)	7(1)	0(1)
C(9)	20(1)	23(1)	17(1)	-5(1)	4(1)	1(1)
C(10)	34(1)	33(1)	42(1)	-4(1)	20(1)	-7(1)
C(11)	29(1)	26(1)	22(1)	2(1)	3(1)	-7(1)
C(12)	17(1)	16(1)	16(1)	-1(1)	2(1)	1(1)
C(13)	15(1)	15(1)	14(1)	1(1)	2(1)	1(1)
C(14)	14(1)	16(1)	14(1)	-1(1)	2(1)	0(1)
C(15)	18(1)	20(1)	16(1)	1(1)	2(1)	1(1)
C(16)	19(1)	29(1)	18(1)	2(1)	-2(1)	4(1)
C(17)	15(1)	33(1)	25(1)	1(1)	-2(1)	-2(1)
C(18)	17(1)	26(1)	20(1)	2(1)	2(1)	-4(1)
C(19)	15(1)	18(1)	13(1)	0(1)	1(1)	0(1)
C(20)	15(1)	14(1)	14(1)	-1(1)	2(1)	0(1)
C(21)	20(1)	18(1)	13(1)	1(1)	2(1)	-2(1)
C(22)	27(1)	21(1)	14(1)	4(1)	0(1)	1(1)
C(23)	26(1)	39(1)	22(1)	9(1)	-2(1)	4(1)
C(24)	29(1)	22(1)	16(1)	2(1)	2(1)	0(1)
C(25)	44(1)	24(1)	20(1)	-1(1)	1(1)	7(1)
C(26)	15(1)	14(1)	14(1)	1(1)	1(1)	-1(1)

C(27)	18(1)	15(1)	16(1)	0(1)	2(1)	0(1)
C(28)	38(1)	31(1)	43(1)	23(1)	-9(1)	-10(1)
C(29)	15(1)	14(1)	14(1)	0(1)	2(1)	-1(1)
C(30)	17(1)	19(1)	15(1)	1(1)	2(1)	0(1)
C(31)	22(1)	36(1)	38(1)	-14(1)	11(1)	1(1)
C(32)	21(1)	28(1)	29(1)	-3(1)	4(1)	5(1)
C(33)	19(1)	43(1)	45(1)	-8(1)	10(1)	-2(1)
C(34)	17(1)	18(1)	18(1)	1(1)	0(1)	0(1)
C(35)	16(1)	24(1)	24(1)	0(1)	-2(1)	2(1)
C(36)	21(1)	22(1)	28(1)	5(1)	-3(1)	5(1)
C(37)	22(1)	16(1)	27(1)	2(1)	0(1)	3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for compound **3a**.

Atom	x	y	z	U(eq)
H(39)	2278	8664	11437	30
H(38)	946	7739	11866	37
H(40A)	2111	4782	7707	52
H(40B)	1240	4205	8104	52
H(40C)	2572	4414	8405	52
H(1A)	-18	6112	8365	53
H(1B)	-424	5281	8099	53
H(1C)	531	5782	7682	53
H(8A)	1393	10359	10516	26
H(8B)	2498	10048	10995	26
H(10)	-1168	7868	11688	43
H(11A)	1638	4888	9494	39
H(11B)	284	4692	9217	39

H(11C)	613	5547	9459	39
H(15)	1592	8970	8357	22
H(16)	-504	8676	8217	27
H(17)	-1434	7801	8930	29
H(18)	-318	7201	9809	25
H(23A)	4938	6109	11246	44
H(23B)	4550	5731	11945	44
H(23C)	4506	6644	11852	44
H(24A)	2316	6646	12184	33
H(24B)	2261	5729	12247	33
H(24C)	1319	6169	11754	33
H(25A)	1994	5318	10826	44
H(25B)	3105	4915	11230	44
H(25C)	3362	5383	10553	44
H(26)	5443	9346	9153	17
H(28A)	4798	10905	7527	56
H(28B)	6166	11152	7742	56
H(28C)	5023	11472	8151	56
H(29)	4031	8609	8098	17
H(31A)	7104	7806	7157	48
H(31B)	6900	6932	7377	48
H(31C)	7666	7502	7858	48
H(32)	-640	9871	10713	31
H(33)	-1964	8938	11142	43
H(34)	6278	8347	9751	21
H(35)	7523	7499	10389	26
H(36)	7082	6193	10404	29
H(37)	5436	5681	9765	26

Table 6. Torsion angles [deg] for compound **3a**.

C(9)-C(39)-C(38)-C(10)	0.3(2)
C(3)-O(1)-C(2)-C(11)	60.54(16)
C(3)-O(1)-C(2)-C(1)	177.88(12)
C(3)-O(1)-C(2)-C(40)	-64.24(16)
C(2)-O(1)-C(3)-O(6)	5.2(2)
C(2)-O(1)-C(3)-N(1)	-172.59(10)
C(4)-N(1)-C(3)-O(6)	172.11(12)
C(12)-N(1)-C(3)-O(6)	-12.99(19)
C(4)-N(1)-C(3)-O(1)	-9.91(17)
C(12)-N(1)-C(3)-O(1)	164.99(11)
C(3)-N(1)-C(4)-O(7)	-21.9(2)
C(12)-N(1)-C(4)-O(7)	162.58(12)
C(3)-N(1)-C(4)-C(5)	159.50(11)
C(12)-N(1)-C(4)-C(5)	-16.02(12)
O(7)-C(4)-C(5)-C(13)	-160.90(12)
N(1)-C(4)-C(5)-C(13)	17.73(11)
O(7)-C(4)-C(5)-C(29)	-35.14(16)
N(1)-C(4)-C(5)-C(29)	143.48(10)
O(7)-C(4)-C(5)-C(6)	78.38(14)
N(1)-C(4)-C(5)-C(6)	-103.00(11)
C(7)-N(2)-C(6)-C(14)	66.91(15)
C(26)-N(2)-C(6)-C(14)	-117.23(11)
C(7)-N(2)-C(6)-C(20)	-50.23(15)
C(26)-N(2)-C(6)-C(20)	125.63(11)
C(7)-N(2)-C(6)-C(5)	-166.76(11)
C(26)-N(2)-C(6)-C(5)	9.09(13)
C(13)-C(5)-C(6)-N(2)	98.63(11)
C(29)-C(5)-C(6)-N(2)	-27.06(11)
C(4)-C(5)-C(6)-N(2)	-147.42(9)

C(13)-C(5)-C(6)-C(14)	-135.02(10)
C(29)-C(5)-C(6)-C(14)	99.30(11)
C(4)-C(5)-C(6)-C(14)	-21.06(13)
C(13)-C(5)-C(6)-C(20)	-21.74(13)
C(29)-C(5)-C(6)-C(20)	-147.43(9)
C(4)-C(5)-C(6)-C(20)	92.22(11)
C(8)-O(2)-C(7)-O(5)	-12.21(19)
C(8)-O(2)-C(7)-N(2)	169.60(11)
C(6)-N(2)-C(7)-O(5)	175.71(12)
C(26)-N(2)-C(7)-O(5)	0.08(19)
C(6)-N(2)-C(7)-O(2)	-6.05(16)
C(26)-N(2)-C(7)-O(2)	178.32(10)
C(7)-O(2)-C(8)-C(9)	-170.81(11)
C(38)-C(39)-C(9)-C(32)	1.3(2)
C(38)-C(39)-C(9)-C(8)	-173.04(13)
O(2)-C(8)-C(9)-C(32)	-104.28(14)
O(2)-C(8)-C(9)-C(39)	69.88(15)
C(39)-C(38)-C(10)-C(33)	-1.9(2)
C(3)-N(1)-C(12)-C(37)	12.4(2)
C(4)-N(1)-C(12)-C(37)	-171.92(12)
C(3)-N(1)-C(12)-C(13)	-168.15(11)
C(4)-N(1)-C(12)-C(13)	7.48(13)
C(37)-C(12)-C(13)-C(34)	1.31(19)
N(1)-C(12)-C(13)-C(34)	-178.15(11)
C(37)-C(12)-C(13)-C(5)	-175.91(11)
N(1)-C(12)-C(13)-C(5)	4.63(13)
C(29)-C(5)-C(13)-C(34)	45.81(17)
C(4)-C(5)-C(13)-C(34)	169.52(12)
C(6)-C(5)-C(13)-C(34)	-71.99(16)
C(29)-C(5)-C(13)-C(12)	-137.33(11)

C(4)-C(5)-C(13)-C(12)	-13.61(12)
C(6)-C(5)-C(13)-C(12)	104.87(11)
N(2)-C(6)-C(14)-C(15)	37.60(17)
C(20)-C(6)-C(14)-C(15)	161.32(12)
C(5)-C(6)-C(14)-C(15)	-81.59(15)
N(2)-C(6)-C(14)-C(19)	-137.99(10)
C(20)-C(6)-C(14)-C(19)	-14.27(12)
C(5)-C(6)-C(14)-C(19)	102.82(11)
C(19)-C(14)-C(15)-C(16)	-1.25(18)
C(6)-C(14)-C(15)-C(16)	-176.42(12)
C(14)-C(15)-C(16)-C(17)	0.3(2)
C(15)-C(16)-C(17)-C(18)	0.4(2)
C(16)-C(17)-C(18)-C(19)	-0.3(2)
C(17)-C(18)-C(19)-C(14)	-0.63(19)
C(17)-C(18)-C(19)-N(3)	173.33(12)
C(15)-C(14)-C(19)-C(18)	1.43(19)
C(6)-C(14)-C(19)-C(18)	177.49(11)
C(15)-C(14)-C(19)-N(3)	-173.60(11)
C(6)-C(14)-C(19)-N(3)	2.45(13)
C(21)-N(3)-C(19)-C(18)	17.6(2)
C(20)-N(3)-C(19)-C(18)	-162.45(13)
C(21)-N(3)-C(19)-C(14)	-167.86(11)
C(20)-N(3)-C(19)-C(14)	12.11(13)
C(21)-N(3)-C(20)-O(8)	-21.0(2)
C(19)-N(3)-C(20)-O(8)	158.98(12)
C(21)-N(3)-C(20)-C(6)	159.14(11)
C(19)-N(3)-C(20)-C(6)	-20.83(12)
N(2)-C(6)-C(20)-O(8)	-33.95(17)
C(14)-C(6)-C(20)-O(8)	-158.91(12)
C(5)-C(6)-C(20)-O(8)	78.89(14)

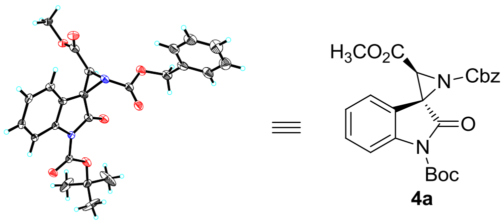
N(2)-C(6)-C(20)-N(3)	145.86(10)
C(14)-C(6)-C(20)-N(3)	20.89(11)
C(5)-C(6)-C(20)-N(3)	-101.30(10)
C(22)-O(10)-C(21)-O(9)	5.6(2)
C(22)-O(10)-C(21)-N(3)	-171.87(10)
C(20)-N(3)-C(21)-O(9)	174.12(12)
C(19)-N(3)-C(21)-O(9)	-5.91(19)
C(20)-N(3)-C(21)-O(10)	-8.25(17)
C(19)-N(3)-C(21)-O(10)	171.72(10)
C(21)-O(10)-C(22)-C(23)	175.32(11)
C(21)-O(10)-C(22)-C(24)	-66.63(14)
C(21)-O(10)-C(22)-C(25)	57.77(15)
C(7)-N(2)-C(26)-C(27)	-53.49(15)
C(6)-N(2)-C(26)-C(27)	130.59(11)
C(7)-N(2)-C(26)-C(29)	-171.27(11)
C(6)-N(2)-C(26)-C(29)	12.80(13)
C(28)-O(12)-C(27)-O(11)	1.1(2)
C(28)-O(12)-C(27)-C(26)	176.69(12)
N(2)-C(26)-C(27)-O(11)	-42.01(16)
C(29)-C(26)-C(27)-O(11)	70.77(15)
N(2)-C(26)-C(27)-O(12)	142.39(11)
C(29)-C(26)-C(27)-O(12)	-104.83(12)
C(13)-C(5)-C(29)-C(30)	36.13(14)
C(4)-C(5)-C(29)-C(30)	-81.87(12)
C(6)-C(5)-C(29)-C(30)	159.90(10)
C(13)-C(5)-C(29)-C(26)	-88.01(11)
C(4)-C(5)-C(29)-C(26)	153.99(10)
C(6)-C(5)-C(29)-C(26)	35.76(11)
N(2)-C(26)-C(29)-C(30)	-154.34(10)
C(27)-C(26)-C(29)-C(30)	86.67(12)

N(2)-C(26)-C(29)-C(5)	-30.24(11)
C(27)-C(26)-C(29)-C(5)	-149.23(10)
C(31)-O(3)-C(30)-O(4)	-2.49(19)
C(31)-O(3)-C(30)-C(29)	177.05(12)
C(5)-C(29)-C(30)-O(4)	-126.32(14)
C(26)-C(29)-C(30)-O(4)	-6.58(18)
C(5)-C(29)-C(30)-O(3)	54.14(13)
C(26)-C(29)-C(30)-O(3)	173.88(10)
C(39)-C(9)-C(32)-C(33)	-1.4(2)
C(8)-C(9)-C(32)-C(33)	172.78(13)
C(38)-C(10)-C(33)-C(32)	1.7(3)
C(9)-C(32)-C(33)-C(10)	-0.1(2)
C(12)-C(13)-C(34)-C(35)	0.01(18)
C(5)-C(13)-C(34)-C(35)	176.57(12)
C(13)-C(34)-C(35)-C(36)	-1.17(19)
C(34)-C(35)-C(36)-C(37)	1.1(2)
C(13)-C(12)-C(37)-C(36)	-1.40(19)
N(1)-C(12)-C(37)-C(36)	177.94(12)
C(35)-C(36)-C(37)-C(12)	0.2(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for compound **3a** [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)



CCDC number: CCDC 980257

Table 1. Crystal data and structure refinement for compound **4a**.

Identification code	compound **4a**
Empirical formula	C24 H24 N2 O7
Formula weight	452.45
Temperature	100(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P -1
Unit cell dimensions	a = 6.647(8) A alpha = 87.147(15) deg.
	b = 12.469(15) A beta = 79.429(16) deg.
	c = 14.173(16) A gamma = 89.310(15) deg.
Volume	1153(2) A^3
Z, Calculated density	2, 1.303 Mg/m^3
Absorption coefficient	0.097 mm^-1
F(000)	476
Crystal size	1.36 x 0.09 x 0.07 mm
Theta range for data collection	1.46 to 27.99 deg.
Limiting indices	-8<=h<=8, -16<=k<=16, -18<=l<=18
Reflections collected / unique	13439 / 5478 [R(int) = 0.1587]
Completeness to theta = 27.99	98.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9933 and 0.8797
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5478 / 0 / 302

Goodness-of-fit on F^2 1.079

Final R indices [I>2sigma(I)] R1 = 0.1679, wR2 = 0.4117

R indices (all data) R1 = 0.2708, wR2 = 0.4605

Largest diff. peak and hole 0.669 and -0.499 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for compound **4a**.

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Atom	x	y	z	U(eq)
O(1)	7631(11)	-2577(4)	8312(4)	51(2)
O(2)	10111(11)	1483(5)	5789(4)	49(2)
O(3)	11866(10)	460(5)	6748(4)	47(2)
O(4)	6603(10)	3344(5)	7970(5)	45(2)
O(5)	3802(9)	2492(4)	7669(4)	36(1)
O(6)	7805(10)	-786(5)	7215(4)	43(2)
O(7)	7132(10)	-2447(4)	9932(4)	41(2)
N(1)	7532(9)	-921(5)	8909(4)	24(1)
C(1)	5361(16)	-4111(9)	8818(13)	98(6)
C(2)	7462(13)	-3766(7)	8333(7)	39(2)
C(3)	7409(10)	-2056(6)	9121(6)	26(2)
C(4)	7728(11)	-404(6)	7991(6)	29(2)
C(5)	7799(12)	809(6)	8145(6)	30(2)
N(2)	9055(10)	1491(5)	7397(5)	32(2)
C(7)	10429(14)	1050(7)	6635(6)	36(2)
C(8)	11654(16)	1208(7)	4951(7)	48(2)
C(9)	13541(17)	1917(7)	4845(7)	48(2)
C(10)	13585(18)	2874(8)	5324(8)	56(3)
C(11)	15329(19)	3500(8)	5190(8)	61(3)
C(12)	17050(20)	3232(10)	4562(9)	71(3)

C(13)	6870(13)	1518(6)	7432(6)	34(2)
C(14)	5785(14)	2556(7)	7723(6)	37(2)
C(15)	2575(14)	3441(6)	7934(7)	41(2)
C(16)	9077(18)	-4288(10)	8763(12)	90(5)
C(17)	7522(10)	-131(6)	9632(5)	25(2)
C(18)	7725(10)	908(5)	9196(5)	24(2)
C(19)	7833(11)	1802(6)	9734(6)	29(2)
C(20)	7700(10)	1636(6)	10727(6)	28(2)
C(21)	7475(11)	600(6)	11150(6)	29(2)
C(22)	7398(10)	-291(6)	10635(5)	25(2)
C(23)	15307(18)	1644(9)	4200(7)	56(3)
C(24)	17040(20)	2287(10)	4041(9)	68(3)
C(6)	7730(40)	-3989(10)	7267(9)	157(11)

Table 3. Bond lengths [A] and angles [deg] for compound **4a**.

O(1)-C(3)	1.330(10)
O(1)-C(2)	1.486(10)
O(2)-C(7)	1.342(10)
O(2)-C(8)	1.472(12)
O(3)-C(7)	1.227(10)
O(4)-C(14)	1.224(10)
O(5)-C(14)	1.338(10)
O(5)-C(15)	1.454(10)
O(6)-C(4)	1.212(10)
O(7)-C(3)	1.209(9)
N(1)-C(4)	1.407(10)
N(1)-C(3)	1.433(10)
N(1)-C(17)	1.455(10)

C(1)-C(2)	1.497(13)
C(1)-H(1A)	0.9800
C(1)-H(1B)	0.9800
C(1)-H(1C)	0.9800
C(2)-C(16)	1.460(14)
C(2)-C(6)	1.527(16)
C(4)-C(5)	1.542(11)
C(5)-N(2)	1.465(10)
C(5)-C(18)	1.492(11)
C(5)-C(13)	1.521(11)
N(2)-C(7)	1.410(11)
N(2)-C(13)	1.444(11)
C(8)-C(9)	1.524(14)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-C(23)	1.399(15)
C(9)-C(10)	1.405(14)
C(10)-C(11)	1.383(15)
C(10)-H(10)	0.9500
C(11)-C(12)	1.364(17)
C(11)-H(11)	0.9500
C(12)-C(24)	1.422(17)
C(12)-H(12)	0.9500
C(13)-C(14)	1.512(12)
C(13)-H(13)	1.0000
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800

C(16)-H(16C)	0.9800
C(17)-C(18)	1.404(10)
C(17)-C(22)	1.413(11)
C(18)-C(19)	1.391(11)
C(19)-C(20)	1.400(11)
C(19)-H(19)	0.9500
C(20)-C(21)	1.395(11)
C(20)-H(20)	0.9500
C(21)-C(22)	1.365(11)
C(21)-H(21)	0.9500
C(22)-H(22)	0.9500
C(23)-C(24)	1.390(16)
C(23)-H(23)	0.9500
C(24)-H(24)	0.9500
C(6)-H(6A)	0.9800
C(6)-H(6B)	0.9800
C(6)-H(6C)	0.9800
C(3)-O(1)-C(2)	121.0(7)
C(7)-O(2)-C(8)	115.1(8)
C(14)-O(5)-C(15)	115.8(6)
C(4)-N(1)-C(3)	126.1(6)
C(4)-N(1)-C(17)	110.1(6)
C(3)-N(1)-C(17)	123.8(6)
C(2)-C(1)-H(1A)	109.5
C(2)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1B)	109.5
C(2)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5
C(16)-C(2)-O(1)	111.4(8)

C(16)-C(2)-C(1)	112.9(11)
O(1)-C(2)-C(1)	110.2(8)
C(16)-C(2)-C(6)	110.8(13)
O(1)-C(2)-C(6)	102.4(8)
C(1)-C(2)-C(6)	108.6(13)
O(7)-C(3)-O(1)	127.0(7)
O(7)-C(3)-N(1)	122.8(7)
O(1)-C(3)-N(1)	110.2(7)
O(6)-C(4)-N(1)	129.6(7)
O(6)-C(4)-C(5)	124.3(7)
N(1)-C(4)-C(5)	106.1(6)
N(2)-C(5)-C(18)	123.9(6)
N(2)-C(5)-C(13)	57.8(5)
C(18)-C(5)-C(13)	130.3(7)
N(2)-C(5)-C(4)	118.1(7)
C(18)-C(5)-C(4)	106.0(6)
C(13)-C(5)-C(4)	114.6(6)
C(7)-N(2)-C(13)	122.6(7)
C(7)-N(2)-C(5)	121.7(7)
C(13)-N(2)-C(5)	63.0(5)
O(3)-C(7)-O(2)	125.2(8)
O(3)-C(7)-N(2)	123.8(8)
O(2)-C(7)-N(2)	110.4(8)
O(2)-C(8)-C(9)	111.3(7)
O(2)-C(8)-H(8A)	109.4
C(9)-C(8)-H(8A)	109.4
O(2)-C(8)-H(8B)	109.4
C(9)-C(8)-H(8B)	109.4
H(8A)-C(8)-H(8B)	108.0
C(23)-C(9)-C(10)	117.3(9)

C(23)-C(9)-C(8)	119.0(9)
C(10)-C(9)-C(8)	123.6(10)
C(11)-C(10)-C(9)	121.3(11)
C(11)-C(10)-H(10)	119.3
C(9)-C(10)-H(10)	119.3
C(12)-C(11)-C(10)	121.4(10)
C(12)-C(11)-H(11)	119.3
C(10)-C(11)-H(11)	119.3
C(11)-C(12)-C(24)	118.8(11)
C(11)-C(12)-H(12)	120.6
C(24)-C(12)-H(12)	120.6
N(2)-C(13)-C(14)	116.6(6)
N(2)-C(13)-C(5)	59.2(5)
C(14)-C(13)-C(5)	121.3(7)
N(2)-C(13)-H(13)	115.9
C(14)-C(13)-H(13)	115.9
C(5)-C(13)-H(13)	115.9
O(4)-C(14)-O(5)	124.8(8)
O(4)-C(14)-C(13)	125.0(8)
O(5)-C(14)-C(13)	110.2(6)
O(5)-C(15)-H(15A)	109.5
O(5)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
O(5)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(2)-C(16)-H(16A)	109.5
C(2)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(2)-C(16)-H(16C)	109.5

H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(18)-C(17)-C(22)	120.5(7)
C(18)-C(17)-N(1)	110.2(6)
C(22)-C(17)-N(1)	129.3(6)
C(19)-C(18)-C(17)	121.2(7)
C(19)-C(18)-C(5)	131.2(7)
C(17)-C(18)-C(5)	107.6(6)
C(18)-C(19)-C(20)	117.9(7)
C(18)-C(19)-H(19)	121.0
C(20)-C(19)-H(19)	121.0
C(21)-C(20)-C(19)	120.2(7)
C(21)-C(20)-H(20)	119.9
C(19)-C(20)-H(20)	119.9
C(22)-C(21)-C(20)	122.9(7)
C(22)-C(21)-H(21)	118.5
C(20)-C(21)-H(21)	118.5
C(21)-C(22)-C(17)	117.3(7)
C(21)-C(22)-H(22)	121.4
C(17)-C(22)-H(22)	121.4
C(24)-C(23)-C(9)	121.5(10)
C(24)-C(23)-H(23)	119.2
C(9)-C(23)-H(23)	119.2
C(23)-C(24)-C(12)	119.6(12)
C(23)-C(24)-H(24)	120.2
C(12)-C(24)-H(24)	120.2
C(2)-C(6)-H(6A)	109.5
C(2)-C(6)-H(6B)	109.5
H(6A)-C(6)-H(6B)	109.5
C(2)-C(6)-H(6C)	109.5

H(6A)-C(6)-H(6C)	109.5
H(6B)-C(6)-H(6C)	109.5

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for compound **4a**.

The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

Atom	U11	U22	U33	U23	U13	U12
O(1)	95(5)	18(3)	35(4)	-7(2)	2(3)	-17(3)
O(2)	71(5)	46(4)	29(3)	-2(3)	-9(3)	-5(3)
O(3)	54(4)	41(4)	43(4)	3(3)	0(3)	-8(3)
O(4)	52(4)	33(3)	57(4)	-5(3)	-25(3)	-6(3)
O(5)	46(3)	26(3)	41(3)	-8(2)	-17(3)	-5(2)
O(6)	64(4)	30(3)	39(4)	-5(3)	-15(3)	-12(3)
O(7)	60(4)	28(3)	33(3)	0(3)	-8(3)	-7(3)
N(1)	22(3)	18(3)	31(3)	-3(3)	-3(2)	-9(2)
C(1)	30(5)	50(7)	206(17)	-49(9)	11(7)	-13(5)
C(2)	34(4)	28(4)	54(6)	-7(4)	0(4)	-3(3)
C(3)	12(3)	28(4)	38(5)	-5(3)	-4(3)	-8(3)
C(4)	29(4)	24(4)	37(5)	-4(3)	-10(3)	-16(3)
C(5)	37(4)	24(4)	32(4)	2(3)	-12(3)	-9(3)
N(2)	43(4)	29(3)	27(4)	0(3)	-10(3)	-11(3)
C(7)	48(5)	31(4)	28(4)	-2(4)	-6(4)	-9(4)
C(8)	70(7)	33(5)	40(5)	-9(4)	-9(5)	-11(4)
C(9)	74(7)	34(5)	38(5)	3(4)	-13(5)	-8(5)
C(10)	69(7)	49(6)	46(6)	6(5)	-6(5)	-2(5)
C(11)	91(9)	42(6)	55(7)	-9(5)	-18(6)	-17(6)
C(12)	86(9)	70(8)	60(8)	-12(6)	-16(7)	-20(7)

C(13)	47(5)	24(4)	34(5)	-8(3)	-18(4)	-9(3)
C(14)	49(5)	33(5)	34(5)	1(4)	-17(4)	-18(4)
C(15)	51(5)	21(4)	50(6)	-11(4)	-4(4)	-4(4)
C(16)	61(7)	59(7)	177(15)	-70(9)	-75(9)	37(6)
C(17)	9(3)	28(4)	36(4)	-2(3)	-2(3)	-5(3)
C(18)	20(3)	20(3)	35(4)	-2(3)	-10(3)	-10(3)
C(19)	19(4)	24(4)	45(5)	-8(3)	-10(3)	-8(3)
C(20)	17(3)	34(4)	33(4)	-15(3)	-6(3)	-2(3)
C(21)	18(3)	39(5)	31(4)	-6(3)	-4(3)	-2(3)
C(22)	15(3)	24(4)	33(4)	1(3)	0(3)	-6(3)
C(23)	81(8)	53(6)	35(5)	-2(5)	-10(5)	-13(6)
C(24)	77(8)	72(8)	55(7)	0(6)	-13(6)	0(6)
C(6)	380(30)	38(7)	38(7)	-4(6)	0(12)	-53(12)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for compound **4a**.

Atom	x	y	z	U(eq)
H(1A)	5088	-3869	9477	147
H(1B)	5270	-4896	8832	147
H(1C)	4348	-3794	8463	147
H(8A)	11052	1296	4362	57
H(8B)	12061	446	5023	57
H(10)	12393	3096	5748	67
H(11)	15326	4128	5542	74
H(12)	18238	3670	4474	85
H(13)	6368	1139	6915	40
H(15A)	2601	3586	8605	61
H(15B)	1160	3317	7859	61
H(15C)	3136	4057	7517	61
H(16A)	10388	-3937	8505	136
H(16B)	9164	-5047	8611	136
H(16C)	8763	-4231	9462	136
H(19)	7992	2503	9436	34
H(20)	7764	2231	11114	33
H(21)	7370	509	11827	35
H(22)	7265	-990	10939	30
H(23)	15321	1004	3862	68
H(24)	18212	2097	3588	82
H(6A)	9052	-3709	6930	236
H(6B)	6625	-3636	6994	236
H(6C)	7679	-4765	7194	236

Table 6. Torsion angles [deg] for compound **4a**.

C(3)-O(1)-C(2)-C(16)	-63.8(12)
C(3)-O(1)-C(2)-C(1)	62.3(12)
C(3)-O(1)-C(2)-C(6)	177.7(12)
C(2)-O(1)-C(3)-O(7)	2.8(12)
C(2)-O(1)-C(3)-N(1)	-176.9(7)
C(4)-N(1)-C(3)-O(7)	-176.0(7)
C(17)-N(1)-C(3)-O(7)	5.7(10)
C(4)-N(1)-C(3)-O(1)	3.8(10)
C(17)-N(1)-C(3)-O(1)	-174.6(6)
C(3)-N(1)-C(4)-O(6)	1.0(13)
C(17)-N(1)-C(4)-O(6)	179.6(8)
C(3)-N(1)-C(4)-C(5)	-179.9(6)
C(17)-N(1)-C(4)-C(5)	-1.4(8)
O(6)-C(4)-C(5)-N(2)	-34.2(11)
N(1)-C(4)-C(5)-N(2)	146.7(6)
O(6)-C(4)-C(5)-C(18)	-178.4(7)
N(1)-C(4)-C(5)-C(18)	2.5(8)
O(6)-C(4)-C(5)-C(13)	31.0(11)
N(1)-C(4)-C(5)-C(13)	-148.1(7)
C(18)-C(5)-N(2)-C(7)	126.9(8)
C(13)-C(5)-N(2)-C(7)	-113.3(8)
C(4)-C(5)-N(2)-C(7)	-10.5(10)
C(18)-C(5)-N(2)-C(13)	-119.8(9)
C(4)-C(5)-N(2)-C(13)	102.8(7)
C(8)-O(2)-C(7)-O(3)	-0.2(12)
C(8)-O(2)-C(7)-N(2)	171.1(6)
C(13)-N(2)-C(7)-O(3)	-136.2(8)
C(5)-N(2)-C(7)-O(3)	-60.0(11)

C(13)-N(2)-C(7)-O(2)	52.3(9)
C(5)-N(2)-C(7)-O(2)	128.5(7)
C(7)-O(2)-C(8)-C(9)	-81.8(9)
O(2)-C(8)-C(9)-C(23)	168.4(9)
O(2)-C(8)-C(9)-C(10)	-15.2(13)
C(23)-C(9)-C(10)-C(11)	-2.4(15)
C(8)-C(9)-C(10)-C(11)	-178.9(10)
C(9)-C(10)-C(11)-C(12)	2.4(17)
C(10)-C(11)-C(12)-C(24)	-0.3(18)
C(7)-N(2)-C(13)-C(14)	-135.8(8)
C(5)-N(2)-C(13)-C(14)	112.3(8)
C(7)-N(2)-C(13)-C(5)	112.0(8)
C(18)-C(5)-C(13)-N(2)	109.3(8)
C(4)-C(5)-C(13)-N(2)	-108.9(7)
N(2)-C(5)-C(13)-C(14)	-104.2(8)
C(18)-C(5)-C(13)-C(14)	5.1(12)
C(4)-C(5)-C(13)-C(14)	146.9(7)
C(15)-O(5)-C(14)-O(4)	-0.4(12)
C(15)-O(5)-C(14)-C(13)	179.8(7)
N(2)-C(13)-C(14)-O(4)	-3.5(12)
C(5)-C(13)-C(14)-O(4)	65.0(11)
N(2)-C(13)-C(14)-O(5)	176.3(7)
C(5)-C(13)-C(14)-O(5)	-115.2(8)
C(4)-N(1)-C(17)-C(18)	-0.4(8)
C(3)-N(1)-C(17)-C(18)	178.2(6)
C(4)-N(1)-C(17)-C(22)	-178.6(7)
C(3)-N(1)-C(17)-C(22)	0.0(10)
C(22)-C(17)-C(18)-C(19)	0.8(10)
N(1)-C(17)-C(18)-C(19)	-177.6(6)
C(22)-C(17)-C(18)-C(5)	-179.6(6)

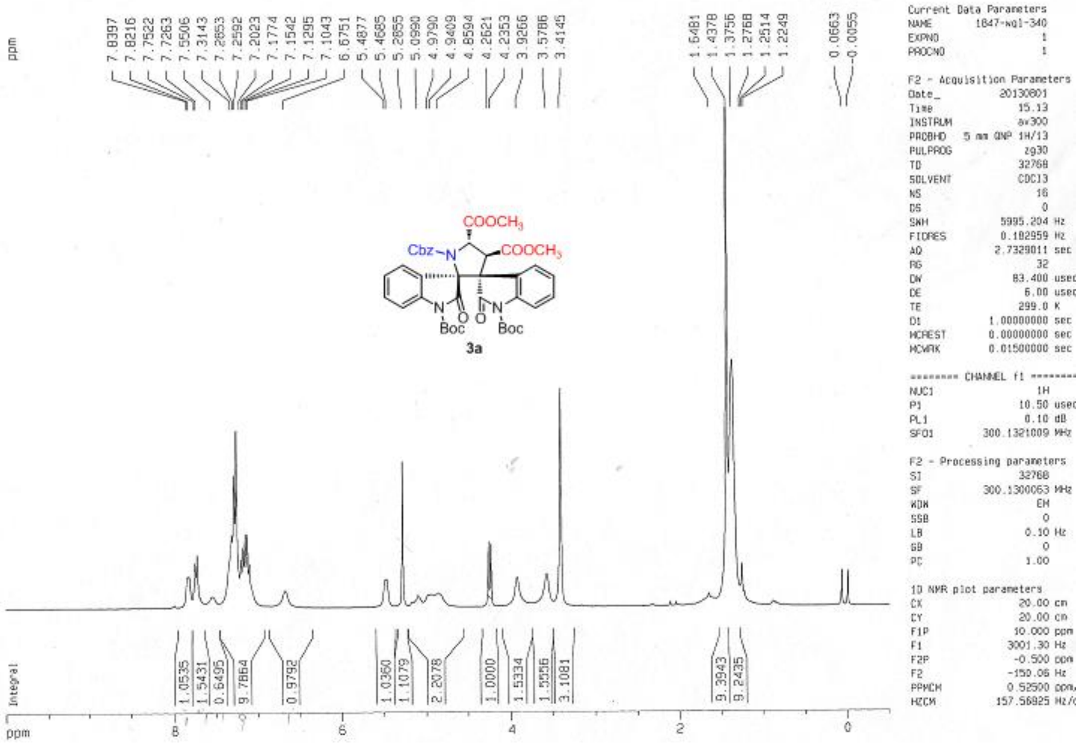
N(1)-C(17)-C(18)-C(5)	2.0(8)
N(2)-C(5)-C(18)-C(19)	35.3(12)
C(13)-C(5)-C(18)-C(19)	-39.0(13)
C(4)-C(5)-C(18)-C(19)	176.8(7)
N(2)-C(5)-C(18)-C(17)	-144.3(7)
C(13)-C(5)-C(18)-C(17)	141.5(8)
C(4)-C(5)-C(18)-C(17)	-2.8(8)
C(17)-C(18)-C(19)-C(20)	-1.0(10)
C(5)-C(18)-C(19)-C(20)	179.5(7)
C(18)-C(19)-C(20)-C(21)	0.2(10)
C(19)-C(20)-C(21)-C(22)	0.8(11)
C(20)-C(21)-C(22)-C(17)	-1.0(10)
C(18)-C(17)-C(22)-C(21)	0.2(10)
N(1)-C(17)-C(22)-C(21)	178.3(6)
C(10)-C(9)-C(23)-C(24)	0.4(15)
C(8)-C(9)-C(23)-C(24)	177.0(9)
C(9)-C(23)-C(24)-C(12)	1.6(17)
C(11)-C(12)-C(24)-C(23)	-1.7(18)

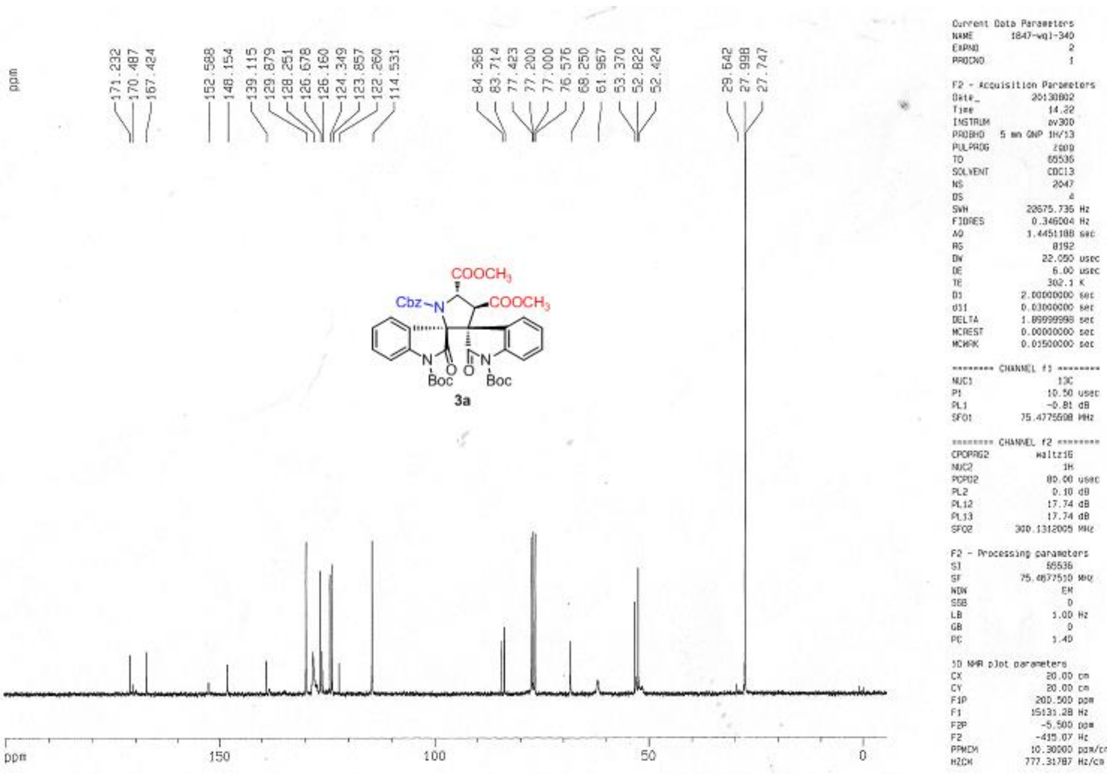
Symmetry transformations used to generate equivalent atoms:

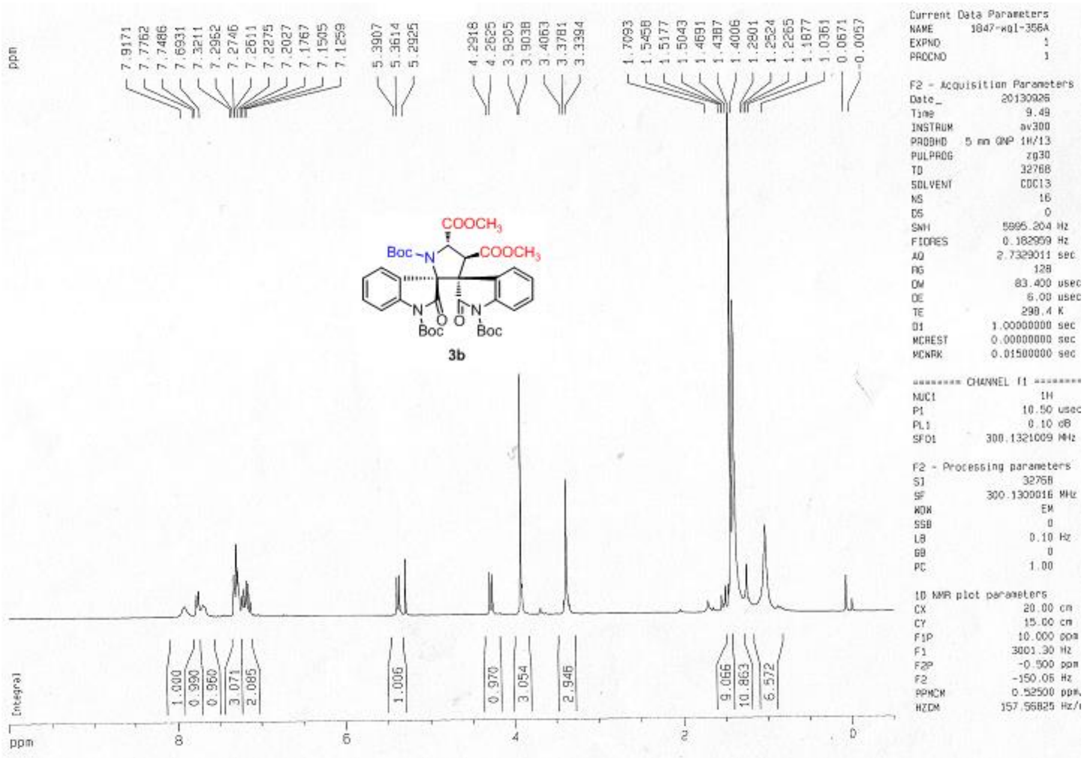
Table 7. Hydrogen bonds for wlx_wql_1 [A and deg.].

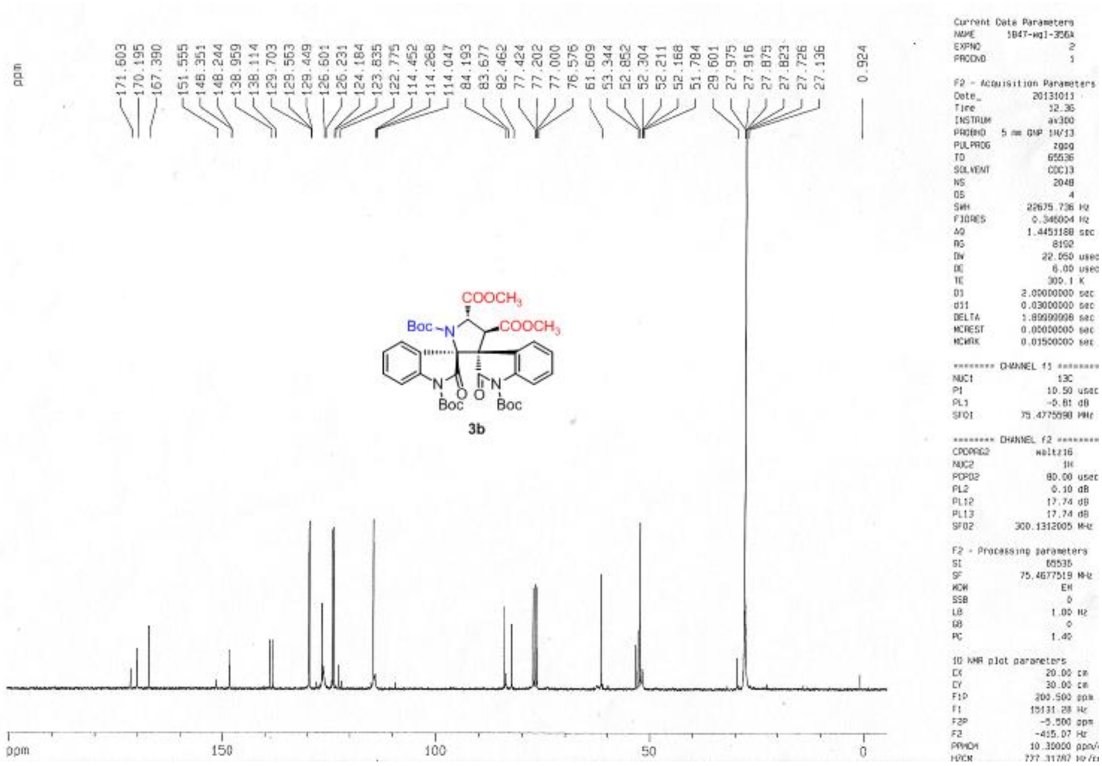
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

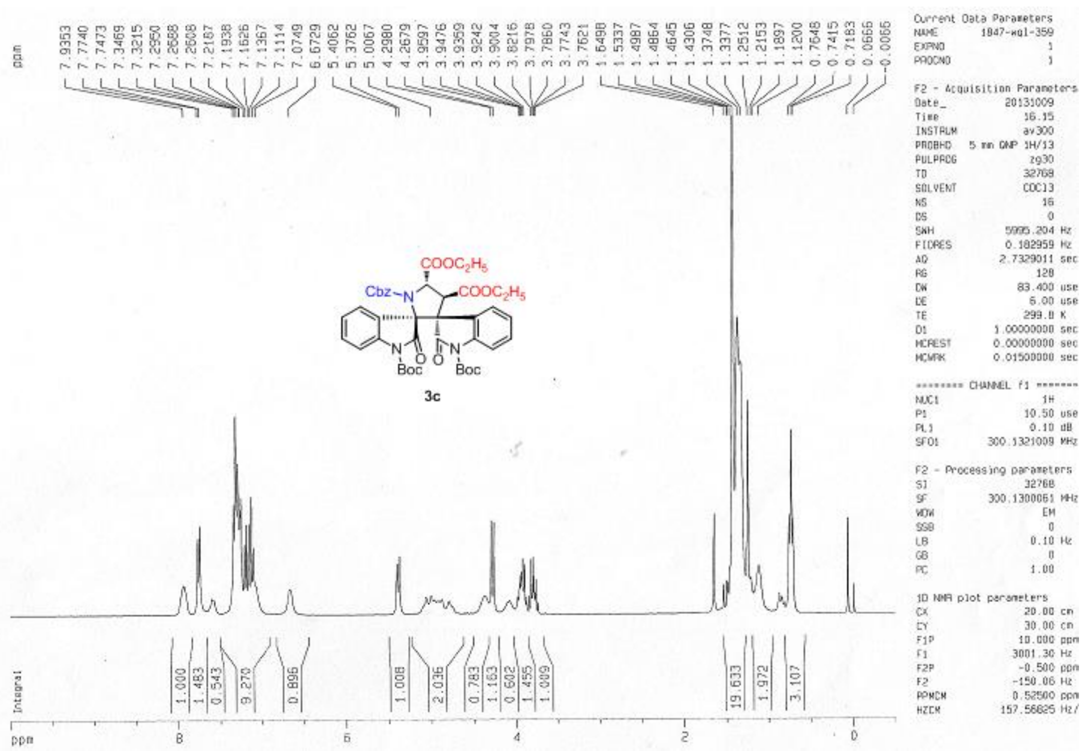
9. Selected ^{1}H NMR and ^{13}C NMR spectra







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7.7740
7.7473
7.3469
7.3215
7.2950
7.2688
7.2608
7.2187
7.1938
7.1626
7.1367
7.1114
7.0749
6.6729
5.4062
5.3762
5.0067
4.2980
4.2679
3.9597
3.9476
3.9359
3.9242
3.9004
3.8216
3.7978
3.7860
3.7743
3.7621
1.6498
1.5337
1.4987
1.4864
1.4645
1.4306
1.3748
1.3377
1.2512
1.2153
1.1897
1.1200
0.7648
0.7415
0.7183
0.0666
-0.0066
Cbz
COOC2H5
COOC2H5
N
N
N
O
O
Boc
Boc
3c
Integral
1.000
1.483
0.543
9.270
0.896
1.008
2.036
0.783
1.163
0.602
1.455
1.089
19.633
1.972
3.107
ppm
8
6
4
2
0
Current Data Parameters
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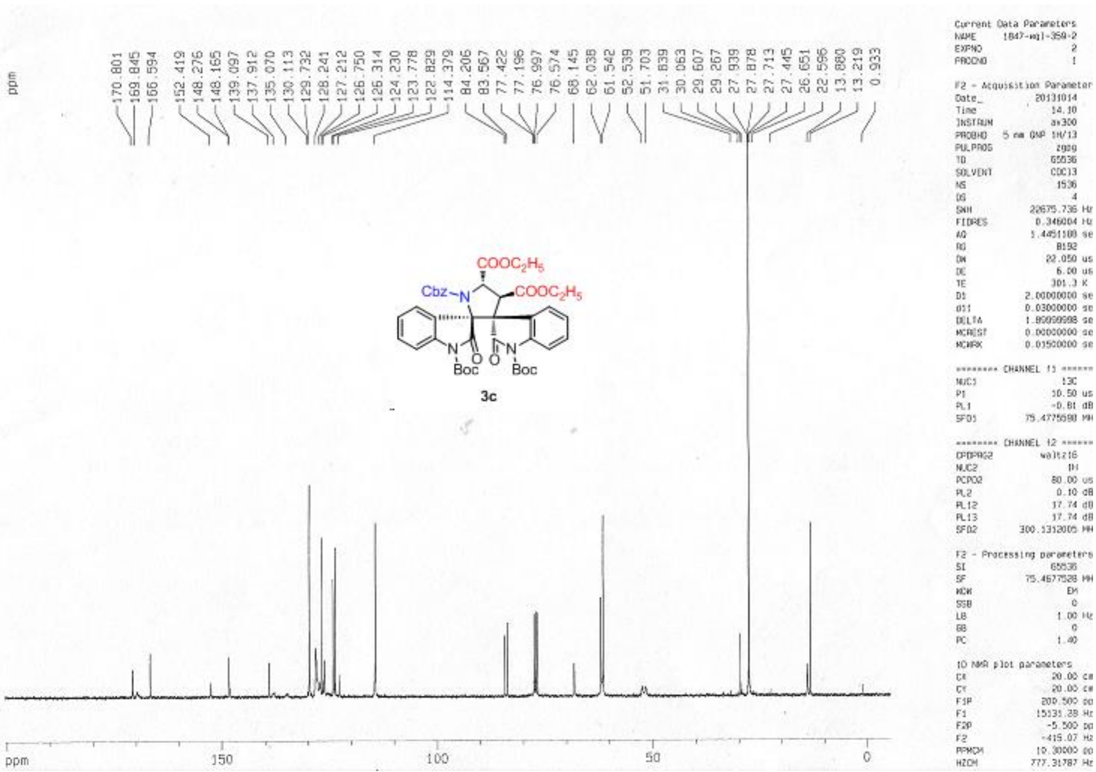
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131009
Time 16.15
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 use
DE 6.00 use
TE 299.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 use
PL1 0.10 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 30.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm
HZCM 157.56825 Hz/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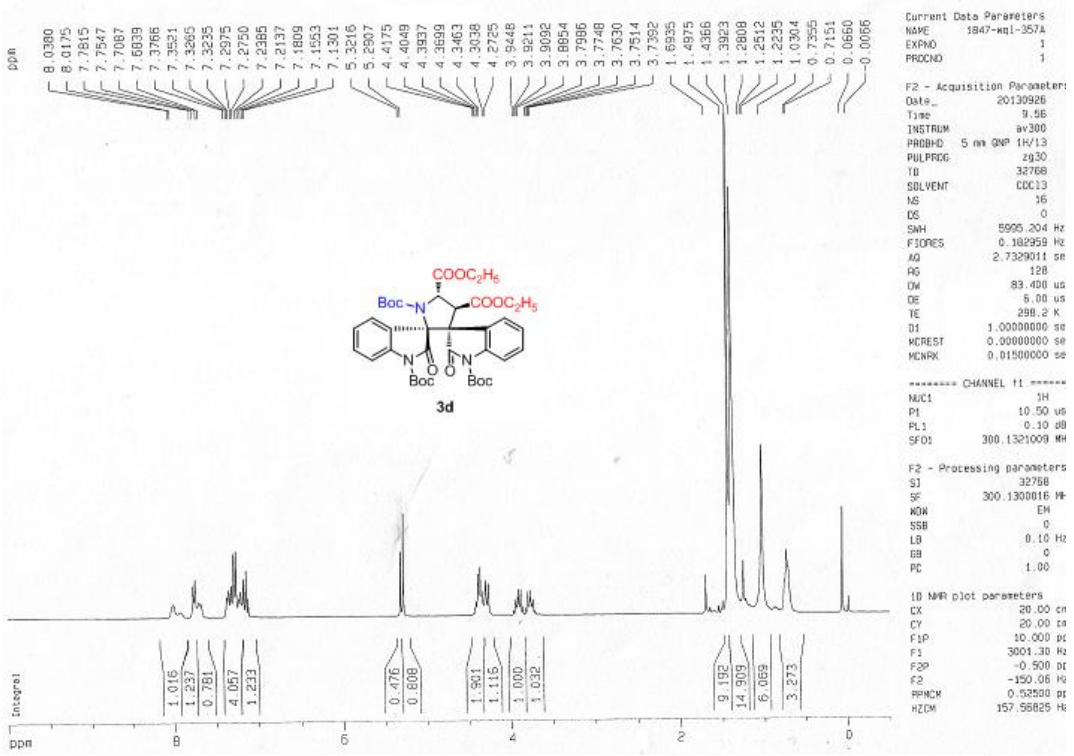
169.845
166.594
152.419
148.276
148.165
139.097
137.912
135.070
130.113
129.732
128.241
127.212
126.750
126.314
124.230
123.778
122.829
114.379
84.206
83.567
77.422
77.196
76.997
76.574
68.145
62.038
61.542
52.539
51.703
31.839
30.063
29.607
29.267
27.939
27.878
27.713
27.445
26.651
22.596
13.880
13.219
0.933
Cbz
COOC2H5
COOC2H5
N
N
N
O
O
Boc
Boc
3c
ppm
150
100
50
0
Current Data Parameters
NAME 1847-wql-359-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameter
Date_ 20131014
Time 14.10
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1536
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451100 se
RG 8192
DW 22.050 us
DE 6.00 us
TE 301.3 K
D1 2.00000000 se
d11 0.03000000 se
DELTA 1.89999998 se
MCREST 0.00000000 se
MCWRK 0.01500000 se
======== CHANNEL f1 ======
NUC1 13C
P1 10.50 us
PL1 -0.81 dB
SFO1 75.4775598 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MH
F2 - Processing parameters
SI 65536
SF 75.4677528 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 20.00 cm
F1P 200.500 pp
F1 15131.28 Hz
F2P -5.500 pp
F2 -415.07 Hz
PPMCM 10.30000 pp
HZCM 777.31787 Hz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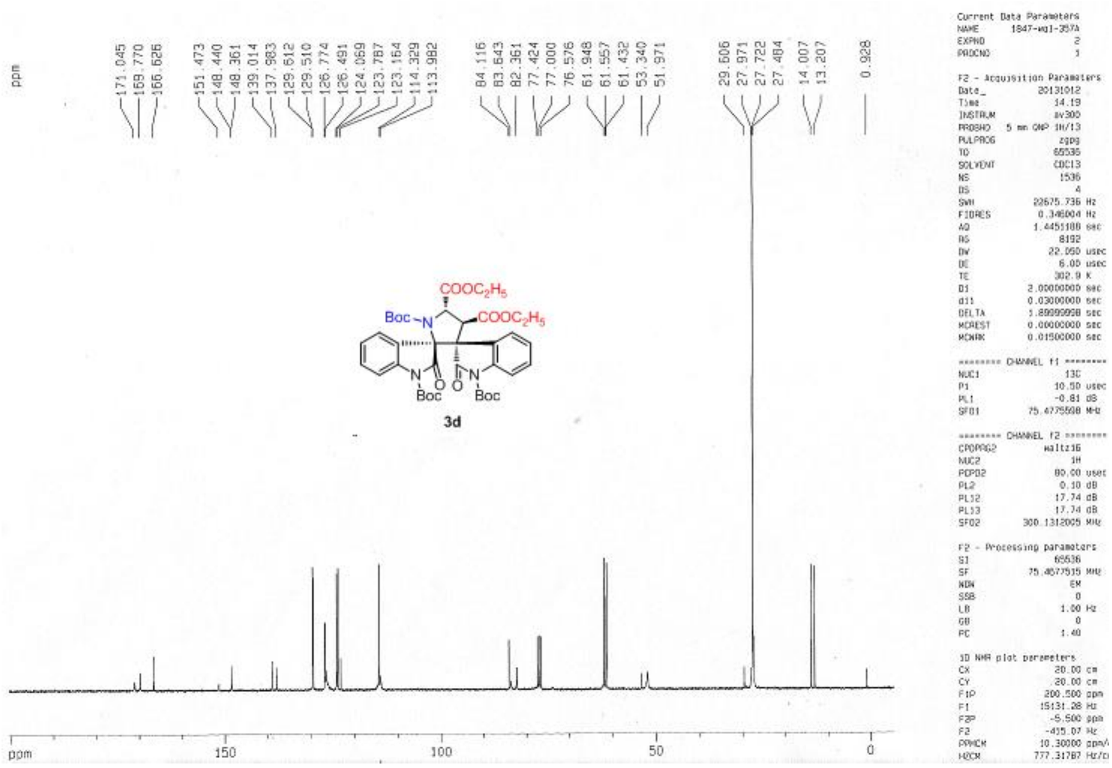
8.0380 8.0175 7.7815 7.7547 7.7087 7.6839 7.3766 7.3521 7.3265 7.3235 7.2975 7.2750 7.2385 7.2137 7.1809 7.1553 7.1301 5.3216 5.2907 4.4175 4.4049 4.3937 4.3699 4.3463 4.3038 4.2725 3.9448 3.9211 3.9092 3.8854 3.7986 3.7748 3.7630 3.7514 3.7392 1.6935 1.4975 1.4366 1.3923 1.2808 1.2512 1.2235 1.0304 0.7355 0.7151 0.0660 -0.0066
COOC2H5
Boc
N
COOC2H5
N
Boc
N
Boc
3d
Integral
1.016 1.237 0.781 4.057 1.233 0.476 0.808 1.901 1.115 1.000 1.032 9.192 14.909 6.069 3.273
8
6
4
2
0
Current Data Parameters
NAME 1847-wql-357A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130926
INSTRUM av300
PULPROG zg30
SOLVENT CDCl3
NUC1 1H
SFO1 300.1321009 MH


ppm
171.045 169.770 166.626 151.473 148.440 148.361 139.014 137.983 129.612 129.510 126.774 126.491 124.069 123.787 123.164 114.329 113.982 84.116 83.643 82.361 77.424 77.000 76.576 61.948 61.557 61.432 53.340 51.971 29.506 27.971 27.722 27.484 14.007 13.207 0.928
COOC2H5
Boc
N
COOC2H5
N
Boc
N
Boc
3d
150
100
50
0
Current Data Parameters
NAME 1847-wql-357A
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131012
INSTRUM av300
PULPROG zgpg
SOLVENT CDCl3
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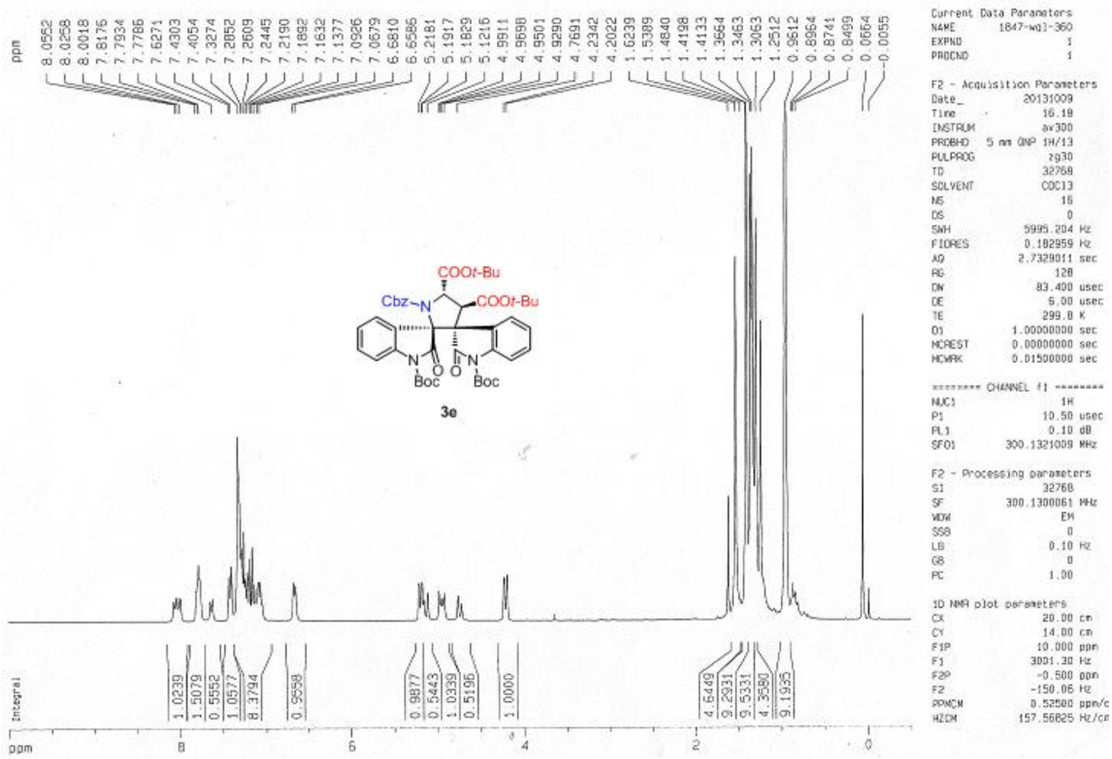
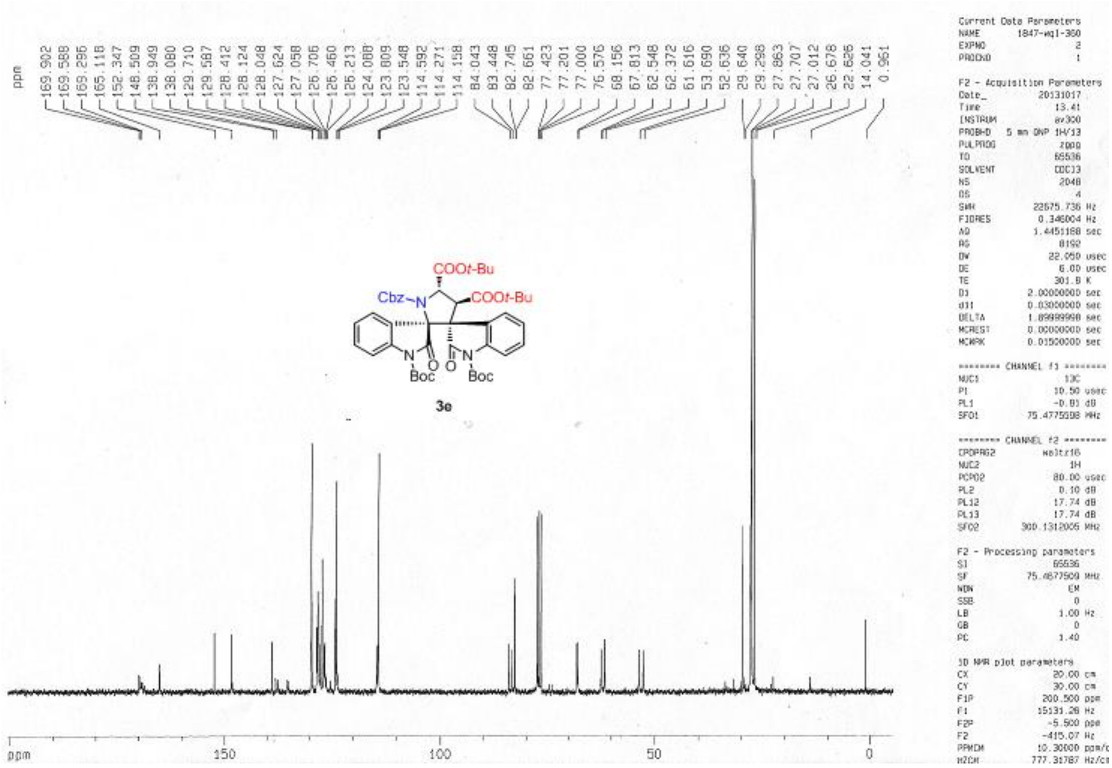
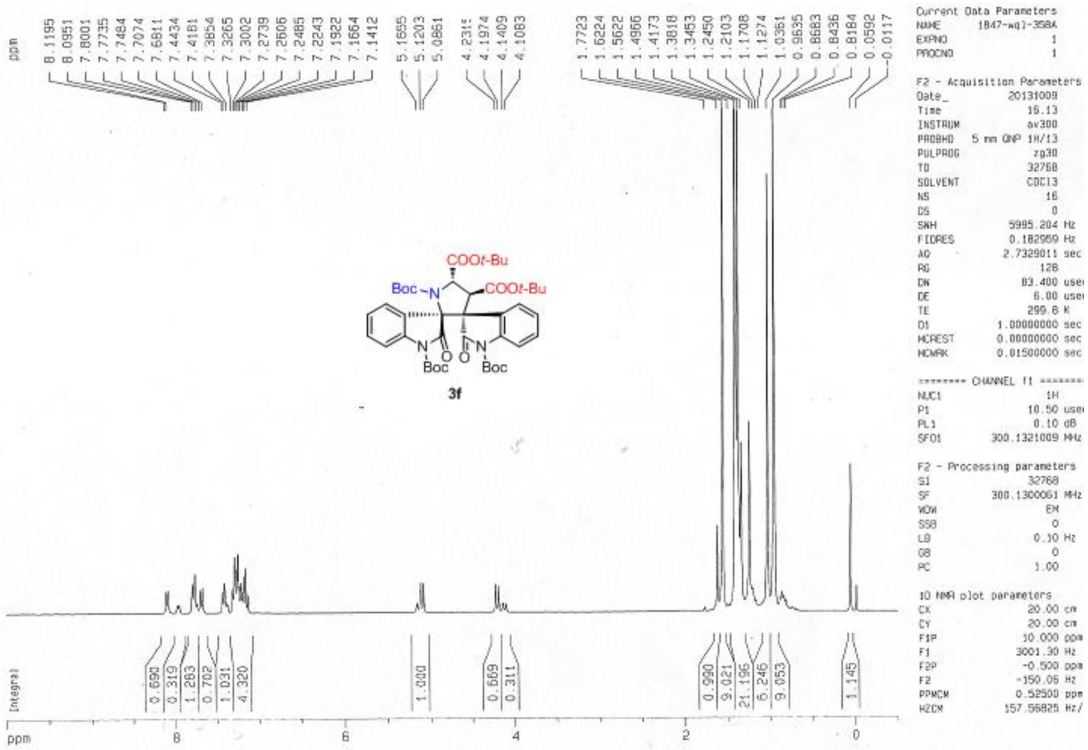
SFO1 75.4775598 MHz

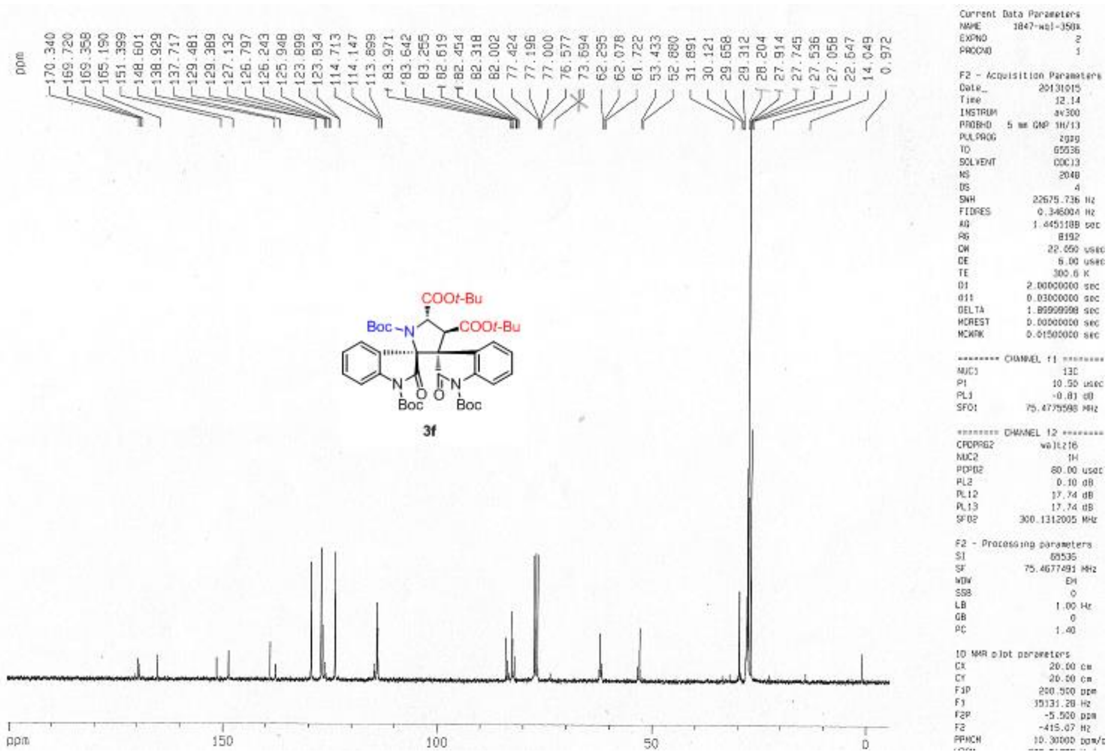


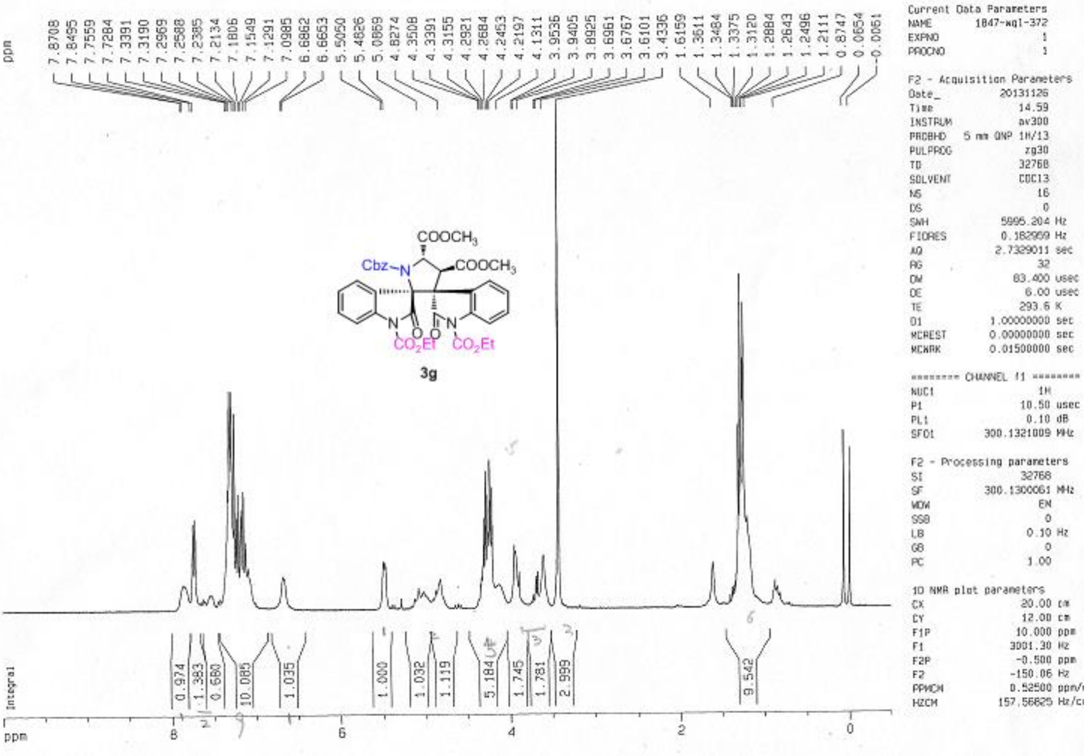
ppm
8.0552 8.0258 8.0018 7.8176 7.7934 7.7786 7.6271 7.4303 7.4054 7.3274 7.2852 7.2609 7.2445 7.2190 7.1892 7.1632 7.1377 7.0926 7.0679 6.6810 6.6586 5.2181 5.1917 5.1829 5.1216 4.9911 4.9698 4.9501 4.9290 4.7691 4.2342 4.2022 1.6239 1.5389 1.4840 1.4198 1.4133 1.3664 1.3463 1.3063 1.2512 0.9612 0.8964 0.8741 0.8499 0.0664 -0.0055
COOt-Bu
Cbz
N
COOt-Bu
N
Boc
O
O
N
Boc
3e
Integral
1.0239 1.5079 0.5552 1.0577 8.3794 0.9558 0.9877 0.5443 1.0339 0.5196 1.0000 4.6449 9.2931 9.5331 4.3580 9.1935
ppm
8 6 4 2 0
Current Data Parameters
NAME 1847-wql-360
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131009
Time 16.18
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 299.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 14.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/c
HZCM 157.56825 Hz/cm



ppm
169.902 169.588 169.295 165.118 152.347 148.509 138.949 138.080 129.710 129.587 128.412 128.124 128.048 127.624 127.058 126.706 126.460 126.213 124.080 123.809 123.548 114.592 114.271 114.158 84.043 83.448 82.745 82.661 77.423 77.201 77.000 76.576 68.156 67.813 62.548 62.372 61.616 53.690 52.636 28.640 28.298 27.863 27.707 27.012 26.678 22.626 14.041 0.961
COOt-Bu
Cbz
N
COOt-Bu
N
Boc
O
O
N
Boc
3e
ppm
150 100 50 0
Current Data Parameters
NAME 1847-wql-360
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131017
Time 13.41
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 301.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677509 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 30.00 cm
F1P 200.500 ppm
F1 15131.28 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm/c
HZCM 777.31787 Hz/cm



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Current Data Parameters
NAME 1847-wql-372
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131126
Time 14.59
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 32
DW 83.400 usec
DE 6.00 usec
TE 293.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm
COOCH3
Cbz
N
COOCH3
N
O
O
N
CO2Et
CO2Et
3g
Integral
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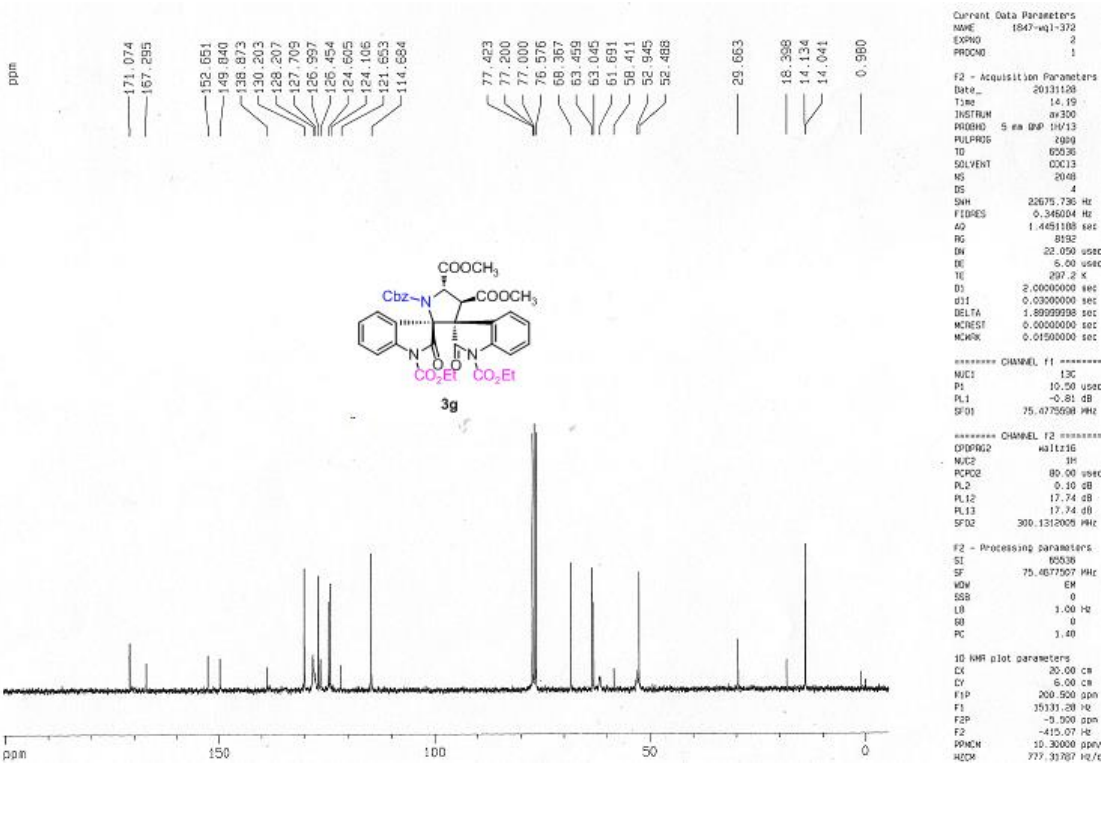
ppm
8 6 4 2 0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Current Data Parameters
NAME 1847-wql-372
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131128
Time 14.19
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 297.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677507 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 200.500 ppm
F1 15131.28 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm/cm
HZCM 777.31787 Hz/cm
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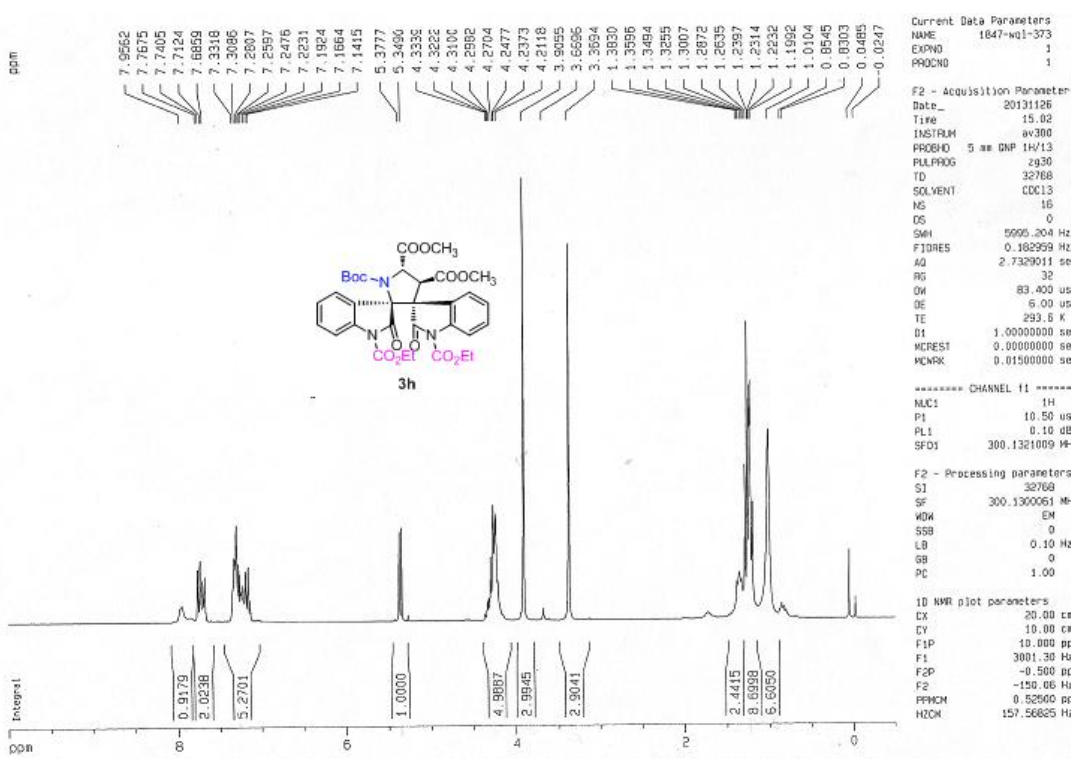
Cbz
N
COOCH3
N
O
O
N
CO2Et
CO2Et
3g
ppm
150 100 50 0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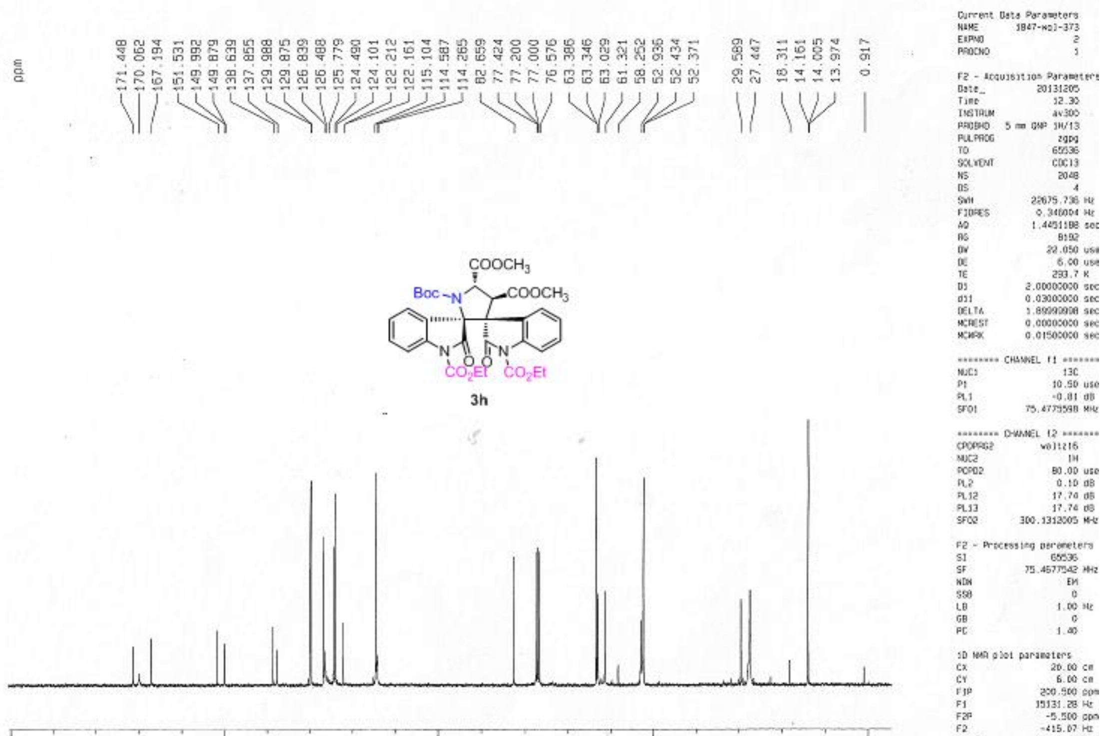
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20131126
Time 15.02
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 se
RG 32
DW 83.400 us
DE 6.00 us
TE 293.6 K
D1 1.00000000 se
MCREST 0.00000000 se
MCWRK 0.01500000 se
======== CHANNEL f1 ======
NUC1 1H
P1 10.50 us
PL1 0.10 dB
SFO1 300.1321009 MH
F2 - Processing parameters
SI 32768
SF 300.1300061 MH
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 pp
F1 3001.30 Hz
F2P -0.500 pp
F2 -150.06 Hz
PPMCM 0.52500 pp
HZCM 157.56825 Hz
COOCH3
Boc
N
COOCH3
N
N
O
O
CO2Et
CO2Et
3h
Integral
0.9179
2.0238
5.2701
1.0000
4.9887
2.9945
2.9041
2.4415
8.6998
6.6050
ppm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PROCNO 1
F2 - Acquisition Parameters
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Time 12.30
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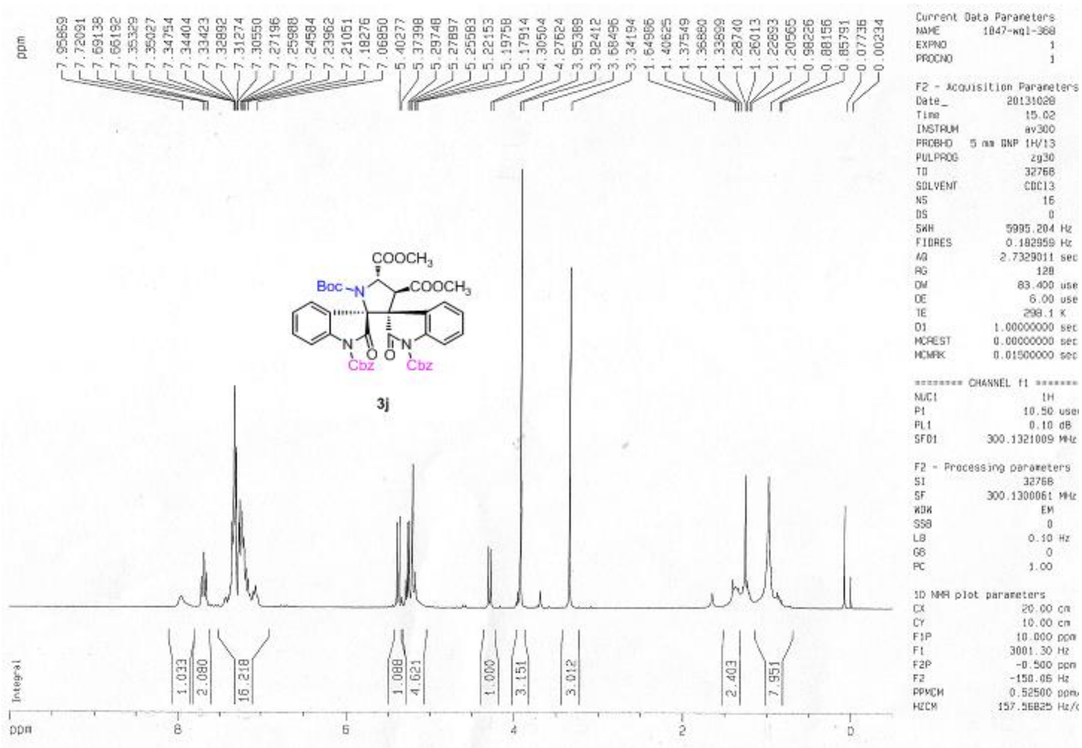
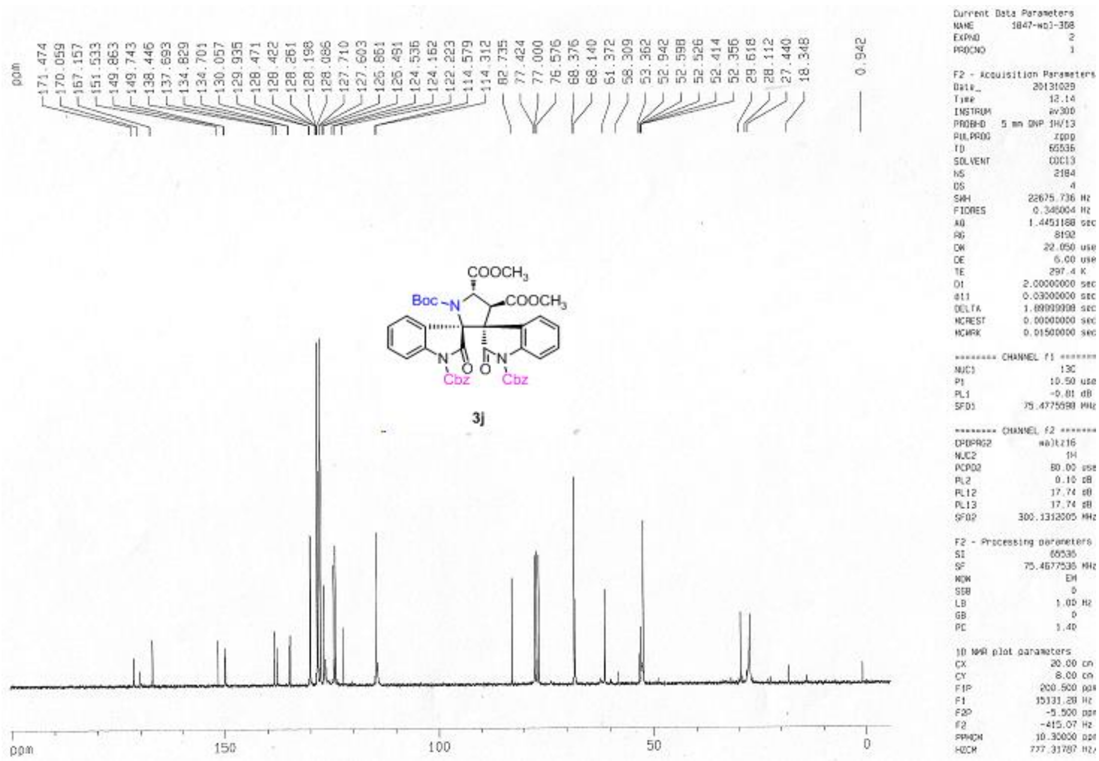
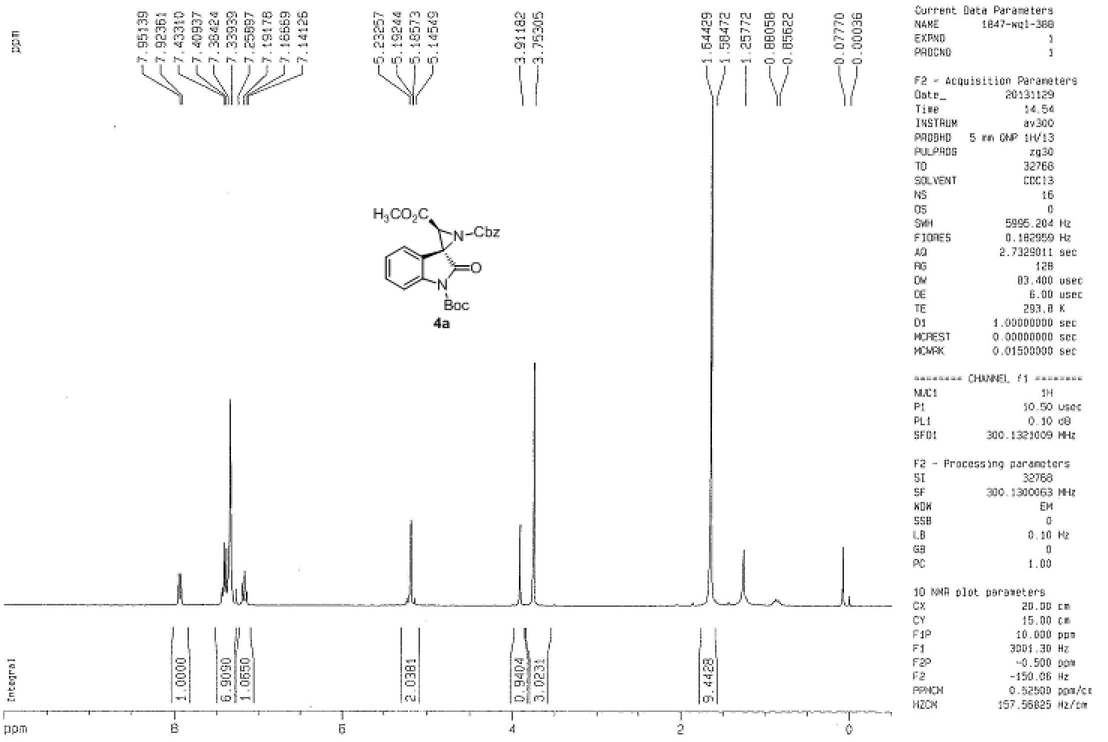
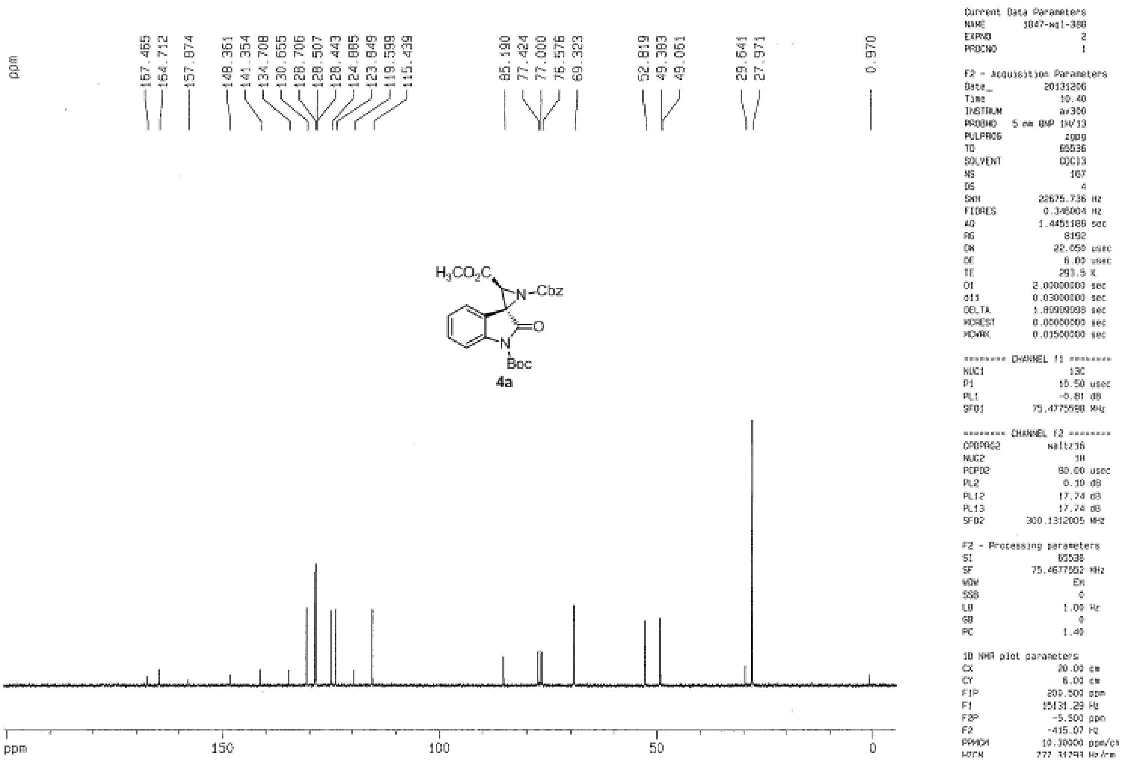
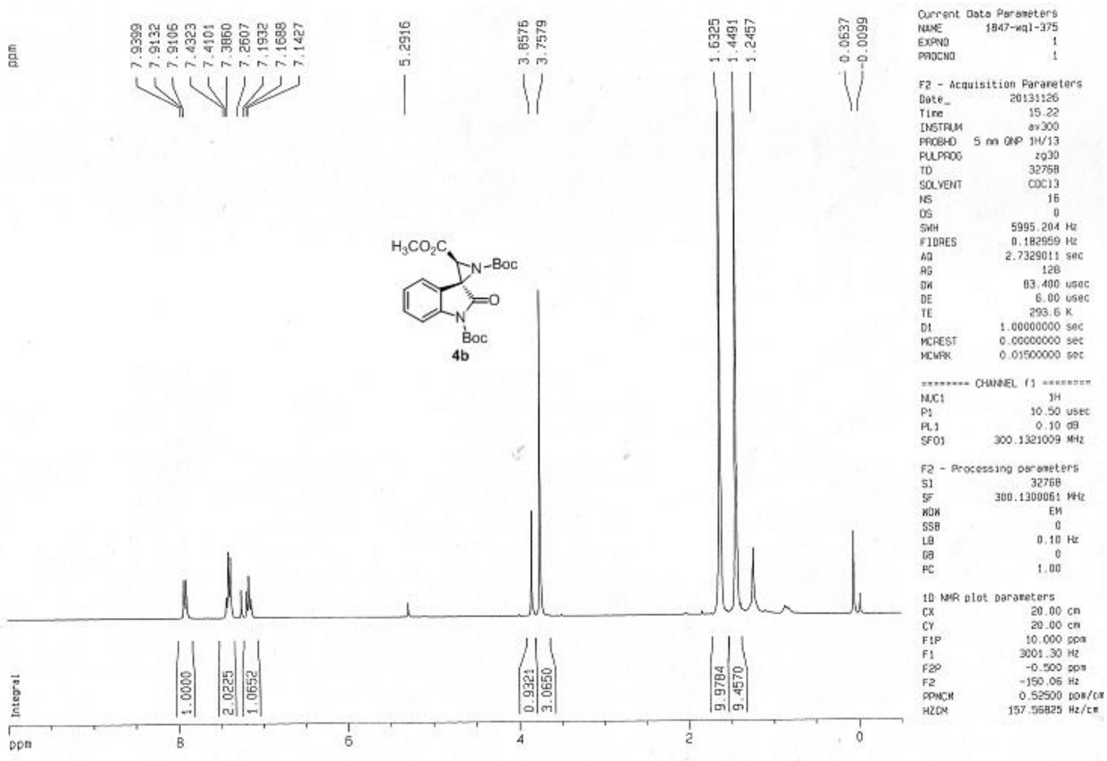
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 use
DE 6.00 use
TE 293.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =======
NUC1 13C
P1 10.50 use
PL1 -0.81 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4577542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 200.500 ppm
F1 15131.28 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm
HZCM 777.31787 Hz/
COOCH3
Boc
N
COOCH3
N
N
O
O
CO2Et
CO2Et
3h
ppm


COOCH3
Boc
N
COOCH3
N
O
Cbz
O
N
Cbz
3j
Current Data Parameters
F2 - Acquisition Parameters
F2 - Processing parameters
1D NMR plot parameters
Integral
ppm

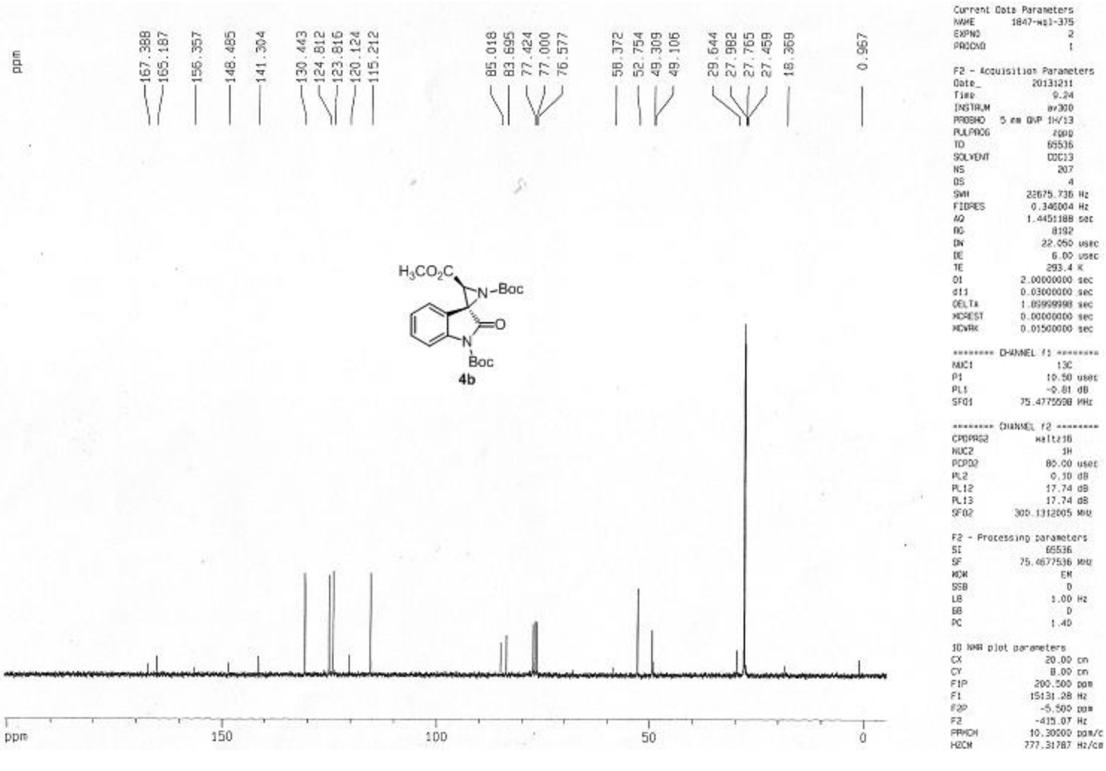

COOCH3
Boc
N
COOCH3
N
O
Cbz
O
N
Cbz
3j
Current Data Parameters
F2 - Acquisition Parameters
F2 - Processing parameters
1D NMR plot parameters
ppm


ppm
7.95139
7.92361
7.43310
7.40937
7.38424
7.33939
7.25897
7.19178
7.16559
7.14126
5.23257
5.19244
5.18573
5.14549
3.91182
3.75305
1.64429
1.58472
1.25772
0.88058
0.85622
0.07770
0.00036
H3CO2C
N–Cbz
N
O
Boc
4a
Integral
1.0000
6.9090
1.0650
2.0381
0.9404
3.0231
9.4428
ppm
8
6
4
2
0
Current Data Parameters
NAME 1847-wql-388
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131129
Time 14.54
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

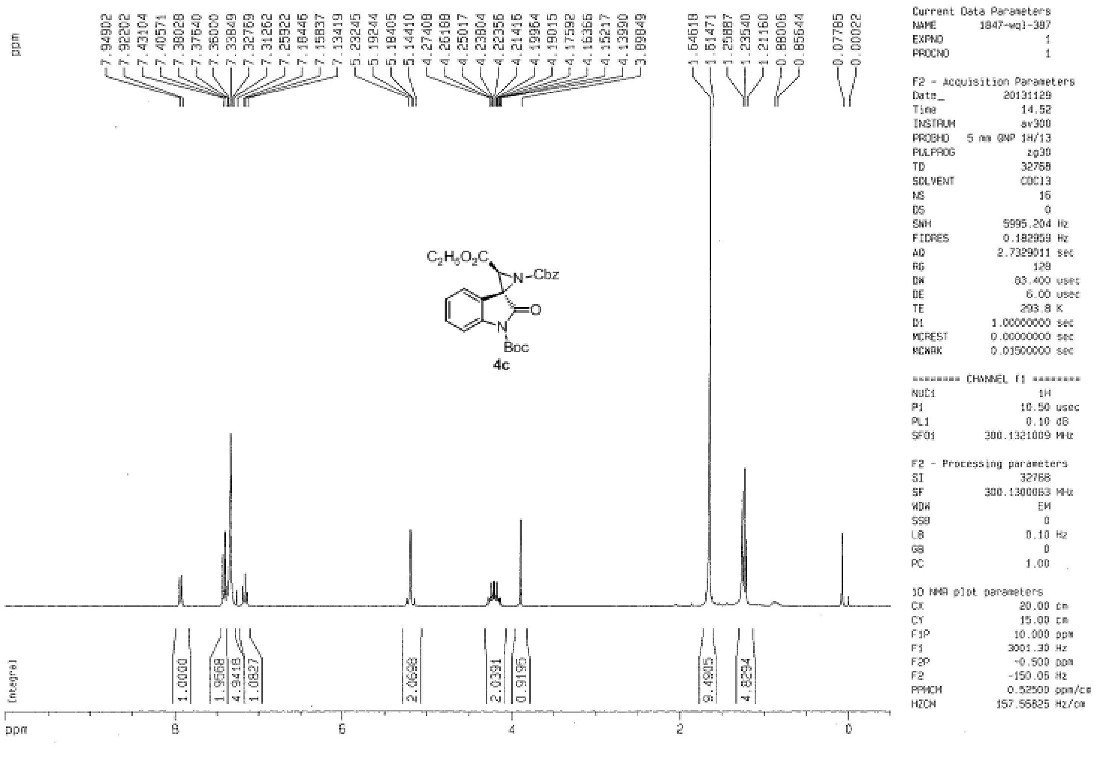

ppm
167.465
164.712
157.874
148.361
141.354
134.708
130.655
128.706
128.507
128.443
124.885
123.849
119.599
115.439
85.190
77.424
77.000
76.576
69.323
52.819
49.383
49.061
29.541
27.971
0.970
H3CO2C
N–Cbz
N
O
Boc
4a
ppm
150
100
50
0
Current Data Parameters
NAME 1847-wql-388
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131206
Time 10.40
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 167
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 293.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.80000008 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677552 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 200.500 ppm
F1 15131.29 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm/cm
HZCM 777.31293 Hz/cm

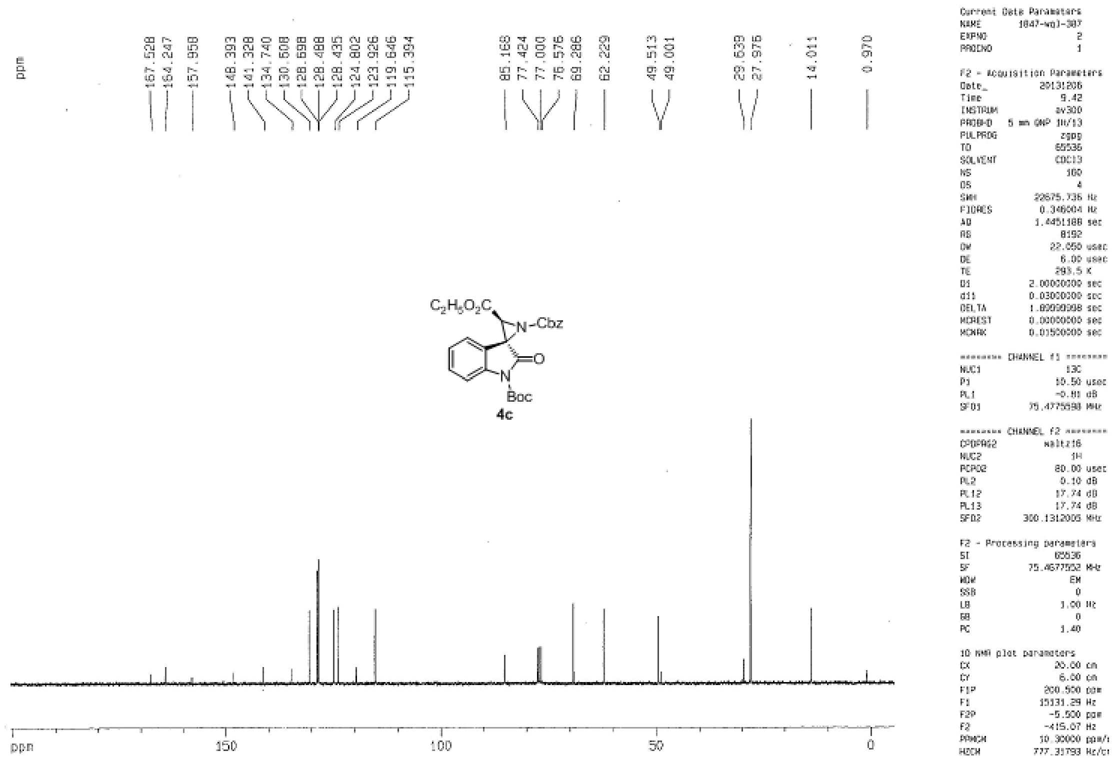


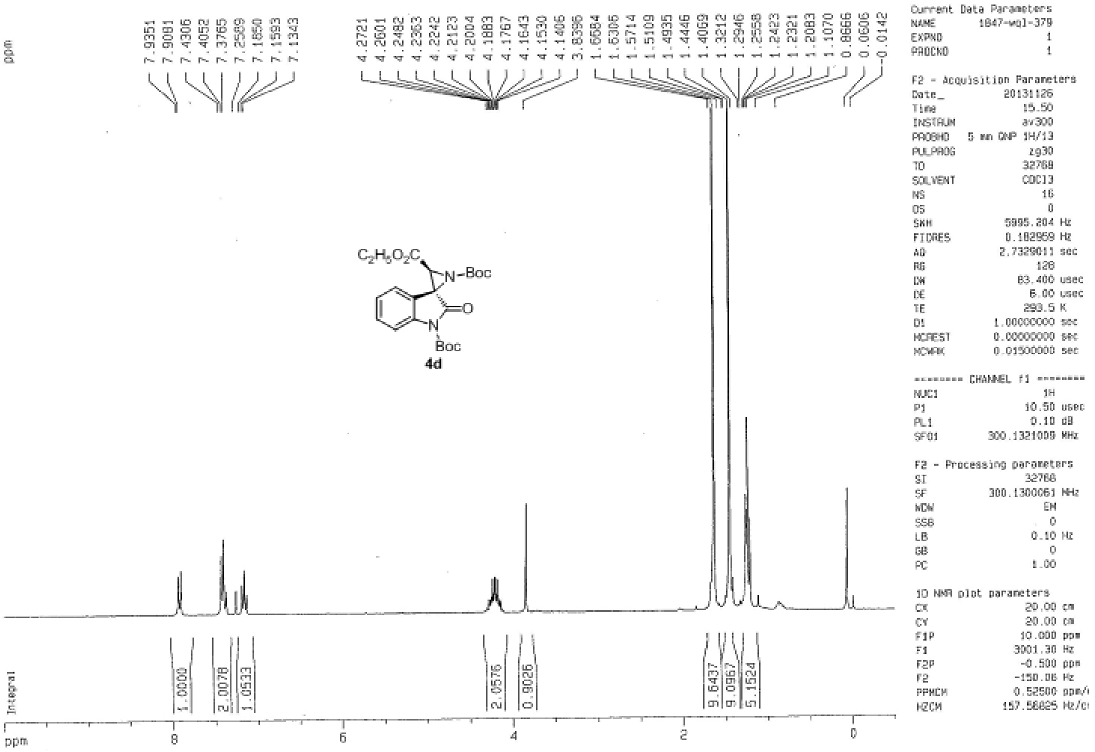
7.9399
7.9132
7.9106
7.4323
7.4101
7.3860
7.2607
7.1932
7.1688
7.1427
5.2916
3.8576
3.7579
1.6325
1.4491
1.2457
0.0537
-0.0099
H3CO2C
N-Boc
O
N
Boc
4b
Integral
1.0000
2.0225
1.0652
0.9321
3.0650
9.9784
9.4570
ppm
8
6
4
2
0
Current Data Parameters
NAME 1847-wql-375
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131126
Time 15.22
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



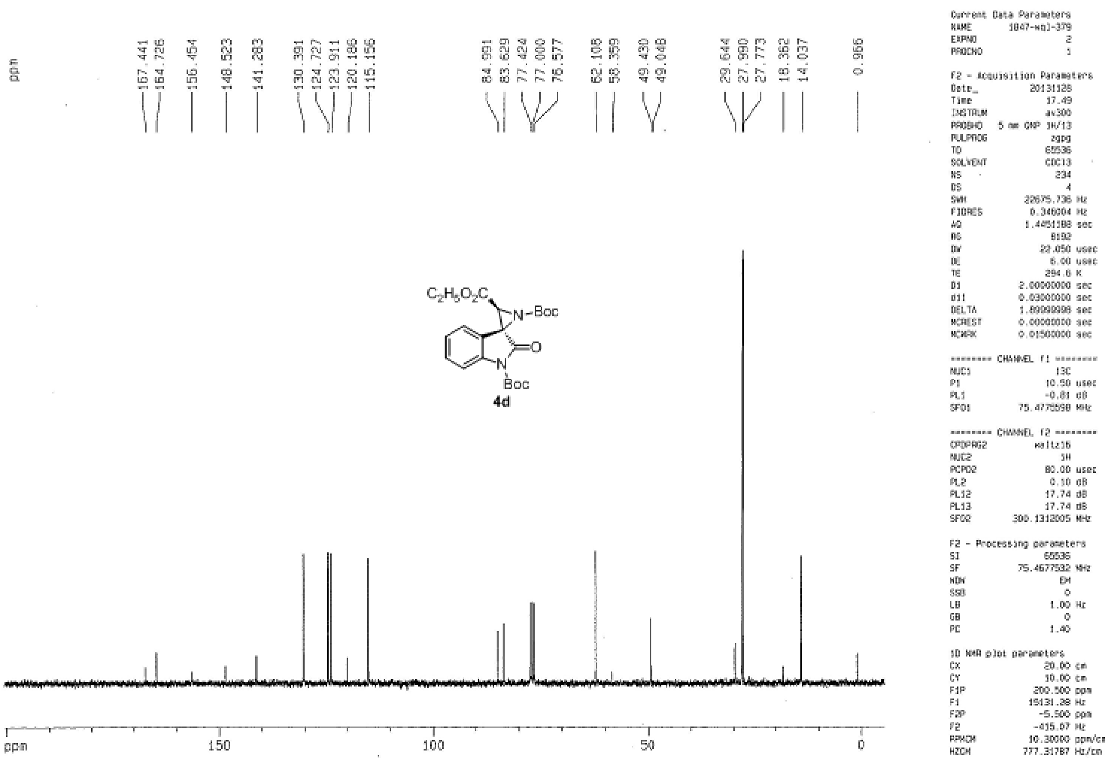
167.388
165.187
156.357
148.485
141.304
130.443
124.812
123.816
120.124
115.212
85.018
83.695
77.424
77.000
76.577
58.372
52.754
49.309
49.106
29.544
27.982
27.765
27.459
18.369
0.967
H3CO2C
N-Boc
O
N
Boc
4b
ppm
150
100
50
0
Current Data Parameters
NAME 1847-wql-375
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131211
Time 9.24
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 207
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 293.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677536 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 200.500 ppm
F1 15131.28 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm/cm
HZCM 777.31787 Hz/cm


C2H5O2C
N–Cbz
O
N
Boc
4c
7.94902
7.92202
7.43104
7.40571
7.38028
7.37640
7.36000
7.33849
7.32769
7.31262
7.25922
7.18446
7.15837
7.13419
5.23245
5.19244
5.18405
5.14410
4.27408
4.26188
4.25017
4.23804
4.22356
4.21416
4.19964
4.19015
4.17592
4.16365
4.15217
4.13990
3.89849
1.64618
1.61471
1.25887
1.23540
1.21160
0.88006
0.85644
0.07785
0.00022
[integral]
1.0000
1.9568
4.9418
1.0827
2.0698
2.0391
0.9195
9.4905
4.8294
ppm
8
6
4
2
0
Current Data Parameters
NAME 1847-wql-387
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131129
Time 14.52
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.05 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

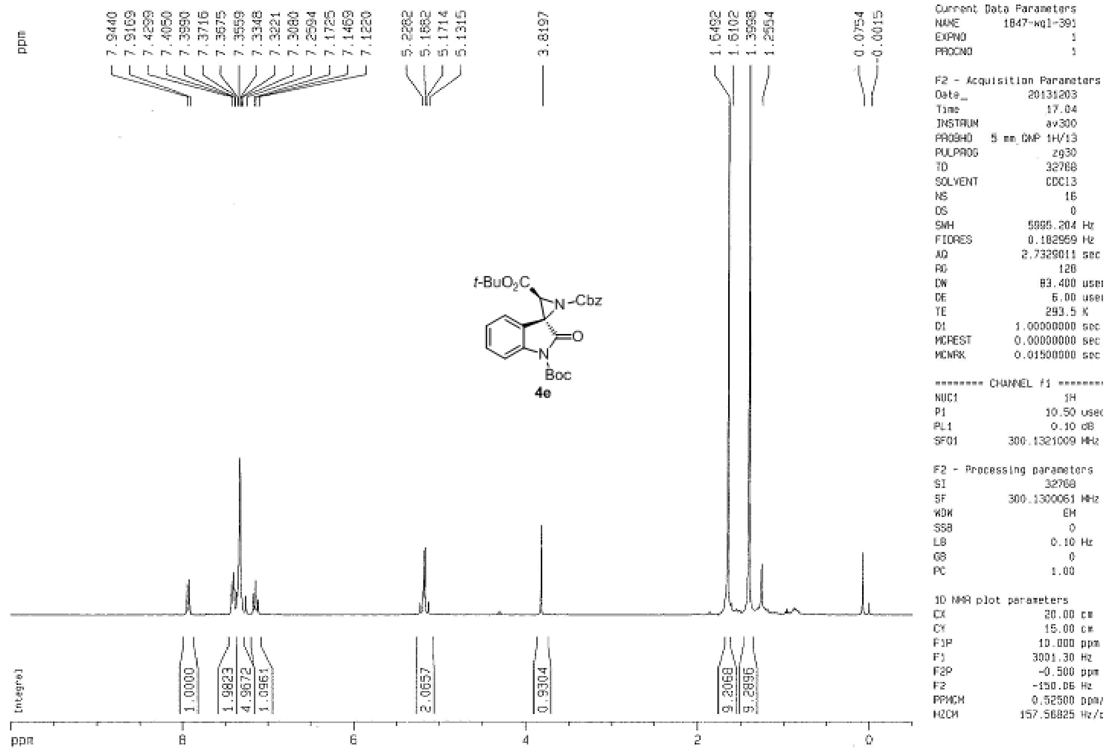

C2H5O2C
N–Cbz
O
N
Boc
4c
167.528
164.247
157.958
148.393
141.328
134.740
130.508
128.698
128.488
128.435
124.802
123.926
119.646
115.394
85.168
77.424
77.000
76.576
69.286
62.229
49.513
49.001
29.639
27.976
14.011
0.970
ppm
150
100
50
0
Current Data Parameters
NAME 1847-wql-387
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131206
Time 9.42
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 293.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677552 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 200.500 ppm
F1 15131.29 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm/cm
HZCM 777.31793 Hz/cm



ppm
7.9351
7.9081
7.4306
7.4052
7.3765
7.2569
7.1850
7.1593
7.1343
4.2721
4.2601
4.2482
4.2363
4.2242
4.2123
4.2004
4.1883
4.1767
4.1643
4.1530
4.1406
3.8396
1.6584
1.6305
1.5714
1.5109
1.4935
1.4446
1.4069
1.3212
1.2946
1.2558
1.2423
1.2321
1.2083
1.1070
0.8666
0.0606
-0.0142
$C_2H_5O_2C$
N–Boc
O
N
Boc
4d
Integral
1.0000
2.0078
1.0533
2.0576
0.9026
9.6437
9.0967
5.1524
ppm
8
6
4
2
0
Current Data Parameters
NAME 1847-wql-379
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131126
Time 15.50
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



ppm
167.441
164.726
156.454
148.523
141.283
130.391
124.727
123.911
120.186
115.156
84.991
83.629
77.424
77.000
76.577
62.108
58.359
49.430
49.048
29.644
27.990
27.773
18.362
14.037
0.966
$C_2H_5O_2C$
N–Boc
O
N
Boc
4d
ppm
150
100
50
0
Current Data Parameters
NAME 1847-wql-379
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131126
Time 17.49
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 234
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 294.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677532 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 200.500 ppm
F1 15131.38 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm/cm
HZCM 777.31787 Hz/cm


ppm
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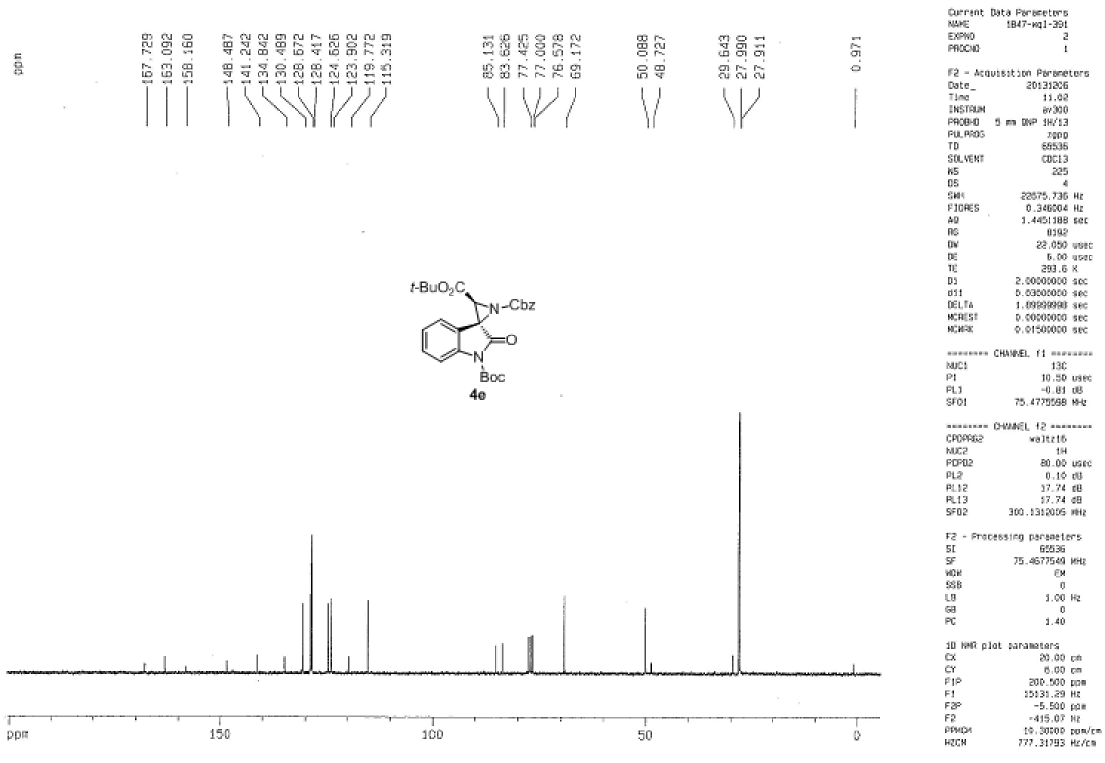
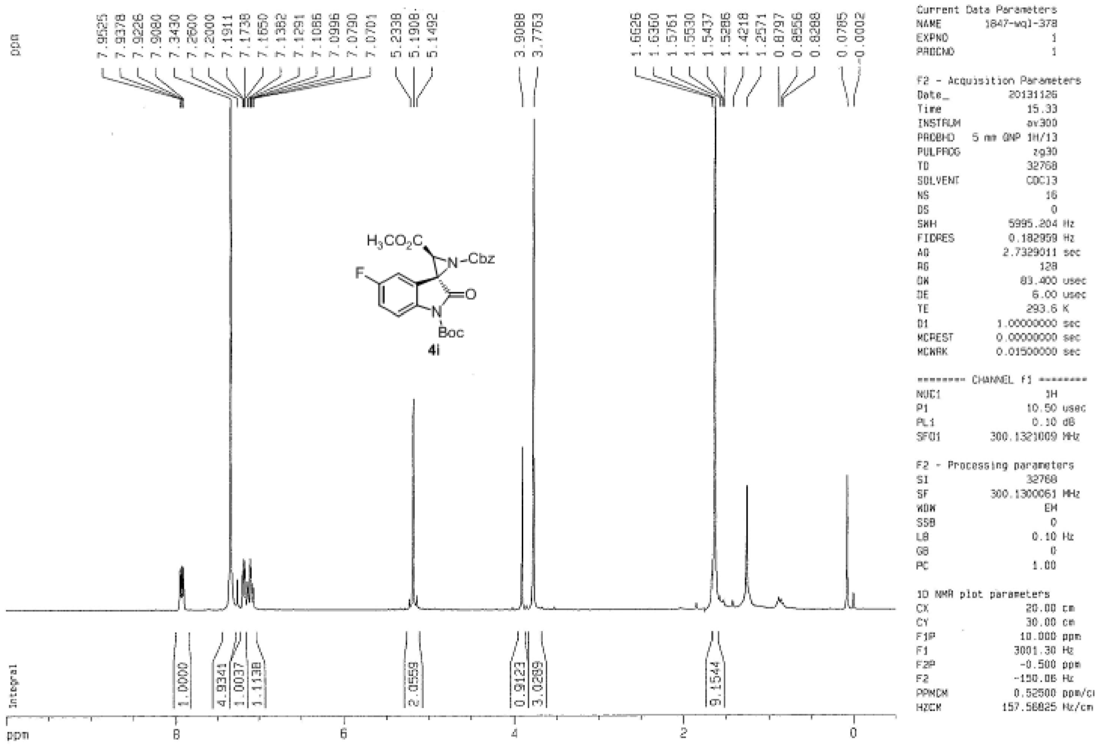
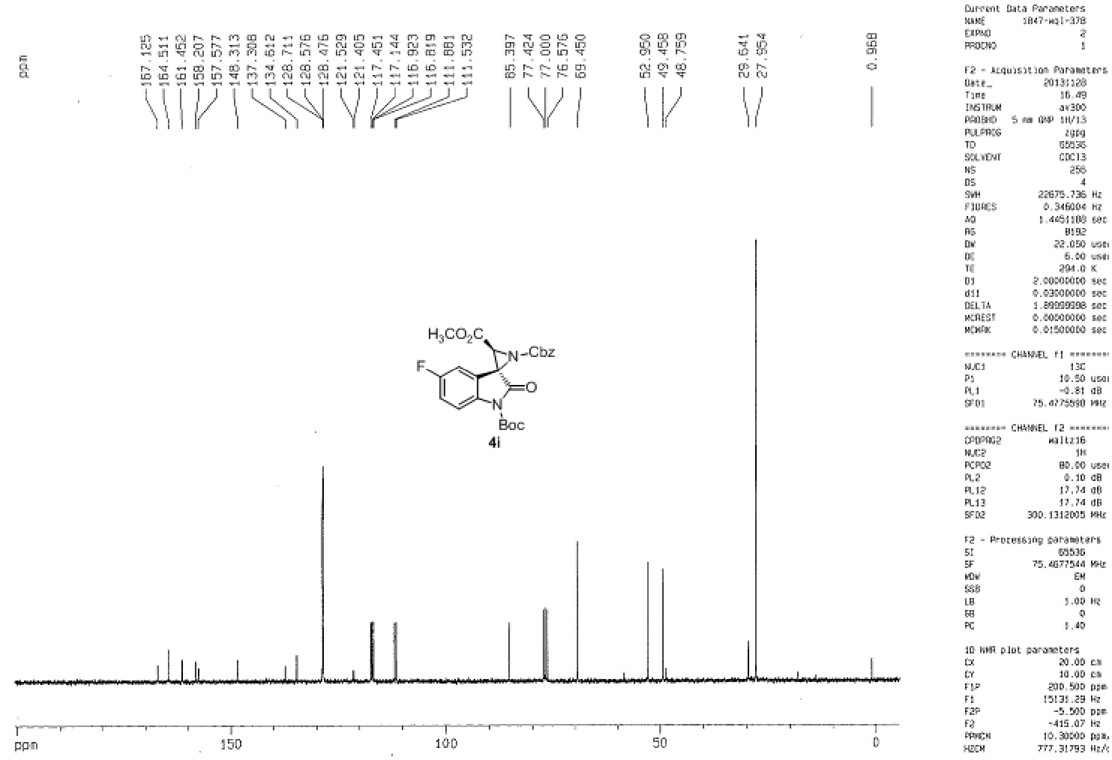
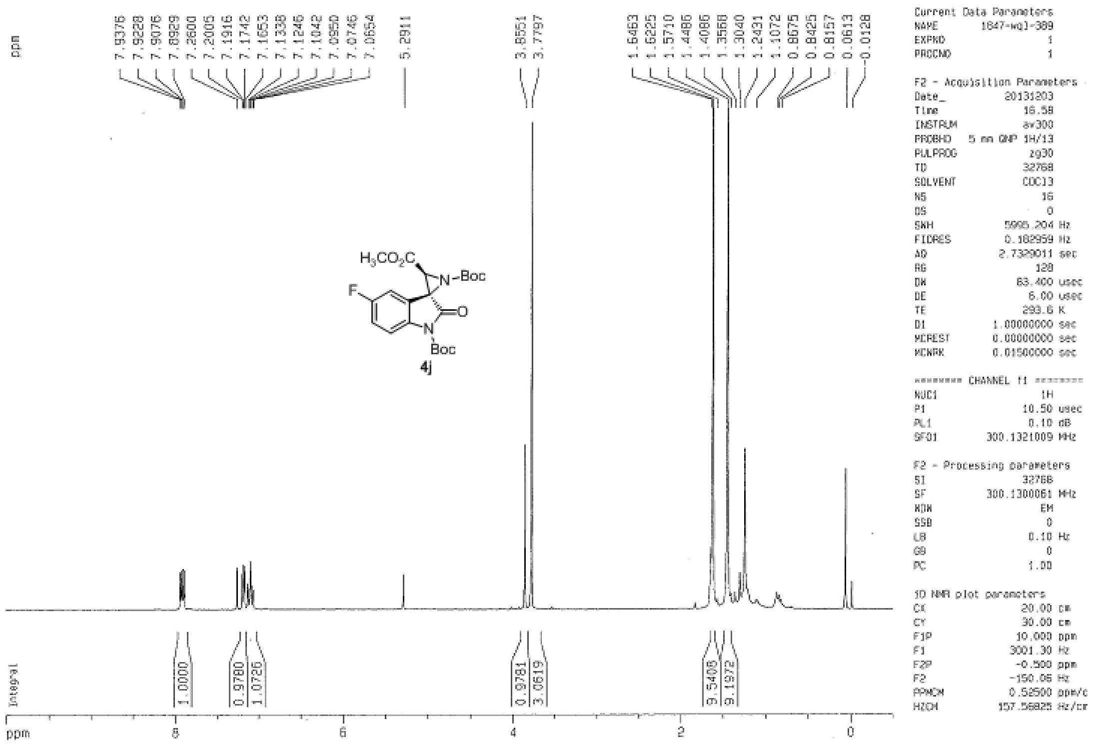
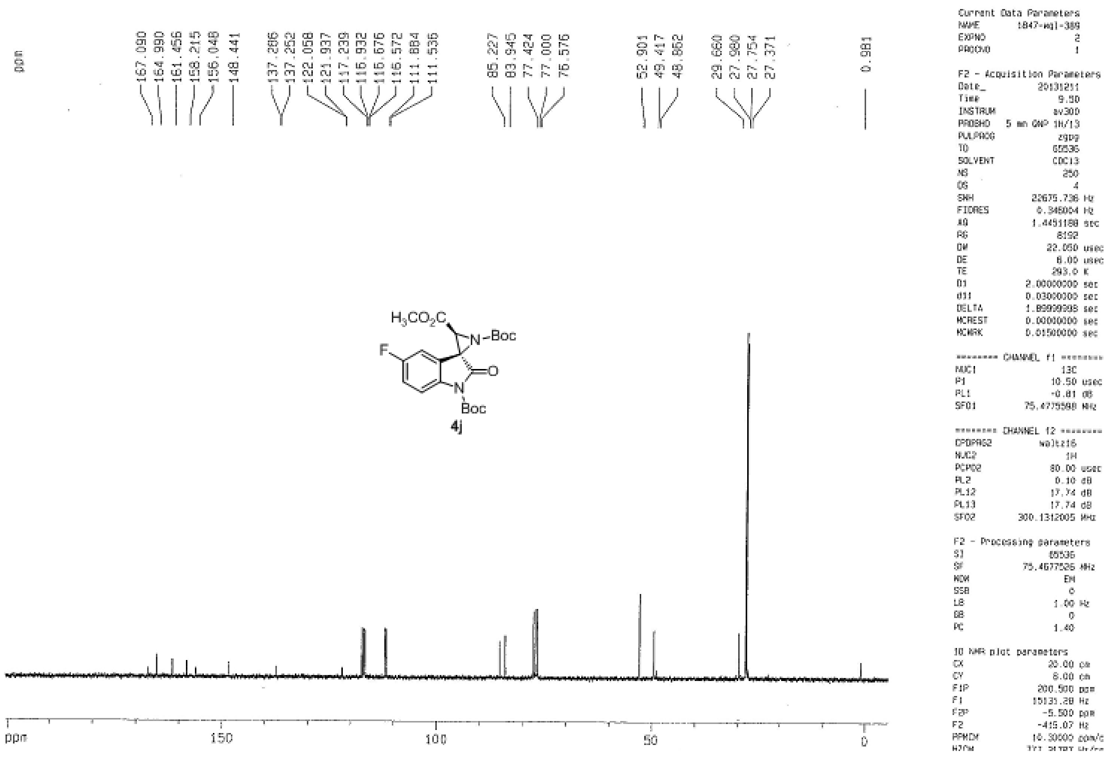
7.9169
7.4299
7.4050
7.3890
7.3716
7.3675
7.3559
7.3348
7.3221
7.3080
7.2594
7.1725
7.1469
7.1220
5.2282
5.1882
5.1714
5.1315
3.8197
1.6492
1.6102
1.3998
1.2554
0.0754
-0.0015
t-BuO2C
N-Cbz
O
N
Boc
4e
[integral]
1.0000
1.9823
4.9672
1.0961
2.0657
0.9304
9.2068
9.2896
ppm
8
6
4
2
0
Current Data Parameters
NAME 1847-wql-391
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131203
Time 17.04
INSTRUM av300
PROBHD 5 mm DNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

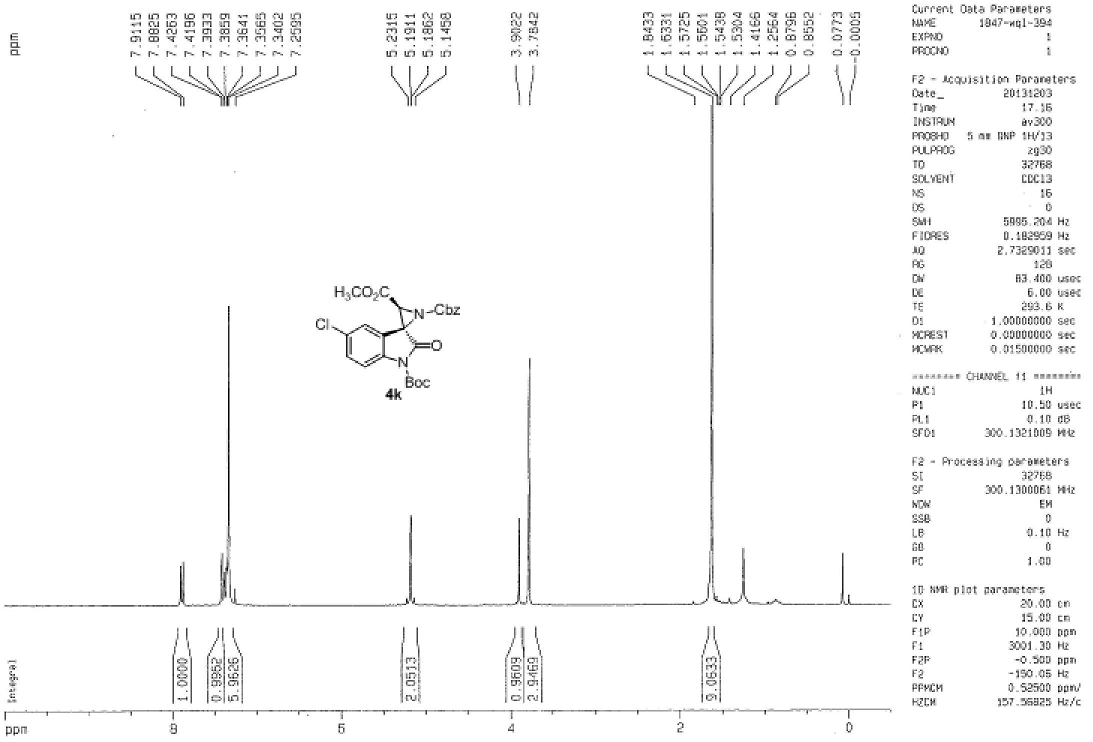

ppm
167.729
163.092
158.160
148.487
141.242
134.842
130.489
128.672
128.417
124.626
123.902
119.772
115.319
85.131
83.626
77.425
77.000
76.578
69.172
50.088
48.727
29.643
27.990
27.911
0.971
t-BuO2C
N-Cbz
O
N
Boc
4e
ppm
150
100
50
0
Current Data Parameters
NAME 1847-wql-391
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131206
Time 11.02
INSTRUM av300
PROBHD 5 mm DNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 225
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 293.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4775568 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677549 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 200.500 ppm
F1 15131.29 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm/cm
HZCM 777.31793 Hz/cm

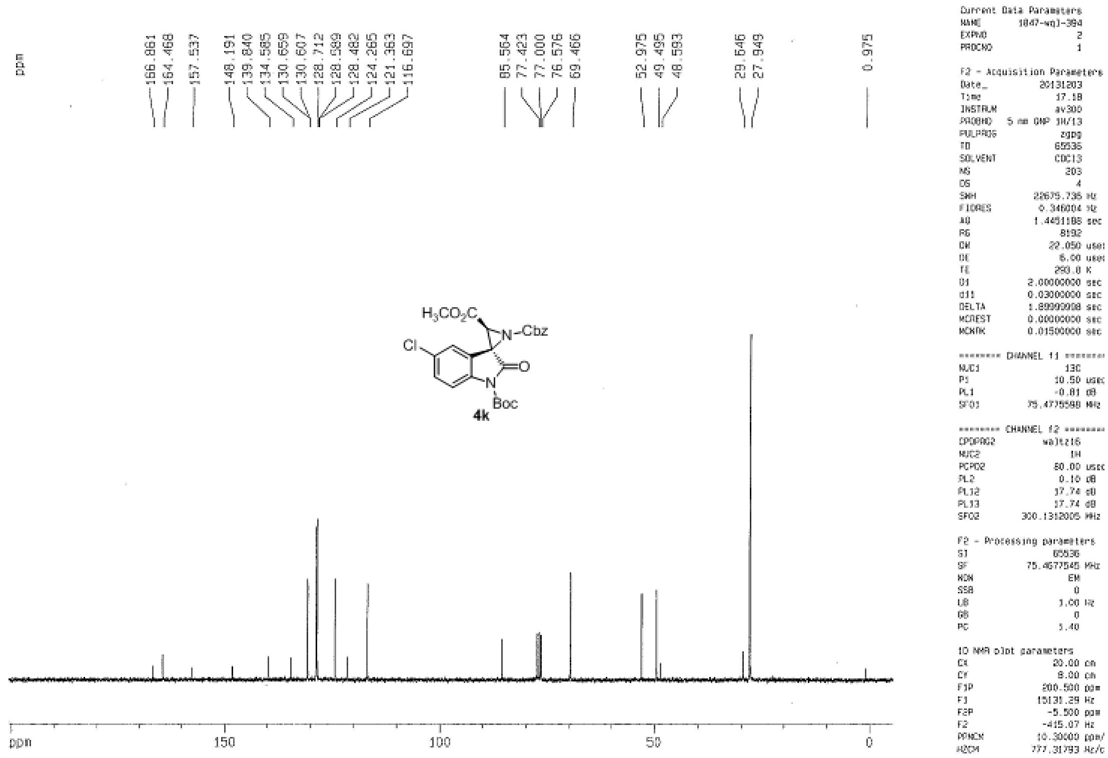

7.9525
7.9378
7.9226
7.9080
7.3430
7.2600
7.2000
7.1911
7.1738
7.1650
7.1382
7.1291
7.1086
7.0996
7.0790
7.0701
5.2338
5.1908
5.1492
3.9088
3.7763
1.6626
1.6360
1.5761
1.5530
1.5437
1.5286
1.4218
1.2571
0.8797
0.8556
0.8288
0.0785
0.0002
H3CO2C
N-Cbz
F
O
N
Boc
4i
1.0000
4.9341
1.0037
1.1138
2.0559
0.9123
3.0289
9.1544
Integral
ppm
Current Data Parameters
NAME 1847-wql-378
SOLVENT CDCl3
INSTRUM av300
SFO1 300.1321009 MHz

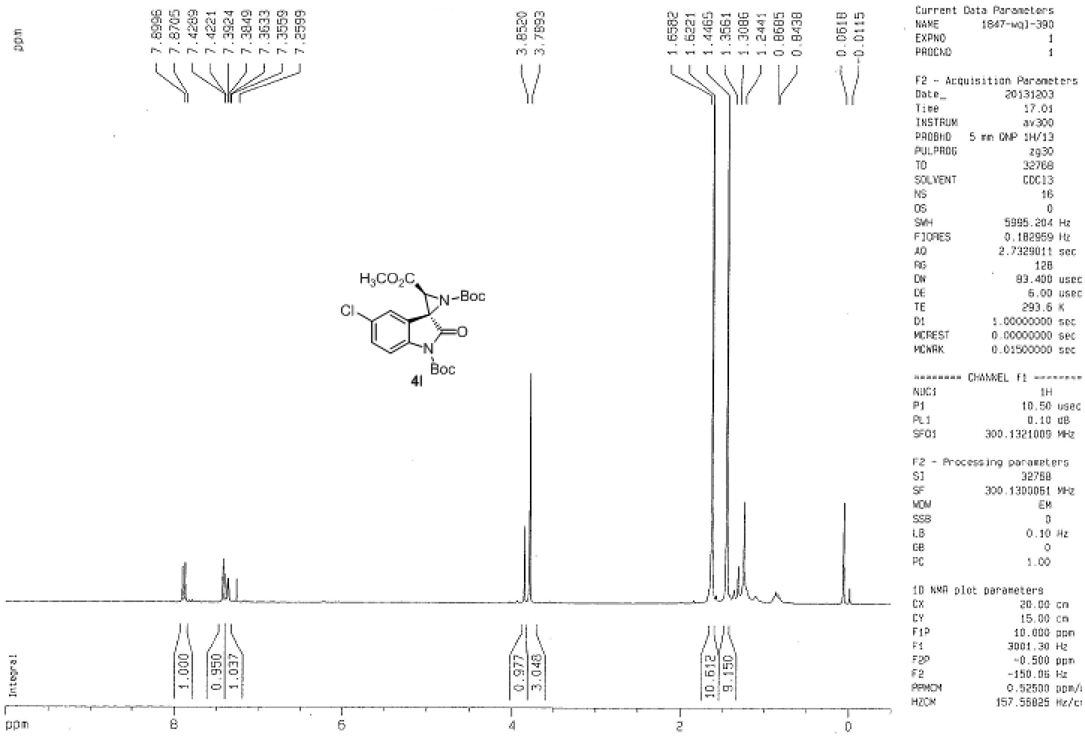

167.125
164.511
161.452
158.207
157.577
148.313
137.308
134.612
128.711
128.576
128.476
121.529
121.405
117.451
117.144
116.923
116.619
111.661
111.532
85.397
77.424
77.000
76.576
69.450
52.950
49.458
48.759
28.641
27.954
0.958
H3CO2C
N-Cbz
F
O
N
Boc
4i
ppm
Current Data Parameters
NAME 1847-wql-378
SOLVENT CDCl3
INSTRUM av300
SFO1 75.4775560 MHz


7.9376
7.9228
7.9076
7.8929
7.2600
7.2005
7.1916
7.1742
7.1653
7.1338
7.1246
7.1042
7.0950
7.0746
7.0654
5.2911
3.8551
3.7797
1.6463
1.6225
1.5710
1.4886
1.4086
1.3568
1.3040
1.2431
1.1072
0.8675
0.8425
0.8157
0.0613
-0.0128
H_3CO_2C
N–Boc
F
O
N
Boc
4j
Integral
1.0000
0.9780
1.0726
0.9781
3.0619
9.5408
9.1972
ppm
8
6
4
2
0

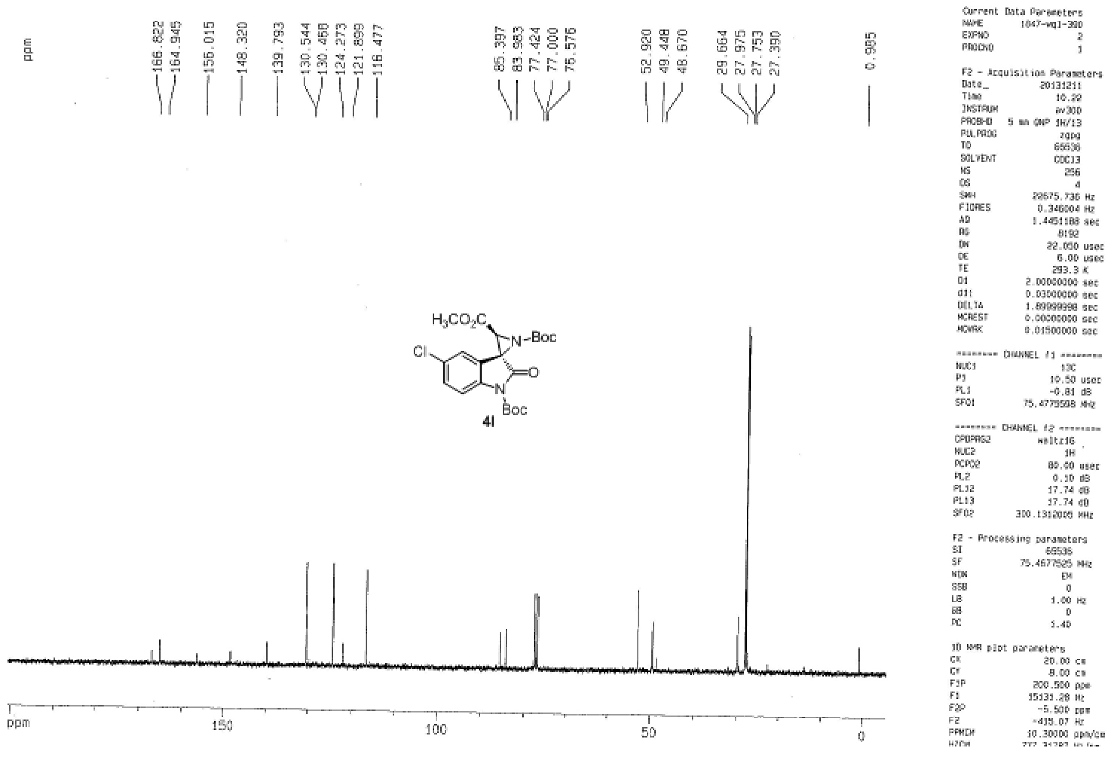

167.090
164.990
161.456
158.215
156.048
148.441
137.286
137.252
122.058
121.937
117.239
116.932
116.676
116.572
111.884
111.536
85.227
83.945
77.424
77.000
76.576
52.901
49.417
48.862
29.660
27.980
27.754
27.371
0.981
H_3CO_2C
N–Boc
F
O
N
Boc
4j
ppm
150
100
50
0


7.9115 7.8825 7.4263 7.4196 7.3933 7.3859 7.3641 7.3565 7.3402 7.2595
5.2315 5.1911 5.1862 5.1458
3.9022 3.7842
1.6433 1.6331 1.5725 1.5601 1.5438 1.5304 1.4166 1.2564 0.8796 0.8552
0.0773 -0.0005
H_3CO_2C
N–Cbz
Cl
O
N
Boc
4k
Integral
1.0000 0.9952 5.9625 2.0513 0.9609 2.9469 9.0633
ppm 8 6 4 2 0
Current Data Parameters
NAME 1847-wql-394
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131203
Time 17.16
INSTRUM av300
PROBHD 5 mm DNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.05 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

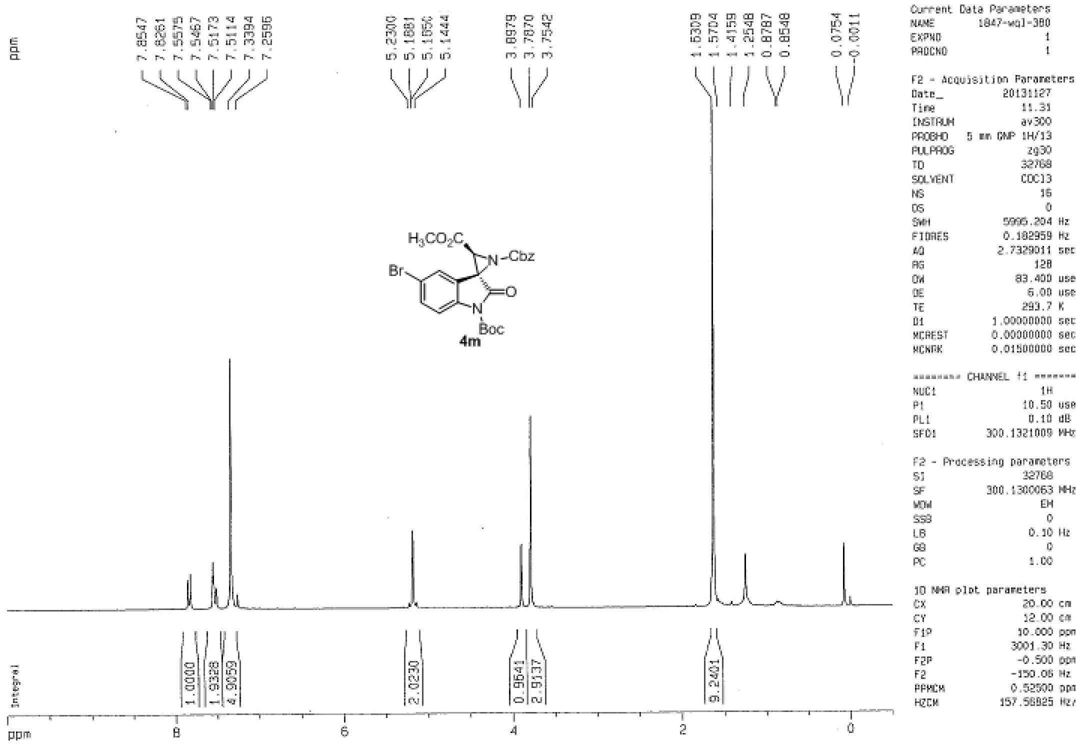

166.861 164.468 157.537 148.191 139.840 134.585 130.659 130.607 128.712 128.589 128.482 124.265 121.363 116.697
85.564 77.423 77.000 76.576 69.466
52.975 49.495 48.593
29.546 27.949
0.975
H_3CO_2C
N–Cbz
Cl
O
N
Boc
4k
ppm 150 100 50 0
Current Data Parameters
NAME 1847-wql-394
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131203
Time 17.18
INSTRUM av300
PROBHD 5 mm DNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 203
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 293.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677545 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 200.500 ppm
F1 15131.29 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm/cm
HZCM 777.31793 Hz/cm

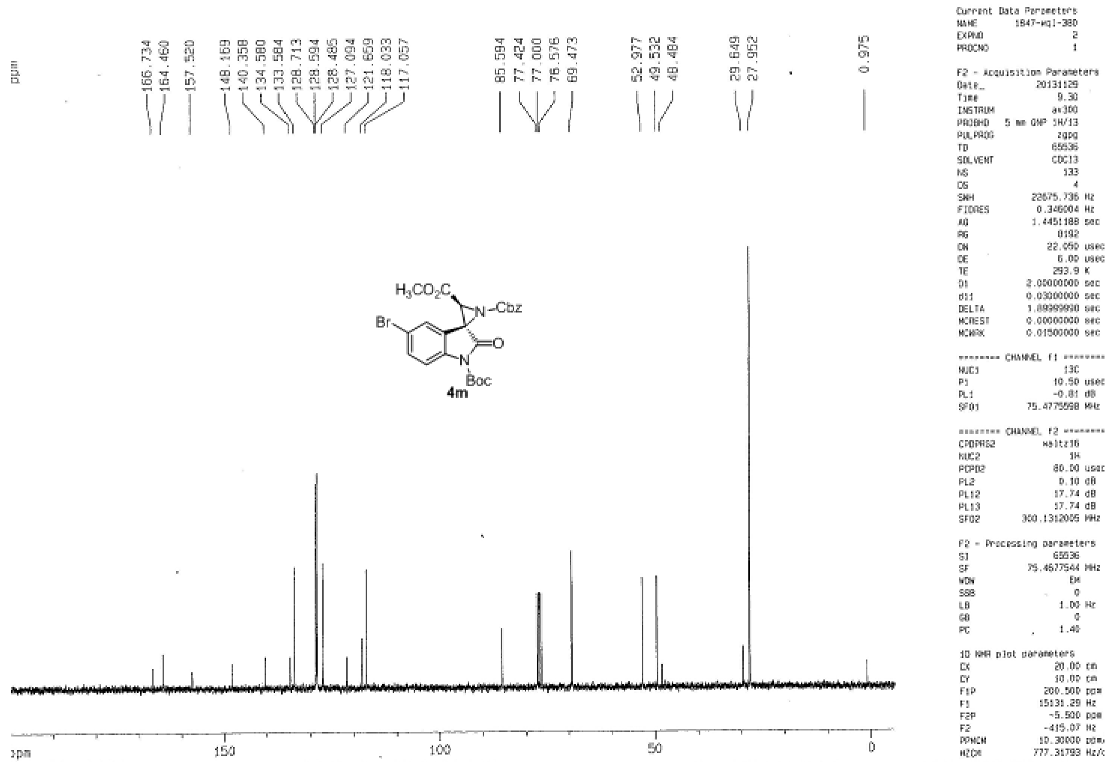


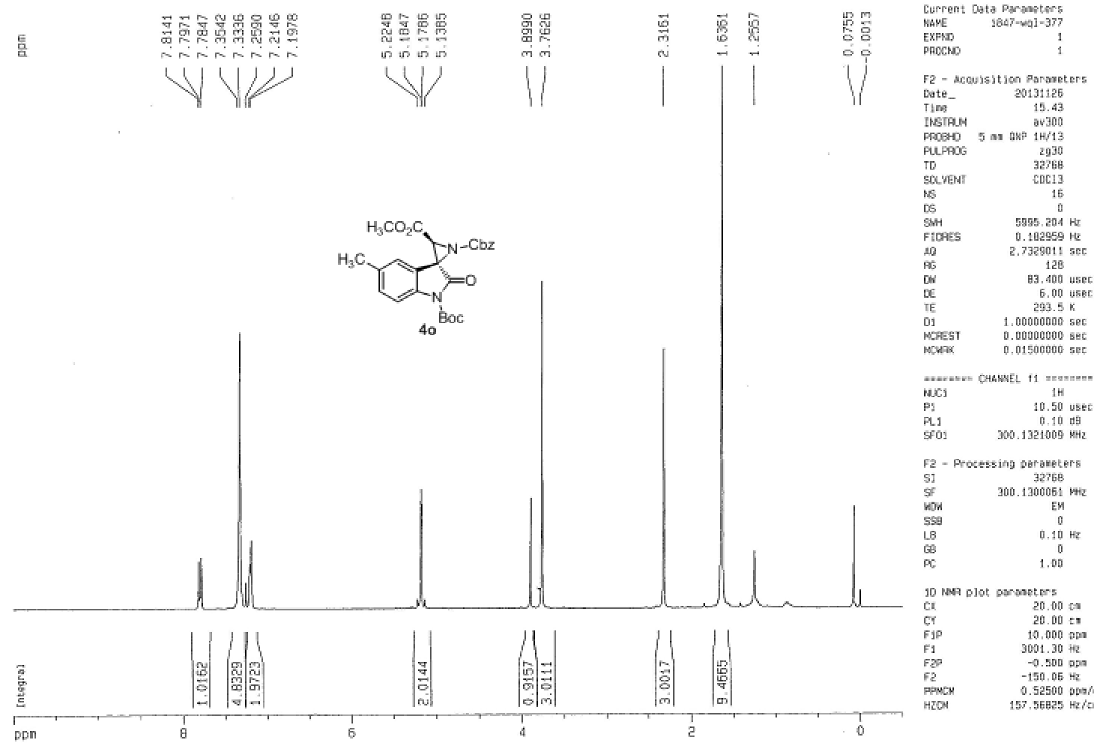
7.8996
7.8705
7.4289
7.4221
7.3924
7.3849
7.3633
7.3559
7.2599
3.8520
3.7893
1.6582
1.6221
1.4465
1.3561
1.3086
1.2441
0.8685
0.8438
0.0618
-0.0115
H3CO2C
N-Boc
Cl
O
N
Boc
4l
Integral
1.000
0.950
1.037
0.977
3.048
10.612
9.150
ppm
8
6
4
2
0
Current Data Parameters
NAME 1847-wql-390
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131203
Time 17.01
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



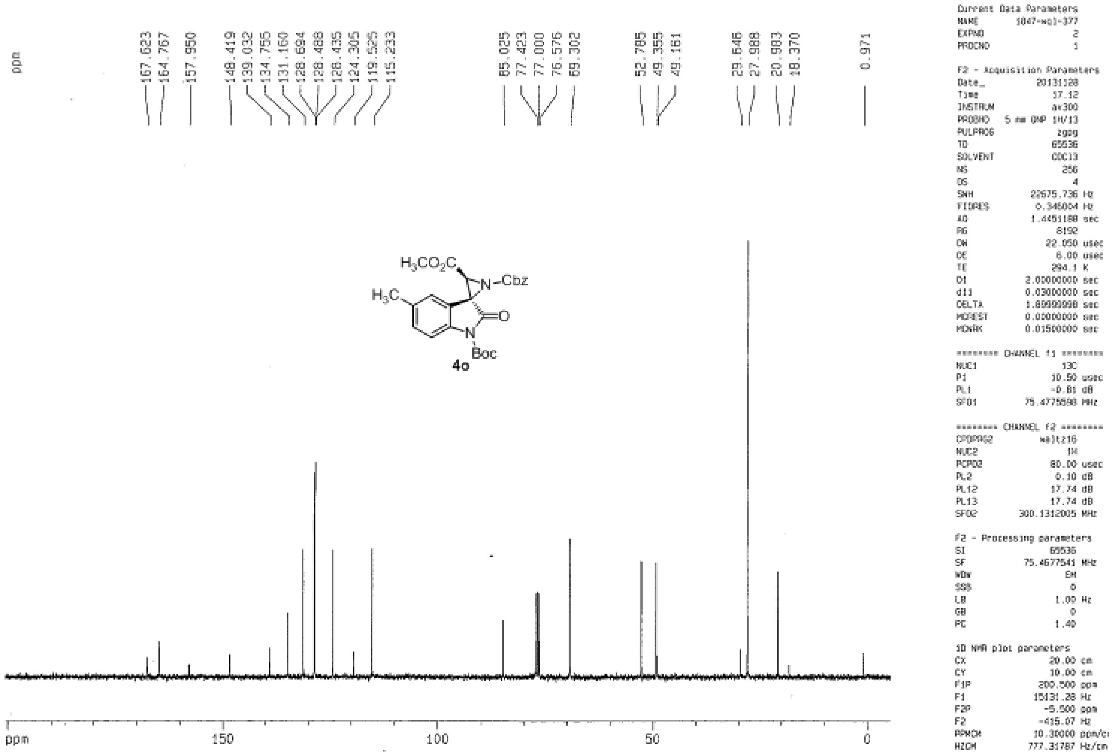
166.822
164.945
155.015
148.320
139.793
130.544
130.458
124.273
121.899
116.477
85.397
83.983
77.424
77.000
76.576
52.920
49.448
48.670
29.664
27.975
27.753
27.390
0.985
H3CO2C
N-Boc
Cl
O
N
Boc
4l
ppm
150
100
50
0
Current Data Parameters
NAME 1847-wql-390
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131211
Time 10.22
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 20675.735 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 293.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677525 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 200.500 ppm
F1 15131.28 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm/cm


7.8547 7.8261 7.5575 7.5467 7.5173 7.5114 7.3394 7.2596
5.2300 5.1881 5.1856 5.1444
3.8979 3.7870 3.7542
1.6309 1.5704 1.4159 1.2548 0.8787 0.8548
0.0754 -0.0011
H_3CO_2C
N-Cbz
Br
O
N
Boc
4m
1.0000 1.9328 4.9059 2.0230 0.9641 2.9137 9.2401
Integral
ppm
Current Data Parameters
NAME 1847-wql-380
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131127
Time 11.31
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 use
DE 6.00 use
TE 293.7 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 use
PL1 0.10 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm
HZCM 157.56825 Hz/

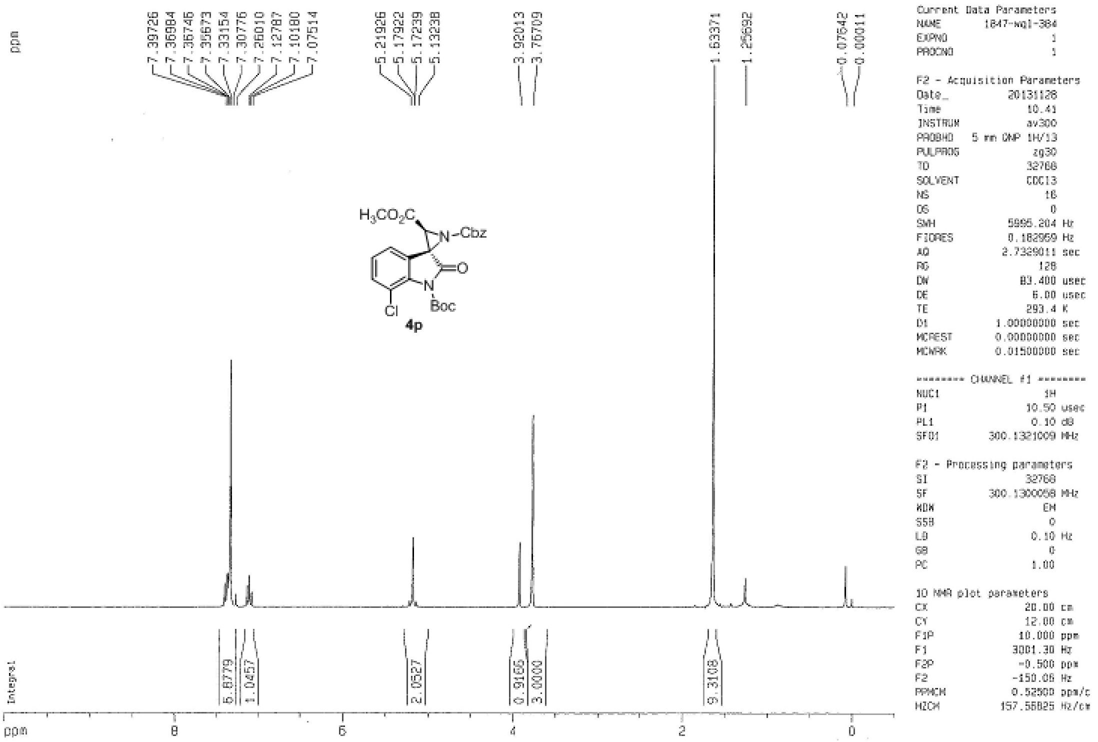

166.734 164.460 157.520 148.169 140.358 134.580 133.584 128.713 128.594 128.485 127.094 121.659 118.033 117.057
85.594 77.424 77.000 76.576 69.473
52.977 49.532 48.484
29.649 27.952
0.975
H_3CO_2C
N-Cbz
Br
O
N
Boc
4m
ppm
150 100 50 0
Current Data Parameters
NAME 1847-wql-380
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131129
Time 9.30
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 133
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 293.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677544 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 200.500 ppm
F1 15131.29 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm
HZCM 777.31793 Hz/c



ppm
7.8141
7.7971
7.7847
7.3542
7.3336
7.2590
7.2146
7.1978
5.2248
5.1847
5.1786
5.1385
3.8990
3.7626
2.3161
1.6361
1.2557
0.0755
-0.0013
H3CO2C
N-Cbz
H3C
N
O
Boc
4o
[integral]
1.0162
4.8329
1.9723
2.0144
0.9157
3.0111
3.0017
9.4665
ppm
8
6
4
2
0
Current Data Parameters
NAME 1847-wql-377
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131126
Time 15.43
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/
HZCM 157.56825 Hz/c



ppm
167.623
164.767
157.950
148.419
139.032
134.755
131.160
128.694
128.488
128.435
124.305
119.525
115.233
85.025
77.423
77.000
76.576
69.302
52.785
49.355
49.161
29.646
27.988
20.983
18.370
0.971
H3CO2C
N-Cbz
H3C
N
O
Boc
4o
ppm
150
100
50
0
Current Data Parameters
NAME 1847-wql-377
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131128
Time 17.12
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 294.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677541 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 200.500 ppm
F1 15131.28 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm/c
HZCM 777.31787 Hz/cm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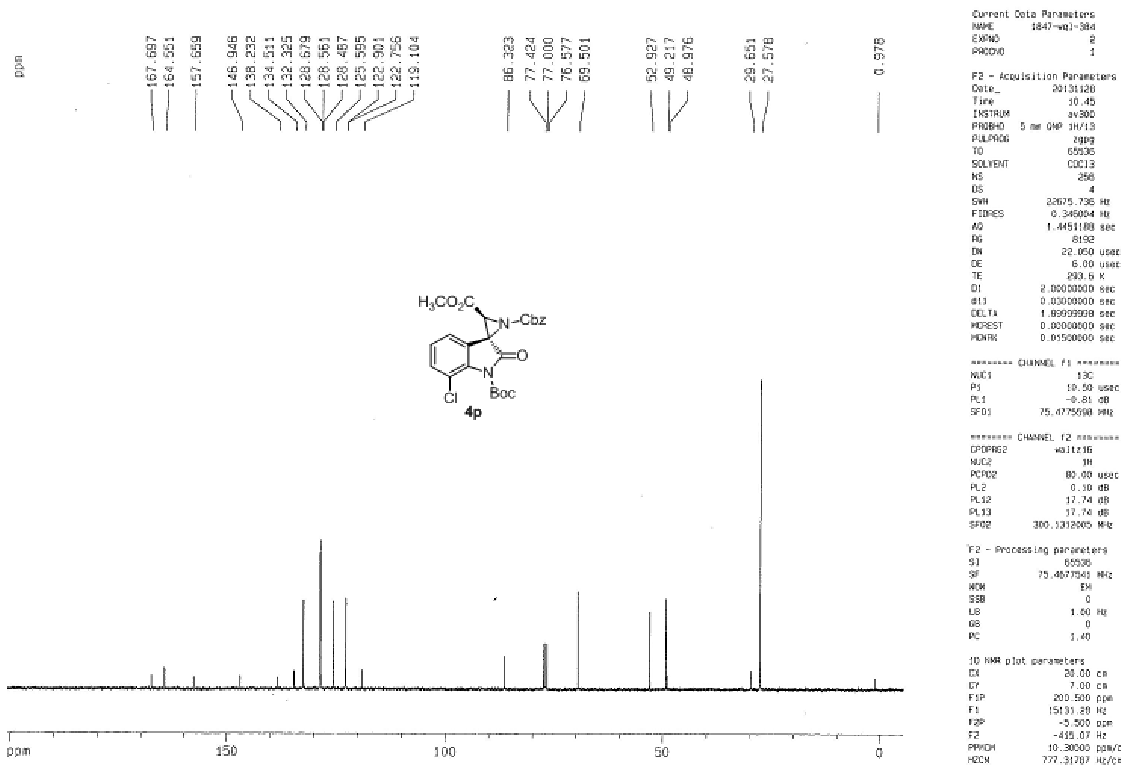
7.36984
7.36746
7.35673
7.33154
7.30776
7.26010
7.12787
7.10180
7.07514
5.21926
5.17922
5.17239
5.13238
3.92013
3.76709
1.63371
1.25692
0.07642
0.00011
H3CO2C
N–Cbz
O
N
Cl
Boc
4p
Integral
5.8779
1.0457
2.0527
0.9165
3.0000
9.3108
ppm
8
6
4
2
0
Current Data Parameters
NAME 1847-wql-384
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131128
Time 10.41
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300058 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm


167.697
164.551
157.659
146.946
138.232
134.511
132.325
128.679
128.561
128.487
125.595
122.901
122.756
119.104
86.323
77.424
77.000
76.577
69.501
52.927
49.217
48.976
29.651
27.578
0.976
H3CO2C
N–Cbz
O
N
Cl
Boc
4p
ppm
150
100
50
0
Current Data Parameters
NAME 1847-wql-384
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131128
Time 10.45
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 293.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677541 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 7.00 cm
F1P 200.500 ppm
F1 15131.28 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm/cm
HZCM 777.31787 Hz/cm