Electronic Supplementary Material (ESI) for RSC Advances

Copper catalyzed three-component synthesis of benzothiazolones from o-iodoanilines, DMF, and potassium sulfide

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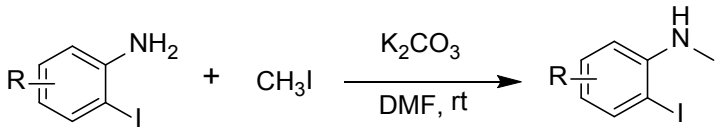
Supporting Information

1) General Information

NMR spectra of the products **2** and **4b−4g** were obtained using Bruker Avance-500 instruments, calibrated to TMS (^{1}H NMR spectra) and $CD(H)Cl_3$ (^{13}C NMR spectra) as the internal reference (0.00 ppm for ^{1}H NMR spectra and 77.00 ppm for ^{13}C NMR spectra). NMR spectra of the product **4h-4k** was recorded using Bruker Avance-500 instruments, calibrated to residual DMSO-d_6 as the internal reference (2.50 ppm for ^{1}H NMR spectra and 40.00 ppm for ^{13}C NMR spectra). High-resolution mass spectra (HRMS) were recorded on a Bruker Apex IV FTMS mass spectrometer using ESI (electrospray ionization). Melting points were measured uncorrected. Reactions were monitored by thin-layer chromatography or GC-MS analysis. Column chromatography (petroleum ether/ethyl acetate) was performed on silica gel (200-300 mesh). Unless otherwise noted, all reactions were run under nitrogen atmosphere.

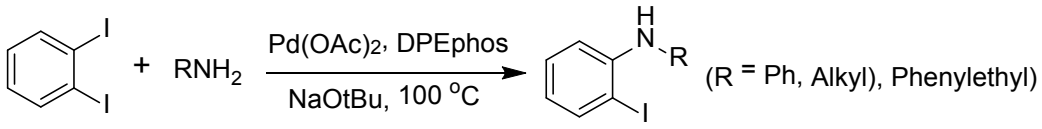
2) Synthesis of Starting Materials

Preparation of 1a and 3b-3g:[1]



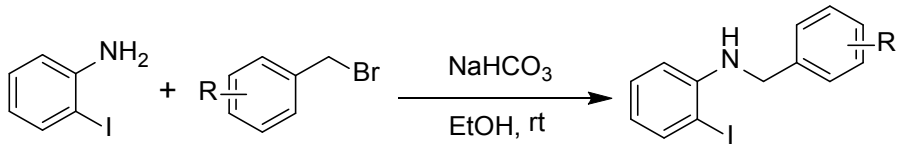
To a solution of the corresponding o-iodoaniline (1.2 equiv) and iodomethane (2 mmol) in DMF (10 mL) was added K_2CO_3 (2 equiv). The resulting mixture was stirred at room temperature for 36 h. Water (10 mL) was added to the reaction mixture. The resulting solution was extracted with diethyl ether (3 × 10 mL). The organic layers were combined and washed with water to remove any remaining DMF and dried over anhydrous Na_2SO_4. The solvent was removed under vaccum and the residue was purified by flash column chromatography on silica gel using petroleum ether/ethyl acetate as the eluent.

Preparation of 1b-1e, 1l-1o and 1q:[2]



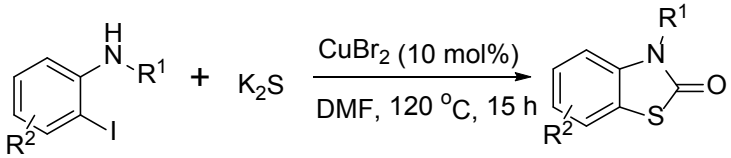
To a schlenk tube were added 1,2-iodobenzene (3 mmol) , the corresponding amine (1.5 equiv), $Pd(OAc)_2$ (0.5 mol%), DPEphos (0.75 mol%), NaOtBu (1.5 equiv) and toluene (8 mL). The resulting mixture was stirred 100 ^{o}C for 24h. the reaction mixture was filtered by a crude column with ethyl acetate as eluent, and evaporated under vacuum. the residue was purified by column chromatography on silica gel to provide the desired product.

Preparation of 1f-1k:[3]



To a schlenk tube were added o-iodoaniline (1.2 equiv), the corresponding benzyl bromide (2mmol), $NaHCO_3$ (2 equiv), and EtOH (10 mL). The resulting mixture was stirred at room temperature for overnight. After completion of the reaciton, the reaction mixture was filtered by a crude column with ethyl acetate as eluent, and evaporated under vacuum. the residue was purified by column chromatography on silica gel to provide the desired product.

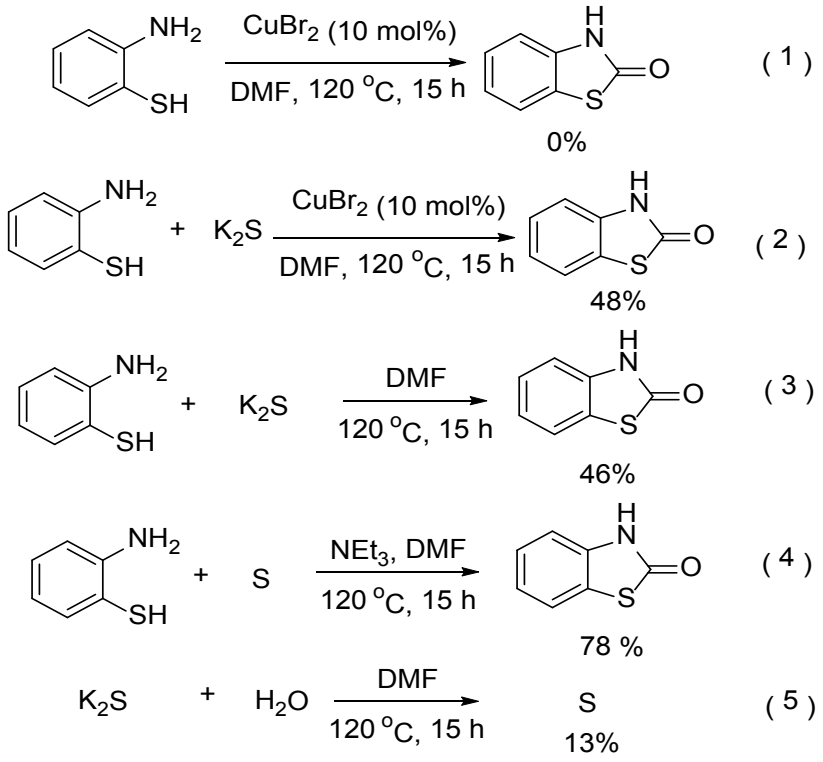
3) Typical Procedures



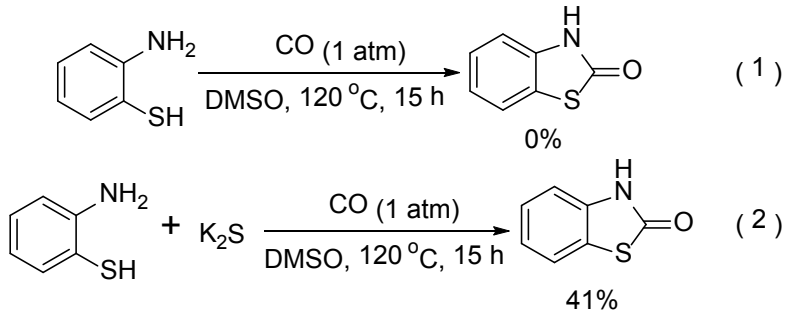
To a schlenk tube were added *o*-haloanilines (0.3 mmol), K_2S (3 equiv), $CuBr_2$ (10 mol%), and DMF (2 mL). Then under the protection of nitrogen, the mixture was stirred at 120 ^{o}C (oil bath temperature) for the indicated time until complete

consumption of starting material as monitored by TLC and GC-MS analysis. After the reaction was finished, the reaction mixture was cooled to room temperature, diluted in ethyl acetate, and washed with water. The aqueous phase was re-extracted with ethyl acetate. The combined organic extracts were dried over Na_2SO_4 and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 15:1) to afford the desired product.

4) Experiments of investigating the reaction mechanism



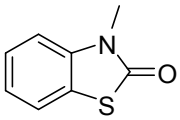
Scheme 1 2-Aminobenzenethiol React with DMF



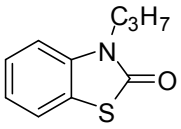
Scheme 2 2-Aminobenzenethiol React with CO

Firstly, 2-aminothiophenol was treated with DMF in the presence of $CuBr_2$, and there was no benzothiazolone product found in the reaction mixture (scheme 1, eq 1). Importantly, when K_2S was added in the above experiment, 48% of benzothiazolone was obtained (scheme 1, eq 2). Surprised, 46% of benzothiazolone was given in the absence of $CuBr_2$ in the above reaction (scheme 1, eq 3). For these results we inferred that K_2S played a key role in carbonylation reaction, and that copper catalyst are not involved in carbonylation reaction. Owing to a small amount of sulfur observed in the experiment 2 and 3, we assume it is not K_2S but sulfur that promoted the carbonylation process.[4] In order to prove our hypothesis, the reaction 2-aminothiophenol with sulfur was run in DMF, and 78% benzothiazolone was afforded (scheme 1, eq 4). Subsequently, we found that 13% of sulfur was afforded when K_2S and equivalent H_2O reacted in DMF (scheme 1, eq 5). Finally, 2-aminothiophenol was treated with CO in DMSO, no product benzothiazolone was found in the reaction mixture (scheme 2, eq 1). Similarly, when K_2S was added in this reaction, 41% of benzothiazolone was isolated (scheme 2, eq 2). These results proved the above results again.

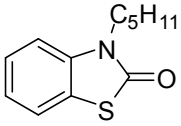
5) Characterization Data



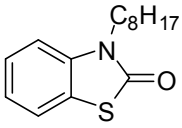
3-methylbenzo[*d*]thiazol-2(3H)-one (2a):[5] Pale yellow solid, isolated yield 77% (38.1 mg); mp: 69.7-70.7 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.42 (dd, J = 8.0 Hz, 1.0 Hz, 1H), 7.33 (td, J = 7.8 Hz, 1.0 Hz, 1H), 7.17 (td, J = 7.8 Hz, 1.0 Hz, 1H), 7.04 (d, J = 8.0 Hz, 1H), 3.45 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 170.00, 137.65, 126.31, 123.15, 122.48, 122.45, 110.36, 28.92; IR (KBr): 1679 (C=O) cm^{-1}.



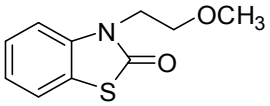
3-propylbenzo[*d*]thiazol-2(3H)-one (2b):[5] Pale yellow oil, isolated yield 68% (39.4 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.42 (dd, *J* = 8.0 Hz, 1.0 Hz, 1H), 7.31 (td, *J* = 8.0 Hz, 1.0 Hz, 1H), 7.15 (td, *J* = 7.8 Hz, 1.0 Hz, 1H), 7.04 (d, *J* = 8.5 Hz, 1H), 3.91 (t, *J* = 7.5 Hz, 2H), 1.81-1.74 (m, 2H), 0.99 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.87, 137.13, 126.16, 122.86, 122.73, 122.55, 110.54, 44.24, 20.89, 11.18; IR (KBr): 1685 (C=O) cm^{-1}.



3-pentylbenzo[*d*]thiazol-2(3H)-one (2c): Pale yellow oil, isolated yield 71% (46.9 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.42 (dd, *J* = 7.5 Hz, 1.0 Hz, 1H), 7.31 (td, *J* = 7.8 Hz, 1.0 Hz, 1H), 7.15 (td, *J* = 7.8 Hz, 1.0 Hz, 1H), 7.04 (d, *J* = 8.5 Hz, 1H), 3.93 (t, *J* = 7.5 Hz, 2H), 1.76-1.71 (m, 2H), 1.38-1.36 (m, 4H), 0.90 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.79, 137.11, 126.16, 122.84, 122.77, 122.55, 110.51, 42.76, 28.83, 27.23, 22.28, 13.86; IR (KBr): 1679 (C=O) cm^{-1}; HRMS (ESI, m/z) calcd for $[C_{12}H_{15}NOS]H^+$: 222.0947; found 222.0947.

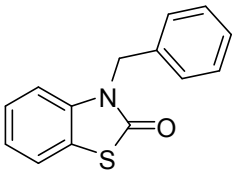


3-octylbenzo[*d*]thiazol-2(3H)-one (2d):[6] Pale yellow oil, isolated yield 81% (64.0 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.41 (dd, *J* = 7.5 Hz, 1.0 Hz, 1H), 7.31 (td, *J* = 7.8 Hz, 1.0 Hz, 1H), 7.14 (td, *J* = 7.5 Hz, 1.0 Hz, 1H), 7.04 (d, *J* = 8.0 Hz, 1H), 3.93 (t, *J* = 7.5 Hz, 2H), 1.76-1.70 (m, 2H), 1.38-1.26 (m, 10H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.77, 137.12, 126.16, 122.84, 122.78, 122.56, 110.52, 42.80, 31.68, 29.16, 29.07, 27.54, 26.75, 22.54, 14.01; IR (KBr): 1679 (C=O) cm^{-1}.

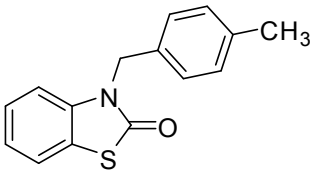


3-(2-methoxyethyl)benzo[*d*]thiazol-2(3H)-one (2e): yellow oil, isolated yield 62% (38.9 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.40 (dd, *J* = 7.5 Hz,

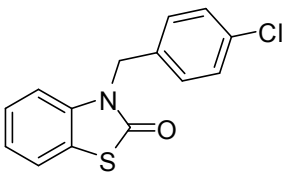
1.0 Hz, 1H), 7.30 (td, *J* = 7.8 Hz, 1.0 Hz, 1H), 7.19-7.13 (m, 2H), 4.13 (t, *J* = 5.8 Hz, 2H), 3.68 (t, *J* = 5.5 Hz, 2H), 3.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 170.13, 137.48, 126.17, 122.98, 122.45, 122.36 111.18, 69.80, 58.98, 42.72; IR (KBr): 1679 (C=O) cm^{-1}; HRMS (ESI, m/z) calcd for $[C_{10}H_{11}NO_2S]H^+$: 210.0583; found 210.0581.



3-benzylbenzo[*d*]thiazol-2(3H)-one (2f):[5] yellow solid, isolated yield 52% (37.7 mg); mp: 83.3-84.1 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.40 (dd, *J* = 8.0 Hz, 1.0 Hz, 1H), 7.32-7.24 (m, 5H), 7.19 (td, *J* = 8.0 Hz, 1.0 Hz, 1H), 7.11 (td, *J* = 7.5 Hz, 1.0 Hz, 1H), 6.95 (d, *J* = 8.0 Hz, 1H), 5.13 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 170.22, 136.88, 135.05, 128.81, 127.81, 127.04, 126.26, 123.17, 122.52 (2C), 111.19, 46.09; IR (KBr): 1665 (C=O) cm^{-1}.

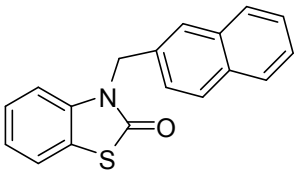


3-(4-methylbenzyl)benzo[*d*]thiazol-2(3H)-one (2g): yellow solid, isolated yield 53% (40.7 mg); mp: 66.2-67.3 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.39 (dd, *J* = 8.0 Hz, 1.0 Hz, 1H), 7.21-7.17 (m, 3H), 7.12-7.08 (m, 3H), 6.96 (d, *J* = 8.0 Hz, 1H), 5.09 (s, 2H), 2.29 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 170.21, 137.55, 136.93, 132.04, 129.46, 127.08, 126.22, 123.10, 122.52, 122.47, 111.21, 45.90, 21.02; IR (KBr): 1662 (C=O) cm^{-1}; HRMS (ESI, m/z) calcd for $[C_{15}H_{13}NOS]H^+$: 256.0791; found 256.0791.

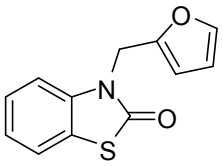


3-(4-chlorobenzyl)benzo[*d*]thiazol-2(3H)-one (2h):[7] yellow solid, isolated yield 61% (50.3 mg); mp: 79.5-80.6 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.43 (dd, *J* = 7.5 Hz, 1.0 Hz, 1H), 7.30-7.27 (m, 2H), 7.24-7.20 (m, 3H), 7.14 (td, *J* =

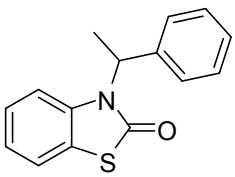
7.5 Hz, 1.0 Hz, 1H), 6.92 (dd, J = 8.0 Hz, 0.5 Hz, 1H), 5.10 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 170.24, 136.63, 133.75, 133.60, 129.04, 128.50, 126.36, 123.38, 122.68, 122.55, 111.01, 45.47; IR (KBr): 1669 (C=O) cm^{-1}.



3-(naphthalen-2-ylmethyl)benzo[*d*]thiazol-2(3H)-one (2i): yellow solid, isolated yield 54% (47.4 mg); mp: 108.9-110.1 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.79-7.75 (m, 3H), 7.70 (s, 1H), 7.45-7.43 (m, 2H), 7.40 (d, J = 8.0 Hz, 2H), 7.15 (td, J = 7.8 Hz, 1.0 Hz, 1H), 7.08 (td, J = 7.5 Hz, 1.0 Hz, 1H), 6.97 (d, J = 8.0 Hz, 1H), 5.27 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 170.34, 136.89, 133.16, 132.81, 132.52, 128.84, 127.71, 127.64, 126.37, 126.28, 126.11, 125.94, 124.82, 123.21, 122.53 (2C), 111.26, 46.33; IR (KBr): 1685 (C=O) cm^{-1}; HRMS (ESI, m/z) calcd for $[C_{18}H_{13}NOS]H^+$: 292.0791; found 292.0792.

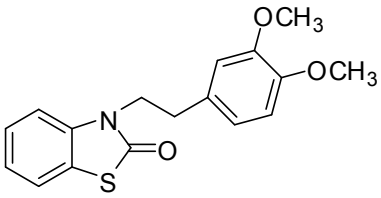


3-(furan-2-ylmethyl)benzo[*d*]thiazol-2(3H)-one (2j): yellow oil, isolated yield 58% (40.2 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.41 (dd, J = 7.5 Hz, 0.5 Hz, 1H), 7.34 (dd, J = 2.0 Hz, 0.5 Hz, 1H), 7.30 (td, J = 7.8 Hz, 1.0 Hz, 1H), 7.24(t, J = 6.0 Hz, 1H), 7.15 (td, J = 7.5 Hz, 1.0 Hz, 1H), 6.36 (d, J = 3.5 Hz, 1H), 6.31 (dd, J = 3.5 Hz, 2.0 Hz, 1H), 5.10 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.82, 148.59, 142.62, 136.74, 126.30, 123.26, 122.51, 122.43, 111.07, 110.55, 109.06, 39.02; IR (KBr): 1682 (C=O) cm^{-1}; HRMS (ESI, m/z) calcd for $[C_{12}H_9NO_2S]H^+$: 232.0427; found 232.0427.

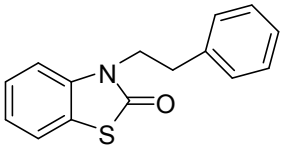


3-(1-phenylethyl)benzo[*d*]thiazol-2(3H)-one (2k): yellow oil, isolated yield 42% (32.1 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.40-7.38 (m, 1H),

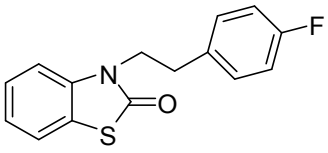
7.34-7.33 (m, 4H), 7.29-7.27 (m, 1H), 7.06-7.04 (m, 2H), 6.73-6.71 (m, 1H), 6.14-6.09 (m, 1H), 1.90 (d, J = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 170.18, 138.58, 135.89, 128.74, 127.58, 126.46, 125.72, 122.65, 122.62, 122.51, 112.75, 51.81, 16.17; IR (KBr): 1665 (C=O) cm^{-1}; HRMS (ESI, m/z) calcd for $[C_{15}H_{13}NOS]H^+$: 256.0791; found 256.0793.



3-(3,4-dimethoxyphenethyl)benzo[*d*]thiazol-2(3H)-one (2l): yellow solid, isolated yield 71% (67.2 mg); mp: 83.9-85.0 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.41 (dd, J = 8.0 Hz, 1.0 Hz, 1H), 7.27 (td, J = 8.0 Hz, 1.0 Hz, 1H), 7.13 (td, J = 7.8 Hz, 1.0 Hz, 1H), 6.95 (d, J = 8.0 Hz, 1H), 6.80-6.75 (m, 2H), 6.67 (d, J = 1.5 Hz, 1H), 4.13 (t, J = 7.5 Hz, 2H), 3.84 (s, 3H), 3.80 (s, 3H), 2.96 (t, J = 7.8 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.64, 148.89, 147.80, 136.83, 130.03, 126.14, 122.89, 122.49, 122.46, 120.65, 111.90, 111.30, 110.41, 55.78, 55.72, 44.19, 33.24; IR (KBr): 1672 (C=O) cm^{-1}; HRMS (ESI, m/z) calcd for $[C_{17}H_{17}NO_3S]H^+$: 316.1002; found 316.1000.

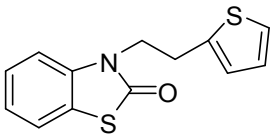


3-phenethylbenzo[*d*]thiazol-2(3H)-one (2m):[8] yellow solid, isolated yield 79% (60.5 mg); mp: 85.8-86.7 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.40 (d, J = 7.5 Hz, 1H), 7.30-7.25 (m, 3H), 7.23-7.21 (m, 3H), 7.13 (t, J = 7.5 Hz, 1H), 6.96 (d, J = 8.0 Hz, 1H), 4.13 (t, J = 8.0 Hz, 2H), 3.00 (t, J = 8.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.64, 137.52, 136.79, 128.70, 128.62 (2C), 126.76, 126.18, 122.91, 122.58, 110.36, 44.09, 33.73; IR (KBr): 1669 (C=O) cm^{-1}.



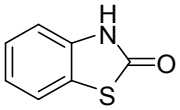
3-(4-fluorophenethyl)benzo[*d*]thiazol-2(3H)-one (2n):

yellow oil, isolated yield 80% (65.5 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.41 (dd, *J* = 7.5 Hz, 0.5 Hz, 1H), 7.28 (td, *J* = 7.8 Hz, 1.5 Hz, 1H), 7.17-7.12 (m, 3H), 6.96 (t, *J* = 9.0 Hz, 3H), 4.12 (t, *J* = 7.8 Hz, 2H), 2.99 (t, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.68, 161.76 (d, *J* = 243.5 Hz), 136.73, 133.22 (d, *J* = 2.9 Hz), 130.20 (d, *J* = 7.9 Hz), 126.22, 123.00, 122.65, 122.59, 115.46 (d, *J* = 21.0 Hz), 110.30, 44.03, 32.91; IR (KBr): 1675 (C=O) cm^{-1}; HRMS (ESI, m/z) calcd for $[C_{15}H_{12}FNOS]H^{+}$: 274.0696; found 274.0700.

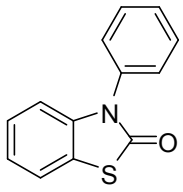


3-(2-(thiophen-2-yl)ethyl)benzo[*d*]thiazol-2(3H)-one (2o):

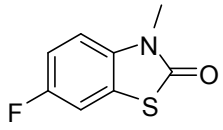
yellow solid, isolated yield 77% (60.3 mg); mp: 82.8-83.8 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.41 (dd, *J* = 8.0 Hz, 1.0 Hz, 1H), 7.27 (td, *J* = 8.0 Hz, 1.0 Hz, 1H), 7.15-7.12 (m, 2H), 6.95 (d, *J* = 8.0 Hz, 1H), 6.90 (dd, *J* = 5.0 Hz, 3.5 Hz, 1H), 6.84 (d, *J* = 2.5 Hz, 1H), 4.17 (t, *J* = 7.5 Hz, 2H), 3.24 (t, *J* = 7.8 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.70, 139.35, 136.75, 127.09, 126.26, 125.76, 124.25, 123.01, 122.62, 122.56, 110.27, 44.12, 27.75; IR (KBr): 1672 (C=O) cm^{-1}; HRMS (ESI, m/z) calcd for $[C_{13}H_{11}NOS_2]H^{+}$: 262.0355; found 262.0355.



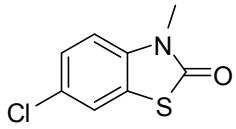
benzo[*d*]thiazol-2(3H)-one (2p):[9] Pale yellow solid, isolated yield 75% (34.0 mg); mp: 134.5-135.3 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 10.44 (s, 1H), 7.39 (d, *J* = 7.5 Hz, 1H), 7.27 (t, *J* = 7.5 Hz, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.14 (t, *J* = 7.8 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 173.37, 135.50, 126.49, 123.88, 123.20, 122.42, 111.88; IR (KBr): 1665 (C=O) cm^{-1}.



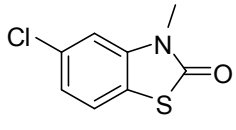
3-phenylbenzo[*d*]thiazol-2(3H)-one (2q):[5] yellow oil, isolated yield 24% (16.4 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.58-7.55 (m, 2H), 7.51-7.48 (m, 1H), 7.47-7.45 (m, 1H), 7.42-7.40 (m, 2H), 7.22-7.16 (m, 2H), 6.80-6.78 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.66, 138.23, 134.82, 129.98, 129.61, 129.18, 127.86, 126.27, 123.54, 122.52, 111.78; IR (KBr): 1695 (C=O) cm^{-1}.



6-fluoro-3-methylbenzo[*d*]thiazol-2(3H)-one (4b):[10] yellow solid, isolated yield 78% (42.8 mg); mp: 91.9-92.9 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.18 (dd, J = 7.5 Hz, 2.5 Hz, 1H), 7.05 (td, J = 8.8 Hz, 2.5 Hz, 1H), 6.97 (dd, J = 9.0 Hz, 4.5 Hz, 1H), 3.44 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.41, 158.93 (d, J = 241.1 Hz), 133.92, 123.60 (d, J = 10.0 Hz), 113.45 (d, J = 23.8 Hz), 110.91 (d, J = 8.4 Hz), 109.84 (d, J = 26.8 Hz), 29.12; IR (KBr): 1679 (C=O) cm^{-1}.

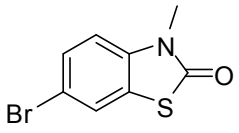


6-chloro-3-methylbenzo[*d*]thiazol-2(3H)-one (4c):[11] Pale yellow solid, isolated yield 83% (49.6 mg); mp: 107.7-108.6 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.39 (d, J = 2.0 Hz, 1H), 7.28 (dd, J = 8.5 Hz, 2.0 Hz, 1H), 6.95 (d, J = 9.0 Hz, 1H), 3.43 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.26, 136.17, 128.43, 126.46, 123.83, 122.20, 111.08, 29.07; IR (KBr): 1682 (C=O) cm^{-1}.

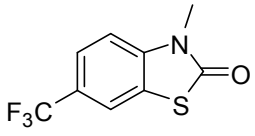


5-chloro-3-methylbenzo[*d*]thiazol-2(3H)-one (4d):[11] Pale yellow solid, isolated yield 77% (46.1 mg); mp: 104.2-104.9 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.32 (d, J = 8.0 Hz, 1H), 7.14 (dd, J = 8.0 Hz, 1.5 Hz, 1H), 7.03 (d, J = 1.5 Hz, 1H), 3.43 (s, 3H); mp: 103.2-103.9 °C; ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.84, 138.55,

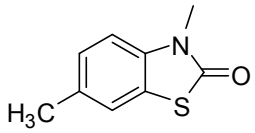
132.33, 123.24, 123.19, 120.70, 110.77, 29.06; IR (KBr): 1685 (C=O) cm^{-1}.



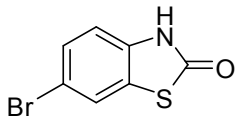
6-bromo-3-methylbenzo[*d*]thiazol-2(3H)-one (4e):[12] Pale yellow solid, isolated yield 55% (40.3 mg); mp: 116.6-117.8 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.53 (d, *J* = 1.5 Hz, 1H), 7.43 (dd, *J* = 8.5 Hz, 1.5 Hz, 1H), 6.90 (d, *J* = 8.5 Hz, 1H), 3.43 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.22, 136.65, 129.30, 124.97, 124.28, 115.54, 111.52, 29.08; IR (KBr): 1679 (C=O) cm^{-1}.



3-methyl-6-(trifluoromethyl)benzo[*d*]thiazol-2(3H)-one (4f): yellow solid, isolated yield 72% (50.5 mg); mp: 51.6-52.7 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.69 (s, 1H), 7.59 (d, *J* = 8.5 Hz, 1H), 7.13 (d, *J* = 8.5 Hz, 1H), 3.49 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.62, 140.16, 125.55 (q, *J* = 32.8 Hz), 123.85 (q, *J* = 270.4 Hz), 123.66 (q, *J* = 3.8 Hz), 123.12, 119.73 (q, *J* = 3.6 Hz), 110.18, 29.20; IR (KBr): 1685 (C=O) cm^{-1}; HRMS (ESI, m/z) calcd for $[C_9H_6F_3NOS]H^+$: 234.0195; found 234.0195.

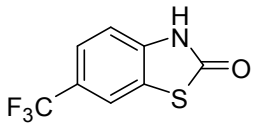


3,6-dimethylbenzo[*d*]thiazol-2(3H)-one (4g):[13] Pale yellow solid, isolated yield 75% (40.3 mg); mp: 68.5-70.0 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.21 (s, 1H), 7.11 (d, *J* = 8.5 Hz, 1H), 6.90 (d, *J* = 8.0 Hz, 1H), 3.41 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.89, 135.41, 132.93, 127.02, 122.65, 122.34, 110.04, 28.86, 20.96; IR (KBr): 1669 (C=O) cm^{-1}.

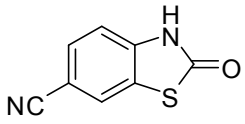


6-bromobenzo[*d*]thiazol-2(3H)-one (4h):[14] Pale yellow solid, isolated yield 53% (36.6 mg); mp: 229.8-230.8 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ: 11.98 (s, 1H), 7.81 (d, *J* = 2.0 Hz, 1H), 7.42 (dd, *J* = 8.5 Hz, 2.0 Hz, 1H), 7.04 (d, *J* = 1.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ: 170.23, 136.13, 129.69, 126.10,

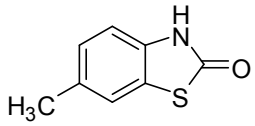
125.51, 114.51, 113.66; IR (KBr): 1672 (C=O) cm^{-1}.



6-(trifluoromethyl)benzo[*d*]thiazol-2(3H)-one (4i):[9] Pale yellow solid, isolated yield 73% (47.9 mg); mp: 130.9-131.8 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ: 12.28 (s, 1H), 8.01 (s, 1H), 7.58 (d, J = 8.5 Hz, 1H), 7.25 (d, J = 8.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ: 170.77, 140.13, 124.89, 124.80 (q, J = 270.1 Hz), 124.08 (q, J = 3.8 Hz), 123.67 (q, J = 32.0 Hz), 120.71 (q, J = 3.9 Hz), 112.21; IR (KBr): 1722(C=O) cm^{-1}.



2-oxo-2,3-dihydrobenzo[*d*]thiazole-6-carbonitrile (4j):[15] Pale yellow solid, isolated yield 14% (7.4 mg); mp: above 230 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ: 12.41 (s, 1H), 8.13 (d, J = 1.5 Hz, 1H), 7.72 (dd, J = 8.5 Hz, 1.5 Hz, 1H), 7.24 (d, J = 8.0 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ: 170.54, 140.73, 131.31, 127.33, 125.09, 119.31, 112.61, 105.23; IR (KBr): 1689 (C=O) cm^{-1}.



6-methylbenzo[*d*]thiazol-2(3H)-one (4k):[9] Pale yellow solid, isolated yield 70% (34.7 mg); mp: 166.3-167.4 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ: 11.74 (s, 1H), 7.32 (s, 1H), 7.06 (d, J = 8.0 Hz, 1H), 6.99 (d, J = 8.0 Hz, 1H), 2.28 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) δ: 170.46, 134.48, 132.32, 127.60, 123.77, 123.12, 111.72, 21.12; IR (KBr): 1655 (C=O) cm^{-1}.

6) References

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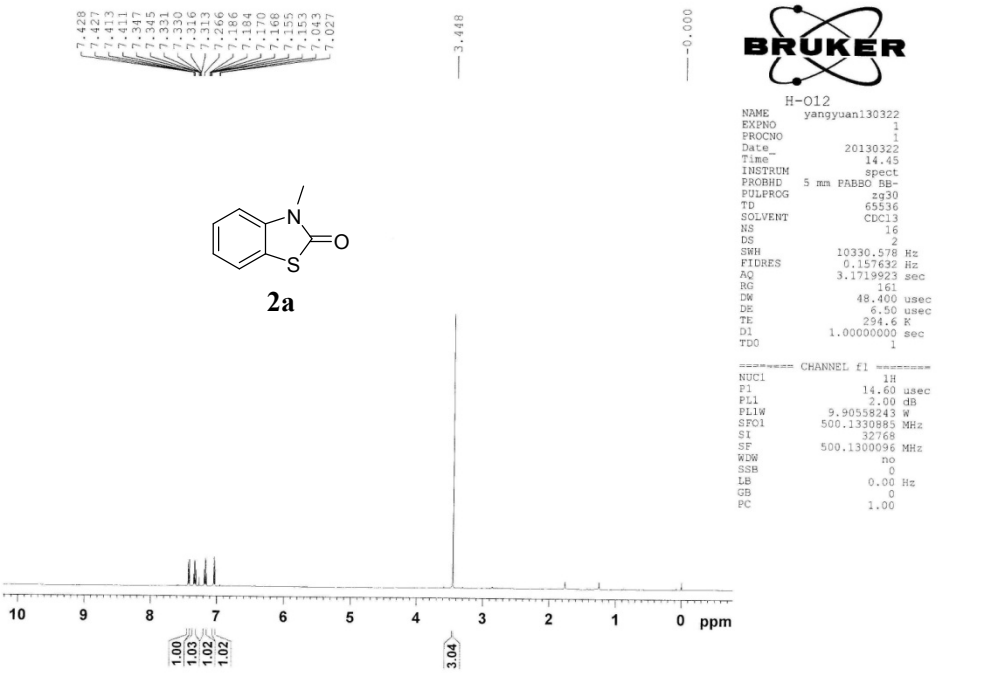
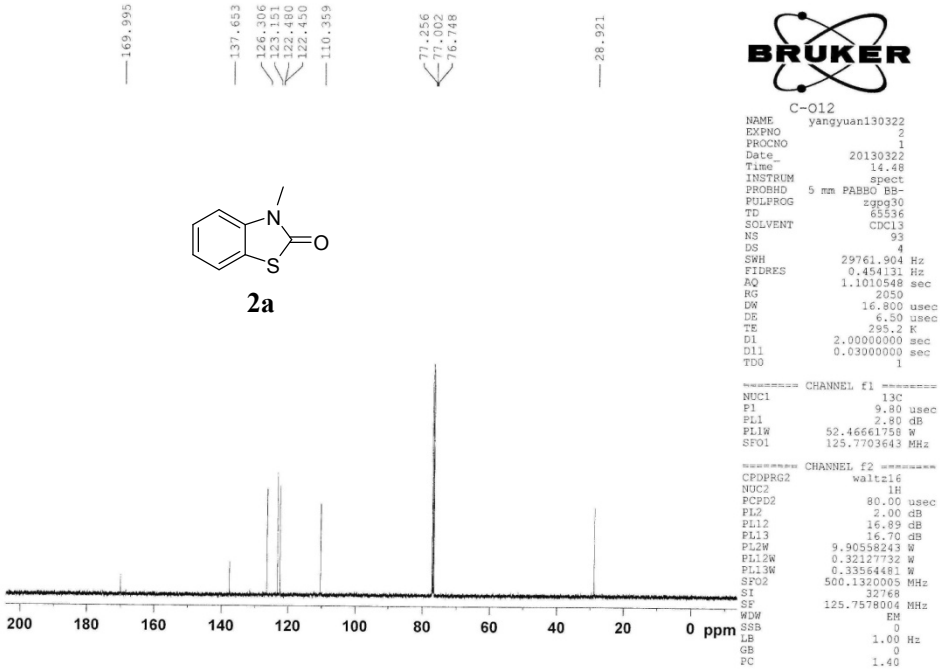
6) Scanned ^{1}H NMR and ^{13}C NMR Spectra of All New Compounds

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DW 16.800 usec
DE 6.50 usec
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D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
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NUC2 1H
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PL13 16.70 dB
PL2W 9.90558243 W
PL12W 0.32127732 W
PL13W 0.33564481 W
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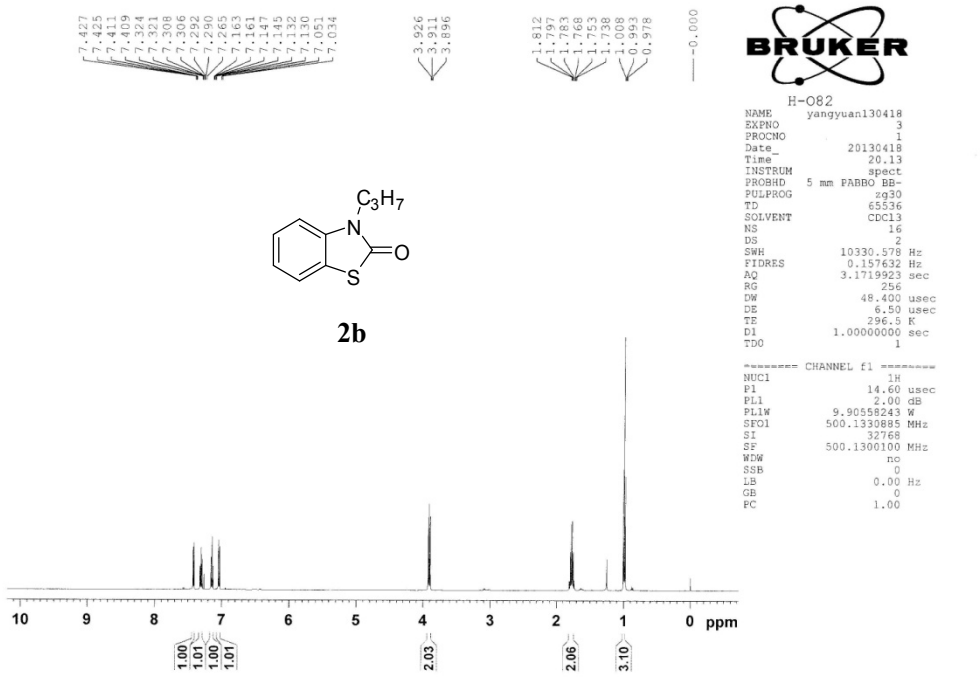
2a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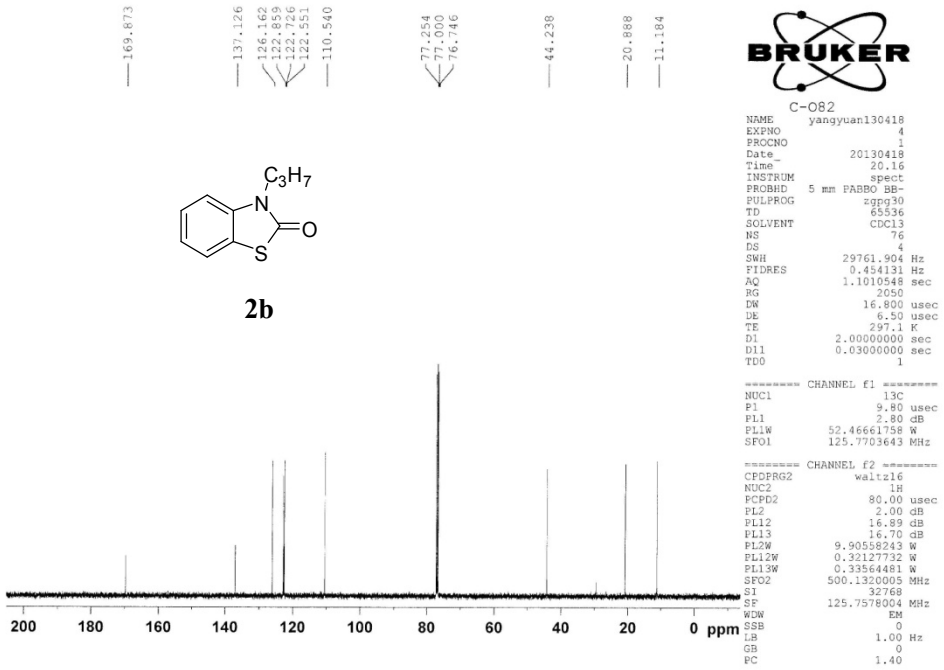
2b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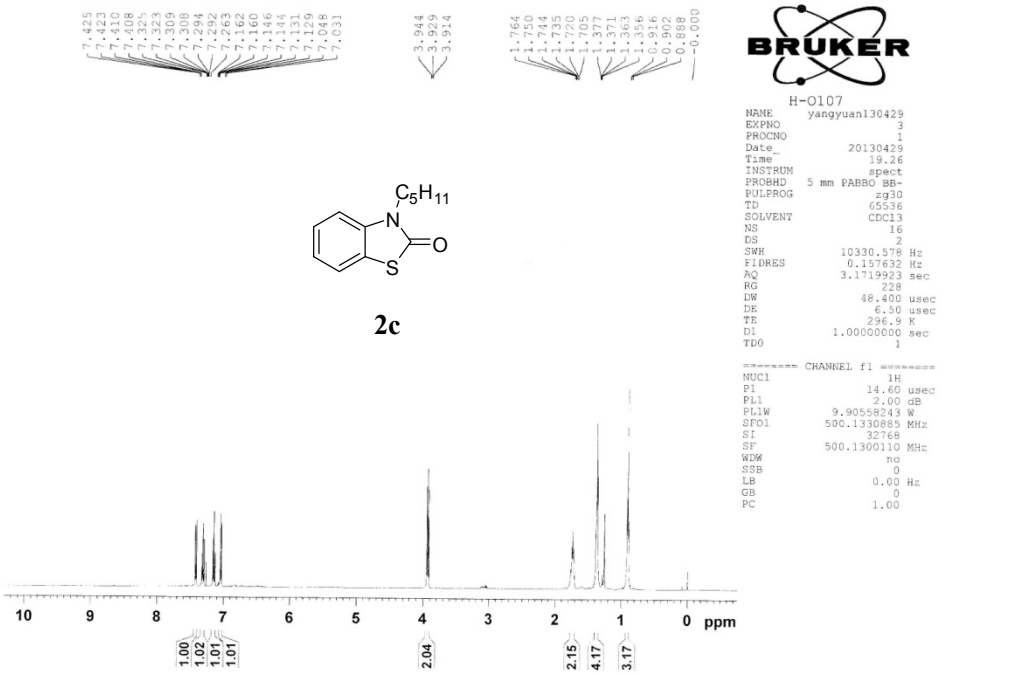
2b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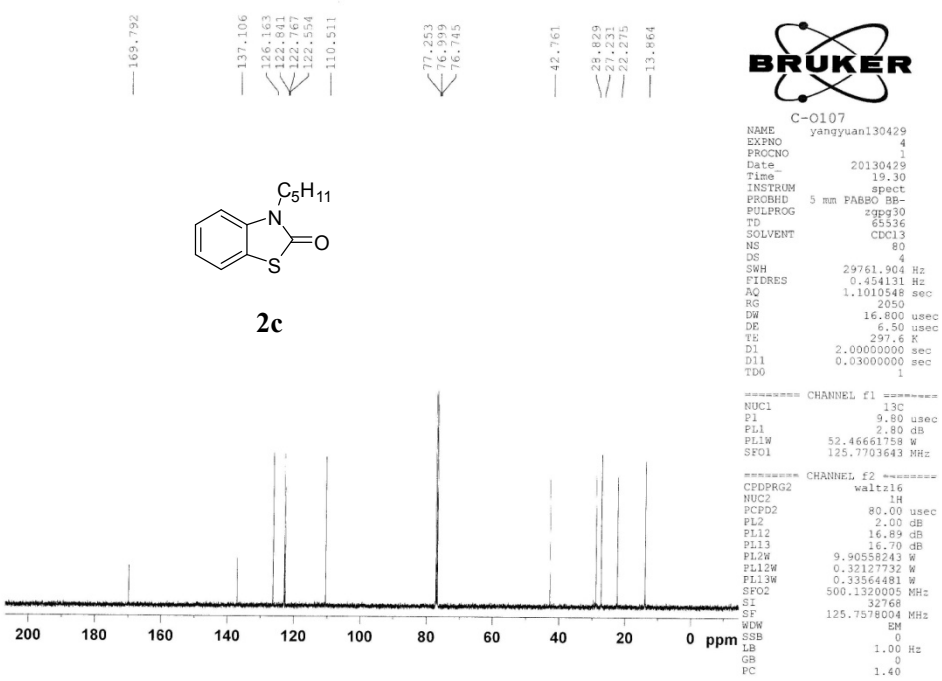
PL1 2.00 dB
PL1W 9.90558243 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300110 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

2c



2c

C-0107

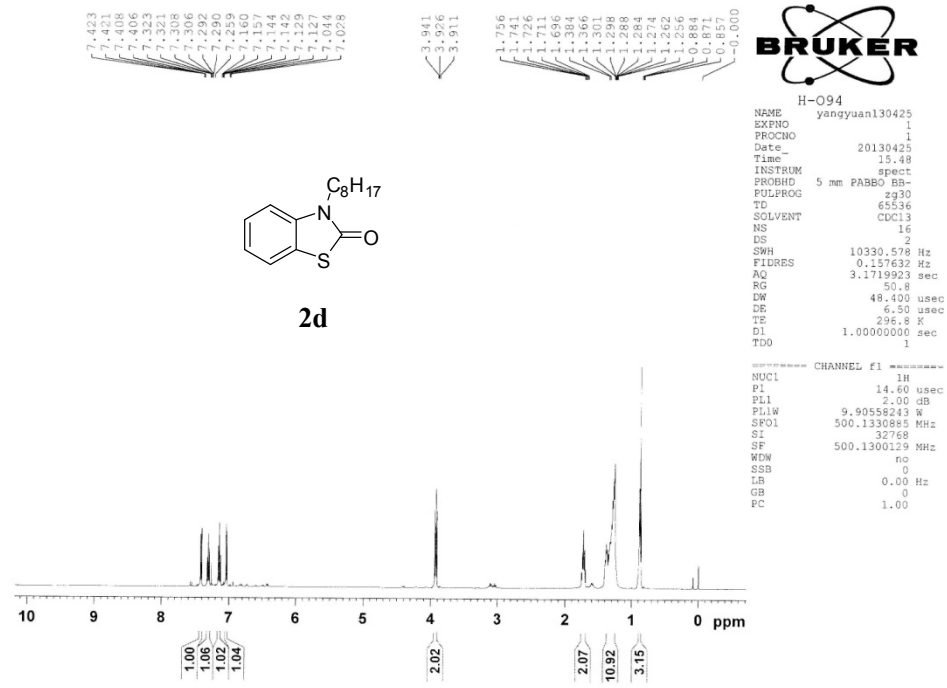
NAME	yangyuan130429
EXPNO	4
PROCNO	1
Date_	20130429
Time	19.30
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	80
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	297.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578004 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



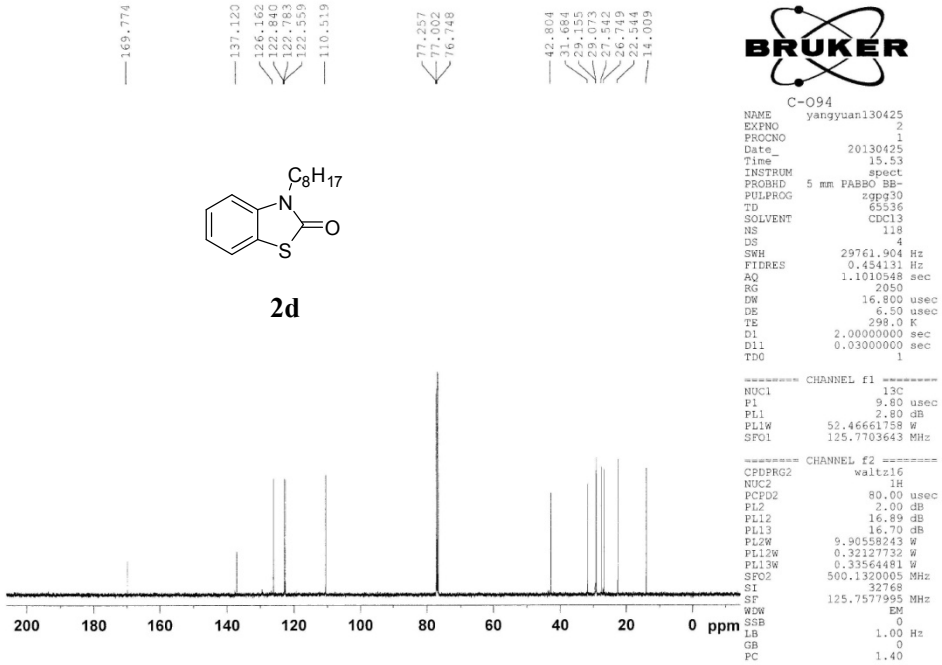
2d

H-094

NAME	yangyuan130425
EXPNO	1
PROCNO	1
Date_	20130425
Time	15.48
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	50.8
DW	48.400 usec
DE	6.50 usec
TE	296.8 K
D1	1.00000000 sec
TD0	1

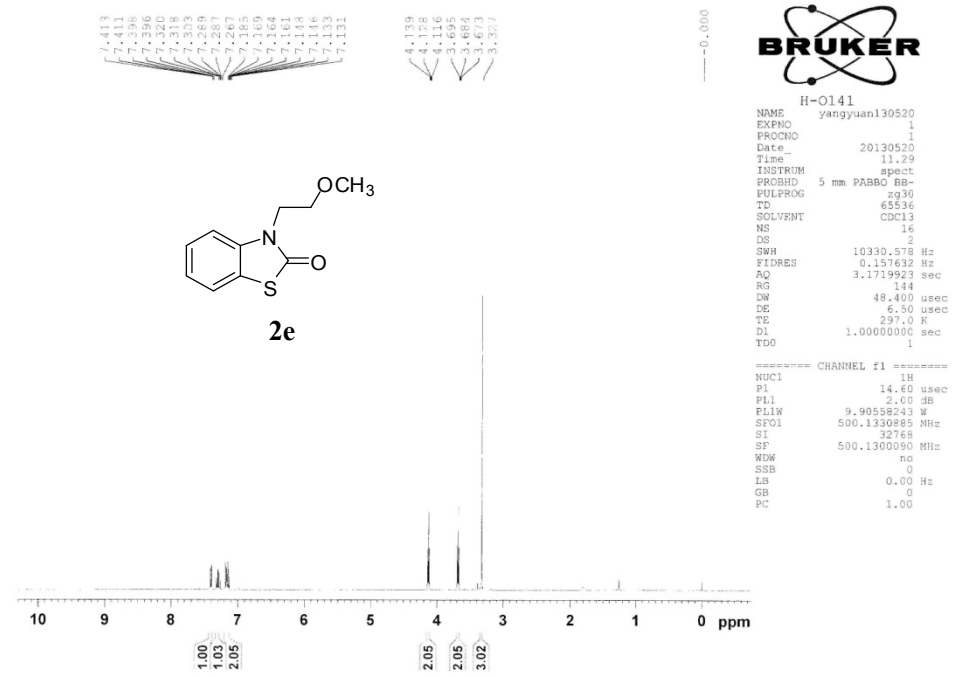
======== CHANNEL f1 ========

NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



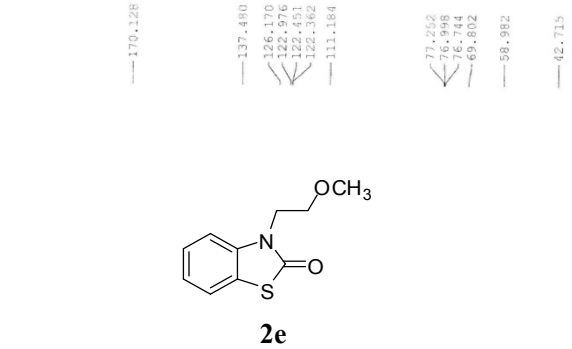
2d

```
C-094
NAME            yangyuan130425
EXPNO                        2
PROCNO                       1
Date_             20130425
Time_                15.53
INSTRUM              spect
PROBHD     5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      118
DS                        4
SWH              29761.904 Hz
FIDRES               0.454131 Hz
AQ                1.1010948 sec
RG                     2050
DW                  16.800 usec
DE                   6.50 usec
TE                  298.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                   9.80 usec
PL1                  2.80 dB
PL1W            52.46661758 W
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                  2.00 dB
PL12                16.89 dB
PL13                16.70 dB
PL2W             9.90558243 W
PL12W            0.32127732 W
PL13W            0.33564481 W
SFO2           500.1320005 MHz
SI                    32768
SF             125.7577995 MHz
WDW                      EM
SSB                       0
LB                   1.00 Hz
GB                        0
PC                    1.40
```



2e

```
H-0141
NAME            yangyuan130520
EXPNO                        1
PROCNO                       1
Date_             20130520
Time_                11.29
INSTRUM              spect
PROBHD     5 mm PABBO BB-
PULPROG              zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH              10330.578 Hz
FIDRES               0.157632 Hz
AQ                3.1719923 sec
RG                      144
DW                  48.400 usec
DE                   6.50 usec
TE                  297.0 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.60 usec
PL1                  2.00 dB
PL1W             9.90558243 W
SFO1           500.1330885 MHz
SI                    32768
SF             500.1300090 MHz
WDW                      no
SSB                       0
LB                   0.00 Hz
GB                        0
PC                    1.00
```

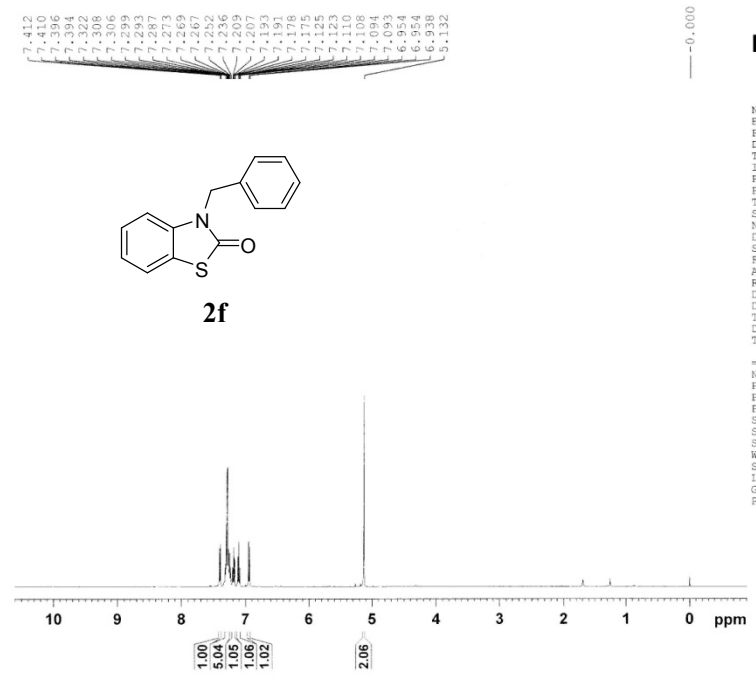


2e

BRUKER

C-0141

NAME	yangyuan130520
EXPNO	2
PROCNO	1
Date_	20130520
Time	11.31
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	92
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	297.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578004 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

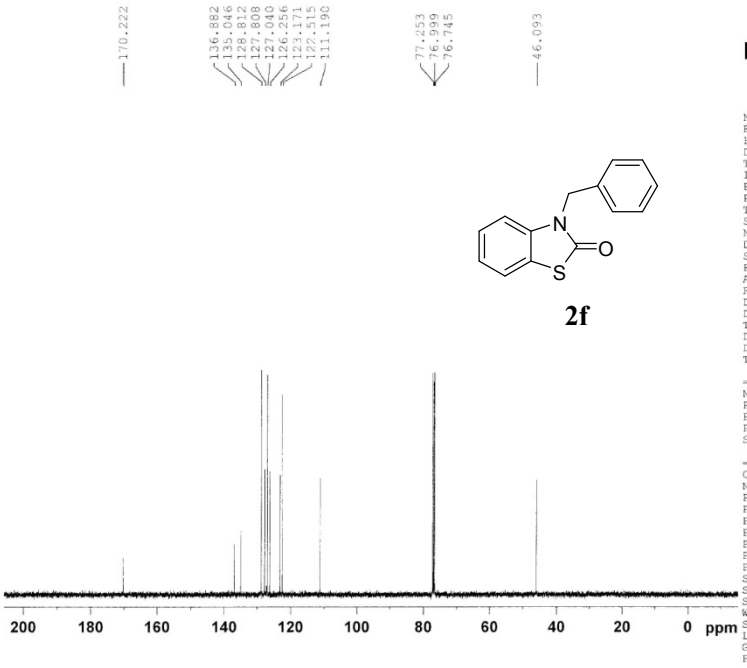


2f

BRUKER

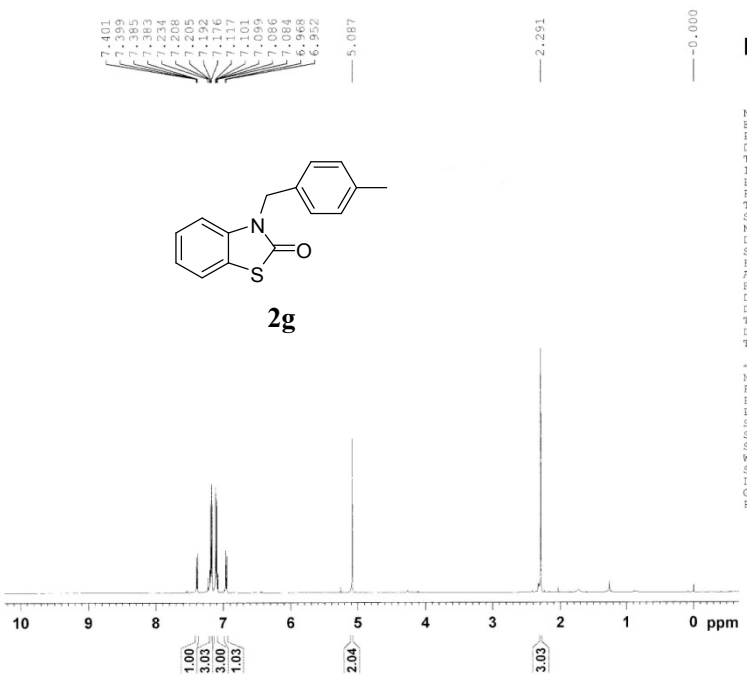
H-093

NAME	yangyuan130424
EXPNO	3
PROCNO	1
Date_	20130424
Time	15.25
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	128
DW	48.400 usec
DE	6.50 usec
TE	297.5 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300242 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



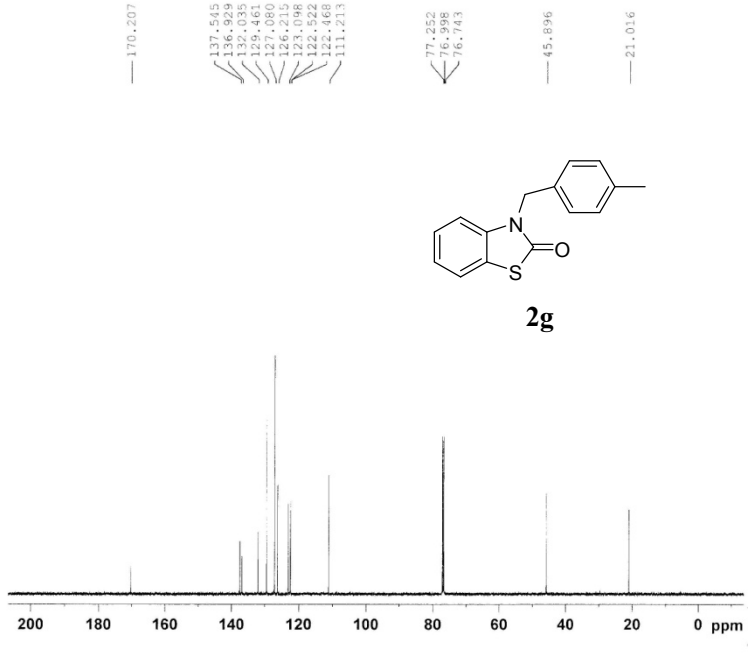
2f

```
C-093
NAME          yangyuan130424
EXPNO                      4
PROCNO                     1
Date_               20130424
Time                   15.27
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        76
DS                         4
SWH                29761.904 Hz
FIDRES             0.454131 Hz
AQ                 1.1010548 sec
RG                      2050
DW                    16.800 usec
DE                     6.50 usec
TE                   298.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                       9.80 usec
PL1                      2.80 dB
PL1W              52.46661758 W
SFO1              125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     2.00 dB
PL12                   16.89 dB
PL13                   16.70 dB
PL2W               9.90558243 W
PL12W              0.32127732 W
PL13W              0.33564481 W
SFO2              500.1320005 MHz
SI                      32768
SF                125.7578040 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```



2g

```
H-098
NAME          yangyuan130426
EXPNO                      1
PROCNO                     1
Date_               20130426
Time                   20.40
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719923 sec
RG                       101
DW                    48.400 usec
DE                     6.50 usec
TE                   296.5 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                       1H
P1                      14.60 usec
PL1                      2.00 dB
PL1W               9.90558243 W
SFO1              500.1330885 MHz
SI                      32768
SF                500.1300262 MHz
WDW                       no
SSB                         0
LB                       0.00 Hz
GB                          0
PC                       1.00
```

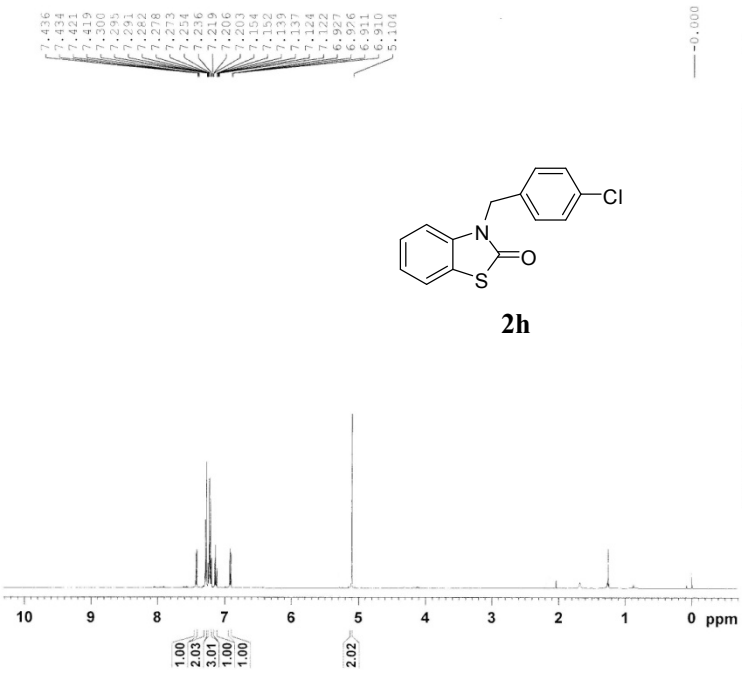


2g

BRUKER

C-098

NAME	yangyuan130426
EXPNO	2
PROCNO	1
Date_	20130426
Time_	20.45
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	92
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	297.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578049 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

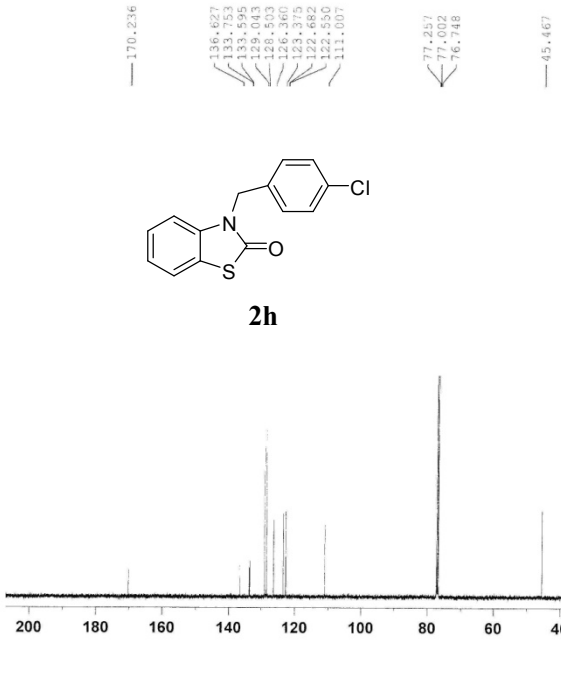


2h

BRUKER

H-0125

NAME	yangyuan130512
EXPNO	5
PROCNO	1
Date_	20130512
Time_	17.37
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	203
DW	48.400 usec
DE	6.50 usec
TE	296.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300154 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

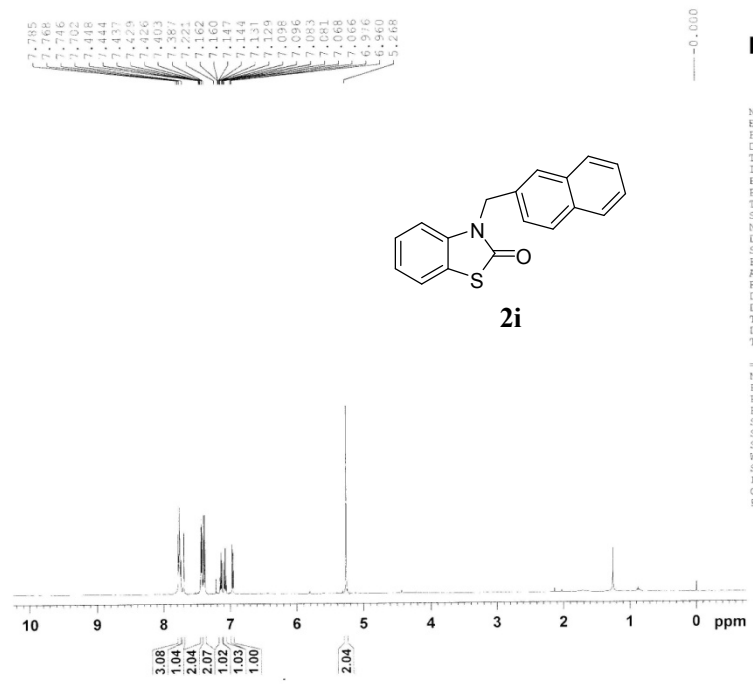


2h

BRUKER

C-0125

NAME	yangyuan130512
EXPNO	6
PROCNO	1
Date_	20130512
Time	17.40
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	80
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	297.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577986 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

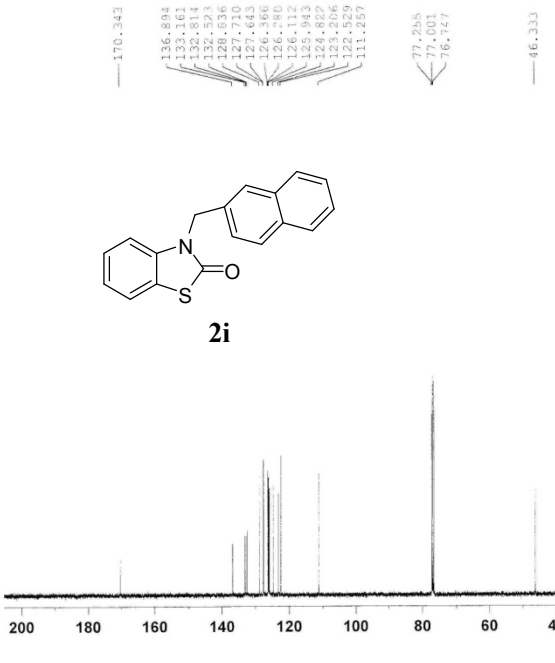


2i

BRUKER

C-0113(1)

NAME	yangyuan130508
EXPNO	3
PROCNO	1
Date_	20130508
Time	17.05
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	128
DW	48.400 usec
DE	6.50 usec
TE	297.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300320 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00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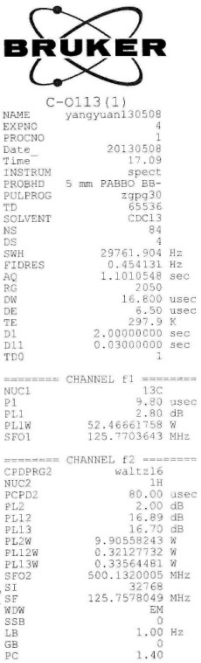
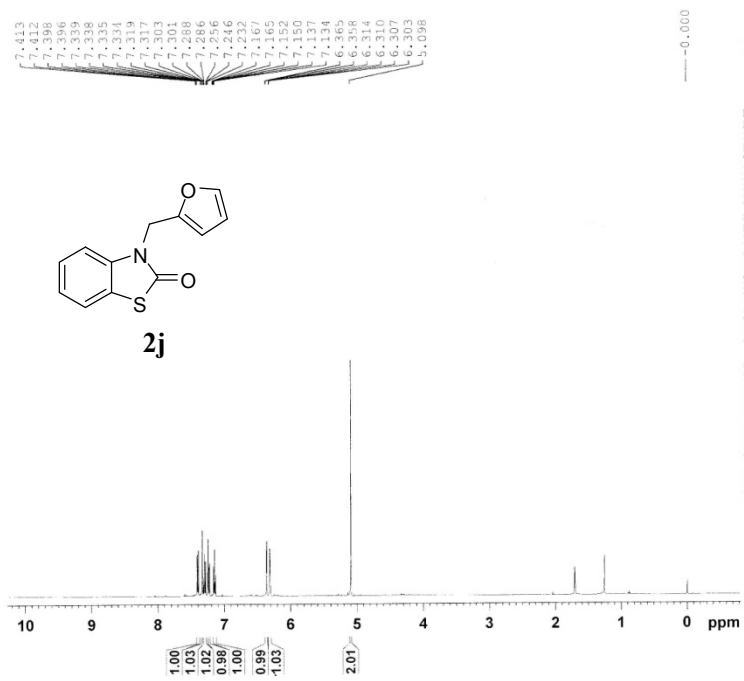
132.223
128.536
127.710
127.643
126.966
126.840
126.112
125.943
125.822
123.626
122.529
111.257
77.255
77.001
76.747
46.333
N
O
S
200
180
160
140
120
100
80
60
40
20
0
ppm

2i


BRUKER
C-0113(1)
NAME yangyuan130508
EXPNO 4
PROCNO 1
Date_ 20130508
Time_ 17.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 84
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 2.80 dB
PL1W 52.46661758 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 16.89 dB
PL13 16.70 dB
PL2W 9.90558243 W
PL12W 0.32127732 W
PL13W 0.33564481 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578049 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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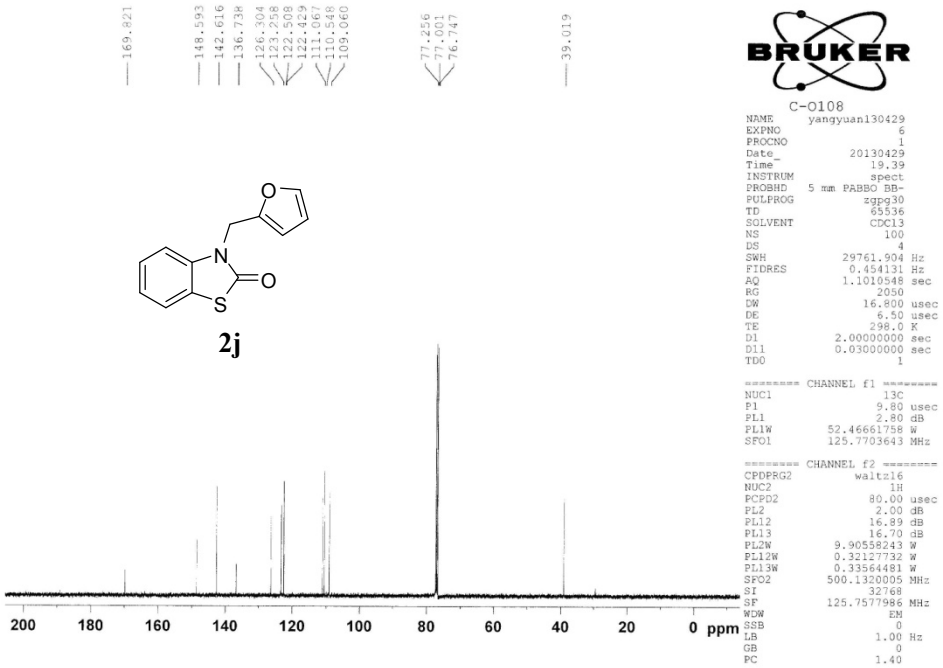
7.317
7.303
7.301
7.288
7.286
7.256
7.246
7.232
7.167
7.165
7.152
7.150
7.137
7.134
6.365
6.358
6.314
6.310
6.307
6.303
5.098
0.000
O
N
O
S
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.03
1.02
0.98
1.00
0.99
1.03
2.01

2j


BRUKER
H-0108
NAME yangyuan130429
EXPNO 5
PROCNO 1
Date_ 20130429
Time_ 19.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 203
DW 48.400 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.60 usec
PL1 2.00 dB
PL1W 9.90558243 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300146 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

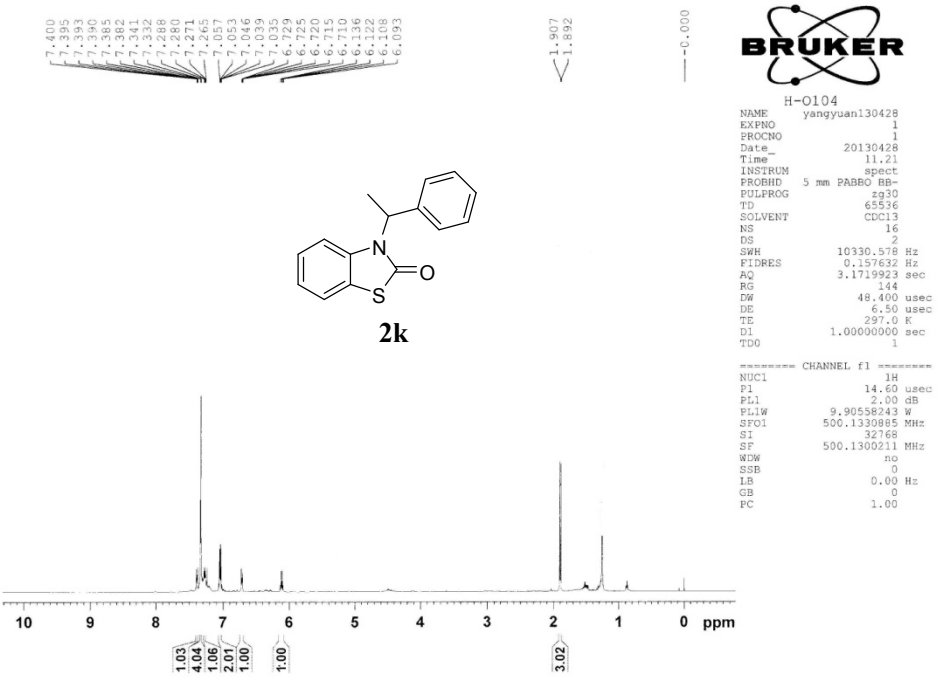


2j

BRUKER

C-0108

NAME	yangyuan130429
EXPNO	6
PROCNO	1
Date_	20130429
Time_	19.39
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	100
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577986 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

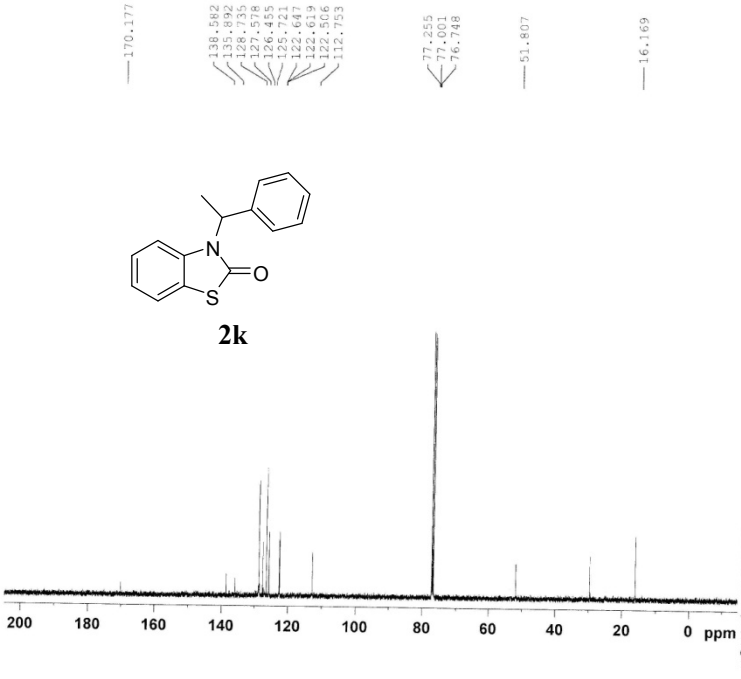


2k

BRUKER

H-0104

NAME	yangyuan130428
EXPNO	1
PROCNO	1
Date_	20130428
Time_	11.21
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	144
DW	48.400 usec
DE	6.50 usec
TE	297.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300211 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



2k

BRUKER

C-0104

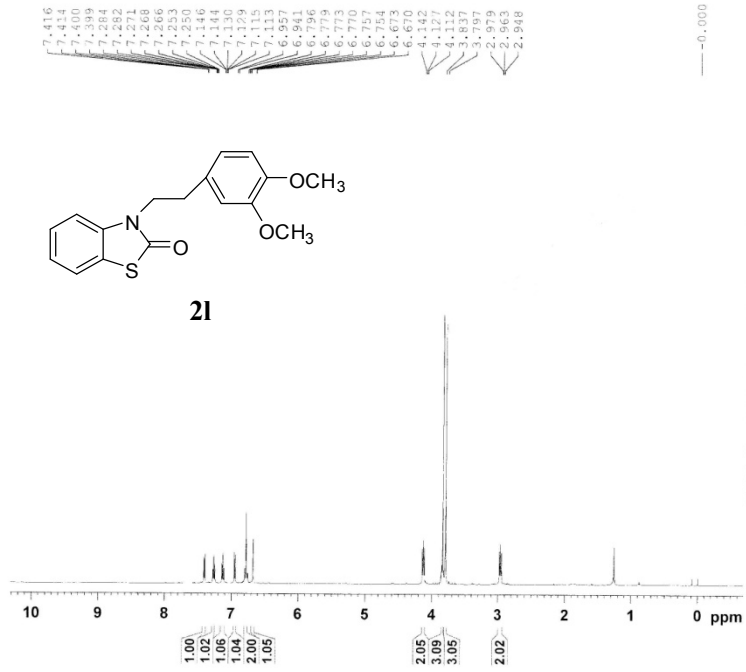
NAME	yangyuan130428
EXPNO	2
PROCNO	1
Date_	20130428
Time	11.25
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	112
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	298.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577977 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



2l

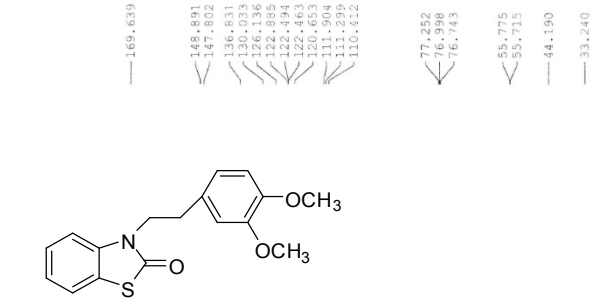
BRUKER

H-0124

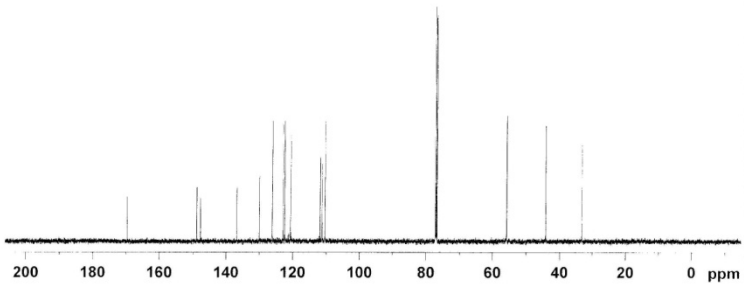
NAME	yangyuan130512
EXPNO	3
PROCNO	1
Date_	20130512
Time	17.26
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	57
DW	48.400 usec
DE	6.50 usec
TE	296.4 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300063 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



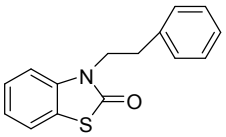
2l



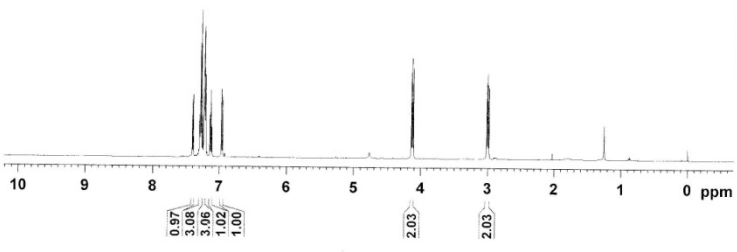
BRUKER

C-0124

NAME	yangyuan130512
EXPNO	4
PROCNO	1
Date_	20130512
Time	17.31
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	108
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	297.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578076 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



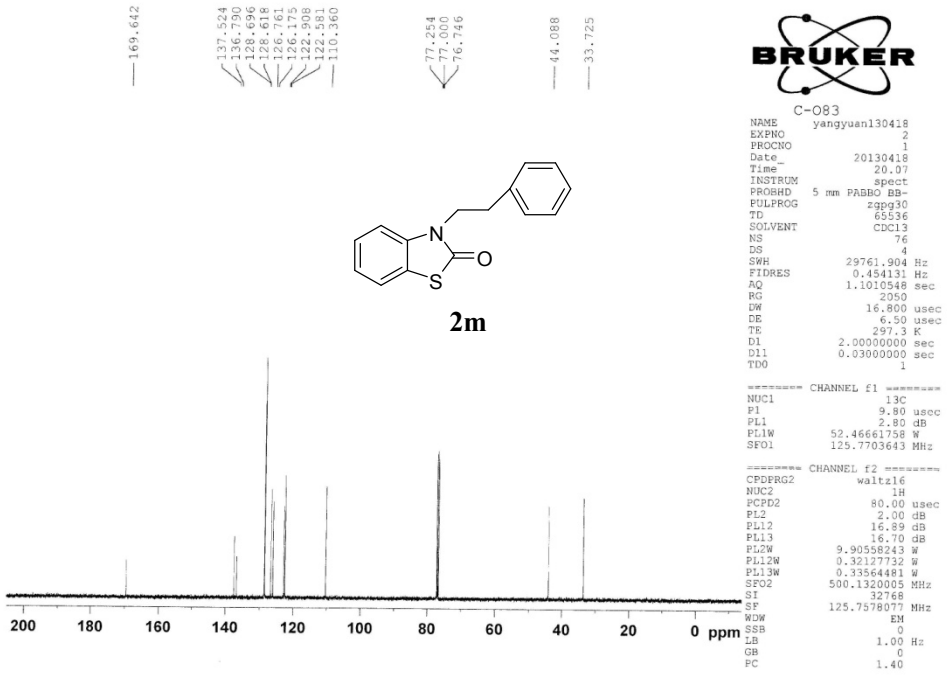
2m

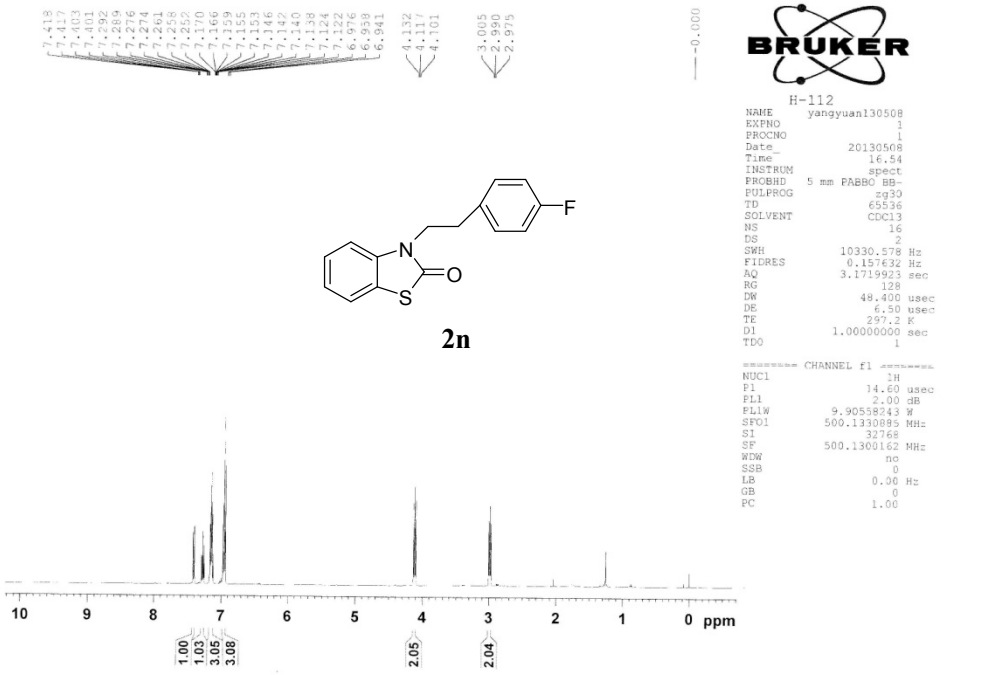


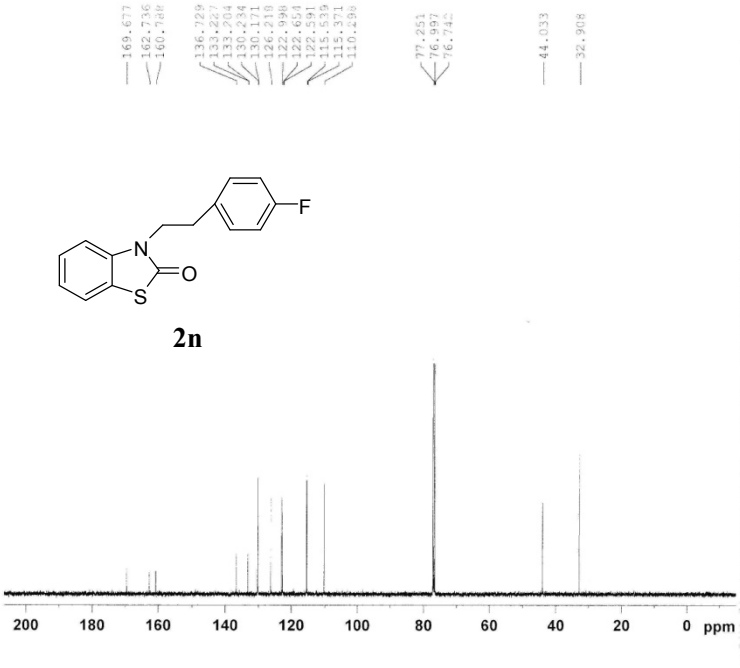
BRUKER

H-083

NAME	yangyuan130418
EXPNO	1
PROCNO	1
Date_	20130418
Time	20.04
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	50.8
DW	48.400 usec
DE	6.50 usec
TE	296.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300267 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00


169.642
137.524
136.790
128.696
128.618
126.761
126.175
122.988
122.581
110.360
77.254
77.000
76.746
44.088
33.725
BRUKER
C-083
NAME yangyuan130418
EXPNO 2
PROCNO 1
Date_ 20130418
Time 20.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 76
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 2.80 dB
PL1W 52.46661758 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 16.89 dB
PL13 16.70 dB
PL2W 9.90558243 W
PL12W 0.32127732 W
PL13W 0.33564481 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578077 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
O
S
2m
200
180
160
140
120
100
80
60
40
20
0 ppm


7.418
7.417
7.403
7.401
7.292
7.289
7.276
7.274
7.261
7.258
7.253
7.170
7.166
7.159
7.155
7.153
7.146
7.142
7.140
7.138
7.124
7.122
6.976
6.959
6.941
4.132
4.117
4.101
3.005
2.990
2.975
-0.000
BRUKER
H-112
NAME yangyuan130508
EXPNO 1
PROCNO 1
Date_ 20130508
Time 16.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719925 sec
RG 128
DW 48.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.60 usec
PL1 2.00 dB
PL1W 9.90558243 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300162 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
N
O
S
F
2n
10
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.03
3.05
3.08
2.05
2.04

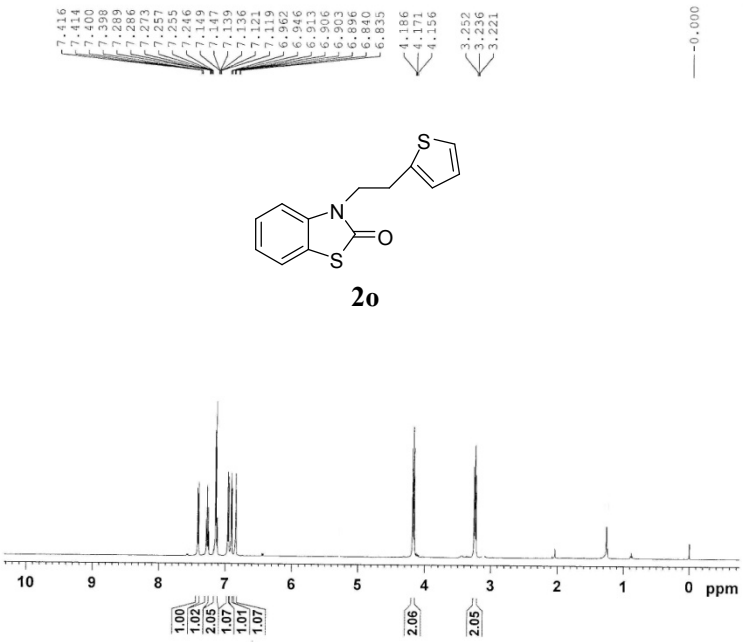


2n

BRUKER

C-112

NAME	yangyuan130508
EXPNO	2
PROCNO	1
Date_	20130508
Time	16.57
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	91
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	297.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578022 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

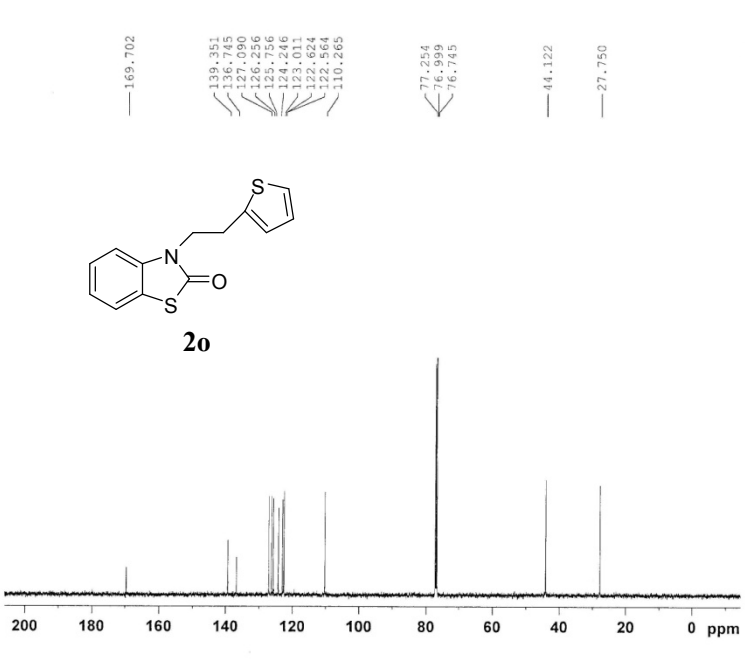


2o

BRUKER

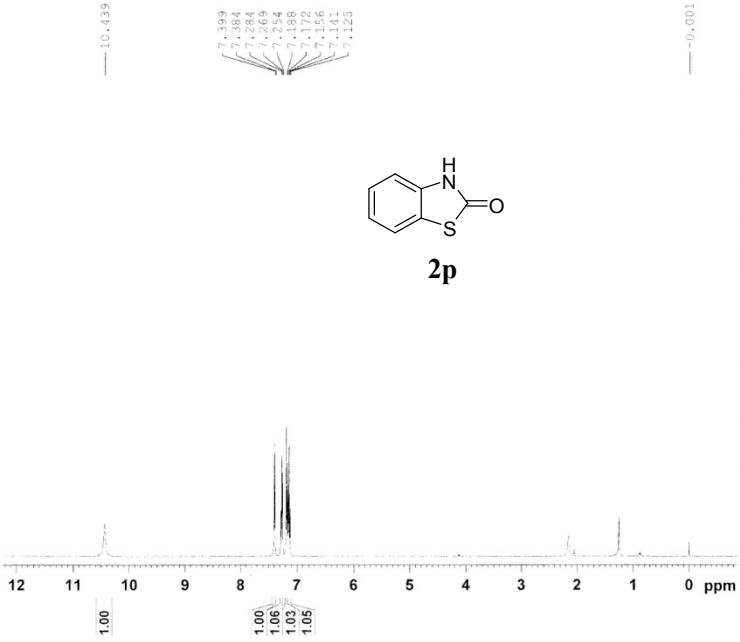
H-0128

NAME	yangyuan130513
EXPNO	3
PROCNO	1
Date_	20130513
Time	16.39
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	128
DW	48.400 usec
DE	6.50 usec
TE	297.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300204 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



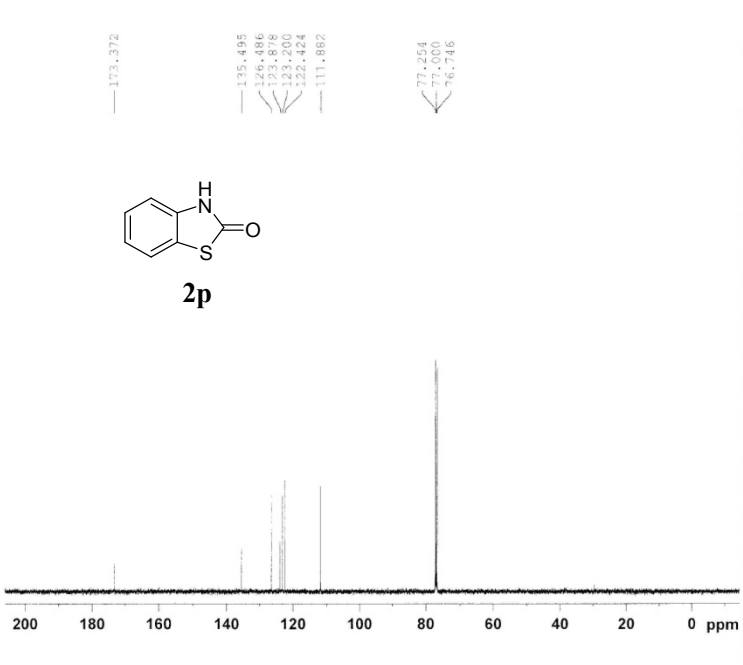
2o

```
C-O128
NAME            yangyuan130513
EXPNO                        4
PROCNO                       1
Date_                 20130513
Time                     16.42
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          72
DS                           4
SWH                  29761.904 Hz
FIDRES               0.454131 Hz
AQ                   1.1010548 sec
RG                        2050
DW                      16.800 usec
DE                        6.50 usec
TE                      298.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        13C
P1                         9.80 usec
PL1                        2.80 dB
PL1W                52.46661758 W
SFO1               125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     80.00 usec
PL2                        2.00 dB
PL12                      16.89 dB
PL13                      16.70 dB
PL2W                9.90558243 W
PL12W               0.32127732 W
PL13W               0.33564481 W
SFO2               500.1320005 MHz
SI                       32768
SF                 125.7578040 MHz
WDW                         EM
SSB                          0
LB                        2.00 Hz
GB                           0
PC                        1.40
```

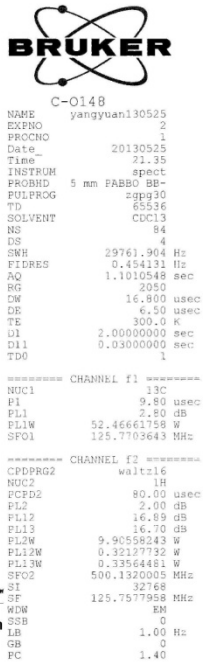


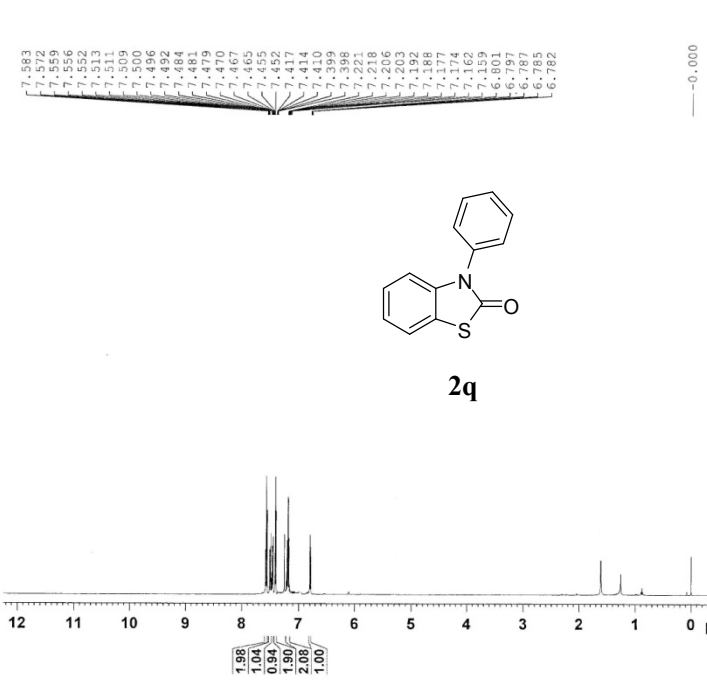
2p

```
H-O148
NAME            yangyuan130525
EXPNO                        1
PROCNO                       1
Date_                 20130525
Time                     21.32
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                  10330.578 Hz
FIDRES               0.157632 Hz
AQ                   3.1719923 sec
RG                         456
DW                      48.400 usec
DE                        6.50 usec
TE                      300.0 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                         1H
P1                        14.60 usec
PL1                        2.00 dB
PL1W                9.90558243 W
SFO1               500.1330885 MHz
SI                       32768
SF                 500.1300140 MHz
WDW                         no
SSB                          0
LB                        0.00 Hz
GB                           0
PC                        1.00
```


173.372
135.495
126.486
123.878
123.200
122.424
111.882
77.254
77.000
76.746
H
N
O
S
200
180
160
140
120
100
80
60
40
20
0 ppm

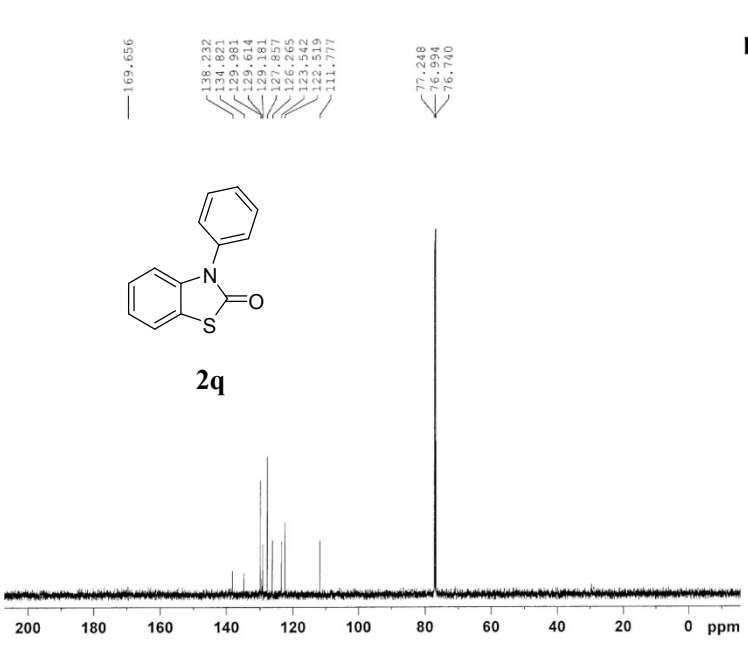
2p


BRUKER
C-0148
NAME yangyuan130525
EXPNO 2
PROCNO 1
Date_ 20130525
Time 21.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 84
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 2.80 dB
PL1W 52.46661758 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 16.89 dB
PL13 16.70 dB
PL2W 9.90558243 W
PL12W 0.32127732 W
PL13W 0.33564481 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577958 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.583
7.572
7.559
7.556
7.552
7.513
7.511
7.509
7.500
7.496
7.492
7.484
7.481
7.479
7.470
7.467
7.465
7.455
7.452
7.417
7.414
7.410
7.399
7.398
7.221
7.218
7.206
7.203
7.192
7.188
7.177
7.174
7.162
7.159
6.801
6.797
6.787
6.785
6.782
0.000
N
O
S
12
11
10
9
8
7
6
5
4
3
2
1
0 ppm
1.98
1.04
0.94
1.90
2.08
1.00

2q


BRUKER
H-J39
NAME yangyuan130627
EXPNO 1
PROCNO 1
Date_ 20120524
Time 10.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 256
DW 48.400 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.60 usec
PL1 2.00 dB
PL1W 9.90558243 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300159 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

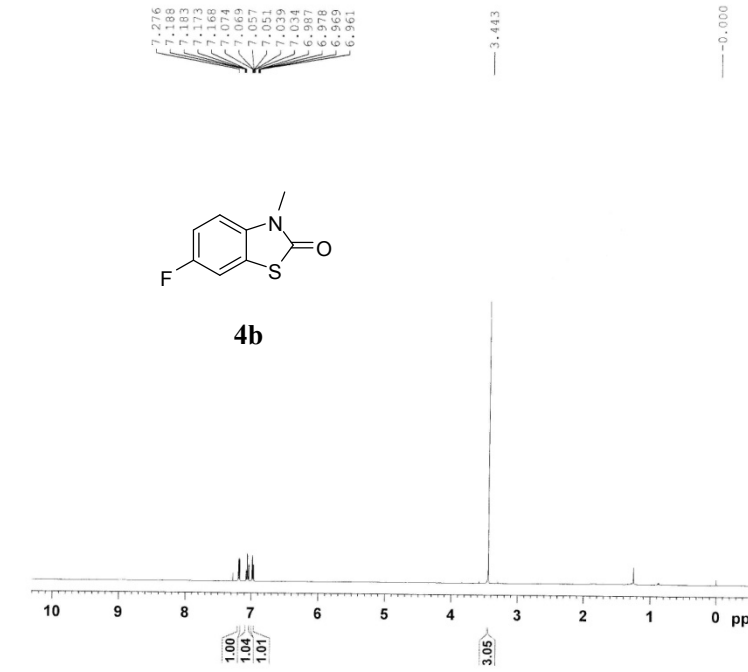


2q

BRUKER

C-J39

```
NAME            yangyuan130627
EXPNO                        2
PROCNO                       1
Date_             20120524
Time                     10.46
INSTRUM                  spect
PROBHD   5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        144
DS                          4
SWH                 29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010548 sec
RG                         2050
DW                       16.800 usec
DE                        6.50 usec
TE                       301.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                        9.80 usec
PL1                       2.80 dB
PL1W               52.46661758 W
SFO1              125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 usec
PL2                       2.00 dB
PL12                     16.89 dB
PL13                     16.70 dB
PL2W                9.90558243 W
PL12W               0.32127732 W
PL13W               0.33564481 W
SFO2              500.1320005 MHz
SI                        32768
SF                125.7577940 MHz
WDW                          EM
SSB                           0
LB                        1.00 Hz
GB                            0
PC                         1.40
```

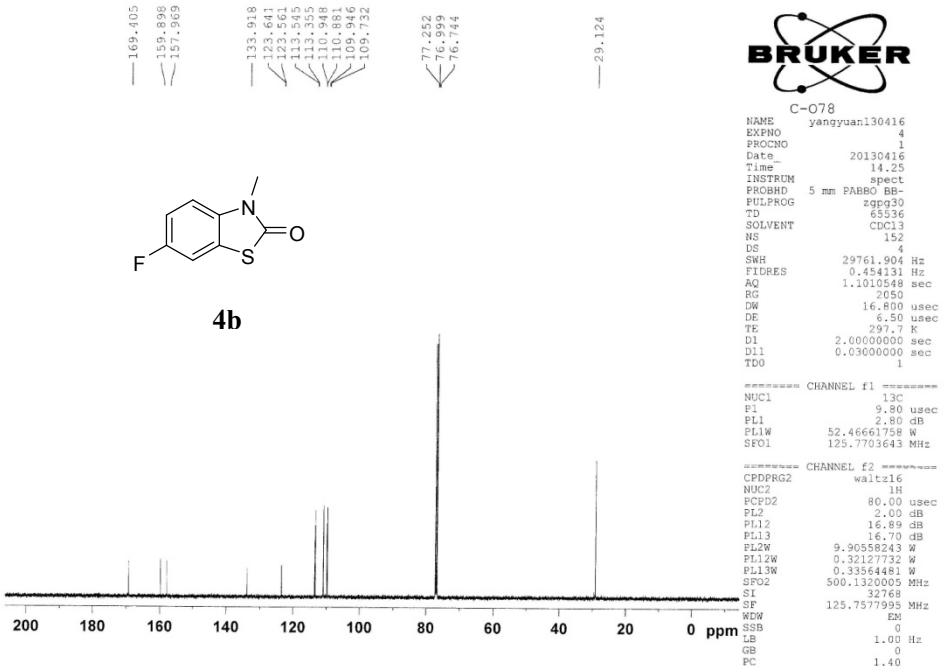


4b

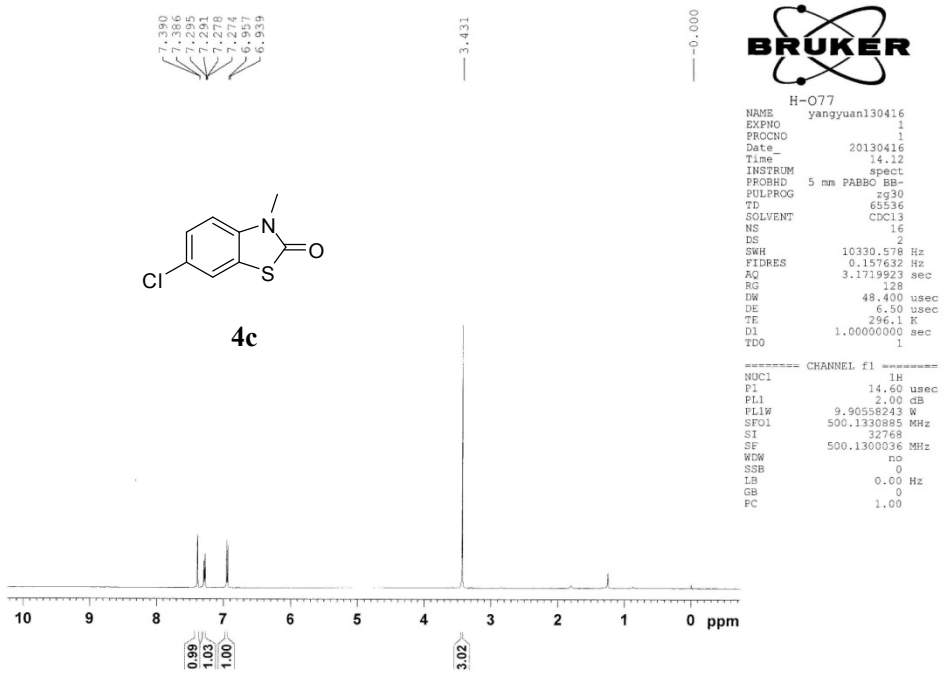
BRUKER

H-078

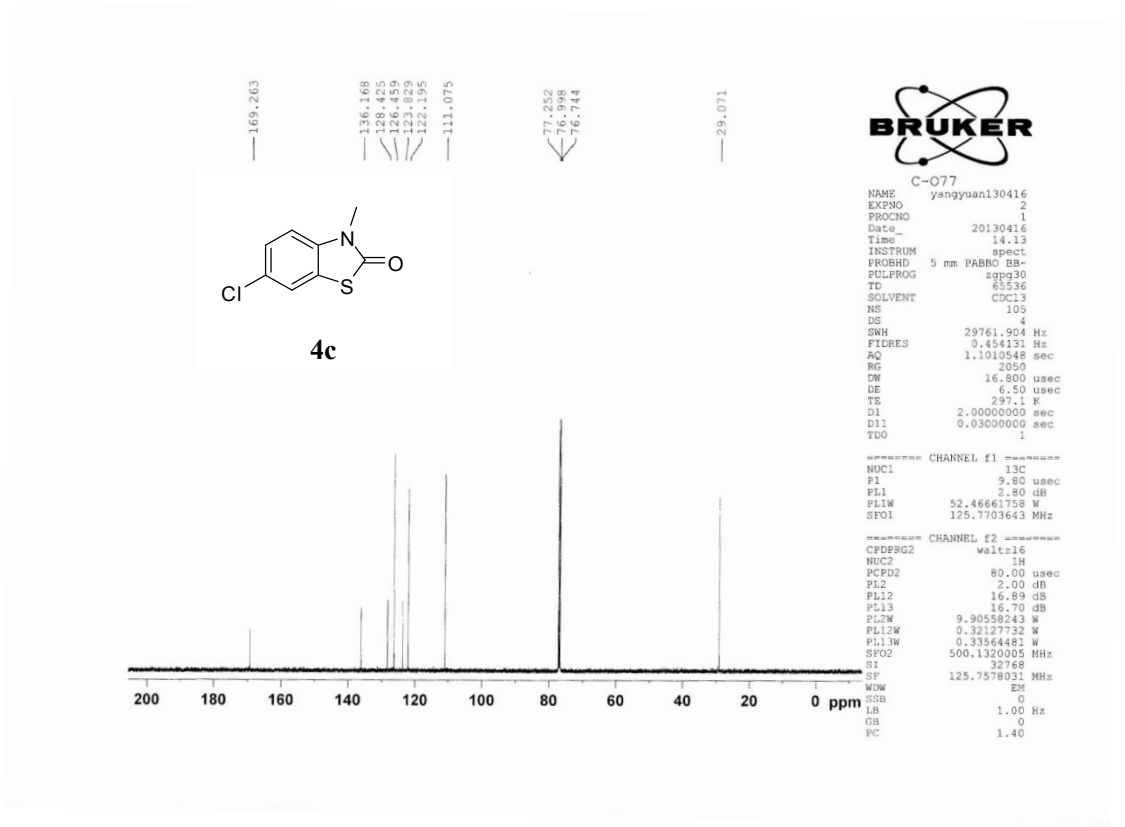
```
NAME            yangyuan130416
EXPNO                        3
PROCNO                       1
Date_             20130416
Time                     14.22
INSTRUM                  spect
PROBHD   5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                 10330.578 Hz
FIDRES                0.157632 Hz
AQ                    3.1719923 sec
RG                         144
DW                       48.400 usec
DE                        6.50 usec
TE                       296.5 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                       14.60 usec
PL1                       2.00 dB
PL1W                9.90558243 W
SFO1              500.1330885 MHz
SI                        32768
SF                500.1300046 MHz
WDW                          no
SSB                           0
LB                        0.00 Hz
GB                            0
PC                         1.00
```

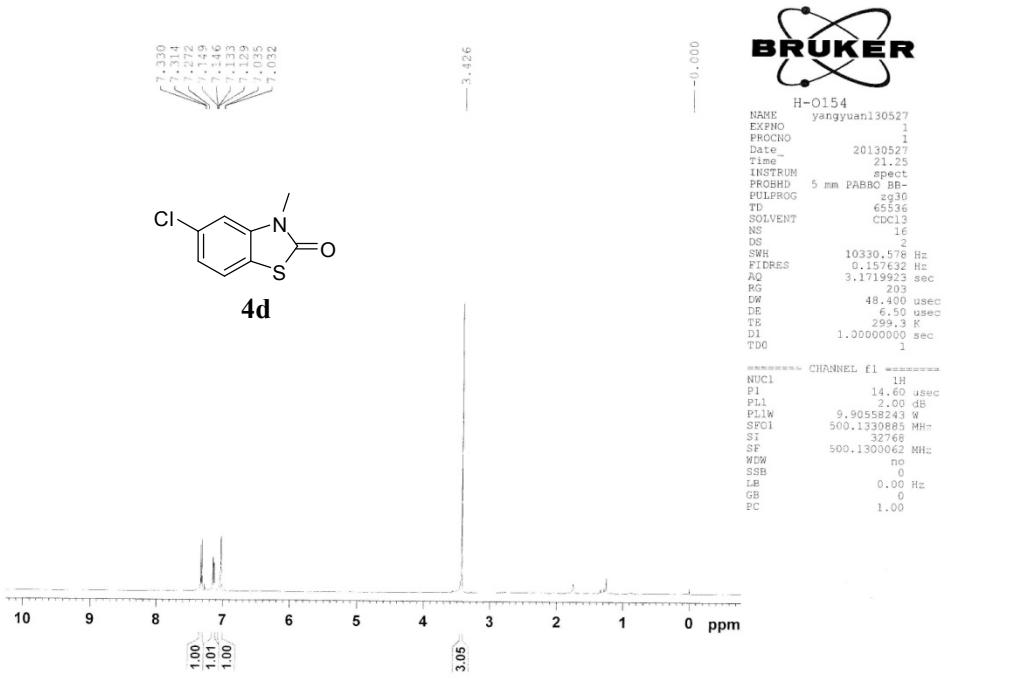

169.405
159.898
157.969
133.918
123.641
123.561
113.545
113.355
110.948
110.881
109.946
109.732
77.252
76.999
76.744
29.124
BRUKER
C-078
NAME yangyuan130416
EXPNO 4
PROCNO 1
Date_ 20130416
Time_ 14.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 152
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 297.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 2.80 dB
PL1W 52.46661758 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 16.89 dB
PL13 16.70 dB
PL2W 9.90558243 W
PL12W 0.32127732 W
PL13W 0.33564481 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577995 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
O
S
F
200 180 160 140 120 100 80 60 40 20 0 ppm

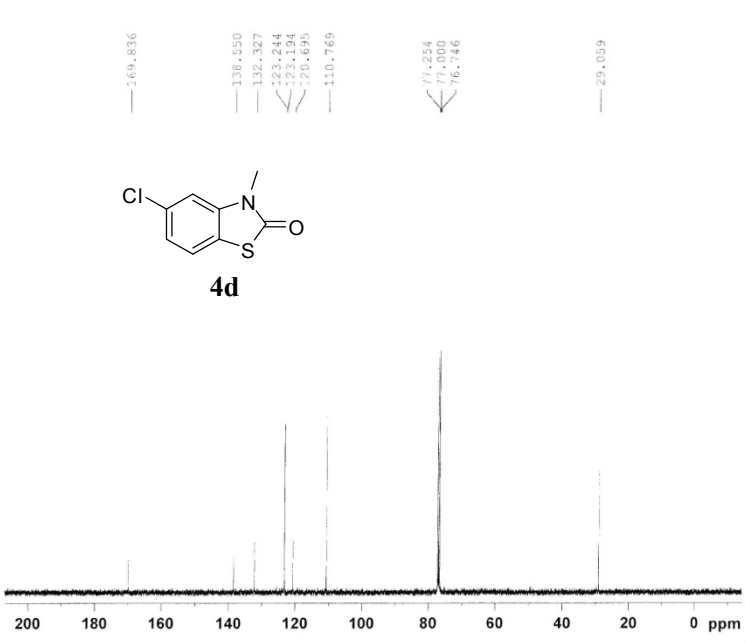
4b


7.390
7.386
7.295
7.291
7.278
7.274
6.957
6.939
3.431
0.000
BRUKER
H-077
NAME yangyuan130416
EXPNO 1
PROCNO 1
Date_ 20130416
Time_ 14.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 128
DW 48.400 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.60 usec
PL1 2.00 dB
PL1W 9.90558243 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300036 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
N
O
S
Cl
10 9 8 7 6 5 4 3 2 1 0 ppm
0.99
1.03
1.00
3.02

4c

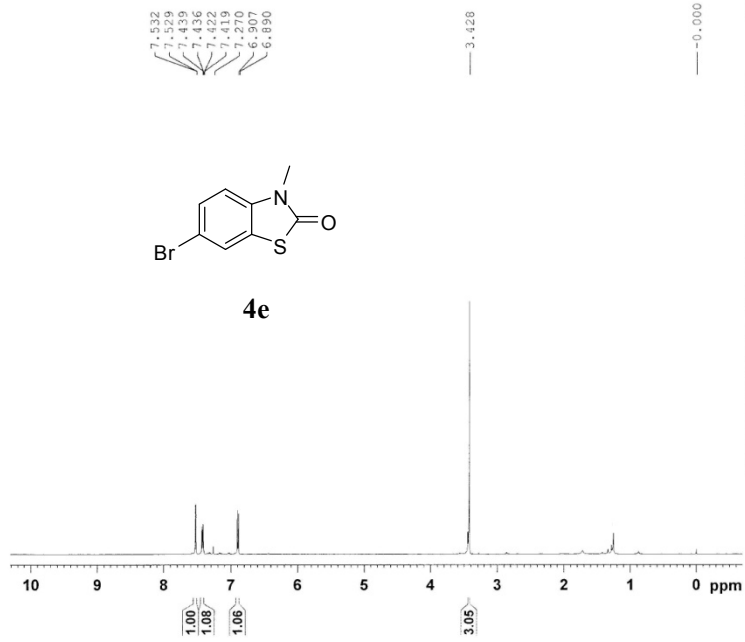

169.263
136.168
128.425
126.459
123.829
122.195
111.075
77.252
76.998
76.744
29.071
Cl
N
O
S
4c
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
C-077
NAME yangyuan130416
EXPNO 2
PROCNO 1
Date_ 20130416
Time 14.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 105
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 2.80 dB
PL1W 52.46661758 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 16.89 dB
PL13 16.70 dB
PL2W 9.90558243 W
PL12W 0.32127732 W
PL13W 0.33564481 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578031 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.330
7.314
7.272
7.149
7.146
7.133
7.129
7.035
7.032
3.426
0.000
Cl
N
O
S
4d
10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
1.01
1.00
3.05
BRUKER
H-0154
NAME yangyuan130527
EXPNO 1
PROCNO 1
Date_ 20130527
Time 21.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 203
DW 48.400 usec
DE 6.50 usec
TE 299.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.60 usec
PL1 2.00 dB
PL1W 9.90558243 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300062 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



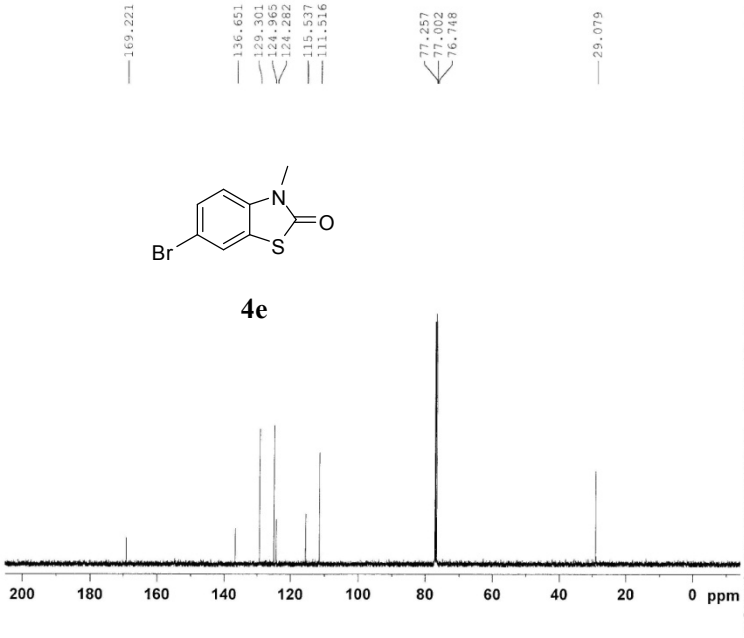
4d

```
BRUKER
C-0154
NAME            yangyuan130527
EXPNO                        2
PROCNO                       1
Date_             20130527
Time                  21.33
INSTRUM               spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      80
DS                       4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ                1.1010548 sec
RG                    2050
DW                  16.800 usec
DE                   6.50 usec
TE                  300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    13C
P1                    9.80 usec
PL1                   2.80 dB
PL1W          52.46661758 W
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   2.00 dB
PL12                 16.89 dB
PL13                 16.70 dB
PL2W           9.90558243 W
PL12W          0.32127732 W
PL13W          0.33564481 W
SFO2         500.1320005 MHz
SI                   32768
SF           125.7577995 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



4e

```
BRUKER
H-0182
NAME            yangyuan130607
EXPNO                        1
PROCNO                       1
Date_             20130607
Time                  11.11
INSTRUM               spect
PROBHD     5 mm PABBO BB-
PULPROG             zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH              10330.578 Hz
FIDRES            0.157632 Hz
AQ                3.1719923 sec
RG                     161
DW                  48.400 usec
DE                   6.50 usec
TE                  298.6 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.60 usec
PL1                   2.00 dB
PL1W           9.90558243 W
SFO1         500.1330885 MHz
SI                   32768
SF           500.1300071 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```

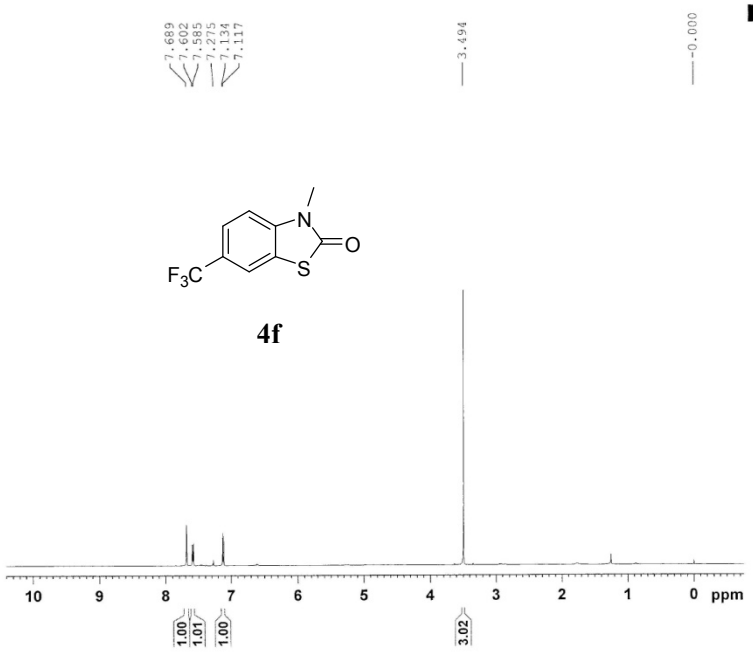


4e

BRUKER

C-0182

NAME	yangyuan130607
EXPNO	2
PROCNO	1
Date_	20130607
Time	11.13
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	81
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	299.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577986 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

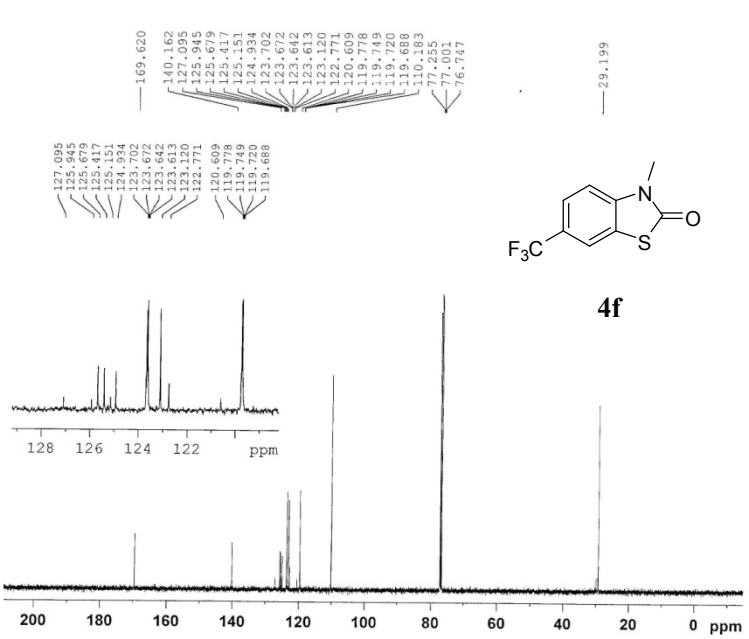


4f

BRUKER

H-081

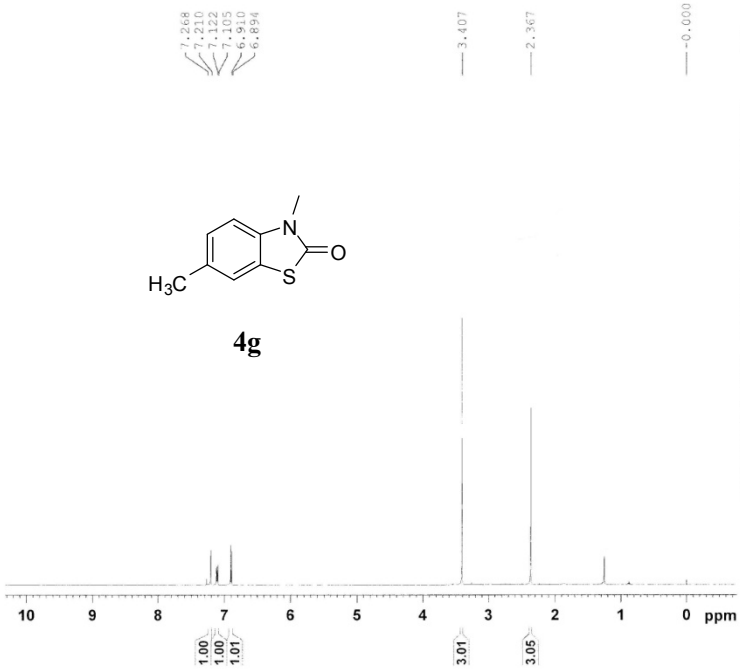
NAME	yangyuan130417
EXPNO	5
PROCNO	1
Date_	20130417
Time	15.48
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	287
DW	48.400 usec
DE	6.50 usec
TE	296.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300048 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



BRUKER

C-081

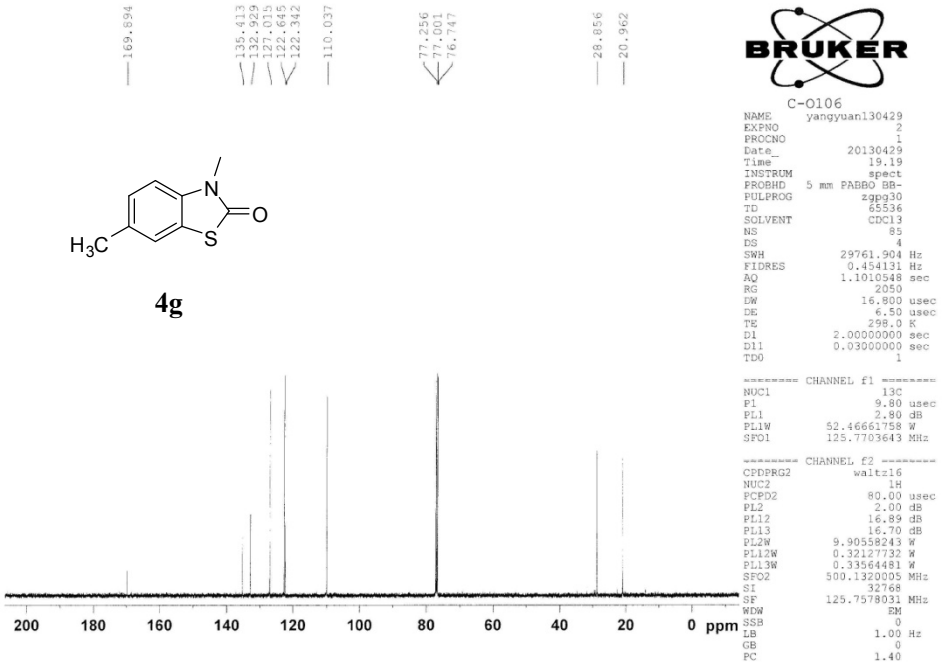
NAME	yangyuan130417
EXPNO	6
PROCNO	1
Date_	20130417
Time	15.51
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	124
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	297.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577977 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

H-0106

NAME	yangyuan130429
EXPNO	1
PROCNO	1
Date_	20130429
Time	19.16
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	128
DW	48.400 usec
DE	6.50 usec
TE	297.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300086 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

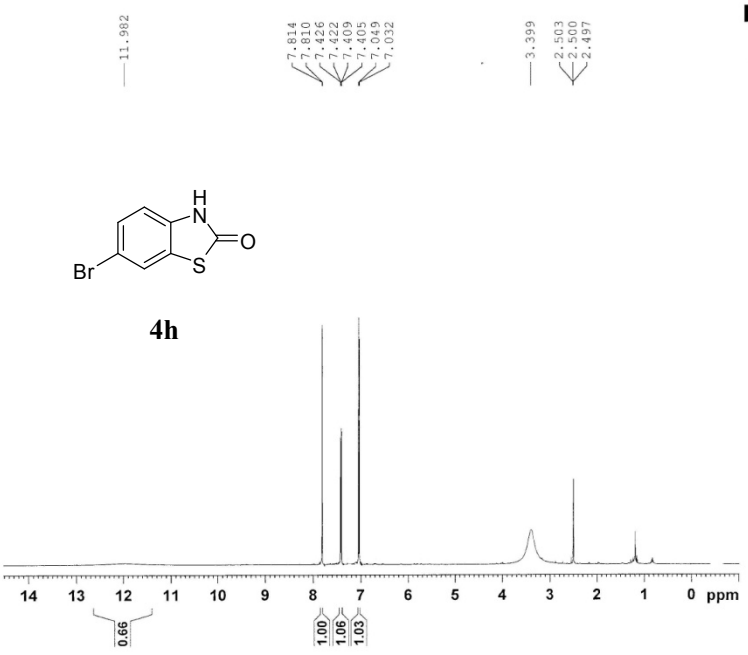


4g

BRUKER

C-0106

NAME	yangyuan130429
EXPNO	2
PROCNO	1
Date_	20130429
Time	19.19
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	85
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578031 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

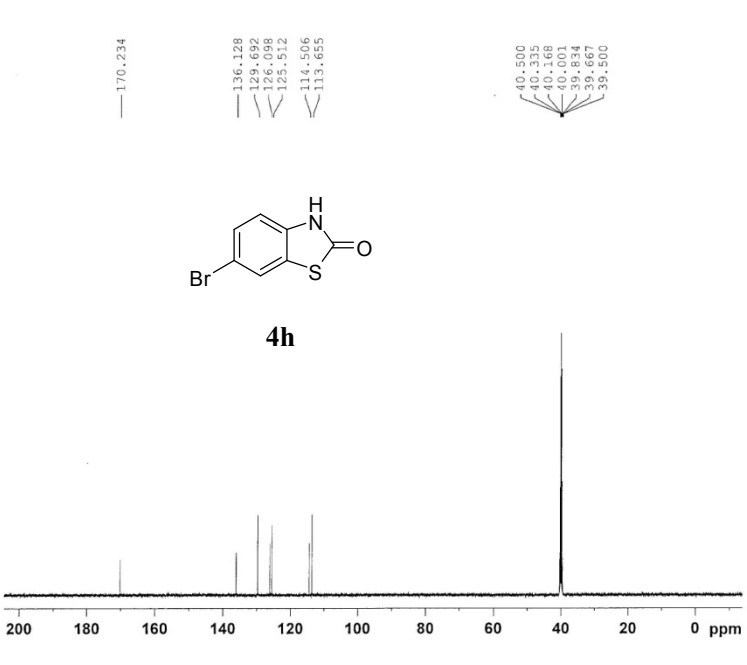


4h

BRUKER

H-J27

NAME	yangyuan130621
EXPNO	9
PROCNO	1
Date_	20130621
Time	16.34
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	456
DW	48.400 usec
DE	6.50 usec
TE	298.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300054 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

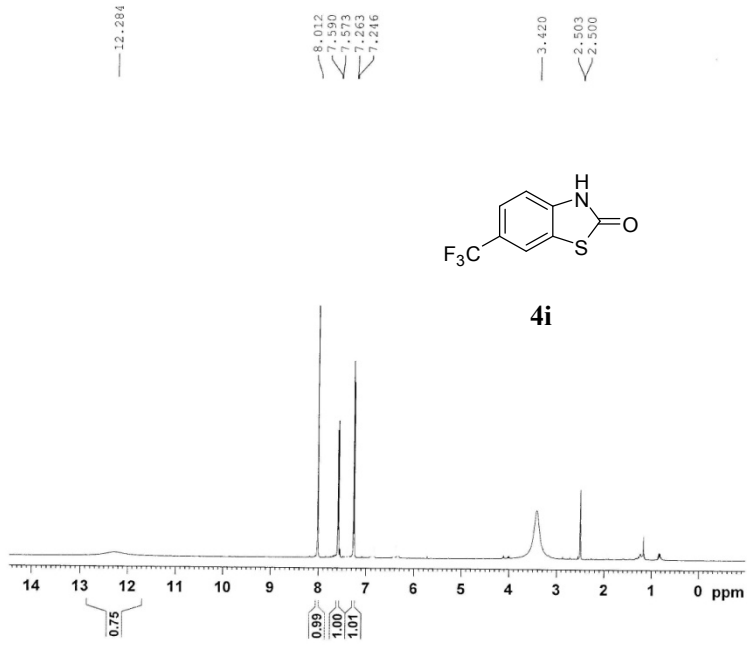


4h

BRUKER

C-J27

NAME	yangyuan130621
EXPNO	10
PROCNO	1
Date_	20130621
Time	16.37
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	80
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	299.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577832 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

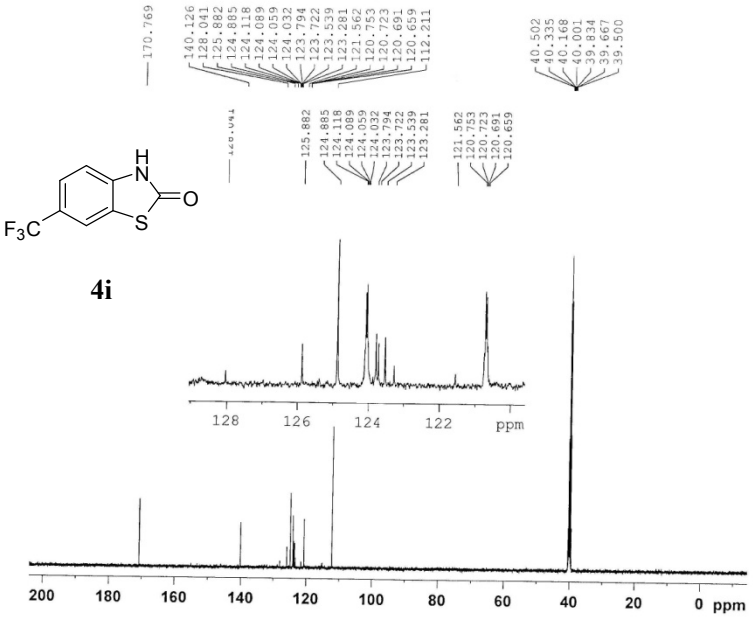


4i

BRUKER

H-J26

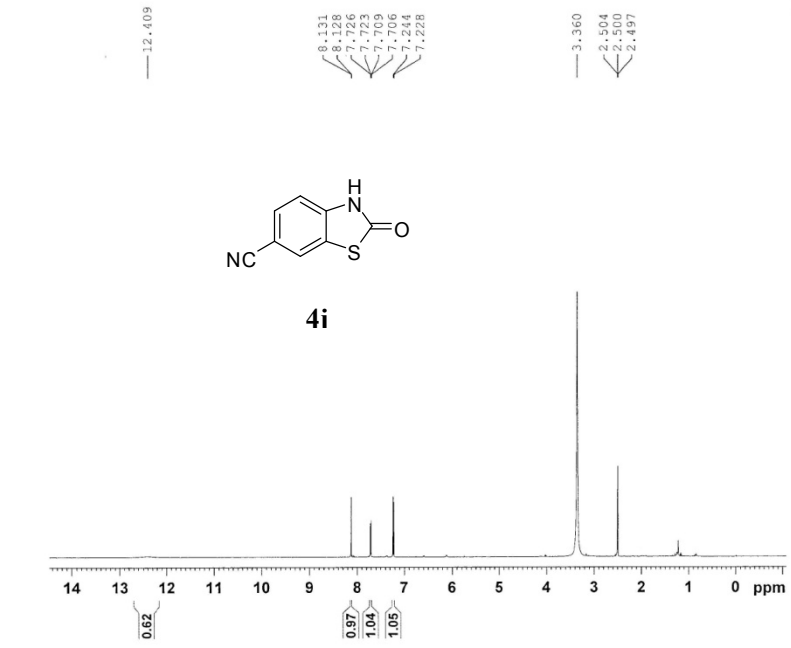
NAME	yangyuan130621
EXPNO	7
PROCNO	1
Date_	20130621
Time	16.21
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	362
DW	48.400 usec
DE	6.50 usec
TE	299.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300039 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



BRUKER

C-J26

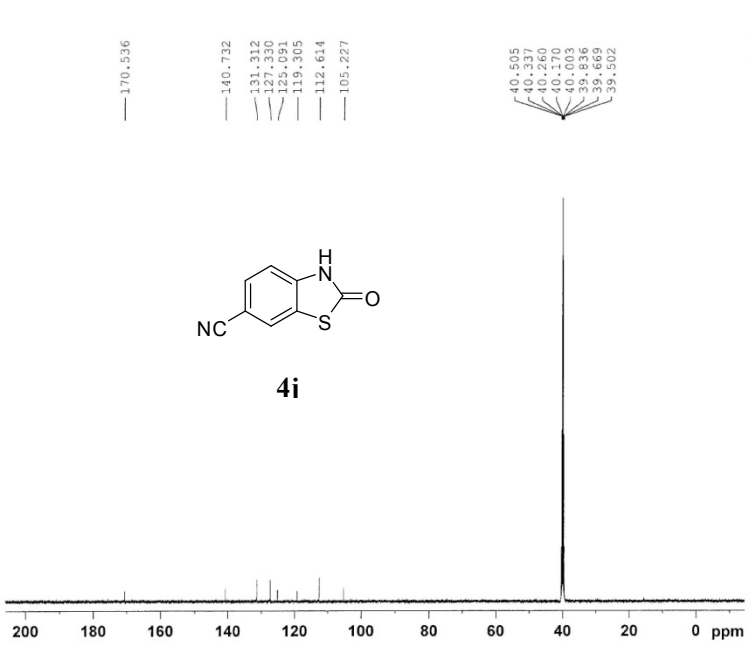
NAME	yangyuan130621
EXPNO	8
PROCNO	1
Date_	20130621
Time	16.23
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	140
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	299.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577777 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

H-J28

NAME	yangyuan130621
EXPNO	11
PROCNO	1
Date_	20130621
Time	16.48
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	575
DW	48.400 usec
DE	6.50 usec
TE	298.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300054 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

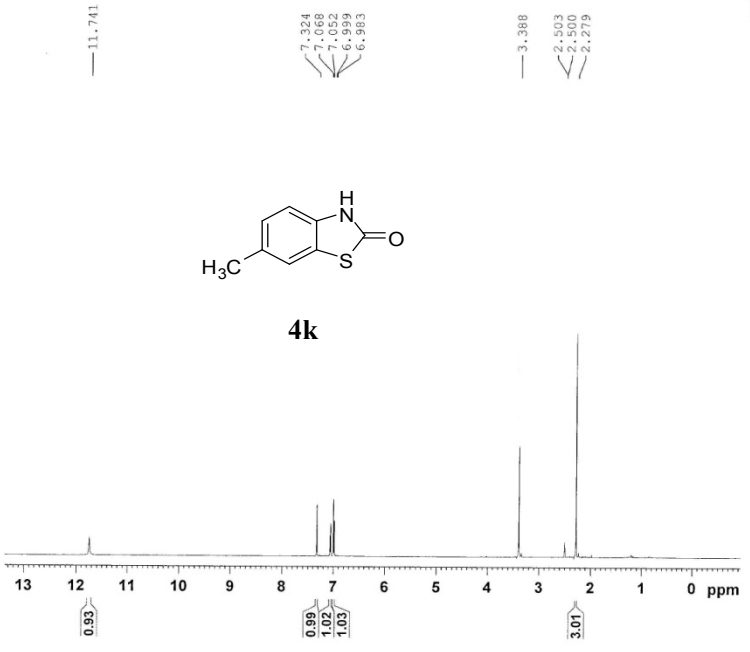


4i

BRUKER

C-J28

NAME	yangyuan130621
EXPNO	12
PROCNO	1
Date_	20130621
Time	16.50
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	200
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	299.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.80 usec
PL1	2.80 dB
PL1W	52.46661758 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.00 dB
PL12	16.89 dB
PL13	16.70 dB
PL2W	9.90558243 W
PL12W	0.32127732 W
PL13W	0.33564481 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577841 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

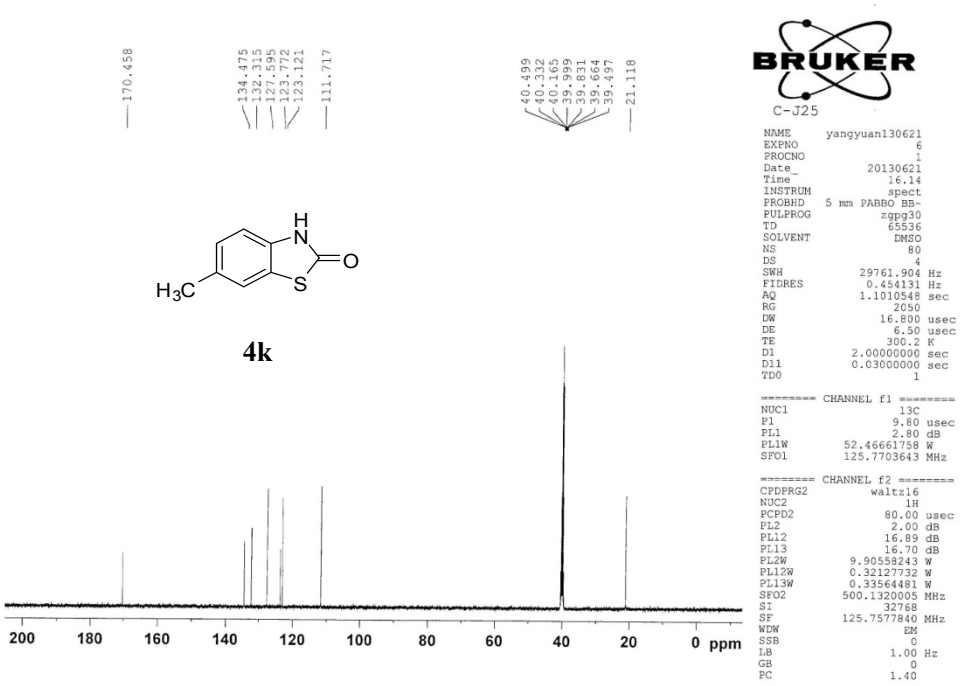


4k

BRUKER

H-J25

NAME	yangyuan130621
EXPNO	5
PROCNO	1
Date_	20130621
Time	16.11
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	362
DW	48.400 usec
DE	6.50 usec
TE	298.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.60 usec
PL1	2.00 dB
PL1W	9.90558243 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300040 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00


170.458
134.475
132.315
127.595
123.772
123.121
111.717
40.499
40.332
40.165
39.999
39.831
39.664
39.497
21.118
H
N
O
S
H3C
4k
200
180
160
140
120
100
80
60
40
20
0 ppm
BRUKER
C-J25
NAME yangyuan130621
EXPNO 6
PROCNO 1
Date_ 20130621
Time 16.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 80
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 2.80 dB
PL1W 52.46661758 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 16.89 dB
PL13 16.70 dB
PL2W 9.90558243 W
PL12W 0.32127732 W
PL13W 0.33564481 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577840 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40