Supporting Information for

Desymmetrization of Cyclohexadienones *via* D-Camphor-derived Triazolium Salts Catalyzed Intramolecular Stetter Reaction

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General methods. Unless stated otherwise, all reactions were carried out in flame-dried glassware under a dry argon atmosphere. All solvents were purified and dried according to standard methods prior to use.

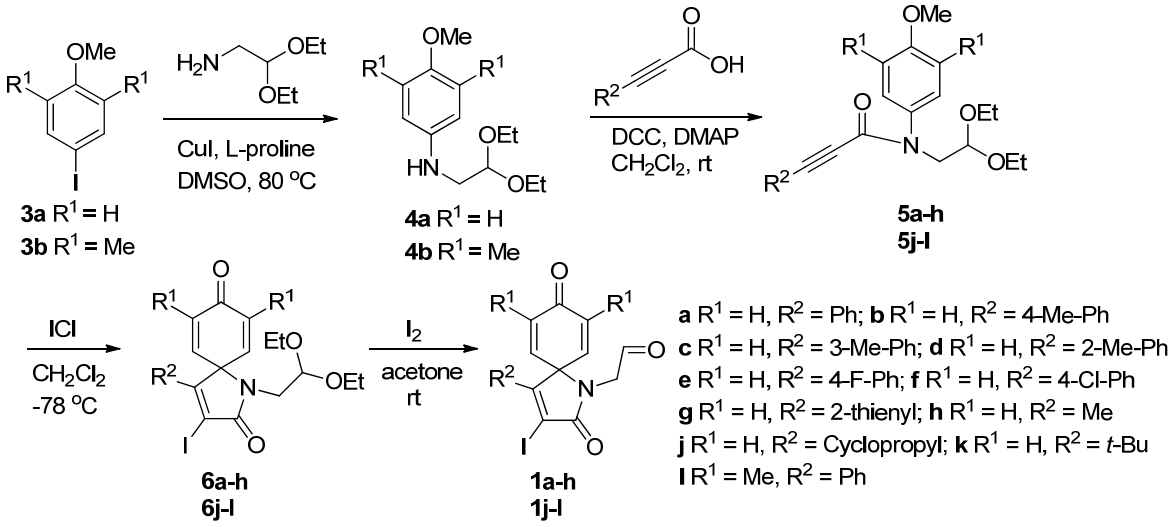
^{1}H and ^{13}C NMR spectra were recorded on Varian instruments (300 MHz and 75 MHz or 400 MHz and 100 MHz, respectively) and internally referenced to tetramethylsilane signal or residual protio solvent signals. Data for ^{1}H NMR are recorded as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, m = multiplet or unresolved, coupling constant(s) in Hz, integration). Data for ^{13}C NMR are reported in terms of chemical shift (δ, ppm).

The D-camphor-derived triazolium salt[1], compound **3b**[2] and 3-substituted-prop-2-ynoic acid[3-4] were prepared according to the reported procedures, compound **3a** is commercially available.

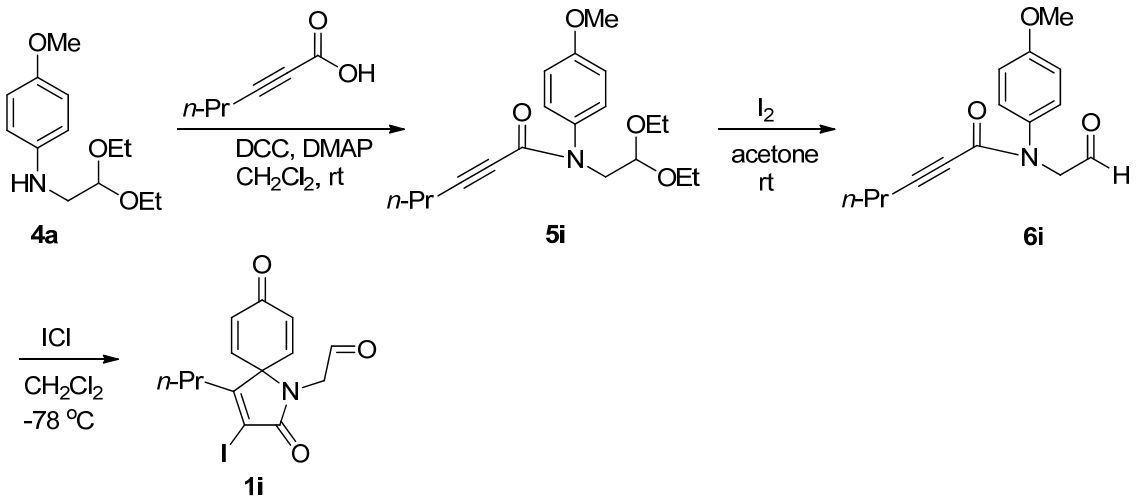
(1) Y. Li, Z. Feng and S.-L. You, *Chem. Commun.* 2008, 2263.
(2) T. M. Salo, J. Juho Helaja and A. M. P. Koskinen, *Tetrahedron Lett.* 2006, **47**, 2977.
(3) H. Sai, T. Ogiku, H. Ohmizu and A. Akio Ohtani, *Chem. Pharm. Bull.* 2006, **54**, 1686.
(4) T. W. Lyons and M. S. Sanford, *Tetrahedron* 2009, **65**, 3211.
(5) J.-W. Sun, Y.-M. Dong, L.-Y. Cao, X.-Y. Wang, S.-Z.Wang and Y.-F. Hu, *J. Org. Chem.* 2004, **69**, 8932.

Substrate synthesis

General procedure for the synthesis of substrates 1a-l



When R^1 = H, R^2 = *n*-Pr, the substrate was synthesized as follows:



1: General procedure for the synthesis of 3-substituted-prop-2-ynoic amide (5)

To a solution of 3-substituted-prop-2-ynoic acid (1.1 eq) in CH_2Cl_2 (0.2 mol/L), DMAP (0.1 eq) was added, then the solution was cooled to 0 °C. (*N*-4,4-Diethoxyalkyl)-4-methoxyaniline (1 eq) and DCC (1.2 eq) were added sequentially. The resulting solution was stirred at room temperature until completion. The precipitated urea was then filtered off by celite and the filtrate was concentrated

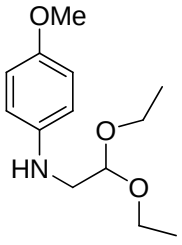
in vacuo. The residue was purified by column chromatography on silica gel to afford the desired compounds.

2: General procedure for dearomatization

ICl (2 eq) in CH_2Cl_2 (0.1 mol/L) was added to a solution of the diethoxyacetal (1 eq) (method **A**) or the corresponding aldehyde (1 eq) (method **B**) in CH_2Cl_2 (0.03 mol/L) at -78 ^oC over a period of 30-60 minutes. The resulting solution was stirred for another 10-60 minutes. After the reaction was complete (monitored by TLC), it was quenched with saturated aqueous Na_2SO_3 solution. The reaction mixture was allowed to warm up to room temperature and extracted three times with CH_2Cl_2. The combined organic layers were dried over Na_2SO_4, concentrated in vacuo, the residue was purified by column chromatography on silica gel to afford the corresponding iodocyclohexadienone.

3: General procedure for acetal removal[5]

To the solution of acetal (1 eq) in acetone (0.04 mol/L), iodine (10 mol%) was added. The resulting solution was stirred at room temperature until completion. The solution was concentrated in vacuo, and the residue was purified by column chromatography on silica gel to afford the desired compounds.

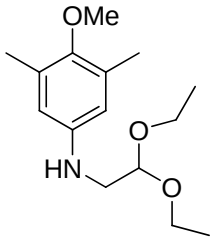


4a

***N*-(2,2-Diethoxyethyl)-4-methoxyaniline**

DMSO (15 mL) was added to a mixture of 4-iodoanisole (2.0 g, 8.55 mmol), CuI (163 mg, 0.85 mmol), L-proline (197 mg, 1.71 mmol) and K_2CO_3 (powdered) (2.38 g, 17.1 mmol) in a round bottom flask under argon, then 2,2-diethoxyethanamine (1.86 mL, 12.83 mmol) was added. The reaction mixture was stirred under argon at 80 ºC for

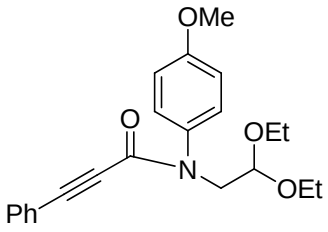
40h, diluted with water and extracted with ethyl acetate (3 x 30 mL). The combined organic layers were washed with water and brine, dried over Na_2SO_4, concentrated in vacuo. The residue was purified by column chromatography on silica gel (EtOAc:PE = 1:10) to afford the product as a pale yellow oil (1.67 g, 82% yield); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.77 (d, *J* = 9.0 Hz, 2H), 6.61 (d, *J* = 8.7 Hz, 2H), 4.67 (t, *J* = 5.4 Hz, 1H), 3.77-3.67 (m, 5H), 3.61-3.51 (m, 2H), 3.20 (d, *J* = 5.7 Hz, 2H), 1.23 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.2, 142.0, 114.7, 114.4, 100.9, 62.2, 55.6, 47.3, 15.3; MS (ESI) 240 ($[M+H]^+$); HRMS (ESI) mass calcd. For $C_{13}H_{22}NO_3$ ($[M+H]^+$): 240.1594. Found 240.1598.



4b

***N*-(2,2-Diethoxyethyl)-4-methoxy-3,5-dimethylaniline**

Pale yellow oil (97% yield), following the procedure for **4a**; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.28 (s, 2H), 4.63 (t, *J* = 4.6 Hz, 1H), 3.72-3.66 (m, 3H), 3.61 (d, *J* = 1.8 Hz, 3H), 3.55-3.50 (m, 2H), 3.17 (d, *J* = 5.4 Hz, 2H), 2.20 (s, 6H), 1.23 (t, *J* = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.5, 143.6, 130.7, 112.7, 100.5, 61.6, 59.3, 46.4, 15.7, 14.9; MS (EI, *m/z*, rel. intensity) 267 ($[M]^+$, 34), 164 (98), 103 (100); HRMS (EI) mass calcd. For $C_{15}H_{25}NO_3$ ($[M]^+$): 267.1834. Found 267.1833.

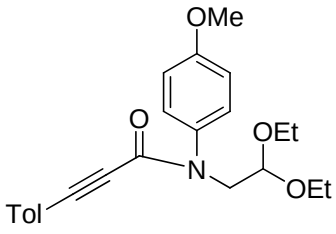


5a

***N*-(2,2-Diethoxyethyl)-*N*-(4-methoxyphenyl)-3-phenylpropiolamide**

Pale yellow solid, following general procedure **1** (93% yield). M.p. 64-66 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29-7.23 (m, 3H), 7.17 (t, *J* = 7.2 Hz, 2H), 7.10 (d, *J* =

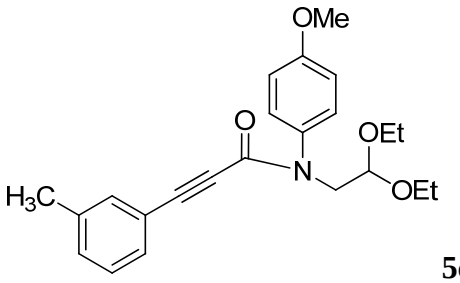
7.2 Hz, 2H), 6.88 (d, *J* = 9.0 Hz, 2H), 4.79 (t, *J* = 5.4 Hz, 1H), 3.84 (d, *J* = 5.4 Hz, 2H), 3.76 (s, 3H), 3.68-3.56 (m, 2H), 3.53-3.43 (m, 2H), 1.13 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.8, 154.4, 134.9, 132.0, 129.6, 129.3, 128.0, 120.0, 113.6, 98.9, 90.8, 82.4, 61.7, 55.1, 50.8, 14.9; MS (ESI) 368 ($[M+H]^+$); HRMS (ESI) mass calcd. For $C_{22}H_{25}NaNO_4$ ($[M+Na]^+$): 390.1676. Found 390.1683.



5b

***N*-(2,2-Diethoxyethyl)-*N*-(4-methoxyphenyl)-3-(p-tolyl)propiolamide**

Pale yellow solid (80% yield), following general procedure **1**. M.p. 54-56 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 (d, *J* = 9.0 Hz, 2H), 7.05 (s, 4H), 6.92 (d, *J* = 9.0 Hz, 2H), 4.84 (t, *J* = 5.7 Hz, 1H), 3.88-3.84 (m, 4H), 3.72-3.61 (m, 2H), 3.59-3.47 (m, 2H), 2.31 (s, 3H), 1.19 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.9, 154.9, 140.4, 135.4, 132.4, 129.6, 129.0, 117.3, 113.9, 99.2, 91.6, 82.3, 62.0, 55.5, 51.1, 21.5, 15.2; MS (ESI) 382 ($[M+H]^+$); HRMS (ESI) mass calcd. For $C_{23}H_{27}NaNO_4$ ($[M+Na]^+$): 404.1832. Found 404.1840.

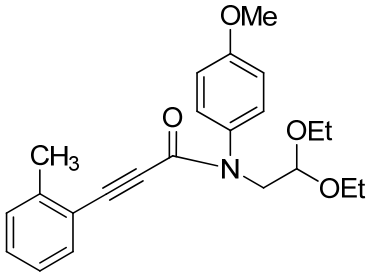


5c

***N*-(2,2-Diethoxyethyl)-*N*-(4-methoxyphenyl)-3-(m-tolyl)propiolamide**

Pale yellow oil (95% yield), following general procedure **1**. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 (d, *J* = 9.3 Hz, 2H), 7.11 (d, *J* = 4.5 Hz, 2H), 6.95-6.92 (m, 4H), 4.84 (t, *J* = 5.4 Hz, 1H), 3.88 (d, *J* = 6.0 Hz, 2H), 3.82 (s, 3H), 3.71-3.61 (m, 2H), 3.59-3.49 (m, 2H), 2.23 (s, 3H), 1.18 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.8,

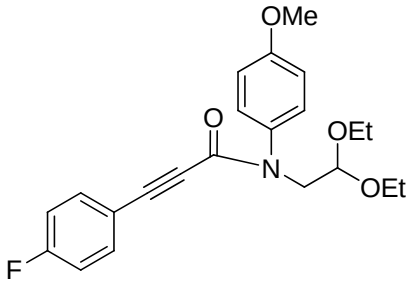
154.5, 137.7, 135.1, 132.7, 130.5, 129.4, 129.2, 127.9, 119.9, 113.7, 99.0, 91.2, 82.2, 61.7, 55.2, 50.8, 20.8, 15.0; MS (ESI) 382 ($[M+H]^+$); HRMS (MALDI) mass calcd. For $C_{23}H_{27}NaNO_4$ ($[M+Na]^+$): 404.1832. Found 404.1843.



5d

***N*-(2,2-Diethoxyethyl)-*N*-(4-methoxyphenyl)-3-(o-tolyl)propiolamide**

Pale yellow oil (98% yield), following general procedure **1**. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (d, *J* = 8.4 Hz, 2H), 7.14 (d, *J* = 7.5 Hz, 1H), 7.07 (d, *J* = 7.5 Hz, 1H), 6.95 (d, *J* = 7.5 Hz, 2H), 6.81 (d, *J* = 8.7 Hz, 2H), 4.75 (t, *J* = 5.7 Hz, 1H), 3.78 (d, *J* = 5.4 Hz, 2H), 3.69 (s, 3H), 3.61-3.51 (m, 2H), 3.49-3.38 (m, 2H), 1.84 (s, 3H), 1.08 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.0, 154.8, 141.2, 135.3, 133.0, 129.8, 129.6, 129.3, 125.4, 120.2, 114.1, 99.2, 99.0, 86.3, 61.9, 55.4, 51.2, 19.8, 15.1; MS (ESI) 404 ($[M+Na]^+$); HRMS (MALDI) mass calcd. For $C_{23}H_{27}NaNO_4$ ($[M+Na]^+$): 404.1832. Found 404.1840.

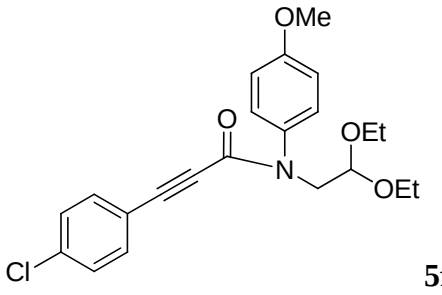


5e

***N*-(2,2-Diethoxyethyl)-3-(4-fluorophenyl)-*N*-(4-methoxyphenyl)propiolamide**

Yellow oil (84% yield), following general procedure **1**. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33 (d, *J* = 9.0 Hz, 2H), 7.15 (dd, *J* = 9.0, 5.4 Hz, 2H), 6.97-6.91 (m, 4H), 4.84 (t, *J* = 5.7 Hz, 1H), 3.88 (d, *J* = 6.0 Hz, 2H), 3.84 (s, 3H), 3.72-3.62 (m, 2H), 3.60-3.50 (m, 2H), 1.19 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.2 (d, *J* = 250.7 Hz),

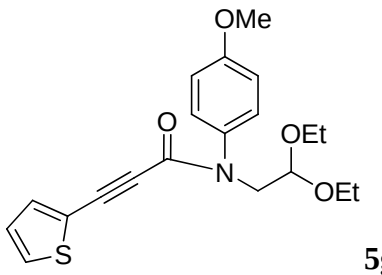
158.9, 154.5, 135.1, 134.4 (d, J = 9.1 Hz), 129.5, 116.3 (d, J = 3.4 Hz), 115.6 (d, J = 22.1 Hz), 113.8, 99.0, 90.0, 82.3, 61.9, 55.3, 50.9, 15.1; MS (ESI) 386 ($[M+H]^+$); HRMS (ESI) mass calcd. For $C_{22}H_{24}FNaNO_4$ ($[M+Na]^+$): 408.1582. Found 408.1583.



5f

3-(4-Chlorophenyl)-*N*-(2,2-diethoxyethyl)-*N*-(4-methoxyphenyl)propiolamide

Orange oil (82% yield), following general procedure **1**. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34 (d, J = 9.0 Hz, 2H), 7.19 (d, J = 8.7 Hz, 2H), 7.06 (d, J = 8.7 Hz, 2H), 6.95 (d, J = 8.7 Hz, 2H), 4.84 (t, J = 5.7 Hz, 1H), 3.89 (d, J = 5.7 Hz, 2H), 3.82 (s, 3H), 3.71-3.61 (m, 2H), 3.59-3.48 (m, 2H), 1.18 (t, J = 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.6, 153.9, 135.5, 134.6, 133.0, 129.2, 128.2, 118.4, 113.5, 98.7, 89.3, 83.1, 61.4, 54.9, 50.6, 14.8; MS (ESI) 424 ($[M+Na]^+$); HRMS (MALDI) mass calcd. For $C_{22}H_{24}ClNaNO_4$ ($[M+Na]^+$): 424.1286. Found 424.1283.

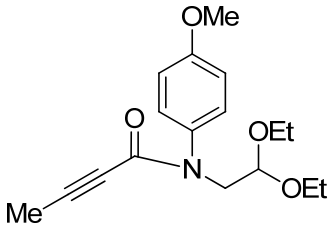


5g

***N*-(2,2-Diethoxyethyl)-*N*-(4-methoxyphenyl)-3-(thiophen-2-yl)propiolamide**

Yellow oil (86% yield), following general procedure **1**. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32-7.29 (m, 3H), 7.04 (d, J = 3.3 Hz, 1H), 6.95-6.87 (m, 3H), 4.84 (t, J = 5.7 Hz, 1H), 3.88 (d, J = 5.7 Hz, 2H), 3.82 (s, 3H), 3.70-3.60 (m, 2H), 3.57-3.478 (m, 2H), 1.17 (t, J = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.7, 154.0, 134.5, 134.4, 129.9, 129.1, 126.8, 119.6, 113.5, 98.7, 86.4, 84.5, 61.5, 55.0, 50.5, 14.8; MS (ESI)

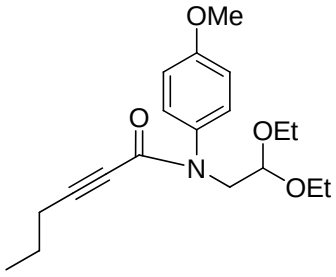
396 ([M+Na]$^+$); HRMS (MALDI) mass calcd. For $C_{20}H_{23}NaNO_4S$ ([M+Na]$^+$): 396.1240. Found 396.1243.



5h

***N*-(2,2-Diethoxyethyl)-*N*-(4-methoxyphenyl)but-2-ynamide**

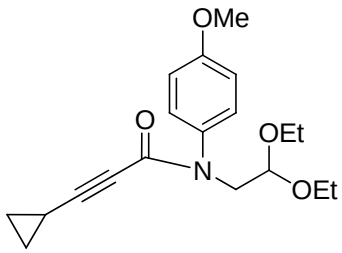
Pale yellow solid (87% yield), following general procedure **1**. M.p. 54-56 ^oC. ^1H NMR (300 MHz, $CDCl_3$) δ 7.23 (d, J = 9.0 Hz, 2H), 6.88 (d, J = 9.0 Hz, 2H), 4.78 (t, J = 5.7 Hz, 1H), 3.83 (s, 3H), 3.79 (d, J = 6.0 Hz, 2H), 3.68-3.58 (m, 2H), 3.56-3.46 (m, 2H), 1.74 (s, 3H), 1.16 (t, J = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.6, 154.6, 135.2, 129.2, 113.7, 99.1, 90.0, 74.0, 61.9, 55.2, 51.0, 15.1, 3.7; MS (ESI) 328 ([M+Na]$^+$); HRMS (MALDI) mass calcd. For $C_{17}H_{23}NaNO_4$ ([M+Na]$^+$): 328.1519. Found 328.1526.



5i

***N*-(2,2-Diethoxyethyl)-*N*-(4-methoxyphenyl)hex-2-ynamide**

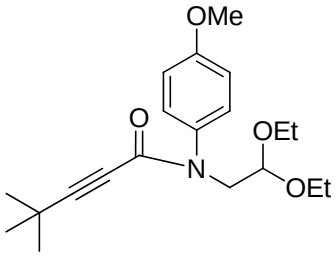
Pale yellow oil (84% yield), following general procedure **1**. ^1H NMR (300 MHz, $CDCl_3$) δ 6.96 (dd, J = 9.0, 2.4 Hz, 2H), 6.61 (dd, J = 9.0, 2.4 Hz, 2H), 4.51-4.48 (m, 1H), 3.53-3.52 (m, 5H), 3.40-3.30 (m, 2H), 3.28-3.17 (m, 2H), 1.81-1.76 (m, 3H), 1.06-0.94 (m, 2H), 0.91-0.85 (m, 6H), 0.46-0.40 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.3, 154.0, 134.7, 128.8, 113.2, 98.5, 93.1, 74.5, 61.1, 54.6, 50.2, 20.2, 19.8, 14.5, 12.3; MS (ESI) 356 ([M+Na]$^+$); HRMS (MALDI) mass calcd. For $C_{19}H_{27}NaNO_4$ ([M+Na]$^+$): 356.1832. Found 356.1842.



5j

3-Cyclopropyl-*N*-(2,2-diethoxyethyl)-*N*-(4-methoxyphenyl)propiolamide

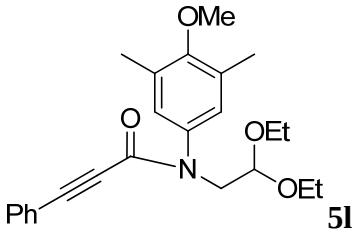
Pale yellow oil (81% yield), following general procedure **1**. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.03 (d, *J* = 8.7 Hz, 2H), 6.70 (d, *J* = 8.7 Hz, 2H), 4.57 (t, *J* = 5.7 Hz, 1H), 3.64 (s, 3H), 3.60 (d, *J* = 5.4 Hz, 2H), 3.49-3.39 (m, 2H), 3.37-3.27 (m, 2H), 0.98 (t, *J* = 6.9 Hz, 6H), 0.79-0.73 (m, 1H), 0.57-0.51 (m, 2H), 0.28-0.23 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.5, 154.2, 135.0, 129.0, 113.3, 98.8, 97.5, 69.6, 61.4, 54.9, 50.4, 14.7, 8.5, -1.1; MS (ESI) 354 ($[M+Na]^+$); HRMS (MALDI) mass calcd. For $C_{19}H_{25}NaNO_4$ ($[M+Na]^+$): 354.1676. Found 354.1680.



5k

***N*-(2,2-Diethoxyethyl)-*N*-(4-methoxyphenyl)-4,4-dimethylpent-2-ynamide**

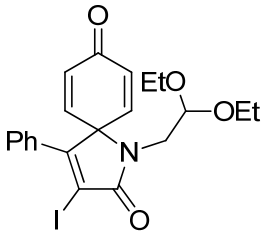
Pale yellow oil (96% yield), following general procedure **1**. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (d, *J* = 9.0 Hz, 2H), 6.88 (d, *J* = 9.0 Hz, 2H), 4.78 (t, *J* = 5.7 Hz, 1H), 3.81 (s, 3H), 3.80 (d, *J* = 6.3 Hz, 2H), 3.69-3.58 (m, 2H), 3.57-3.47 (m, 2H), 1.17 (t, *J* = 7.2 Hz, 6H), 0.97 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.7, 154.9, 135.5, 129.4, 113.6, 101.1, 99.1, 73.4, 61.6, 55.3, 50.7, 29.5, 27.1, 15.0; MS (ESI) 370 ($[M+Na]^+$); HRMS (MALDI) mass calcd. For $C_{20}H_{29}NaNO_4$ ($[M+Na]^+$): 370.1989. Found 370.1998.



5l

***N*-(2,2-Diethoxyethyl)-*N*-(4-methoxy-3,5-dimethylphenyl)-3-phenylpropiolamide**

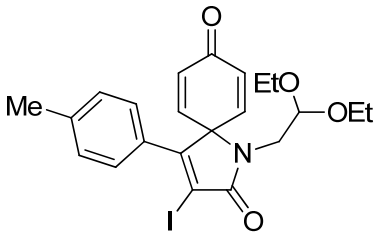
Pale yellow solid (94% yield), following general procedure **1**. M.p. 64-66 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32-7.28 (m, 1 H), 7.28 (t, *J* = 7.5 Hz, 2H), 7.09-7.13 (m, 4 H), 4.86 (t, *J* = 5.7 Hz, 1H), 3.89 (d, *J* = 5.7 Hz, 2H), 3.73 (s, 3H), 3.70-3.62 (m, 2H), 3.59-3.49 (m, 2H), 2.30 (s, 6H), 1.18 (t, *J* = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.0, 153.9, 137.4, 131.8, 130.7, 129.4, 128.1, 127.8, 120.0, 98.8, 90.3, 82.4, 61.5, 59.1, 50.5, 15.5, 14.7; MS (ESI) 418 ($[M+Na]^+$); HRMS (MALDI) mass calcd. For $C_{24}H_{29}NaNO_4$ ($[M+Na]^+$): 419.1989. Found 418.1997.



6a

1-(2,2-Diethoxyethyl)-3-iodo-4-phenyl-1-azaspiro[4.5]deca-3,6,9-triene-2,8-dione

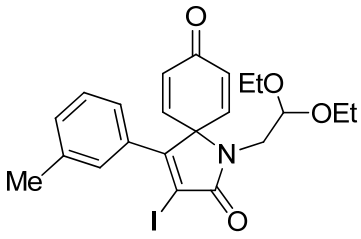
Pale yellow solid (54% yield), following general procedure **2**, method **A**. M.p. 140-142 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43-7.34 (m, 3H), 7.25 (d, *J* = 7.2 Hz, 2H), 6.57 (d, *J* = 9.6 Hz, 2H), 6.38 (d, *J* = 10.2 Hz, 2H), 4.89 (t, *J* = 5.4 Hz, 1H), 3.79-3.69 (m, 2H), 3.60-3.50 (m, 2H), 3.40 (d, *J* = 5.4 Hz, 2H), 1.20 (t, *J* = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 183.8, 167.8, 158.8, 144.1, 132.4, 131.7, 129.9, 128.4, 127.6, 99.1, 97.9, 71.0, 63.0, 45.1, 15.2; MS (ESI) 502 ($[M+Na]^+$); HRMS (MALDI) mass calcd. For $C_{21}H_{22}INaNO_4$ ($[M+Na]^+$): 502.0486. Found 502.0496.



6b

1-(2,2-Diethoxyethyl)-3-iodo-4-(p-tolyl)-1-azaspiro[4.5]deca-3,6,9-triene-2,8-dione

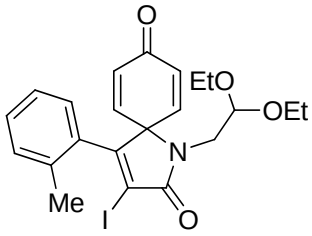
Pale yellow solid (45% yield), following general procedure **2**, method **A**. M.p. 138-140 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.16 (s, 4H), 6.53 (d, *J* = 10.2 Hz, 2H), 6.38 (d, *J* = 10.2 Hz, 2H), 4.89 (t, *J* = 5.7 Hz, 1H), 3.79-3.69 (m, 2H), 3.60-3.50 (m, 2H), 3.38 (d, *J* = 5.7 Hz, 2H), 2.35 (s, 3H), 1.20 (t, *J* = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 183.8, 167.8, 158.7, 144.3, 140.0, 132.2, 129.0, 128.7, 127.4, 99.0, 97.3, 70.9, 62.9, 45.0, 21.2, 15.1; MS (ESI) 516 ($[M+Na]^+$); HRMS (ESI) mass calcd. For $C_{22}H_{24}INaNO_4$ ($[M+Na]^+$): 516.0642. Found 516.0651.



6c

1-(2,2-Diethoxyethyl)-3-iodo-4-(m-tolyl)-1-azaspiro[4.5]deca-3,6,9-triene-2,8-dione

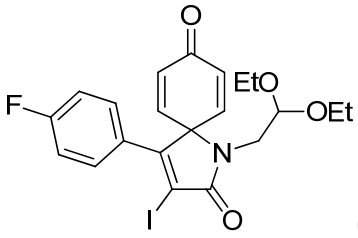
Yellow solid (41% yield), following general procedure **2**, method **A**. M.p. 128-130 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.12-7.04 (m, 2H), 6.92-6.90 (m, 2H), 6.48 (d, *J* = 9.9 Hz, 2H), 6.24 (d, *J* = 9.6 Hz, 2H), 4.76 (t, *J* = 5.4 Hz, 1H), 3.65-3.55 (m, 2H), 3.46-3.36 (m, 2H), 3.27 (d, *J* = 5.1 Hz, 2H), 2.19 (s, 3H), 1.06 (t, *J* = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 183.8, 167.8, 159.0, 144.2, 138.1, 132.2, 131.7, 130.6, 128.3, 128.1, 124.5, 99.0, 97.6, 71.0, 62.9, 45.1, 21.2, 15.1; MS (ESI) 516 ($[M+Na]^+$); HRMS (MALDI) mass calcd. For $C_{22}H_{24}INaNO_4$ ($[M+Na]^+$): 516.0642. Found 516.0660.



6d

1-(2,2-Diethoxyethyl)-3-iodo-4-(o-tolyl)-1-azaspiro[4.5]deca-3,6,9-triene-2,8-dione

White solid (61% yield), following general procedure **2**, method **A**. M.p. 123-124 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28-7.20 (m, 2H), 7.12 (t, *J* = 7.2 Hz, 1H), 6.87 (d, *J* = 7.2 Hz, 1H), 6.71 (dd, *J* = 9.6 Hz, 2.7 Hz, 1H), 6.63 (dd, *J* = 9.6 Hz, 2.7 Hz, 1H), 6.42 (d, *J* = 9.9 Hz, 1H), 6.22 (d, *J* = 10.2 Hz, 1H), 4.90 (t, *J* = 5.4 Hz, 1H), 3.79-3.69 (m, 2H), 3.60-3.50 (m, 2H), 3.47-3.37 (m, 2H), 2.21 (s, 3H), 1.22-1.17 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 183.3, 167.2, 159.8, 143.6, 143.5, 135.1, 132.3, 131.5, 130.22, 130.17, 129.2, 127.8, 124.9, 99.8, 98.8, 72.3, 62.6, 45.1, 19.7, 14.9; MS (ESI) 516 ($[M+Na]^+$); HRMS (MALDI) mass calcd. For $C_{22}H_{24}INaNO_4$ ($[M+Na]^+$): 516.0642. Found [$(M+Na)^+$] 516.0653.

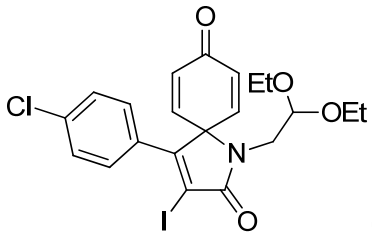


6e

1-(2,2-Diethoxyethyl)-4-(4-fluorophenyl)-3-iodo-1-azaspiro[4.5]deca-3,6,9-triene-2,8-dione(jmq6-74)

White solid (51% yield), following general procedure **2**, method **A**. M.p. 143-145 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29-7.24 (m, 2H), 7.07 (t, *J* = 8.4 Hz, 2H), 6.54 (d, *J* = 10.2 Hz, 2H), 6.39 (d, *J* = 9.9 Hz, 2H), 4.89 (t, *J* = 5.7 Hz, 1H), 3.77-3.69 (m, 2H), 3.60-3.52 (m, 2H), 3.39 (d, *J* = 5.4 Hz, 2H), 1.20 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 183.8, 167.8, 163.4 (d, *J* = 250.1 Hz), 157.9, 144.2, 132.6, 129.9 (d, *J* = 8.5 Hz), 127.9 (d, *J* = 3.4 Hz), 116.0 (d, *J* = 22.1 Hz), 99.2, 98.6, 71.1, 63.2, 45.3,

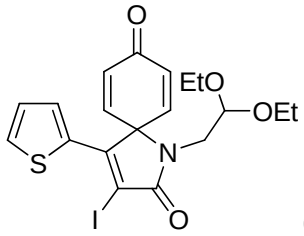
15.3; MS (ESI) 520 ([M+Na]$^+$); HRMS (ESI) mass calcd. For $C_{21}H_{21}FINaNO_4$ ([M+Na]$^+$): 520.0392. Found 520.0404.



6f

4-(4-Chlorophenyl)-1-(2,2-Diethoxyethyl)-3-iodo-1-azaspiro[4.5]deca-3,6,9-triene -2,8-dione

White solid (53% yield), following general procedure **2**, method **A**. M.p. 176-178 ^oC. ^1H NMR (300 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.4 Hz, 2H), 7.22 (d, *J* = 8.9 Hz, 2H), 6.55 (d, *J* = 10.2 Hz, 2H), 6.40 (d, *J* = 9.9 Hz, 2H), 4.89 (t, *J* = 5.4 Hz, 1H), 3.79-3.69 (m, 2H), 3.60-3.49 (m, 2H), 3.39 (d, *J* = 5.4 Hz, 2H), 1.20 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.7, 167.6, 157.6, 144.0, 136.2, 132.6, 130.2, 129.1, 129.0, 99.1, 98.7, 71.0, 63.1, 45.2, 15.2; MS (ESI) 536 ([M+Na]$^+$); HRMS (MALDI) mass calcd. For $C_{21}H_{21}ClINaNO_4$ ([M+Na]$^+$): 536.0096. Found 536.0105.

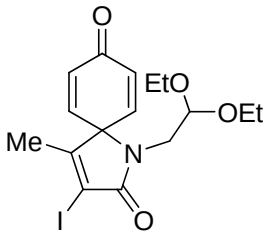


6g

1-(2,2-Diethoxyethyl)-3-iodo-4-(thiophen-2-yl)-1-azaspiro[4.5]deca-3,6,9-triene-2, 8-dione

Yellow solid (51% yield), following general procedure **2**, method **A**. M.p. 89-91 ^oC. ^1H NMR (300 MHz, $CDCl_3$) δ 7.69 (d, *J* = 3.9 Hz, 1H), 7.54 (d, *J* = 8.1 Hz, 1H), 7.11 (t, *J* = 4.5 Hz, 1H), 6.60 (d, *J* = 9.9 Hz, 2H), 6.53 (d, *J* = 10.5 Hz, 2H), 4.92 (t, *J* = 5.4 Hz, 1H), 3.79-3.69 (m, 2H), 3.59-3.49 (m, 2H), 3.34 (d, *J* = 5.4 Hz, 2H), 1.19 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.2, 168.0, 150.2, 145.2, 132.6, 132.4,

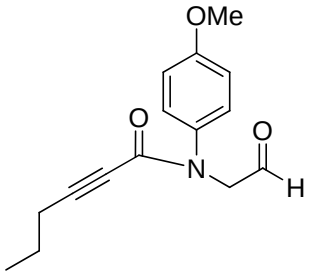
129.8, 129.5, 127.4, 99.2, 93.5, 69.7, 63.4, 44.7, 15.2; MS (ESI) 508 ($[M+Na]^+$); HRMS (MALDI) mass calcd. For $C_{19}H_{20}INaNO_4S$ ($[M+Na]^+$): 508.0050. Found 508.0056.



6h

1-(2,2-Diethoxyethyl)-3-iodo-4-methyl-1-azaspiro[4.5]deca-3,6,9-triene-2,8-dione

Pale yellow solid (47% yield), following general procedure **2**, method **A**. M.p. 94-96 ^oC. ^1H NMR (300 MHz, $CDCl_3$) δ 6.50 (d, J = 10.5 Hz, 2H), 6.45 (d, J = 10.5 Hz, 2H), 4.82 (t, J = 5.4 Hz, 1H), 3.78-3.67 (m, 2H), 3.58-3.48 (m, 2H), 3.37 (d, J = 5.7 Hz, 2H), 1.89 (s, 3H), 1.19 (t, J = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.9, 167.7, 156.9, 145.0, 132.1, 99.0, 95.8, 70.8, 62.8, 45.1, 15.2, 15.0; MS (ESI) 440 ($[M+Na]^+$); HRMS (MALDI) mass calcd. For $C_{16}H_{20}INaNO_4$ ($[M+Na]^+$): 440.0329. Found 440.0341.

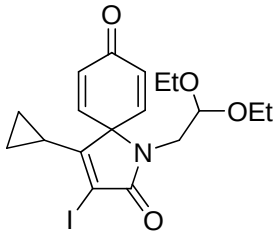


6i

***N*-(4-Methoxyphenyl)-*N*-(2-oxoethyl)hex-2-ynamide**

Pale yellow oil (79% yield), following general procedure **3**. ^1H NMR (300 MHz, $CDCl_3$) δ 9.61 (s, 1H), 7.27 (d, J = 8.7 Hz, 2H), 6.90 (d, J = 8.7 Hz, 2H), 4.48 (s, 2H), 3.81 (s, 3H), 2.09 (t, J = 6.9 Hz, 2H), 1.37-1.25 (m, 2H), 0.72 (t, J = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.1, 159.3, 155.0, 134.8, 129.2, 114.3, 95.4, 74.3, 58.8, 55.5, 20.9, 20.7, 13.1; MS (ESI) 260 ($[M+H]^+$); HRMS (MALDI) mass calcd. For

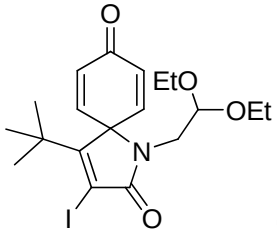
$C_{15}H_{18}NO_3$ ($[M+H]^+$): 260.1281. Found 260.1284.



6j

4-Cyclopropyl-1-(2,2-diethoxyethyl)-3-iodo-1-azaspiro[4.5]deca-3,6,9-triene-2,8-dione

Pale yellow solid (35% yield), following general procedure **2**, method **A**. M.p. 72-74 ^oC. ^1H NMR (300 MHz, $CDCl_3$) δ 6.50 (s, 4H), 4.82 (t, *J* = 5.7 Hz, 1H), 3.76-3.66 (m, 2H), 3.57-3.47 (m, 2H), 3.31 (d, *J* = 5.4 Hz, 2H), 1.38-1.26 (m, 3H), 1.18 (t, *J* = 7.2 Hz, 6H), 0.99-0.94 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.1, 167.9, 158.9, 145.1, 132.0, 99.0, 88.0, 71.4, 62.8, 44.8, 15.0, 11.1, 7.6; MS (ESI) 466 ($[M+Na]^+$); HRMS (MALDI) mass calcd. For $C_{18}H_{22}INaNO_4$ ($[M+Na]^+$): 466.0486. Found 466.0490.

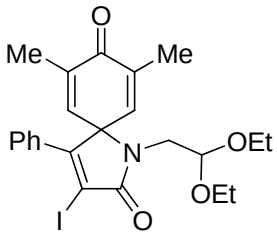


6k

4-(tert-Butyl)-1-(2,2-diethoxyethyl)-3-iodo-1-azaspiro[4.5]deca-3,6,9-triene-2,8-dione

Modified general procedure **2**, method **A**, N-(2,2-Diethoxyethyl)-N-(4-methoxyphenyl)-4,4- dimethylpent-2-ynamide (1.39 g, 4.0 mmol), CH_2Cl_2 135 mL, ICl (408 uL, 8.0 mmol) in 80 mL CH_2Cl_2, stirred for 30 min, then the solution was slowly warmed to room temperature for 12h, quenched with saturated aqueous Na_2SO_3 solution and extracted three times with CH_2Cl_2. The combined organic layers were dried over Na_2SO_4, concentrated in vacuo. The residue was purified by column chromatography on silica gel (EtOAc:PE, 1:3) to afford the product as a pale yellow

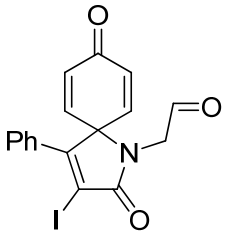
solid (380 mg, 28% yield); M.p. 148-150 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.50 (s, 4H), 4.82 (t, *J* = 5.4 Hz, 1H), 3.76-3.66 (m, 2H), 3.55-3.45 (m, 2H), 3.15 (d, *J* = 5.7 Hz, 2H), 1.40 (s, 9H), 1.18 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.2, 168.2, 166.1, 145.0, 132.2, 99.1, 96.1, 70.5, 63.3, 43.9, 36.0, 28.4, 15.2; MS (ESI) 482 ($[M+Na]^+$); HRMS (MALDI) mass calcd. For $C_{19}H_{26}INaNO_4$ ($[M+Na]^+$): 482.0799. Found 482.0801.



6l

1-(2,2-Diethoxyethyl)-3-iodo-7,9-dimethyl-4-phenyl-1-azaspiro[4.5]deca-3,6,9-triene-2,8-dione

Pale yellow solid (54% yield), following general procedure **2**, method **A**. M.p. 153-155 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27-7.21 (m, 3H), 7.13-7.10 (m, 2H), 6.22 (s, 2H), 4.80 (t, *J* = 5.7 Hz, 1H), 3.71-3.60 (m, 2H), 3.51-3.41 (m, 2H), 3.26 (d, *J* = 5.4 Hz, 2H), 1.77 (s, 3H), 1.12 (t, *J* = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 185.3, 167.8, 160.0, 139.0, 138.8, 132.2, 129.6, 128.3, 127.5, 99.4, 96.9, 71.5, 63.0, 45.1, 16.0, 15.3; MS (ESI) 530 ($[M+Na]^+$); HRMS (MALDI) mass calcd. For $C_{23}H_{26}INaNO_4$ ($[M+Na]^+$): 530.0799. Found 530.0798.

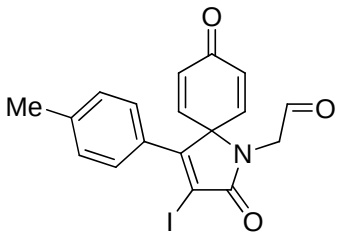


1a

2-(3-Iodo-2,8-dioxo-4-phenyl-1-azaspiro[4.5]deca-3,6,9-trien-1-yl)acetaldehyde

Pale yellow solid (86% yield), following general procedure **3**. M.p. 93-95 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.85 (s, 1H), 7.43-7.37 (m, 3H), 7.31 (d, *J* = 6.9 Hz, 2H), 6.64 (d, *J* = 9.6 Hz, 2H), 6.39 (d, *J* = 9.9 Hz, 2H), 4.21 (s, 2H); ^{13}C NMR (75 MHz,

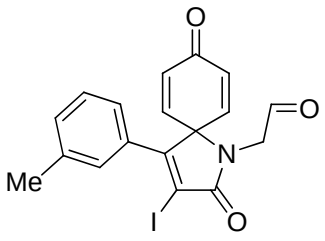
$CDCl_3$) δ 194.9, 183.5, 167.5, 159.3, 143.1, 133.0, 131.6, 130.2, 128.6, 127.5, 96.9, 70.1, 50.5; IR (thin film): ν_{max} (cm^{-1}) = 2919, 1694, 1667, 1628, 1383, 1112, 1059, 724, 698; MS (ESI) 406 ($[M+H]^+$); HRMS (ESI) mass calcd. For $C_{17}H_{13}INO_3$ ($[M+H]^+$): 405.9935. Found 405.9939.



1b

2-(3-Iodo-2,8-dioxo-4-(p-tolyl)-1-azaspiro[4.5]deca-3,6,9-trien-1-yl)acetaldehyde

White solid (96% yield), following general procedure **3**. M.p. 163-164 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.58 (s, 1H), 7.21 (m, 4H), 6.63 (d, *J* = 9.6 Hz, 2H), 6.40 (d, *J* = 9.6 Hz, 2H), 4.21 (s, 2H), 2.36 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.0, 183.6, 167.6, 159.2, 143.3, 140.5, 132.9, 129.3, 128.6, 127.4, 96.3, 70.0, 50.5, 21.3; IR (thin film): ν_{max} (cm^{-1}) = 2921, 2854, 1733, 1699, 1667, 1627, 1507, 1386, 1060, 998, 873, 823, 749; MS (ESI) 420 ($[M+H]^+$); HRMS (ESI) mass calcd. For $C_{18}H_{15}INO_3$ 420.0091 ($[M+H]^+$). Found 420.0083.

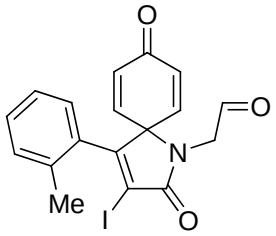


1c

2-(3-Iodo-2,8-dioxo-4-(m-tolyl)-1-azaspiro[4.5]deca-3,6,9-trien-1-yl)acetaldehyde

White solid (72% yield), following general procedure **3**. M.p. 80-82 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.58 (s, 1H), 7.27-7.25 (m, 2H), 7.09-7.06 (m, 2H), 6.60 (d, *J* = 9.9 Hz, 2H), 6.40 (d, *J* = 10.2 Hz, 2H), 4.19 (s, 2H), 2.36 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 194.9, 183.6, 167.6, 159.6, 143.2, 138.4, 132.9, 131.5, 131.0, 128.5, 128.1, 124.5, 96.7, 70.1, 50.5, 21.3; IR (thin film): ν_{max} (cm^{-1}) = 2922, 2854, 1733, 1700, 1668, 1629, 1507, 1385, 1061, 874, 823, 750; MS (ESI) 420 ($[M+H]^+$); HRMS

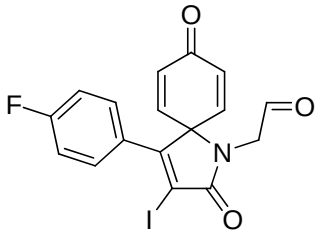
(MALDI) mass calcd. For $C_{18}H_{15}INO_3$ ($[M+H]^+$): 420.0091. Found 420.0105.



1d

2-(3-Iodo-2,8-dioxo-4-(o-tolyl)-1-azaspiro[4.5]deca-3,6,9-trien-1-yl)acetaldehyde

White solid (67% yield), following general procedure **3**. M.p. 210-211 ^oC. ^1H NMR (300 MHz, d_6-DMSO) δ 9.51 (s, 1H), 7.26 (s, 2H), 7.15 (s, 1H), 6.99 (d, J = 8.1 Hz, 2H), 6.87 (d, J = 9.9 Hz, 2H), 6.35 (d, J = 9.6 Hz, 1H), 6.21(d, J = 9.6 Hz, 1H), 4.20 (s, 2H), 2.19 (s, 3H); ^{13}C NMR (75 MHz, d_6-DMSO) δ 198.3, 183.8, 167.4, 160.0, 144.2, 144.0, 135.5, 132.5, 132.0, 131.0, 130.4, 129.4, 128.4, 125.2, 100.8, 71.7, 50.9, 19.7; IR (thin film): ν_{max} (cm^{-1}) = 2921, 1738, 1700, 1663, 1626, 1423, 1381, 1115, 1059, 741, 700; MS (ESI) 420 ($[M+H]^+$); HRMS (MALDI) mass calcd. For $C_{18}H_{15}INO_3$ ($[M+H]^+$): 420.0091. Found 420.0101.

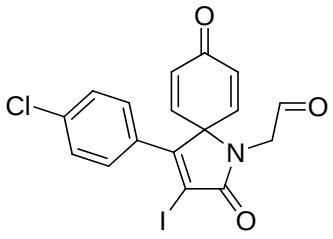


1e

2-(4-(4-Fluorophenyl)-3-iodo-2,8-dioxo-1-azaspiro[4.5]deca-3,6,9-trien-1-yl)aceta ldehyde

White solid (96% yield), following general procedure **3**. M.p. 186-188 ^oC. ^1H NMR (300 MHz, $CDCl_3$) δ 9.56 (s, 1H), 7.33-7.29 (m, 2H), 7.07 (t, J = 8.4 Hz, 2H), 6.59 (d, J = 9.6 Hz, 2H), 6.38 (d, J = 10.2 Hz, 2H), 4.18 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 194.7, 183.4, 167.4, 163.5 (d, J = 250.7 Hz), 158.3, 143.1, 133.1, 129.8 (d, J = 8.6 Hz), 127.6 (d, J = 4.0 Hz), 116.1 (d, J = 22.1 Hz), 97.5, 70.1, 50.6; IR (thin film): ν_{max} (cm^{-1}) = 2920, 2127, 1706, 1663, 1627, 1503, 1383, 1231, 1159, 1062, 843; MS (ESI) 478 ($[M+CH_3OH+Na]^+$); HRMS (ESI) mass calcd. For $C_{18}H_{15}FINaNO_4$ ($[M+CH_3OH$

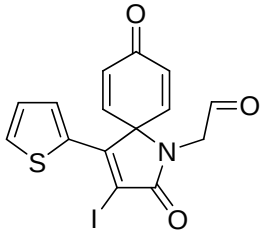
+Na]$^+$): 477.9922. Found 477.9918.



1f

2-(4-(4-Chlorophenyl)-3-iodo-2,8-dioxo-1-azaspiro[4.5]deca-3,6,9-trien-1-yl)acetaldehyde

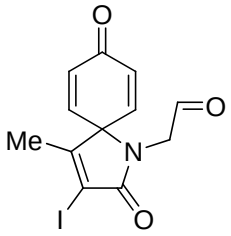
White solid (82% yield), following general procedure **3**. M.p. 180-181 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.58 (s, 1H), 7.38 (d, *J* = 8.7 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 6.64 (d, *J* = 10.2 Hz, 2H), 6.41 (d, *J* = 9.9 Hz, 2H), 4.23 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 194.9, 182.3, 167.2, 158.0, 142.9, 136.2, 133.0, 129.9, 129.0, 128.9, 97.6, 69.9, 50.5; IR (thin film): ν_{max} (cm^{-1}) = 2912, 1704, 1668, 1626, 1385, 1091; MS (ESI) 440 ([M+H]$^+$); HRMS (MALDI) mass calcd. For $C_{17}H_{12}ClINO_3$ ([M+H]$^+$): 439.9567. Found 439.9562.



1g

2-(3-Iodo-2,8-dioxo-4-(thiophen-2-yl)-1-azaspiro[4.5]deca-3,6,9-trien-1-yl)acetaldehyde

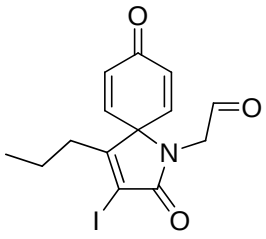
Yellow solid (81% yield), following general procedure **3**. M.p. 165-167 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.56 (s, 1H), 7.69 (d, *J* = 3.6 Hz, 1H), 7.57 (d, *J* = 5.1 Hz, 1H), 7.12 (t, *J* = 5.2 Hz, 1H), 6.69 (d, *J* = 9.6 Hz, 2H), 6.53 (d, *J* =9.6 Hz, 2H), 4.20 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.2, 183.6, 167.4, 150.3, 144.2, 132.6, 132.1, 129.8, 129.6, 127.4, 91.8, 68.6, 49.7; IR (thin film): ν_{max} (cm^{-1}) = 2919, 1665, 1628, 1383, 1062, 711; MS (ESI) 410 ([M-H]$^-$); HRMS (MALDI) mass calcd. For $C_{15}H_{11}INO_3S$ ([M+H]$^+$): 411.9499. Found 411.9514.



1h

2-(3-Iodo-4-methyl-2,8-dioxo-1-azaspiro[4.5]deca-3,6,9-trien-1-yl)acetaldehyde

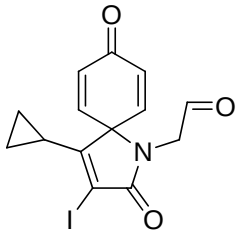
Pale yellow solid (96% yield), following general procedure **3**. M.p. 92-94 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.56 (s, 1H), 6.51 (s, 4H), 4.20 (s, 2H), 1.94 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.3, 183.5, 167.4, 157.8, 143.7, 132.8, 94.9, 70.0, 50.6, 15.3; IR (thin film): ν_{max} (cm^{-1}) = 2923, 1733, 1690, 1665, 1626, 1387, 1063; MS (ESI) 344 ($[M+H]^+$); HRMS (ESI) mass calcd. For $C_{12}H_{11}INO_3$ 343.9778 ($[M+H]^+$). Found 343.9785.



1i

2-(3-Iodo-2,8-dioxo-4-propyl-1-azaspiro[4.5]deca-3,6,9-trien-1-yl)acetaldehyde

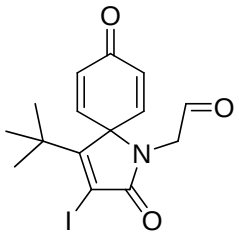
White solid (54% yield), following general procedure **2**, method **B**. M.p. 150-152 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.55 (s, 1H), 6.53-6.45 (m, 4H), 4.16 (s, 2H), 2.18 (t, J = 7.8 Hz, 2H), 1.59-1.48 (m, 2H), 0.97 (t, J = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.1, 183.8, 167.6, 161.2, 143.6, 132.9, 95.4, 70.2, 50.6, 31.5, 21.6, 14.2; IR (thin film): ν_{max} (cm^{-1}) = 2926, 2853, 1699, 1667, 1626, 1414, 1385, 1307, 1058, 850; MS (ESI) 372 ($[M+H]^+$); HRMS (MALDI) mass calcd. For $C_{14}H_{15}INO_3$ ($[M+H]^+$): 372.0091. Found 372.0103.



1j

2-(4-Cyclopropyl-3-iodo-2,8-dioxo-1-azaspiro[4.5]deca-3,6,9-trien-1-yl)acetal-dehyde

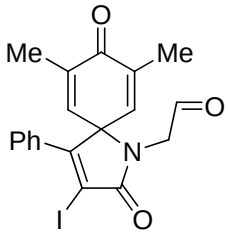
Yellow solid (81% yield), following general procedure **3**. M.p. 166-168 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.53 (s, 1H), 6.55-6.48 (m, 4H), 4.12 (s, 2H), 1.38-136 (m, 3H), 1.02-0.98 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.2, 183.9, 167.8, 160.2, 144.1, 132.9, 88.0, 70.5, 50.4, 11.9, 8.0; IR (thin film): ν_{max} (cm^{-1}) = 2921, 2857, 1737, 1699, 1666, 1627, 1414, 1384, 1308, 1058, 1110; MS (ESI) 368 ($[M-H]^-$); HRMS (MALDI) mass calcd. For $C_{14}H_{13}INO_3$ ($[M+H]^+$): 369.9935. Found 369.9938.



1k

2-(4-(tert-Butyl)-3-iodo-2,8-dioxo-1-azaspiro[4.5]deca-3,6,9-trien-1-yl)acetal-dehyde

Pale yellow solid (67% yield), following general procedure **3**. M.p. 187-189 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.48 (s, 1H), 6.59 (d, J = 10.2 Hz, 2H), 6.48 (d, J = 9.9 Hz, 2H), 4.00 (s, 2H), 1.42 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.0, 183.8, 167.8, 166.9, 144.3, 132.2, 95.2, 69.6, 49.2, 36.0, 28.4; IR (thin film): ν_{max} (cm^{-1}) = 2944, 2866, 1723, 1694, 1664, 1625, 1417, 1390, 1307, 1052, 1009, 878, 850, 796, 750; MS (ESI) 384 ($[M-H]^-$); HRMS (MALDI) mass calcd. For $C_{15}H_{17}INO_3$ ($[M+H]^+$): 386.0248. Found 386.0256.

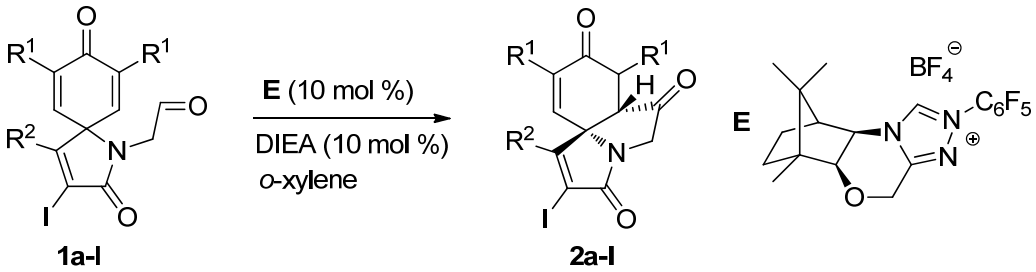


1l

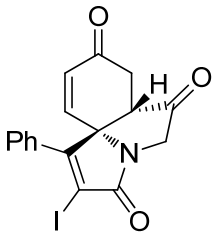
2-(3-Iodo-7,9-dimethyl-2,8-dioxo-4-phenyl-1-azaspiro[4.5]deca-3,6,9-trien-1-yl) acetaldehyde

Off white solid (96% yield), following general procedure **3**. M.p. 214-216 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.56 (s, 1H), 7.42-7.34 (m, 3H), 7.26-7.24 (m, 2H), 6.32 (s, 2H), 4.13 (s, 2H), 1.85 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.2, 184.9, 167.3, 160.3, 140.0, 137.5, 131.8, 129.8, 128.4, 127.3, 95.8, 70.5, 50.3, 15.8; IR (thin film): ν_{max} (cm^{-1}) = 2920, 2838, 1729, 1692, 1668, 1638, 1388, 1037, 913, 756, 699; MS (ESI) 434 ($[M+H]^+$); HRMS (MALDI) mass calcd. For $C_{19}H_{17}INO_3$ ($[M+H]^+$): 434.0248. Found 434.0255.

General procedure for desymmetrization of cyclohexadienones via intramolecular Stetter reaction



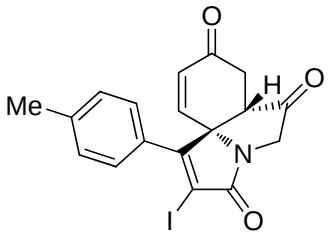
A flame-dried Schlenk tube was cooled to room temperature and filled with argon. To this flask were added triazolium salt **E** (9.7 mg, 0.02 mmol, 10 mol%), *o*-xylene (2.0 mL), DIEA (3.3 uL, 0.02 mmol, 10 mol%). The reaction mixture was stired at 25°C for 20 minutes. The substrate (0.2 mmol) was then added. After the reaction was complete (monitored by TLC), the reaction mixture was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel to afford the product.



2a

(6aS,10aR)-2-Iodo-1-phenyl-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

White solid, 81% yield, 91% ee [Daicel Chiralcel OD-H, *n*-hexane/2-propanol = 60/40, v = 0.7 mL·min^{-1}, λ = 254 nm, t (major) = 22.9 min, t (minor) = 30.4 min]; $[\alpha]_D^{20}$ = +94 (c = 0.1, $CHCl_3$). M.p. 225 °C (decomposed). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46-7.44 (m, 3H), 7.30-7.29 (m, 2H), 6.62 (d, J = 10.2 Hz, 2H), 6.20 (d, J = 9.9 Hz, 2H), 4.45 (d, J = 18.9 Hz, 1H), 3.63 (d, J = 18.9 Hz, 1H), 2.96-2.87 (m, 2H), 2.12 (dd, J = 18.0, 6.6 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.8, 192.5, 169.5, 164.4, 143.2, 133.7, 132.4, 130.2, 129.1, 127.1, 97.5, 72.4, 50.9, 50.1, 32.7; IR (thin film): ν_{max} (cm^{-1}) = 2922, 1765, 1707, 1682, 1632, 1382, 1346, 1255, 1098, 765, 696; MS (ESI) 406 ([M+H]$^+$); HRMS (MALDI) calcd for $C_{17}H_{13}INO_3$ ([M+H]$^+$): 405.9935. Found: 405.9947.

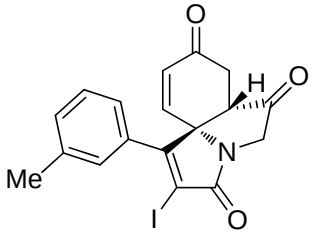


2b

(6aS,10aR)-2-Iodo-1-(p-tolyl)-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

White solid, 55% yield, 80% ee [Daicel Chiralcel OD-H, *n*-hexane/2-propanol = 60/40, v = 0.8 mL·min^{-1}, λ = 254 nm, t (major) = 26.4 min, t (minor) = 34.9 min]; $[\alpha]_D^{20}$ = +62 (c = 0.2, $CHCl_3$). M.p. 174-176 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 (d, J = 7.5 Hz, 2H), 7.22 (d, J = 7.5 Hz, 2H), 6.63 (d, J = 10.2 Hz, 2H), 6.24 (d, J = 9.9 Hz, 2H), 4.47 (d, J = 19.2 Hz, 1H), 3.64 (d, J = 18.6 Hz, 2H), 2.96 (d, J = 17.7 Hz, 1H), 2.86 (d, J = 5.7 Hz, 1H), 2.40 (s, 3H), 2.16 (dd, J = 18.0, 6.9 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.9, 192.6, 169.7, 164.6, 143.4, 140.6, 133.7, 129.8, 129.4,

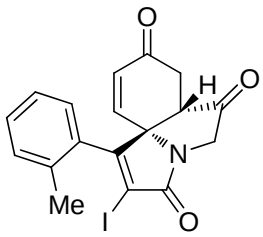
127.0, 97.0, 72.5, 51.2, 50.2, 32.8, 21.4; IR (thin film): ν_{max} (cm^{-1}) = 2921, 1766, 1633, 1381, 1078, 778, 694; MS (ESI) 420 ($[M+H]^+$); HRMS (MALDI) calcd for $C_{18}H_{15}INO_3$ ($[M+H]^+$): 420.0091. Found: ($[M+H]^+$) 420.0103.



2c

(6aS,10aR)-2-Iodo-1-(m-tolyl)-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

Pale yellow solid, 60% yield, 71% ee [Daicel Chiralcel OD-H, *n*-hexane/2-propanol = 60/40, v = 0.8 mL·min^{-1}, λ = 254 nm, t (major) = 24.7 min, t (minor) = 30.1 min]; $[\alpha]_D^{20}$ = +75 (c = 0.2, $CHCl_3$). M.p. 176-178 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38-7.28 (m, 2H), 7.10-7.08 (m, 2H), 6.62 (d, J = 10.5 Hz, 2H), 6.22 (d, J = 10.2 Hz, 2H), 4.47 (d, J = 19.2 Hz, 1H), 3.64 (d, J = 19.2 Hz, 2H), 2.97 (d, J = 18.6 Hz, 1H), 2.87 (d, J = 6.9 Hz, 1H), 2.39 (s, 3H), 2.17 (dd, J = 18.0, 6.9 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.8, 192.6, 169.7, 164.8, 143.3, 139.0, 133.7, 132.4, 131.1, 129.1, 127.6, 124.1, 97.3, 72.5, 51.1, 50.2, 32.8, 21.4; IR (thin film): ν_{max} (cm^{-1}) = 2922, 2853, 1768, 1672, 1634, 1382, 1256, 1092, 777, 691; MS (ESI) 420 ($[M+H]^+$); HRMS (MALDI) calcd for $C_{18}H_{15}INO_3$ ($[M+H]^+$): 420.0091. Found: 420.0106.

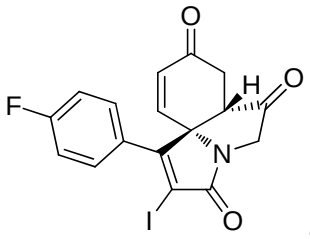


2d

(6aS,10aR)-2-Iodo-1-(*o*-tolyl)-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

White solid, 52% yield, 89% ee [Daicel Chiralcel OD-H, *n*-hexane/2-propanol = 60/40, v = 0.8 mL·min^{-1}, λ = 254 nm, t_R (major) = 22.8, 27.7 min, t_R (minor) = 34.9, 39.3 min]; $[\alpha]_D^{20}$ = +110 (c = 0.2, CH_2Cl_2). M.p. 227-229 °C. ^{1}H NMR (300 MHz,

$CDCl_3$) δ 7.41-7.20 (m, 6H), 7.07 (d, *J* = 7.5 Hz, 1H), 6.92 (d, *J* = 7.8 Hz, 1H), 6.65 (d, *J* = 10.2 Hz, 1H), 6.54 (d, *J* = 9.9 Hz, 1H), 6.19 (d, *J* = 10.2 Hz, 1H), 6.06 (d, *J* = 10.2 Hz, 1H), 4.53 (d, *J* = 6.9 Hz, 1H), 4.46 (d, *J* = 7.2 Hz, 1H), 3.66 (d, *J* = 19.2 Hz, 2H), 3.10-3.04 (m, 2H), 2.98-2.87 (m, 2H), 2.49 (dd, *J* = 18.3, 7.5 Hz, 1H), 2.33 (s, 3H), 2.27 (s, 3H), 2.02 (dd, *J* = 17.7, 7.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.65, 208.64, 208.37, 192.3, 192.2, 169.29, 169.27, 165.9, 164.9, 143.1, 135.3, 134.3, 133.8, 133.3, 131.8, 131.3, 131.2, 131.0, 130.2, 129.9, 127.6, 126.3, 126.2, 126.0, 99.65, 99.55, 73.4, 73.1, 51.3, 51.0, 50.2, 50.1, 32.9, 32.1, 20.0, 19.8; IR (thin film): ν_{max} (cm^{-1}) = 2922, 1767, 1685, 1623, 1363, 1255, 1083, 986, 765, 693; MS (ESI) 420 ($[M+H]^+$); HRMS (MALDI) calcd for $C_{18}H_{15}INO_3$ ($[M+H]^+$): 420.0091. Found: 420.0102.

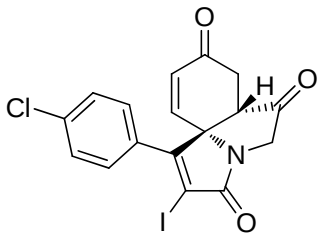


2e

(6aS,10aR)-1-(4-Fluorophenyl)-2-iodo-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

White solid, 55% yield, 84% ee [Daicel Chiralcel OD-H, *n*-hexane/2-propanol = 60/40, *v* = 0.8 mL·min^{-1}, λ = 254 nm, t (major) = 30.3 min, t (minor) = 38.6 min]; $[\alpha]_D^{20}$ = +128 (c = 0.1, $CHCl_3$). M.p. 195-197 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.33 (m, 2H), 7.18 (t, *J* = 8.4 Hz, 2H), 6.63 (dd, *J* = 9.9, 1.2 Hz, 2H), 6.26 (d, *J* = 10.2 Hz, 2H), 4.48 (dd, *J* = 18.9, 1.5 Hz, 1H), 3.66 (d, *J* = 18.9 Hz, 2H), 2.99 (d, *J* = 18.0 Hz, 1H), 2.86 (d, *J* = 6.9 Hz, 1H), 2.15 (ddd, *J* = 18.0, 7.2, 1.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.5, 192.2, 169.4, 163.5 (d, *J* = 251.0 Hz), 162.3, 143.1, 134.0, 129.4 (d, *J* = 8.6 Hz), 128.4 (d, *J* = 3.4 Hz), 116.6 (d, *J* = 21.9 Hz), 98.2 (d, *J* = 0.7 Hz), 72.4 (d, *J* = 0.8 Hz), 51.1, 50.2, 32.7, 25.3; IR (thin film): ν_{max} (cm^{-1}) = 2921, 2853, 1765, 1707, 1638, 1504, 1382, 1346, 1232, 1160, 1099, 985, 848, 784, 697; MS (ESI) 424 ($[M+H]^+$); HRMS (MALDI) calcd for $C_{17}H_{12}FINO_3$ ($[M+H]^+$): 423.9840.

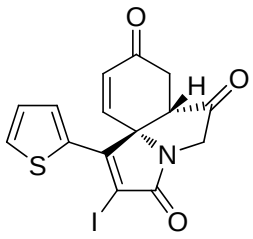
Found: 423.9848.



2f

(6aS,10aR)-1-(4-Chlorophenyl)-2-iodo-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

Pale yellow solid, 54% yield, 86% ee [Daicel Chiralcel OD-H, *n*-hexane /2-propanol = 60/40, *v* = 0.8 mL·min^{-1}, λ = 254 nm, t (major) = 33.8 min, t (minor) = 42.3 min]; $[\alpha]_D^{20}$ = +105 (c = 0.1, $CHCl_3$). M.p. 107-109 ^oC. ^1H NMR (300 MHz, $CDCl_3$) δ 7.47 (d, *J* = 7.8 Hz, 2H), 7.28 (d, *J* = 7.5 Hz, 2H), 6.62 (d, *J* = 10.2 Hz, 1H), 6.26 (d, *J* = 9.9 Hz, 1H), 4.48 (d, *J* = 18.6 Hz, 1H), 3.66 (d, *J* = 18.9 Hz, 1H), 3.00 (d, *J* = 17.7 Hz, 1H), 2.84 (d, *J* = 6.0 Hz, 1H), 2.16 (dd, *J* = 18.0, 6.9 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.4, 192.1, 169.3, 163.1, 142.9, 136.7, 134.1, 130.8, 129.7, 128.6, 98.4, 72.4, 51.2, 50.2, 32.7; IR (thin film): ν_{max} (cm^{-1}) = 2920, 1767, 1683, 1633, 1428, 1380, 1092, 876, 615; MS (ESI) 440 ($[M+H]^+$); HRMS (MALDI) calcd for $C_{17}H_{12}ClINO_3$ ($[M+H]^+$): 439.9545. Found: 439.9557.

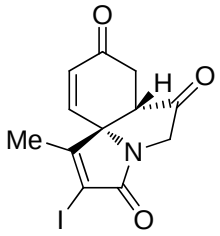


2g

(6aS,10aR)-2-Iodo-1-(thiophen-2-yl)-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

Yellow solid, 54% yield, 75% ee [Daicel Chiralcel OD-H, *n*-hexane/2-propanol = 60/40, *v* = 0.8 mL·min^{-1}, λ = 254 nm, t (major) = 39.1 min, t (minor) = 64.9 min]; $[\alpha]_D^{20}$ = +70 (c = 0.1, $CHCl_3$). M.p. 201-202 ^oC. ^1H NMR (400 MHz, $CDCl_3$) δ 7.80

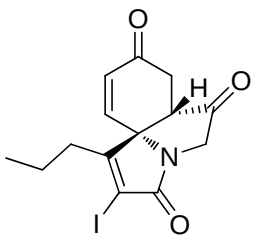
(d, *J* = 4.0 Hz, 1H), 7.61 (d, *J* = 4.8 Hz, 1H), 7.20 (t, *J* = 4.8 Hz, 1H), 6.69 (dd, *J* = 10.0, 0.8 Hz, 1H), 6.40 (d, *J* = 10.4 Hz, 1H), 4.49 (dd, *J* = 19.2, 4.0 Hz, 1H), 3.63 (d, *J* = 18.8 Hz, 1H), 3.13 (d, *J* = 18.0 Hz, 1H), 2.91 (d, *J* = 7.6 Hz, 1H), 2.73 (dd, *J* = 18.4, 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.2, 192.9, 170.0, 155.6, 144.1, 134.6, 133.0, 130.2, 130.0, 127.9, 93.9, 72.2, 51.2, 50.0, 33.4; IR (thin film): ν_{max} (cm^{-1}) = 2920, 2850, 1766, 1692, 1675, 1631, 1384, 1254, 1096, 712; MS (ESI) 412 ($[M+H]^+$); HRMS (MALDI) calcd for $C_{15}H_{11}INO_3S$ ($[M+H]^+$): 410.9499. Found: 411.9510.



2h

(6aS,10aR)-2-Iodo-1-methyl-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

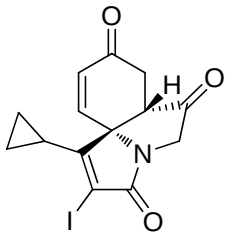
White solid, 58% yield, 86% ee [Daicel Chiralpak IC, *n*-hexane/2-propanol = 60/40, *v* = 0.8 mL·min^{-1}, λ = 254 nm, t (major) = 50.3 min, t (minor) = 69.9 min]; $[\alpha]_D^{20}$ = +182 (c = 0.2, $CHCl_3$). M.p. 83-85 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.42 (dd, *J* = 10.5, 0.9 Hz, 1H), 6.29 (d, *J* = 10.5 Hz, 1H), 4.42 (d, *J* = 18.9 Hz, 1H), 3.59 (d, *J* = 19.2 Hz, 1H), 3.21 (dt, *J* = 17.1, 4.5 Hz, 1H), 2.74-2.67 (m, 2H), 2.19 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.8, 192.4, 170.0, 163.0, 144.2, 133.8, 96.0, 71.8, 50.8, 50.2, 32.6, 17.1; IR (thin film): ν_{max} (cm^{-1}): 2921, 2848, 1766, 1683, 1633, 1383, 1259, 1104; MS (ESI) 365 ($[M+Na]^+$); HRMS (MALDI) calcd for $C_{12}H_{10}INaNO_3$ ($[M+Na]^+$): 365.9598. Found: 365.9603.



2i

(6aS,10aR)-2-Iodo-1-propyl-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

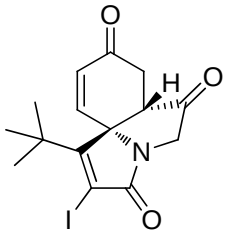
Pale yellow solid, 60% yield, 85% ee [Daicel Chiralcel OD-H, *n*-hexane/2-propanol = 60/40, *v* = 0.8 mL·min^{-1}, λ = 254 nm, t (major) = 19.4 min, t (minor) = 23.7 min]; $[\alpha]_D^{20}$ = +236 (c = 0.1, $CHCl_3$). M.p. 186-187 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.41 (dd, *J* = 10.4, 1.2 Hz, 1H), 6.28 (d, *J* = 10.4 Hz, 1H), 4.41 (d, *J* = 18.8 Hz, 1H), 3.58 (d, *J* = 19.2 Hz, 1H), 3.21 (dt, *J* = 16.8, 4.8 Hz, 1H), 2.77-2.70 (m, 2H), 2.48-2.40 (m, 2H), 1.68-1.59 (m, 2H), 1.05 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.9, 192.5, 169.9, 166.4, 144.2, 133.5, 96.2, 71.9, 50.7, 49.9, 32.84, 32.83, 21.7, 14.4; IR (thin film): ν_{max} (cm^{-1}) = 2924, 1768, 1696, 1634, 1260, 1079, 778, 693; MS (ESI) 372 ([M+H]$^+$); HRMS (MALDI) calcd for $C_{14}H_{15}INO_3$ ([M+H]$^+$): 372.0091. Found: 372.0105.



2j

(6aS,10aR)-1-Cyclopropyl-2-iodo-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

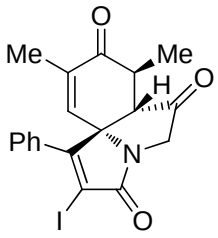
White solid, 85% yield, 88% ee [Daicel Chiralcel OD-H, *n*-hexane/2-propanol = 60/40, *v* = 0.8 mL·min^{-1}, λ = 254 nm, t (major) = 25.2 min, t (minor) = 33.1 min]; $[\alpha]_D^{20}$ = +177 (c = 0.1, $CHCl_3$). M.p. 201-203 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.47 (d, *J* = 10.2 Hz, 1H), 6.31 (d, *J* = 10.2 Hz, 1H), 4.41 (d, *J* = 18.9 Hz, 1H), 3.57 (d, *J* = 19.2 Hz, 1H), 3.23 (d, *J* = 17.4 Hz, 1H), 2.89-2.77 (m, 2H), 1.66-1.49 (m, 2H), 1.44-1.36 (m, 2H), 1.20-1.05 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.1, 192.8, 170.3, 164.9, 144.6, 133.6, 87.6, 72.9, 51.0, 50.1, 32.8, 11.9, 8.2, 7.7; IR (thin film): ν_{max} (cm^{-1}) = 2921, 1763, 1698, 1677, 1638, 1382, 1355, 1258, 1208, 1099, 1029, 779, 761, 714; MS (ESI) 370 ([M+H]$^+$); HRMS (MALDI) calcd for $C_{14}H_{13}INO_3$ ([M+H]$^+$): 369.9935. Found: 369.9942.



2k

(6aS,10aR)-1-(tert-Butyl)-2-iodo-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

White solid, 75% yield, 94% ee [Daicel Chiralcel OD-H, *n*-hexane/2-propanol = 60/40, v = 0.8 mL·min^{-1}, λ = 254 nm, t (major) = 23.4 min, t (minor) = 30.8 min]; $[\alpha]_D^{20}$ = +153 (c = 0.2, $CHCl_3$). M.p. 203-205 ^oC. ^1H NMR (300 MHz, $CDCl_3$) δ 6.50 (dd, J = 10.5, 1.2 Hz, 1H), 6.27 (d, J = 10.2 Hz, 1H), 4.44 (dd, J = 19.2, 0.9 Hz, 1H), 3.50 (d, J = 19.2 Hz, 1H), 3.23 (d, J = 18.3 Hz, 1H), 3.09-3.00 (m, 1H), 2.88 (d, J = 7.5 Hz, 2H), 1.50 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.1, 192.9, 171.5, 169.8, 145.5, 132.9, 96.3, 73.3, 49.6, 48.8, 35.9, 35.0, 29.6; IR (thin film): ν_{max} (cm^{-1}): 2921, 1766, 1703, 1676, 1382, 1250, 1088, 776, 694; MS (ESI) 386 ([M+H]$^+$); HRMS (MALDI) calcd for $C_{15}H_{17}INO_3$ ([M+H]$^+$): 386.0248. Found: 386.0260.



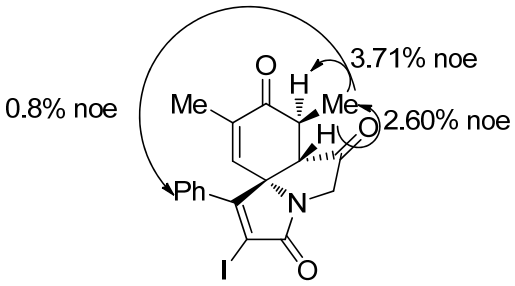
2l

(6aS,7S,10aR)-2-Iodo-7,9-dimethyl-1-phenyl-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

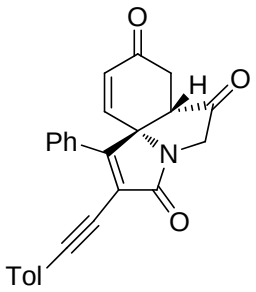
White solid, 9% yield, 99% ee [Daicel Chiralcel OD-H, *n*-hexane/2-propanol = 60/40, v = 0.8 mL·min^{-1}, λ = 254 nm, t (major) = 22.0 min, t (minor) = 70.4 min]; M.p. 215-217 ^oC. ^1H NMR (300 MHz, $CDCl_3$) δ 7.47-7.45 (m, 3H), 7.27-7.23 (m, 2H), 6.49 (s, 1H), 4.49 (d, J = 18.9 Hz, 1H), 3.64 (d, J = 18.9 Hz, 1H), 3.08 (q, J = 7.8 Hz, 1H), 2.51 (s, 1H), 1.91 (s, 3H), 0.41 (d, J = 8.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.5, 196.6, 169.4, 165.1, 140.5, 137.5, 133.4, 130.2, 129.0, 127.9, 98.4, 73.6, 57.6,

49.8, 37.9, 17.7, 16.7; IR (thin film): ν_{max} (cm^{-1}) = 2922, 1764, 1704, 1679, 1625, 1368, 1119, 758, 697; MS (ESI) 434 ($[M+H]^+$); HRMS (MALDI) calcd for $C_{19}H_{17}INO_3$ ($[M+H]^+$): 434.0248. Found: 434.0256.

NOE experiment of **2l**:



2l

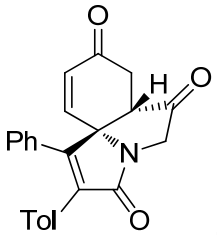


2aa

(6aS,10aR)-1-Phenyl-2-(p-tolylethynyl)-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

Under argon, compound **2a** (40.5 mg, 0.1 mmol), CuI (5.7 mg, 0.03 mmol) and $PdCl_2(PPh_3)_2$ (21.0 mg, 0.03 mmol) were placed in a Schlenk tube equipped with a stir bar. Then Et_3N/toluene (2mL/2mL) and 4-ethynyltoluene (116.0 mg, 1.0 mmol) were added. The reaction was stirred at room temperature for 48h. After the reaction was complete, the reaction mixture was filtrated over a pad of celite, and extracted with CH_2Cl_2. The combined organic layers were dried over Na_2SO_4. The solvent was evaporated under reduced pressure and the residue was purified by column chromatography on silica gel (EtOAc:PE, 1:2) affording compound **2aa** as a white solid (30 mg, 76% yield, 98% ee). [Daicel Chiralcel OD-H, *n*-hexane/2-propanol = 60/40, v = 0.8 mL · min^{-1}, λ = 254 nm, t (major) = 20.6 min, t (minor) = 28.0 min]; $[\alpha]_D^{20}$ = +200 (c = 0.1, $CHCl_3$). M.p. 186-188 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76

(d, *J* = 6.3 Hz, 2H), 7.51-7.43 (m, 3H), 7.35 (d, *J* = 7.2 Hz, 2H), 7.14 (d, *J* = 7.2 Hz, 2H), 6.75 (d, *J* = 9.9 Hz, 1H), 6.34 (d, *J* = 9.9 Hz, 1H), 4.50 (d, *J* = 19.2 Hz, 1H), 3.60 (d, *J* = 19.2 Hz, 1H), 3.04 (d, *J* = 19.2 Hz, 1H), 2.87 (d, *J* = 6.6 Hz, 3H), 2.41-2.36 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.5, 193.0, 169.9, 159.2, 145.0, 139.9, 133.6, 132.0, 131.8, 130.7, 129.2, 129.0, 127.6, 120.1, 118.7, 99.9, 80.0, 69.3, 51.3, 49.6, 33.3, 21.6; IR (thin film): ν_{max} (cm^{-1}) = 2918, 2850, 1767, 1717, 1693, 1637, 1413, 1385, 1342, 1211, 1084, 816, 767, 693; MS (ESI) 394 ($[M+H]^+$); HRMS (MALDI) calcd for $C_{26}H_{20}NO_3$ ($[M+H]^+$): 394.1438. Found: 394.1447.

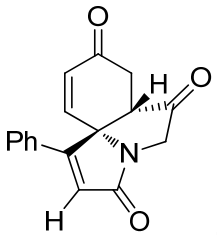


2ab

(6aS,10aR)-1-Phenyl-2-(p-tolyl)-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trion e

Under argon, $Pd(OAc)_2$ (4.8 mg, 0.02 mmol), PPh_3 (12.8 mg, 0.04 mmol), 4-tolylboronic acid (20.4 mg, 0.15 mmol), compound **2a** (40.5 mg, 0.1 mmol) and K_2CO_3 (27.8 mg, 0.2 mmol) were placed in a Schlenk tube equipped with a stir bar. Benzene/H_2O (5/1, 4.8 mL) was added, and the resulting heterogeneous reaction mixture was stirred vigorously for 48 hours at 60 ºC. The reaction mixture was then filtered through a short pad of celite and concentrated under reduced pressure, the residue was purified by column chromatography on silica gel (EtOAc:PE, 1:2) to afford compound **2ab** as a white solid (36.0 mg, 97% yield, 98% ee). [Daicel Chiralcel OD-H, *n*-hexane/2-propanol = 60/40, *v* = 0.8 mL·min^{-1}, λ = 254 nm, t (major) = 21.2 min, t (minor) = 29.0 min]; $[\alpha]_D^{20}$ = +193 (c = 0.1, $CHCl_3$). M.p. >250 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 (m, 5H), 7.17 (d, *J* = 5.7 Hz, 2H), 7.08 (d, *J* = 7.8 Hz, 2H), 6.67 (d, *J* = 9.9 Hz, 1H), 6.21 (d, *J* = 10.5 Hz, 1H), 4.54 (d, *J* = 18.6 Hz, 1H), 3.64 (d, *J* = 18.9 Hz, 1H), 2.99 (d, *J* = 18.0 Hz, 1H), 2.88 (d, *J* = 7.2 Hz, 1H), 2.31 (s, 3H), 2.20 (dd, *J* = 18.0, 7.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 210.0,

193.0, 172.0, 154.7, 145.0, 138.9, 134.1, 133.6, 132.7, 129.4, 129.19, 129.16, 129.0, 127.9, 126.8, 68.9, 51.3, 49.8, 33.0, 21.3; IR (thin film): ν_{max} (cm^{-1}): 2922, 2852, 1769, 1696, 1635, 1378, 1333, 1085, 775, 696; MS (EI, *m/z*, rel. intensity) 369 ($[M]^+$, 100), 312 (56); HRMS (EI) calcd for $C_{24}H_{19}NO_3$ ($[M]^+$): 369.1365. Found: 369.1364.

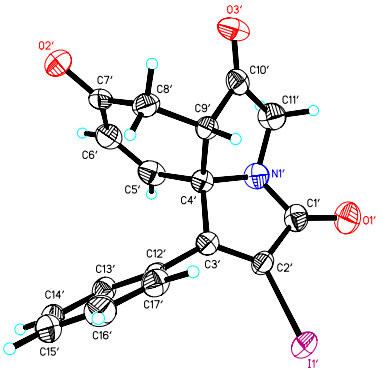


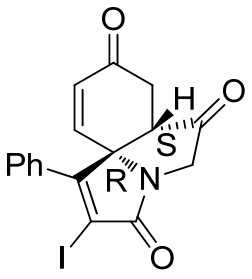
2ac

(6aS,10aR)-1-Phenyl-6a,7-dihydropyrrolo[2,1-i]indole-3,6,8(5H)-trione

CH_3OH/CH_2Cl_2 (4 mL/1 mL) was added to a Schlenk tube containing compound **2a** (40.5 mg, 0.1 mmol) equipped with a stir bar, then 10% Pd/C (20 mg, 50% wt) was added. The vial was sealed up, and then it was evacuated and filled with hydrogen (three cycles). The reaction was stirred at room temperature for 120h. After the reaction was complete, the reaction mixture was filtrated over a pad of celite and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (EtOAc:PE, 1:2) to afford compound **2ac** as a white solid (26.0 mg, 64% yield, 99% ee). [Daicel Chiralcel OD-H, *n*-hexane/2-propanol = 60/40, *v* = 0.8 mL·min^{-1}, λ = 254 nm, t (major) = 43.6 min, t (minor) = 70.7 min]; $[\alpha]_D^{20}$ = +92 (c = 0.1, $CHCl_3$). M.p. 183-185 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54-7.43 (m, 5H), 6.75 (d, *J* = 9.9 Hz, 1H), 6.54 (s, 1H), 6.35 (d, *J* = 10.2 Hz, 1H), 4.45 (d, *J* = 18.6 Hz, 1H), 3.56 (d, *J* = 19.2 Hz, 1H), 3.10 (d, *J* = 19.3 Hz, 1H), 2.82 (d, *J* = 6.6 Hz, 1H), 2.55 (dd, *J* = 18.0, 7.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 210.1, 193.1, 172.4, 162.4, 145.3, 133.4, 131.7, 130.9, 129.3, 126.8, 124.1, 70.1, 51.1, 49.2, 33.4; IR (thin film): ν_{max} (cm^{-1}) = 2920, 2852, 1762, 1634, 1444, 1380, 1093, 768, 694; MS (EI, *m/z*, rel. intensity) 279 ($[M]^+$, 22), 251 (100), 223 (33), 167 (57); HRMS (EI) calcd for $C_{17}H_{13}NO_3$ ($[M]^+$): 279.0895. Found: 279.0901.

X-ray of enantiopure (6aS,10aR)-**2a**



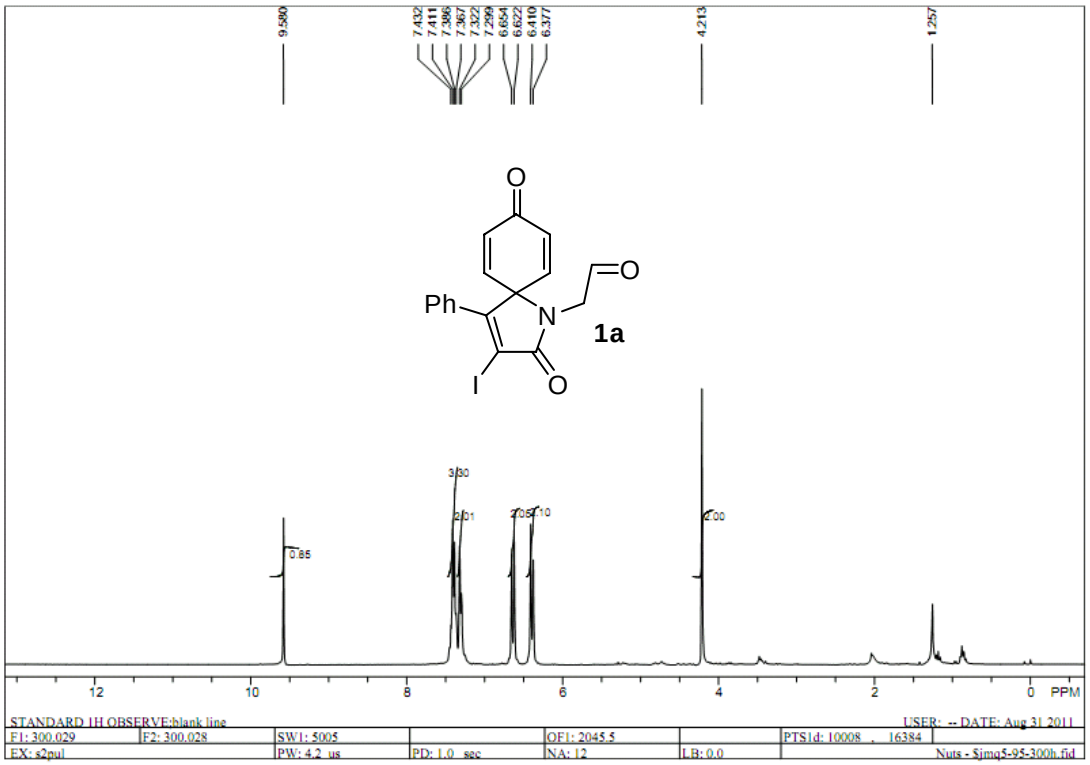


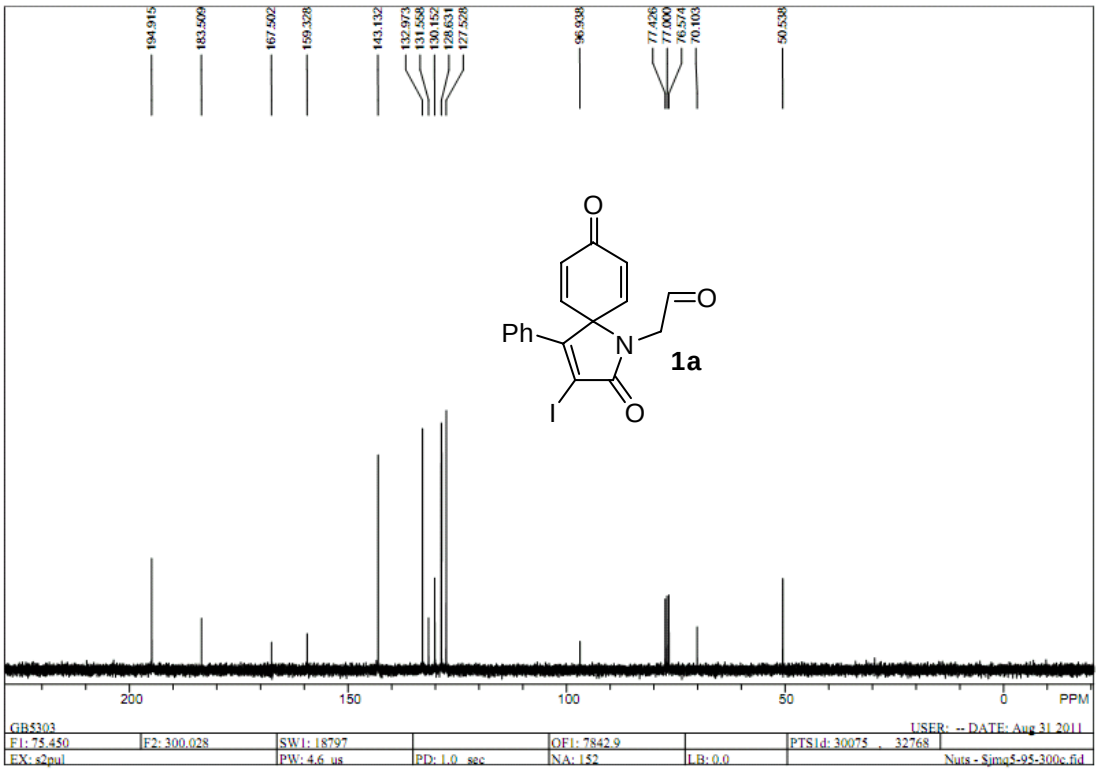
2a

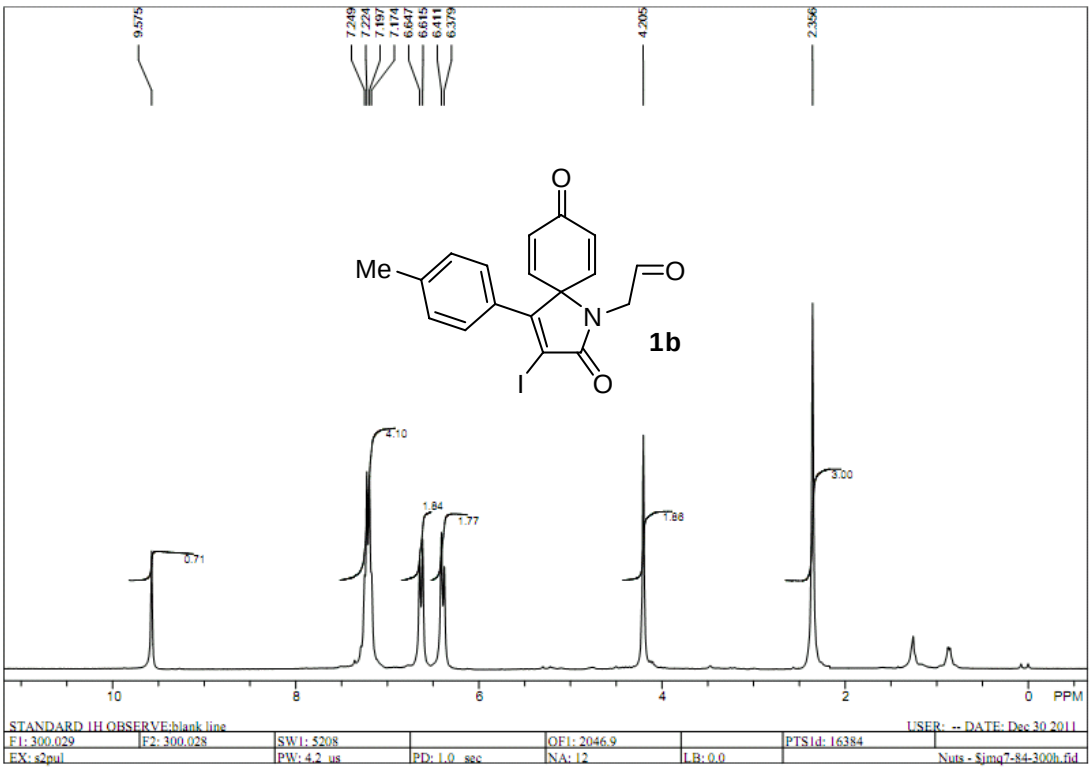
Table 1. Crystal data and structure refinement for cd211527.

Identification code	cd211527
Empirical formula	C17 H12 I N O3
Formula weight	405.18
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, P2(1)2(1)2(1)
Unit cell dimensions	a = 9.5004(5) A alpha = 90 deg.
	b = 14.8567(7) A beta = 90 deg.
	c = 21.8984(11) A gamma = 90 deg.
Volume	3090.8(3) A^3
Z, Calculated density	8, 1.741 Mg/m^3
Absorption coefficient	2.084 mm^-1
F(000)	1584
Crystal size	0.316 x 0.203 x 0.157 mm
Theta range for data collection	1.66 to 26.00 deg.
Limiting indices	-11<=h<=11, -18<=k<=16, -25<=l<=27
Reflections collected / unique	18928 / 6087 [R(int) = 0.0283]
Completeness to theta = 26.00	100.0 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.49889
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6087 / 0 / 397
Goodness-of-fit on F^2	1.061
Final R indices [I>2sigma(I)]	R1 = 0.0362, wR2 = 0.0799
R indices (all data)	R1 = 0.0408, wR2 = 0.0824
Absolute structure parameter	-0.01(2)
Largest diff. peak and hole	0.821 and -0.327 e.A^-3

NMR and HPLC Spectra



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6.411
6.379
4.205
2.356
Me
O
N
I
1b
4.10
1.84
1.77
1.88
3.00
0.71
10
8
6
4
2
0
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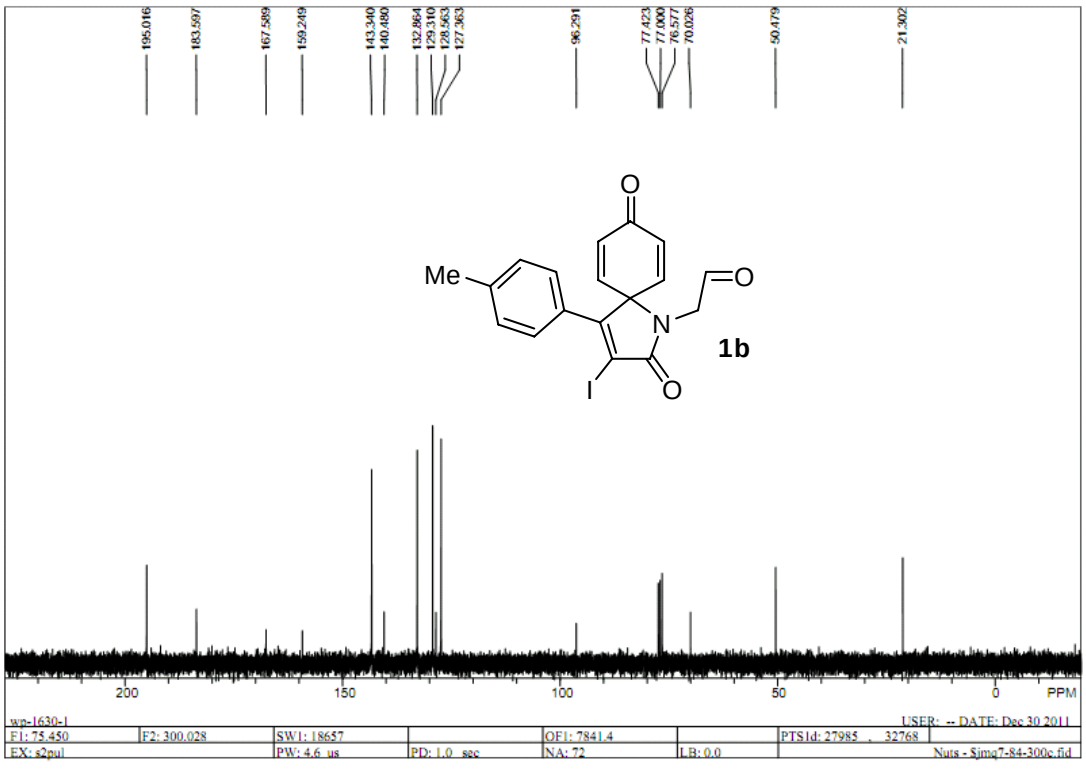
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127.363
96.291
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77.000
76.577
70.026
50.479
21.302
Me
O
N
I
1b
200
150
100
50
0
PPM
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PTS1d: 27985 , 32768
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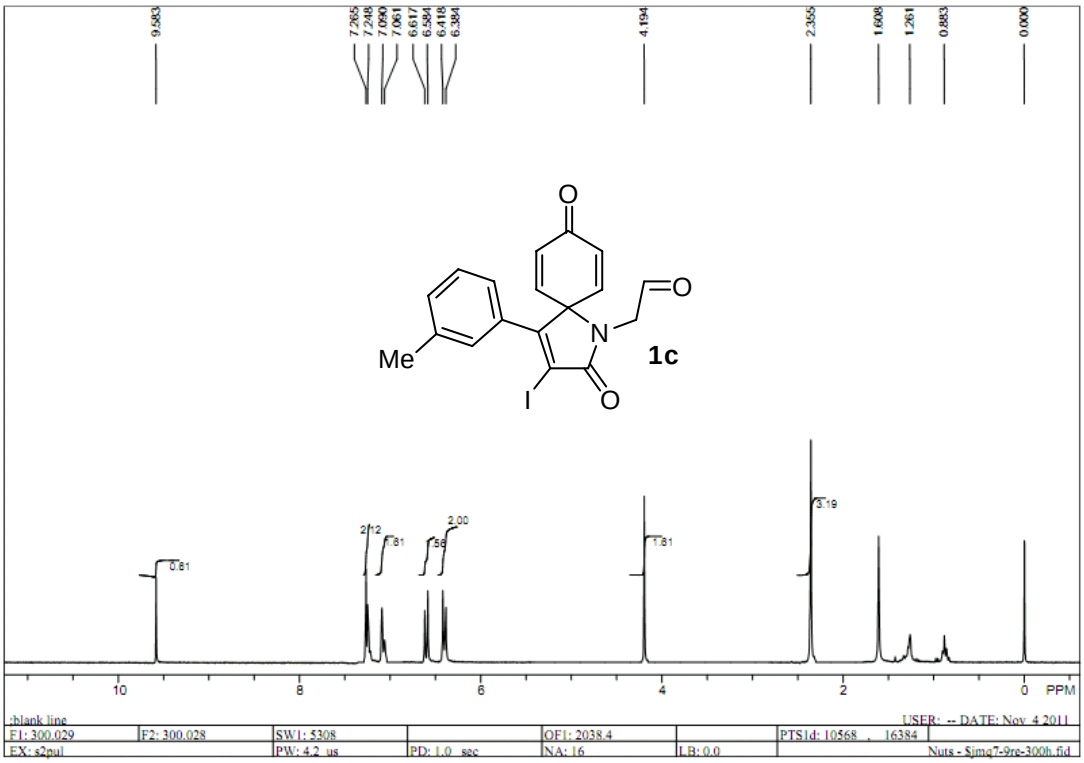
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4.194
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1.608
1.261
0.883
0.000
O
O
N
Me
I
O
1c
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2.00
1.58
1.81
3.19
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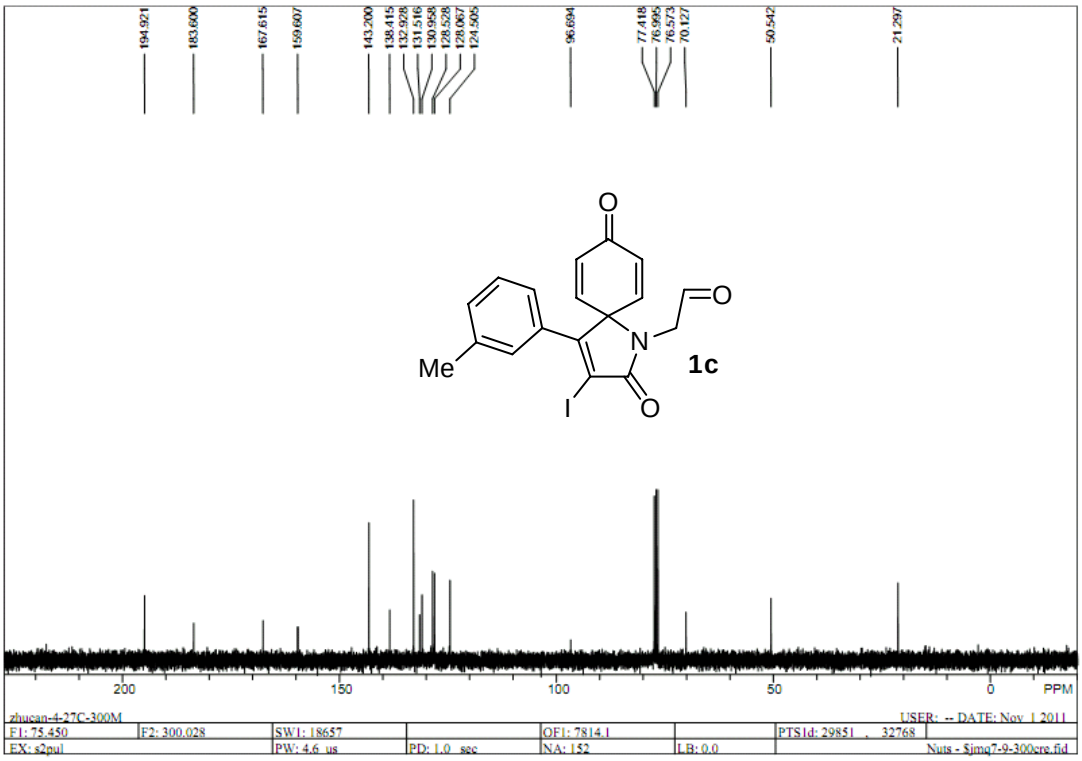
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77.418
76.995
76.573
70.127
50.542
21.297
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O
N
Me
I
O
1c
200
150
100
50
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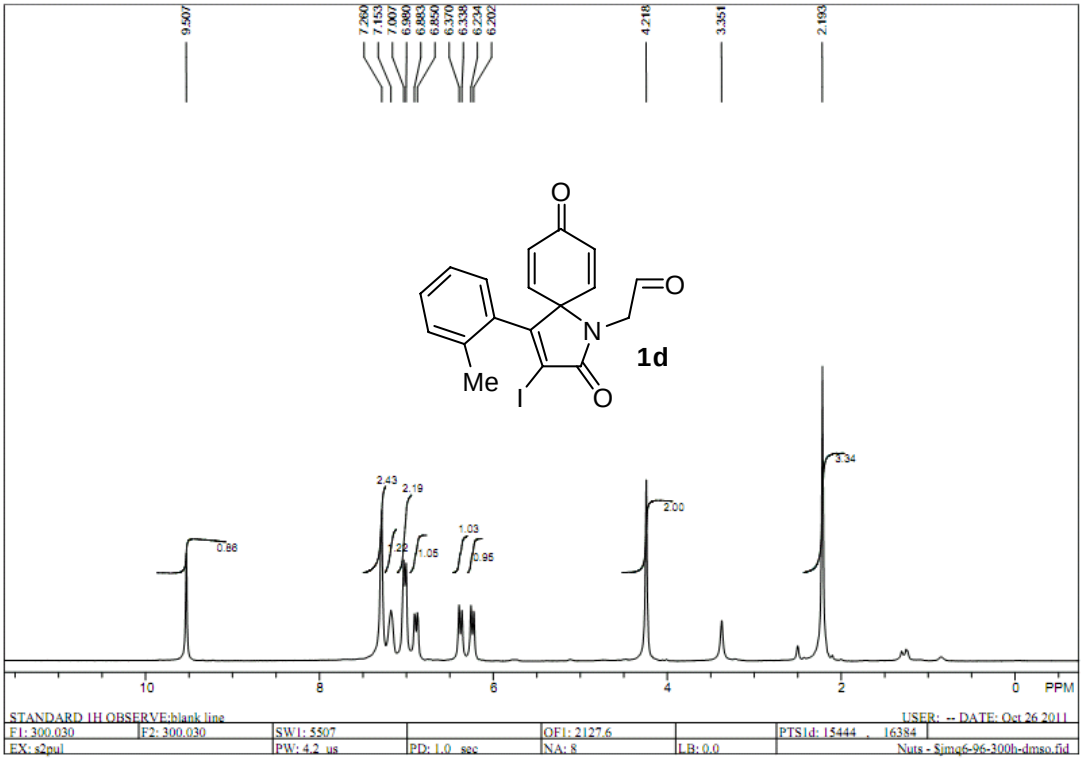
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6.234
6.202
4.218
3.351
2.193
O
O
N
Me
I
O
1d
0.86
2.43
2.19
1.22
1.05
1.03
0.95
2.00
3.34
10
8
6
4
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LB: 0.0
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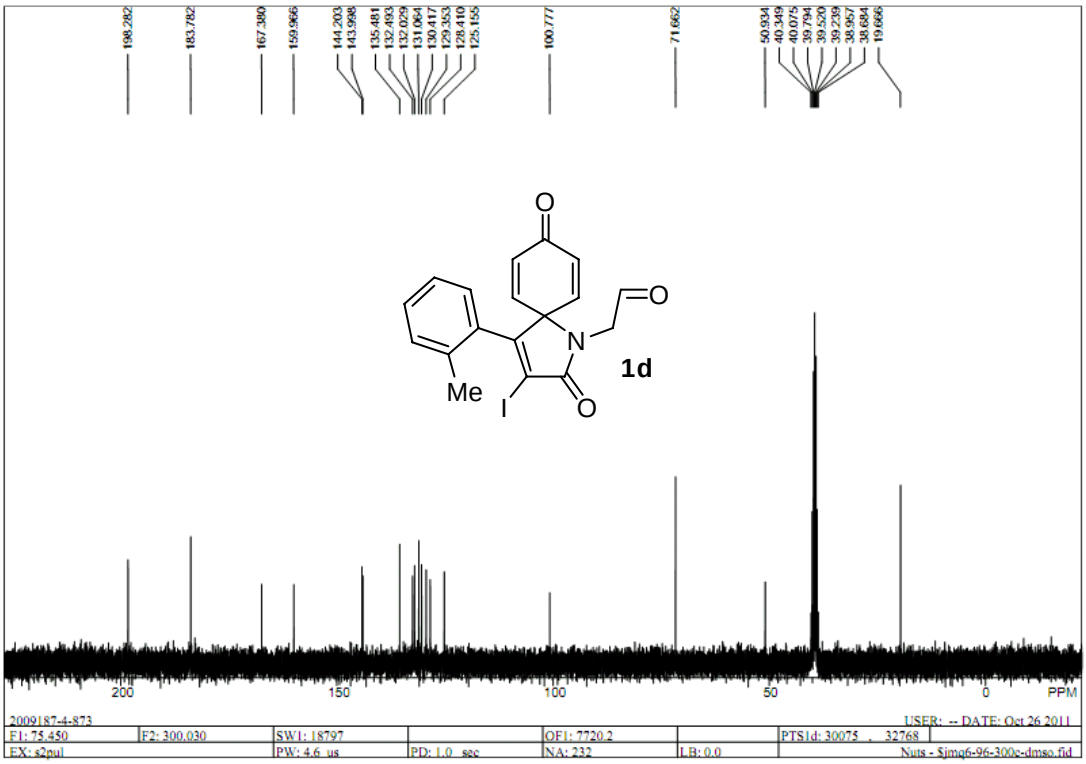
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128.410
125.155
100.777
71.662
50.934
40.349
40.075
39.794
39.520
39.239
38.957
38.684
19.666
O
O
N
Me
I
O
1d
200
150
100
50
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PPM
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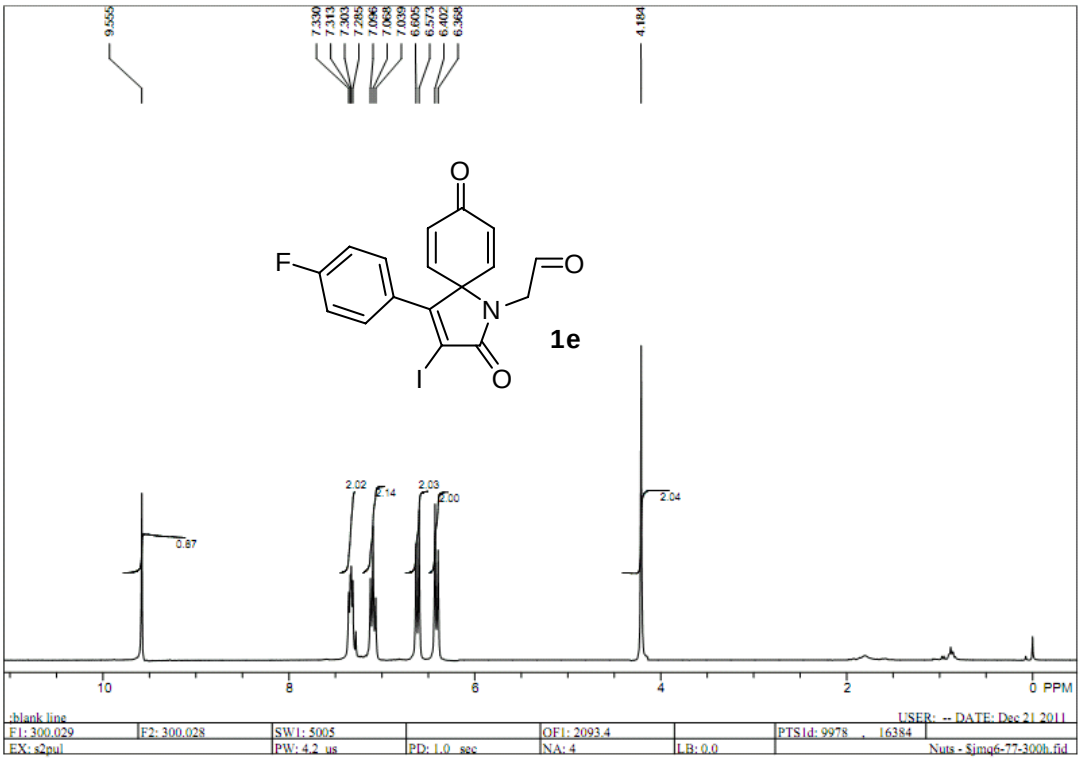
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4.184
O
F
N
O
O
I
1e
2.02
2.14
2.03
2.00
2.04
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10
8
6
4
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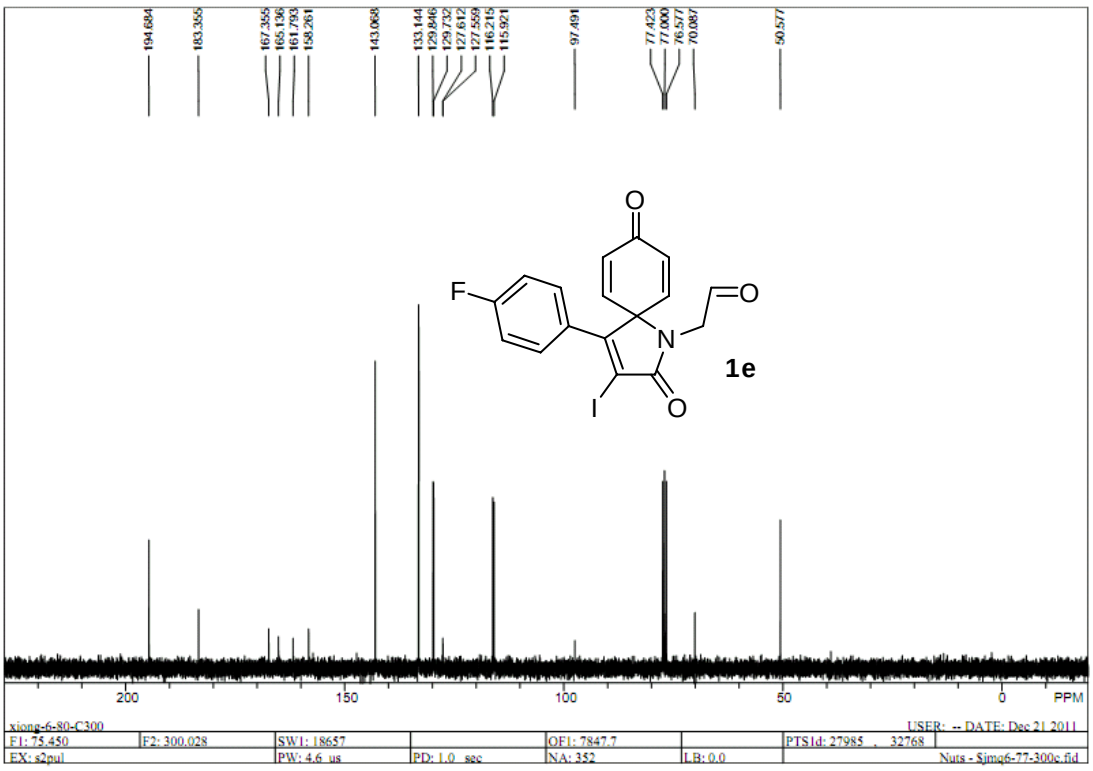
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115.921
97.491
77.423
77.000
76.577
70.087
50.577
O
F
N
O
O
I
1e
200
150
100
50
0
PPM
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USER: -- DATE: Dec 21 2011
F1: 75.450
F2: 300.028
SW1: 18657
OF1: 7847.7
PTS1d: 27985 , 32768
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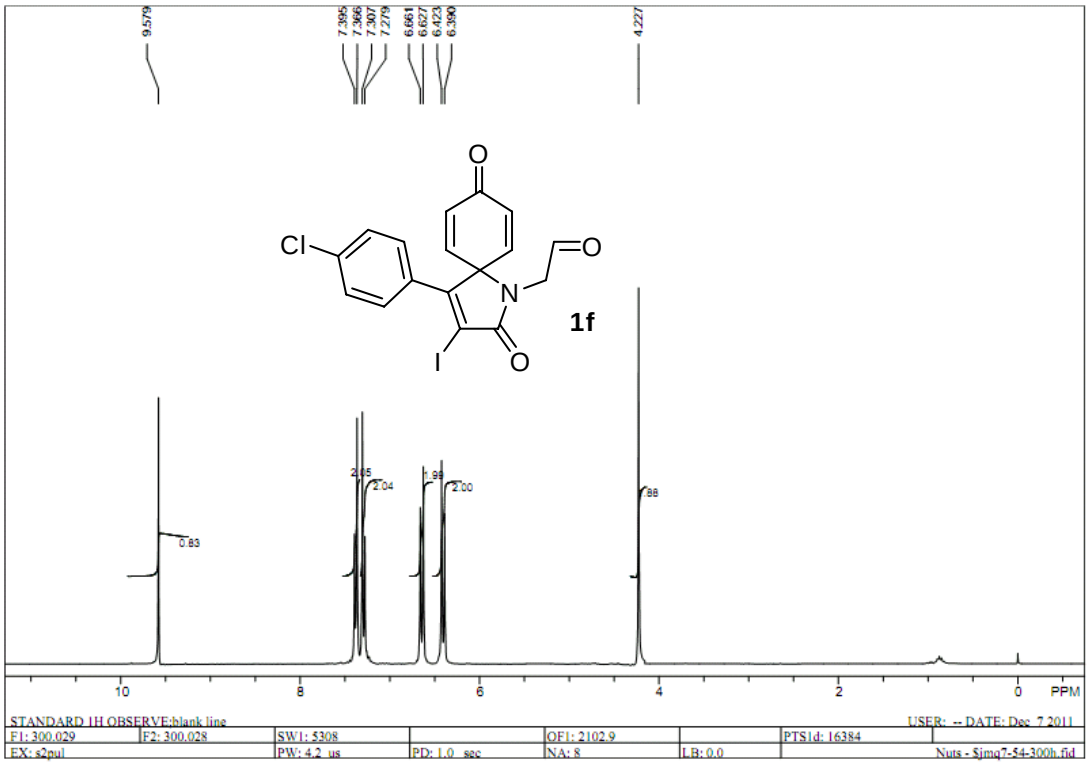
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6.390
4.227
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I
1f
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2.04
1.99
2.00
3.88
10
8
6
4
2
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LB: 0.0
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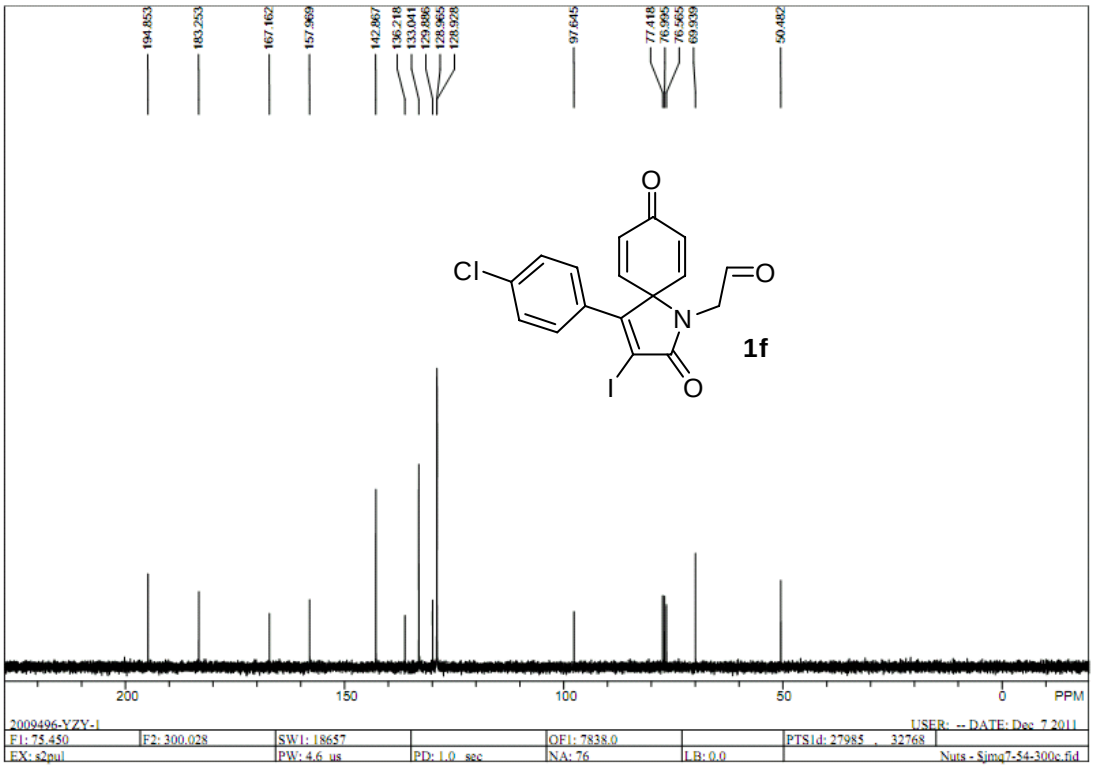
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136.218
133.041
129.886
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128.928
97.645
77.418
76.995
76.565
69.939
50.482
Cl
O
N
I
1f
200
150
100
50
0
PPM
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USER: -- DATE: Dec 7 2011
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F2: 300.028
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OF1: 7838.0
PTS1d: 27985 , 32768
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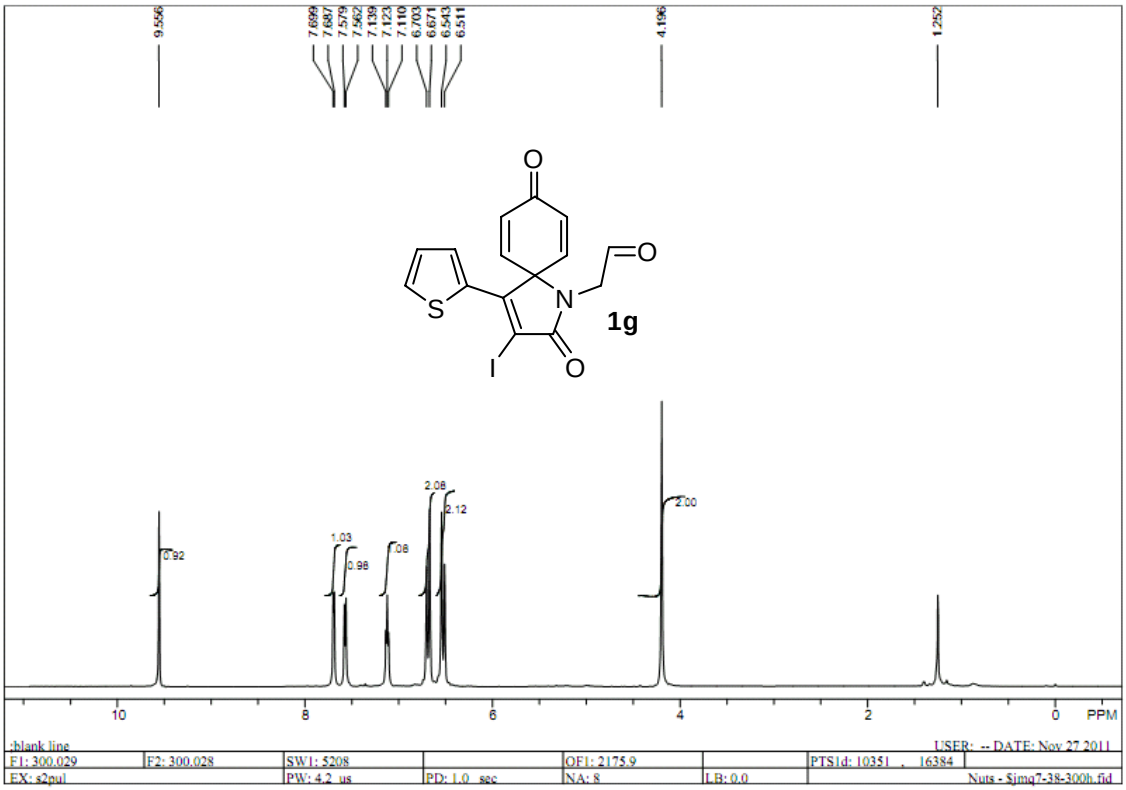
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7.110
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6.671
6.543
6.511
4.196
1.252
1g
0.92
1.03
0.98
1.08
2.08
2.12
2.00
10
8
6
4
2
0
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PTS1d: 10351 , 16384
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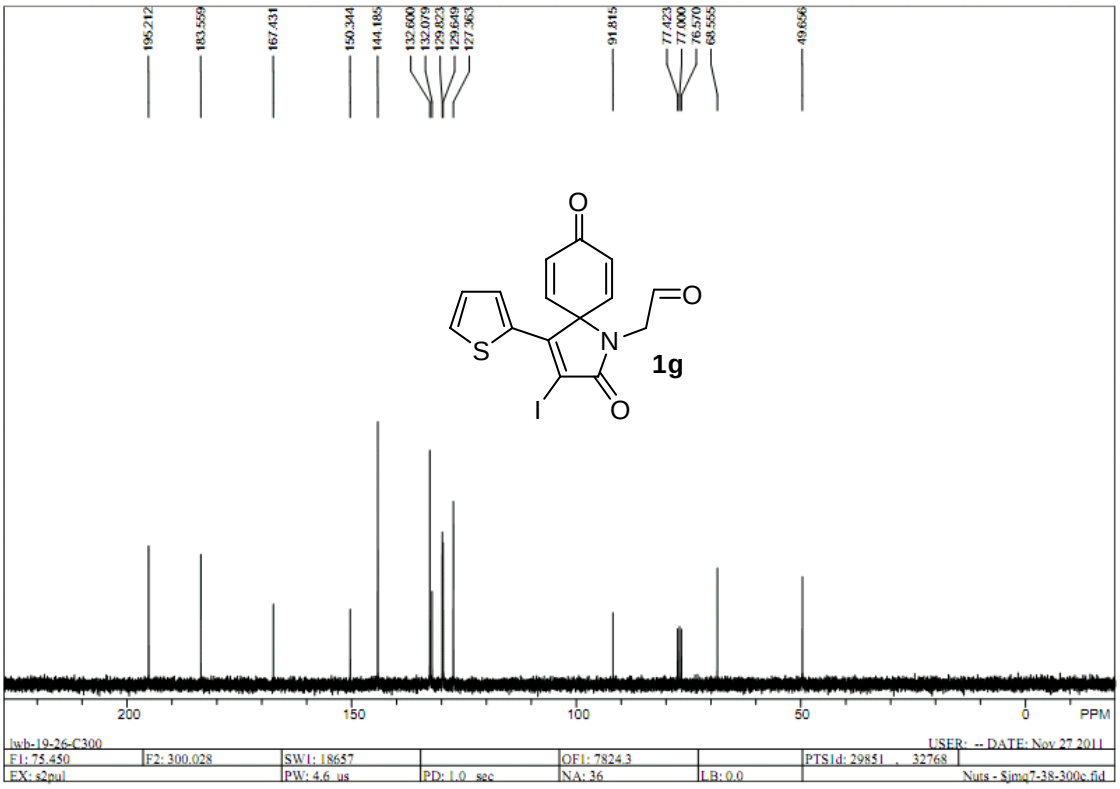
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127.363
91.815
77.423
77.000
76.570
68.555
49.696
1g
200
150
100
50
0
PPM
lwh-19-26-C300
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F1: 75.450
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PTS1d: 29851 , 32768
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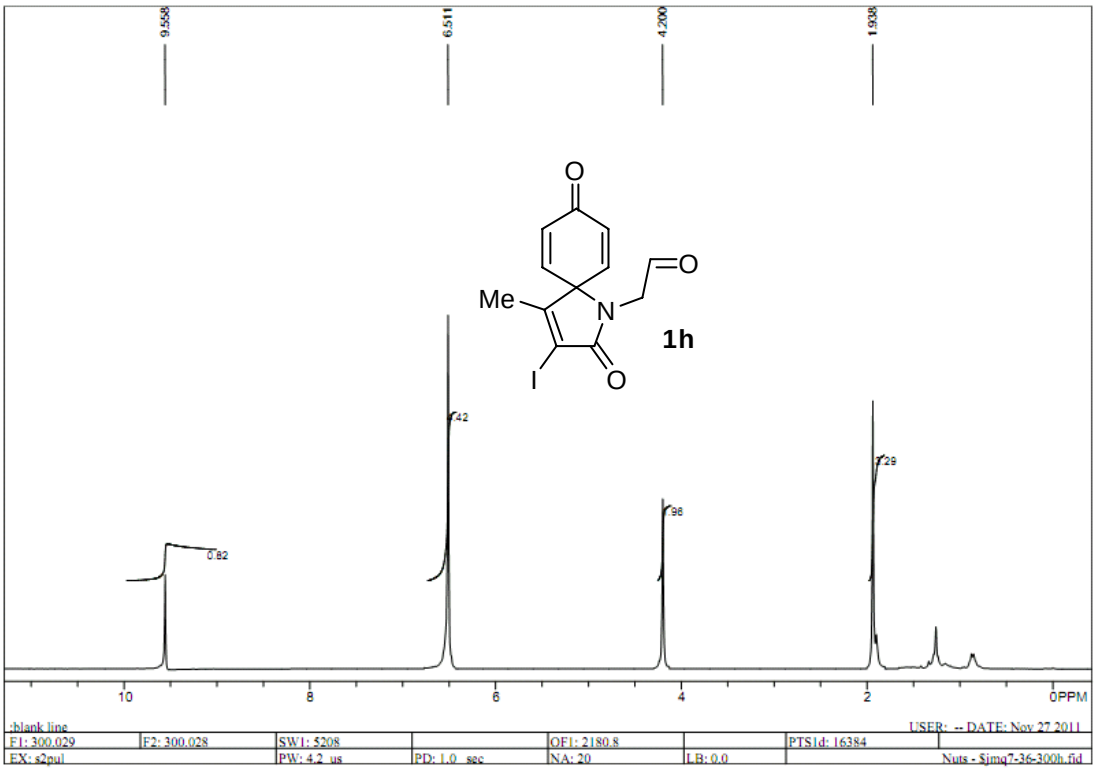
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8
6
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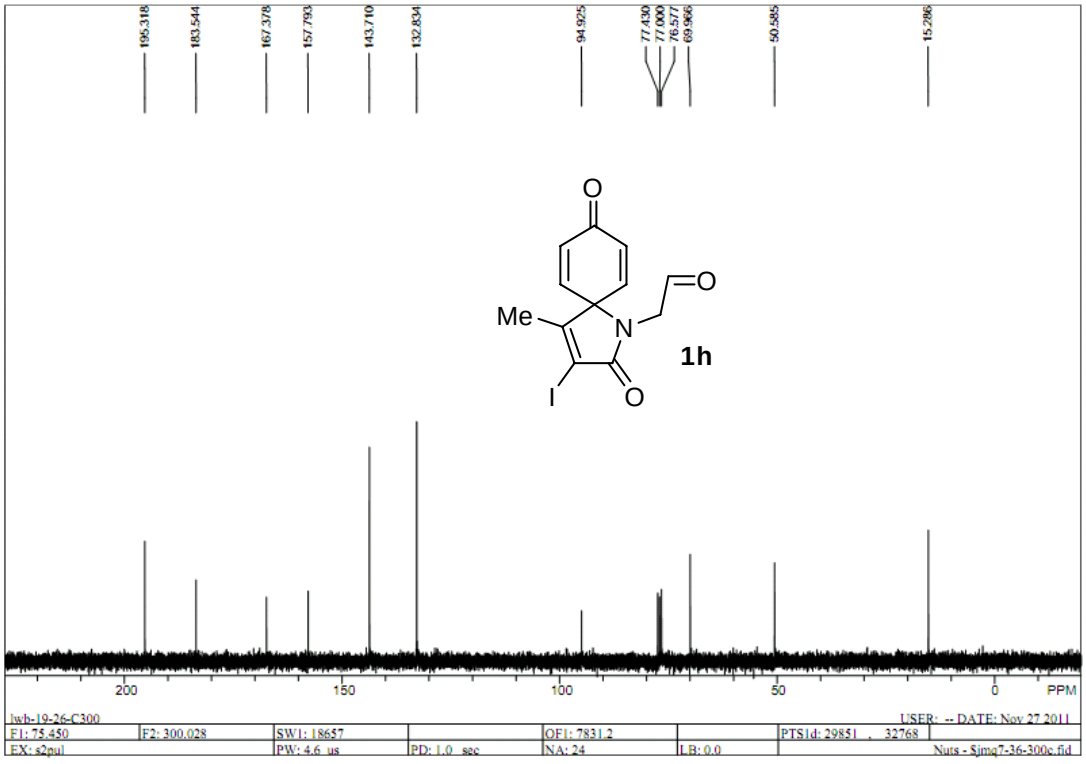
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15.286
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O
I
O
1h
200
150
100
50
0
PPM
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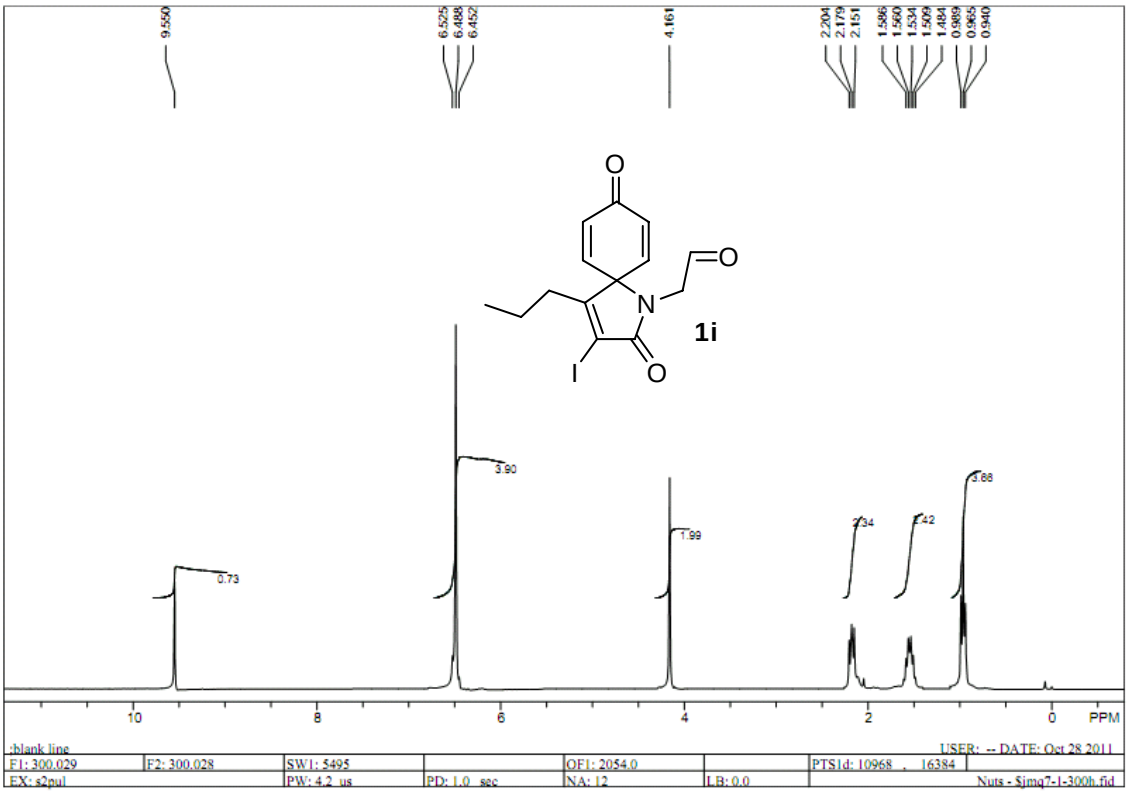
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0.969
0.965
0.940
O
O
N
O
I
1i
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1.99
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PTS1d: 10968 , 16384
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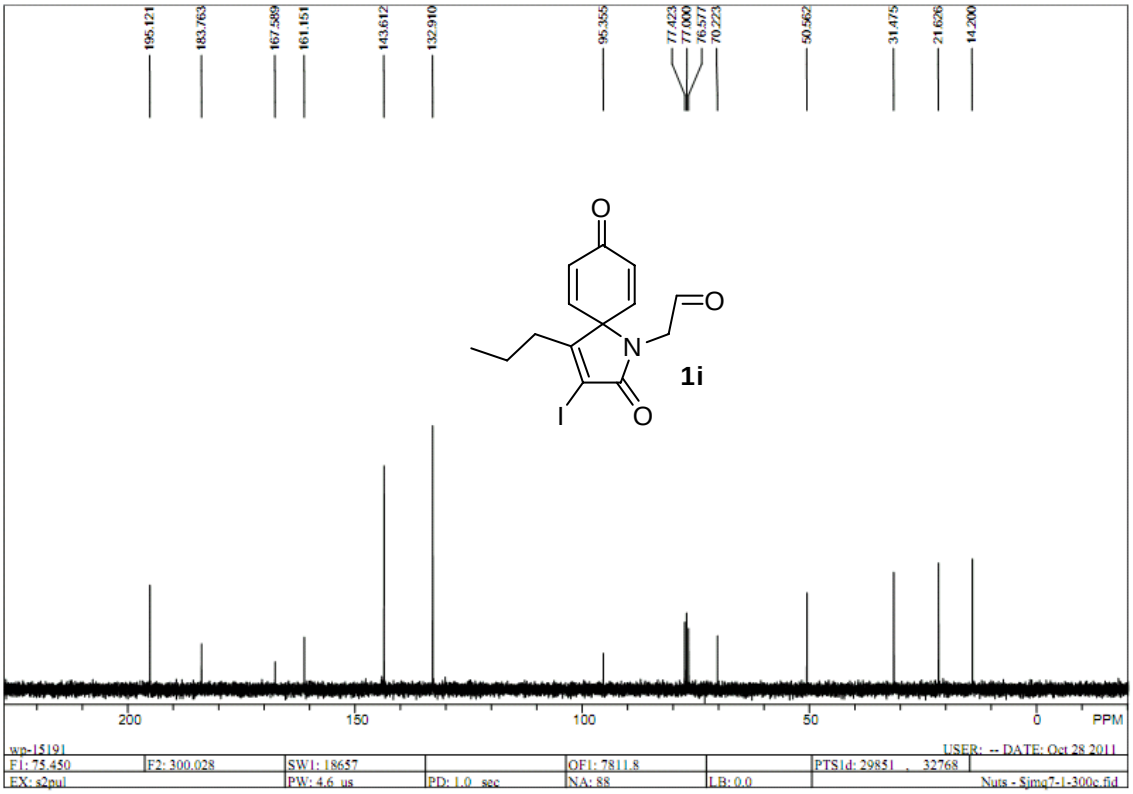
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50.562
31.475
21.626
14.200
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O
N
O
I
1i
200
150
100
50
0
PPM
wp-15191
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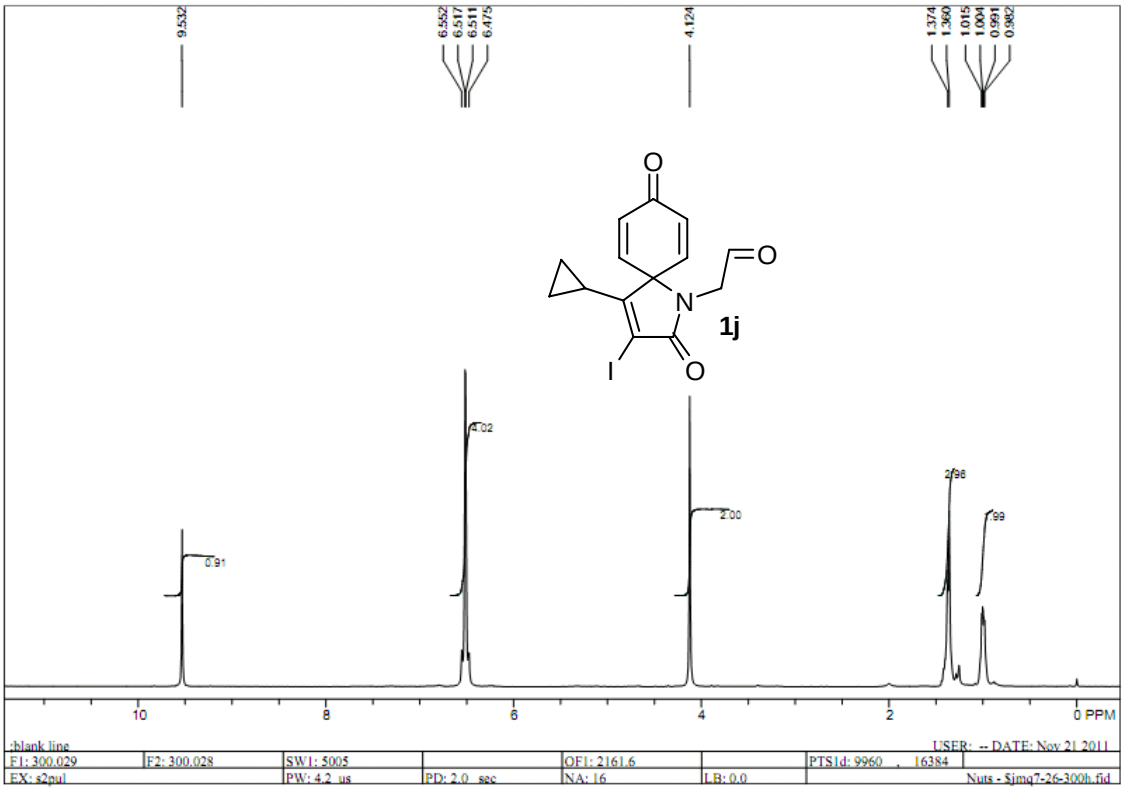
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1.004
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2.00
2.98
1.99
0.91
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8
6
4
2
0 PPM
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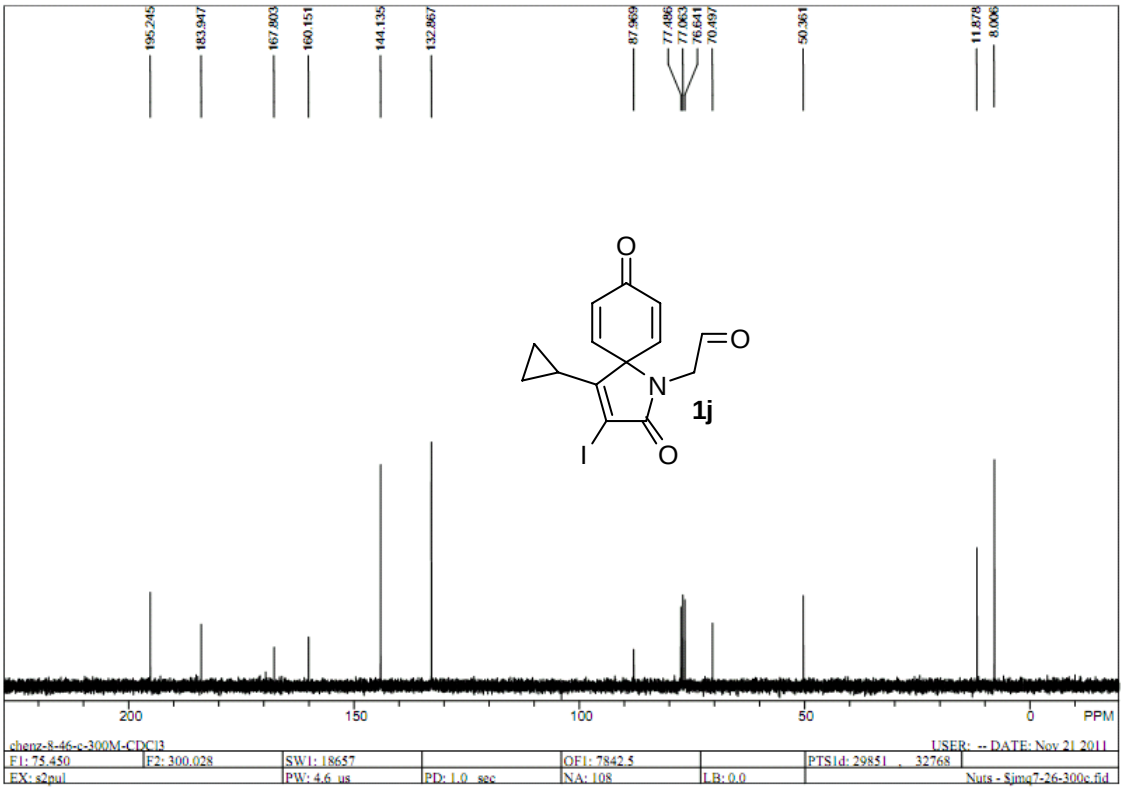
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11.878
8.006
1j
200
150
100
50
0
PPM
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OF1: 7842.5
PTS1d: 29851 , 32768
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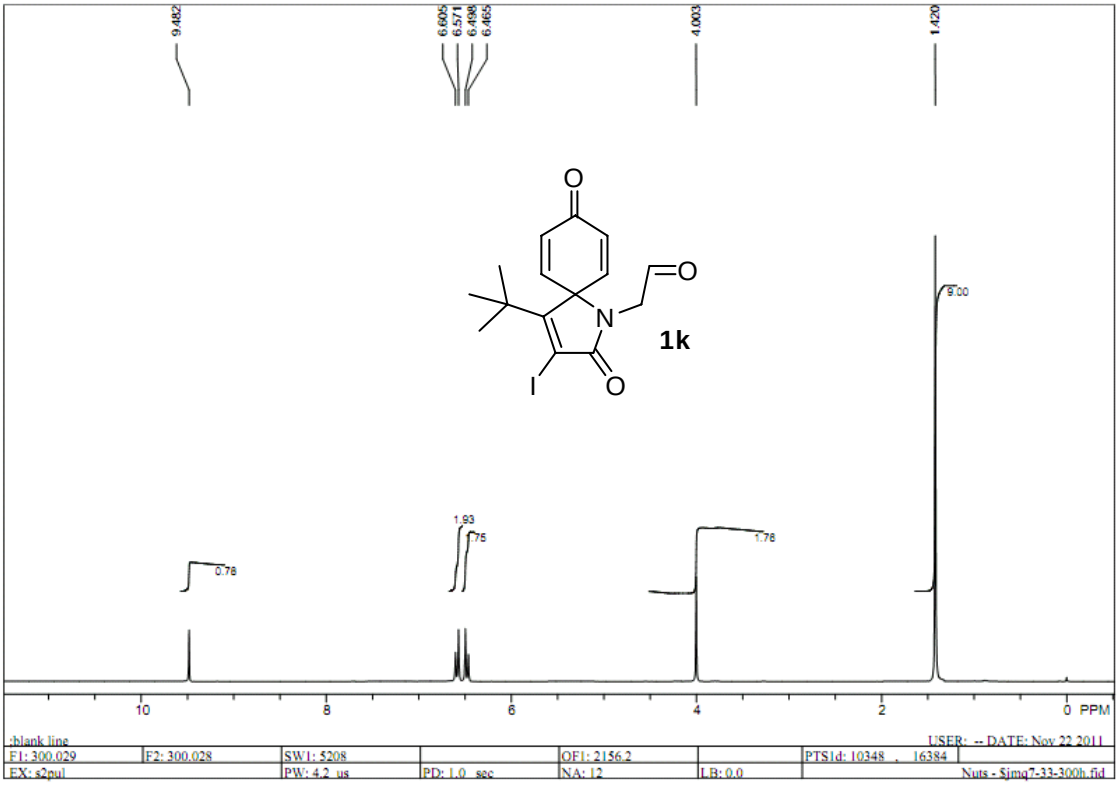
LB: 0.0
Nuts - $jmq7-26-300c.fid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6.571
6.498
6.465
4.003
1.420
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1.93
1.75
1.78
0.78
10
8
6
4
2
0 PPM
:blank line
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F2: 300.028
SW1: 5208
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PTS1d: 10348 , 16384
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PW: 4.2 us
PD: 1.0 sec
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LB: 0.0
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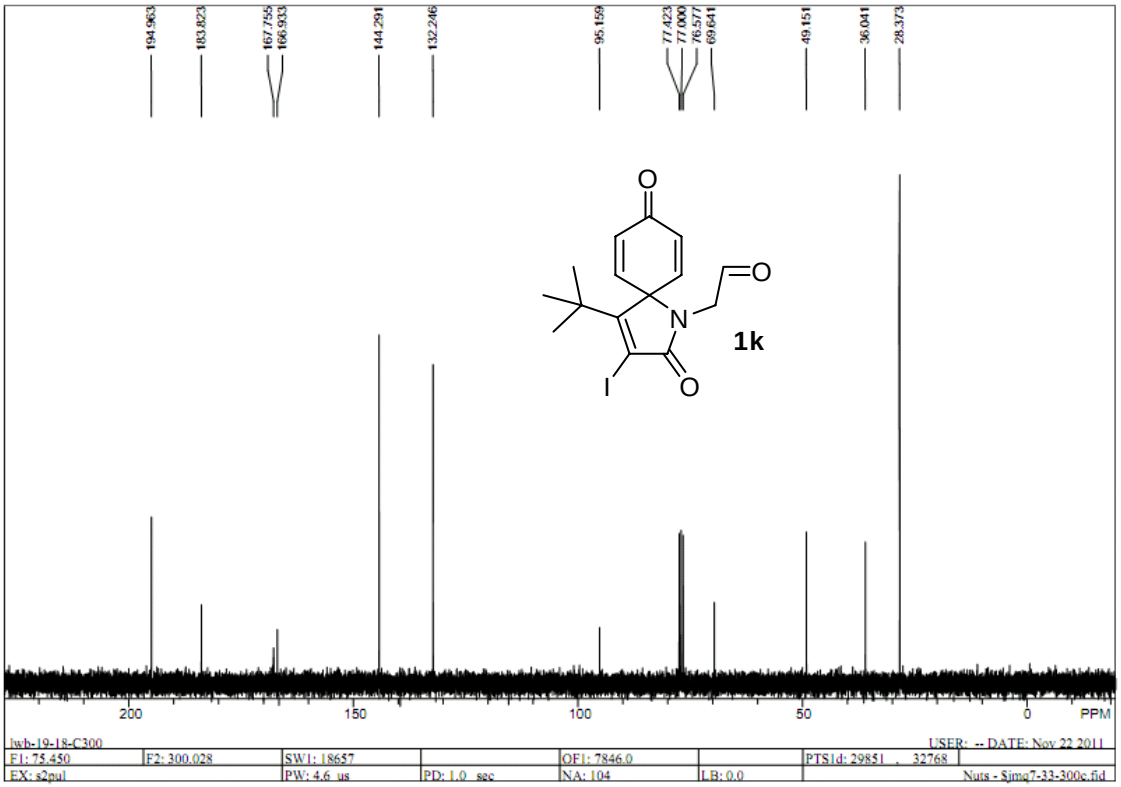
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167.755
166.933
144.291
132.246
95.159
77.423
77.000
76.577
69.641
49.151
36.041
28.373
1k
200
150
100
50
0 PPM
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SW1: 18657
OF1: 7846.0
PTS1d: 29851 , 32768
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PD: 1.0 sec
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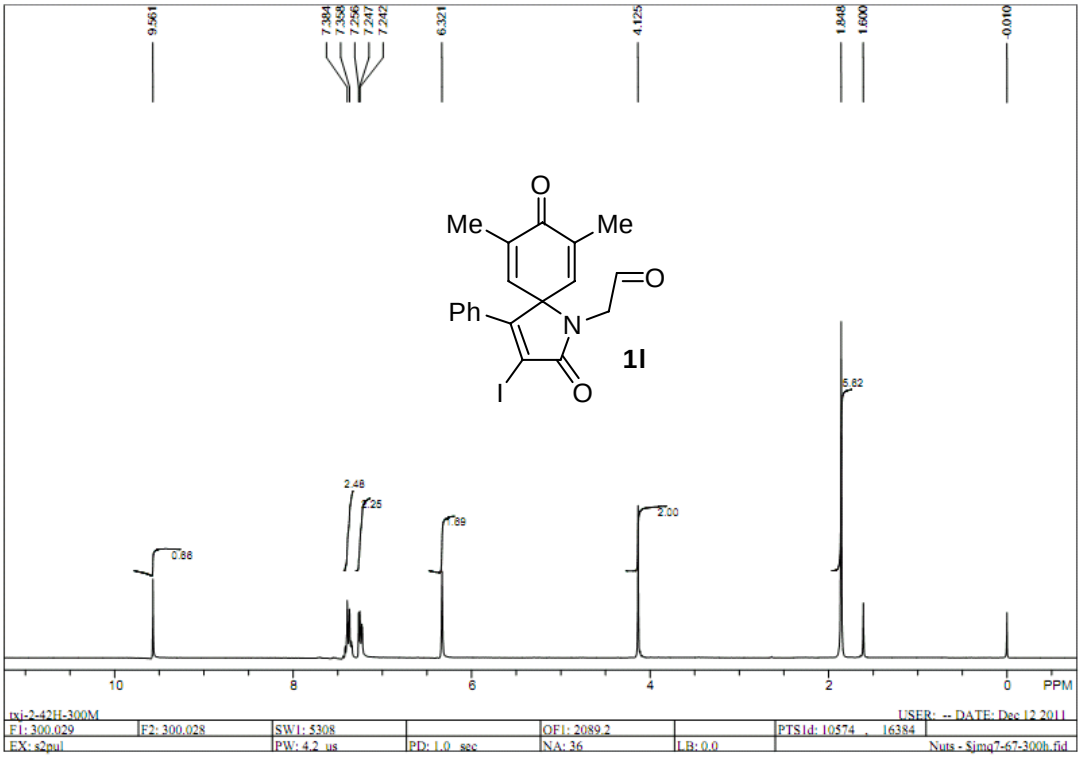
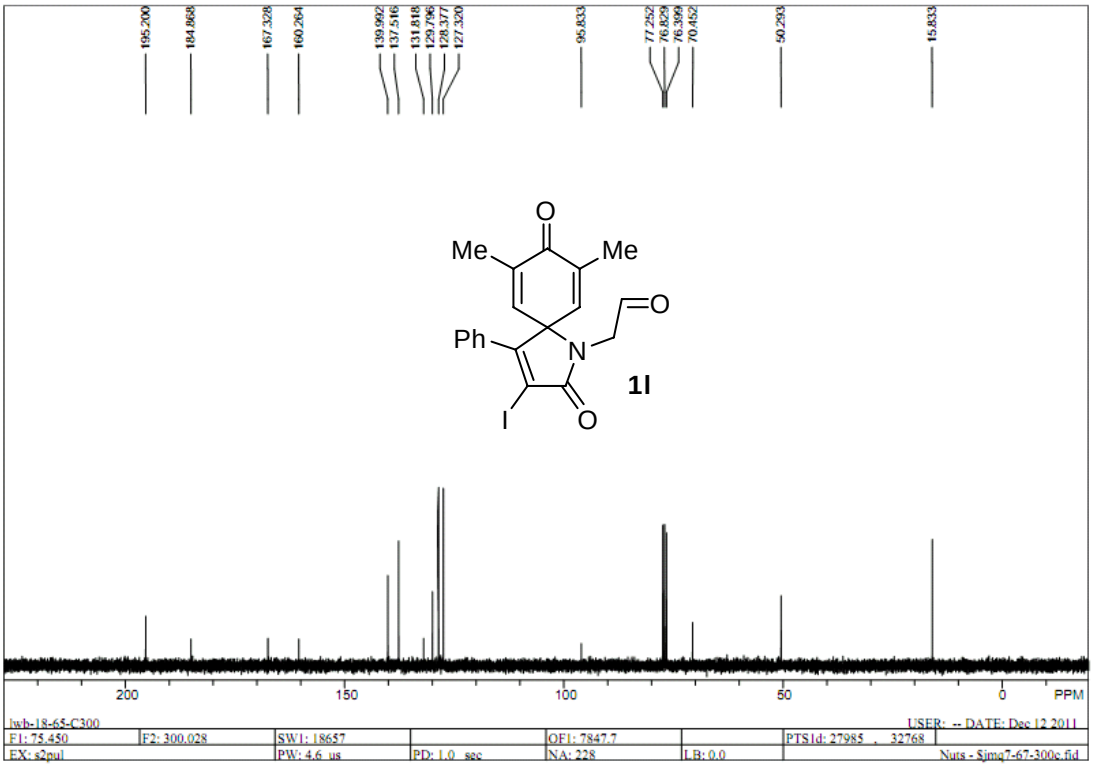
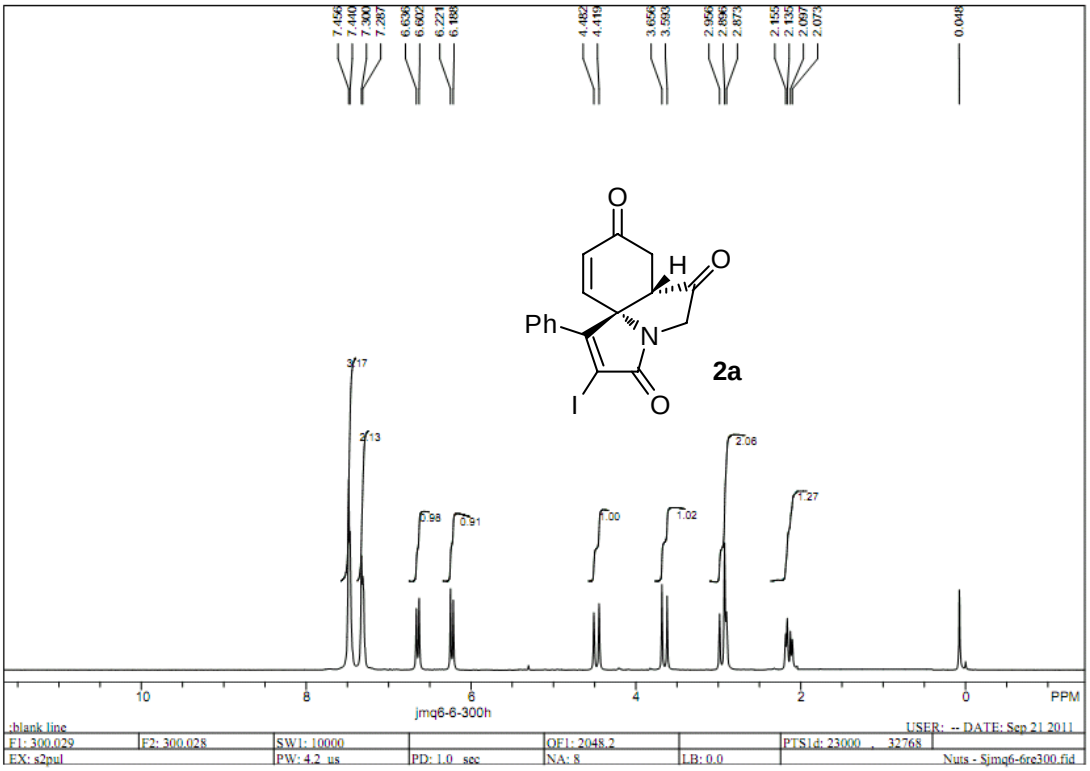
LB: 0.0
Nuts - Sjmq7-33-300c.fid


9.561
7.384
7.358
7.256
7.247
7.242
6.321
4.125
1.848
1.600
0.010
Me
Me
O
O
Ph
N
I
O
1l
6.82
2.48
2.25
1.89
2.00
0.86
10
8
6
4
2
0
PPM
rxj-2-42H-300M
USER: -- DATE: Dec 12 2011
F1: 300.029
F2: 300.028
SW1: 5308
OF1: 2089.2
PTS1d: 10574 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 36
LB: 0.0
Nuts - Sjmq7-67-300h.fid


195.200
184.868
167.328
160.264
139.992
137.516
131.818
129.796
128.377
127.320
96.833
77.252
76.829
76.399
70.452
50.293
15.833
Me
Me
O
O
Ph
N
I
O
1l
200
150
100
50
0
PPM
lwb-18-65-C300
USER: -- DATE: Dec 12 2011
F1: 75.450
F2: 300.028
SW1: 18657
OF1: 7847.7
PTS1d: 27985 , 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
NA: 228
LB: 0.0
Nuts - Sjmq7-67-300c.fid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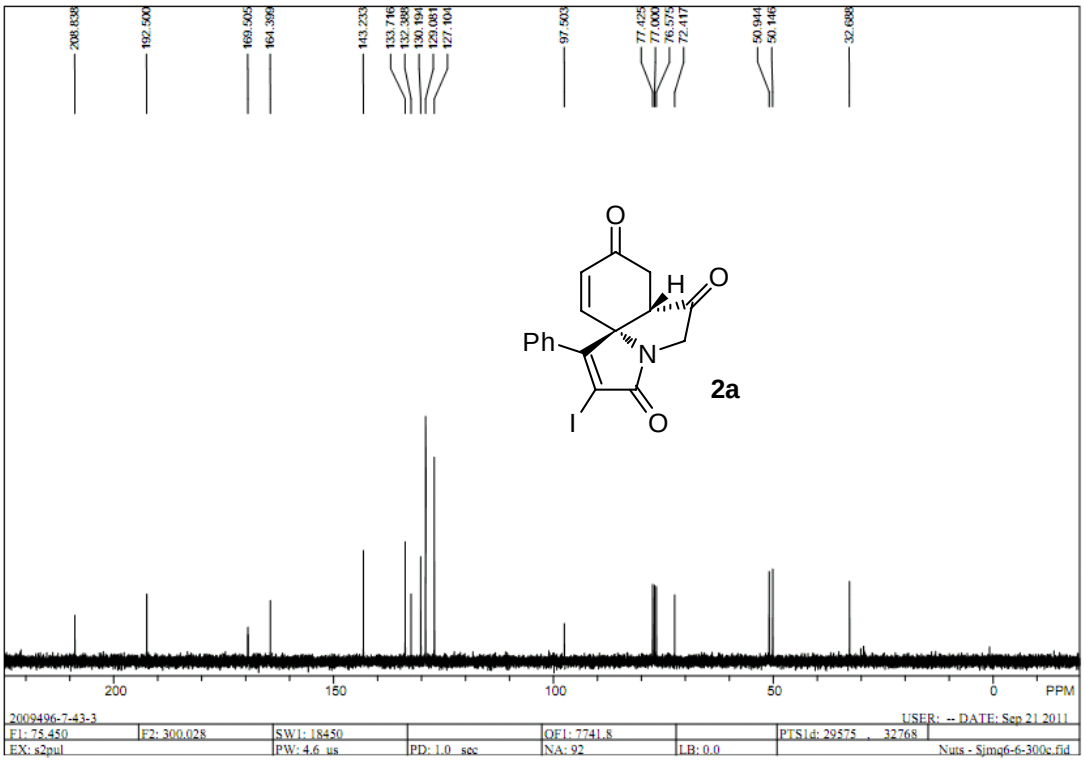
7.300
7.287
6.636
6.602
6.221
6.188
4.482
4.419
3.656
3.593
2.956
2.896
2.873
2.155
2.135
2.097
2.073
0.048
O
H
O
Ph
N
I
O
2a
3.17
2.13
0.98
0.91
1.00
1.02
2.06
1.27
10
8
6
4
2
0
PPM
jmq6-6-300h
:blank line
USER: -- DATE: Sep 21 2011
F1: 300.029
F2: 300.028
SW1: 10000
OF1: 2048.2
PTS1d: 23000 , 32768
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 8
LB: 0.0
Nuts - Sjmq6-6re300.fid


208.838
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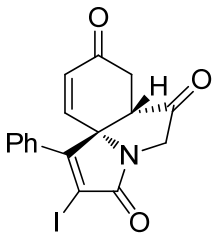
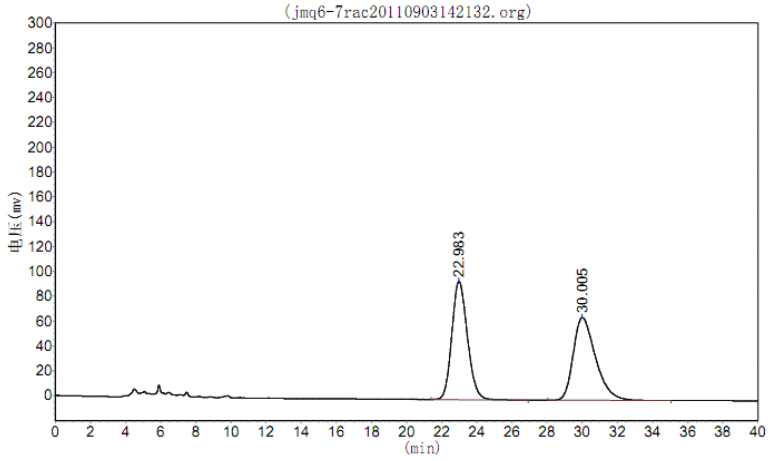
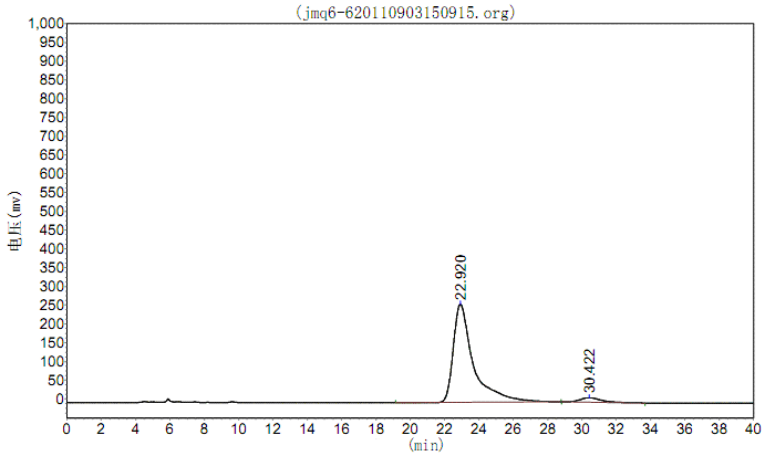
169.505
164.399
143.233
133.746
132.388
130.194
129.081
127.104
97.503
77.425
77.000
76.575
72.417
50.944
50.146
32.688
O
H
O
Ph
N
I
O
2a
200
150
100
50
0
PPM
2009496-7-43-3
USER: -- DATE: Sep 21 2011
F1: 75.450
F2: 300.028
SW1: 18450
OF1: 7741.8
PTS1d: 29575 , 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
NA: 92
LB: 0.0
Nuts - Sjmq6-6-300c.fid



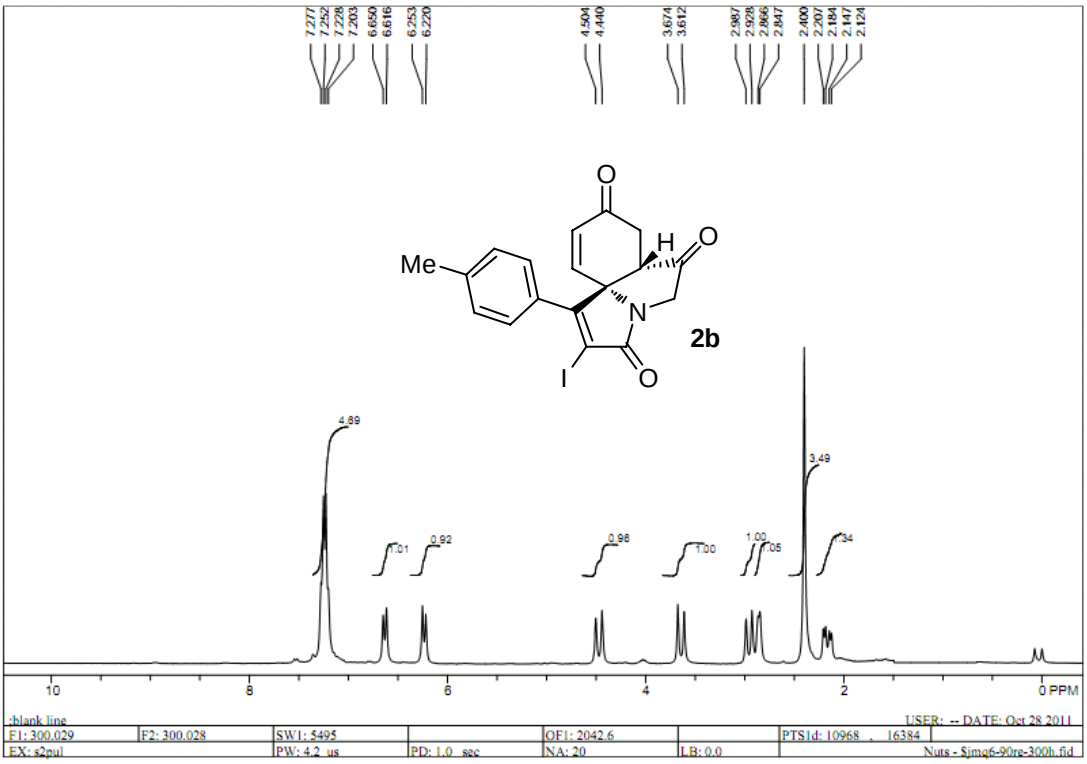
2a



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	22.983	94982.844	5851248.500	49.8538
2	30.005	66481.789	5885561.500	50.1462
Total		161464.633	11736810.000	100.0000



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	22.920	261075.406	21432194.000	95.5026
2	30.422	11519.874	1009282.813	4.4974
Total		272595.280	22441476.813	100.0000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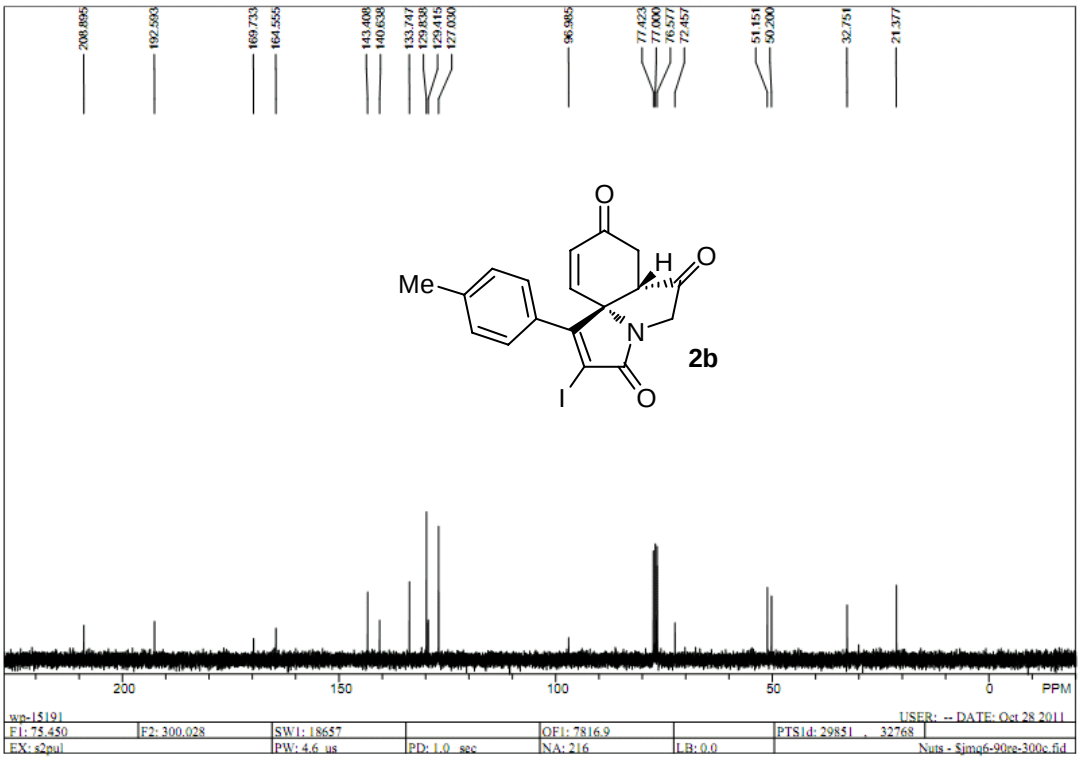
7.252
7.228
7.203
6.650
6.616
6.253
6.220
4.504
4.440
3.674
3.612
2.987
2.928
2.866
2.847
2.400
2.207
2.184
2.147
2.124
Me
H
O
N
I
2b
4.89
3.49
1.01
0.92
0.98
1.00
1.00
1.05
0.34
10
8
6
4
2
0 PPM
-blank line
USER: -- DATE: Oct 28 2011
F1: 300.029
F2: 300.028
SW1: 5495
OF1: 2042.6
PTS1d: 10968 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 20
LB: 0.0
Nuts - $jmq6-90re-300h.fid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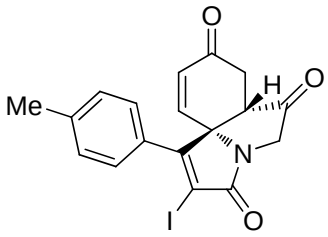
169.733
164.555
143.408
140.638
133.747
129.838
129.415
127.030
96.985
77.423
77.000
76.577
72.457
51.151
50.260
32.751
21.377
Me
H
O
N
I
2b
200
150
100
50
0
PPM
wp-15191
USER: -- DATE: Oct 28 2011
F1: 75.450
F2: 300.028
SW1: 18657
OF1: 7816.9
PTS1d: 29851 , 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
NA: 216
LB: 0.0
Nuts - $jmq6-90re-300c.fid



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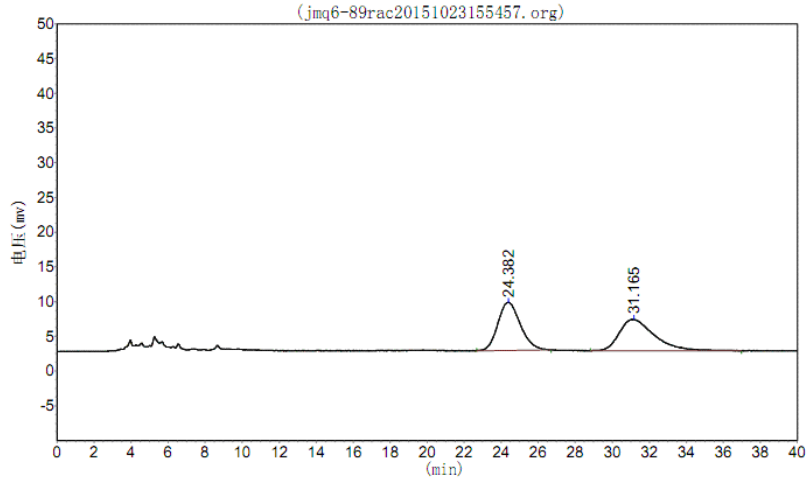
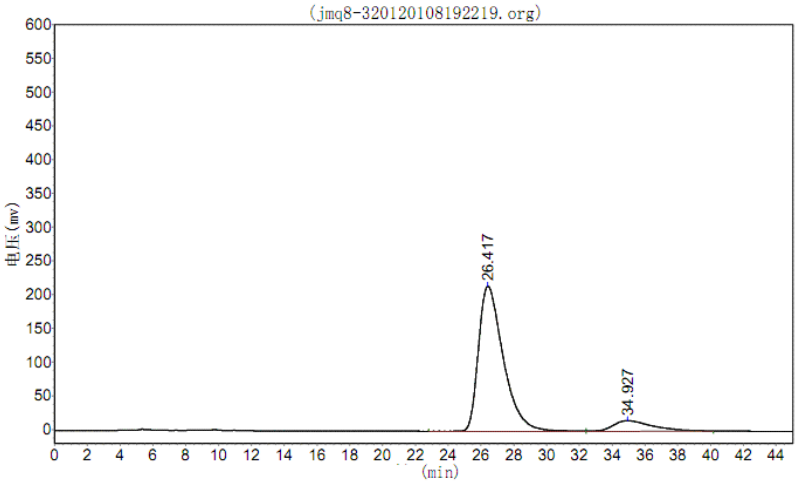
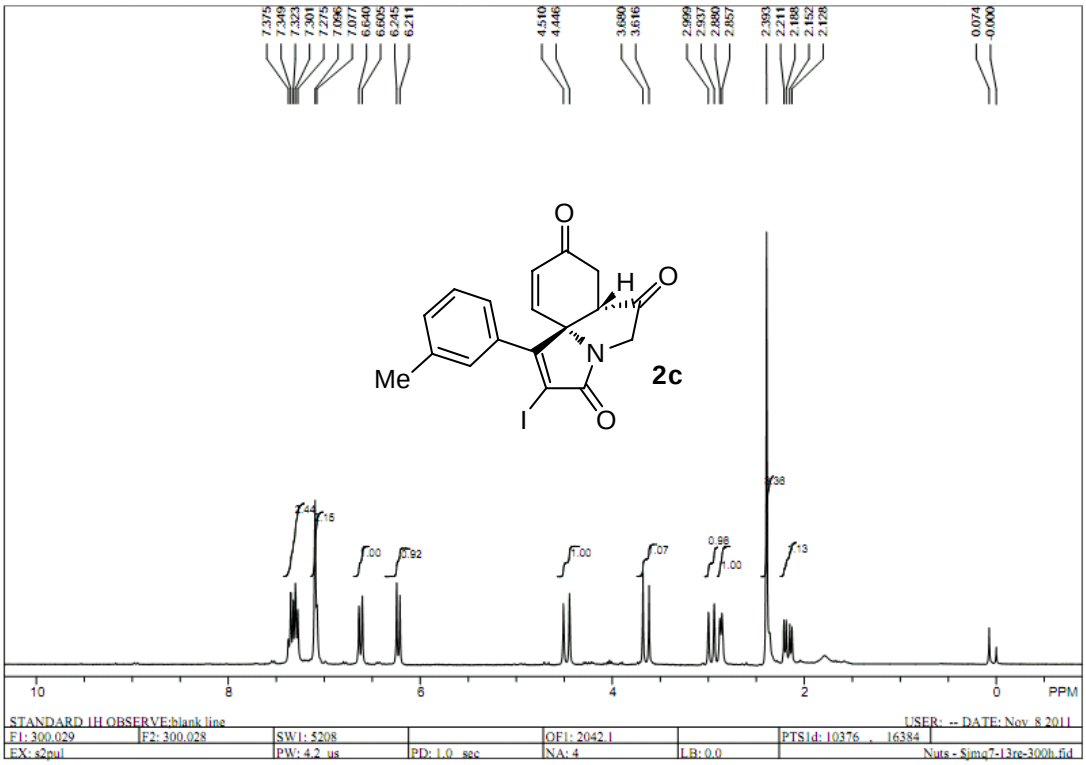


Peak No.	R. Time	Peak Height	Peak Area	Percent
1	24.382	6927.291	584525.813	49.4863
2	31.165	4500.196	596660.188	50.5137
Total		11427.487	1181186.000	100.0000



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	26.417	215008.063	22605450.000	89.7579
2	34.927	15488.554	2579474.750	10.2421
Total		230496.616	25184924.750	100.0000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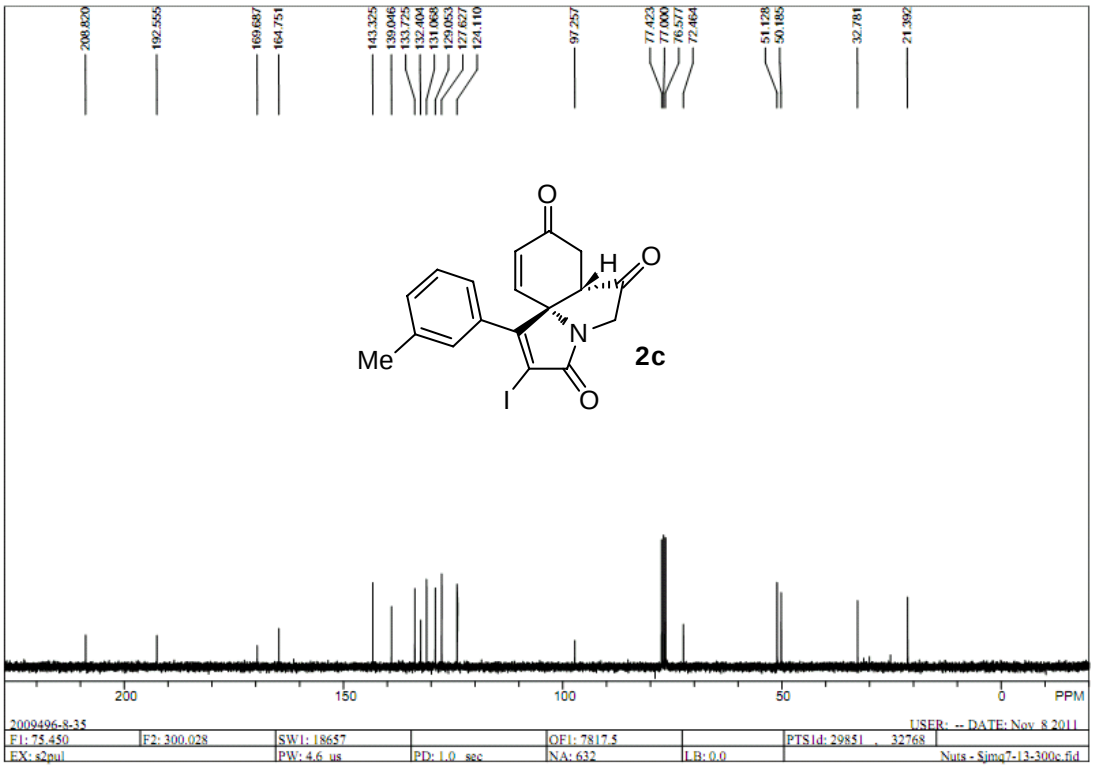
7.301
7.275
7.096
7.077
6.640
6.605
6.245
6.211
4.510
4.446
3.680
3.616
2.999
2.937
2.880
2.857
2.393
2.211
2.188
2.152
2.128
0.074
0.000
O
H
O
N
Me
2c
I
O
1.44
1.15
1.00
0.92
1.00
1.07
0.98
1.00
3.38
1.13
10
8
6
4
2
0
PPM
STANDARD 1H OBSERVE:blank line
USER: -- DATE: Nov 8 2011
F1: 300.029
F2: 300.028
SW1: 5208
OF1: 2042.1
PTS1d: 10376 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 4
LB: 0.0
Nuts - Sjmq7-13re-300h.fid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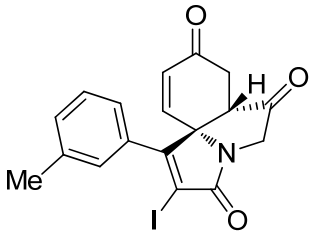
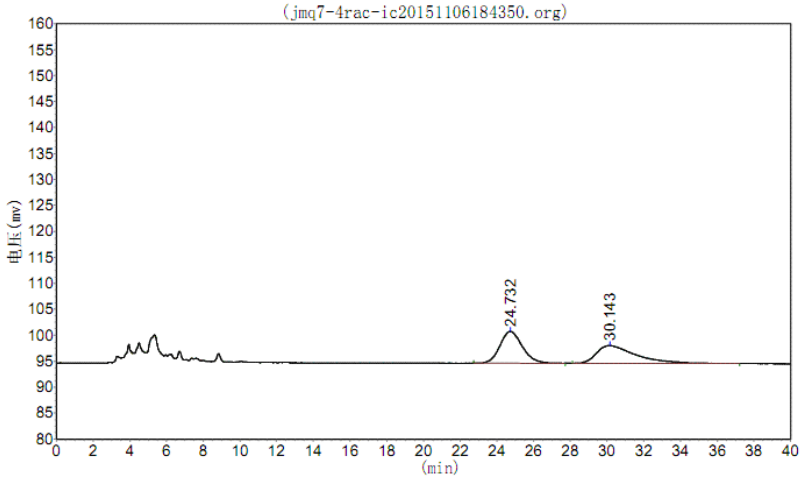
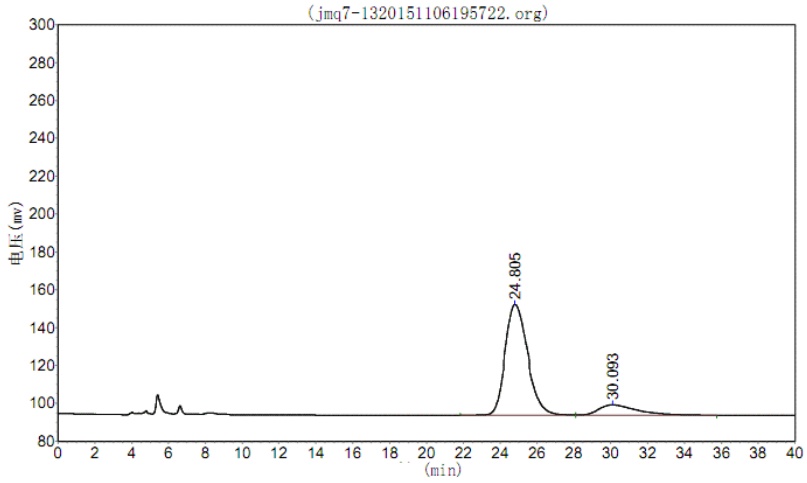
164.751
143.325
139.046
133.725
132.404
131.068
129.053
127.627
124.110
97.257
77.423
77.000
76.577
72.464
51.128
50.185
32.781
21.392
O
H
O
N
Me
2c
I
O
200
150
100
50
0
PPM
2009496-8-35
USER: -- DATE: Nov 8 2011
F1: 75.450
F2: 300.028
SW1: 18657
OF1: 7817.5
PTS1d: 29851 , 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
NA: 632
LB: 0.0
Nuts - Sjmq7-13-300c.fid



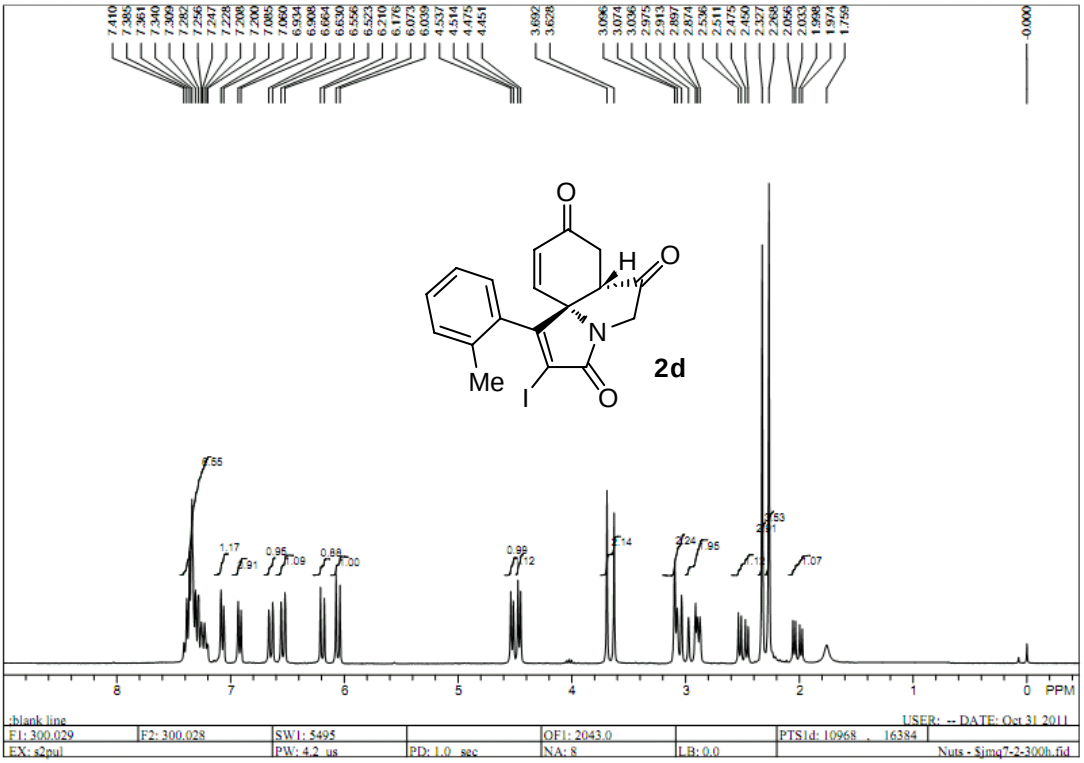
2c

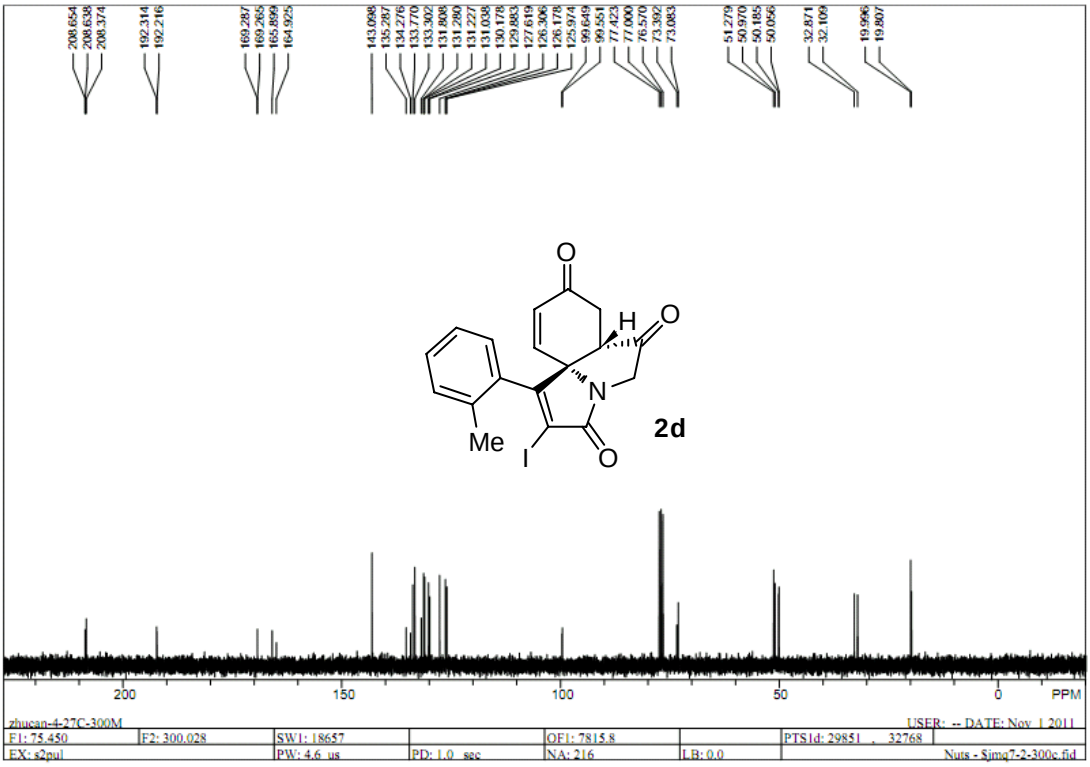


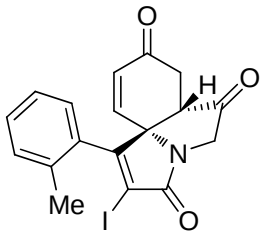
Peak No.	R. Time	Peak Height	Peak Area	Percent
1	24.732	6095.193	518561.813	50.4839
2	30.143	3426.758	508621.563	49.5161
Total		9521.951	1027183.375	100.0000



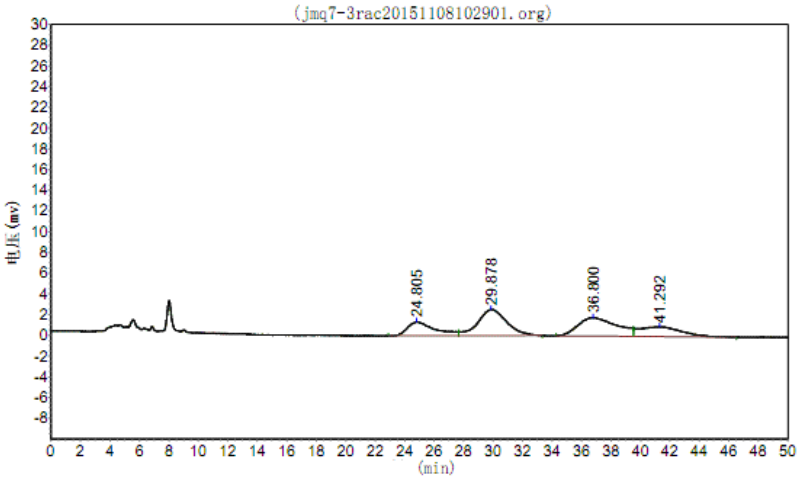
Peak No.	R. Time	Peak Height	Peak Area	Percent
1	24.805	58587.203	5019201.500	85.6634
2	30.093	5649.789	840012.438	14.3366
Total		64236.992	5859213.938	100.0000


2d
Me
I
PPM
:blank line
F1: 300.029
F2: 300.028
SW1: 5495
OF1: 2043.0
PTS1d: 10968 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 8
LB: 0.0
Nuts - $jmq7-2-300h.fid

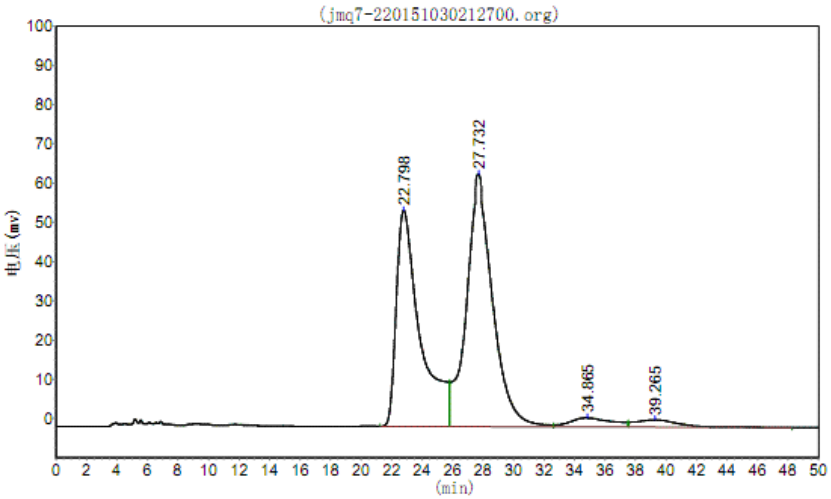

2d
Me
I
PPM
zhucan-4-27C-300M
F1: 75.450
F2: 300.028
SW1: 18657
OF1: 7815.8
PTS1d: 29851 , 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
NA: 216
LB: 0.0
Nuts - $jmq7-2-300c.fid



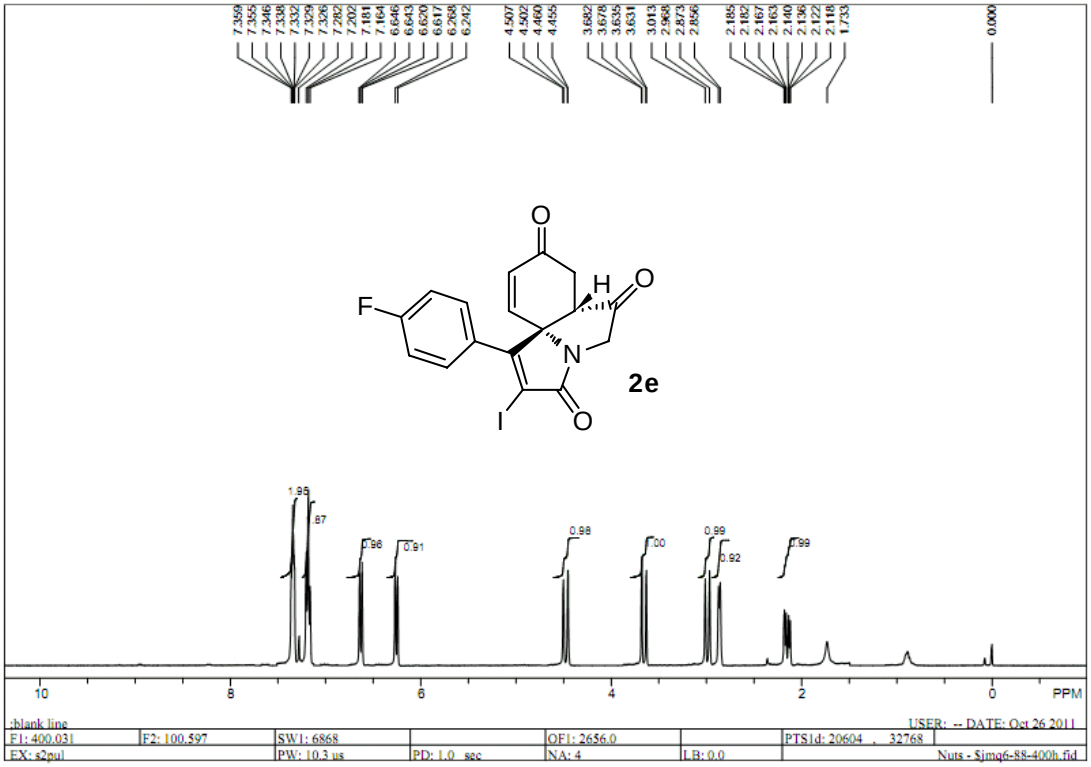
2d



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	24.805	1344.226	175959.891	17.0314
2	29.878	2514.892	342507.219	33.1517
3	36.800	1776.848	324146.813	31.3746
4	41.292	937.688	190537.484	18.4424
Total		6573.655	1033151.406	100.0000



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	22.798	54999.484	5968632.500	41.3929
2	27.732	64398.902	7642182.000	52.9991
3	34.865	2293.226	447360.344	3.1025
4	39.265	1888.032	361288.281	2.5056
Total		123579.645	14419463.125	100.0000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7.332
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7.282
7.202
7.181
7.164
6.646
6.643
6.620
6.617
6.268
6.242
4.507
4.502
4.460
4.455
3.682
3.678
3.635
3.631
3.013
2.968
2.873
2.856
2.185
2.182
2.167
2.163
2.140
2.136
2.122
2.118
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O
H
O
F
N
I
O
2e
1.95
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0.91
0.98
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0.92
0.99
10
8
6
4
2
0
PPM
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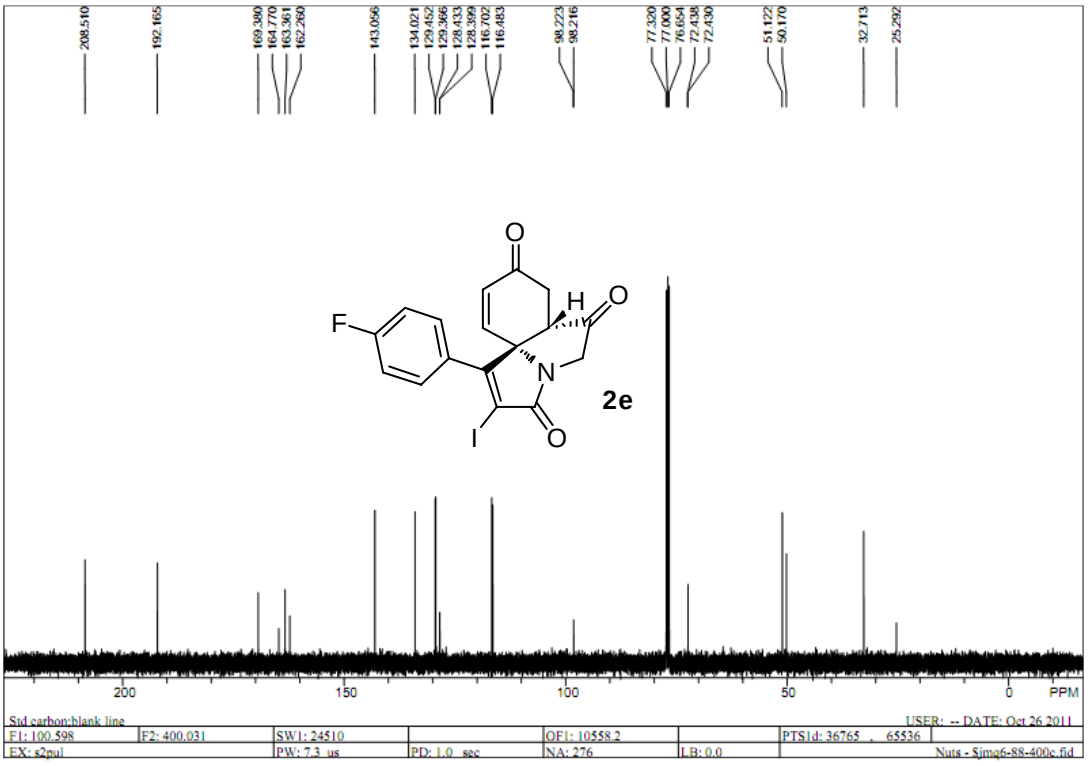
USER: -- DATE: Oct 26 2011
F1: 400.031
F2: 100.597
SW1: 6868
OF1: 2656.0
PTS1d: 20604 , 32768
EX: s2pul
PW: 10.3 us
PD: 1.0 sec
NA: 4
LB: 0.0
Nuts - Sjmq6-88-400h.fid


208.510
192.165
169.380
164.770
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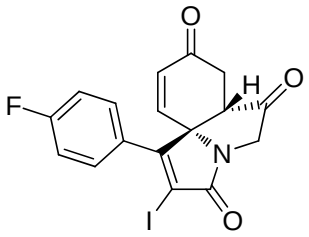
162.260
143.056
134.021
129.452
129.366
128.433
128.399
116.702
116.483
98.223
98.216
77.320
77.000
76.654
72.438
72.430
51.122
50.170
32.713
25.292
O
H
O
F
N
I
O
2e
200
150
100
50
0
PPM
Std carbon;blank line
USER: -- DATE: Oct 26 2011
F1: 100.598
F2: 400.031
SW1: 24510
OF1: 10558.2
PTS1d: 36765 , 65536
EX: s2pul
PW: 7.3 us
PD: 1.0 sec
NA: 276
LB: 0.0
Nuts - Sjmq6-88-400c.fid



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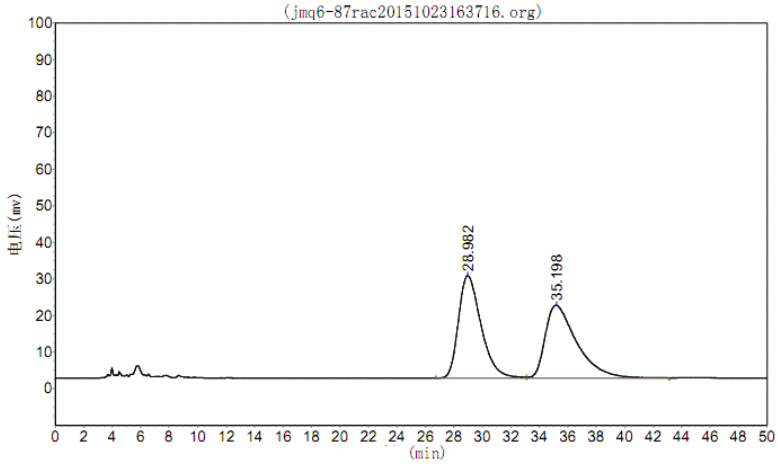
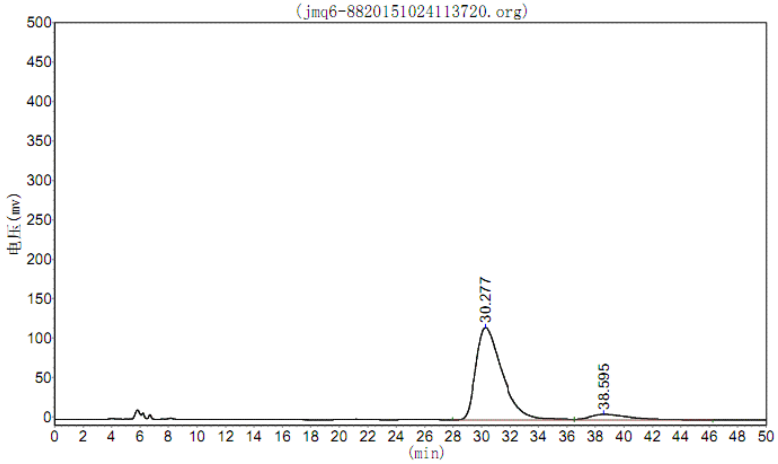
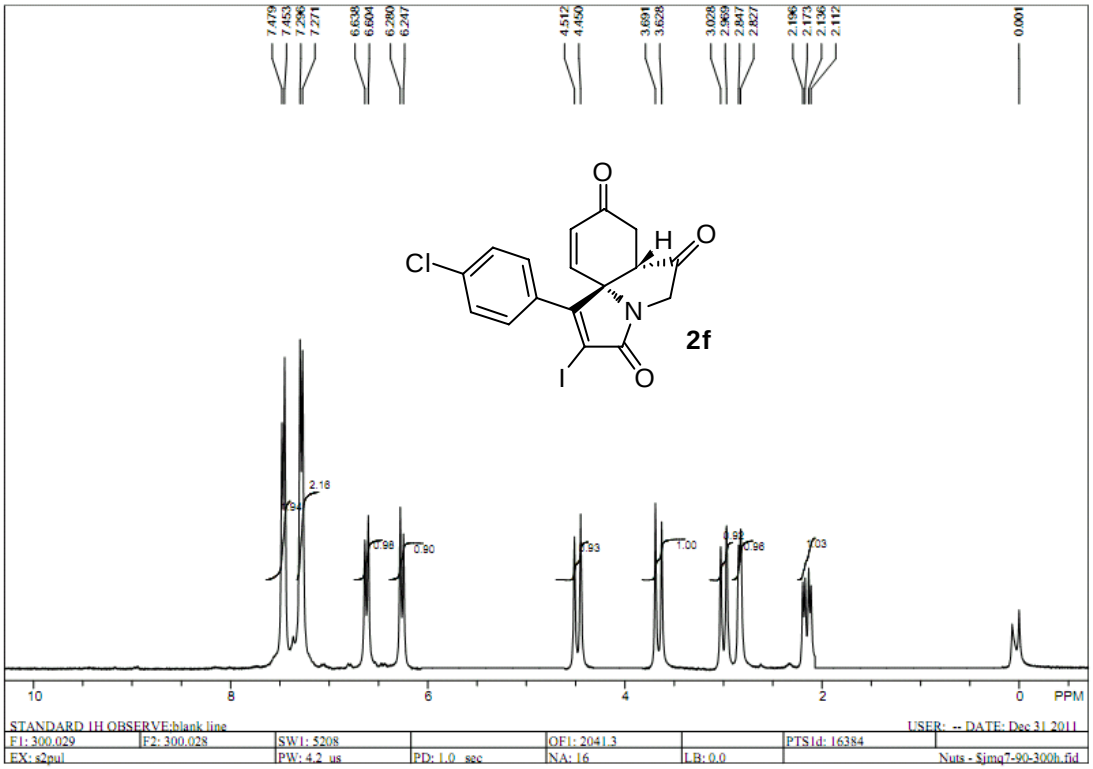


Peak No.	R. Time	Peak Height	Peak Area	Percent
1	28.982	28065.439	3116736.500	50.0637
2	35.198	19967.947	3108804.250	49.9363
Total		48033.387	6225540.750	100.0000



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	30.277	116635.992	14998557.000	92.0355
2	38.595	6902.286	1297934.750	7.9645
Total		123538.278	16296491.750	100.0000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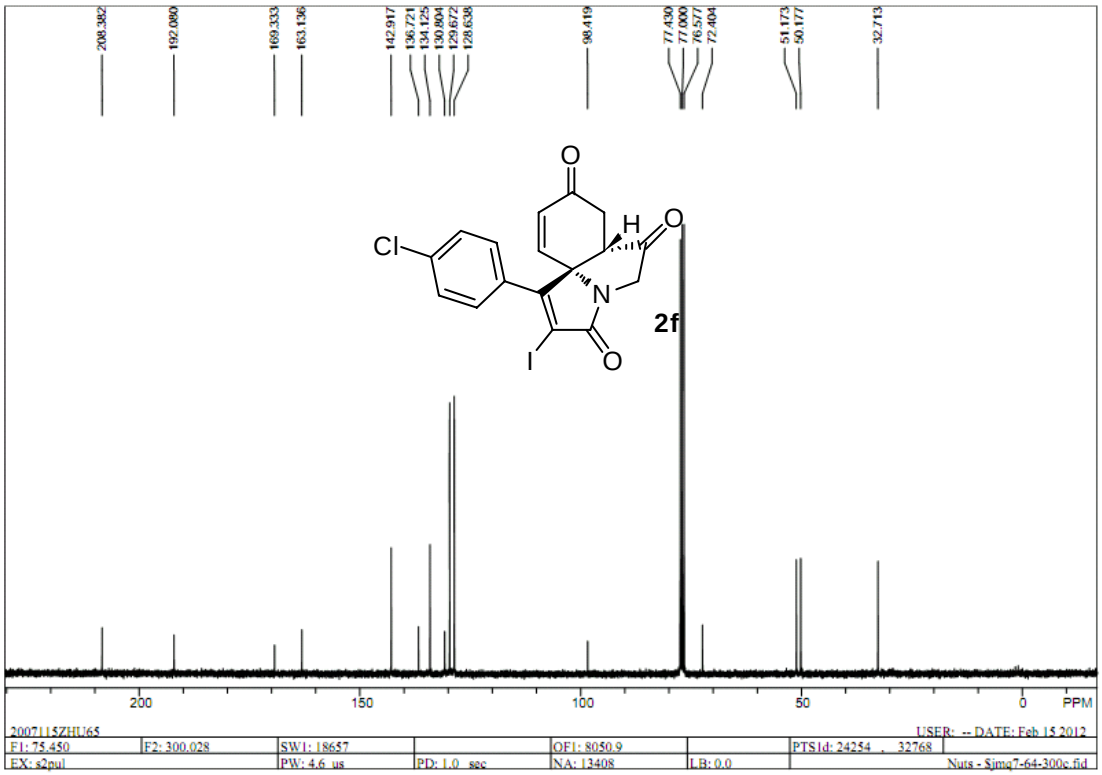
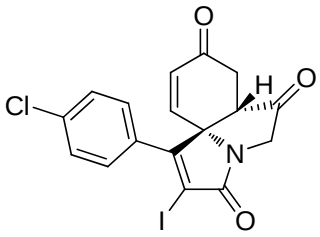
7.271
6.638
6.604
6.280
6.247
4.512
4.450
3.691
3.628
3.028
2.969
2.847
2.827
2.196
2.173
2.136
2.112
0.001
O
H
O
Cl
N
I
O
2f
2.18
1.94
0.98
0.90
0.93
1.00
0.98
1.03
10
8
6
4
2
0
PPM
STANDARD 1H OBSERVE:blank line
USER: -- DATE: Dec 31 2011
F1: 300.029
F2: 300.028
SW1: 5208
OF1: 2041.3
PTS1d: 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 16
LB: 0.0
Nuts - $jmq7-90-300h.fid


208.382
192.080
169.333
163.136
142.917
136.721
134.125
130.804
129.672
128.638
98.419
77.430
77.000
76.577
72.404
51.173
50.177
32.713
O
H
O
Cl
N
I
O
2f
200
150
100
50
0
PPM
2007115ZHU65
USER: -- DATE: Feb 15 2012
F1: 75.450
F2: 300.028
SW1: 18657
OF1: 8050.9
PTS1d: 24254 . 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
NA: 13408
LB: 0.0
Nuts - $jmq7-64-300c.fid



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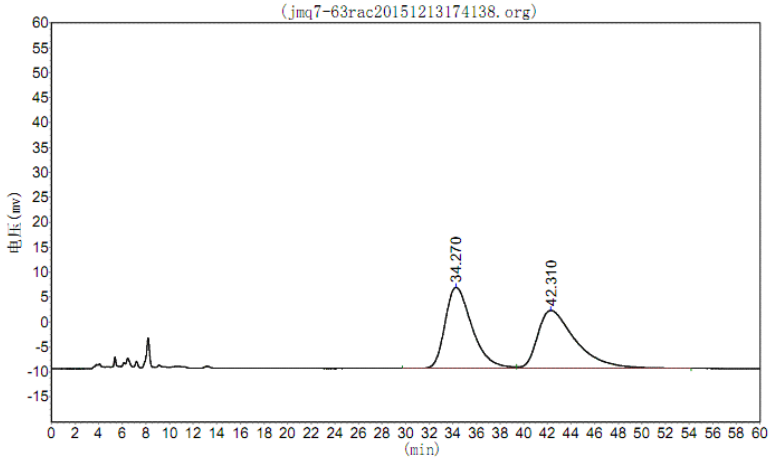
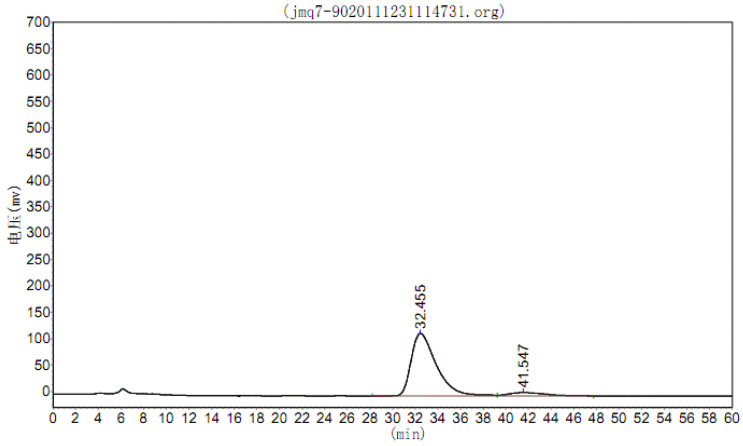
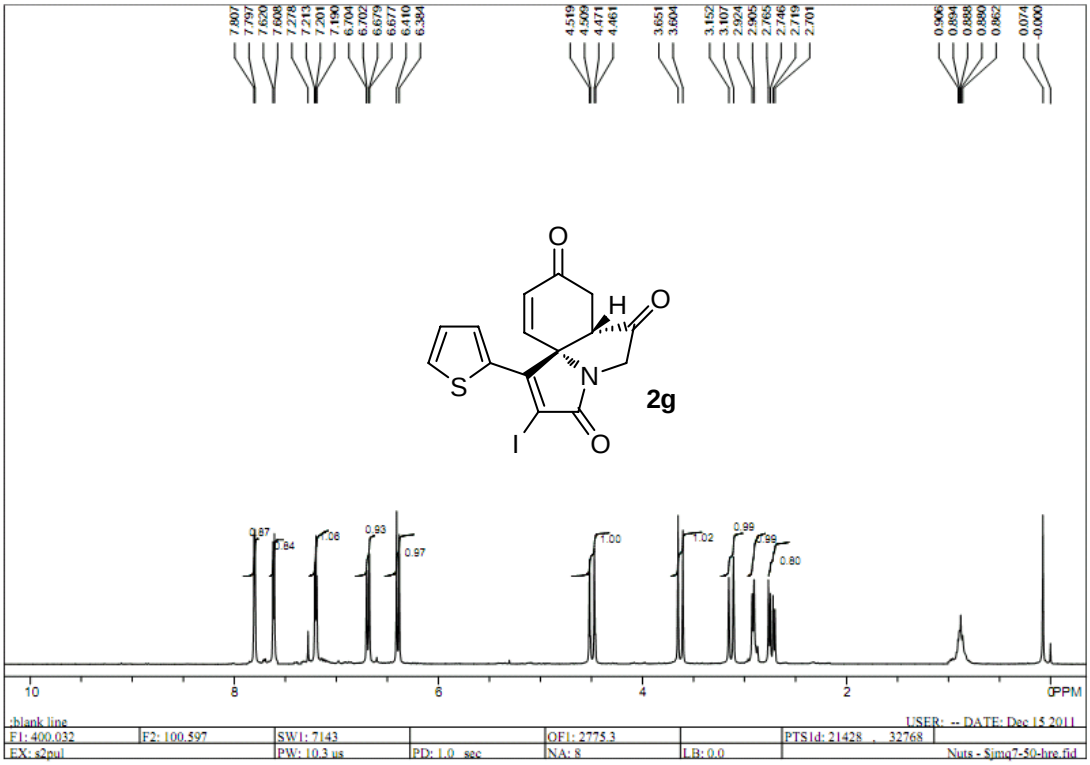


Peak No.	R. Time	Peak Height	Peak Area	Percent
1	34.270	16280.342	2581356.000	49.8651
2	42.310	11641.716	2595319.250	50.1349
Total		27922.058	5176675.250	100.0000



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	32.455	118558.094	18391662.000	92.8061
2	41.547	6509.462	1425629.375	7.1939
Total		125067.556	19817291.375	100.0000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7.797
7.620
7.608
7.278
7.213
7.201
7.190
6.704
6.702
6.679
6.667
6.410
6.384
4.519
4.509
4.471
4.461
3.651
3.604
3.152
3.107
2.924
2.905
2.765
2.746
2.719
2.701
0.906
0.894
0.888
0.880
0.862
0.074
0.000
O
H
O
S
N
I
O
2g
0.87
0.84
1.08
0.93
0.97
1.00
1.02
0.99
0.99
0.80
10
8
6
4
2
0PPM
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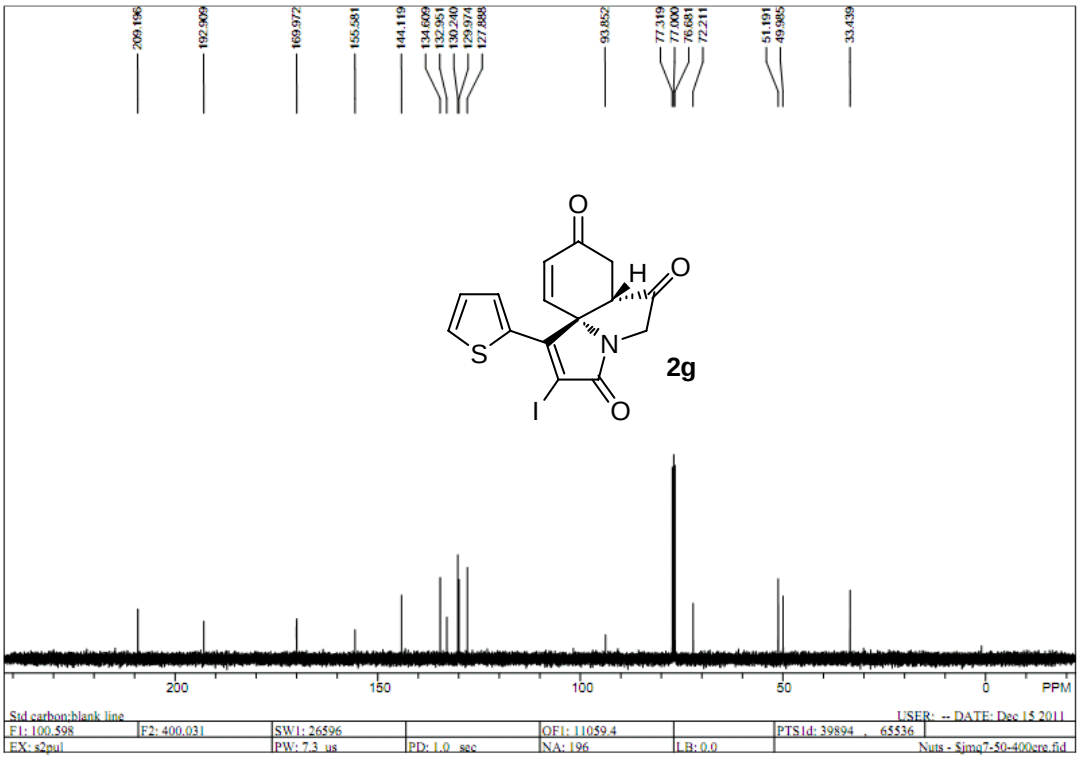
USER: -- DATE: Dec 15 2011
F1: 400.032
F2: 100.597
SW1: 7143
OF1: 2775.3
PTS1d: 21428 , 32768
EX: s2pul
PW: 10.3 us
PD: 1.0 sec
NA: 8
LB: 0.0
Nuts - Sjmq7-50-hre.fid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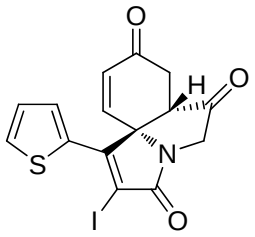
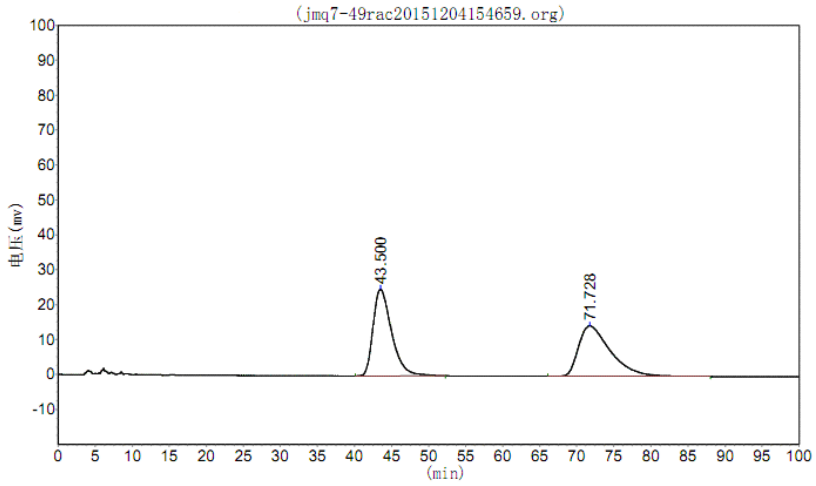
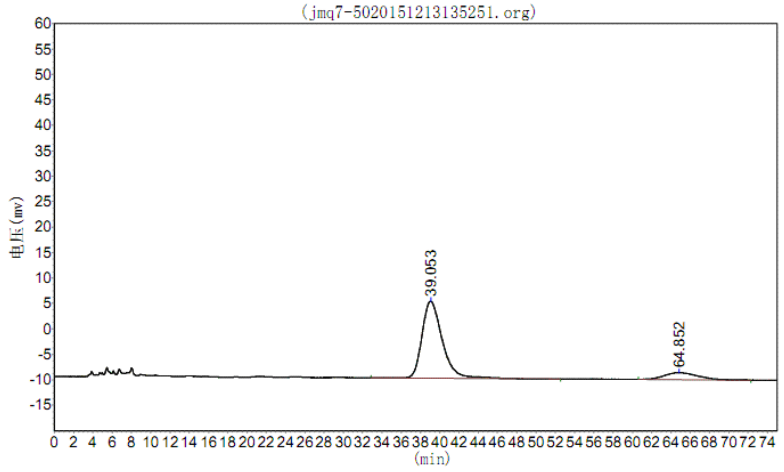
169.972
155.581
144.119
134.609
132.951
130.240
129.974
127.888
93.852
77.319
77.000
76.681
72.211
51.191
49.985
33.439
O
H
O
S
N
I
O
2g
200
150
100
50
0
PPM
Std carbon:blank line
USER: -- DATE: Dec 15 2011
F1: 100.598
F2: 400.031
SW1: 26596
OF1: 11059.4
PTS1d: 39894 , 65536
EX: s2pul
PW: 7.3 us
PD: 1.0 sec
NA: 196
LB: 0.0
Nuts - Sjmq7-50-400cre.fid



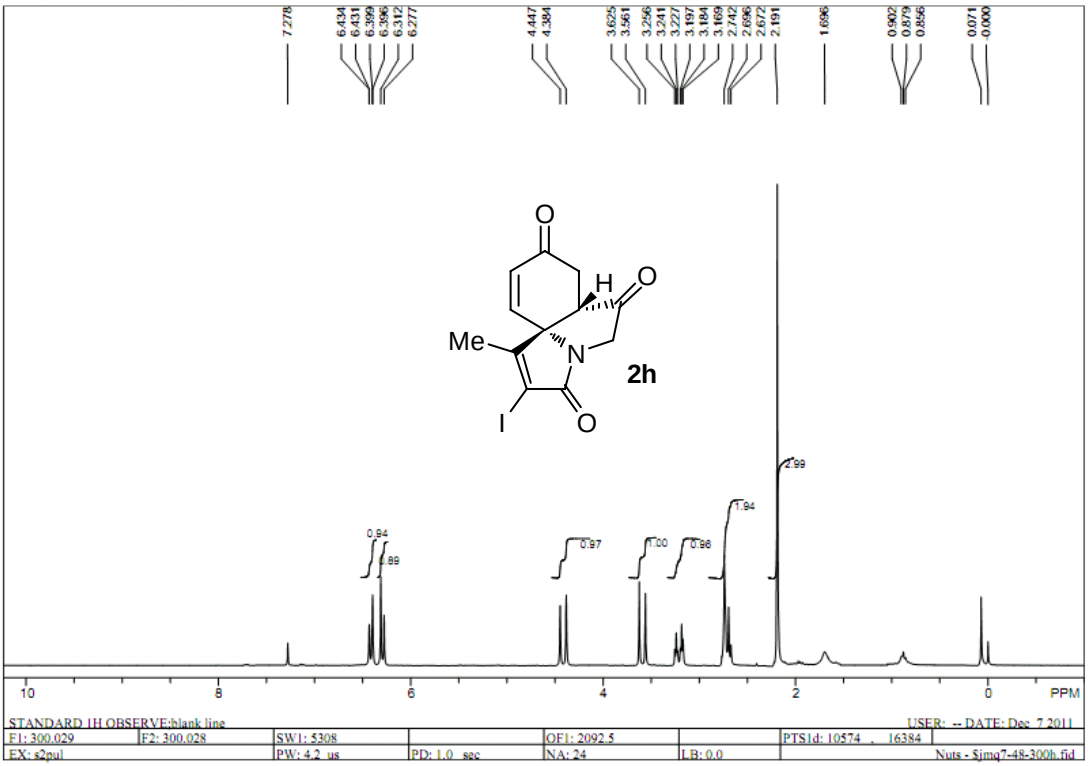
2g



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	43.500	24822.111	4342897.000	50.0326
2	71.728	14445.268	4337233.500	49.9674
Total		39267.379	8680130.500	100.0000



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	39.053	15117.240	2219172.500	87.4047
2	64.852	1371.756	319790.938	12.5953
Total		16488.996	2538963.438	100.0000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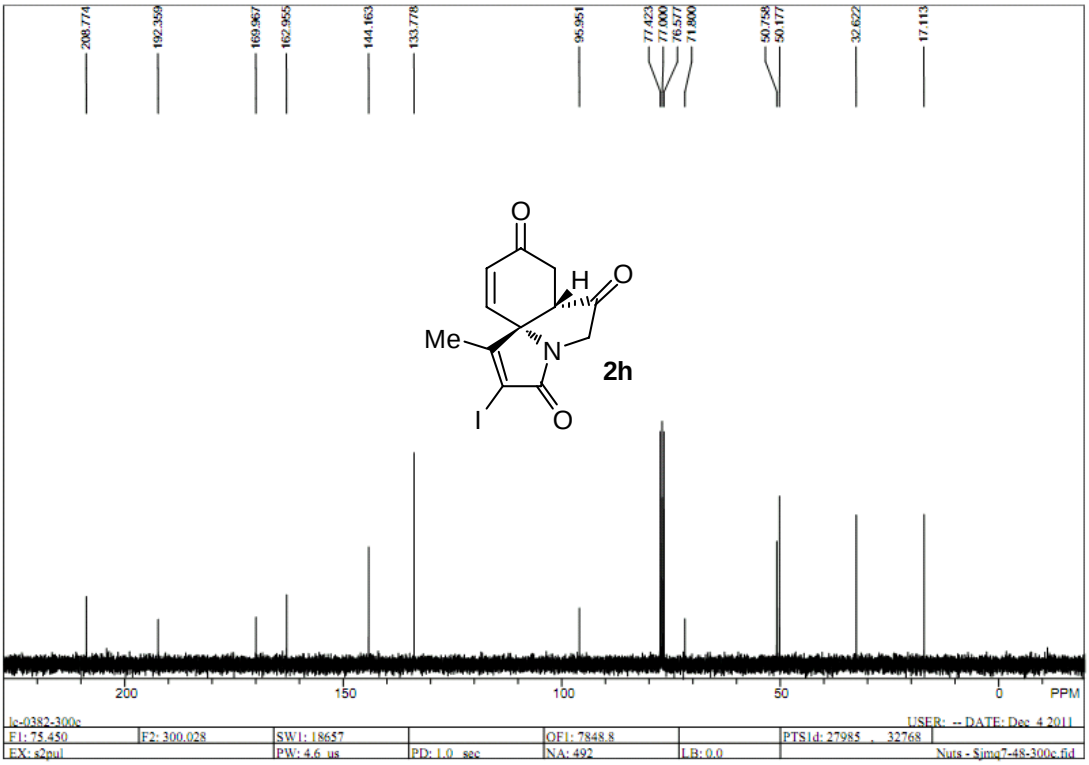
6.399
6.396
6.312
6.277
4.447
4.384
3.625
3.561
3.256
3.241
3.227
3.197
3.184
3.169
2.742
2.696
2.672
2.191
1.696
0.902
0.879
0.856
0.071
0.000
O
H
O
Me
N
2h
I
O
0.94
0.89
0.97
1.00
0.98
1.94
2.99
10
8
6
4
2
0
PPM
STANDARD 1H OBSERVE;blank line
USER: -- DATE: Dec 7 2011
F1: 300.029
F2: 300.028
SW1: 5308
OF1: 2092.5
PTS1d: 10574 . 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 24
LB: 0.0
Nuts - $jmq7-48-300h.fid


208.774
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169.967
162.955
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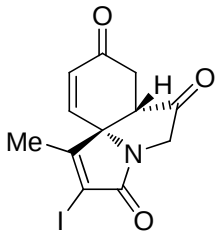
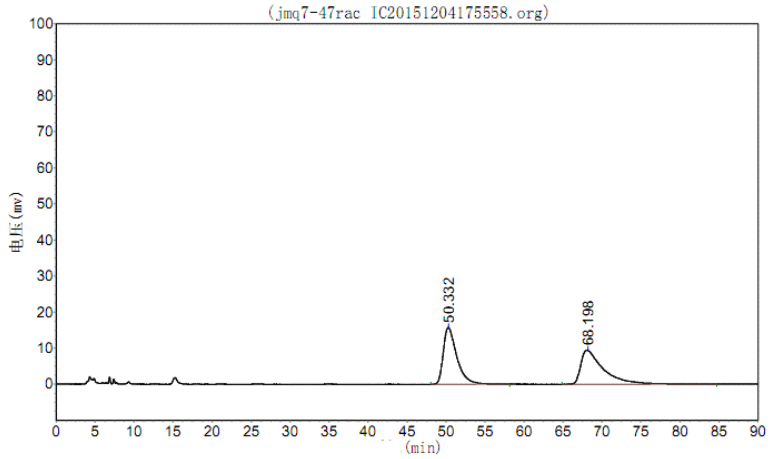
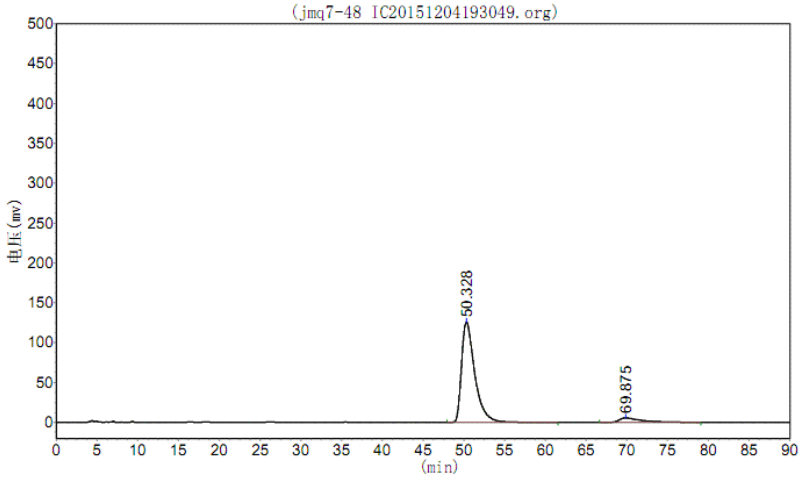
95.951
77.423
77.000
76.577
71.800
50.758
50.177
32.622
17.113
O
H
O
Me
N
2h
I
O
200
150
100
50
0
PPM
jc-0382-300c
USER: -- DATE: Dec 4 2011
F1: 75.450
F2: 300.028
SW1: 18657
OF1: 7848.8
PTS1d: 27985 . 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
NA: 492
LB: 0.0
Nuts - $jmq7-48-300c.fid



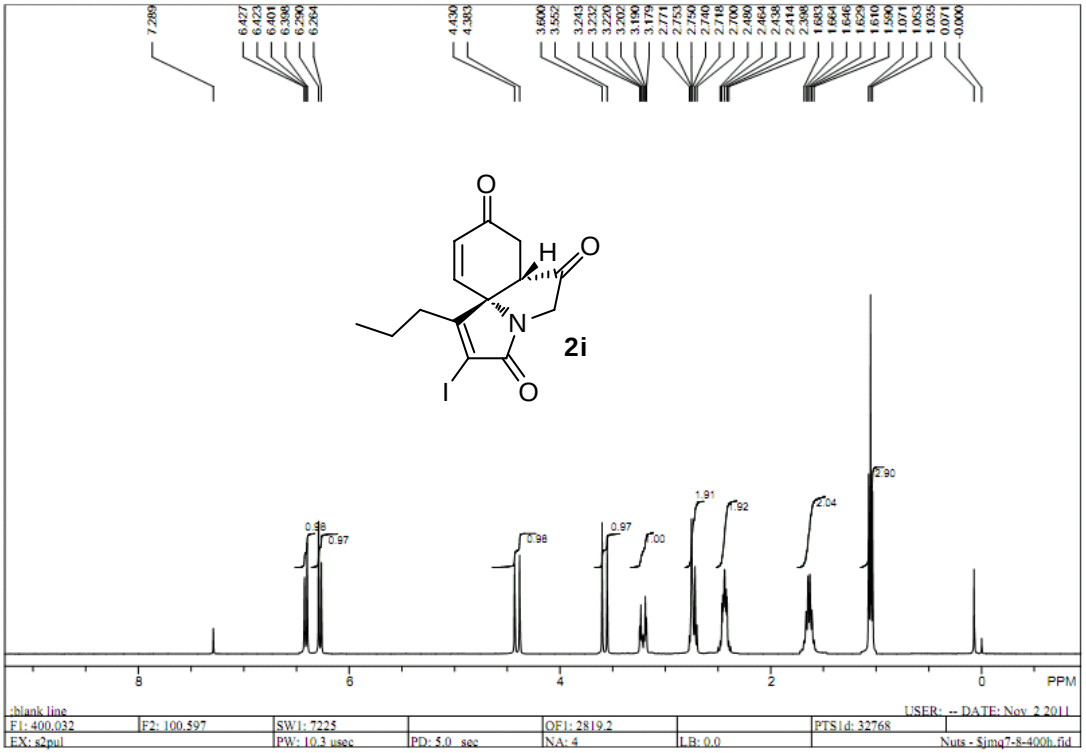
2h



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	50.332	15760.967	1917566.750	49.9181
2	68.198	9443.953	1923859.375	50.0819
Total		25204.920	3841426.125	100.0000



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	50.328	125702.047	14276741.000	92.8413
2	69.875	5393.104	1100829.750	7.1587
Total		131095.151	15377570.750	100.0000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6.401
6.398
6.290
6.264
4.430
4.383
3.600
3.552
3.243
3.232
3.220
3.202
3.190
3.179
2.771
2.753
2.750
2.740
2.718
2.700
2.480
2.464
2.438
2.414
2.398
1.683
1.664
1.646
1.629
1.610
1.590
1.071
1.053
1.035
0.071
-0.000
O
H
O
N
2i
I
O
0.98
0.97
0.98
0.97
1.00
1.91
1.92
2.04
2.90
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6
4
2
0
PPM
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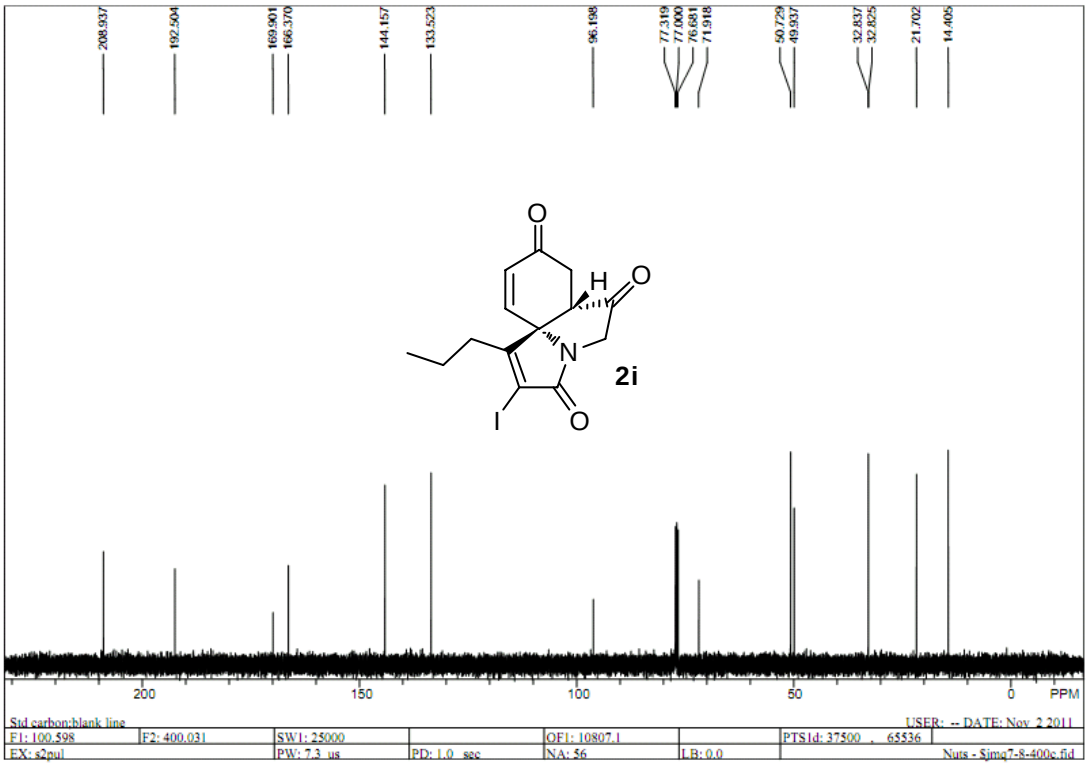
USER: -- DATE: Nov 2 2011
F1: 400.032
F2: 100.597
SW1: 7225
OF1: 2819.2
PTS1d: 32768
EX: s2pul
PW: 10.3 usec
PD: 5.0 sec
NA: 4
LB: 0.0
Nuts - $jmq7-8-400h.fid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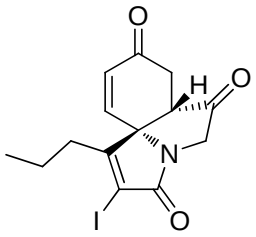
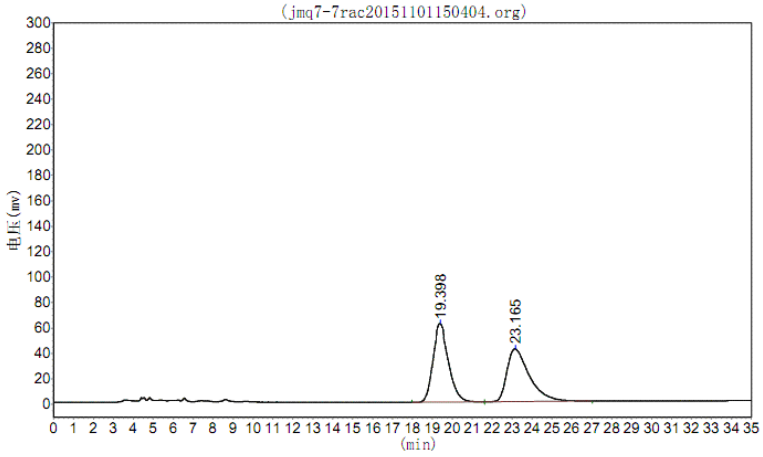
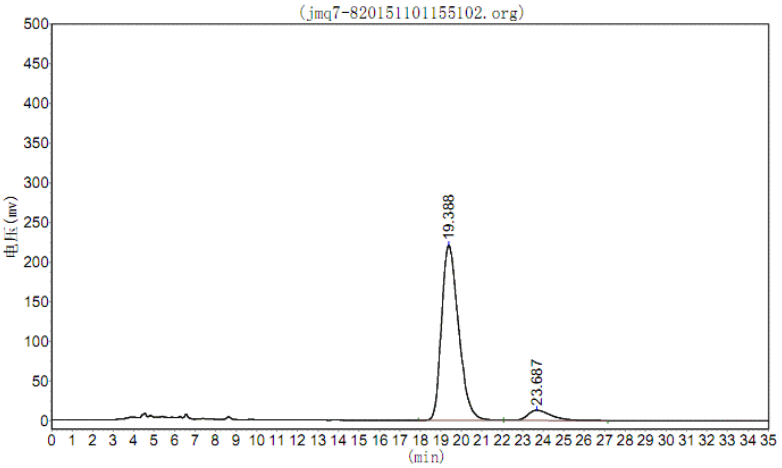
169.901
166.370
144.157
133.523
96.198
77.319
77.000
76.681
71.918
50.729
49.937
32.837
32.825
21.702
14.405
O
H
O
N
2i
I
O
200
150
100
50
0
PPM
Std carbon:blank line
USER: -- DATE: Nov 2 2011
F1: 100.598
F2: 400.031
SW1: 25000
OF1: 10807.1
PTS1d: 37500 , 65536
EX: s2pul
PW: 7.3 us
PD: 1.0 sec
NA: 56
LB: 0.0
Nuts - $jmq7-8-400c.fid



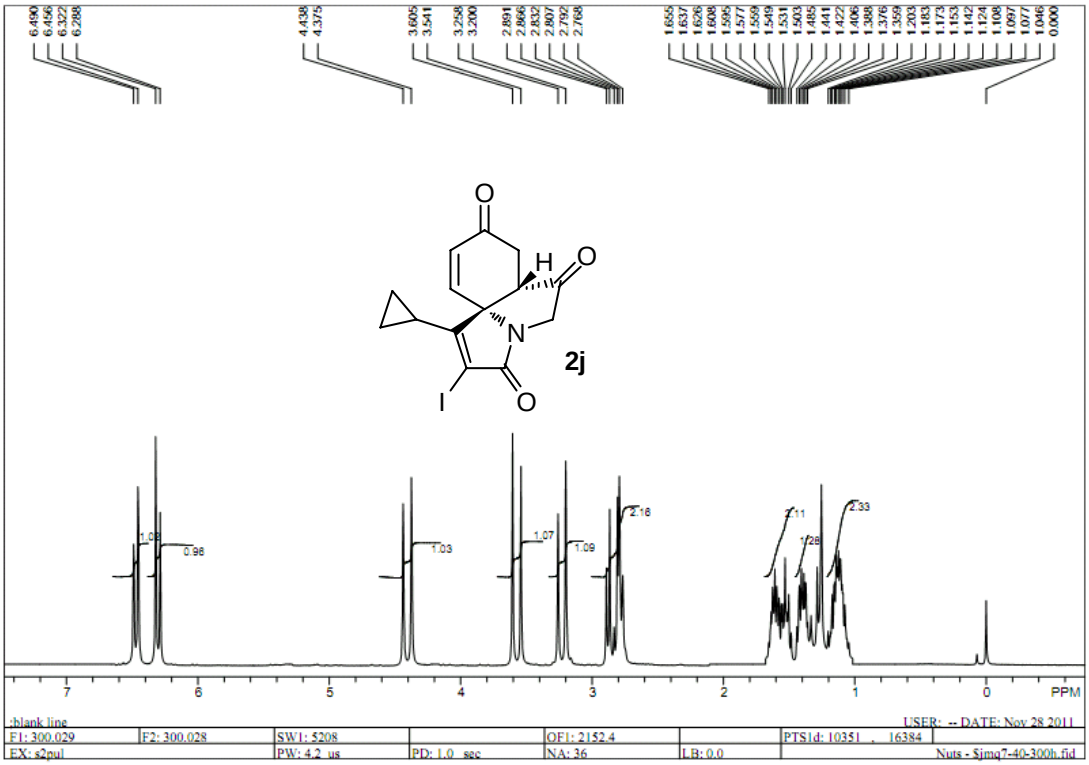
2i



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	19.398	61906.191	3313582.000	50.4766
2	23.165	41490.691	3251013.250	49.5234
Total		103396.883	6564595.250	100.0000



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	19.388	220360.063	12744896.000	92.3985
2	23.687	13079.066	1048501.313	7.6015
Total		233439.129	13793397.312	100.0000


6.490
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6.288
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4.375
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3.541
3.258
3.200
2.891
2.866
2.832
2.807
2.792
2.768
1.655
1.637
1.626
1.608
1.595
1.577
1.559
1.549
1.531
1.503
1.485
1.441
1.422
1.406
1.388
1.376
1.359
1.203
1.183
1.173
1.153
1.142
1.124
1.108
1.097
1.077
1.046
0.000
O
H
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2j
I
O
1.08
0.98
1.03
1.07
1.09
2.16
2.11
1.28
2.33
7
6
5
4
3
2
1
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PPM
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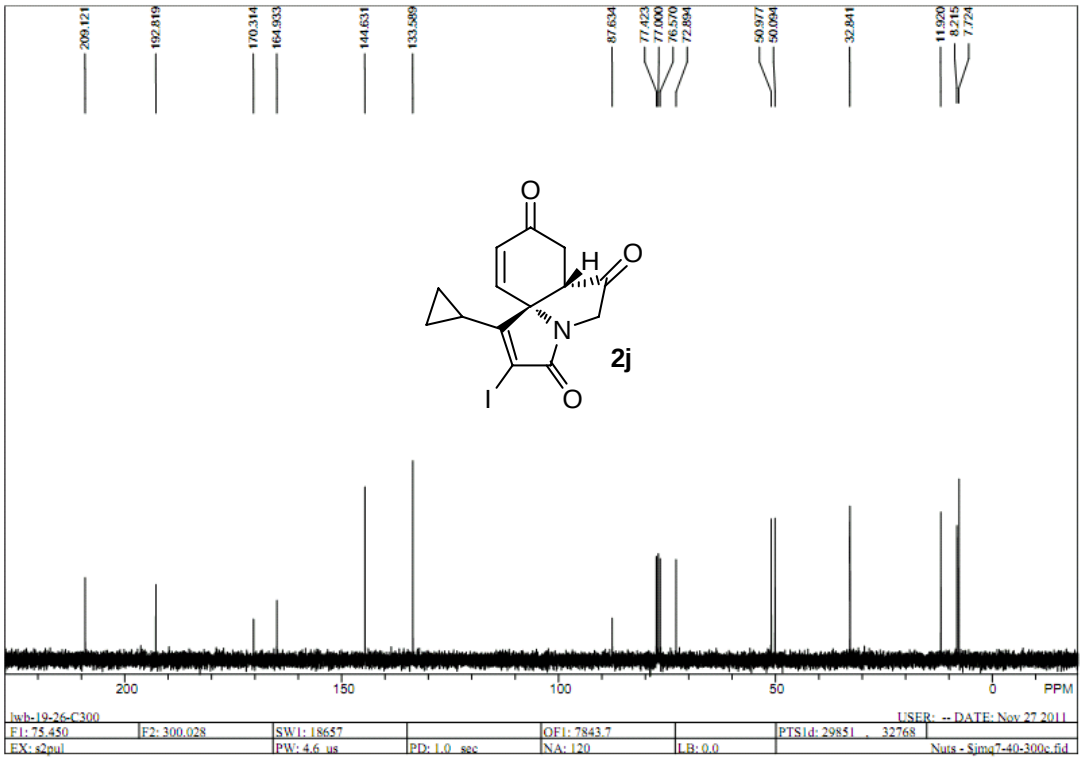
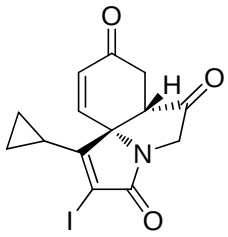
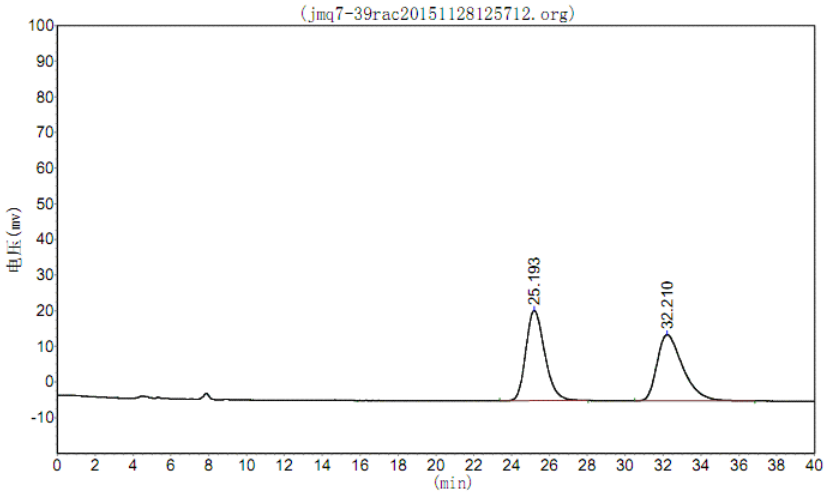
USER: -- DATE: Nov 28 2011
F1: 300.029
F2: 300.028
SW1: 5208
OF1: 2152.4
PTS1d: 10351 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 36
LB: 0.0
Nuts - Sjmq7-40-300h.fid

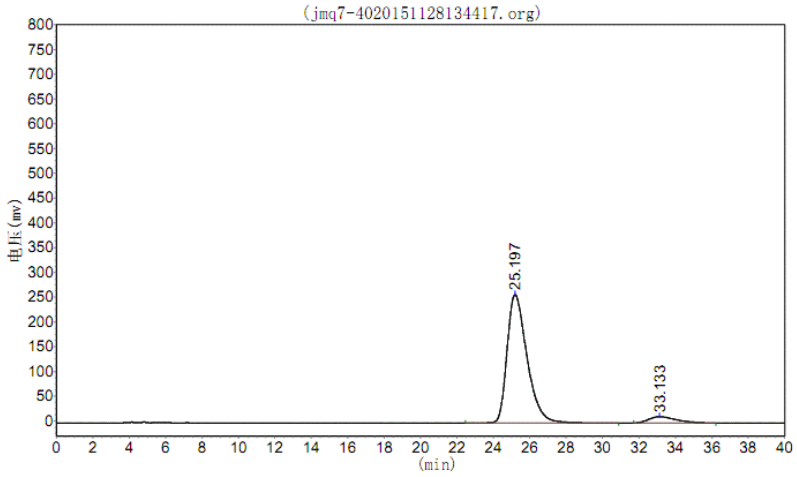

209.121
192.819
170.314
164.933
144.631
133.589
87.634
77.423
77.000
76.570
72.894
50.977
50.094
32.841
11.920
8.215
7.724
O
H
O
N
2j
I
O
200
150
100
50
0
PPM
lwb-19-26-C300
USER: -- DATE: Nov 27 2011
F1: 75.450
F2: 300.028
SW1: 18657
OF1: 7843.7
PTS1d: 29851 , 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
NA: 120
LB: 0.0
Nuts - Sjmq7-40-300c.fid



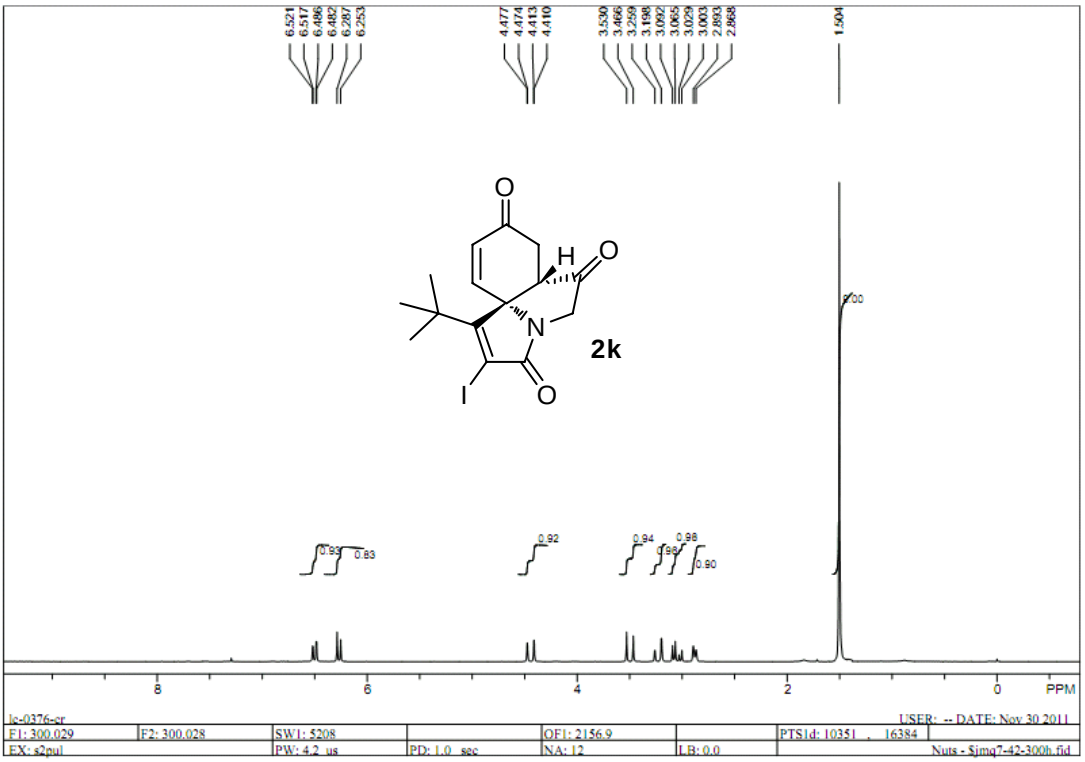
2j

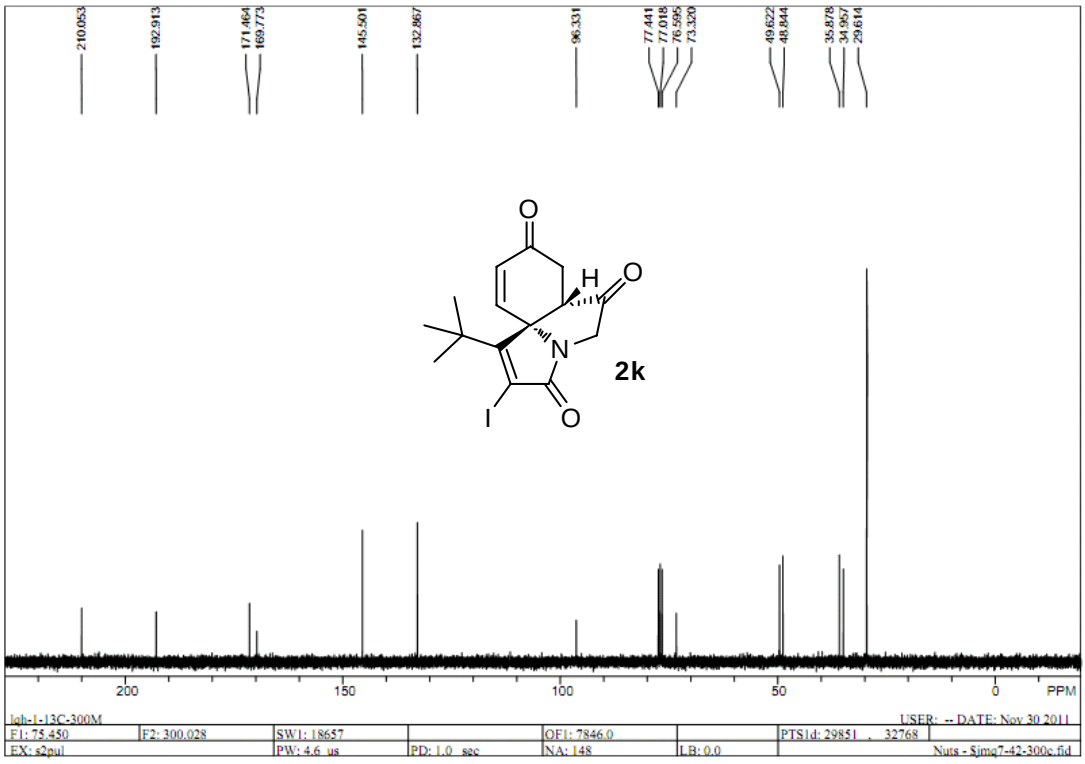


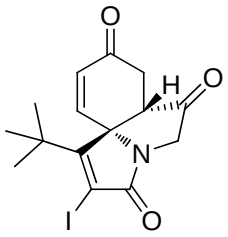
Peak No.	R. Time	Peak Height	Peak Area	Percent
1	25.193	25353.822	1758060.250	49.9994
2	32.210	18557.279	1758100.875	50.0006
Total		43911.102	3516161.125	100.0000



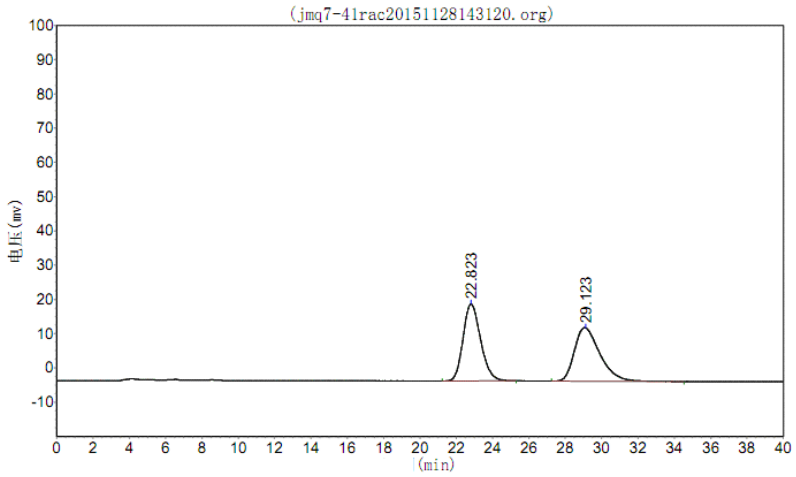
Peak No.	R. Time	Peak Height	Peak Area	Percent
1	25.197	258860.109	19690630.000	93.8311
2	33.133	12838.799	1294557.125	6.1689
Total		271698.908	20985187.125	100.0000


6.521
6.517
6.496
6.482
6.287
6.253
4.477
4.474
4.413
4.410
3.530
3.466
3.259
3.198
3.092
3.065
3.029
3.003
2.893
2.868
1.504
2k
0.83
0.92
0.94
0.98
0.90
8
6
4
2
0
PPM
lc-0376-cr
USER: -- DATE: Nov 30 2011
F1: 300.029
F2: 300.028
SW1: 5208
OF1: 2156.9
PTS1d: 10351 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 12
LB: 0.0
Nuts - Sjmq7-42-300h.fid

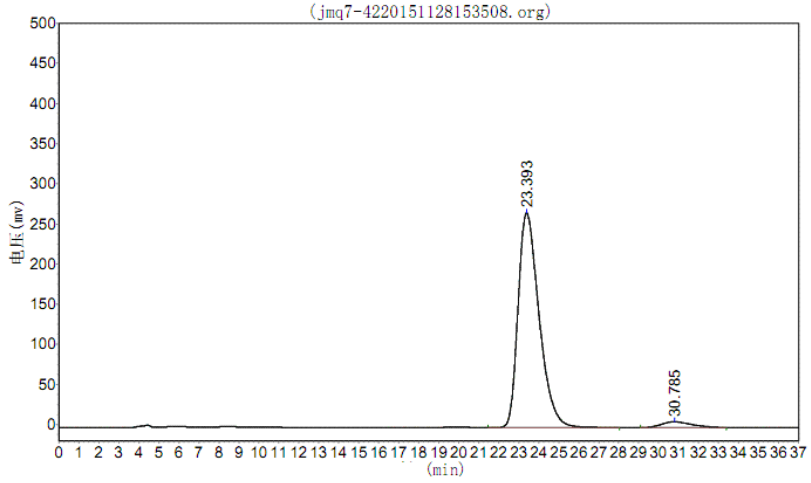

210.053
192.913
171.464
169.773
145.501
132.867
96.331
77.441
77.018
76.595
73.320
49.622
48.844
35.878
34.967
29.614
2k
200
150
100
50
0
PPM
lgh-1-13C-300M
USER: -- DATE: Nov 30 2011
F1: 75.450
F2: 300.028
SW1: 18657
OF1: 7846.0
PTS1d: 29851 , 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
NA: 148
LB: 0.0
Nuts - Sjmq7-42-300c.fid



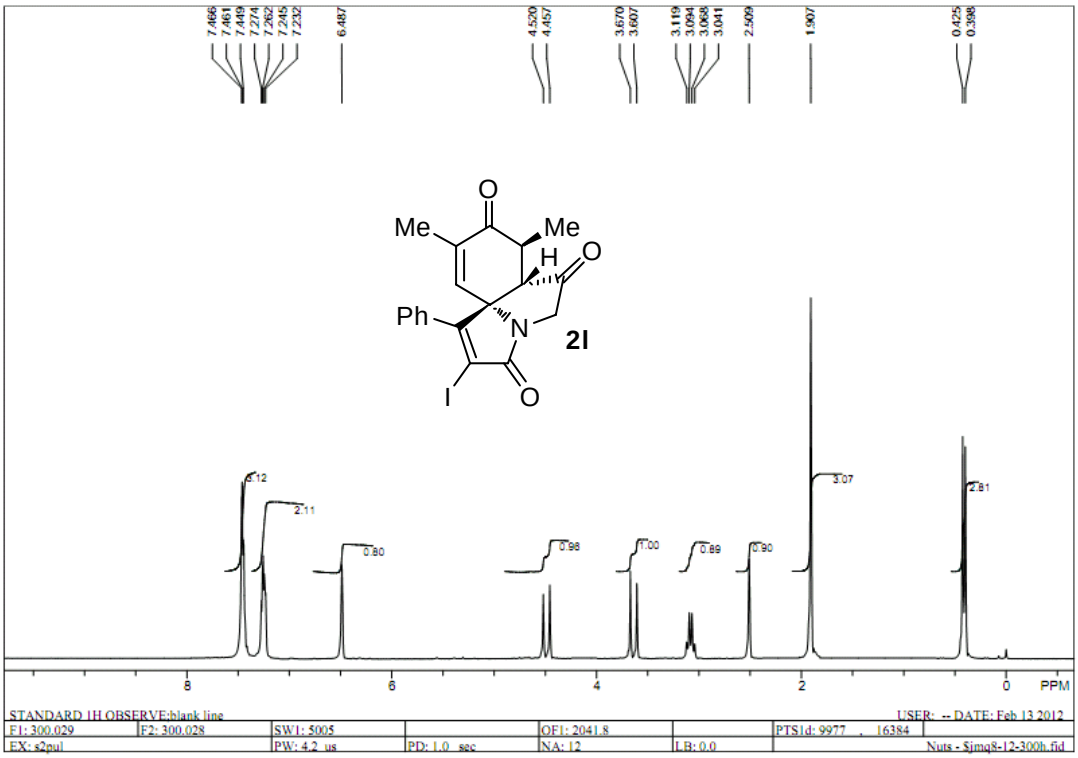
2k



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	22.823	22495.670	1512650.250	49.8480
2	29.123	15657.495	1521876.250	50.1520
Total		38153.165	3034526.500	100.0000



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	23.393	267421.938	19523174.000	96.2971
2	30.785	7280.936	750720.375	3.7029
Total		274702.873	20273894.375	100.0000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7.461
7.449
7.274
7.262
7.245
7.232
6.487
4.520
4.457
3.670
3.607
3.119
3.094
3.068
3.041
2.509
1.907
0.425
0.398
Me
Me
H
O
Ph
N
O
I
O
2l
6.12
2.11
0.80
0.98
1.00
0.89
0.90
3.07
2.81
8
6
4
2
0
PPM
STANDARD 1H OBSERVE:blank line
USER: -- DATE: Feb 13 2012
F1: 300.029
F2: 300.028
SW1: 5005
OF1: 2041.8
PTS1d: 9977 , 16384
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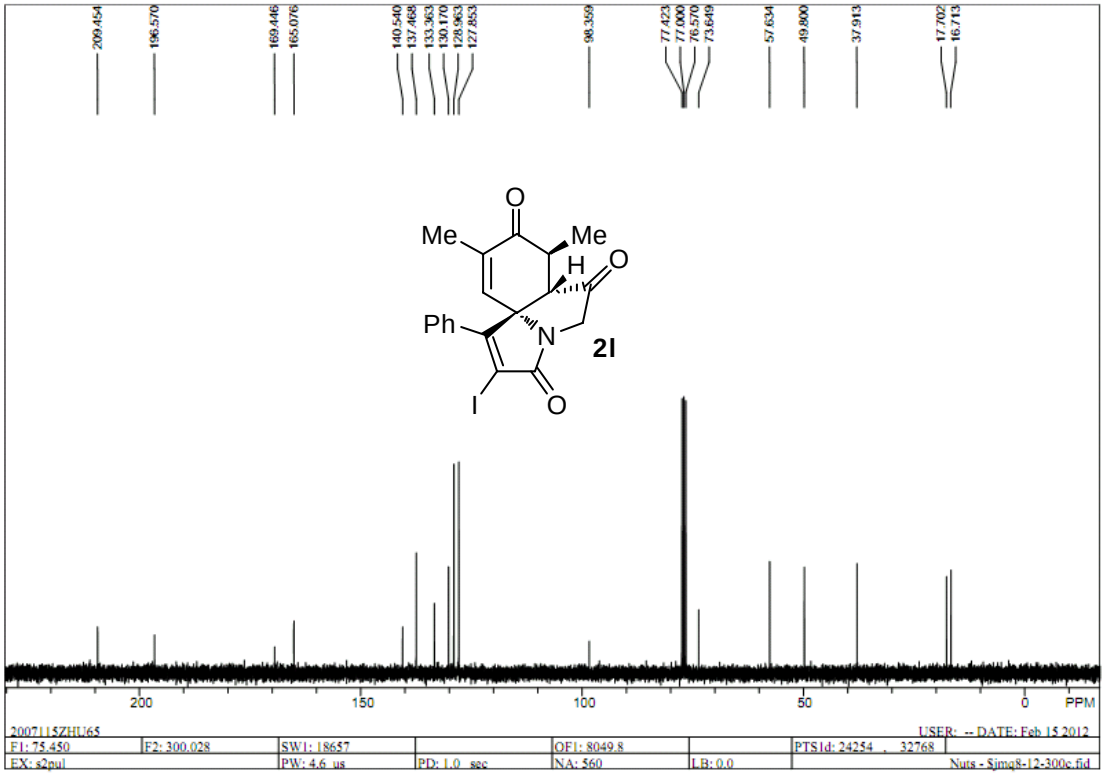
PW: 4.2 us
PD: 1.0 sec
NA: 12
LB: 0.0
Nuts - Sjmq8-12-300h.fid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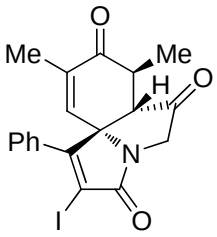
169.446
165.076
140.540
137.468
133.363
130.170
128.963
127.853
98.369
77.423
77.000
76.570
73.649
57.634
49.800
37.913
17.702
16.713
Me
Me
H
O
Ph
N
O
I
O
2l
200
150
100
50
0
PPM
2007115ZHU65
USER: -- DATE: Feb 15 2012
F1: 75.450
F2: 300.028
SW1: 18657
OF1: 8049.8
PTS1d: 24254 , 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
NA: 560
LB: 0.0
Nuts - Sjmq8-12-300c.fid



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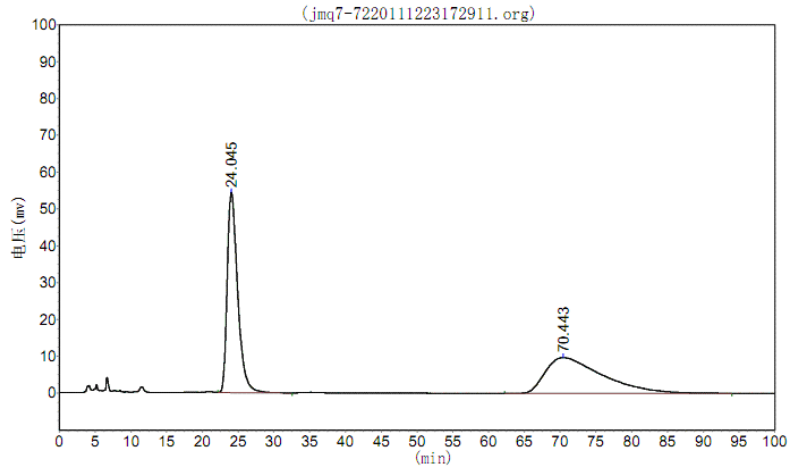
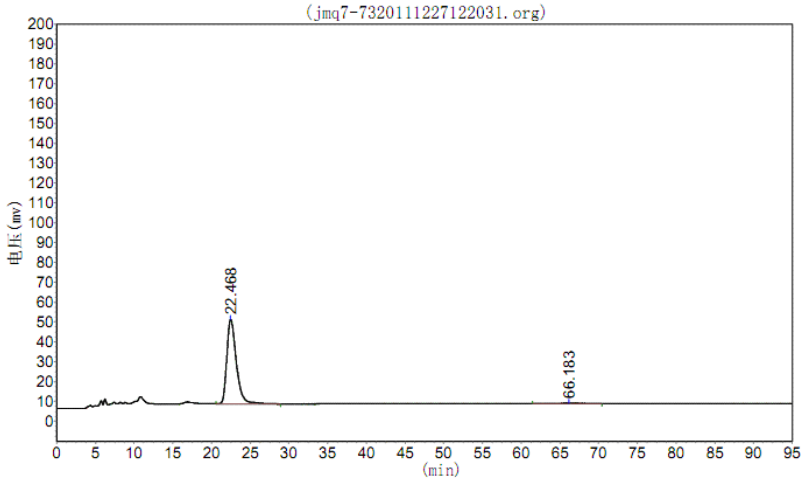
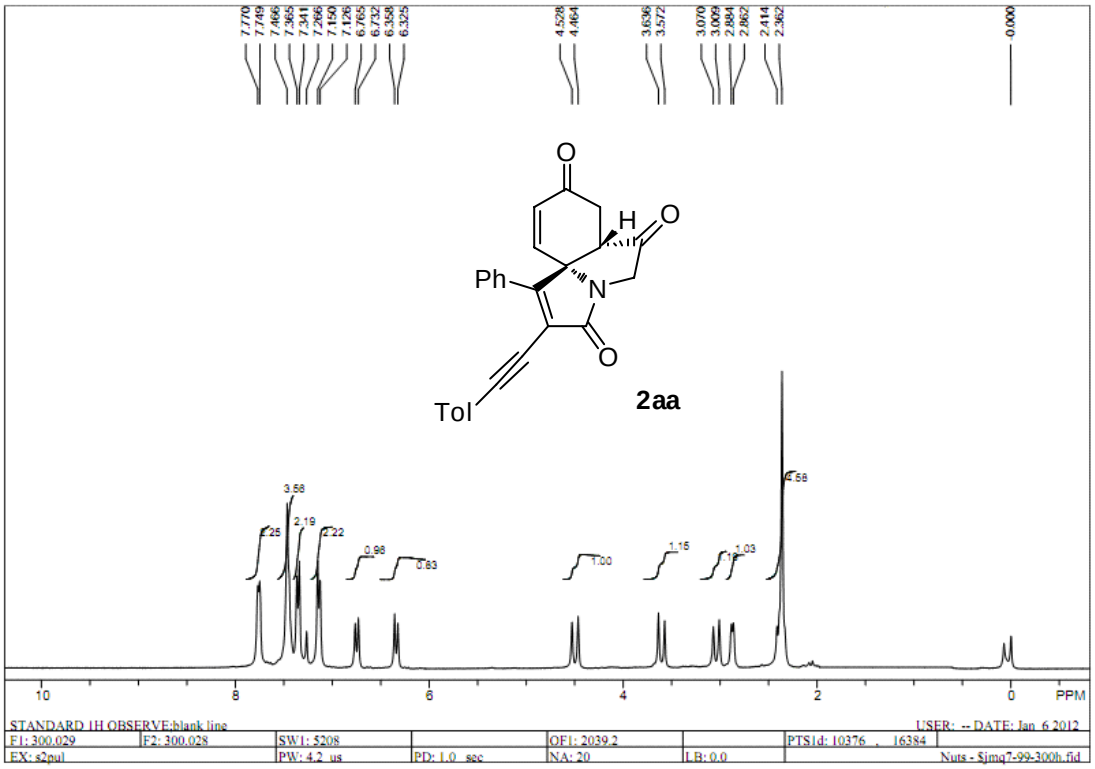


Peak No.	R. Time	Peak Height	Peak Area	Percent
1	24.045	54255.301	5516012.000	50.0742
2	70.443	9796.000	5499657.500	49.9258
Total		64051.301	11015669.500	100.0000



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	22.468	42579.059	3637235.250	99.3891
2	66.183	91.556	22356.002	0.6109
Total		42670.615	3659591.252	100.0000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7.466
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7.266
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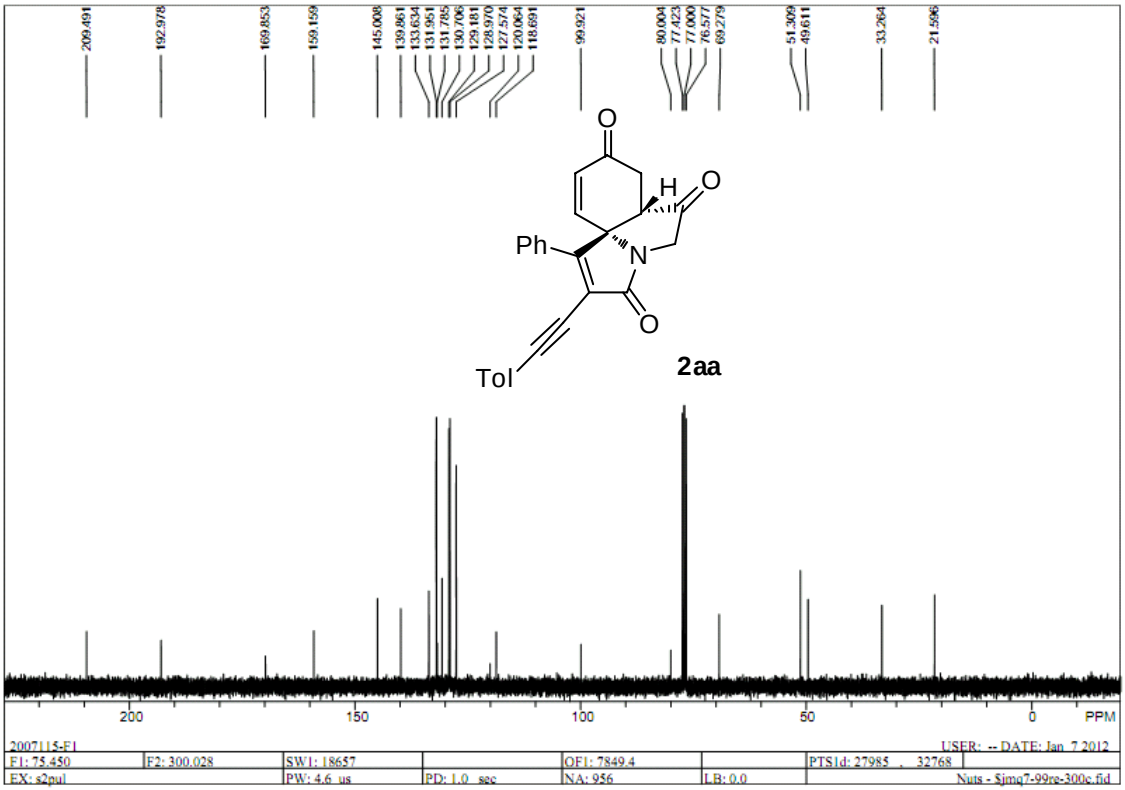
7.126
6.765
6.732
6.358
6.325
4.528
4.464
3.636
3.572
3.070
3.009
2.884
2.862
2.414
2.362
0.000
O
H
O
Ph
N
O
Tol
2aa
3.58
2.19
2.22
0.98
0.83
1.00
1.15
1.03
10
8
6
4
2
0
PPM
STANDARD 1H OBSERVE:blank line
USER: -- DATE: Jan 6 2012
F1: 300.029
F2: 300.028
SW1: 5208
OF1: 2039.2
PTS1d: 10376 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 20
LB: 0.0
Nuts - $jmq7-99-300h.fid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192.978
169.853
159.159
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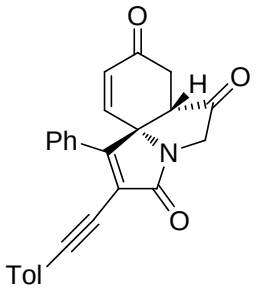
139.861
133.634
131.951
131.785
130.706
129.181
128.970
127.574
120.064
118.691
99.921
80.004
77.423
77.000
76.577
69.279
51.309
49.611
33.264
21.596
O
H
O
Ph
N
O
Tol
2aa
200
150
100
50
0
PPM
2007115-F1
USER: -- DATE: Jan 7 2012
F1: 75.450
F2: 300.028
SW1: 18657
OF1: 7849.4
PTS1d: 27985 , 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
NA: 956
LB: 0.0
Nuts - $jmq7-99re-300c.fid



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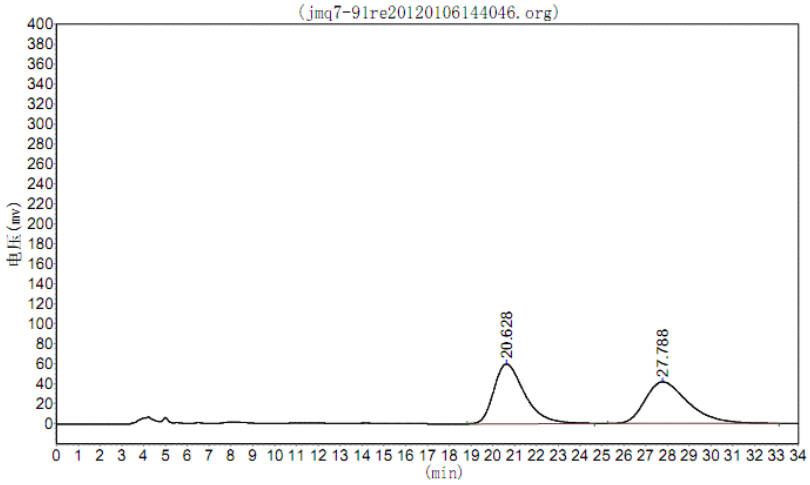
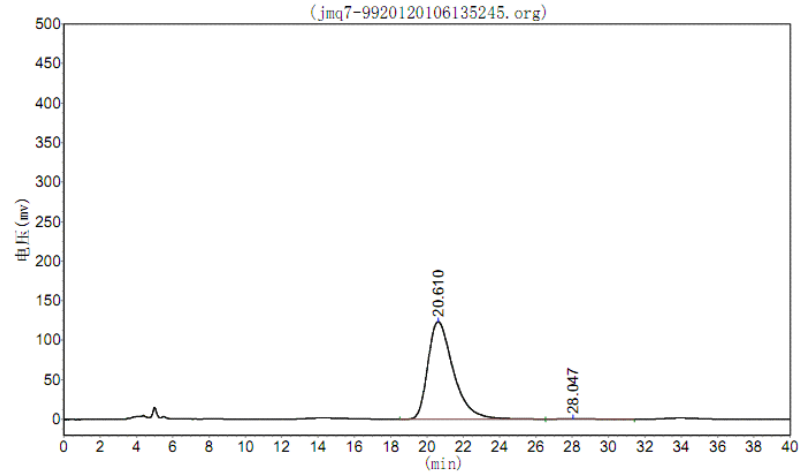
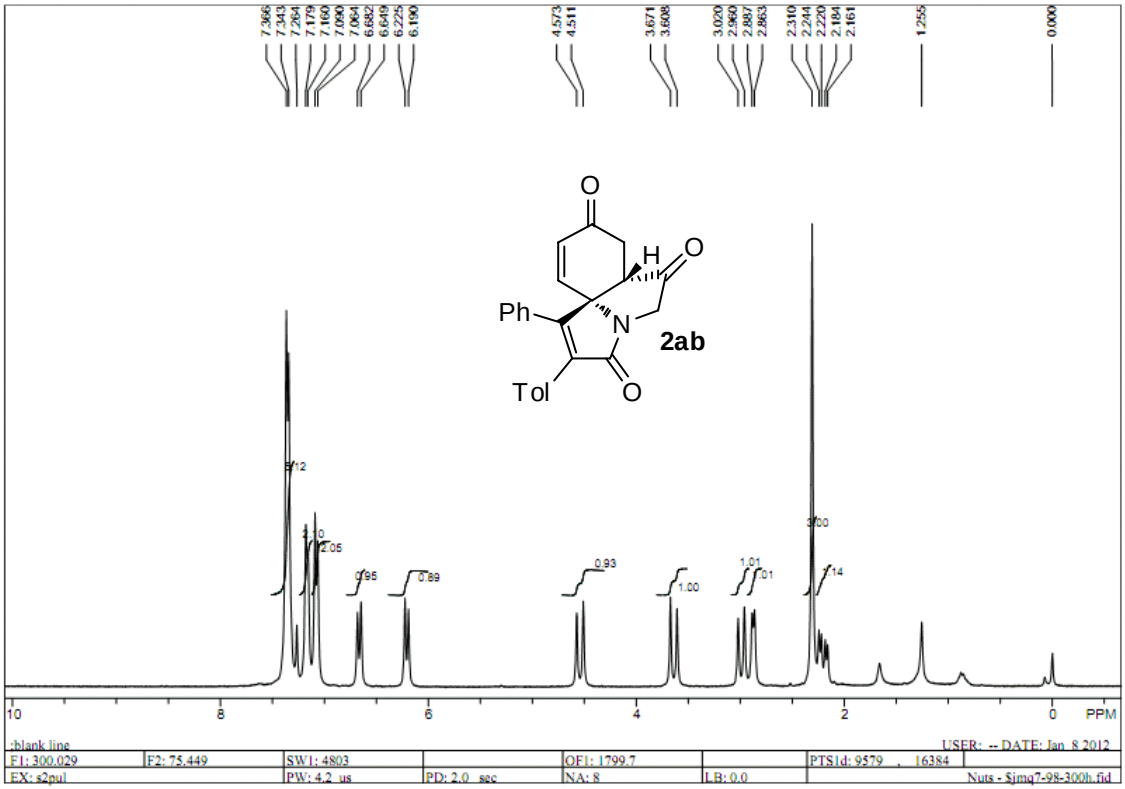


Peak No.	R. Time	Peak Height	Peak Area	Percent
1	20.628	59790.543	5852300.500	50.4636
2	27.788	41714.363	5744769.500	49.5364
Total		101504.906	11597070.000	100.0000



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	20.610	123709.750	12139788.000	99.1197
2	28.047	683.486	107812.367	0.8803
Total		124393.236	12247600.367	100.0000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7.179
7.160
7.090
7.064
6.882
6.649
6.225
6.190
4.573
4.511
3.671
3.608
3.020
2.960
2.887
2.863
2.310
2.244
2.220
2.184
2.161
1.255
0.000
O
H
O
Ph
N
2ab
Tol
O
9.12
2.00
2.05
0.95
0.89
0.93
1.00
1.01
1.01
3.00
3.14
10
8
6
4
2
0
PPM
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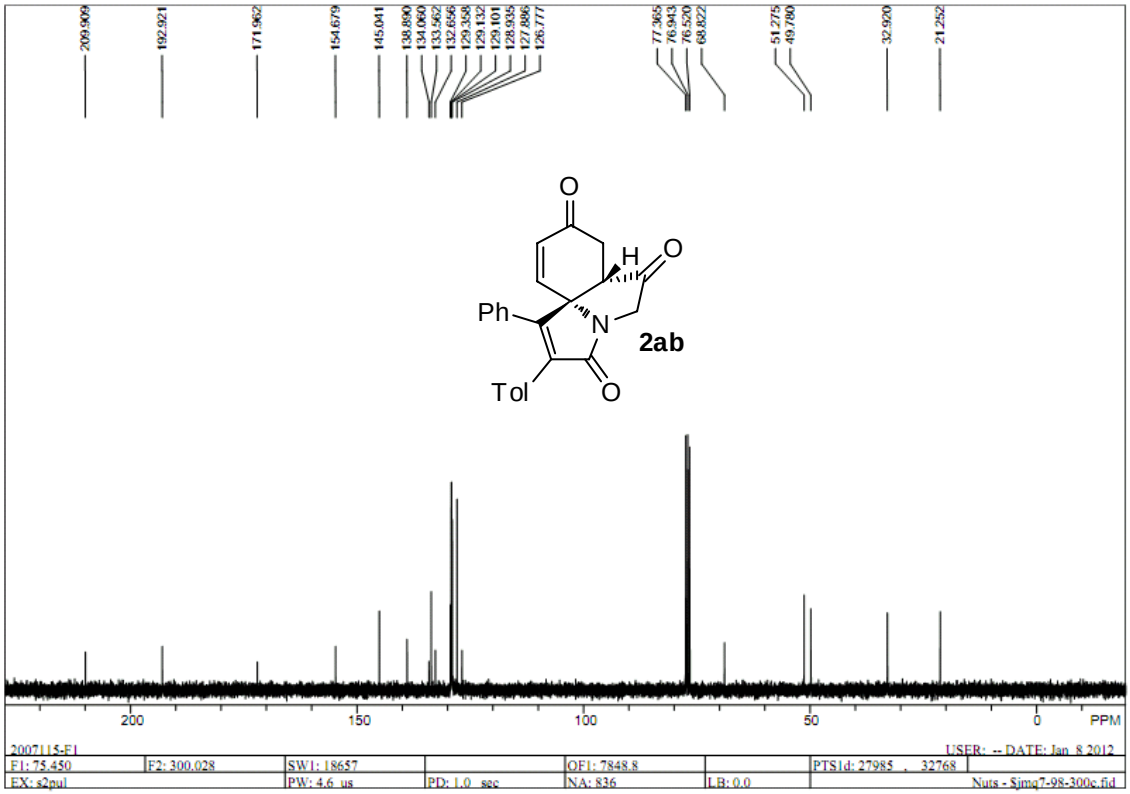
USER: -- DATE: Jan 8 2012
F1: 300.029
F2: 75.449
SW1: 4803
OF1: 1799.7
PTS1d: 9579 , 16384
EX: s2pul
PW: 4.2 us
PD: 2.0 sec
NA: 8
LB: 0.0
Nuts - Sjmq7-98-300h.fid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154.679
145.041
138.890
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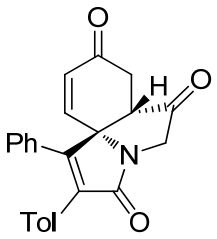
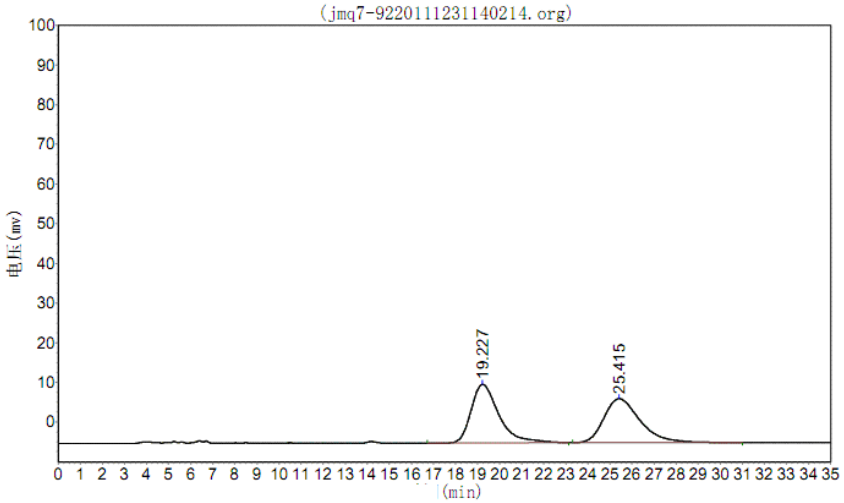
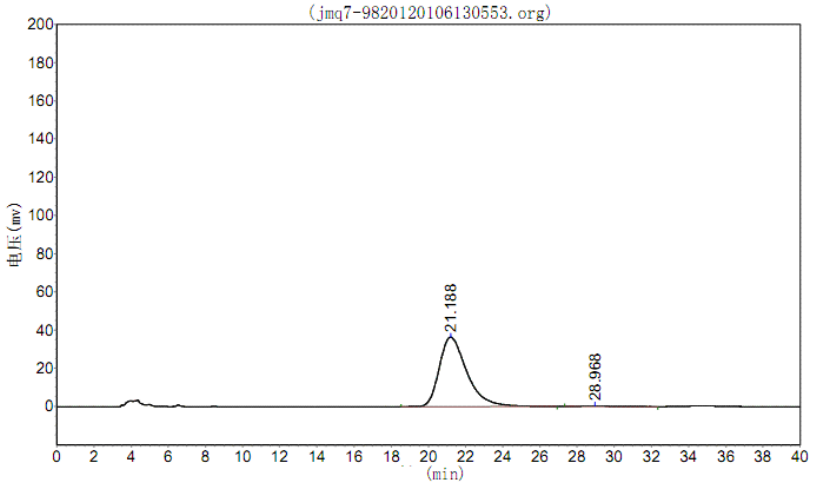
133.562
132.656
129.358
129.132
129.101
128.935
127.886
126.777
77.365
76.943
76.520
68.822
51.275
49.780
32.920
21.252
O
H
O
Ph
N
2ab
Tol
O
200
150
100
50
0
PPM
2007115-F1
USER: -- DATE: Jan 8 2012
F1: 75.450
F2: 300.028
SW1: 18657
OF1: 7848.8
PTS1d: 27985 , 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
NA: 836
LB: 0.0
Nuts - Sjmq7-98-300c.fid



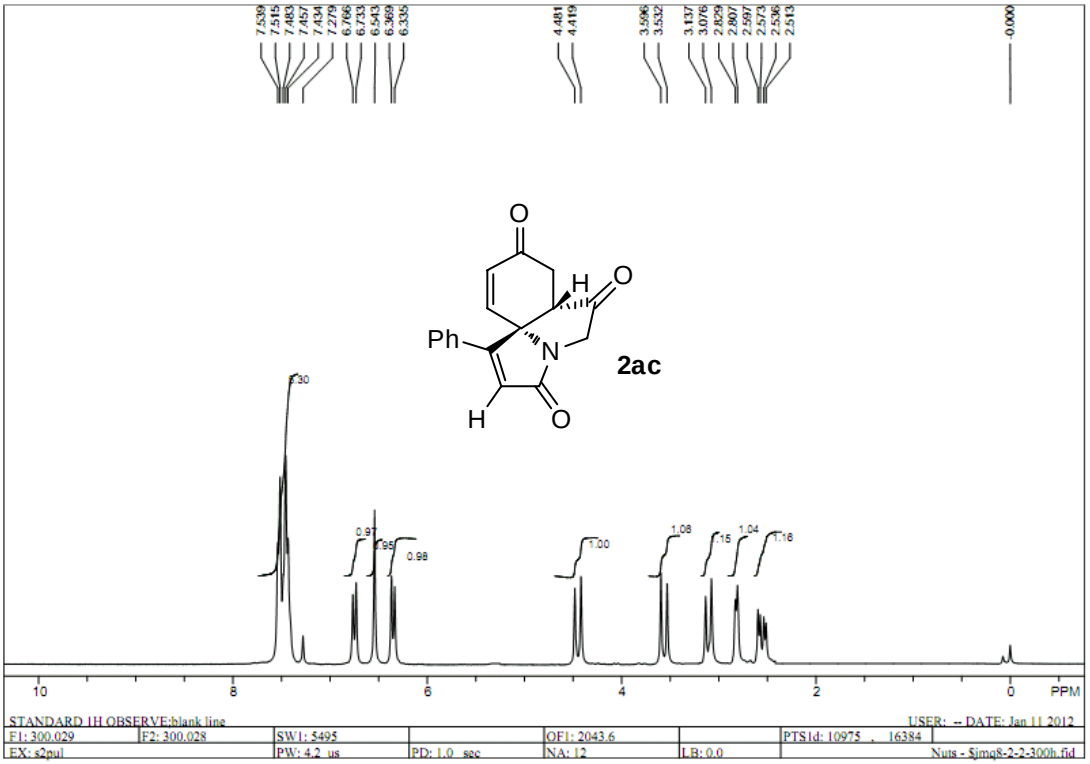
2ab



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	19.227	14708.955	1284387.375	50.5517
2	25.415	11064.061	1256354.750	49.4483
Total		25773.016	2540742.125	100.0000



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	21.188	36379.832	3780922.000	99.0288
2	28.968	307.194	37079.301	0.9712
Total		36687.026	3818001.301	100.0000


7.539
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7.483
7.457
7.434
7.279
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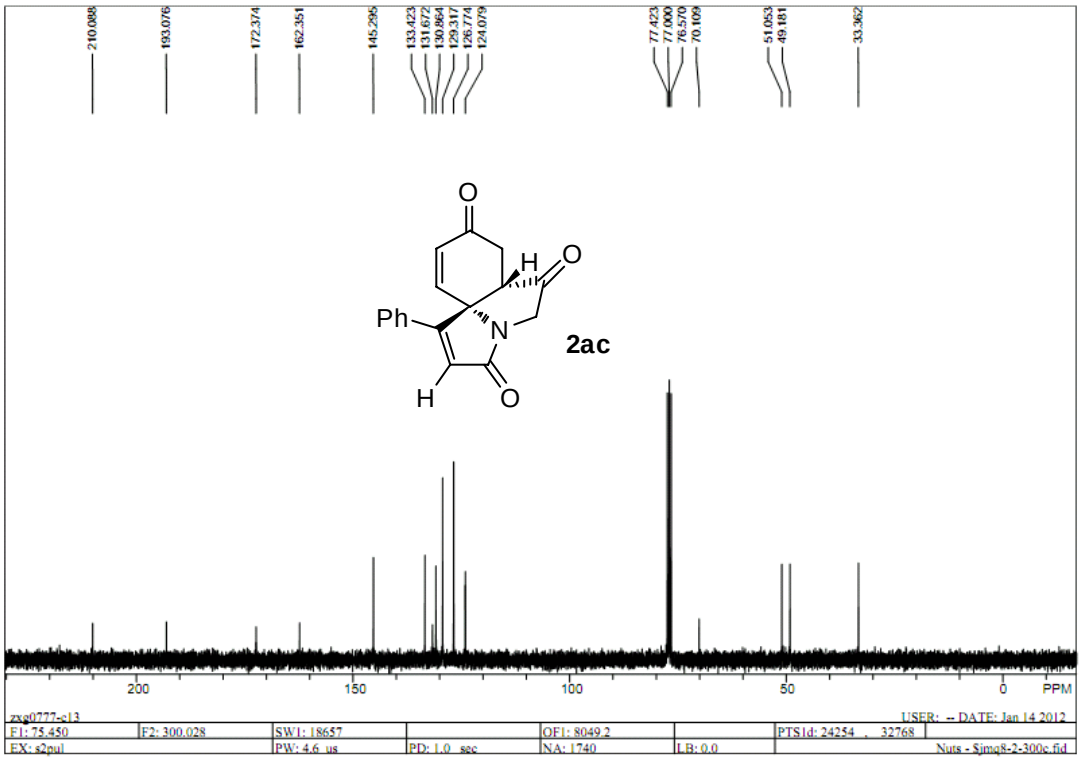
6.733
6.543
6.369
6.335
4.481
4.419
3.596
3.532
3.137
3.076
2.829
2.807
2.597
2.573
2.536
2.513
0.000
O
H
O
Ph
N
2ac
H
O
1.30
0.97
0.95
0.98
1.00
1.08
1.15
1.04
1.16
10
8
6
4
2
0
PPM
STANDARD 1H OBSERVE:blank line
USER: -- DATE: Jan 11 2012
F1: 300.029
F2: 300.028
SW1: 5495
OF1: 2043.6
PTS1d: 10975 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 12
LB: 0.0
Nuts - Sjmq8-2-2-300h.fid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130.864
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126.774
124.079
77.423
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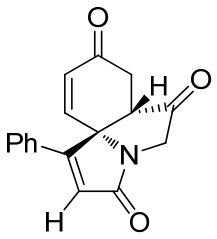
76.570
70.109
51.053
49.181
33.362
O
H
O
Ph
N
2ac
H
O
200
150
100
50
0
PPM
zxg0777-c13
USER: -- DATE: Jan 14 2012
F1: 75.450
F2: 300.028
SW1: 18657
OF1: 8049.2
PTS1d: 24254 , 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
NA: 1740
LB: 0.0
Nuts - Sjmq8-2-300c.fid



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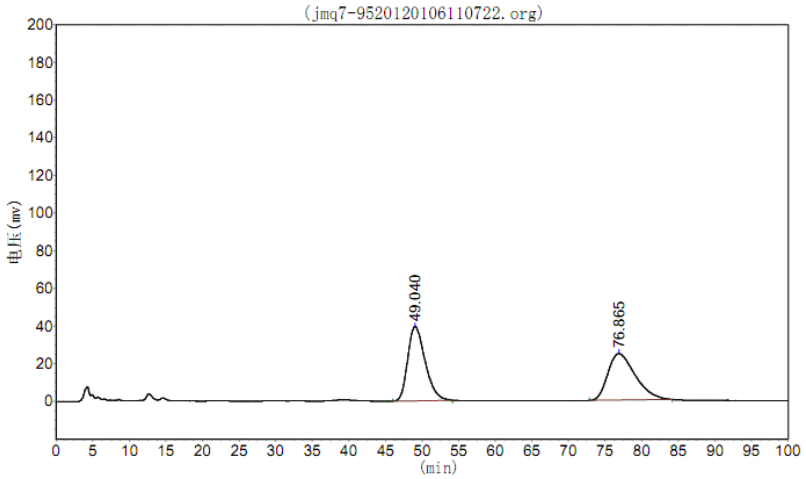
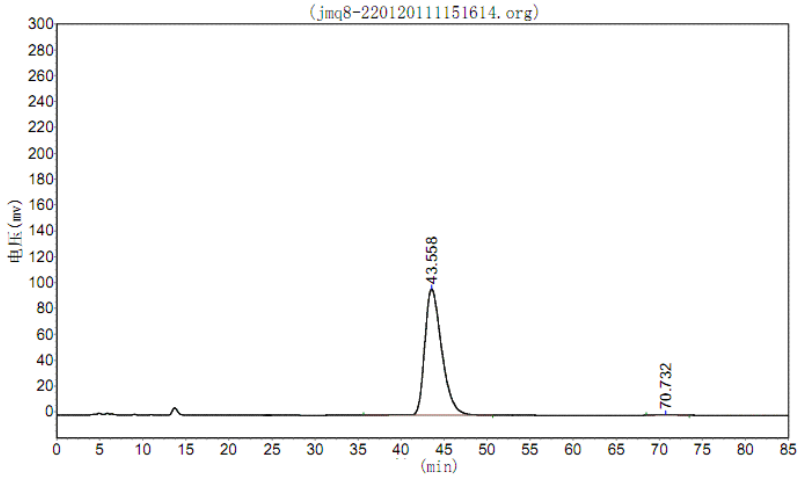


Peak No.	R. Time	Peak Height	Peak Area	Percent
1	49.040	39408.277	6520712.000	50.3801
2	76.865	24721.996	6422332.000	49.6199
Total		64130.273	12943044.000	100.0000



Peak No.	R. Time	Peak Height	Peak Area	Percent
1	43.558	97660.727	13425020.000	99.2866
2	70.732	578.410	96467.695	0.7134
Total		98239.137	13521487.695	100.0000