Supporting Information

for

One-pot synthesis of tetracyclic fused imidazo[1,2-a]pyridines via a three-component reaction

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Experimental procedures, characterization and spectral data for synthesized compounds and X-ray data for compound **1o**

General Information

Unless noted otherwise, all reactions were performed under a nitrogen atmosphere, and materials obtained from commercial suppliers were used without further purification. ^{1}H NMR spectra were recorded on a 300 MHz, 400 MHz or 500 MHz spectrometer using residual solvent signal (DMSO δ = 2.50 ppm) as internal standard. All coupling constants are reported in hertz (Hz). ^{13}C NMR spectra were recorded on the same instruments, and chemical shifts were measured relative to solvent resonances (DMSO δ = 39.9 ppm). High-resolution mass spectra were obtained on a quadrupole time-of-flight (QqTOF) mass spectrometer utilizing electrospray ionization (ESI) method.

Typical procedure for the multicomponent reaction

To a solution of isatin **3** (1.0 mmol), 2-aminopyridine **2** (1.35 mmol) and isocyanide **4** (1.35 mmol) in 4 mL of *n*-butyl alcohol was added $HClO_4$ (1.0 mmol), and the reaction mixture was stirred under reflux for 8 h. After cooling to room temperature, the precipitate was collected by filtration, rinsed with ethanol and dried to afford the target compound **1**.

*13-(tert-Butylamino)-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1a**):* 171 mg (42%) as a slight yellow solid; mp 252.3−253.7°C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.25 (*s*, 9H), 5.63 (*s*, 1H), 7.35 (*d*, *J* = 8.4 Hz, 1H), 7.41 (*t*, *J* = 7.2 Hz, 1H), 7.61-7.65 (*m*, 1H), 7.88-7.92 (*m*, 1H), 8.27-8.32 (*m*, 1H), 8.70 (*d*, *J* = 7.2 Hz, 1H), 8.95 (*d*, *J* = 8.8 Hz, 1H), 9.14 (*d*, *J* = 6.8 Hz, 1H), 12.29 (*s*, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 30.4, 58.1, 110.7, 115.2, 117.5, 119.8, 123.1, 123.3, 125.3, 125.6, 127.3, 131.9, 134.7, 135.8, 137.2, 145.0; HRMS (*m/z*, ESI) calc. for $C_{18}H_{19}N_4O$ (+) 307.1553, found 307.1552.

*13-(tert-Butylamino)-10-chloro-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1b**):* 234 mg (53%) as a slight yellow solid; mp 248.8−250.6 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.25 (*s*, 9H), 5.69 (*s*, 1H), 7.40-7.43 (*m*, 2H), 7.62-7.66 (*m*, 1H), 8.35 (*dd*, *J* = 2.0, 9.6 Hz, 1H), 8.69 (*dd*, *J* = 1.2, 8.4 Hz, 1H), 8.95 (*d*, *J* = 9.6 Hz, 1H), 9.40 (*d*, *J* = 1.2 Hz, 1H), 12.53 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 30.2, 58.8, 110.5, 116.1, 116.8, 123.9, 124.2, 125.7, 125.8, 126.8, 132.4, 133.8, 135.9, 136.3, 144.4; HRMS (*m/z*, ESI) calc. for $C_{18}H_{18}N_4OCl$ (+) 341.1164, found 341.1152.

*13-(tert-Butylamino)-10-methyl-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1c**):* 160 mg (38%) as a brown solid; mp 258.9−260.4 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.25 (*s*, 9H), 2.59 (*s*, 3H), 5.54 (*s*, 1H), 7.34-7.41 (*m*, 2H), 7.59-7.63 (*m*, 1H), 8.15 (*d*, *J* = 9.2 Hz, 1H), 8.68 (*d*, *J* = 8.0 Hz, 1H), 8.84 (*d*, *J* = 9.2 Hz, 1H), 8.95 (*s*, 1H), 12.26 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 18.2, 30.4, 58.3, 110.7, 114.5, 116.4, 123.5, 123.8, 125.0, 125.1, 125.8, 130.5, 132.2, 133.8, 135.5, 138.7, 144.7; HRMS (*m/z*, ESI) calc. for $C_{19}H_{21}N_4O$ (+) 321.1710, found 321.1700.

*10-Bromo-13-(tert-butylamino)-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1d**):* 267 mg (55%) as a slight yellow solid; mp 267.8−269.1 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ 1.24 (*s*, 9H), 5.56 (*s*, 1H), 7.36-7.45 (*m*, 2H), 7.62-7.67 (*m*, 1H), 8.43 (*dd*, *J* = 1.8, 9.6 Hz, 1H), 8.69 (*d*, *J* = 7.2 Hz, 1H), 8.87 (*d*, *J* = 9.6 Hz, 1H), 9.38 (*s*,

1H), 12.45 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 30.2, 58.7, 110.4, 114.2, 116.2, 116.6, 123.9, 124.1, 125.5, 125.9, 127.3, 132.6, 134.1, 135.4, 138.8, 144.4; HRMS (*m/z*, ESI) calc. for $C_{18}H_{18}N_4OBr$ (+) 385.0659, found 385.0653.

13-(*tert*-Butylamino)-2-methoxy-10-methyl-6-oxo-5,6-dihydropyrido[2',1':2,3]imi-dazo[1,5-*c*]quin-azolin-12-ium perchlorate (**1e**): 225 mg (50%) as a grey solid; mp 270.8−272.7 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ 1.27 (*s*, 9H), 2.59 (*s*, 3H), 3.87 (*s*, 3H), 5.64 (*s*, 1H), 7.26-7.28 (*m*, 2H), 8.13-8.17 (*m*, 2H), 8.84 (*d*, *J* = 9.3 Hz, 1H), 8.95 (*s*, 1H), 12.17 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 18.2, 30.5, 56.2, 58.1, 108.8, 111.4, 114.5, 117.8, 120.0, 123.5, 124.9, 125.1, 129.2, 130.5, 133.9, 138.6, 144.4, 155.4; HRMS (*m/z*, ESI) calc. for $C_{20}H_{23}N_4O_2$ (+) 351.1816, found 351.1801.

*13-(tert-Butylamino)-10-chloro-2-methoxy-6-oxo-5,6-dihydropyrido[2',1':2,3]imi-dazo[1,5-c]quinazolin-12-ium perchlorate (**1f**):* 184 mg (39%) as a yellow solid; mp 238.4−240.5 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.27 (*s*, 9H), 3.88 (*s*, 3H), 5.66 (*s*, 1H), 7.28-7.34 (*m*, 2H), 8.18 (*d*, *J* = 2.8 Hz, 1H), 8.34 (*dd*, *J* = 2.0, 9.6 Hz, 1H), 8.96 (*d*, *J* = 9.6 Hz, 1H), 9.33 (*d*, *J* = 1.6 Hz, 1H) 12.20 (*br*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 30.3, 56.2, 58.5, 108.6, 111.0, 116.2, 118.1, 120.5, 124.1, 125.2, 125.7, 127.0, 129.5, 133.9, 136.4, 144.2, 155.5; HRMS (*m/z*, ESI) calc. for $C_{19}H_{20}N_4O_2Cl$ (+) 371.1269, found 371.1277.

*10-Bromo-13-(tert-butylamino)-2-methoxy-6-oxo-5,6-dihydropyrido[2',1':2,3]imi-dazo[1,5-c]quinazolin -12-ium perchlorate (**1g**):* 247 mg (48%) as a brown solid; mp 249.5−251.3 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ 1.26 (*s*, 9H), 3.88 (*s*, 3H), 5.65 (*s*, 1H), 7.28-7.32 (*m*, 2H), 8.17 (*d*, *J* = 2.1 Hz, 1H), 8.42 (*dd*, *J* = 1.5, 9.6 Hz, 1H), 8.87 (*d*, *J* = 9.6 Hz, 1H), 9.37 (*s*, 1H) 12.35 (*s*, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 30.4, 56.3, 58.5, 108.7, 111.1, 114.2, 116.3, 118.1, 120.5, 123.9, 125.5, 127.2, 129.4, 134.2, 138.8, 144.2, 155.6; HRMS (*m/z*, ESI) calc. for $C_{19}H_{20}N_4O_2Br$ (+) 415.0764, found 415.0764.

*13-(tert-Butylamino)-2-chloro-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1h**):* 163 mg (37%) as a slight yellow solid; mp 285.7−287.4 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ 1.25 (*s*, 9H), 5.77 (*s*, 1H), 7.35 (*d*, *J* = 9.0 Hz, 1H), 7.67 (*dd*, *J* = 2.4, 8.7 Hz, 1H), 7.89 (*t*, *J* = 6.9 Hz, 1H), 8.32 (*t*, *J* = 7.8 Hz, 1H), 8.71 (*d*, *J* = 2.1 Hz, 1H), 8.95 (*d*, *J* = 9.0 Hz, 1H), 9.17 (*d*, *J* = 6.6 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 30.1, 58.1, 111.9, 114.8, 118.3, 120.0, 123.6, 124.0, 124.5, 127.2, 131.7, 132.3, 134.4, 134.8, 136.5, 144.4; HRMS (*m/z*, ESI) calc. for $C_{18}H_{18}N_4OCl$ (+) 341.1164, found 341.1156.

*10-Bromo-13-(tert-butylamino)-2-chloro-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1i**):* 213 mg (41%) as a grey solid; mp 265.6−267.3 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.25 (*s*, 9H), 5.68 (*s*, 1H), 7.38 (*d*, *J* = 8.4 Hz, 1H), 7.71 (*d*, *J* = 8.4 Hz, 1H), 8.47 (*d*, *J* = 9.6 Hz, 1H), 8.70 (*s*, 1H), 8.87 (*d*, *J* = 9.2 Hz, 1H), 9.40 (*s*, 1H), 12.57 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 30.2, 58.9, 111.9, 114.6, 116.2, 118.5, 124.4, 124.5, 124.8, 127.5, 127.9, 132.4, 134.2, 134.5, 139.5, 144.2; HRMS (*m/z*, ESI) calc. for $C_{18}H_{17}N_4OClBr$ (+) 419.0269, found 419.0251.

*2-Bromo-13-(tert-butylamino)-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1j**):* 189 mg (39%) as a slight yellow solid; mp 284.8−286.5 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.25 (*s*, 9H), 5.76 (*s*, 1H), 7.31 (*s*, 1H), 7.81-7.92 (*m*, 2H), 8.33 (*s*, 1H), 8.86-8.96 (*m*, 2H), 9.18 (*s*, 1H), 12.44 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 30.4, 58.4, 115.2, 115.5, 118.5, 120.4, 121.6, 123.8, 124.4, 127.7, 134.7, 135.2, 137.2, 137.7, 144.6; HRMS (*m/z*, ESI) calc. for $C_{18}H_{18}N_4OBr$ (+) 385.0659, found 385.0658.

*2,10-Dibromo-13-(tert-butylamino)-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1k**):* 197 mg (35%) as a slight yellow solid; mp 259.7−261.6 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.25 (*s*, 9H), 5.68 (*s*, 1H), 7.31 (*d*, *J* = 8.8 Hz, 1H), 7.82 (*d*, *J* = 7.2 Hz, 1H), 8.47 (*d*, *J* = 9.2 Hz, 1H), 8.84-8.89 (*m*, 2H), 9.39 (*s*, 1H), 12.56 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 30.2, 58.9, 112.3, 114.5, 115.6, 116.2, 118.7, 124.2, 124.5, 127.4, 127.8, 134.2, 134.7, 135.1, 139.5, 144.2; HRMS (*m/z*, ESI) calc. for $C_{18}H_{17}N_4OBr_2$ (+)

462.9764, found 462.9748.

*13-(tert-Butylamino)-10-chloro-2,4-dimethyl-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1l**):* 188 mg (40%) as a brown solid; mp 242.3−244.2 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ 1.24 (*s*, 9H), 2.38 (*s*, 3H), 2.43 (*s*, 3H), 5.54 (*s*, 1H), 7.34 (*s*, 1H), 8.33 (*d*, *J* = 9.9 Hz, 1H), 8.43 (*s*, 1H), 8.95 (*d*, *J* = 9.3 Hz, 1H), 9.32 (*s*, 1H) 11.50 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 18.0, 20.8, 30.3, 58.7, 110.5, 116.3, 123.4, 123.8, 125.2, 125.4, 125.8, 127.1, 131.8, 132.9, 133.7, 134.9, 136.3, 144.7; HRMS (*m/z*, ESI) calc. for $C_{20}H_{22}N_4OCl$ (+) 369.1477, found 369.1477.

*13-(tert-Butylamino)-10-chloro-4-fluoro-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1m**):* 174 mg (38%) as a yellow solid; mp 236.5−238.4 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ 1.24 (*s*, 9H), 5.60 (*s*, 1H), 7.43-7.45 (*m*, 1H), 7.56-7.62 (*m*, 1H), 8.39 (*dd*, *J* = 1.8, 9.3 Hz, 1H), 8.50 (*d*, *J* = 7.8 Hz, 1H), 8.96 (*d*, *J* = 9.6 Hz, 1H), 9.36 (*d*, *J* = 1.5 Hz, 1H), 12.57 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 30.2, 58.8, 112.6, 116.2, 118.1, 118.2, 121.6, 124.3, 124.5, 124.6, 125.1, 125.5, 127.3, 134.1, 137.1, 144.3; HRMS (*m/z*, ESI) calc. for $C_{18}H_{17}N_4OClF$ (+) 359.1069, found 359.1069.

*13-(Cyclohexylamino)-10-methyl-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1n**):* 232 mg (52%) as a grey solid; mp 244.9−246.6 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.13-1.22 (*m*, 3H), 1.38-1.43 (*m*, 2H), 1.56-1.59 (*m*, 1H), 1.65-1.72 (*m*, 2H), 1.92-1.94 (*m*, 2H), 2.60 (*s*, 3H), 3.04-3.07 (*m*, 1H), 5.70 (*d*, *J* = 6.0 Hz, 1H), 7.35 (*d*, *J* = 8.0 Hz, 1H), 7.44 (*t*, *J* = 7.6 Hz, 1H), 7.62 (*t*, *J* = 8.0 Hz, 1H), 8.14 (*d*, *J* = 9.2 Hz, 1H), 8.44 (*d*, *J* = 8.0 Hz, 1H), 8.82 (*d*, *J* = 9.2 Hz, 1H), 8.88 (*s*, 1H), 12.25 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 18.3, 25.1, 25.6, 33.6, 57.1, 110.8, 114.5, 116.5, 122.7, 124.5, 124.7, 124.9, 131.0, 131.9, 133.3, 135.2, 138.5, 144.6; HRMS (*m/z*, ESI) calc. for $C_{21}H_{23}N_4O$ (+) 347.1866, found 347.1852.

*10-Chloro-13-(cyclohexylamino)-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1o**):* 224 mg (48%) as a brown solid; mp 258.7−260.4 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.13-1.17 (*m*, 3H), 1.36-1.41 (*m*, 2H), 1.56-1.59 (*m*, 1H), 1.65-1.72 (*m*, 2H), 1.92-1.96 (*m*, 2H), 3.06 (*m*, 1H), 5.74 (*d*, *J* = 4.4 Hz, 1H), 7.38 (*d*, *J* = 8.0 Hz, 1H), 7.48 (*t*, *J* = 7.6 Hz, 1H), 7.64 (*d*, *J* = 7.6 Hz, 1H), 8.35 (*dd*, *J* = 1.6, 9.6 Hz, 1H), 8.43 (*d*, *J* = 8.0 Hz, 1H), 8.92 (*d*, *J* = 9.6 Hz, 1H), 9.32 (*s*, 1H), 12.44 (*s*, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 25.2, 25.7, 33.6, 57.4, 110.5, 116.1, 116.7, 123.4, 124.8, 124.9, 125.1, 125.5, 127.5, 132.4, 133.3, 135.3, 136.4, 144.4; HRMS (*m/z*, ESI) calc. for $C_{20}H_{20}N_4OCl$ (+) 367.1320, found 367.1313.

*10-Bromo-13-(cyclohexylamino)-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1p**):* 230 mg (45%) as a brown solid; mp 286.6−288.7 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ 1.12-1.21 (*m*, 3H), 1.38-1.44 (*m*, 2H), 1.56-1.57 (*m*, 1H), 1.65-1.73 (*m*, 2H), 1.92-1.95 (*m*, 2H), 3.03-3.04 (*m*, 1H), 5.73 (*d*, *J* = 6.9 Hz, 1H), 7.38 (*d*, *J* = 8.1 Hz, 1H), 7.45-7.50 (*m*, 1H), 7.63-7.68 (*m*, 1H), 8.41-8.45 (*m*, 2H), 8.84 (*d*, *J* = 9.6 Hz, 1H), 9.36 (*s*, 1H), 12.43 (*s*, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 25.2, 25.7, 33.6, 57.5, 110.5, 114.7, 116.2, 116.7, 123.1, 124.8, 125.1, 125.4, 126.7, 132.4, 133.6, 135.3, 138.7, 144.4; HRMS (*m/z*, ESI) calc. for $C_{20}H_{20}N_4OBr$ (+) 411.0815, found 411.0800.

*10-Chloro-13-(cyclohexylamino)-2-methoxy-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1q**):* 234 mg (47%) as a brown solid; mp 269.5−271.2 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ 1.13-1.22 (*m*, 3H), 1.38-1.43 (*m*, 2H), 1.56-1.59 (*m*, 1H), 1.65-1.72 (*m*, 2H), 1.92-1.95 (*m*, 2H), 3.07-3.10 (*m*, 1H), 3.88 (*s*, 3H), 5.81 (*d*, *J* = 6.6 Hz, 1H), 7.28-7.36 (*m*, 2H), 7.90 (*d*, *J* = 2.1 Hz, 1H), 8.33 (*dd*, *J* = 1.5, 9.3 Hz, 1H), 8.92 (*d*, *J* = 9.6Hz, 1H), 9.33 (*s*, 1H), 12.34 (*br*, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 25.1, 25.6, 33.7, 56.1, 57.5, 107.6, 111.0, 116.1, 118.1, 120.3, 123.0, 124.7, 125.5, 127.3, 129.0, 133.2, 136.1, 144.1, 156.0; HRMS (*m/z*, ESI) calc. for $C_{21}H_{22}N_4O_2Cl$ (+) 397.1426, found 397.1419.

*10-Bromo-13-(cyclohexylamino)-2-methoxy-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1r**):* 206 mg (38%) as a green solid; mp 275.2−277.3 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 1.15-1.22 (*m*, 3H), 1.40-1.44 (*m*, 2H), 1.57-1.60 (*m*, 1H), 1.69-1.72 (*m*, 2H), 1.92-1.96 (*m*, 2H), 3.08-3.10 (*m*, 1H), 3.88 (*s*, 3H), 5.78 (*d*, *J* = 6.2 Hz, 1H), 7.28-7.35 (*m*, 2H), 7.90 (*s*, 1H), 8.41 (*d*, *J* = 9.5 Hz, 1H), 8.85 (*d*, *J* = 9.5 Hz, 1H), 9.36 (*s*, 1H), 12.31 (*s*, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 25.1, 25.7, 33.8, 56.2, 57.7, 107.8, 111.1, 114.6, 116.2, 118.2, 120.3, 123.0, 125.4, 126.6, 129.1, 133.6, 138.5, 144.1, 156.2; HRMS (*m/z*, ESI) calc. for $C_{21}H_{22}N_4O_2Br$ (+) 441.0921, found 441.0900.

*2,10-Dichloro-13-(cyclohexylamino)-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1s**):* 256 mg (51%) as a slight purple solid; mp 249.1−250.8 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 1.16-1.22 (*m*, 3H), 1.38-1.42 (*m*, 2H), 1.56-1.59 (*m*, 1H), 1.65-1.72 (*m*, 2H), 1.92-1.94 (*m*, 2H), 3.03-3.06 (*m*, 1H), 5.88 (*d*, *J* = 6.8 Hz, 1H), 7.39 (*d*, *J* = 8.8 Hz, 1H), 7.71-7.73 (*m*, 1H), 8.38-8.44 (*m*, 2H), 8.92 (*d*, *J* = 9.6 Hz, 1H), 9.31 (*s*, 1H), 12.55 (*s*, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 25.1, 25.7, 33.8, 57.6, 112.0, 116.1, 118.7, 122.2, 124.2, 125.1, 126.1, 127.7, 128.6, 132.2, 133.5, 134.3, 137.0, 144.2; HRMS (*m/z*, ESI) calc. for $C_{20}H_{19}N_4OCl_2$ (+) 401.0930, found 401.0917.

*10-Bromo-2-chloro-13-(cyclohexylamino)-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1t**):* 218 mg (40%) as a slight purple solid; mp 278.8−280.8 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.12-1.22 (*m*, 3H), 1.35-1.43 (*m*, 2H), 1.56-1.59 (*m*, 1H), 1.65-1.72 (*m*, 2H), 1.92-1.99 (*m*, 2H), 3.02-3.06 (*m*, 1H), 5.88 (*d*, *J* = 6.8 Hz, 1H), 7.38 (*d*, *J* = 8.8 Hz, 1H), 7.71 (*dd*, *J* = 2.4, 8.8 Hz, 1H), 8.42-8.48 (*m*, 2H), 8.84 (*d*, *J* = 9.6 Hz, 1H), 9.36 (*d*, *J* = 1.2 Hz, 1H), 12.56 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 25.1, 25.6, 33.7, 57.6, 112.0, 114.8, 116.1, 118.6, 121.7, 124.0, 125.9, 126.9, 128.5, 132.0, 133.6, 134.2, 139.2, 144.2; HRMS (*m/z*, ESI) calc. for $C_{20}H_{19}N_4OClBr$ (+) 445.0425, found 445.0419.

*2-Bromo-10-chloro-13-(cyclohexylamino)-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1u**):* 186 mg (34%) as a slight yellow solid; mp 286.8−288.7 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ 1.12-1.21 (*m*, 3H), 1.38-1.42 (*m*, 2H), 1.57-1.58 (*m*, 1H), 1.69-1.73 (*m*, 2H), 1.91-1.95 (*m*, 2H), 3.04 (*m*, 1H), 5.90 (*d*, *J* = 6.9 Hz, 1H), 7.31 (*d*, *J* = 8.7 Hz, 1H), 7.82 (*dd*, *J* = 2.1, 8.7 Hz, 1H), 8.37 (*dd*, *J* = 1.8, 9.6 Hz, 1H), 8.56 (*d*, *J* = 1.8 Hz, 1H), 8.91 (*d*, *J* = 9.6 Hz, 1H), 9.32 (*d*, *J* = 1.2 Hz, 1H), 12.57 (*s*, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 25.1, 25.6, 33.7, 57.5, 112.4, 116.0, 116.2, 118.7, 121.8, 125.0, 126.1, 127.0, 127.6, 133.3, 134.5, 134.8, 136.8, 144.2; HRMS (*m/z*, ESI) calc. for $C_{20}H_{19}N_4OClBr$ (+) 445.0425, found 445.0430.

*2,10-Dibromo-13-(cyclohexylamino)-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1v**):* 219 mg (37%) as a green solid; mp 278.2−280.3 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 1.14-1.21 (*m*, 3H), 1.38-1.41(*m*, 2H), 1.57-1.58 (*m*, 1H), 1.69-1.73 (*m*, 2H), 1.91-1.94 (*m*, 2H), 3.03-3.05 (*m*, 1H), 5.86 (*d*, *J* = 7.0 Hz, 1H), 7.32 (*d*, *J* = 8.8 Hz, 1H), 7.82 (*d*, *J* = 8.9 Hz, 1H), 8.45 (*d*, *J* = 9.5 Hz, 1H), 8.55 (*s*, 1H), 8.84 (*d*, *J* = 9.5 Hz, 1H), 9.35 (*s*, 1H), 12.53 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 25.1, 25.6, 33.7, 57.6, 112.4, 114.8, 116.1, 116.2, 118.8, 121.6, 125.9, 126.8, 127.0, 133.5, 134.7, 139.1, 144.2; HRMS (*m/z*, ESI) calc. for $C_{20}H_{19}N_4OBr_2$ (+) 488.9920, found 488.9906.

*13-(Cyclohexylamino)-2-iodo-10-methyl-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1w**):* 206 mg (36%) as a slight yellow solid; mp 285.7−287.8 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.15-1.22 (*m*, 3H), 1.40-1.49 (*m*, 2H), 1.58-1.60 (*m*, 1H), 1.71-1.78 (*m*, 2H), 1.92-1.95 (*m*, 2H), 2.60 (*s*, 3H), 3.01-3.04 (*m*, 1H), 5.83 (*d*, *J* = 6.4 Hz, 1H), 7.14 (*d*, *J* = 8.4 Hz, 1H), 7.91 (*d*, *J* = 8.8 Hz, 1H), 8.16 (*d*, *J* = 9.2 Hz, 1H), 8.75 (*s*, 1H), 8.81 (*d*, *J* = 9.2 Hz, 1H), 8.89 (*s*, 1H), 12.33 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 18.3, 25.1, 25.7, 33.8, 57.3, 87.7, 113.0, 114.5, 118.5, 121.1, 124.6, 125.3, 131.2, 132.9, 133.4, 134.8, 138.9, 139.9, 144.4; HRMS (*m/z*, ESI) calc. for $C_{21}H_{22}N_4OI$ (+) 473.0833, found 473.0811.

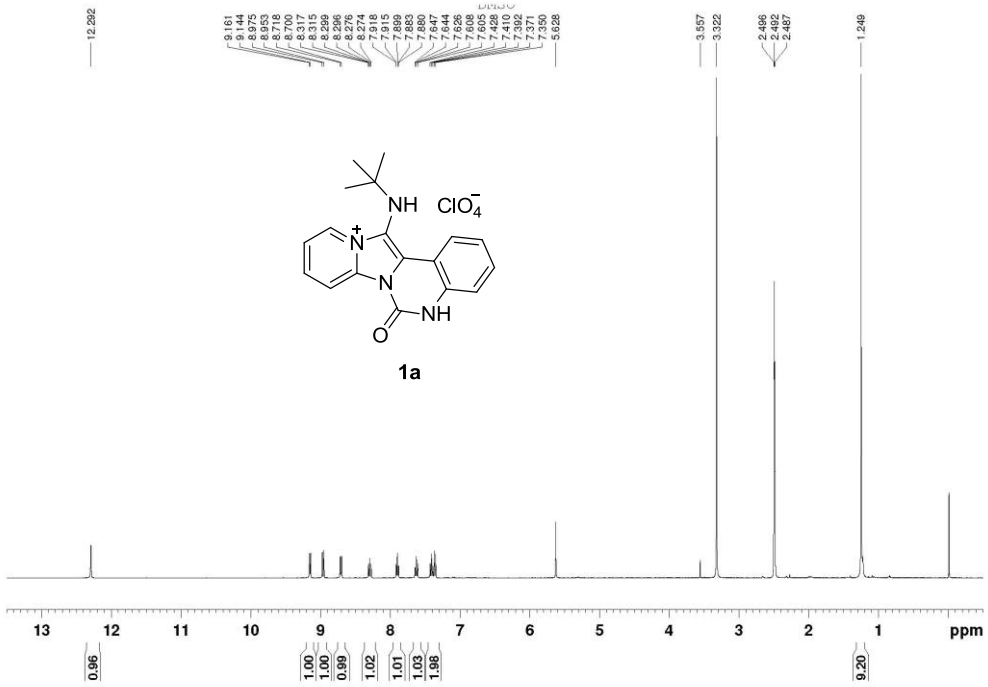
*10-Bromo-13-(cyclohexylamino)-4-fluoro-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1x**):* 175 mg (33%) as a slight yellow solid; mp 254.6−256.7 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.14-1.22 (*m*, 3H), 1.38-1.444 (*m*, 2H), 1.58-1.60 (*m*, 1H), 1.67-1.76 (*m*, 2H), 1.91-1.94 (*m*, 2H), 3.03-3.05 (*m*, 1H), 5.77 (*d*, *J* = 6.4 Hz, 1H), 7.46-7.51 (*m*, 1H), 7.57-7.62 (*m*, 1H), 8.23 (*d*, *J* = 7.6 Hz, 1H), 8.46 (*d*, *J* = 9.2 Hz, 1H), 8.87 (*d*, *J* = 9.2 Hz, 1H), 9.39 (*s*, 1H), 12.53 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 25.1, 25.6, 33.5, 57.5, 112.7, 114.8, 116.2, 117.8, 118.0, 120.7, 123.8, 124.0, 125.2, 125.9, 126.9, 133.7, 139.2, 144.2; HRMS (*m/z*, ESI) calc. for $C_{20}H_{19}N_4OBrF$ (+) 429.0721, found 429.0725.

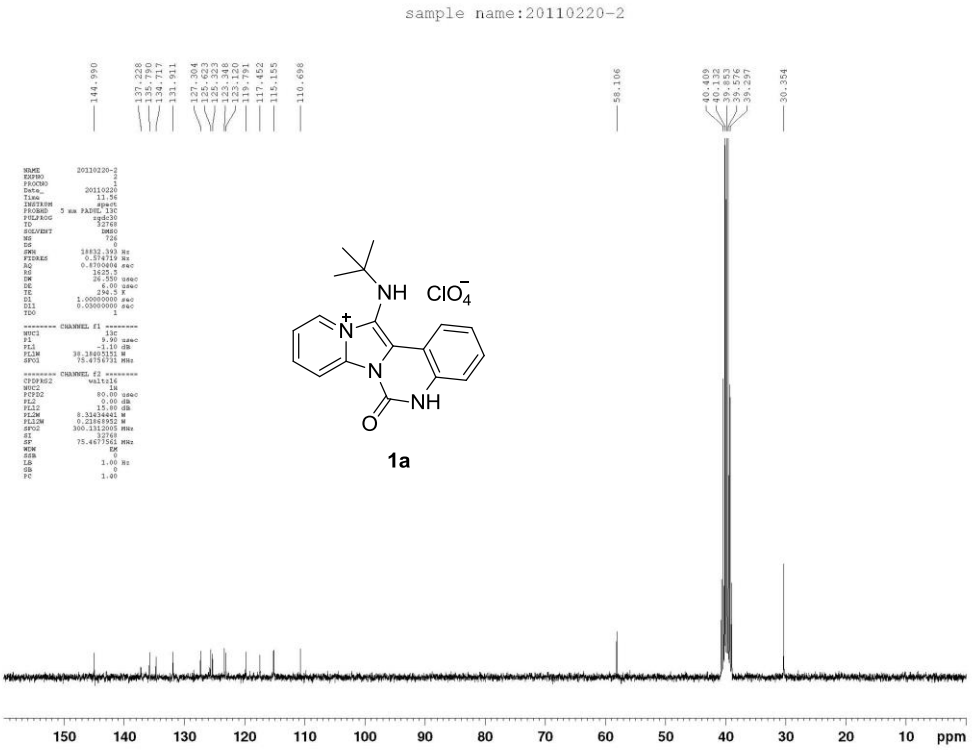
*10-Chloro-13-(cyclohexylamino)-2,4-dimethyl-6-oxo-5,6-dihydropyrido[2',1':2,3]imidazo[1,5-c]quinazolin-12-ium perchlorate (**1y**):* 188 mg (38%) as a yellow solid; mp 212.4−214.2 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ 1.14-1.22 (*m*, 3H), 1.38-1.43 (*m*, 2H), 1.54-1.60 (*m*, 1H), 1.66-1.72 (*m*, 2H), 1.89-1.93 (*m*, 2H), 2.38 (*s*, 3H), 2.42 (*s*, 3H), 3.01-3.04 (*m*, 1H), 5.66 (*d*, *J* = 6.9 Hz, 1H), 7.31 (*s*, 1H), 8.10 (*s*, 1H), 8.24 (*dd*, *J* = 1.8, 9.3 Hz, 1H), 8.95 (*d*, *J* = 9.3 Hz, 1H), 9.26 (*s*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 18.1, 21.0, 25.1, 25.6, 33.7, 57.4, 110.4, 116.2, 122.4, 123.8, 124.2, 124.8, 126.4, 127.1, 132.6, 132.7, 134.3, 135.0, 144.9; HRMS (*m/z*, ESI) calc. for $C_{22}H_{24}N_4OCl$ (+) 395.1633, found 395.1632.

Note: Yields and NMR shifts of the compounds (**1a**, **1e**, **1f**, **1l**, **1m**, **1r**, **1u**, **1v** and **1w**) were already published in CN105017253 A.

^{1}H NMR and ^{13}C NMR spectra

1a

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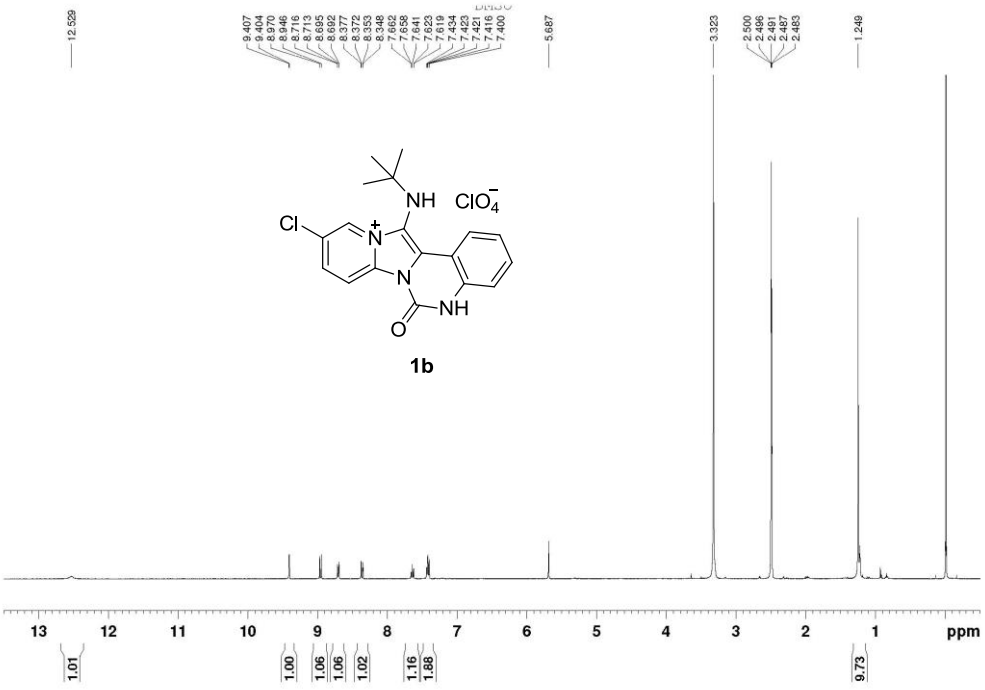
1a

1b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9
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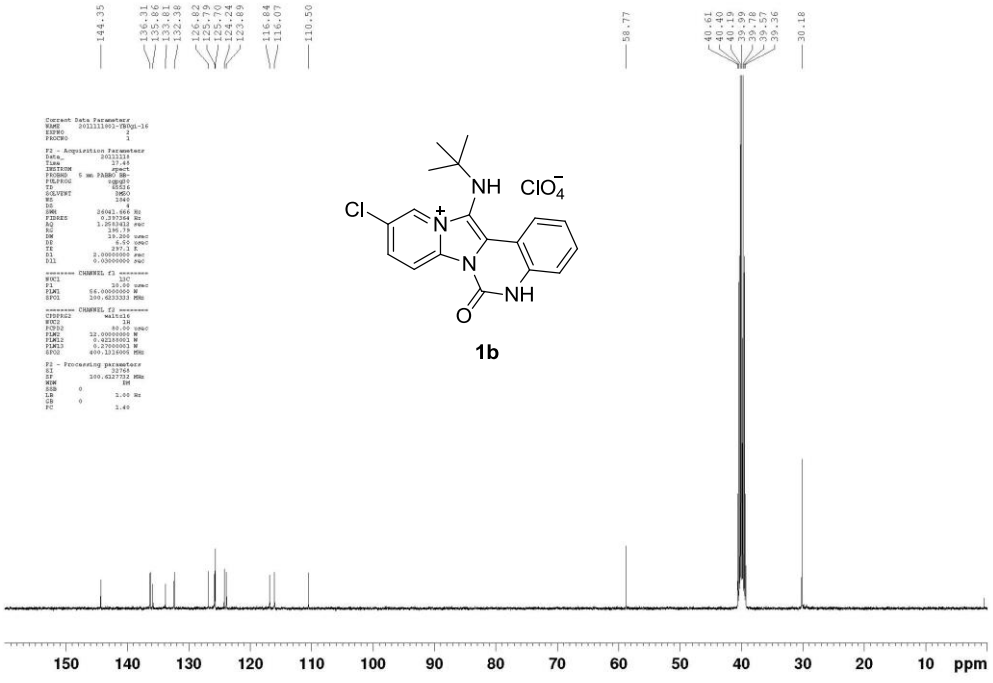
1b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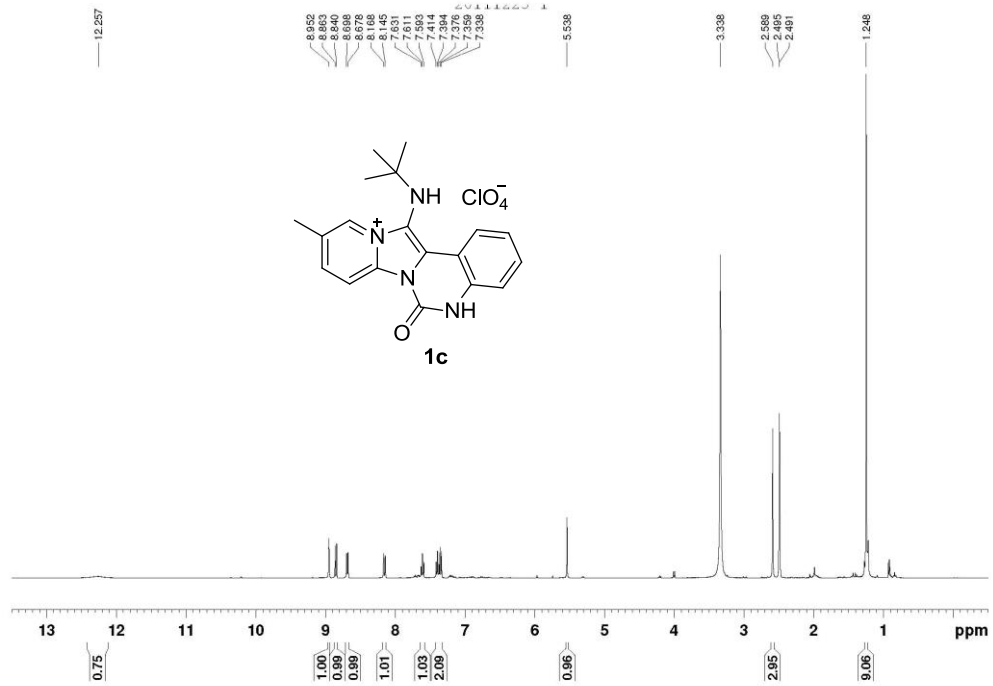
1b

1c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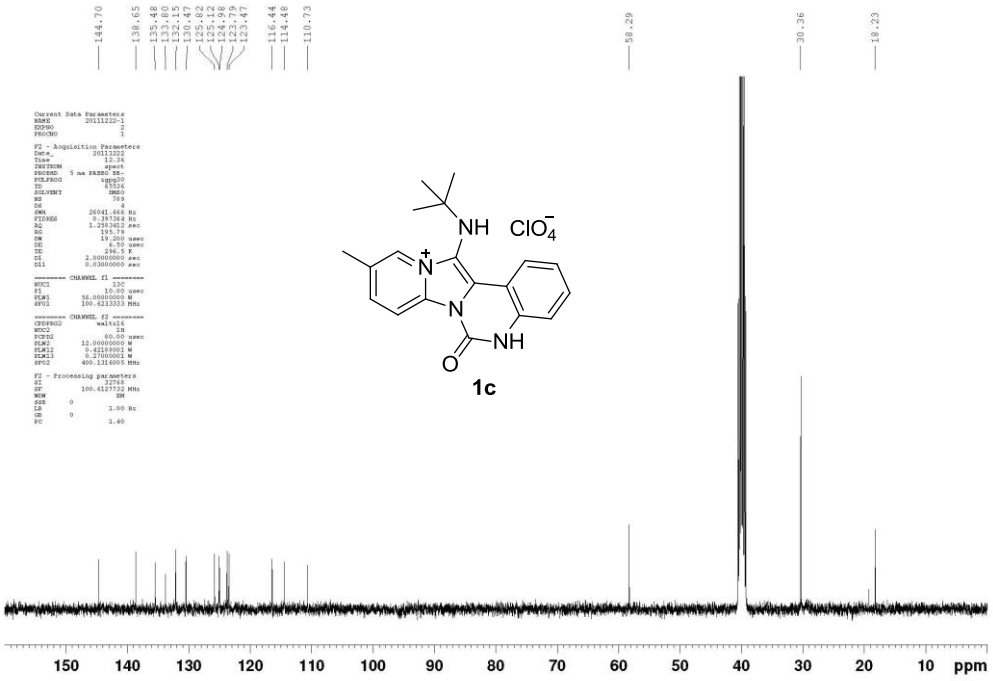
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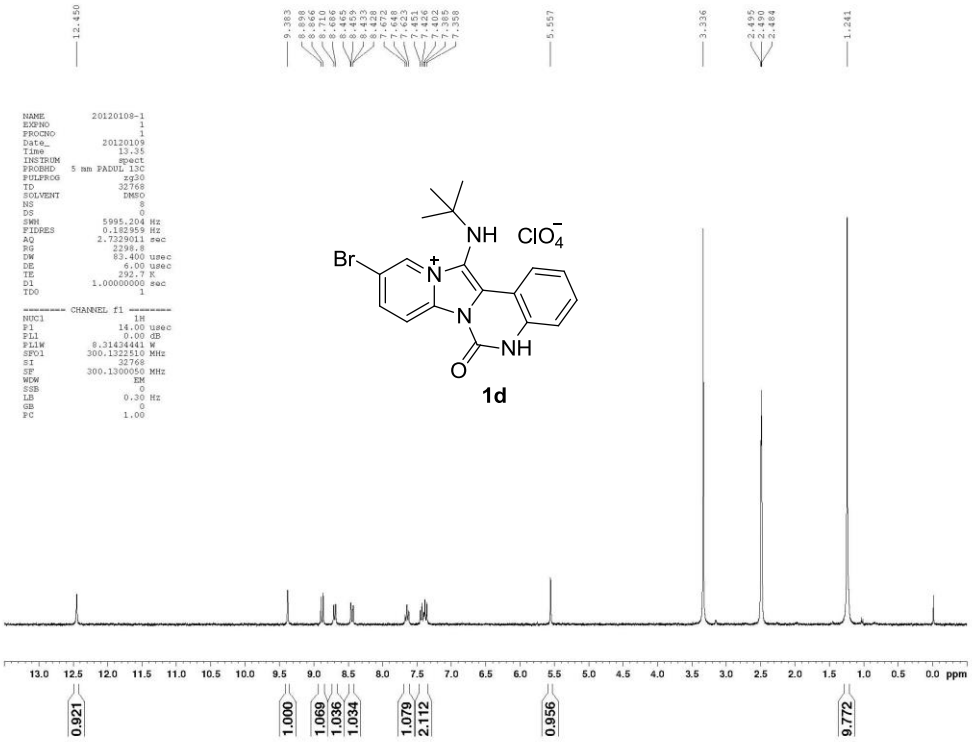
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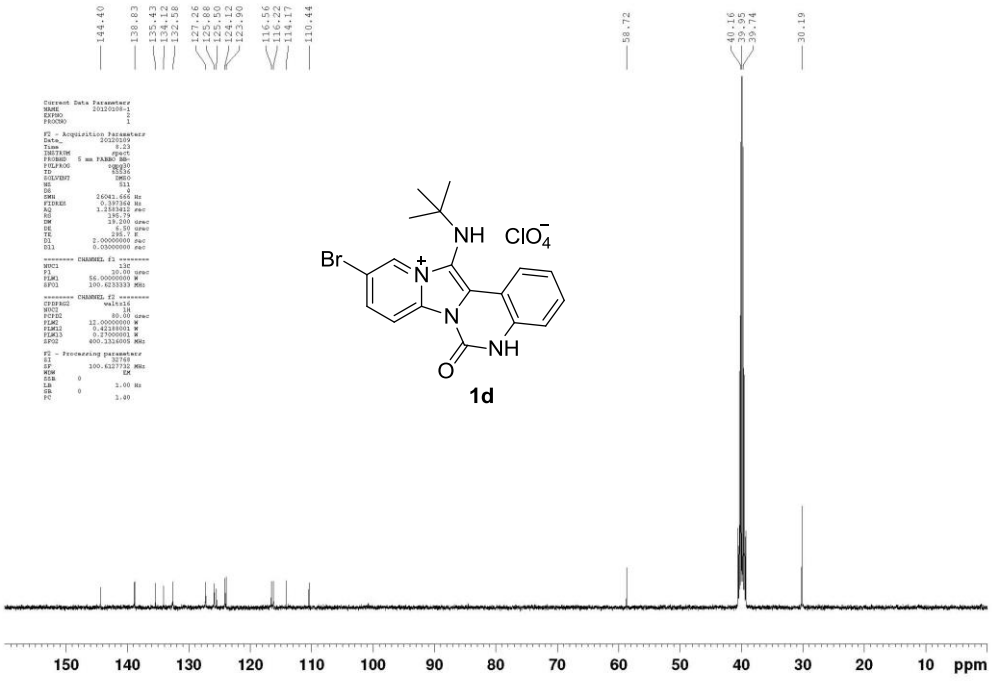
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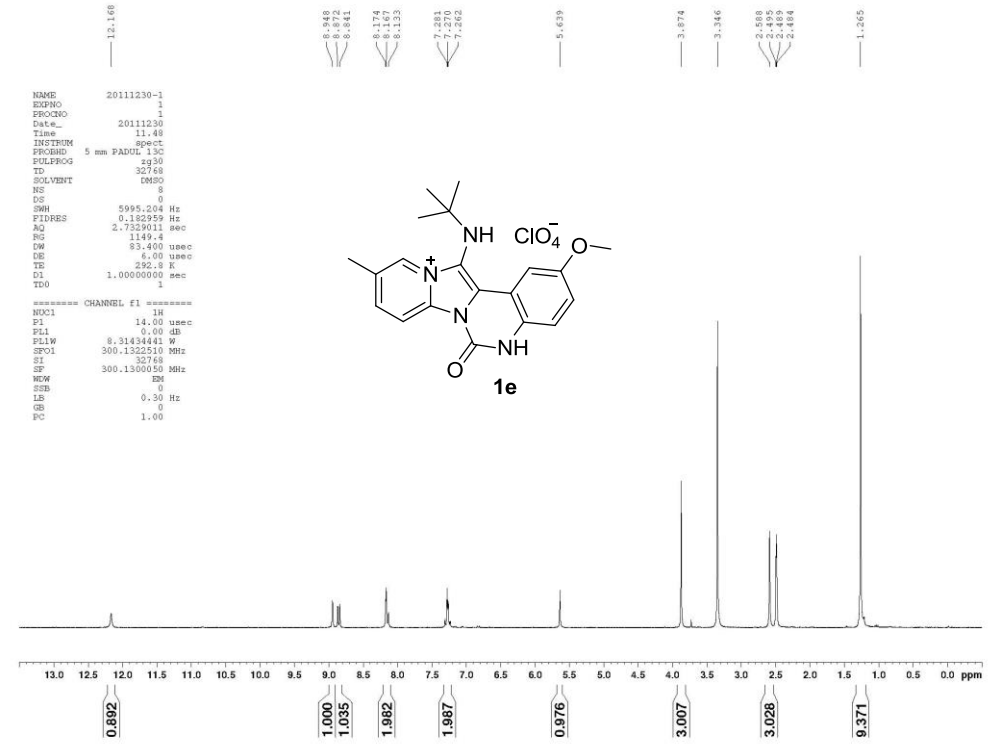
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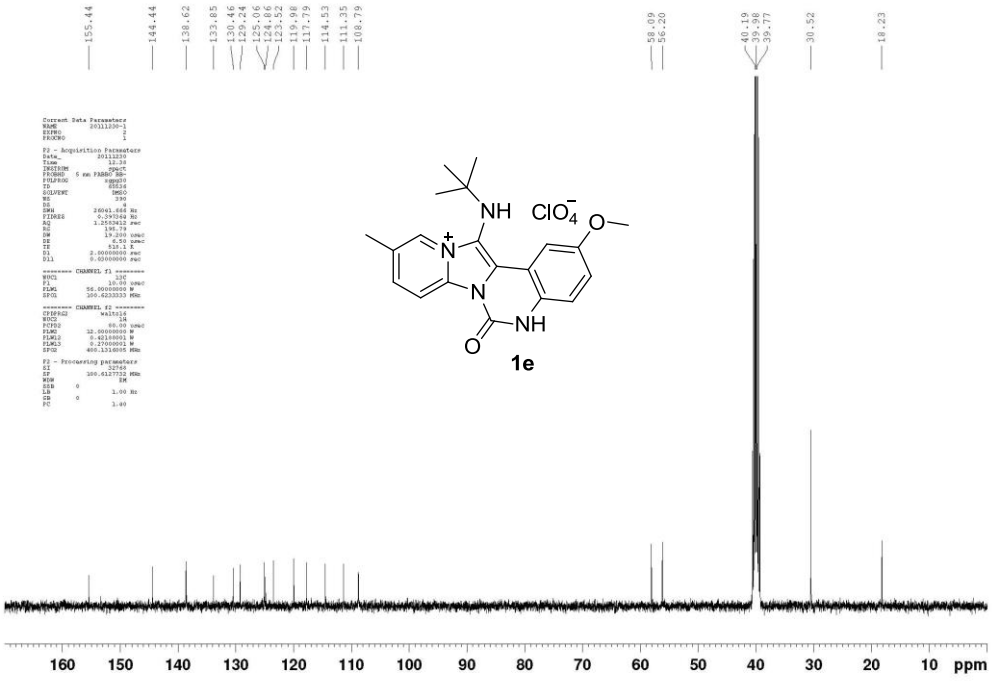
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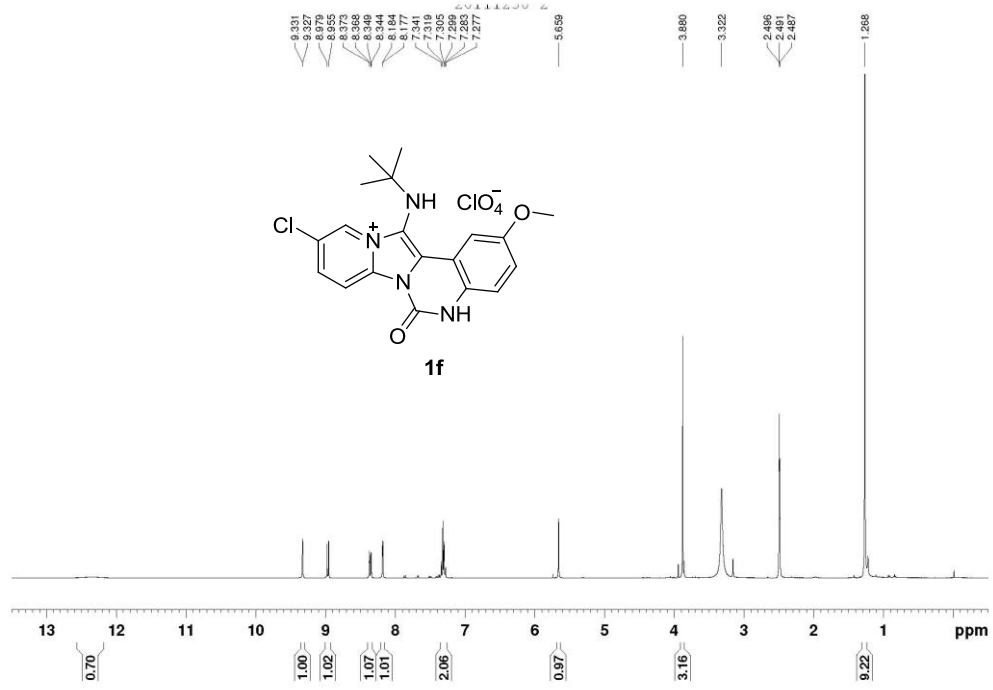
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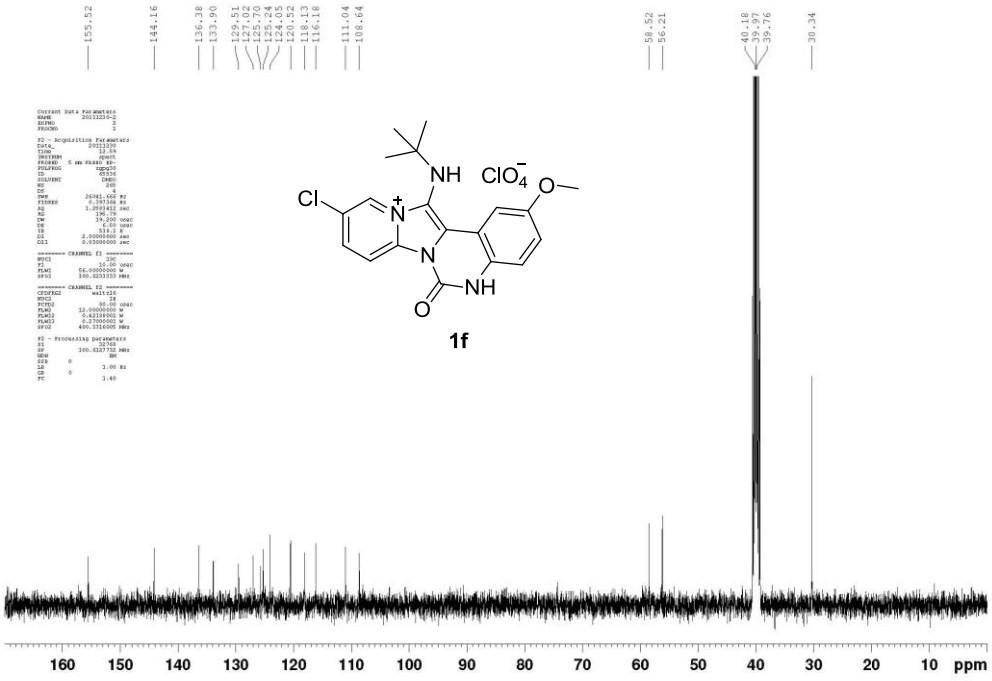
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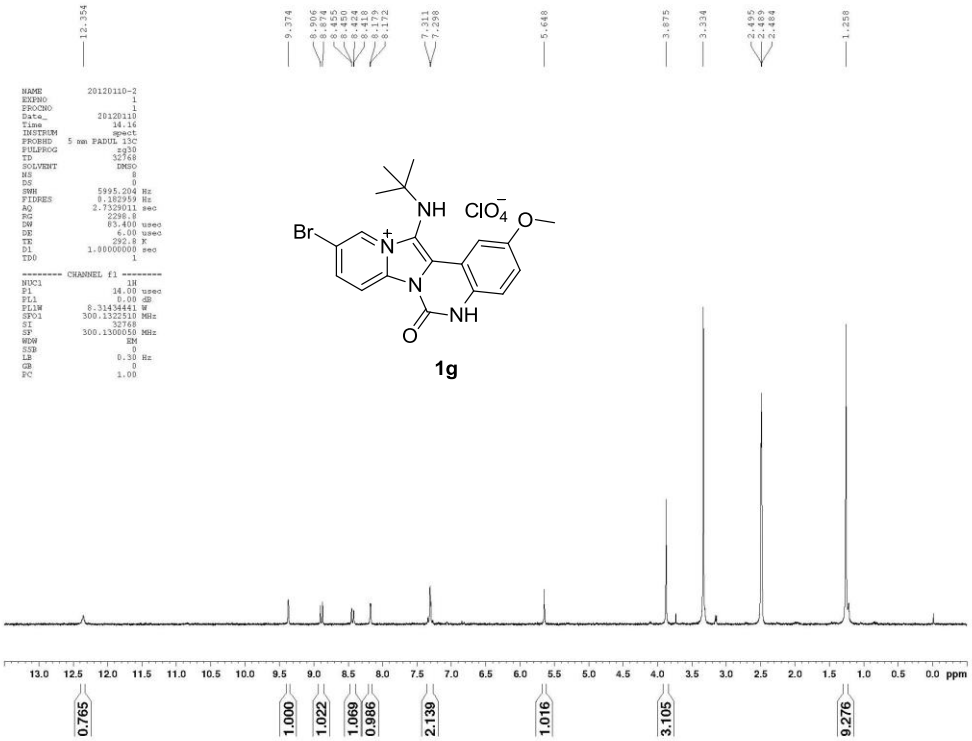
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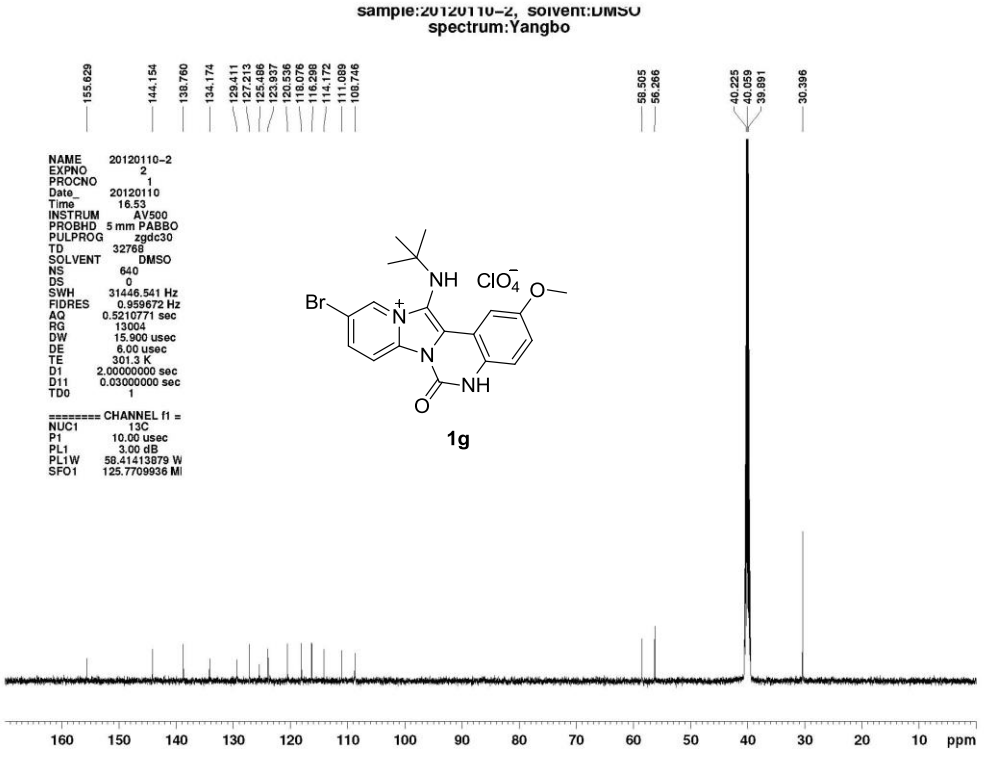
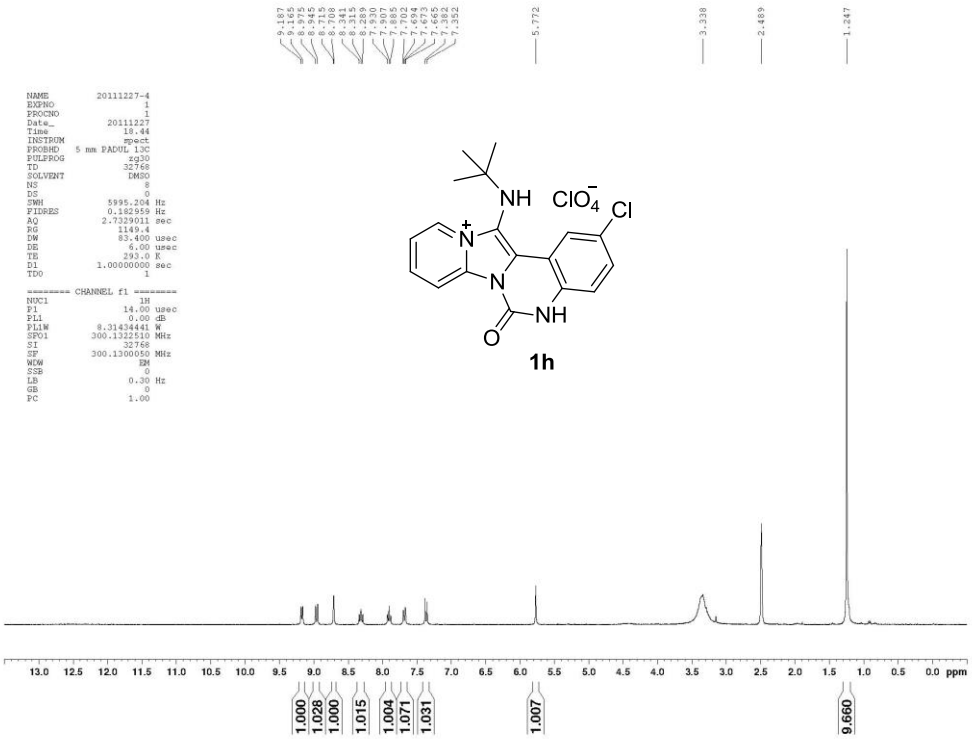
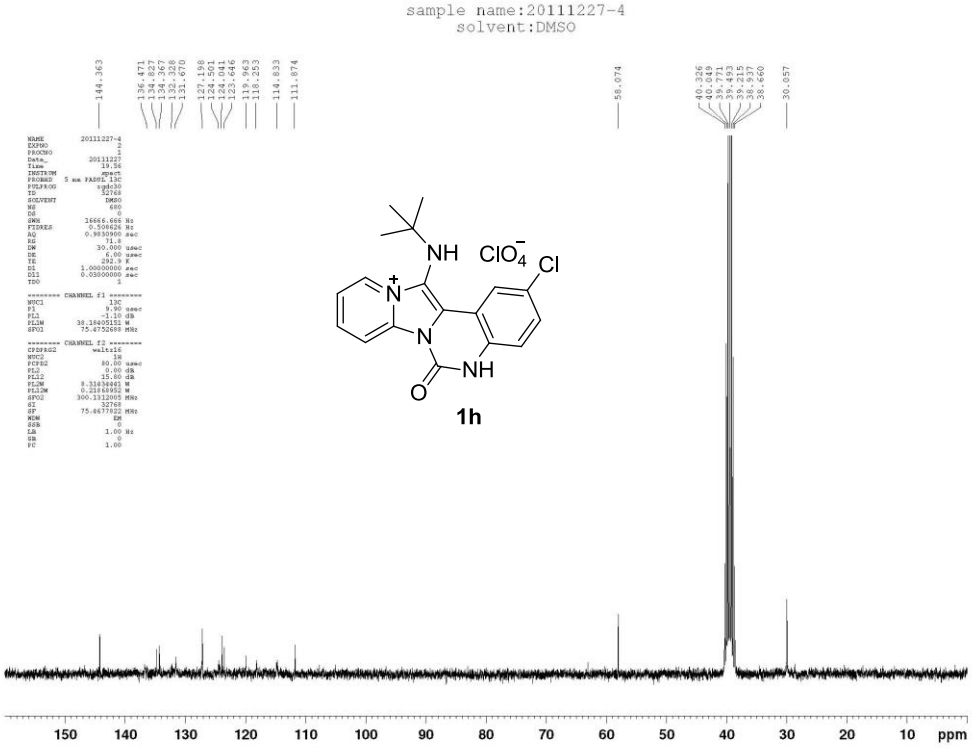
3.105
9.276

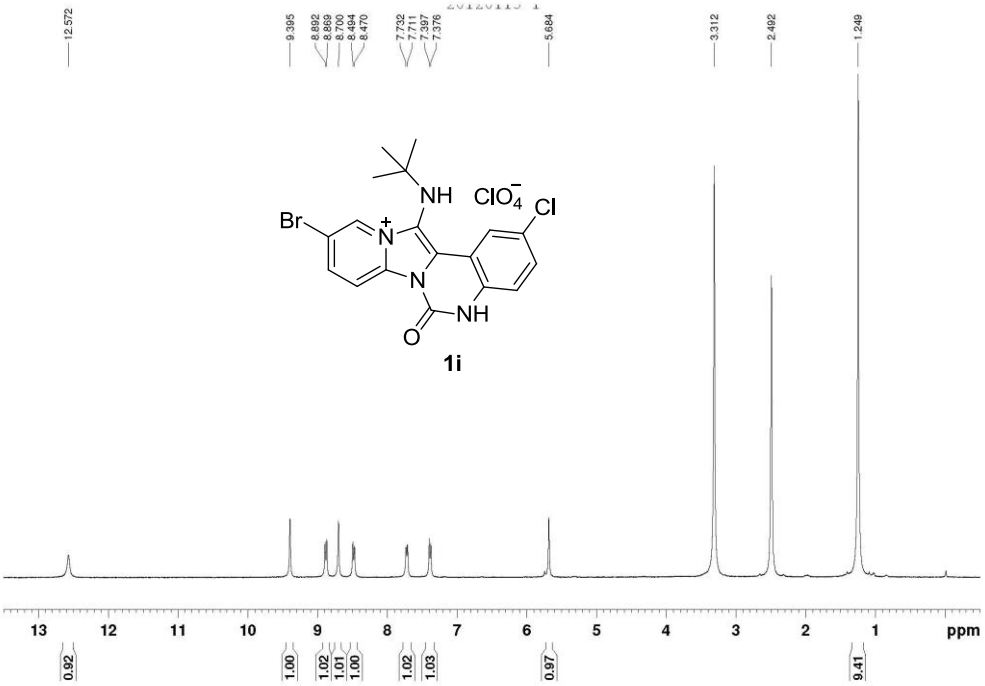

sample:20120110-2, solvent:DMSO
spectrum:Yangbo
155.629
144.154
138.760
134.174
129.411
127.213
125.486
123.937
120.536
118.076
116.298
114.172
111.089
108.746
58.505
56.266
40.225
40.059
39.891
30.396
NAME 20120110-2
EXPNO 2
PROCNO 1
Date_ 20120110
Time 16.53
INSTRUM AV500
PROBHD 5 mm PABBO
PULPROG zgdc30
TD 32768
SOLVENT DMSO
NS 640
DS 0
SWH 31446.541 Hz
FIDRES 0.959672 Hz
AQ 0.5210771 sec
RG 13004
DW 15.900 usec
DE 6.00 usec
TE 301.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1 =
NUC1 13C
P1 10.00 usec
PL1 3.00 dB
PL1W 58.41413879 W
SFO1 125.7709936 MI
NH
ClO4
O
Br
N
NH
O
1g
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

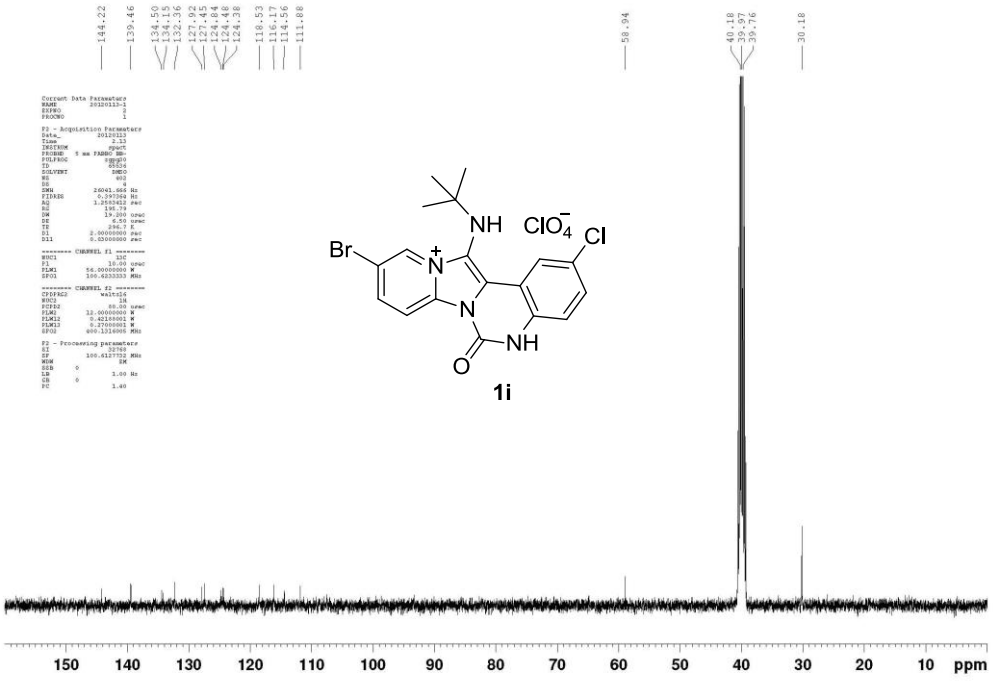
1h


NAME 20111227-4
EXPNO 1
PROCNO 1
Date_ 20111227
Time 18.44
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 1149.4
DW 83.400 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 300.1322510 MHz
SI 32768
SF 300.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NH
ClO4
Cl
N
NH
O
1h
9.187
9.165
8.975
8.945
8.715
8.700
8.341
8.315
8.030
7.930
7.907
7.885
7.802
7.694
7.673
7.665
7.652
7.352
5.772
3.338
2.489
1.247
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.000
1.028
1.000
1.015
1.004
1.071
1.031
1.007
9.660


sample name:20111227-4
solvent:DMSO
NAME 20111227-4
EXPNO 2
PROCNO 1
Date_ 20111227
Time 19.56
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 680
DS 0
SWH 16666.666 Hz
FIDRES 0.508626 Hz
AQ 0.9830900 sec
RG 71.8
DW 30.000 usec
DE 6.00 usec
TE 292.9 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.90 usec
PL1 -1.10 dB
PL1W 38.18905151 W
SFO1 75.4752688 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.80 dB
PL13 8.31434441 W
PL12W 0.21868952 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677822 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
NH
ClO4
Cl
N
NH
O
1h
144.363
136.471
134.227
134.367
132.328
131.670
127.198
124.501
124.041
123.646
119.963
118.253
114.833
111.874
58.074
40.396
40.049
39.771
39.493
39.215
38.937
38.660
30.057
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

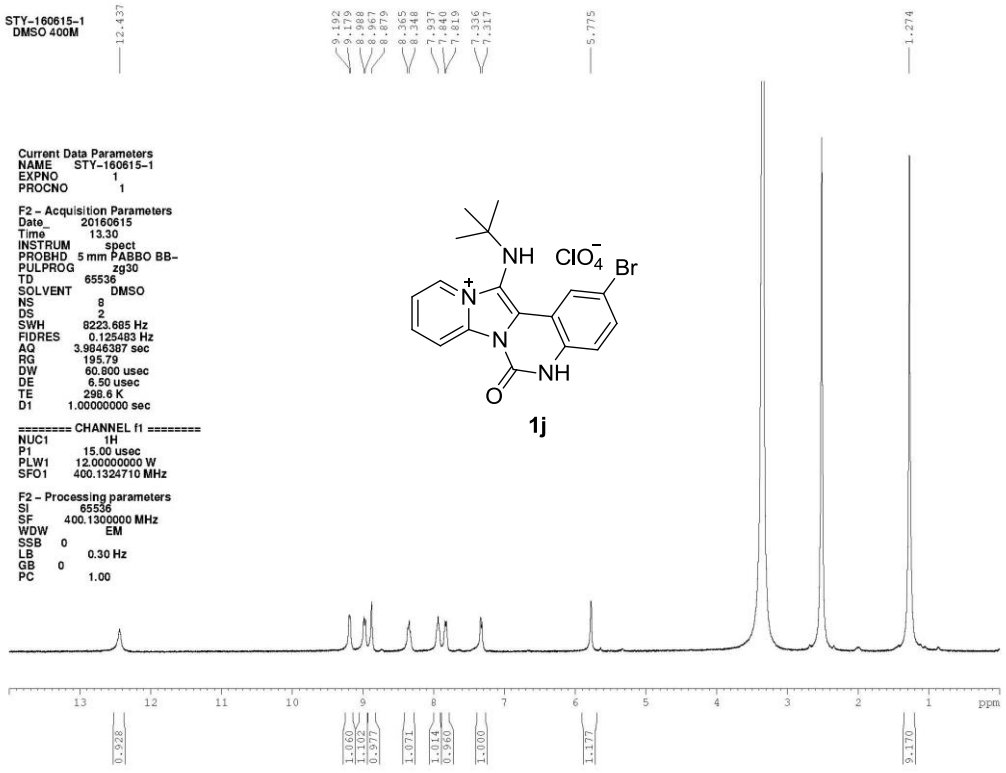
1i

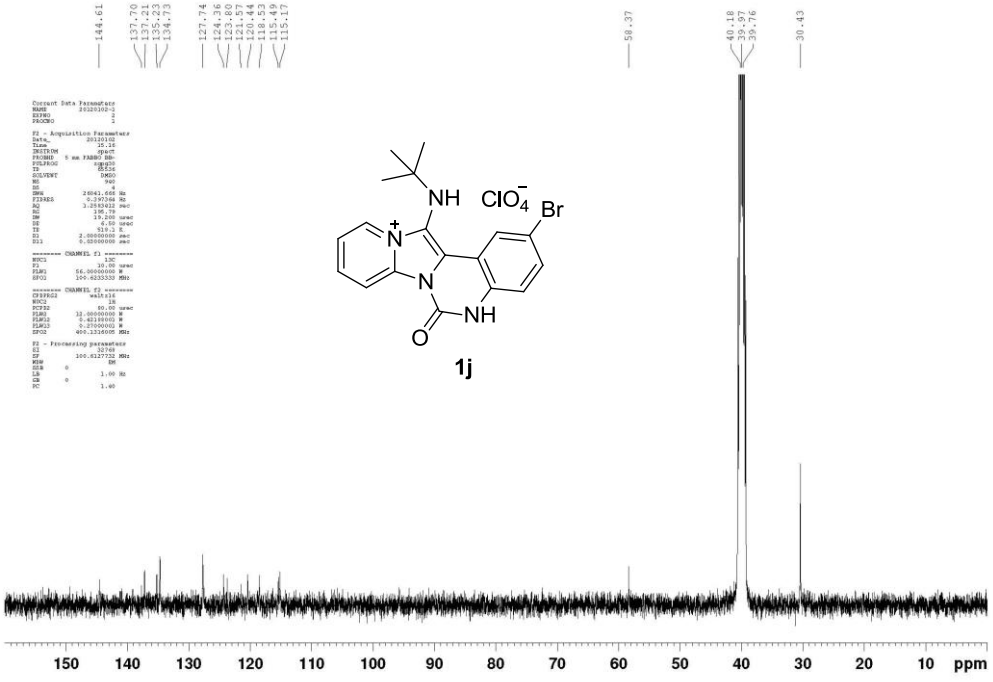

12.572
9.395
8.892
8.868
8.709
8.494
8.470
7.732
7.711
7.393
7.376
5.684
3.312
2.492
1.249
NH
ClO4−
Cl
Br
N+
N
NH
O
1i
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.92
1.00
1.02
1.01
1.00
1.02
1.03
0.97
9.41


144.22
139.46
134.50
134.15
132.36
127.92
127.45
124.84
124.48
124.38
118.53
116.17
114.56
111.88
58.94
40.18
39.97
39.76
30.18
Br
NH
ClO4
Cl
N
N
NH
O
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

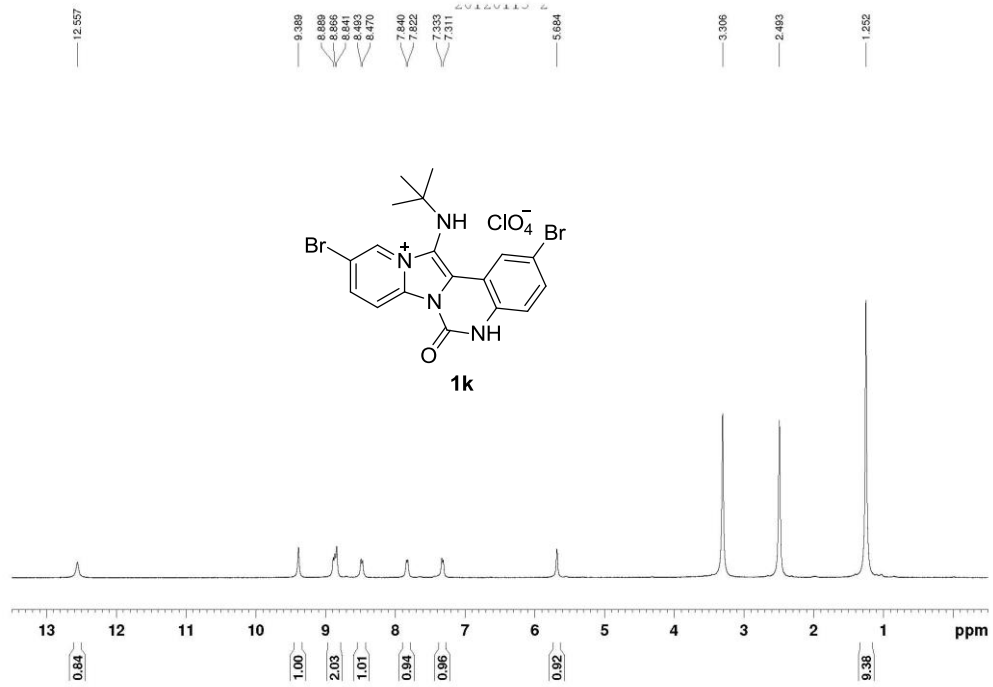
1i

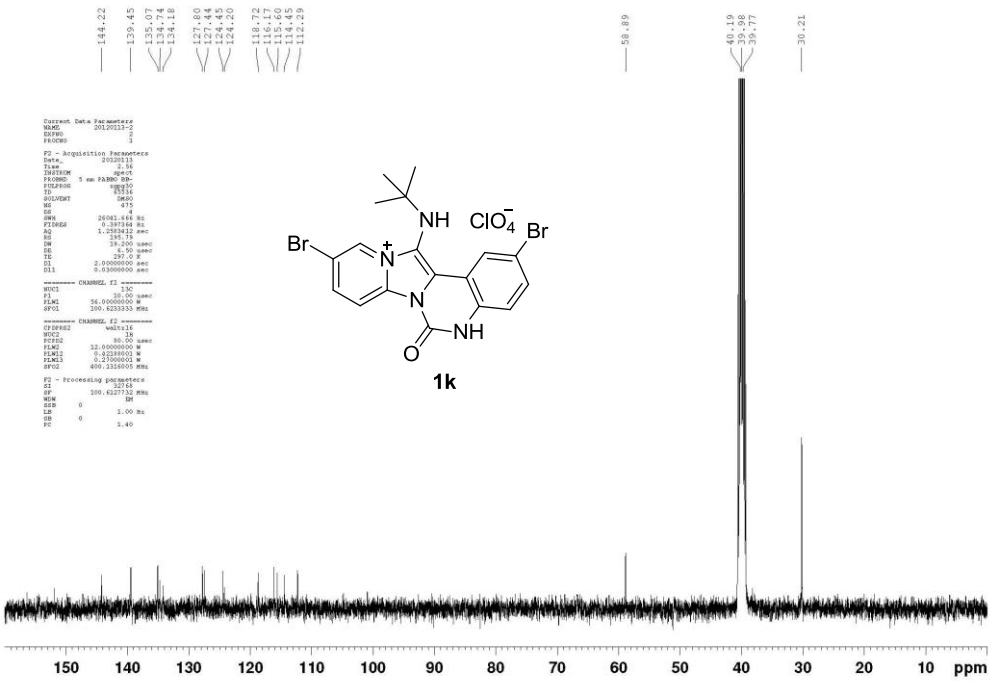
1j


STY-160615-1
DMSO 400M
12.437
9.192
9.179
8.988
8.967
8.879
8.365
8.348
7.937
7.840
7.819
7.336
7.317
5.775
1.274
Current Data Parameters
NAME STY-160615-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160615
Time 13.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 195.79
DW 60.800 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NH
ClO4
Br
N
NH
O
1j
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.928
1.060
1.102
0.977
1.071
1.014
0.960
1.000
1.177
9.170

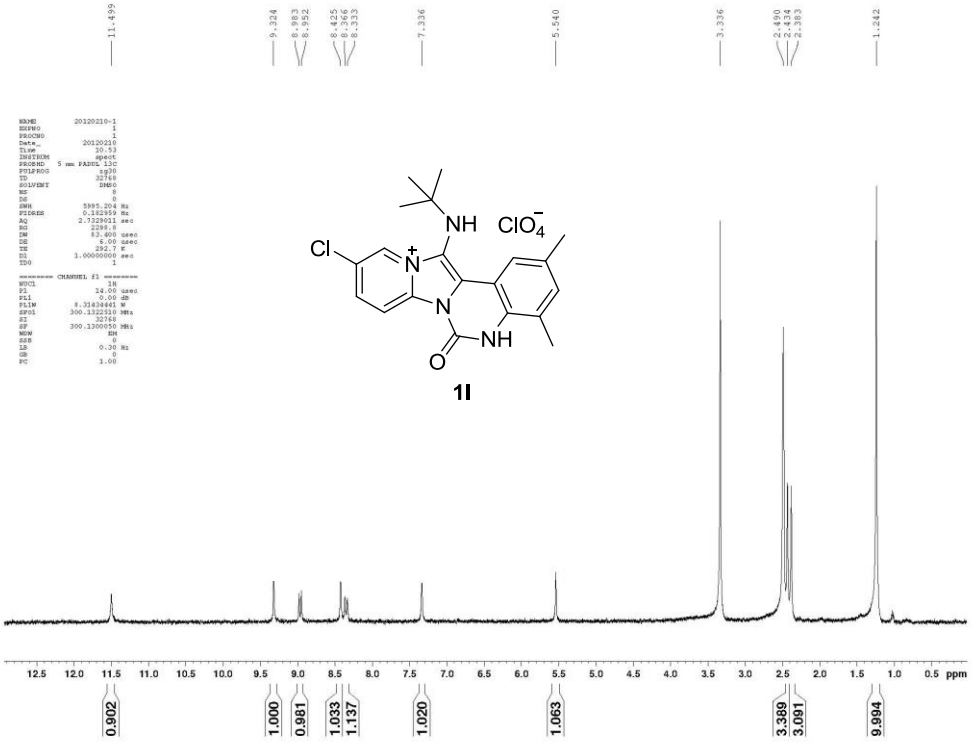

144.61
137.70
137.21
135.23
134.73
127.74
124.36
123.80
121.57
120.44
118.53
115.49
115.17
58.37
40.18
39.97
39.76
30.43
NH
ClO4
Br
N
NH
O
1j
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

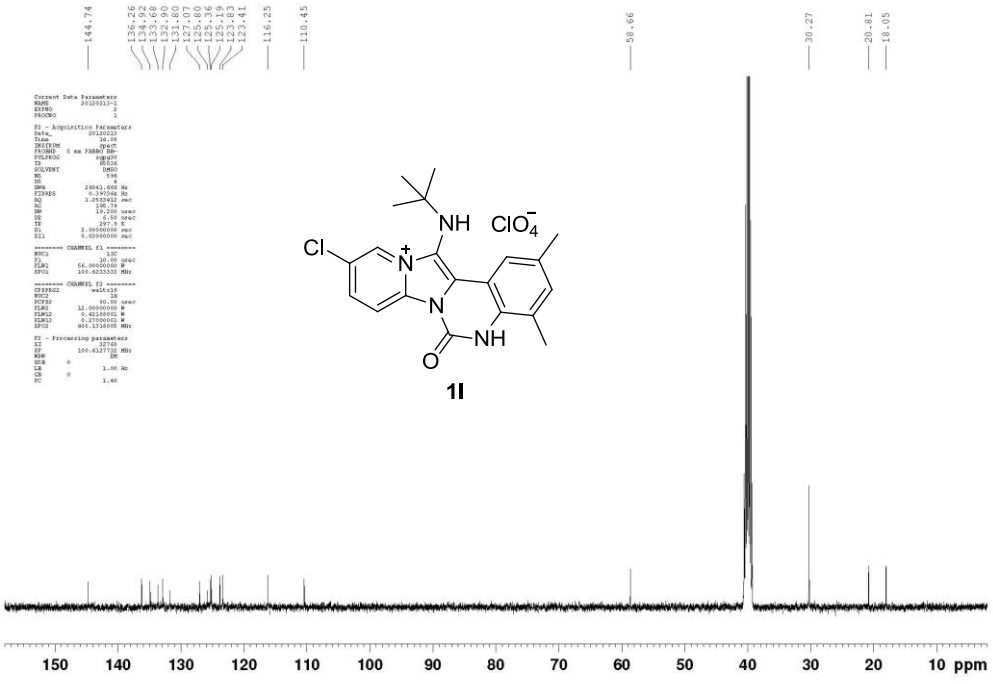
1k


12.557
9.389
8.899
8.866
8.841
8.493
8.470
7.840
7.822
7.333
7.311
5.684
3.306
2.493
1.252
NH
ClO4
Br
Br
N
N
NH
O
1k
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.84
1.00
2.03
1.01
0.94
0.96
0.92
9.38


144.22
139.45
135.07
134.74
134.18
127.60
127.44
124.45
124.20
118.72
116.77
116.60
114.45
112.29
58.89
40.19
39.98
39.77
30.21
NH
ClO4
Br
Br
N
N
NH
O
1k
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

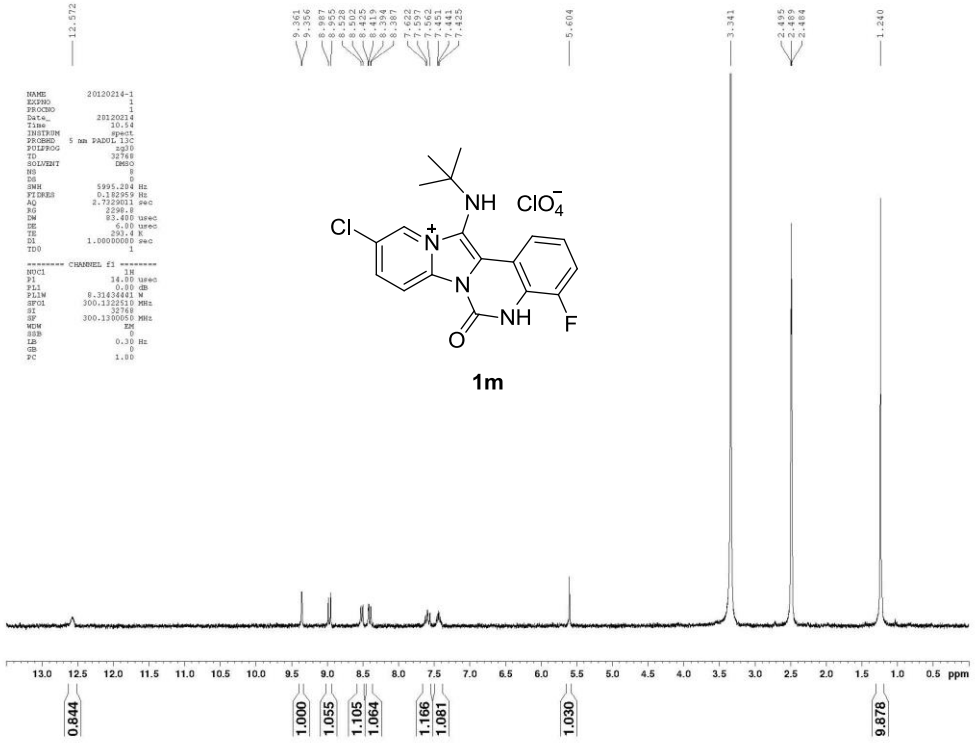
1l


11.499
9.324
9.003
8.992
8.425
8.366
8.333
7.336
5.540
3.396
2.490
2.434
2.383
1.242
NAME 20120210-1
EXPNO 1
PROCNO 1
Date_ 20120210
Time 20.53
INSTRUM spect
PROBHD 5 mm PABBO 13C
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 2286.8
DW 83.400 usec
DE 6.00 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
PLW 8.31838441 W
SFO1 300.1322510 MHz
SI 32768
SF 300.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NH
ClO4
Cl
N
N
NH
O
1l
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.902
1.000
0.981
1.033
1.137
1.020
1.063
3.389
3.091
9.994

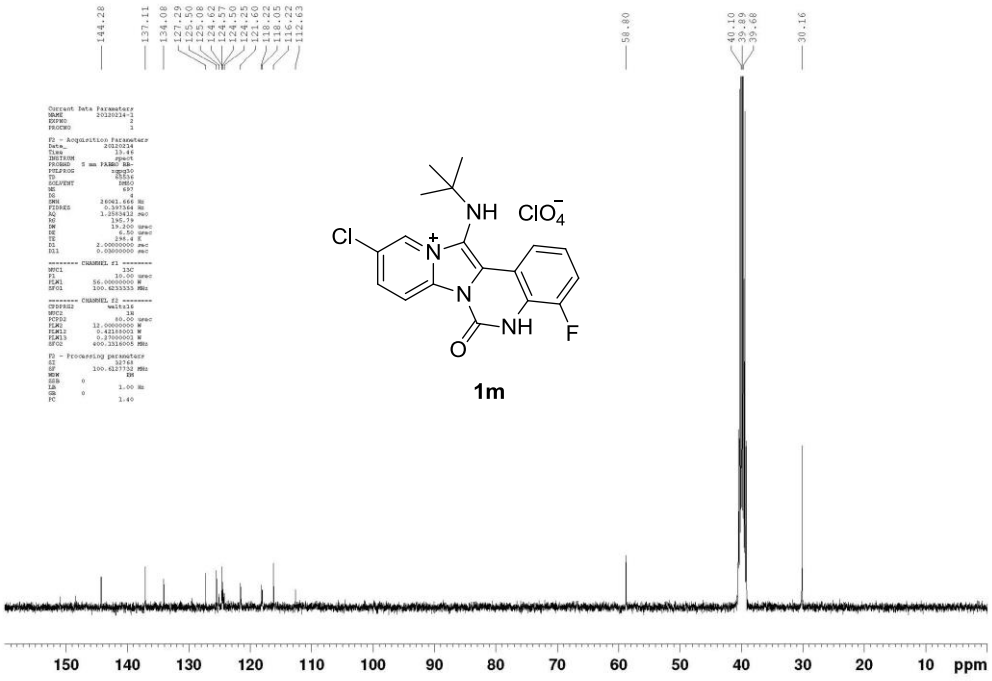

144.74
136.26
134.92
134.68
132.90
131.80
127.07
125.80
125.36
125.19
123.83
123.41
116.25
110.45
58.66
30.27
20.81
18.05
NH
ClO4
Cl
N
N
NH
O
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

1l

1m

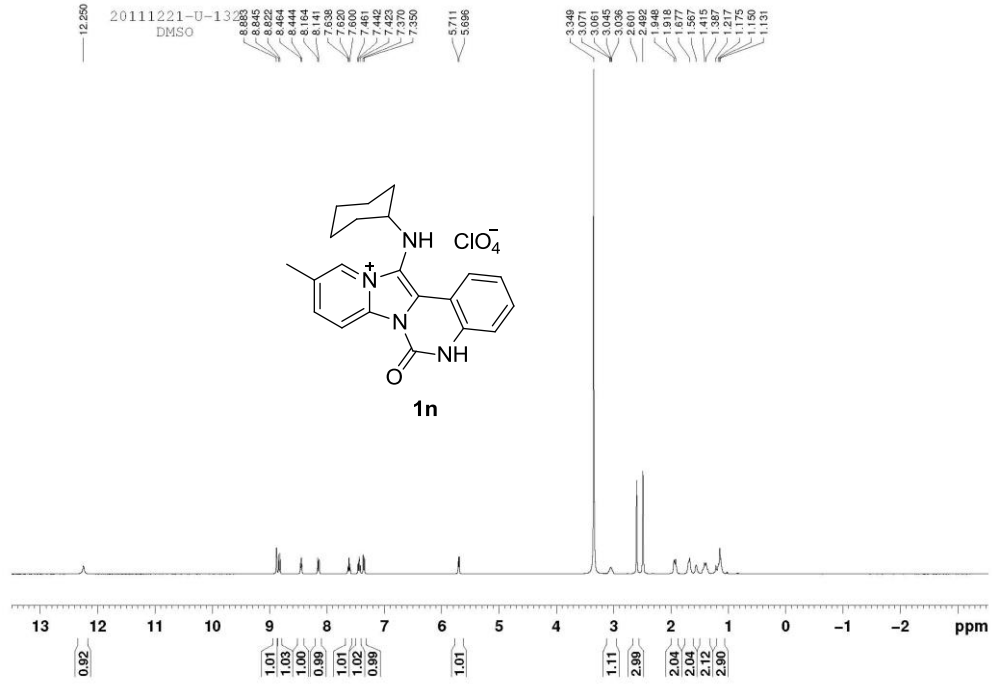

12.572
9.361
9.036
8.807
8.955
8.528
8.502
8.435
8.419
8.394
8.387
7.652
7.562
7.562
7.451
7.441
7.425
5.604
3.341
2.495
2.469
2.484
1.240
NAME 20120214-1
EXPNO 1
PROCNO 1
Date_ 20120214
Time 10.54
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 2296.8
DW 83.400 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 300.1322510 MHz
SI 32768
SF 300.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NH
ClO4
Cl
N
N
NH
F
O
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.844
1.000
1.055
1.105
1.064
1.166
1.081
1.030
9.878

1m

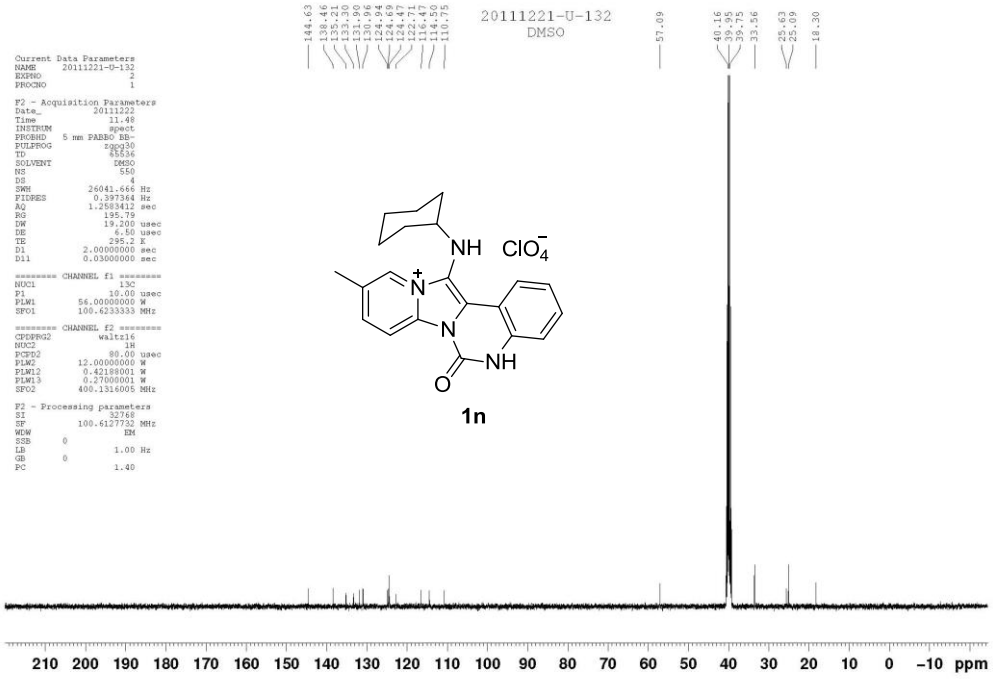

144.28
137.11
134.08
127.29
125.50
125.08
124.62
124.57
124.50
124.25
121.60
118.22
118.05
116.22
112.63
59.80
40.10
39.89
39.68
30.16
NH
ClO4
Cl
N
N
NH
F
O
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

1m

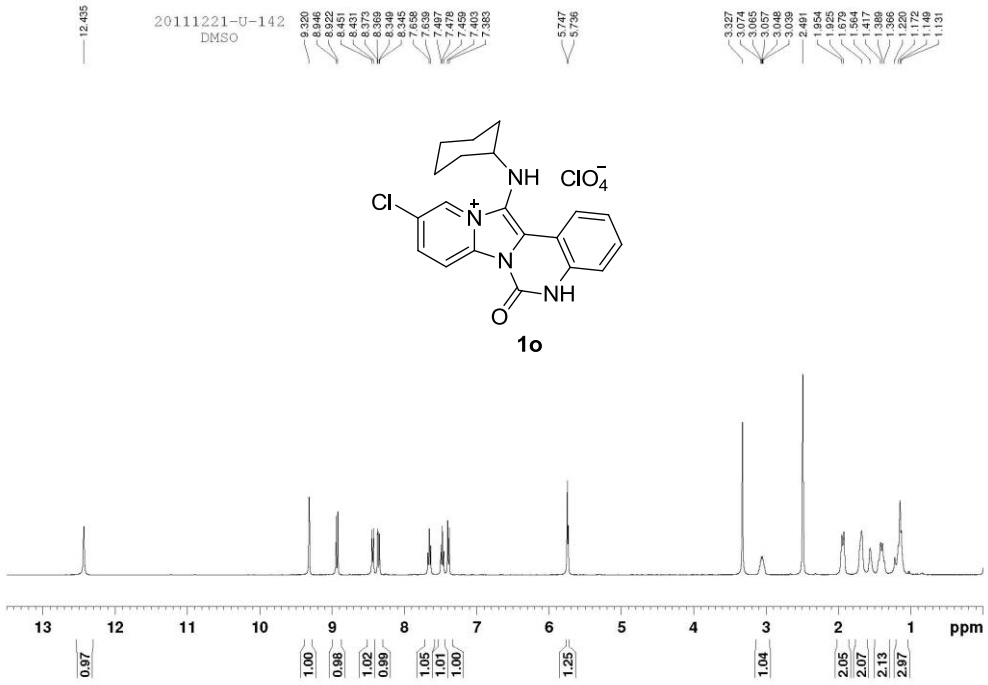
1n

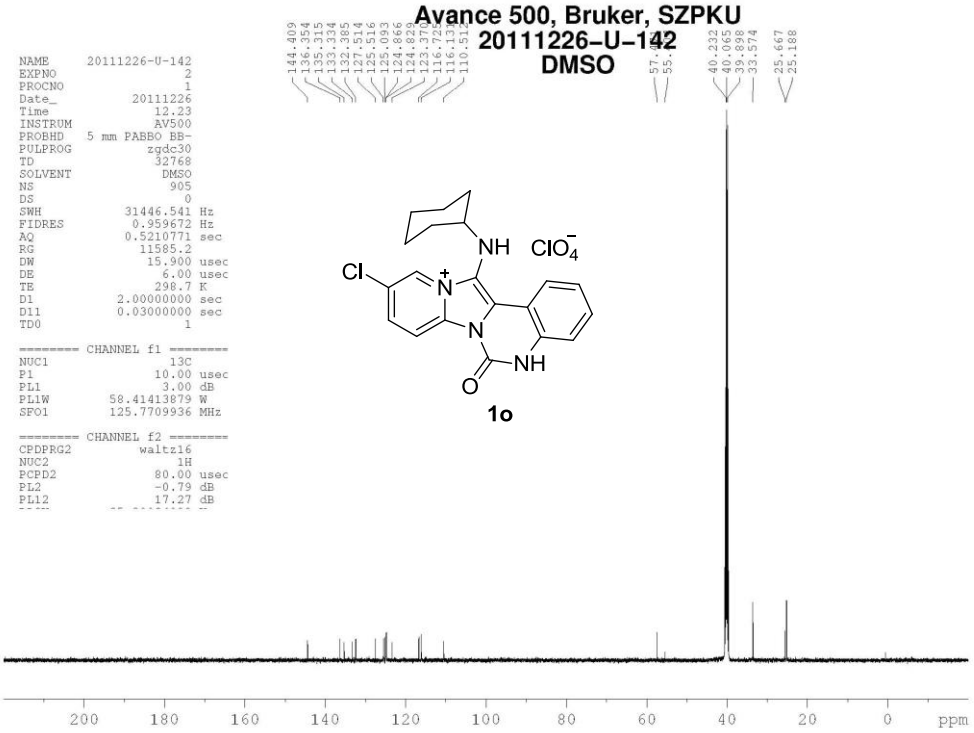

12.250
20111221-U-132
DMSO
8.883
8.845
8.822
8.464
8.444
8.164
8.141
7.638
7.620
7.600
7.461
7.442
7.423
7.370
7.350
5.711
5.696
3.349
3.071
3.061
3.045
3.036
2.601
2.492
1.948
1.918
1.677
1.567
1.415
1.387
1.277
1.175
1.150
1.131
NH
ClO4
N
N
NH
O
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
ppm
0.92
1.01
1.03
1.00
0.99
1.01
1.02
0.99
1.01
1.11
2.99
2.04
2.04
2.12
2.90

1n

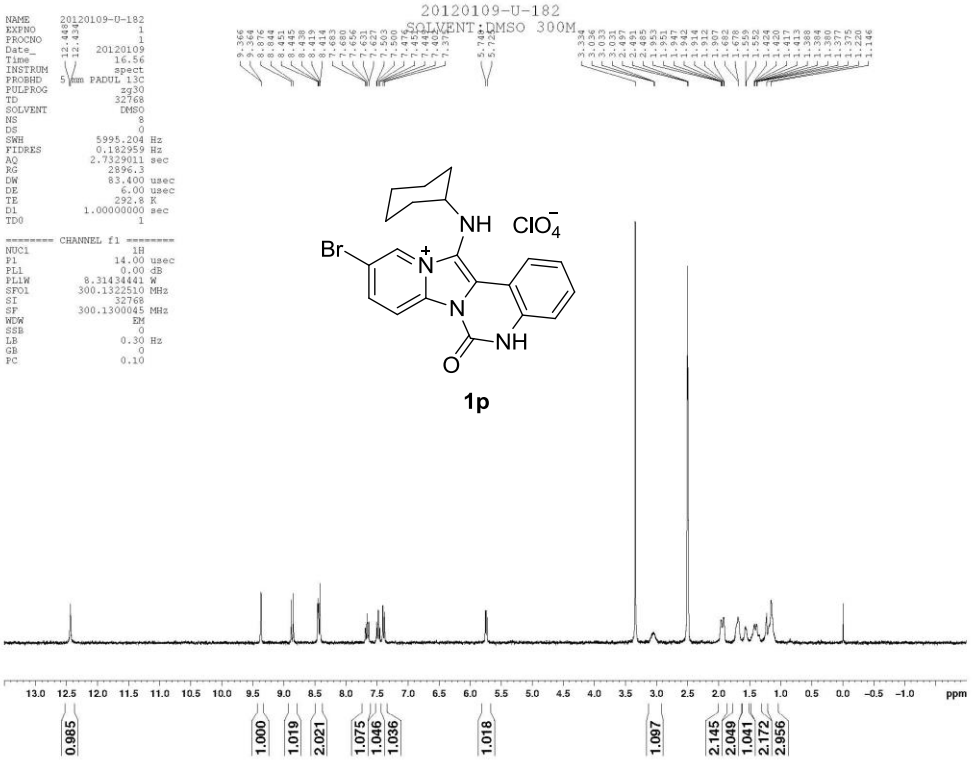

144.63
138.46
135.21
133.00
131.90
130.96
124.94
124.69
124.47
122.71
116.47
114.50
110.75
20111221-U-132
DMSO
57.09
40.16
39.95
39.75
33.56
25.63
23.09
10.30
Current Data Parameters
NAME 20111221-U-132
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111222
Time 11.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 550
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42188001 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127732 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
NH
ClO4
N
N
NH
O
1n
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

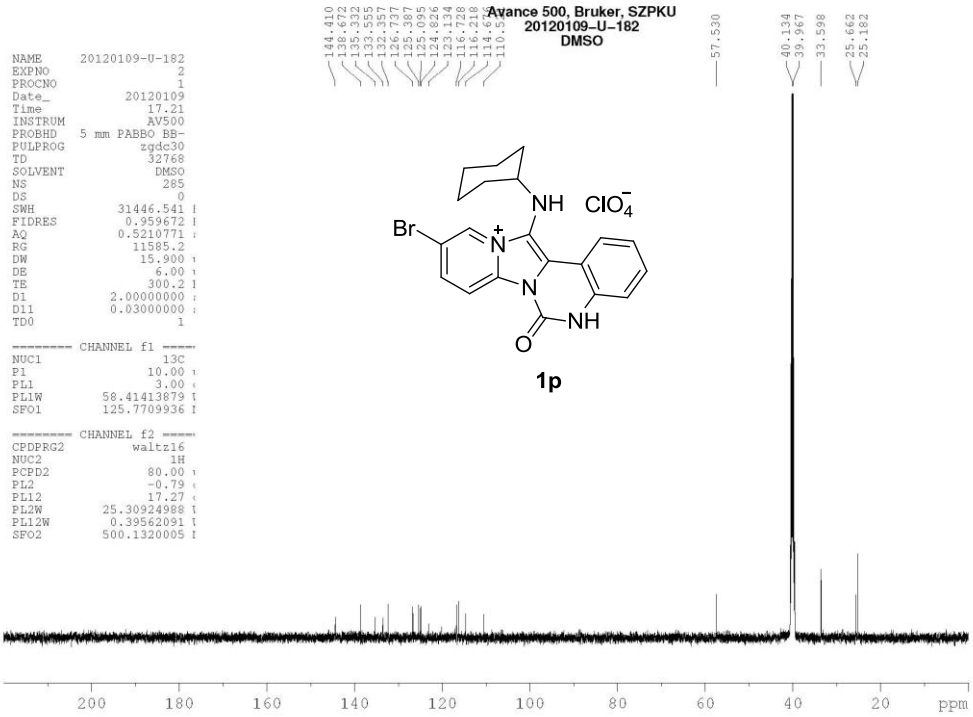
1o


20111221-U-142
DMSO
12.435
9.320
8.946
8.902
8.451
8.431
8.373
8.369
8.349
8.345
7.658
7.639
7.497
7.478
7.459
7.403
7.383
5.747
5.736
3.327
3.074
3.065
3.057
3.048
3.039
2.491
1.954
1.925
1.679
1.564
1.417
1.389
1.366
1.290
1.172
1.149
1.131
NH
ClO4
Cl
N
N
NH
O
1o
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.97
1.00
0.98
1.02
0.99
1.05
1.01
1.00
1.25
1.04
2.05
2.07
2.13
2.97

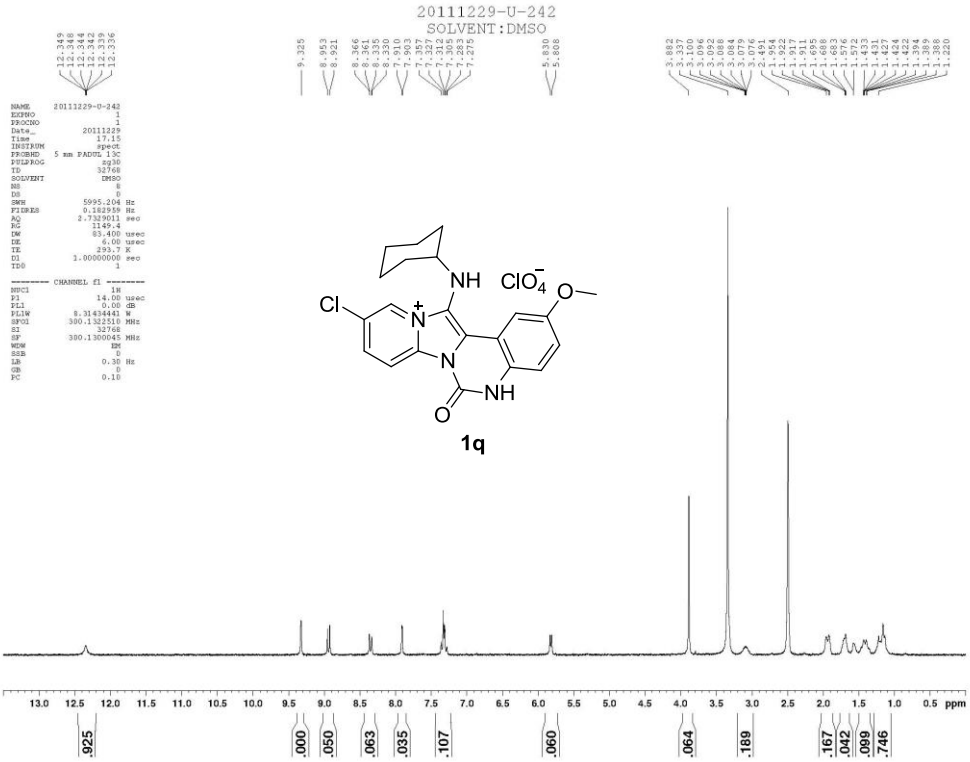

Avance 500, Bruker, SZPKU
20111226-U-142
DMSO
144.409
136.354
135.315
133.334
132.385
127.514
125.516
125.093
124.866
124.829
123.370
116.725
116.137
110.512
57.
55.
40.232
40.065
39.898
33.574
25.667
25.188
NAME 20111226-U-142
EXPNO 2
PROCNO 1
Date_ 20111226
Time 12.23
INSTRUM AV500
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 32768
SOLVENT DMSO
NS 905
DS 0
SWH 31446.541 Hz
FIDRES 0.959672 Hz
AQ 0.5210771 sec
RG 11585.2
DW 15.900 usec
DE 6.00 usec
TE 298.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.00 dB
PL1W 58.41413879 W
SFO1 125.7709936 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -0.79 dB
PL12 17.27 dB
NH
ClO4
Cl
N
N
NH
O
1o
200
180
160
140
120
100
80
60
40
20
0
ppm

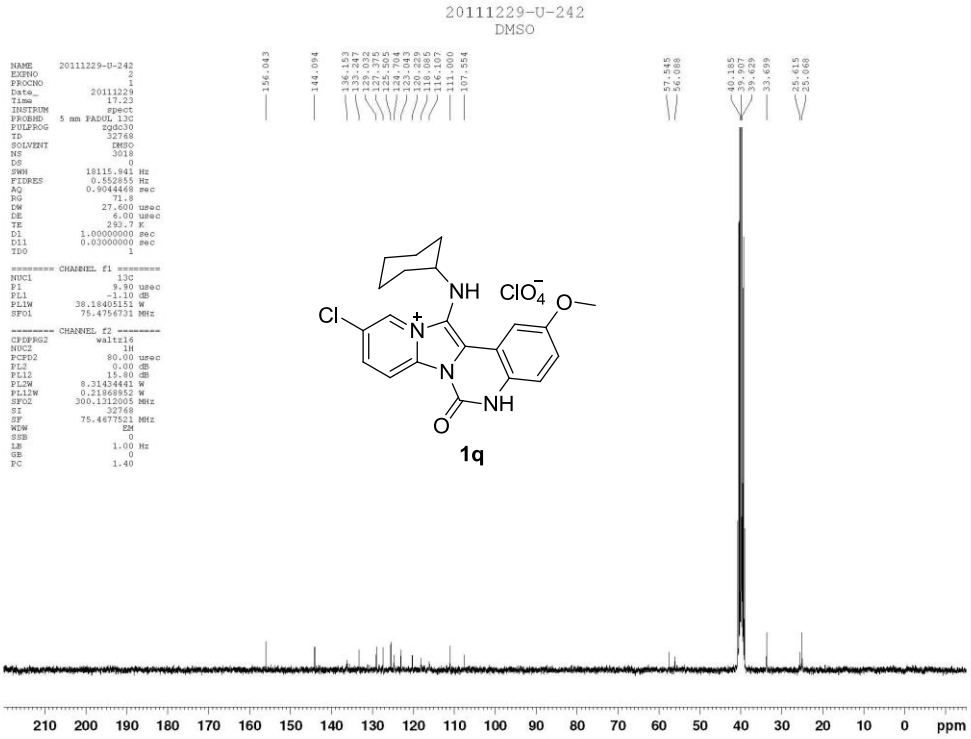
1p


20120109-U-182
SOLVENT:DMSO 300M
NAME 20120109-U-182
EXPNO 1
PROCNO 1
Date_ 20120109
Time 16.56
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 2896.3
DW 83.400 usec
DE 6.00 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
PLW 8.31434441 W
SFO1 300.1322510 MHz
SI 32768
SF 300.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.10
NH
ClO4
Br
N
N
NH
O
1p
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 ppm
0.985
1.000
1.019
2.021
1.075
1.046
1.036
1.018
1.097
2.145
2.049
1.041
2.172
2.956

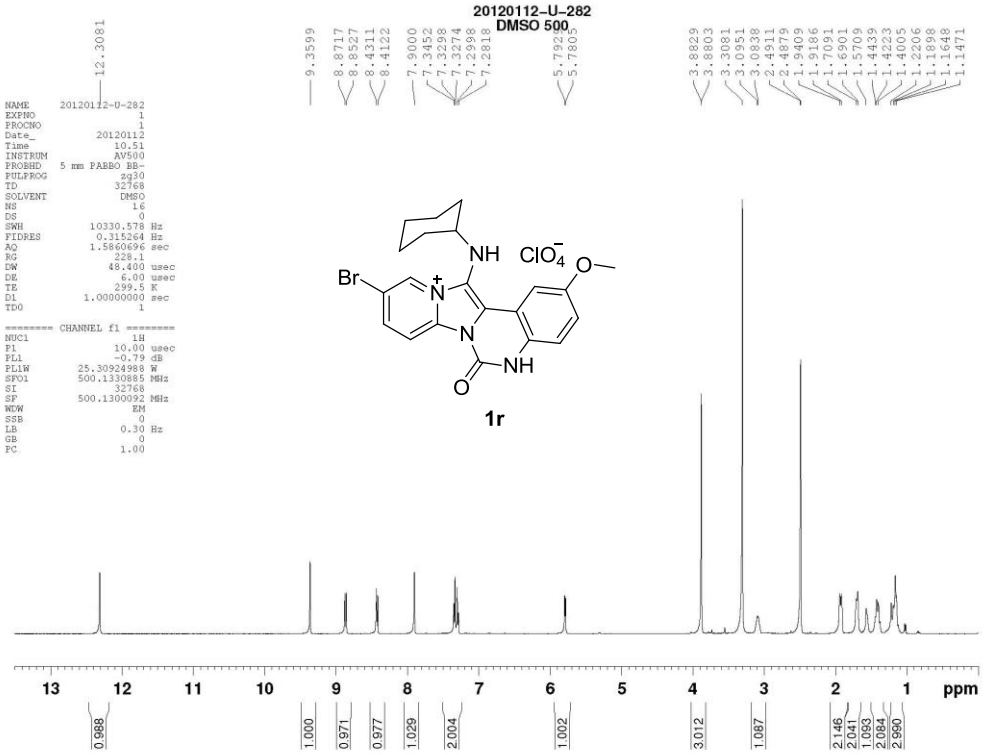

Avance 500, Bruker, SZPKU
20120109-U-182
DMSO
144.410
138.672
135.332
133.555
132.357
126.737
125.387
125.095
123.134
116.728
116.218
114.672
110.555
57.530
40.134
39.967
33.598
25.662
25.182
NAME 20120109-U-182
EXPNO 2
PROCNO 1
Date_ 20120109
Time 17.21
INSTRUM AV500
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 32768
SOLVENT DMSO
NS 285
DS 0
SWH 31446.541 H
FIDRES 0.959672 H
AQ 0.5210771 s
RG 11585.2
DW 15.900 u
DE 6.00 u
TE 300.2 K
D1 2.00000000 s
D11 0.03000000 s
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 u
PL1 3.00 d
PL1W 58.41413879 W
SFO1 125.7709936 M
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 u
PL2 -0.79 d
PL12 17.27 d
PL2W 25.30924988 W
PL12W 0.39562091 W
SFO2 500.1320005 M
NH
ClO4
Br
N
N
NH
O
1p
200 180 160 140 120 100 80 60 40 20 ppm

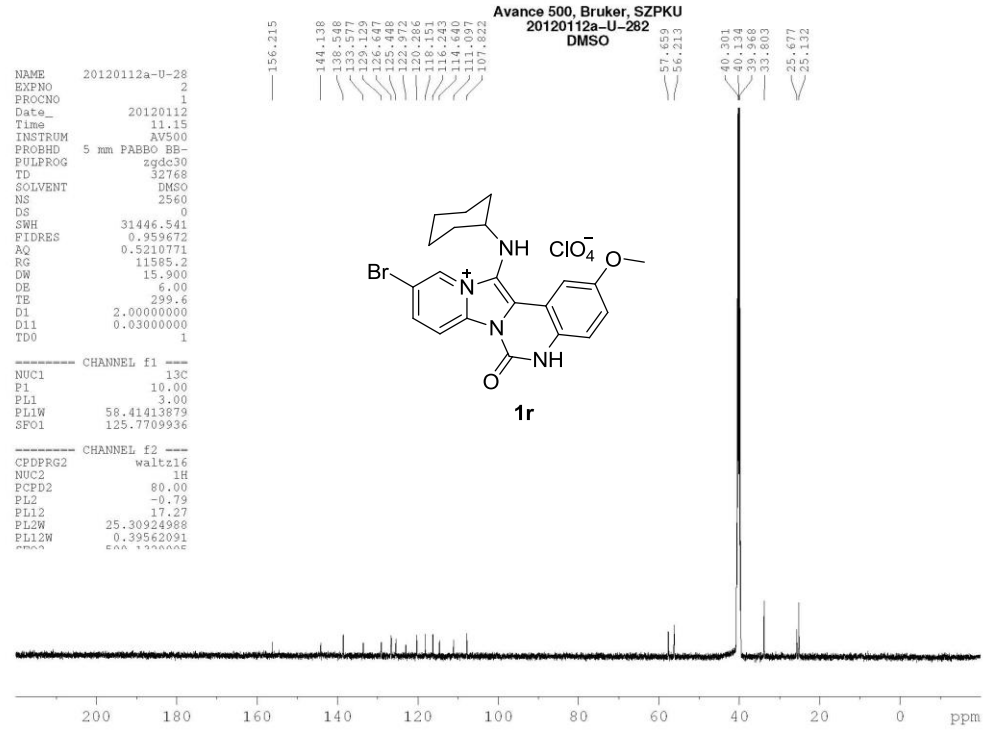
1q


20111229-U-242
SOLVENT:DMSO
NAME 20111229-U-242
EXPNO 1
PROCNO 1
Date_ 20111229
Time 17.15
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 1149.4
DW 83.400 usec
DE 6.00 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 300.1322510 MHz
SI 32768
SF 300.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.10
NH
ClO4−
Cl
O
N
N
NH
O
1q
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
.925
.000
.050
.063
.035
.107
.060
.064
.189
.167
.042
.099
.746

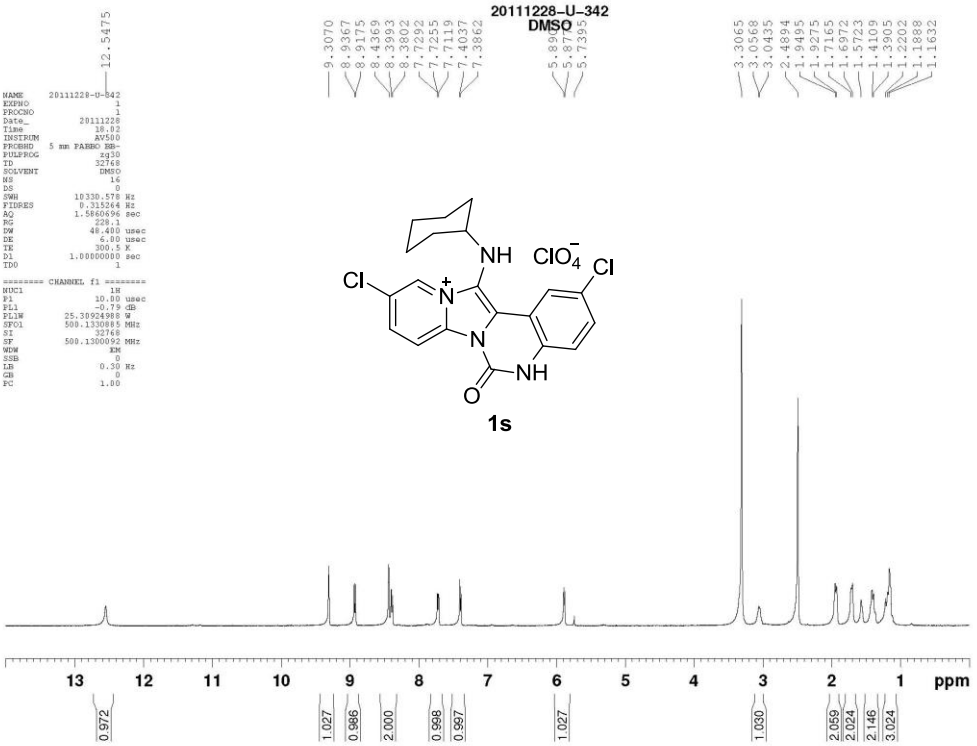

20111229-U-242
DMSO
NAME 20111229-U-242
EXPNO 2
PROCNO 1
Date_ 20111229
Time 17.23
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgdc30
TD 32768
SOLVENT DMSO
NS 3018
DS 0
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 71.8
DW 27.600 usec
DE 6.00 usec
TE 293.7 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.90 usec
PL1 -1.10 dB
PL1W 38.18405151 W
SFO1 75.4756731 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.80 dB
PL2W 8.31434441 W
PL12W 0.21868952 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677521 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
156.043
144.094
136.153
133.247
129.032
125.595
124.704
123.043
120.858
118.085
116.107
111.000
107.554
57.545
56.088
40.185
39.907
39.628
33.699
25.615
25.068
NH
ClO4
O
Cl
N
N
NH
O
1q
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

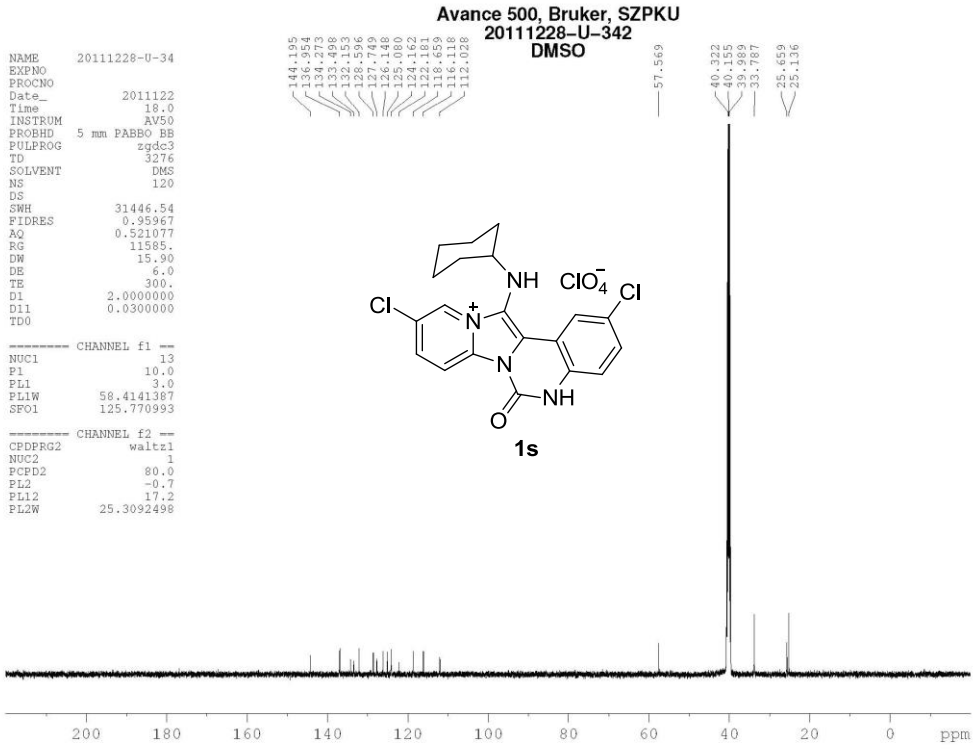
1r


20120112-U-282
DMSO 500
1r
ppm

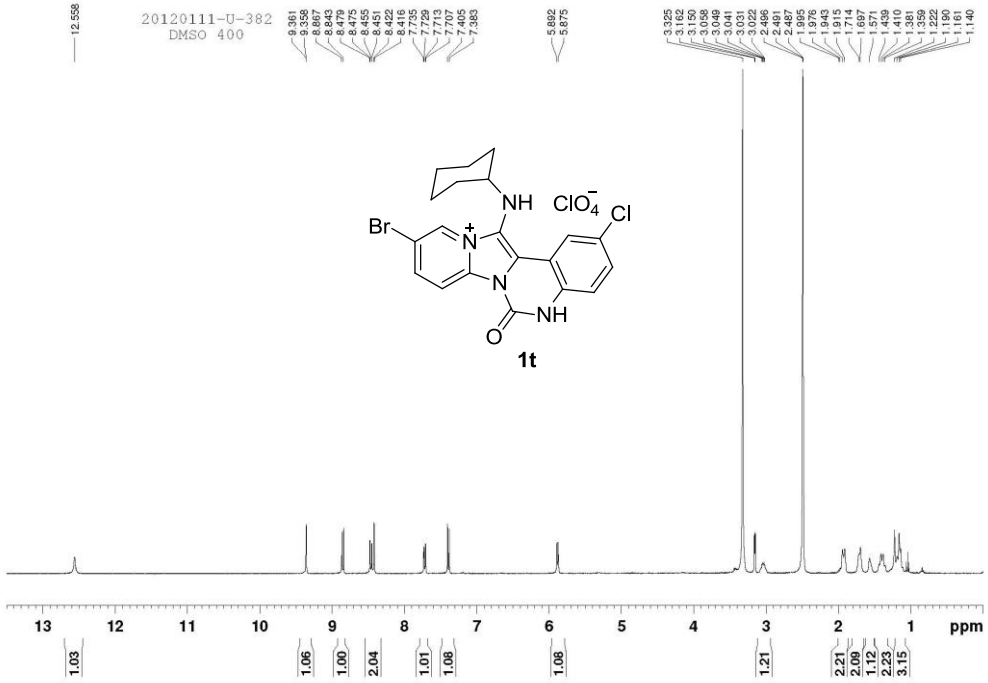

Avance 500, Bruker, SZPKU
20120112a-U-282
DMSO
1r
ppm

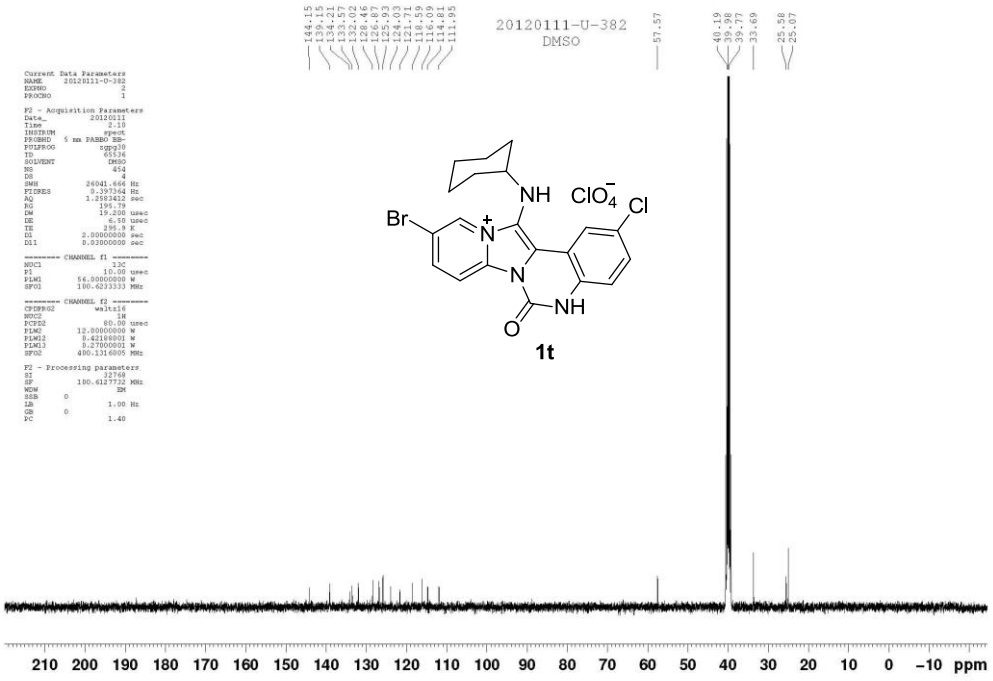
1s


20111228-U-342
DMSO
1s
ppm

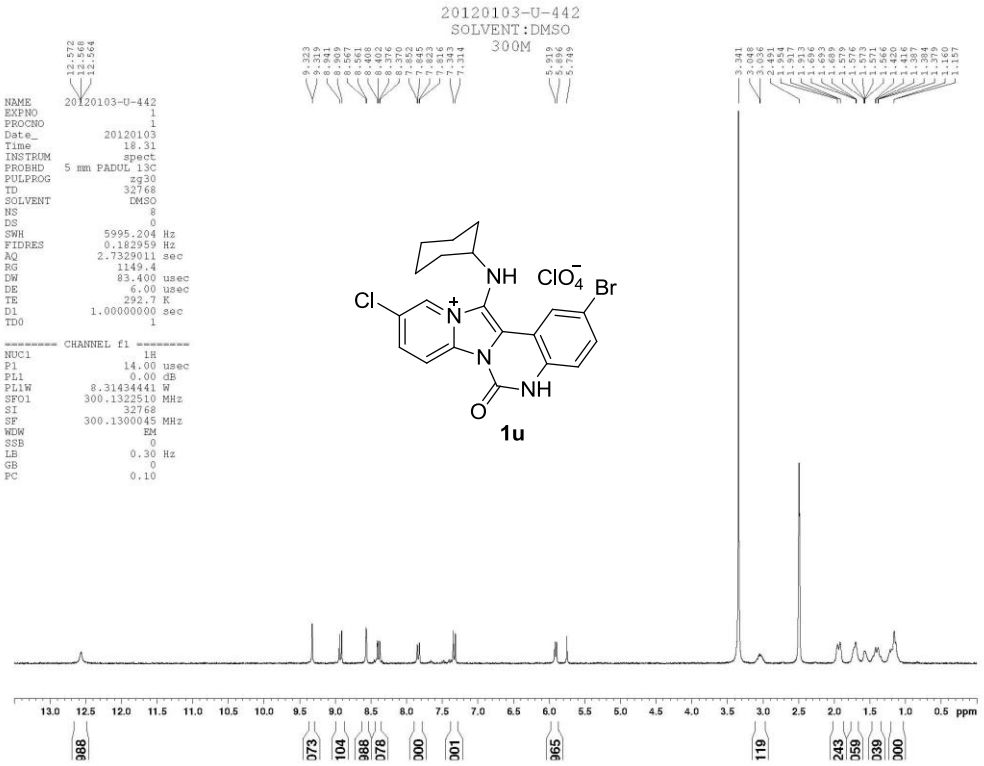

Avance 500, Bruker, SZPKU
20111228-U-342
DMSO
1s
ppm

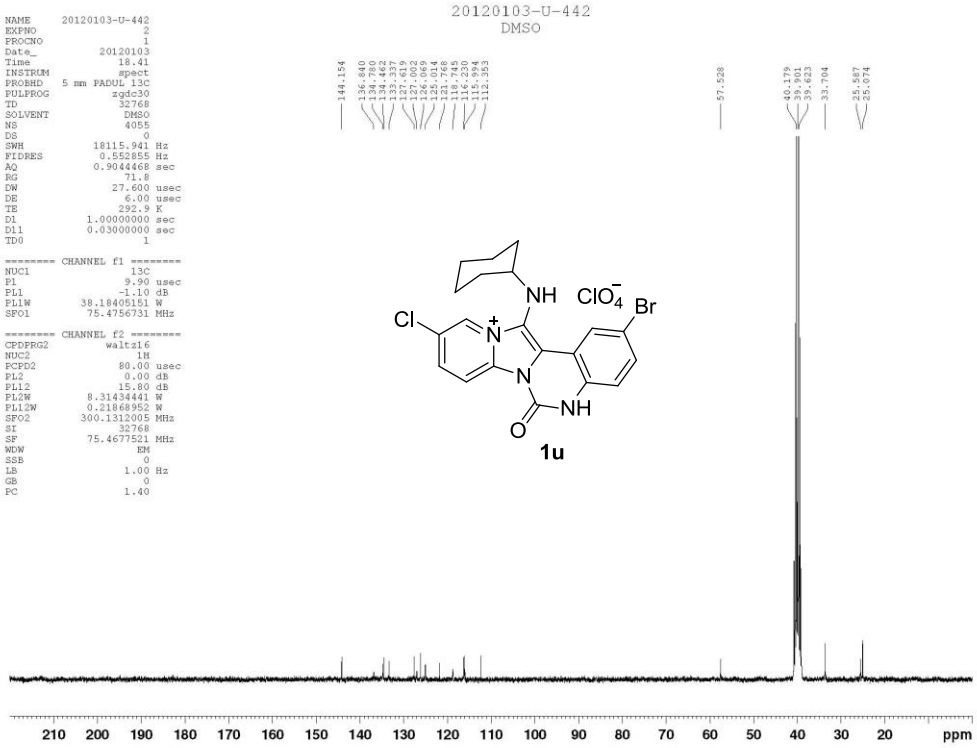
1t


20120111-U-382
DMSO 400
12.558
9.361
9.358
8.847
8.845
8.479
8.475
8.455
8.451
8.422
8.416
7.735
7.729
7.713
7.707
7.405
7.383
5.892
5.875
3.325
3.152
3.150
3.058
3.049
3.041
3.031
3.022
2.496
2.491
2.487
1.995
1.976
1.943
1.915
1.714
1.697
1.571
1.439
1.410
1.381
1.359
1.222
1.190
1.161
1.140
NH
ClO4−
Cl
Br
N+
N
NH
O
1t
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.03
1.06
1.00
2.04
1.01
1.08
1.08
1.21
2.21
2.09
1.12
2.23
3.15

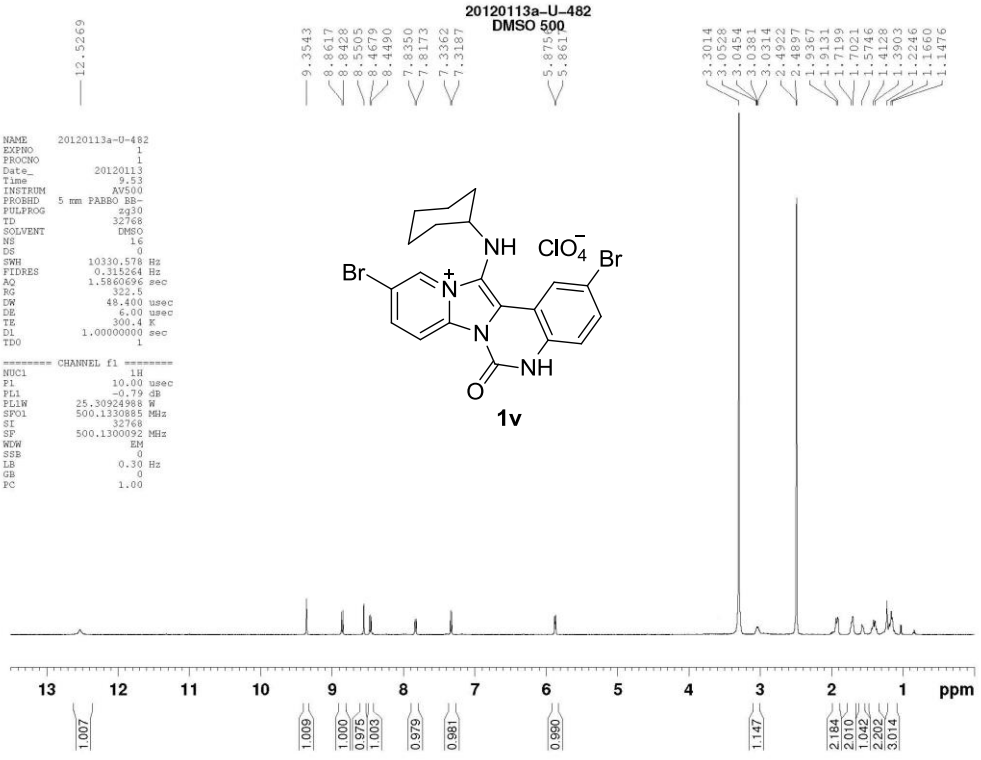

144.15
143.15
134.21
133.57
132.02
128.46
126.87
125.93
124.03
121.71
118.59
116.09
114.81
111.95
20120111-U-382
DMSO
57.57
40.19
39.98
39.77
33.69
25.58
25.07
Current Data Parameters
NAME 20120111-U-382
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120111
Time 2.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 454
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583422 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42189901 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127732 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
NH
ClO4−
Cl
Br
N+
N
NH
O
1t
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 ppm

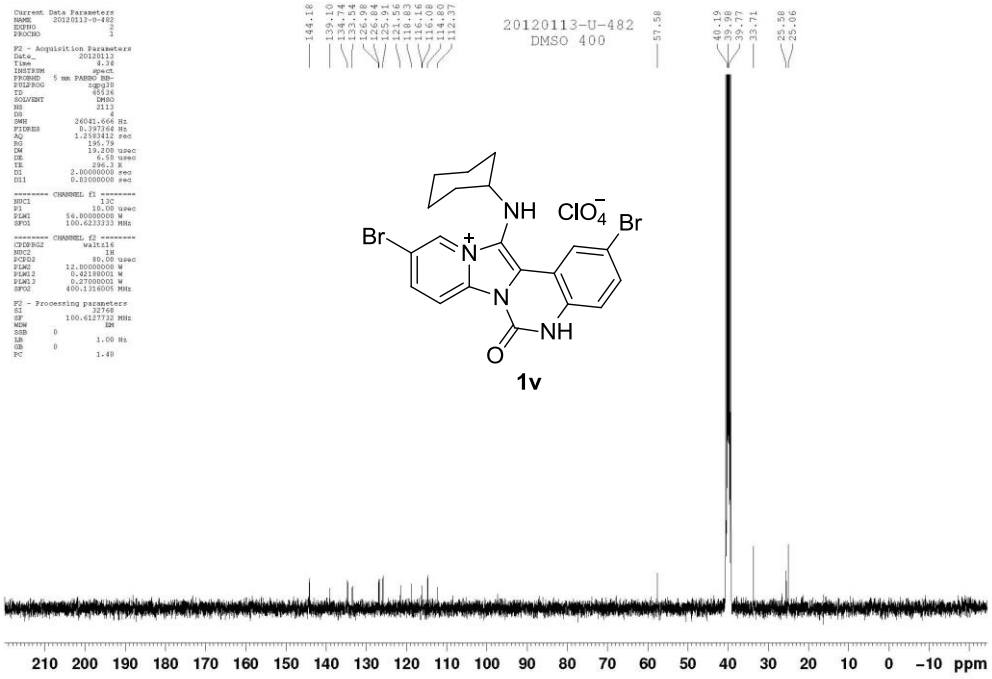
1u


20120103-U-442
SOLVENT:DMSO
300M
NAME 20120103-U-442
EXPNO 1
PROCNO 1
Date_ 20120103
Time 18.31
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 1149.4
DW 83.400 usec
DE 6.00 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 300.1322510 MHz
SI 32768
SF 300.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.10
NH
ClO4
Br
Cl
N
N
NH
O
1u
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

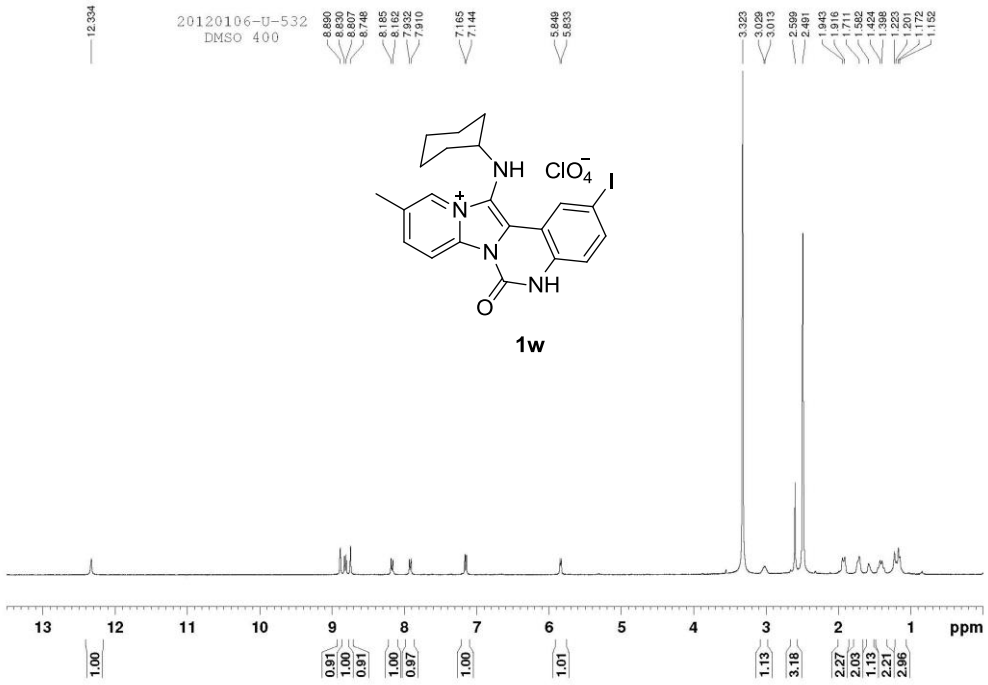

20120103-U-442
DMSO
NAME 20120103-U-442
EXPNO 2
PROCNO 1
Date_ 20120103
Time 18.41
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgdc30
TD 32768
SOLVENT DMSO
NS 4055
DS 0
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 71.8
DW 27.600 usec
DE 6.00 usec
TE 292.9 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.90 usec
PL1 -1.10 dB
PL1W 38.18405151 W
SFO1 75.4756731 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.80 dB
PL2W 8.31434441 W
PL12W 0.21868952 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677521 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
144.154
136.840
134.780
133.462
131.337
127.619
127.002
126.004
121.768
118.745
116.280
115.994
112.393
57.528
40.179
39.901
39.623
33.704
25.587
25.074
NH
ClO4
Br
Cl
N
N
NH
O
1u
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

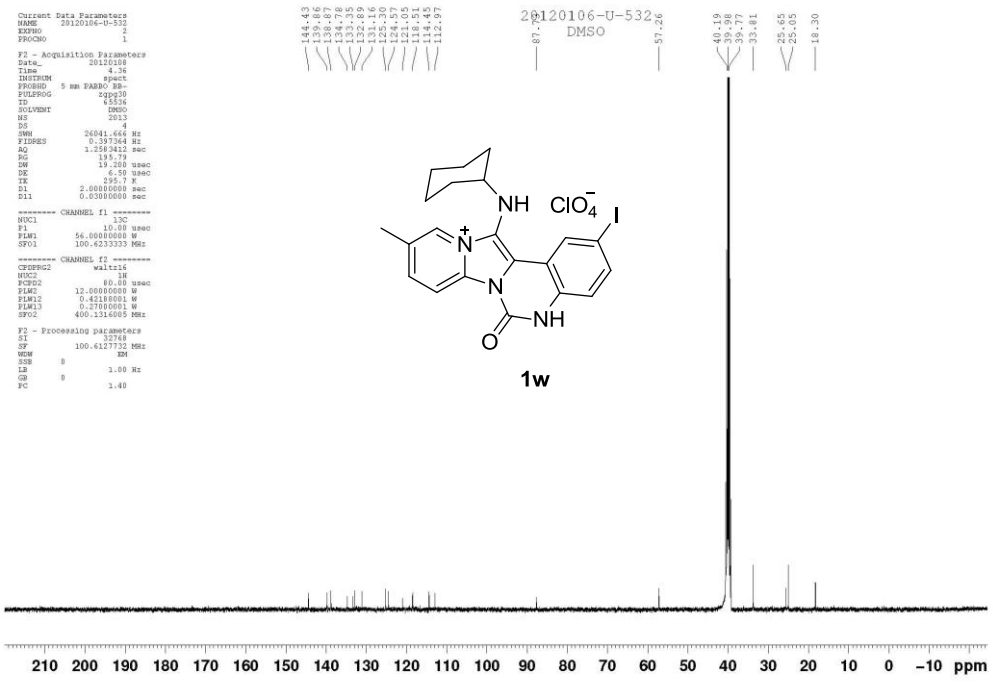
1v


20120113a-U-482
DMSO 500
12.5269
9.3543
8.8617
8.8428
8.5505
8.4679
8.4490
7.8350
7.8173
7.3362
7.3187
5.8754
5.8611
3.3014
3.0528
3.0454
3.0381
3.0314
2.4922
2.4897
1.9367
1.9131
1.7199
1.7021
1.5746
1.4128
1.3903
1.2246
1.1660
1.1476
NAME 20120113a-U-482
EXPNO 1
PROCNO 1
Date_ 20120113
Time 9.53
INSTRUM AV500
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 322.5
DW 48.400 usec
DE 6.00 usec
TE 300.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -0.79 dB
PL1W 25.30924988 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NH
ClO4
Br
Br
N
N
NH
O
1v
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.007
1.009
1.000
0.975
1.003
0.979
0.981
0.990
1.147
2.184
2.010
1.042
2.202
3.014

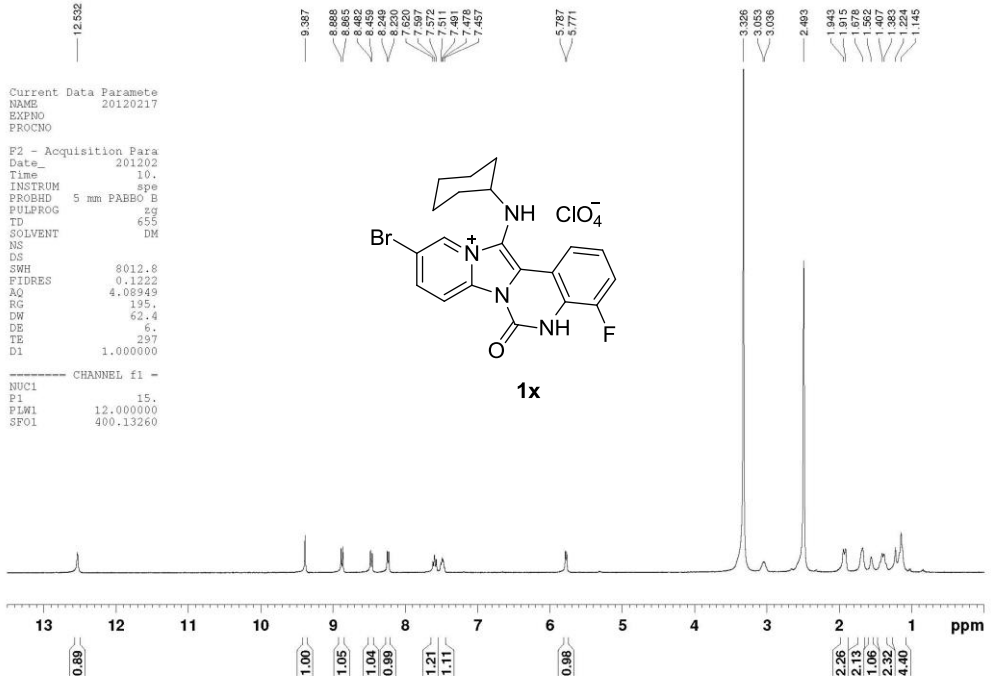

20120113-U-482
DMSO 400
144.18
139.10
134.74
133.54
126.84
125.91
122.56
116.03
115.56
115.08
114.80
112.37
57.58
40.19
39.98
39.77
33.71
25.58
25.06
NH
ClO4
Br
Br
N
N
NH
O
1v
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 ppm

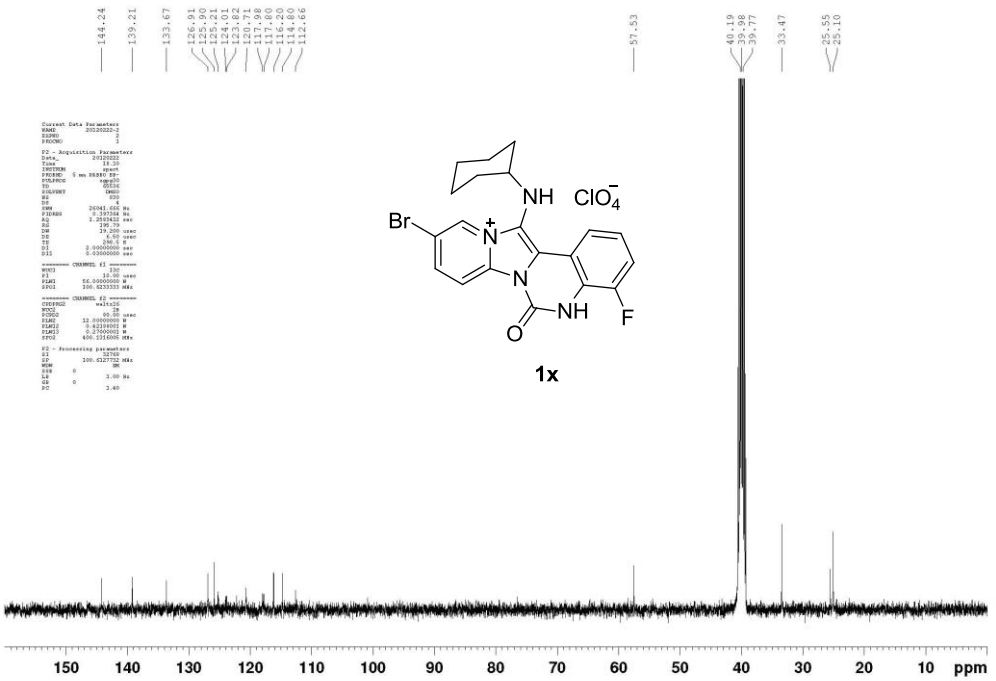
1w


20120106-U-532
DMSO 400
12.334
8.890
8.830
8.807
8.748
8.185
8.162
7.932
7.910
7.165
7.144
5.849
5.833
3.323
3.029
3.013
2.599
2.491
1.943
1.916
1.771
1.582
1.424
1.398
1.223
1.201
1.172
1.152
NH
ClO4
I
N
N
NH
O
1w
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
0.91
1.00
0.91
1.00
0.97
1.00
1.01
1.13
3.18
2.27
2.03
1.13
2.21
2.96

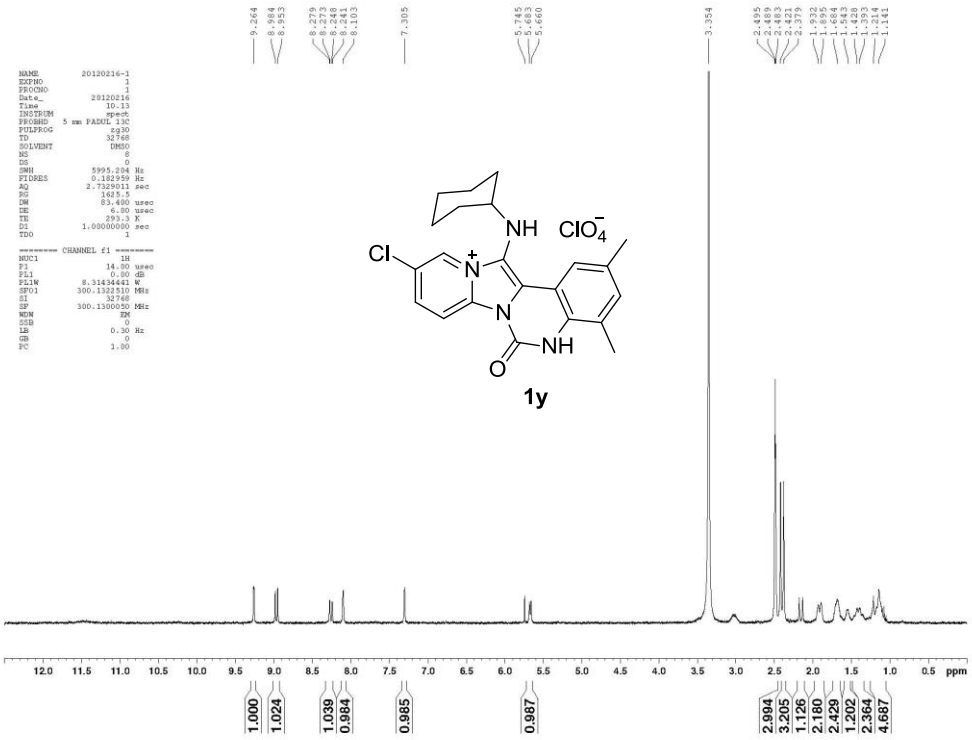

Current Data Parameters
NAME 20120106-U-532
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120108
Time 4.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2013
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 199.79
DW 19.200 usec
DE 6.50 usec
TE 295.7 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42180001 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127732 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
144.43
139.66
138.87
134.78
133.85
132.88
131.16
125.30
124.57
121.05
118.53
114.45
112.97
87.76
57.26
40.19
39.98
39.77
33.01
25.65
25.05
18.30
20120106-U-532
DMSO
NH
ClO4
I
N
N
NH
O
1w
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm

1x


12.532
9.387
8.888
8.865
8.492
8.456
8.249
8.230
7.620
7.597
7.572
7.511
7.491
7.478
7.457
5.787
5.771
3.326
3.053
3.036
2.493
1.943
1.915
1.678
1.562
1.407
1.383
1.264
1.145
Current Data Paramete
NAME 20120217
EXPNO
PROCNO
P2 - Acquisition Para
Date_ 201202
Time 10.
INSTRUM spe
PROBHD 5 mm PABBO B
PULPROG zg
TD 655
SOLVENT DM
NS
DS
SWH 8012.8
FIDRES 0.1222
AQ 4.08949
RG 195.
DW 62.4
DE 6.
TE 297
D1 1.000000
======== CHANNEL f1 =
NUC1
P1 15.
PLW1 12.000000
SFO1 400.13260
NH
ClO4
Br
N
N
NH
F
O
1x
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.89
1.00
1.05
1.04
0.99
1.21
1.11
0.98
2.26
2.13
1.06
2.32
4.40


144.24
139.21
133.67
126.91
125.90
125.21
124.01
123.72
120.71
117.98
117.80
116.20
116.00
112.66
57.53
40.19
39.98
39.77
33.47
25.55
25.10
NH
ClO4
Br
N
N
NH
F
O
1x
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

1y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2.495
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DW 83.400 usec
DE 6.50 usec
TE 293.3 K
D1 1.00000000 sec
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SFO1 300.1322510 MHz
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SP 300.1300050 MHz
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LB 0.30 Hz
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NH
ClO4
Cl
N
N
NH
O
1y
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1.024
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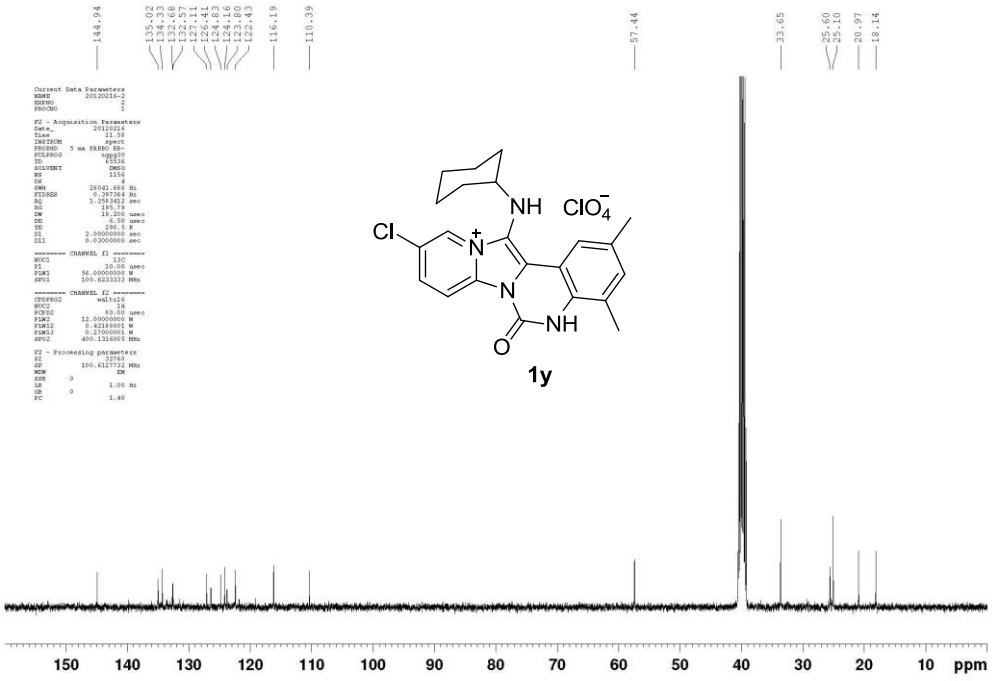
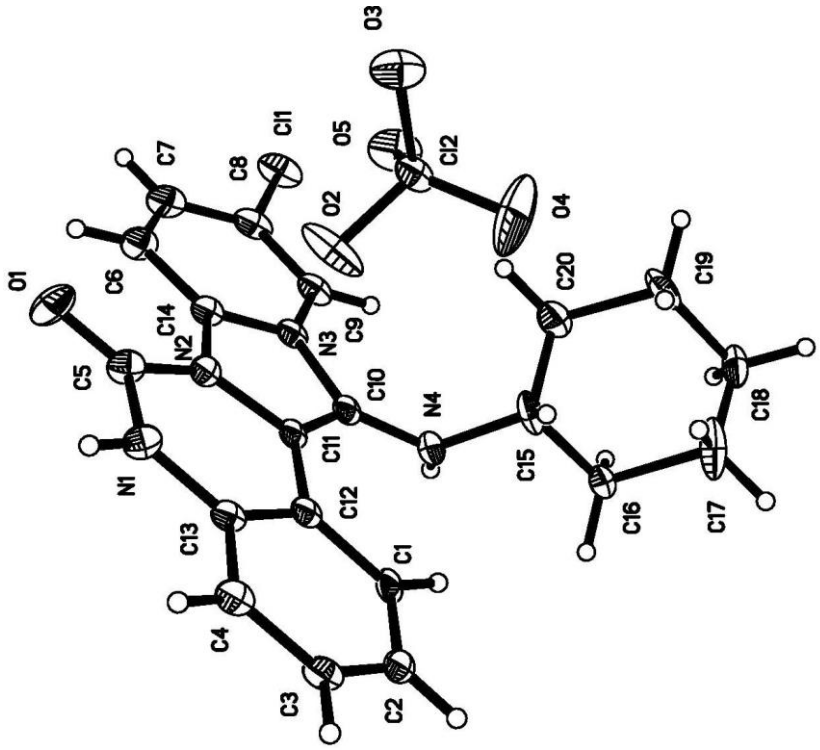


X-ray data for compound 1o



ORTEP structure for compound **1o**

Table 1. Crystal data and structure refinement for compound **1o**.

Identification code	03071a	
Empirical formula	C20 H20 Cl2 N4 O5	
Formula weight	467.30	
Temperature	150(2) K	
Wavelength	1.54184 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 9.9958(2) Å	α= 90°.
	b = 15.0623(3) Å	β= 119.167(2)°.
	c = 15.2761(4) Å	γ = 90°.
Volume	2008.34(8) $Å^3$	
Z	4	
Density (calculated)	1.545 Mg/m^3	
Absorption coefficient	3.287 mm^{-1}	
F(000)	968	
Crystal size	0.38 x 0.34 x 0.25 mm^3	
Theta range for data collection	4.43 to 66.86°.	
Index ranges	-11<=h<=11, -14<=k<=17, -18<=l<=18	
Reflections collected	10354	
Independent reflections	3542 [R(int) = 0.0349]	
Completeness to theta = 66.86°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.4937 and 0.3681	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3542 / 24 / 366	
Goodness-of-fit on F^2	1.095	
Final R indices [I>2sigma(I)]	R1 = 0.0535, wR2 = 0.1350	

R indices (all data)	R1 = 0.0616, wR2 = 0.1439
Largest diff. peak and hole	0.346 and -0.858 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for compound **1o**.

U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2496(3)	10243(2)	3302(2)	32(1)
C(2)	1349(3)	10548(2)	3488(2)	36(1)
C(3)	1150(3)	10162(2)	4237(2)	35(1)
C(4)	2082(3)	9481(2)	4818(2)	32(1)
C(5)	5412(3)	8140(2)	5161(2)	32(1)
C(6)	8023(3)	7772(2)	4638(2)	37(1)
C(7)	8947(3)	7710(2)	4208(2)	40(1)
C(8)	8624(3)	8197(2)	3346(2)	36(1)
C(9)	7418(3)	8771(2)	2929(2)	31(1)
C(10)	5204(3)	9391(2)	3079(2)	27(1)
C(11)	4690(3)	9203(2)	3737(2)	25(1)
C(12)	3452(3)	9551(2)	3875(2)	27(1)
C(13)	3254(3)	9173(2)	4639(2)	27(1)
C(14)	6757(3)	8335(2)	4174(2)	28(1)
C(15)	3453(3)	9754(2)	1300(2)	37(1)
C(16)	3218(13)	10672(5)	657(4)	52(3)
C(17)	1349(10)	10290(7)	-336(5)	61(2)
C(18)	1947(13)	9871(8)	-909(9)	43(3)
C(19)	2333(14)	8940(5)	-272(5)	52(2)
C(20)	3987(16)	9194(9)	737(11)	44(3)
C(15B)	3453(3)	9754(2)	1300(2)	37(1)
C(16B)	2551(11)	10483(6)	753(6)	39(2)
C(17B)	1916(14)	10566(7)	-422(6)	49(3)

C(18B)	2252(17)	9620(9)	-859(12)	35(3)
C(19B)	2963(13)	8914(11)	-311(9)	64(4)
C(20B)	3830(20)	9007(10)	797(14)	50(5)
Cl(1)	9776(1)	8073(1)	2807(1)	47(1)
N(1)	4211(3)	8495(2)	5223(2)	31(1)
N(2)	5631(2)	8553(1)	4400(2)	26(1)
N(3)	6496(2)	8830(1)	3364(2)	26(1)
N(4)	4695(3)	9980(2)	2310(2)	35(1)
O(1)	6240(2)	7555(1)	5675(2)	46(1)
Cl(2)	2792(1)	7038(1)	2155(1)	33(1)
O(2)	3053(4)	7401(3)	3097(3)	72(1)
O(3)	2480(5)	6082(3)	2161(4)	50(1)
O(4)	1580(5)	7456(4)	1388(4)	106(2)
O(5)	4181(3)	7094(2)	2121(3)	49(1)
Cl(2B)	2792(1)	7038(1)	2155(1)	33(1)
O(2B)	4164(18)	7195(10)	2911(11)	78(5)
O(3B)	2014(19)	6292(10)	1917(13)	39(4)
O(4B)	1700(20)	7652(12)	2041(18)	93(7)
O(5B)	2822(17)	7331(7)	1133(9)	58(4)

Table 3. Bond lengths [Å] and angles [°] for compound **1o**.

C(1)-C(2)	1.387(4)
C(1)-C(12)	1.397(4)
C(1)-H(1)	0.9500
C(2)-C(3)	1.382(4)
C(2)-H(2)	0.9500
C(3)-C(4)	1.379(4)
C(3)-H(3)	0.9500
C(4)-C(13)	1.404(3)
C(4)-H(4)	0.9500
C(5)-O(1)	1.203(3)
C(5)-N(1)	1.358(3)
C(5)-N(2)	1.426(3)
C(6)-C(7)	1.373(4)
C(6)-C(14)	1.395(4)
C(6)-H(6)	0.9500
C(7)-C(8)	1.400(5)
C(7)-H(7)	0.9500
C(8)-C(9)	1.363(4)
C(8)-Cl(1)	1.723(3)
C(9)-N(3)	1.377(3)
C(9)-H(9)	0.9500
C(10)-N(4)	1.359(4)
C(10)-C(11)	1.361(4)
C(10)-N(3)	1.423(3)
C(11)-N(2)	1.394(3)
C(11)-C(12)	1.450(3)
C(12)-C(13)	1.397(4)

C(13)-N(1)	1.387(4)
C(14)-N(3)	1.357(4)
C(14)-N(2)	1.368(3)
C(15)-N(4)	1.473(3)
C(15)-C(20)	1.479(14)
C(15)-C(16)	1.645(9)
C(15)-H(15A)	1.0000
C(16)-C(17)	1.833(14)
C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900
C(17)-C(18)	1.425(15)
C(17)-H(17A)	0.9900
C(17)-H(17B)	0.9900
C(18)-C(19)	1.641(15)
C(18)-H(18A)	0.9900
C(18)-H(18B)	0.9900
C(19)-C(20)	1.664(17)
C(19)-H(19A)	0.9900
C(19)-H(19B)	0.9900
C(20)-H(20A)	0.9900
C(20)-H(20B)	0.9900
C(16B)-C(17B)	1.591(12)
C(16B)-H(16C)	0.9900
C(16B)-H(16D)	0.9900
C(17B)-C(18B)	1.675(16)
C(17B)-H(17C)	0.9900
C(17B)-H(17D)	0.9900
C(18B)-C(19B)	1.326(19)
C(18B)-H(18C)	0.9900

C(18B)-H(18D)	0.9900
C(19B)-C(20B)	1.48(2)
C(19B)-H(19C)	0.9900
C(19B)-H(19D)	0.9900
C(20B)-H(20C)	0.9900
C(20B)-H(20D)	0.9900
N(1)-H(1A)	0.84(3)
N(4)-H(4A)	0.7692
Cl(2)-O(4)	1.362(4)
Cl(2)-O(5)	1.417(2)
Cl(2)-O(2)	1.439(3)
Cl(2)-O(3)	1.474(4)

C(2)-C(1)-C(12)	120.0(3)
C(2)-C(1)-H(1)	120.0
C(12)-C(1)-H(1)	120.0
C(3)-C(2)-C(1)	119.9(3)
C(3)-C(2)-H(2)	120.1
C(1)-C(2)-H(2)	120.1
C(4)-C(3)-C(2)	121.4(2)
C(4)-C(3)-H(3)	119.3
C(2)-C(3)-H(3)	119.3
C(3)-C(4)-C(13)	119.1(3)
C(3)-C(4)-H(4)	120.5
C(13)-C(4)-H(4)	120.5
O(1)-C(5)-N(1)	126.4(3)
O(1)-C(5)-N(2)	121.4(2)
N(1)-C(5)-N(2)	112.2(2)
C(7)-C(6)-C(14)	117.0(3)

C(7)-C(6)-H(6)	121.5
C(14)-C(6)-H(6)	121.5
C(6)-C(7)-C(8)	120.9(3)
C(6)-C(7)-H(7)	119.6
C(8)-C(7)-H(7)	119.6
C(9)-C(8)-C(7)	121.3(2)
C(9)-C(8)-Cl(1)	119.3(2)
C(7)-C(8)-Cl(1)	119.4(2)
C(8)-C(9)-N(3)	117.5(3)
C(8)-C(9)-H(9)	121.2
N(3)-C(9)-H(9)	121.2
N(4)-C(10)-C(11)	131.1(2)
N(4)-C(10)-N(3)	123.6(2)
C(11)-C(10)-N(3)	105.3(2)
C(10)-C(11)-N(2)	108.6(2)
C(10)-C(11)-C(12)	133.4(2)
N(2)-C(11)-C(12)	118.0(2)
C(1)-C(12)-C(13)	119.7(2)
C(1)-C(12)-C(11)	123.4(2)
C(13)-C(12)-C(11)	116.9(2)
N(1)-C(13)-C(12)	120.2(2)
N(1)-C(13)-C(4)	119.8(2)
C(12)-C(13)-C(4)	120.0(2)
N(3)-C(14)-N(2)	106.4(2)
N(3)-C(14)-C(6)	121.4(2)
N(2)-C(14)-C(6)	132.2(3)
N(4)-C(15)-C(20)	112.5(6)
N(4)-C(15)-C(16)	103.5(3)
C(20)-C(15)-C(16)	96.9(7)

N(4)-C(15)-H(15A)	114.1
C(20)-C(15)-H(15A)	114.1
C(16)-C(15)-H(15A)	114.1
C(15)-C(16)-C(17)	91.3(7)
C(15)-C(16)-H(16A)	113.4
C(17)-C(16)-H(16A)	113.4
C(15)-C(16)-H(16B)	113.4
C(17)-C(16)-H(16B)	113.4
H(16A)-C(16)-H(16B)	110.7
C(18)-C(17)-C(16)	94.8(8)
C(18)-C(17)-H(17A)	112.8
C(16)-C(17)-H(17A)	112.8
C(18)-C(17)-H(17B)	112.8
C(16)-C(17)-H(17B)	112.8
H(17A)-C(17)-H(17B)	110.2
C(17)-C(18)-C(19)	93.5(10)
C(17)-C(18)-H(18A)	113.0
C(19)-C(18)-H(18A)	113.0
C(17)-C(18)-H(18B)	113.0
C(19)-C(18)-H(18B)	113.0
H(18A)-C(18)-H(18B)	110.4
C(18)-C(19)-C(20)	101.1(8)
C(18)-C(19)-H(19A)	111.5
C(20)-C(19)-H(19A)	111.5
C(18)-C(19)-H(19B)	111.5
C(20)-C(19)-H(19B)	111.5
H(19A)-C(19)-H(19B)	109.4
C(15)-C(20)-C(19)	101.1(9)
C(15)-C(20)-H(20A)	111.6

C(19)-C(20)-H(20A)	111.6
C(15)-C(20)-H(20B)	111.6
C(19)-C(20)-H(20B)	111.6
H(20A)-C(20)-H(20B)	109.4
C(17B)-C(16B)-H(16C)	107.0
C(17B)-C(16B)-H(16D)	107.0
H(16C)-C(16B)-H(16D)	106.7
C(16B)-C(17B)-C(18B)	109.6(9)
C(16B)-C(17B)-H(17C)	109.7
C(18B)-C(17B)-H(17C)	109.7
C(16B)-C(17B)-H(17D)	109.7
C(18B)-C(17B)-H(17D)	109.7
H(17C)-C(17B)-H(17D)	108.2
C(19B)-C(18B)-C(17B)	125.4(13)
C(19B)-C(18B)-H(18C)	106.0
C(17B)-C(18B)-H(18C)	106.0
C(19B)-C(18B)-H(18D)	106.0
C(17B)-C(18B)-H(18D)	106.0
H(18C)-C(18B)-H(18D)	106.3
C(18B)-C(19B)-C(20B)	118.8(13)
C(18B)-C(19B)-H(19C)	107.6
C(20B)-C(19B)-H(19C)	107.6
C(18B)-C(19B)-H(19D)	107.6
C(20B)-C(19B)-H(19D)	107.6
H(19C)-C(19B)-H(19D)	107.1
C(19B)-C(20B)-H(20C)	107.4
C(19B)-C(20B)-H(20D)	107.4
H(20C)-C(20B)-H(20D)	106.9
C(5)-N(1)-C(13)	127.0(2)

C(5)-N(1)-H(1A)	113(2)
C(13)-N(1)-H(1A)	120(2)
C(14)-N(2)-C(11)	109.4(2)
C(14)-N(2)-C(5)	125.0(2)
C(11)-N(2)-C(5)	125.6(2)
C(14)-N(3)-C(9)	121.9(2)
C(14)-N(3)-C(10)	110.3(2)
C(9)-N(3)-C(10)	127.8(2)
C(10)-N(4)-C(15)	121.6(2)
C(10)-N(4)-H(4A)	120.6
C(15)-N(4)-H(4A)	112.4
O(4)-Cl(2)-O(5)	114.7(3)
O(4)-Cl(2)-O(2)	109.6(4)
O(5)-Cl(2)-O(2)	108.07(19)
O(4)-Cl(2)-O(3)	110.8(3)
O(5)-Cl(2)-O(3)	105.9(2)
O(2)-Cl(2)-O(3)	107.5(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for compound **1o**.

The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ \ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	18(1)	50(2)	20(1)	-8(1)	1(1)	7(1)
C(2)	18(1)	49(2)	28(1)	-10(1)	1(1)	11(1)
C(3)	16(1)	47(2)	38(2)	-17(1)	11(1)	1(1)
C(4)	16(1)	42(2)	35(1)	-12(1)	11(1)	-2(1)
C(5)	24(1)	35(1)	40(2)	-2(1)	18(1)	2(1)
C(6)	21(1)	42(2)	44(2)	-8(1)	13(1)	6(1)
C(7)	19(1)	44(2)	52(2)	-13(1)	14(1)	3(1)
C(8)	17(1)	45(2)	47(2)	-22(1)	16(1)	-8(1)
C(9)	20(1)	42(2)	33(1)	-16(1)	14(1)	-9(1)
C(10)	15(1)	35(1)	23(1)	-10(1)	3(1)	0(1)
C(11)	13(1)	33(1)	21(1)	-10(1)	2(1)	0(1)
C(12)	12(1)	38(1)	25(1)	-13(1)	3(1)	1(1)
C(13)	15(1)	33(1)	30(1)	-11(1)	8(1)	-2(1)
C(14)	14(1)	34(1)	31(1)	-10(1)	8(1)	-1(1)
C(15)	20(1)	71(2)	18(1)	-8(1)	7(1)	-4(1)
C(16)	71(6)	46(4)	20(3)	-4(2)	7(3)	16(4)
C(17)	38(4)	104(7)	26(3)	21(4)	4(3)	-13(4)
C(18)	39(5)	54(6)	22(3)	5(4)	4(3)	-2(4)
C(19)	65(6)	50(4)	27(3)	-19(3)	11(4)	-20(4)
C(20)	53(4)	47(5)	32(4)	-6(4)	20(3)	-1(4)
C(15B)	20(1)	71(2)	18(1)	-8(1)	7(1)	-4(1)
C(16B)	29(4)	61(5)	21(3)	11(3)	6(3)	-10(3)
C(17B)	53(6)	57(6)	24(4)	-3(4)	9(4)	4(4)
C(18B)	35(5)	43(5)	23(4)	-4(4)	11(4)	-2(4)

C(19B)	28(5)	129(11)	40(5)	-43(6)	20(4)	-22(6)
C(20B)	89(9)	24(5)	26(5)	-6(4)	20(5)	-29(5)
Cl(1)	27(1)	59(1)	64(1)	-28(1)	30(1)	-10(1)
N(1)	24(1)	34(1)	41(1)	0(1)	19(1)	3(1)
N(2)	14(1)	34(1)	27(1)	-5(1)	7(1)	2(1)
N(3)	14(1)	35(1)	27(1)	-13(1)	8(1)	-3(1)
N(4)	31(1)	39(1)	24(1)	-5(1)	5(1)	0(1)
O(1)	39(1)	43(1)	65(1)	19(1)	33(1)	19(1)
Cl(2)	19(1)	42(1)	36(1)	-15(1)	13(1)	-6(1)
O(2)	82(3)	93(3)	64(2)	-55(2)	53(2)	-48(2)
O(3)	38(2)	39(2)	79(3)	-11(2)	34(2)	-11(2)
O(4)	41(2)	145(5)	91(4)	58(4)	-1(2)	20(3)
O(5)	37(2)	54(2)	78(2)	-11(2)	46(2)	-9(1)
Cl(2B)	19(1)	42(1)	36(1)	-15(1)	13(1)	-6(1)
O(2B)	72(6)	67(6)	68(6)	-8(4)	12(4)	-10(4)
O(3B)	39(6)	28(5)	45(5)	-5(4)	17(4)	-6(4)
O(4B)	113(15)	87(11)	120(16)	24(11)	89(14)	66(10)
O(5B)	113(12)	39(6)	60(8)	6(5)	72(8)	3(6)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for compound **1o**.

	x	y	z	U(eq)
H(1)	2630	10505	2784	39
H(2)	702	11021	3102	43
H(3)	354	10370	4354	42
H(4)	1933	9223	5331	38
H(6)	8236	7447	5225	44
H(7)	9817	7331	4499	48
H(9)	7222	9118	2359	38
H(15A)	2499	9534	1285	45
H(16A)	3984	10749	430	62
H(16B)	3156	11212	1006	62
H(17A)	850	9866	-90	73
H(17B)	644	10785	-699	73
H(18A)	2873	10169	-845	52
H(18B)	1174	9798	-1624	52
H(19A)	2450	8439	-648	63
H(19B)	1532	8794	-92	63
H(20A)	4523	8656	1120	53
H(20B)	4671	9527	555	53
H(16C)	3157	11025	1065	47
H(16D)	1653	10501	863	47
H(17C)	2426	11067	-561	58
H(17D)	801	10686	-764	58
H(18C)	1240	9408	-1386	42
H(18D)	2830	9792	-1207	42

H(19C)	3683	8697	-534	77
H(19D)	2185	8444	-471	77
H(20C)	4930	9064	992	60
H(20D)	3720	8443	1088	60
H(1A)	4130(40)	8290(20)	5710(30)	38
H(4A)	5231	10341	2299	42

Table 6. Torsion angles [°] for compound **1o**.

C(12)-C(1)-C(2)-C(3)	-0.4(4)
C(1)-C(2)-C(3)-C(4)	0.8(4)
C(2)-C(3)-C(4)-C(13)	-0.2(4)
C(14)-C(6)-C(7)-C(8)	0.4(4)
C(6)-C(7)-C(8)-C(9)	2.4(4)
C(6)-C(7)-C(8)-Cl(1)	-177.9(2)
C(7)-C(8)-C(9)-N(3)	-2.4(4)
Cl(1)-C(8)-C(9)-N(3)	177.97(18)
N(4)-C(10)-C(11)-N(2)	-178.9(2)
N(3)-C(10)-C(11)-N(2)	0.1(3)
N(4)-C(10)-C(11)-C(12)	-0.7(5)
N(3)-C(10)-C(11)-C(12)	178.3(2)
C(2)-C(1)-C(12)-C(13)	-0.4(4)
C(2)-C(1)-C(12)-C(11)	-179.1(2)
C(10)-C(11)-C(12)-C(1)	-2.3(4)
N(2)-C(11)-C(12)-C(1)	175.7(2)
C(10)-C(11)-C(12)-C(13)	178.9(3)
N(2)-C(11)-C(12)-C(13)	-3.0(3)
C(1)-C(12)-C(13)-N(1)	-179.1(2)
C(11)-C(12)-C(13)-N(1)	-0.3(3)
C(1)-C(12)-C(13)-C(4)	1.0(4)
C(11)-C(12)-C(13)-C(4)	179.7(2)
C(3)-C(4)-C(13)-N(1)	179.4(2)
C(3)-C(4)-C(13)-C(12)	-0.7(4)
C(7)-C(6)-C(14)-N(3)	-3.2(4)
C(7)-C(6)-C(14)-N(2)	-179.5(3)
N(4)-C(15)-C(16)-C(17)	-163.7(4)

C(20)-C(15)-C(16)-C(17)	81.1(8)
C(15)-C(16)-C(17)-C(18)	-90.7(9)
C(16)-C(17)-C(18)-C(19)	85.5(8)
C(17)-C(18)-C(19)-C(20)	-80.8(10)
N(4)-C(15)-C(20)-C(19)	172.4(5)
C(16)-C(15)-C(20)-C(19)	-79.9(9)
C(18)-C(19)-C(20)-C(15)	76.4(10)
C(16B)-C(17B)-C(18B)-C(19B)	-1(2)
C(17B)-C(18B)-C(19B)-C(20B)	-14(2)
O(1)-C(5)-N(1)-C(13)	179.6(3)
N(2)-C(5)-N(1)-C(13)	0.4(4)
C(12)-C(13)-N(1)-C(5)	1.7(4)
C(4)-C(13)-N(1)-C(5)	-178.3(3)
N(3)-C(14)-N(2)-C(11)	-1.3(3)
C(6)-C(14)-N(2)-C(11)	175.4(3)
N(3)-C(14)-N(2)-C(5)	175.4(2)
C(6)-C(14)-N(2)-C(5)	-7.9(4)
C(10)-C(11)-N(2)-C(14)	0.8(3)
C(12)-C(11)-N(2)-C(14)	-177.7(2)
C(10)-C(11)-N(2)-C(5)	-175.9(2)
C(12)-C(11)-N(2)-C(5)	5.6(3)
O(1)-C(5)-N(2)-C(14)	0.4(4)
N(1)-C(5)-N(2)-C(14)	179.7(2)
O(1)-C(5)-N(2)-C(11)	176.6(3)
N(1)-C(5)-N(2)-C(11)	-4.1(4)
N(2)-C(14)-N(3)-C(9)	-179.6(2)
C(6)-C(14)-N(3)-C(9)	3.3(4)
N(2)-C(14)-N(3)-C(10)	1.4(3)
C(6)-C(14)-N(3)-C(10)	-175.8(2)

C(8)-C(9)-N(3)-C(14)	-0.4(3)
C(8)-C(9)-N(3)-C(10)	178.5(2)
N(4)-C(10)-N(3)-C(14)	178.1(2)
C(11)-C(10)-N(3)-C(14)	-0.9(3)
N(4)-C(10)-N(3)-C(9)	-0.9(4)
C(11)-C(10)-N(3)-C(9)	-179.9(2)
C(11)-C(10)-N(4)-C(15)	-85.6(3)
N(3)-C(10)-N(4)-C(15)	95.6(3)
C(20)-C(15)-N(4)-C(10)	-78.4(7)
C(16)-C(15)-N(4)-C(10)	178.2(4)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for compound **1o** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(4)-H(4A)...O(3)#1	0.77	2.31	3.026(5)	156.1
N(1)-H(1A)...O(5)#2	0.84(3)	2.22(4)	3.047(4)	171(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y+1/2,-z+1/2 #2 x,-y+3/2,z+1/2