

## Supporting Information

### **Yb(OTf)<sub>3</sub> catalyzed [3+2] annulations of D–A cyclopropanes with $\beta$ -oxodithioesters: a regioselective synthesis of tetrahydrothiophenes**

Shuwen Wang, Weisi Guo, Lirong Wen\* and Ming Li\*

*State Key Laboratory Base of Eco-Chemical Engineering, College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, Qingdao 266042, P. R. China.*

\*Corresponding author: E-mail address: wenlirong@qust.edu.cn; liming928@qust.edu.cn

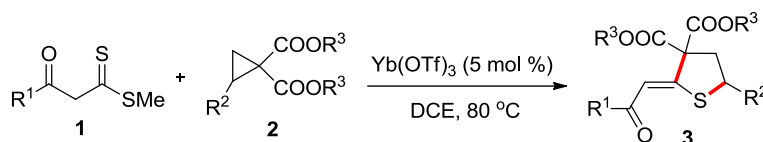
## Table of Contents

General methods.....	S2
General procedure for the preparation of compounds <b>3</b> .....	S2
X-ray structure of <b>3g</b> and <b>3j</b> .....	S3
Characterization of compounds <b>3</b> .....	S4
<sup>1</sup> H NMR and <sup>13</sup> C NMR Spectra of compounds <b>3</b> .....	S12

## General methods

All reagents and solvents were obtained from commercial suppliers and used without further purification. All reagents were weighed and handled in air at room temperature. Melting points were recorded on a RY-1 microscopic melting apparatus and uncorrected.  $^1\text{H}$  NMR spectra were recorded on 500 MHz and  $^{13}\text{C}$  NMR spectra were recorded on 125 MHz by using a Bruker Avance 500 spectrometer. Chemical shifts were reported in parts per million ( $\delta$ ) relative to tetramethylsilane (TMS). IR spectra were recorded on a Nicolet iS10 FT-IR spectrometer and only major peaks are reported in  $\text{cm}^{-1}$ . Mass spectra were performed on an Ultima Global spectrometer with an ESI source. The X-ray single-crystal diffraction was performed on Saturn 724+ instrument (for **3g**) and an Agilent Supernova CCD diffractometer (for **3j**). The  $\beta$ -oxodithioesters **1**<sup>1</sup> and D-A cyclopropanes **2**<sup>2</sup> were synthesized according to the literatures.

## General procedure for the preparation of tetrahydrothiophene derivatives **3**



A solution of  $\beta$ -oxodithioesters **1** (0.2 mmol) and D-A cyclopropanes **2** (0.2 mmol) was stirred in 1,2-DCE (2 mL) with  $\text{Yb(OTf)}_3$  (0.01 mmol) as the catalyst. The mixture was heated to reflux for appropriate reaction time. After completion of the reaction as indicated by TLC (petroleum ether/EtOAc, 15:1, v/v), the solvent was removed under vacuum, and the residue was purified by column chromatography (petroleum ether/EtOAc, 20:1, v/v) to afford products **3**.

(Z)-diethyl

2-(2-(3-chlorophenyl)-2-oxoethylidene)-5-phenyldihydrothiophene-3,3(2H)-dicarboxylate (3g)  
(CCDC: 993221)

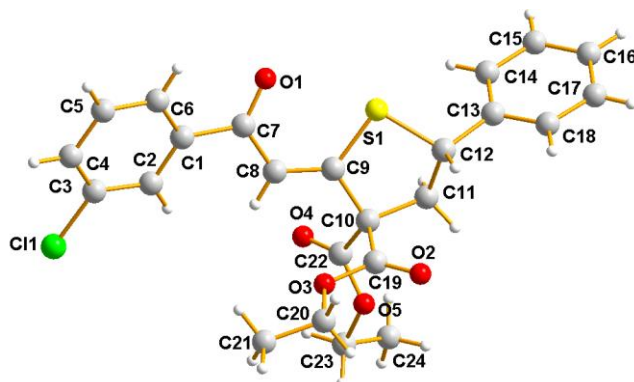


Figure S1. X-Ray Structure of 3g

(Z)-diethyl

2-(2-oxo-2-(thiophen-2-yl)ethylidene)-5-phenyldihydrothiophene-3,3(2H)-dicarboxylate (3j)  
(CCDC: 1041149)

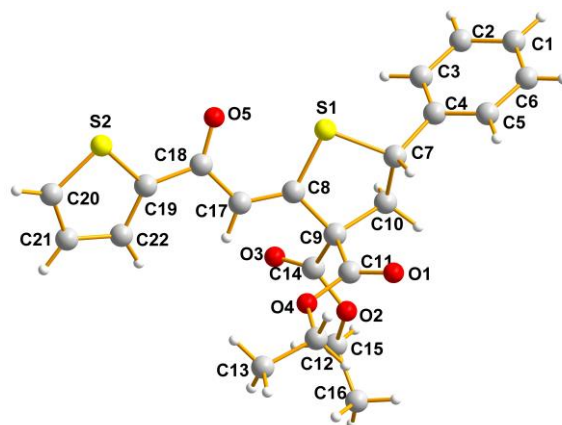
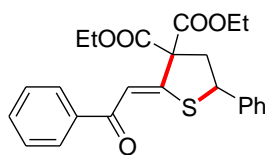


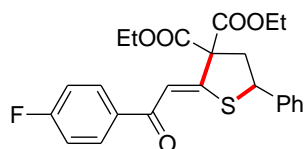
Figure S2. X-Ray Structure of 3j

**(Z)-diethyl 2-(2-oxo-2-phenylethylidene)-5-phenyldihydrothiophene-3,3(2H)-dicarboxylate (3a)**



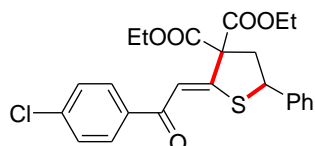
Pale yellow solid; yield 93 %; mp 92–93 °C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3061, 3030, 2981, 2937, 1733, 1636, 1579, 1541, 1231, 764, 699;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  = 7.97 (d,  $J$  = 7.4 Hz, 2H, ArH), 7.54 (t,  $J$  = 7.4 Hz, 1H, ArH), 7.45–7.49 (m, 5H, ArH and C=CH), 7.36 (t,  $J$  = 7.4 Hz, 2H, ArH), 7.31 (t,  $J$  = 7.2 Hz, 1H, ArH), 4.74 (dd,  $J$  = 5.0, 11.8 Hz, 1H,  $\text{CH}_2$ ), 4.26–4.37 (m, 4H,  $\text{OCH}_2\text{CH}_3$ ), 3.18 (dd,  $J$  = 5.0, 13.1 Hz, 1H,  $\text{CH}_2$ ); 2.74 (t,  $J$  = 12.4, 1H, CH); 1.30–1.34 (m, 6H,  $\text{OCH}_2\text{CH}_3$ );  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  = 188.4, 168.1, 167.7, 162.2, 138.2, 138.0, 132.4, 128.8, 128.6, 128.2, 128.0, 114.8, 70.4, 62.8, 62.6, 52.1, 45.3, 14.0; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for  $\text{C}_{24}\text{H}_{25}\text{O}_5\text{S}$  [(M + H)<sup>+</sup>], 425.1423; found, 425.1423.

**(Z)-diethyl 2-(2-(4-fluorophenyl)-2-oxoethylidene)-5-phenyldihydrothiophene-3,3(2H)-dicarboxylate (3b)**



Pale yellow solid; yield 65 %; mp 78–79 °C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3064, 2982, 2937, 1734, 1640, 1599, 1541, 1506, 1229, 855, 700;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  = 7.98–8.00 (m, 2H, ArH), 7.47 (d,  $J$  = 7.4 Hz, 2H, ArH); 7.41 (s, 1H, C=CH), 7.36 (t,  $J$  = 7.4 Hz, 2H, ArH), 7.31 (t,  $J$  = 7.3 Hz, 1H, ArH), 7.14 (t,  $J$  = 8.6 Hz, 2H, ArH), 4.75 (dd,  $J$  = 5.1, 11.8 Hz, 1H,  $\text{CH}_2$ ), 4.26–4.37 (m, 4H,  $\text{OCH}_2\text{CH}_3$ ), 3.18 (dd,  $J$  = 5.1, 13.1 Hz, 1H,  $\text{CH}_2$ ); 2.73 (t,  $J$  = 12.4, 1H, CH); 1.30–1.34 (m, 6H,  $\text{OCH}_2\text{CH}_3$ );  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  = 186.9, 168.0, 167.6, 165.3 (d,  $J_{\text{CF}}^1$  = 253.8 Hz), 162.6, 137.9, 134.6, 130.5 (d,  $J_{\text{CF}}^3$  = 8.4 Hz), 128.8, 128.2, 127.9, 115.6 (d,  $J_{\text{CF}}^2$  = 21.8 Hz), 114.5, 70.54, 62.9, 62.6, 52.2, 45.2, 14.0; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for  $\text{C}_{24}\text{H}_{24}\text{O}_5\text{SF}$  [(M + H)<sup>+</sup>], 443.1328; found, 443.1332.

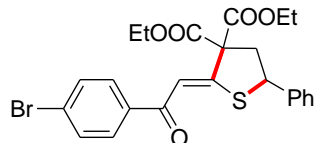
**(Z)-diethyl 2-(2-(4-chlorophenyl)-2-oxoethylidene)-5-phenyldihydrothiophene-3,3(2H)-dicarboxylate (3c)**



Yellow oil; yield 74 %; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3062, 3030, 2981, 2937, 1734, 1637, 1590, 1570, 1539, 1238, 852, 763, 699;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  = 7.91 (d,  $J$  = 8.5 Hz, 2H, ArH), 7.43–7.47 (m, 4H, ArH), 7.41 (s, 1H, C=CH), 7.36 (t,  $J$  = 7.4 Hz, 2H, ArH), 7.31 (t,  $J$  = 7.2 Hz, 1H, ArH), 4.75 (dd,  $J$  = 5.1, 11.8 Hz, 1H,  $\text{CH}_2$ ), 4.26–4.36 (m, 4H,  $\text{OCH}_2\text{CH}_3$ ), 3.18 (dd,  $J$  = 5.1, 13.1 Hz, 1H,  $\text{CH}_2$ ), 2.73 (t,  $J$  = 12.5, 1H, CH); 1.30–1.33 (m, 6H,  $\text{OCH}_2\text{CH}_3$ );  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  = 187.1, 168.0, 167.6, 163.0, 138.8, 137.8, 136.6, 129.4, 128.9, 128.8, 128.2, 128.0,

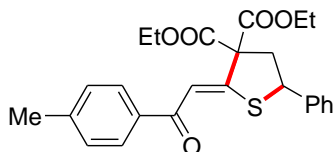
114.4, 70.5, 62.9, 62.7, 52.3, 45.3, 14.0; HRMS (ESI-TOF<sup>+</sup>): *m/z* calcd for C<sub>24</sub>H<sub>24</sub>O<sub>5</sub>SCl [(M + H)<sup>+</sup>], 459.1033; found, 459.1023.

**(Z)-diethyl 2-(2-(4-bromophenyl)-2-oxoethylidene)-5-phenylthiophene-3,3(2H)-dicarboxylate (3d)**



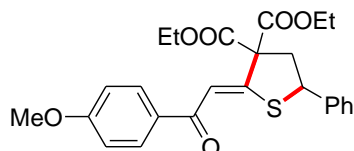
Pale yellow oil; yield 78 %; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3062, 3030, 2981, 2938, 1735, 1637, 1586, 1566, 1537, 1236, 813, 763, 699; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta$  = 7.83 (d, *J* = 8.4 Hz, 2H, ArH), 7.61 (d, *J* = 8.4 Hz, 2H, ArH), 7.46 (d, *J* = 7.4 Hz, 2H, ArH), 7.39 (s, 1H, C=CH), 7.36 (t, *J* = 7.4 Hz, 2H, ArH), 7.31 (t, *J* = 7.2 Hz, 1H, ArH), 4.75 (dd, *J* = 5.0, 11.6 Hz, 1H, CH<sub>2</sub>), 4.25–4.37 (m, 4H, OCH<sub>2</sub>CH<sub>3</sub>), 3.18 (dd, *J* = 5.0, 13.1 Hz, 1H, CH<sub>2</sub>), 2.73 (t, *J* = 12.5, 1H, CH), 1.30–1.33 (m, 6H, OCH<sub>2</sub>CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta$  = 171.1, 137.5, 137.4, 136.2, 132.7, 129.6, 128.8, 128.0, 126.6, 126.5, 125.2, 122.5, 119.6, 60.4, 53.4, 34.1, 31.6, 22.7, 22.3, 14.1; HRMS (ESI-TOF<sup>+</sup>): *m/z* calcd for C<sub>24</sub>H<sub>24</sub>O<sub>5</sub>SBr [(M + H)<sup>+</sup>], 503.0528; found, 503.0534.

**(Z)-diethyl 2-(2-(4-methylphenyl)-2-oxoethylidene)-5-phenylthiophene-3,3(2H)-dicarboxylate (3e)**



Yellow oil; yield 87 %; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3062, 3030, 2981, 2938, 1733, 1637, 1607, 1570, 1541, 1243, 861, 763, 699; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta$  = 7.88 (d, *J* = 8.2 Hz, 2H, ArH), 7.47 (d, *J* = 7.4 Hz, 2H, ArH), 7.44 (s, 1H, C=CH), 7.35 (t, *J* = 7.4 Hz, 2H, ArH), 7.27–7.31 (m, 2H, ArH), 4.73 (dd, *J* = 5.1, 11.8 Hz, 1H, CH<sub>2</sub>), 4.28–4.33 (m, 4H, OCH<sub>2</sub>CH<sub>3</sub>), 3.17 (dd, *J* = 5.1, 13.0 Hz, 1H, CH<sub>2</sub>); 2.74 (t, *J* = 12.4, 1H, CH); 2.41 (s, 3H, CH<sub>3</sub>), 1.29–1.34 (m, 6H, OCH<sub>2</sub>CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta$  = 188.03, 168.0, 167.7, 161.5, 143.1, 138.0, 135.6, 129.2, 128.7, 128.0, 127.9, 114.8, 70.3, 62.7, 62.6, 52.0, 45.2, 21.6, 14.0; HRMS (ESI-TOF<sup>+</sup>): *m/z* calcd for C<sub>25</sub>H<sub>27</sub>O<sub>5</sub>S [(M + H)<sup>+</sup>], 439.1579; found, 439.1568.

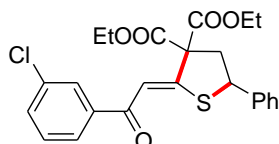
**(Z)-diethyl 2-(2-(4-methoxyphenyl)-2-oxoethylidene)-5-phenylthiophene-3,3(2H)-dicarboxylate (3f)**



Yellow solid; yield 95 %; mp 95–96 °C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3062, 3031, 2955, 2925, 1733, 1632, 1600, 1572, 1263, 1239, 856, 765, 697; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta$  = 7.97 (d, *J* = 8.8 Hz, 2H, ArH), 7.47 (d, *J* = 7.6 Hz, 2H, ArH), 7.42 (s, 1H, C=CH), 7.35 (t, *J* = 7.4 Hz, 2H, ArH),

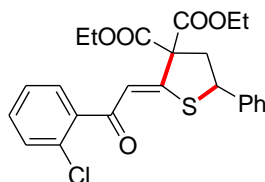
7.27–7.31 (m, 1H, ArH), 6.95 (d,  $J = 8.8$  Hz, 2H, ArH), 4.72 (dd,  $J = 4.6, 12.3$  Hz, 1H, CH<sub>2</sub>), 4.27–4.35 (m, 4H, OCH<sub>2</sub>CH<sub>3</sub>), 3.87 (s, 3H, OCH<sub>3</sub>), 3.16 (dd,  $J = 4.6, 13.1$  Hz, 1H, CH<sub>2</sub>), 2.73 (t,  $J = 12.6$ , 1H, CH), 1.30–1.34 (m, 6H, OCH<sub>2</sub>CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta = 187.1, 168.2, 167.8, 163.0, 161.0, 138.1, 131.2, 130.2, 128.7, 128.1, 128.0, 114.7, 113.8, 70.3, 62.7, 62.6, 55.4, 45.3, 29.7, 14.0$ ; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for C<sub>25</sub>H<sub>27</sub>O<sub>6</sub>S [(M + H)<sup>+</sup>], 455.1528; found, 455.1534.

**(Z)-diethyl 2-(2-(3-chlorophenyl)-2-oxoethylidene)-5-phenylthiophene-3,3(2H)-dicarboxylate (3g)**



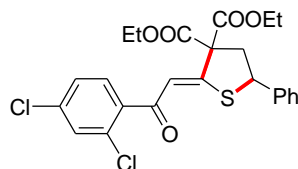
Pale yellow solid; yield 68 %; mp 99–101 °C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3062, 3027, 2980, 2936, 1755, 1731, 1639, 1594, 1568, 1541, 1278, 1234, 791, 702; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta = 7.93$  (s, 1H, ArH), 7.84 (d,  $J = 8.0$  Hz, 1H, ArH), 7.51 (d,  $J = 7.9$  Hz, 1H, ArH), 7.47 (d,  $J = 7.5$  Hz, 2H, ArH), 7.41 (t,  $J = 7.7$  Hz, 1H, ArH), 7.39 (s, 1H, C=CH), 7.35 (t,  $J = 7.3$  Hz, 2H, ArH), 7.30–7.33 (m, 1H, ArH), 4.76 (dd,  $J = 5.0, 11.9$  Hz, 1H, CH<sub>2</sub>), 4.28–4.37 (m, 4H, OCH<sub>2</sub>CH<sub>3</sub>), 3.19 (dd,  $J = 5.1, 13.1$  Hz, 1H, CH<sub>2</sub>), 2.74 (t,  $J = 12.3$ , 1H, CH), 1.31–1.35 (m, 6H, OCH<sub>2</sub>CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta = 187.0, 168.0, 167.5, 163.5, 140.0, 137.8, 134.8, 132.3, 130.0, 128.8, 128.2, 128.1, 128.0, 126.0, 114.4, 70.5, 62.9, 62.7, 52.3, 45.3, 14.0$ ; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for C<sub>24</sub>H<sub>24</sub>O<sub>5</sub>SCl [(M + H)<sup>+</sup>], 459.1033; found, 459.1045.

**(Z)-diethyl 2-(2-(2-chlorophenyl)-2-oxoethylidene)-5-phenylthiophene-3,3(2H)-dicarboxylate (3h)**



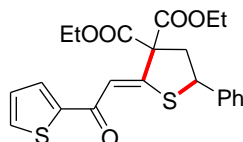
Yellow oil; yield 68%; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3063, 3030, 2981, 2938, 1734, 1638, 1590, 1537, 1245, 1223, 765, 699; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta = 7.59$  (dd,  $J = 1.6, 7.9$  Hz, 1H, ArH), 7.48 (d,  $J = 7.4$  Hz, 2H, ArH), 7.30–7.42 (m, 6H, ArH), 7.22 (s, 1H, C=CH), 4.76 (dd,  $J = 5.1, 11.8$  Hz, 1H, CH<sub>2</sub>), 4.21–4.35 (m, 4H, OCH<sub>2</sub>CH<sub>3</sub>), 3.18 (dd,  $J = 5.1, 13.1$  Hz, 1H, CH<sub>2</sub>); 2.77 (t,  $J = 12.5$ , 1H, CH); 1.26–1.32 (m, 6H, OCH<sub>2</sub>CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta = 189.7, 167.8, 167.5, 161.9, 139.3, 137.7, 131.6, 131.2, 130.3, 130.2, 128.8, 128.2, 127.9, 127.0, 118.6, 70.4, 62.8, 62.7, 52.3, 45.2, 14.0$ ; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for C<sub>24</sub>H<sub>24</sub>O<sub>5</sub>SCl [(M + H)<sup>+</sup>], 459.1033; found, 459.1042.

**(Z)-diethyl 2-(2-(2,4-dichlorophenyl)-2-oxoethylidene)-5-phenylthiophene-3,3(2H)-dicarboxylate (3i)**



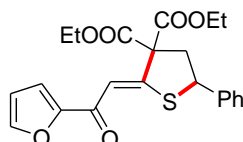
Yellow oil; yield 73%; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3064, 3030, 2982, 2937, 1755, 1734, 1637, 1584, 1537, 1246, 1220, 763, 699;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  = 7.55 (d,  $J$  = 8.3 Hz, 1H, ArH), 7.47 (d,  $J$  = 7.4 Hz, 2H, ArH), 7.43 (d,  $J$  = 1.8 Hz, 1H, ArH), 7.36 (t,  $J$  = 7.4 Hz, 2H, ArH), 7.30–7.33 (m, 2H, ArH), 7.19 (s, 1H, C=CH), 4.76 (dd,  $J$  = 5.0, 11.9 Hz, 1H,  $\text{CH}_2$ ), 4.21–4.37 (m, 4H,  $\text{OCH}_2\text{CH}_3$ ), 3.17 (dd,  $J$  = 5.0, 13.1 Hz, 1H,  $\text{CH}_2$ ); 2.76 (t,  $J$  = 12.5, 1H, CH); 1.29 (q,  $J$  = 7.6, 6H,  $\text{OCH}_2\text{CH}_3$ );  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  = 188.4, 167.8, 167.4, 162.8, 137.6, 137.2, 132.3, 131.3, 130.2, 128.8, 128.3, 127.9, 127.4, 118.2, 70.5, 62.9, 62.7, 52.5, 45.2, 14.0; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for  $\text{C}_{24}\text{H}_{24}\text{O}_5\text{SCl}_2$  [(M + H)<sup>+</sup>], 493.0643; found, 493.0652.

**(Z)-diethyl 2-(2-(thiophen-2-yl)ethylidene)-5-phenylthiophene-3,3(2H)-dicarboxylate (3j)**



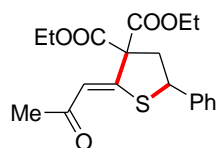
Pale yellow solid; yield 85 %; mp 80–81 °C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3111, 3056, 2991, 2901, 1759, 1731, 1619, 1550, 1516, 1494, 1238, 741, 700;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  = 7.73 (d,  $J$  = 3.7 Hz, 1H, ArH), 7.61 (d,  $J$  = 4.7 Hz, 1H, ArH), 7.46 (d,  $J$  = 7.7 Hz, 2H, ArH), 7.35 (t,  $J$  = 7.4 Hz, 2H, ArH), 7.30 (t,  $J$  = 7.1 Hz, 1H, ArH), 7.27 (s, 1H, C=CH), 7.14 (d,  $J$  = 4.5 Hz, 1H, ArH), 4.72 (dd,  $J$  = 5.3, 11.8 Hz, 1H,  $\text{CH}_2$ ), 4.26–4.38 (m, 4H,  $\text{OCH}_2\text{CH}_3$ ), 3.16 (dd,  $J$  = 5.2, 12.9 Hz, 1H,  $\text{CH}_2$ ), 2.73 (t,  $J$  = 12.4, 1H, CH), 1.31–1.35 (m, 6H,  $\text{OCH}_2\text{CH}_3$ );  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  = 181.0, 167.9, 167.6, 161.6, 145.5, 137.9, 133.0, 130.6, 128.8, 128.1, 128.0, 115.0, 70.3, 62.8, 62.6, 52.2, 45.3, 14.0; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for  $\text{C}_{22}\text{H}_{23}\text{O}_5\text{S}_2$  [(M + H)<sup>+</sup>], 431.0987; found, 431.0980.

**(Z)-diethyl 2-(2-(furan-2-yl)-2-oxoethylidene)-5-phenylthiophene-3,3(2H)-dicarboxylate (3k)**



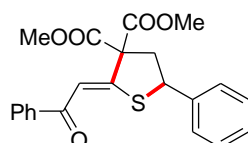
Pale yellow solid; yield 86 %; mp 82–83 °C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3138, 3063, 2982, 2938, 1781, 1732, 1634, 1573, 1247, 761, 699;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  = 7.57 (s, 1H, ArH), 7.46 (d,  $J$  = 7.7 Hz, 2H, ArH), 7.35 (t,  $J$  = 7.4 Hz, 2H, ArH), 7.29–7.31 (m, 2H, ArH), 7.21 (d,  $J$  = 3.3 Hz, 1H, ArH), 6.54 (t,  $J$  = 7.7 Hz, 1H, ArH), 4.72 (dd,  $J$  = 5.0, 11.9 Hz, 1H,  $\text{CH}_2$ ), 4.26–4.38 (m, 4H,  $\text{OCH}_2\text{CH}_3$ ), 3.15 (dd,  $J$  = 5.0, 13.1 Hz, 1H,  $\text{CH}_2$ ), 2.74 (t,  $J$  = 12.5, 1H, CH), 1.30–1.35 (m, 6H,  $\text{OCH}_2\text{CH}_3$ );  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  = 177.3, 167.9, 167.7, 161.8, 153.8, 145.7, 137.9, 128.8, 128.2, 128.0, 116.1, 114.5, 112.5, 70.4, 62.8, 62.7, 52.3, 45.3, 14.0; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for  $\text{C}_{22}\text{H}_{23}\text{O}_6\text{S}$  [(M + H)<sup>+</sup>], 415.1215; found, 415.1228.

**(Z)-diethyl 2-(2-oxopropylidene)-5-phenyldihydrothiophene-3,3(2H)-dicarboxylate (3l)**



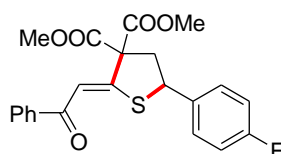
Yellow oil; yield 56 %; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3445, 2987, 2930, 1747, 1720, 1670, 1557, 1495, 1456, 1271, 1256, 770, 709;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  = 7.43 (d,  $J$  = 7.4 Hz, 2H, ArH), 7.34 (t,  $J$  = 7.4 Hz, 2H, ArH), 7.29 (t,  $J$  = 7.2 Hz, 1H, ArH), 6.65 (s, 1H, CH=C), 4.65 (dd,  $J$  = 5.0, 11.9 Hz, 1H,  $\text{CH}_2$ ), 4.25–4.33 (m, 4H,  $\text{OCH}_2\text{CH}_3$ ), 3.11 (dd,  $J$  = 5.0, 13.0 Hz, 1H,  $\text{CH}_2$ ); 2.69 (t,  $J$  = 12.4 Hz, 1H, CH); 2.28 (s, 3H,  $\text{CH}_3$ ), 1.29–1.34 (m, 6H,  $\text{OCH}_2\text{CH}_3$ );  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  = 195.9, 168.0, 167.7, 159.6, 137.9, 128.8, 128.2, 127.9, 118.3, 70.1, 62.7, 62.6, 52.0, 45.2, 30.3, 14.0; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for  $\text{C}_{19}\text{H}_{23}\text{O}_5\text{S}$  [(M + H)<sup>+</sup>], 363.1266; found, 363.1265.

**(Z)-dimethyl 2-(2-oxo-2-phenylethylidene)-5-phenyldihydrothiophene-3,3(2H)-dicarboxylate (3m)**



Pale yellow solid; yield 91 %; mp 116–117 °C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3060, 2951, 1734, 1634, 1597, 1535, 1232, 874, 771, 703;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  = 7.97 (d,  $J$  = 7.4 Hz, 2H, ArH), 7.54 (t,  $J$  = 7.3 Hz, 1H, ArH), 7.46–7.49 (m, 4H, ArH), 7.41 (s, 1H, CH=C), 7.36 (t,  $J$  = 7.4 Hz, 2H, ArH), 7.31 (t,  $J$  = 7.2 Hz, 1H, ArH), 4.73 (dd,  $J$  = 5.0, 11.8 Hz, 1H,  $\text{CH}_2$ ), 3.85 (s, 6H,  $\text{OCH}_3$ ), 3.18 (dd,  $J$  = 5.1, 13.0 Hz, 1H,  $\text{CH}_2$ ); 2.76 (t,  $J$  = 12.4, 1H, CH);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  = 188.4, 168.6, 168.2, 161.9, 138.2, 137.8, 132.4, 128.8, 128.6, 128.2, 128.0, 127.9, 114.9, 70.3, 53.7, 53.5, 52.2, 45.3; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for  $\text{C}_{22}\text{H}_{21}\text{O}_5\text{S}$  [(M + H)<sup>+</sup>], 397.1110; found, 397.1115.

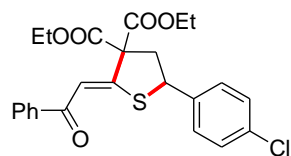
**(Z)-dimethyl 5-(4-fluorophenyl)-2-(2-oxo-2-phenylethylidene)dihydrothiophene-3,3(2H)-dicarboxylate (3n)**



Pale yellow solid; yield 85%; mp 78–79 °C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3067, 2954, 1742, 1731, 1636, 1597, 1549, 1508, 1257, 1231, 865, 764, 706;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  = 7.97 (d,  $J$  = 7.4 Hz, 2H, ArH), 7.55 (t,  $J$  = 7.3 Hz, 1H, ArH), 7.42–7.49 (m, 5H, ArH), 7.04 (d,  $J$  = 8.6 Hz, 2H, ArH), 4.71 (dd,  $J$  = 5.0, 11.8 Hz, 1H,  $\text{CH}_2$ ), 3.86 (s,  $\text{OCH}_3$ ), 3.85 (s,  $\text{OCH}_3$ ), 3.16 (dd,  $J$  = 5.0, 13.0 Hz, 1H,  $\text{CH}_2$ ), 2.74 (t,  $J$  = 12.4 Hz, 1H, CH);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  = 188.4, 168.5, 168.1, 161.5, 138.0, 133.6, 132.5, 129.5 (d,  $J_{\text{CF}}^3$  = 7.5 Hz), 128.6, 128.0, 115.7 (d,  $J_{\text{CF}}^2$  = 21.6 Hz), 115.0, 70.2, 53.8, 53.5, 51.3, 45.4; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for  $\text{C}_{22}\text{H}_{20}\text{O}_5\text{SF}$  [(M + H)<sup>+</sup>], 415.1015; found, 415.1025.

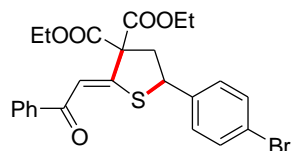


**(Z)-diethyl 5-(4-chlorophenyl)-2-(2-oxo-2-phenylethylidene)dihydrothiophene-3,3(2H)-dicarboxylate (3o)**



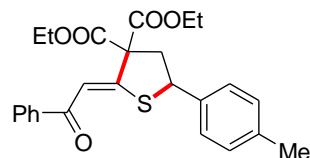
Pale yellow solid; yield 67%; mp 120–121 °C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3060, 2986, 2926, 1754, 1728, 1634, 1579, 1541, 1231, 861, 773, 692;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  = 7.97 (d,  $J$  = 7.55 Hz, 1H, ArH), 7.48–7.49 (m, 3H, ArH), 7.41 (d,  $J$  = 8.5 Hz, 2H, ArH), 7.33 (d,  $J$  = 8.5 Hz, 2H, ArH), 4.71 (dd,  $J$  = 5.1, 11.8 Hz, 1H,  $\text{CH}_2$ ), 4.25–4.37 (m, 4H,  $\text{OCH}_2\text{CH}_3$ ), 3.16 (dd,  $J$  = 5.0, 13.0 Hz, 1H,  $\text{CH}_2$ ); 2.67 (t,  $J$  = 12.4 Hz, 1H, CH), 1.29–1.34 (m, 6H,  $\text{OCH}_2\text{CH}_3$ );  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  = 188.4, 167.9, 167.5, 161.7, 138.1, 133.6, 132.5, 129.3, 1289, 128.6, 128.0, 115.7, 115.0, 70.3, 62.8, 62.7, 51.4, 45.3, 14.0; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for  $\text{C}_{24}\text{H}_{24}\text{O}_5\text{SCl}$  [(M + H)<sup>+</sup>], 459.1033; found, 459.1035.

**(Z)-diethyl 5-(4-bromophenyl)-2-(2-oxo-2-phenylethylidene)dihydrothiophene-3,3(2H)-dicarboxylate (3p)**



Yellow solid; yield 62 %; mp 112–113 °C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3062, 2983, 2935, 1753, 1728, 1634, 1596, 1579, 1542, 1489, 1250, 1232, 823, 772, 698;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  = 7.97 (d,  $J$  = 7.3 Hz, 2H, ArH), 7.55 (t,  $J$  = 7.2 Hz, 1H, ArH), 7.48 (t,  $J$  = 8.7 Hz, 5H, ArH), 7.35 (d,  $J$  = 8.5 Hz, 2H, ArH), 4.69 (dd,  $J$  = 5.0, 11.8 Hz, 1H,  $\text{CH}_2$ ), 4.25–4.36 (m, 4H,  $\text{OCH}_2\text{CH}_3$ ), 3.16 (dd,  $J$  = 5.0, 12.9 Hz, 1H,  $\text{CH}_2$ ); 2.66 (t,  $J$  = 12.4 Hz, 1H, CH); 1.32 (q,  $J$  = 7.3 Hz, 6H,  $\text{OCH}_2\text{CH}_3$ );  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  = 188.5, 167.9, 167.5, 161.7, 138.1, 137.1, 132.5, 131.9, 129.7, 128.6, 128.0, 122.0, 115.1, 70.3, 62.9, 62.7, 51.4, 45.2, 14.0; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for  $\text{C}_{24}\text{H}_{24}\text{O}_5\text{SBr}$  [(M + H)<sup>+</sup>], 503.0528; found, 503.0516.

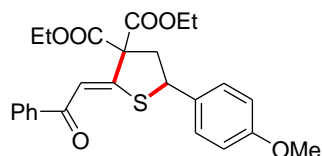
**(Z)-diethyl 2-(2-oxo-2-phenylethylidene)-5-p-tolyldihydrothiophene-3,3(2H)-dicarboxylate (3q)**



Yellow solid; yield 90 %; mp 91–92 °C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3083, 3052, 2984, 2925, 1752, 1728, 1635, 1596, 1541, 1365, 1269, 1230, 817, 776, 692;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  = 7.97 (d,  $J$  = 7.3 Hz, 2H, ArH), 7.54 (t,  $J$  = 7.3 Hz, 1H, ArH), 7.47 (t,  $J$  = 7.6 Hz, 2H, ArH), 7.44 (s, 1H, CH=C), 7.36 (d,  $J$  = 8.0 Hz, 2H, ArH), 7.16 (d,  $J$  = 7.9 Hz, 2H, ArH), 4.71 (dd,  $J$  = 5.0, 11.9 Hz, 1H,  $\text{CH}_2$ ), 4.27–4.37 (m, 4H,  $\text{OCH}_2\text{CH}_3$ ), 3.14 (dd,  $J$  = 5.1, 13.1 Hz, 1H,  $\text{CH}_2$ ); 2.72 (t,  $J$  = 12.7 Hz, 1H, CH), 2.35 (s, 1H,  $\text{CH}_3$ ), 1.32 (q,  $J$  = 6.8 Hz, 6H,  $\text{OCH}_2\text{CH}_3$ );  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  = 188.4,

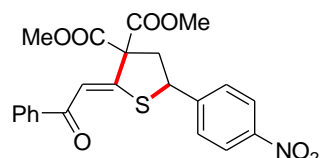
168.1, 167.7, 162.4, 160.8, 138.3, 138.0, 134.9, 132.4, 129.4, 128.6, 128.0, 127.8, 70.4, 62.8, 62.6, 60.0, 52.0, 45.4, 21.1, 14.0; HRMS (ESI-TOF<sup>+</sup>): *m/z* calcd for C<sub>25</sub>H<sub>27</sub>O<sub>5</sub>S [(M + H)<sup>+</sup>], 439.1579; found, 439.1582.

**(Z)-diethyl 5-(4-methoxyphenyl)-2-(2-oxo-2-phenylethylidene)dihydrothiophene-3,3(2H)-dicarboxylate (3r)**



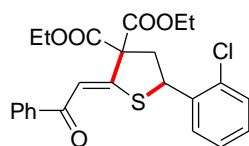
Yellow solid; yield 90%; mp 87–88 °C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3083, 2980, 2835, 1740, 1726, 1634, 1539, 1240, 827, 779, 702; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta$  = 7.97 (d, *J* = 7.5 Hz, 2H, ArH), 7.54 (t, *J* = 7.2 Hz, 1H, ArH), 7.47 (t, *J* = 7.5 Hz, 2H, ArH), 7.43 (s, 1H, CH=C), 7.39 (d, *J* = 8.4 Hz, 2H, ArH), 6.88 (d, *J* = 8.4 Hz, 2H, ArH), 4.71 (dd, *J* = 4.8, 11.8 Hz, 1H, CH<sub>2</sub>), 4.26–4.34 (m, 4H, OCH<sub>2</sub>CH<sub>3</sub>), 3.81 (s, 1H, OCH<sub>3</sub>), 3.13 (dd, *J* = 4.8, 13.0 Hz, 1H, CH<sub>2</sub>); 2.71 (t, *J* = 12.5 Hz, 1H, CH), 1.32–1.34 (m, 6H, OCH<sub>2</sub>CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta$  = 188.4, 168.1, 167.7, 162.3, 159.4, 138.3, 132.4, 129.7, 129.1, 128.6, 128.0, 114.7, 114.1, 70.4, 62.7, 62.6, 55.3, 51.7, 45.4, 14.0; HRMS (ESI-TOF<sup>+</sup>): *m/z* calcd for C<sub>25</sub>H<sub>27</sub>O<sub>6</sub>S [(M + H)<sup>+</sup>], 455.1528; found, 455.1532.

**(Z)-diethyl 5-(4-nitrophenyl)-2-(2-oxo-2-phenylethylidene)dihydrothiophene-3,3(2H)-dicarboxylate (3s)**



Yellow solid; yield 87 %; mp 137–138 °C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3056, 2957, 2857, 1746, 1730, 1693, 1635, 1519, 1347, 1263, 1230, 868, 773, 699; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta$  = 8.22 (d, *J* = 8.7 Hz, 2H, ArH), 7.98 (d, *J* = 7.5 Hz, 2H, ArH), 7.65 (d, *J* = 8.7 Hz, 2H, ArH), 7.47–7.58 (m, 5H, ArH), 4.81 (dd, *J* = 5.2, 11.7 Hz, 1H, CH<sub>2</sub>), 3.87 (s, OCH<sub>3</sub>), 3.86 (s, OCH<sub>3</sub>), 3.23 (dd, *J* = 5.2, 13.0 Hz, 1H, CH<sub>2</sub>); 2.71 (t, *J* = 12.4 Hz, 1H, CH); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta$  = 188.5, 168.2, 167.9, 160.5, 147.7, 145.7, 137.8, 132.7, 129.0, 128.7, 128.1, 125.8, 124.3, 124.0, 115.5, 70.0, 54.0, 53.7, 51.2, 45.0; HRMS (ESI-TOF<sup>+</sup>): *m/z* calcd for C<sub>22</sub>H<sub>20</sub>NO<sub>7</sub>S [(M + H)<sup>+</sup>], 442.0960; found, 442.0968.

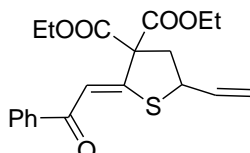
**(Z)-diethyl 5-(2-chlorophenyl)-2-(2-oxo-2-phenylethylidene)dihydrothiophene-3,3(2H)-dicarboxylate (3t)**



Yellow oil; yield 94%; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3061, 2982, 2937, 1736, 1637, 1598, 1542, 1232, 759, 704; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta$  = 7.98 (d, *J* = 7.5 Hz, 2H, ArH), 7.71–7.73 (m, 1 H),

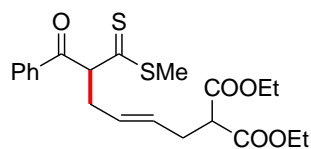
7.45–7.55 (m, 4 H, ArH + CH=C), 7.39 (d,  $J = 7.9$  Hz, 1 H, ArH), 7.21–7.29 (m, 2 H, ArH), 5.27 (dd,  $J = 5.2, 11.2$  Hz, 1H, CH<sub>2</sub>), 4.25–4.37 (m, 4H, OCH<sub>2</sub>CH<sub>3</sub>), 3.26 (dd,  $J = 5.2, 13.0$  Hz, 1H, CH<sub>2</sub>), 2.64–2.69 (m, 1H, CH), 1.27–1.34 (m, 6H, OCH<sub>2</sub>CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta = 188.2, 167.7, 167.4, 161.6, 138.0, 135.5, 133.9, 132.3, 129.6, 129.0, 128.5, 128.0, 127.8, 127.2, 115.0, 70.0, 62.7, 62.5, 48.1, 43.6, 13.8$ ; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for C<sub>25</sub>H<sub>27</sub>O<sub>5</sub>S [(M + H)<sup>+</sup>], 439.1579; found, 439.1582.

**(Z)-diethyl 2-(2-oxo-2-phenylethylidene)-5-vinyldihydrothiophene-3,3(2H)-dicarboxylate (3v)**



Yellow oil; yield 12 %; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3084, 2925, 1733, 1636, 1597, 1540, 1231, 773, 701; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta = 7.95$  (d,  $J = 7.5$  Hz, 2H, ArH), 7.53 (t,  $J = 7.3$  Hz, 1H, ArH), 7.46 (t,  $J = 7.5$  Hz, 2H, ArH), 7.38 (s, 1H, CH=C), 5.79–5.86 (m, 1H, CH=CH<sub>2</sub>), 5.35 (d,  $J = 16.9$  Hz, 1H, CH=CH<sub>2</sub>), 5.20 (d,  $J = 10.1$  Hz, 1H, CH=CH<sub>2</sub>), 4.23–4.37 (m, 4H, OCH<sub>2</sub>CH<sub>3</sub>), 4.16–4.21 (m, 1H, CH), 2.99 (dd,  $J = 5.2, 13.0$  Hz, 1H, CH<sub>2</sub>), 2.46–2.50 (m, 1H, CH<sub>2</sub>), 1.27–1.37 (m, 6H, OCH<sub>2</sub>CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta = 188.3, 168.1, 167.7, 162.3, 138.2, 135.6, 132.4, 128.6, 128.0, 118.7, 115.0, 70.3, 62.7, 62.6, 51.1, 42.9, 14.0$ ; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for C<sub>20</sub>H<sub>23</sub>O<sub>5</sub>S [(M + H)<sup>+</sup>], 375.1266; found, 375.1275.

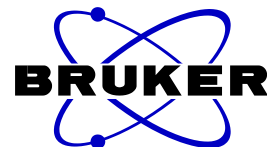
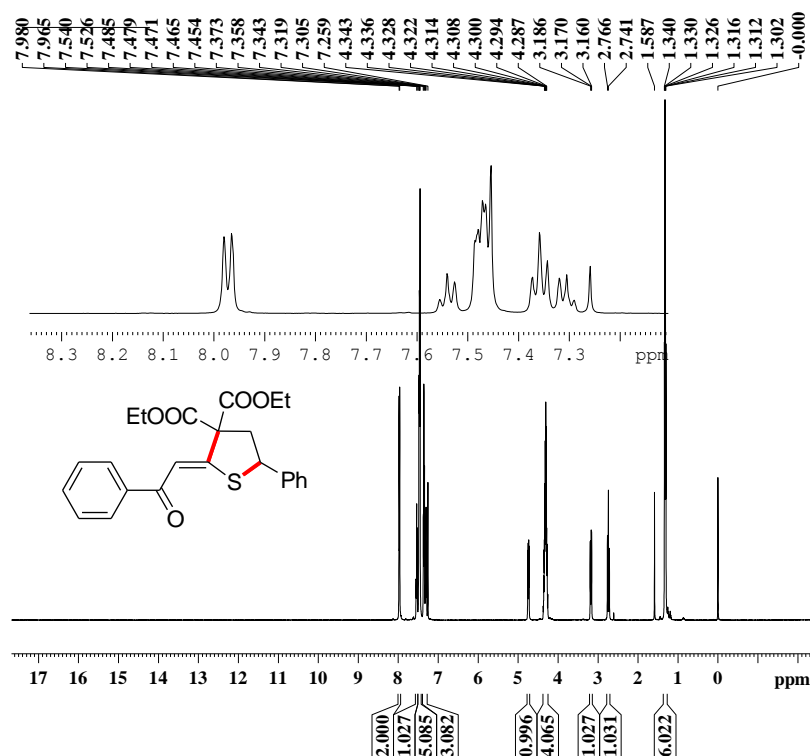
**(E)-diethyl 2-(5-benzoyl-6-(methylthio)-6-thioxohex-2-enyl)malonate (5)**



Yellow oil; yield 51%; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3086, 2981, 2935, 1735, 1732, 1690, 1690, 1597, 1447, 1369, 1267, 689, 659; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta = 8.02$  (d,  $J = 7.7$  Hz, 2H, ArH), 7.55 (t,  $J = 7.3$  Hz, 1H, ArH), 7.44 (t,  $J = 7.7$  Hz, 2H, ArH), 5.47–5.57 (m, 2H, CH=CH), 5.24 (t,  $J = 7.1$  Hz, 1H, CH), 4.09–4.19 (m, 4H, OCH<sub>2</sub>CH<sub>3</sub>), 3.37 (t,  $J = 7.6$  Hz, 1H, CH(COOEt)<sub>2</sub>), 2.92–2.98 (m, 1H, CH<sub>2</sub>), 2.69–2.74 (m, 1H, CH<sub>2</sub>), 2.59 (s, 3H, SCH<sub>3</sub>), 2.55 (t,  $J = 6.6$  Hz, 1H, CH<sub>2</sub>), 1.23 (t,  $J = 7.1$  Hz, 6H, OCH<sub>2</sub>CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta = 231.7, 193.3, 168.8, 136.1, 133.2, 129.3, 128.8, 128.6, 68.0, 61.3, 52.0, 37.2, 31.6, 20.0, 14.0$ ; HRMS (ESI-TOF<sup>+</sup>):  $m/z$  calcd for C<sub>21</sub>H<sub>27</sub>O<sub>5</sub>S<sub>2</sub> [(M + H)<sup>+</sup>], 423.1300; found, 423.1309.

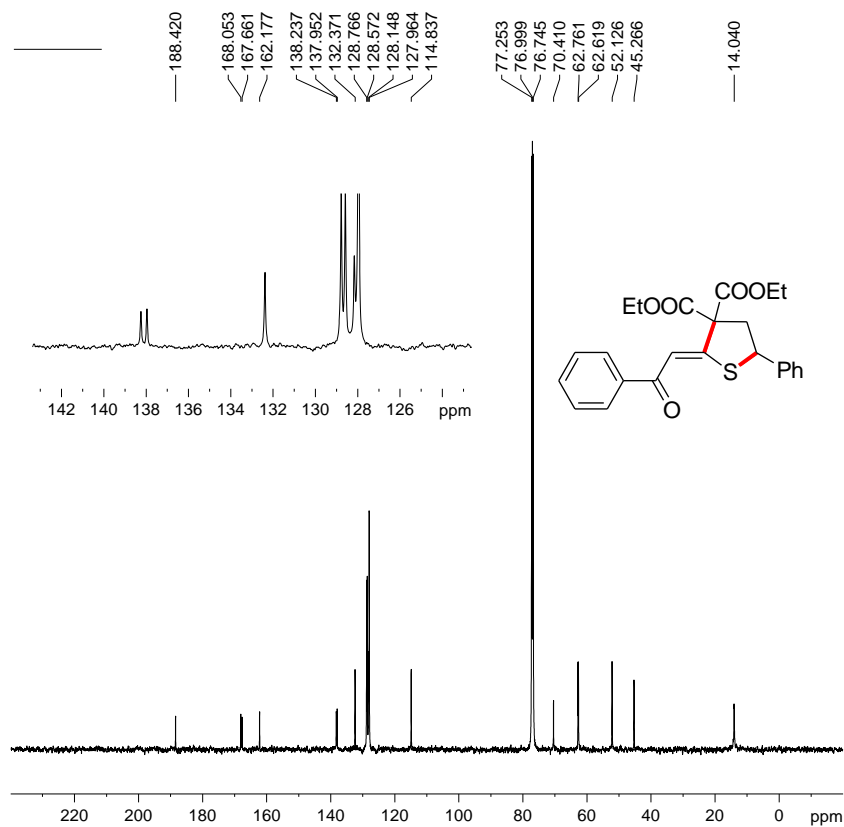
**References**

- (a) K. Riza, R. B. Nicholas, *J. Org. Chem.*, 1981, **46**, 197–201; (b) S. Mohammad, A. Zeinab, M. Barahman, *Synthesis*, 2010, 392–394.
- (a) R. A. Novikov, V. P. Timofeev and Y. V. Tomilov, *J. Org. Chem.*, 2012, **77**, 5993–6006; (b) A. F. G. Goldberg, N. R. O'Connor, R. A. Craig II and B. M. Stoltz, *Org. Lett.*, 2012, **14**, 5314–5317; (c) J. Moran, A. G. Smith, R. M. Carris, J. S. Johnson and M. J. Krische, *J. Am. Chem. Soc.*, 2011, **133**, 18618–18621.



NAME WSW140530  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20140605  
 Time 15.33  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 144  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 673.2 K  
 D1 1.00000000 sec  
 TD0 1

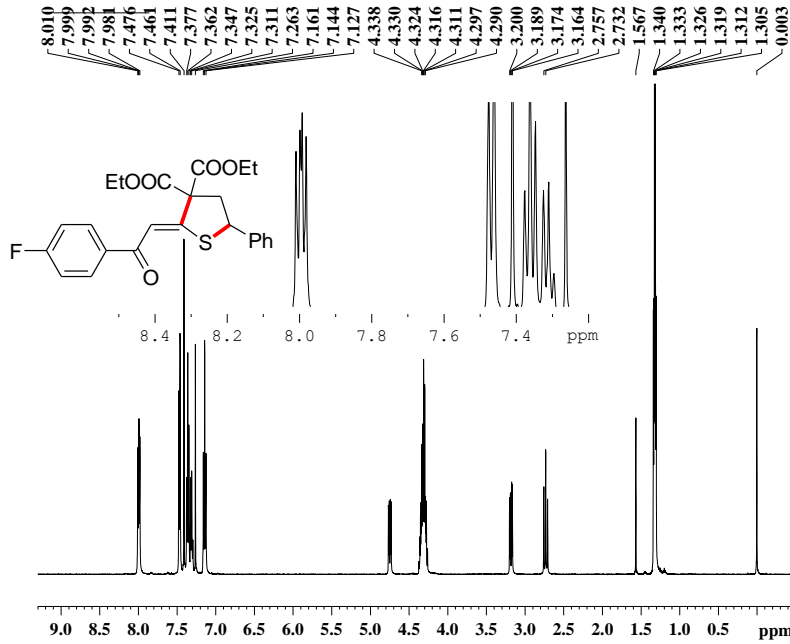
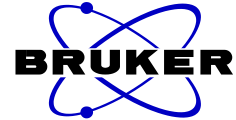
===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300114 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00



NAME WSW140530  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20140609  
 Time 13.47  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 721  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 1820  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 299.3 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 20

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

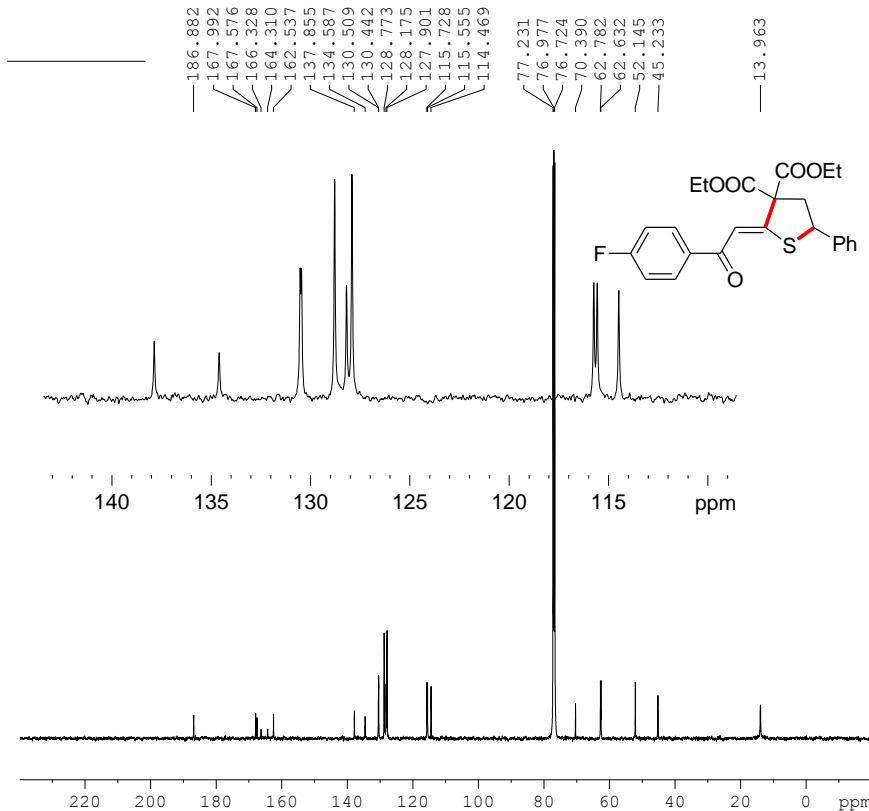
===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326481 MHz  
 WDW EM  
 SSB 0  
 LB 8.00 Hz  
 GB 0  
 PC 1.00



NAME WSW-140512  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20140528  
 Time 16.00  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 8  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 362  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 673.2 K  
 D1 1.0000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300095 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

WSW140512 13C 1D 2014 05 30

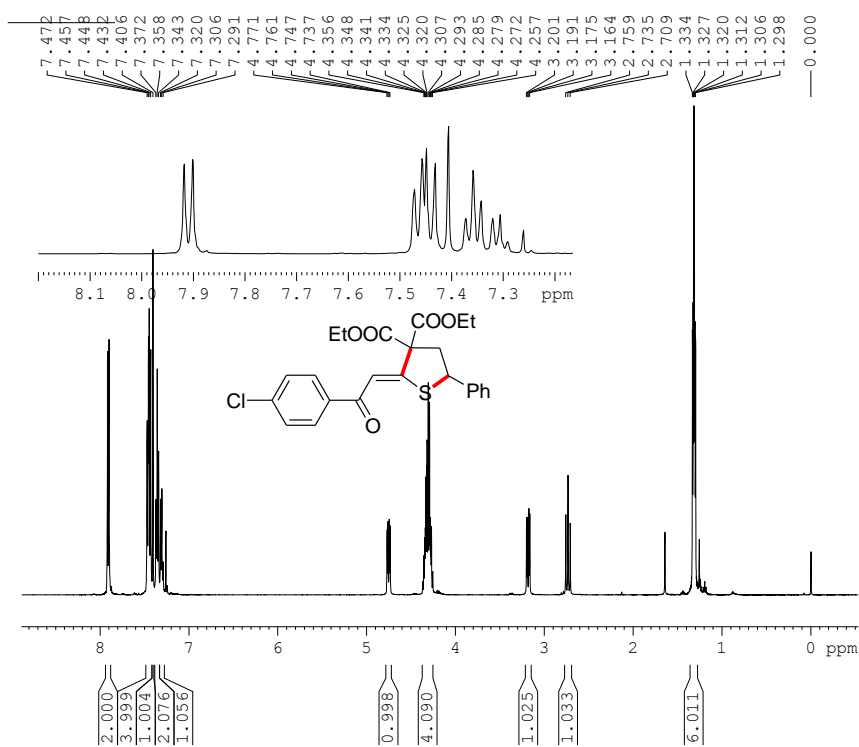


NAME WSW140512  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20140530  
 Time 15.00  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 1639  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 2300  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 673.2 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 2

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326490 MHz  
 WDW EM  
 SSB 0  
 LB 8.00 Hz  
 GB 0  
 PC 1.00

WSW-2-b 1H 1D 2013 10 15



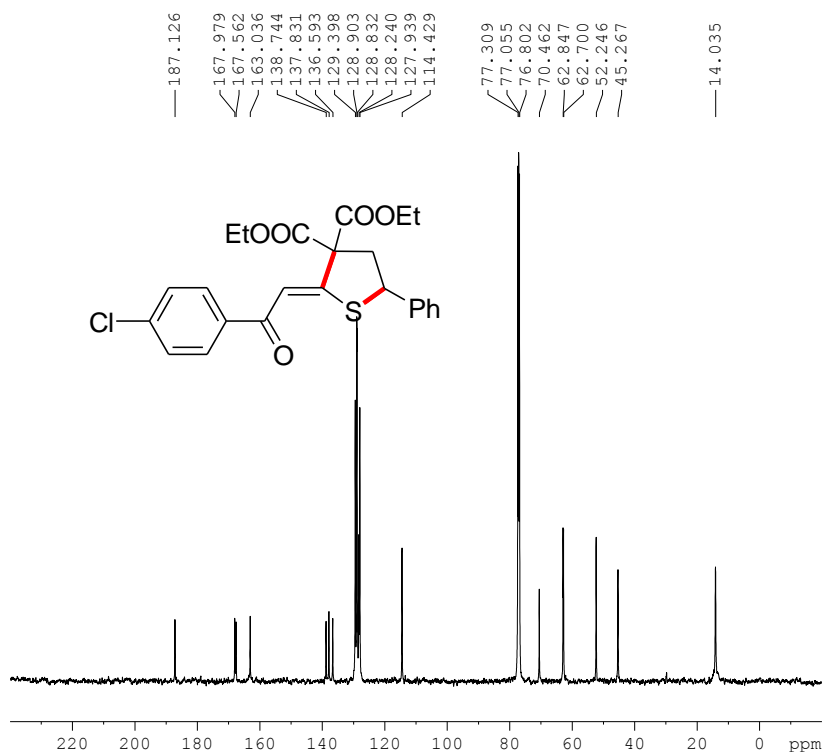
```

NAME          WSW-2-b
EXPNO         1
PROCNO        1
Date_         20131015
Time          9.43
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            16384
SOLVENT       CDC13
NS            8
DS            1
SWH           10000.000 Hz
FIDRES        0.610352 Hz
AQ            0.8193000 sec
RG            161
DW            50.000 usec
DE            8.00 usec
TE            297.5 K
D1            1.00000000 sec
D1            1
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            13.00 usec
PL1           2.00 dB
SFO1          500.0338500 MHz
SI            16384
SF            500.0300087 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            4.00
  
```

wsw-2-b 13C 2013 10 15



```

NAME          WSW-2-b
EXPNO         2
PROCNO        1
Date_         20131015
Time          20.48
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDC13
NS            13258
DS            2
SWH           32679.738 Hz
FIDRES        0.498653 Hz
AQ            1.0027661 sec
RG            6500
DW            15.300 usec
DE            6.00 usec
TE            298.7 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0           20
  
```

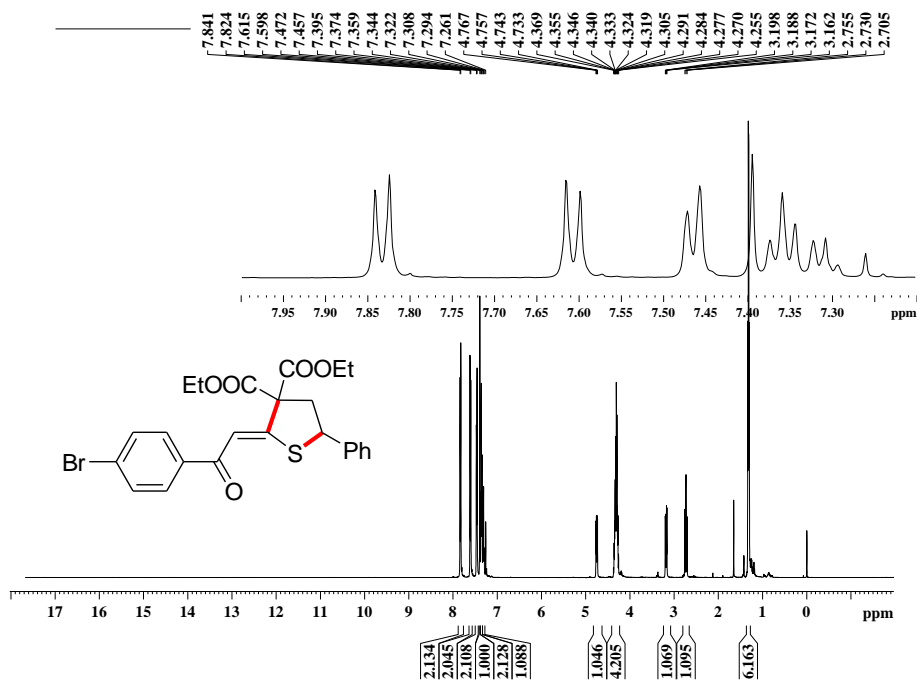
```

===== CHANNEL f1 =====
NUC1          13C
P1            12.20 usec
PL1           3.00 dB
SFO1          125.7464750 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.00 dB
PL12          17.70 dB
PL13          17.70 dB
SFO2          500.0355000 MHz
SI            32768
SF            125.7326443 MHz
WDW           EM
SSB           0
LB            12.00 Hz
GB            0
PC            1.00
  
```

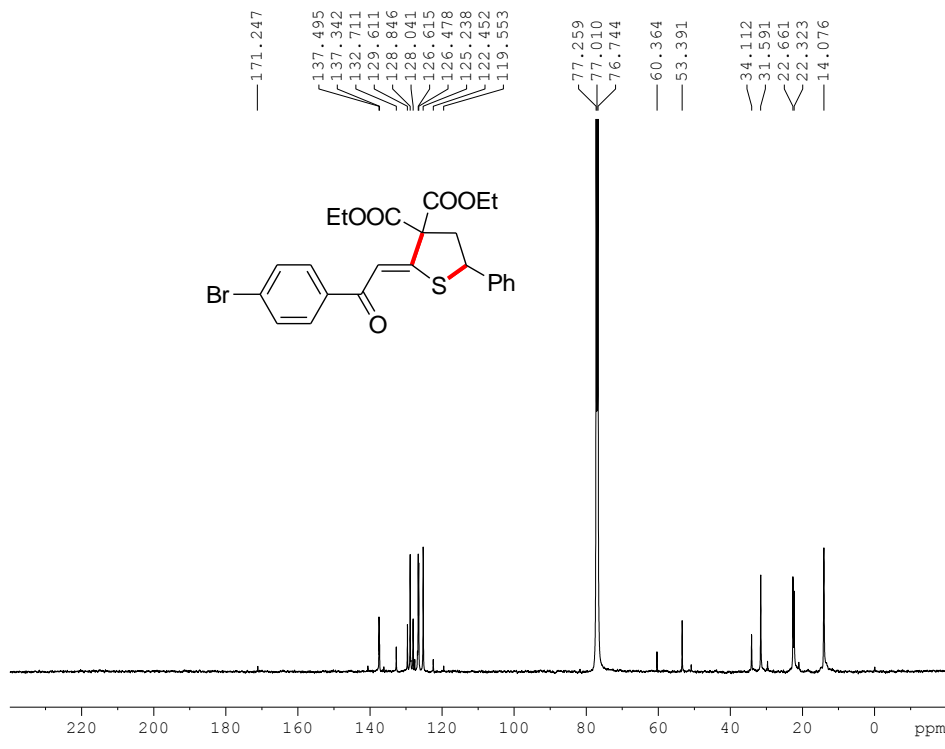
WSW140920-3 1H 1D 2015 01 08



NAME WSW140920-3  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20150108  
 Time 16.10  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 161  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 290.9 K  
 D1 1.00000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300099 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

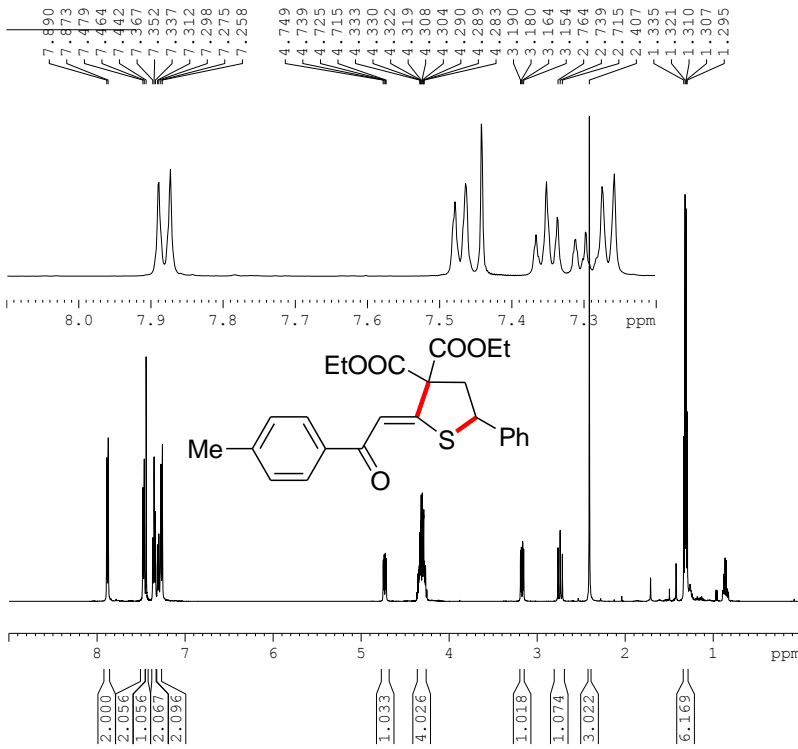
WSW140920-3 13C 1D 2015 01 10



NAME WSW140920-3  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20150110  
 Time 22.36  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 14017  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 5790  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 673.2 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 20

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz  
 ===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 FCPD2 80.00 usec  
 FL2 2.00 dB  
 FL12 17.70 dB  
 FL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326433 MHz  
 WDW EM  
 SSB 0  
 LB 10.00 Hz  
 GB 0  
 PC 1.00

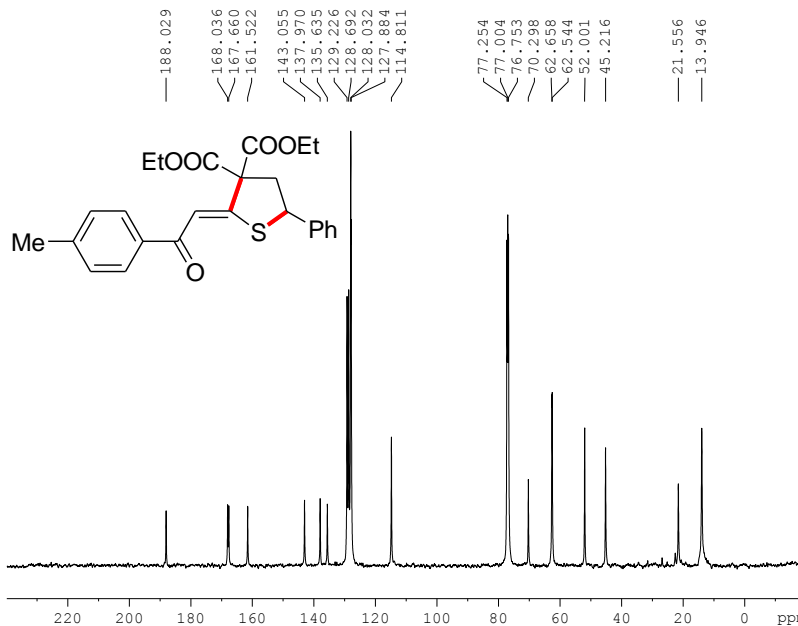
WSW-2-F 1H 1D 2013 12 13



NAME WSW-2-F  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20131213  
 Time\_ 18.28  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDC13  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 80.6  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 294.1 K  
 D1 1.00000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300117 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

WSW-2-f 13C 2013 12 16



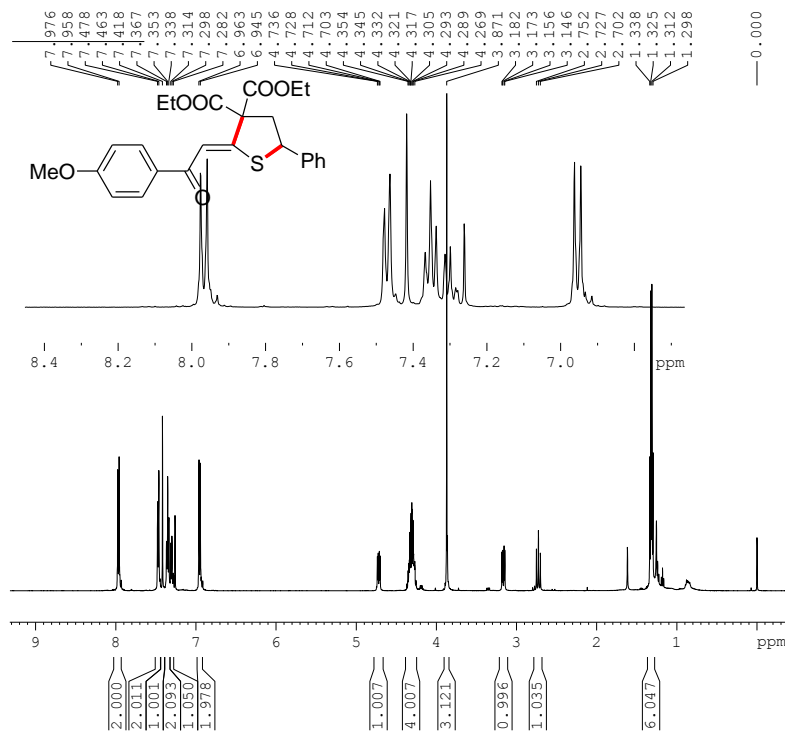
NAME WSW-2-F  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20131216  
 Time\_ 13.16  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 800  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 1290  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 298.5 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TDO 20

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326559 MHz  
 WDW EM  
 SSB 0  
 LB 20.00 Hz  
 GB 0  
 PC 2.00



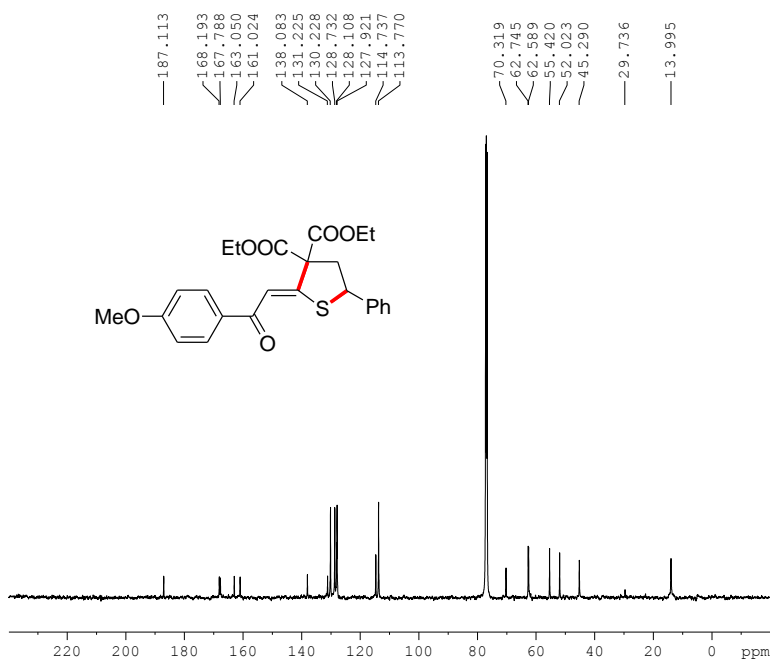
ISW-2-c 1H 1D 2013 11 0'



NAME WSW-2-c  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20131107  
 Time 16.13  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 8  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 161  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 673.2 K  
 D1 1.00000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0388500 MHz  
 SI 16384  
 SF 500.0300087 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 4.00

WSW-2-C 13C 2013 11 08

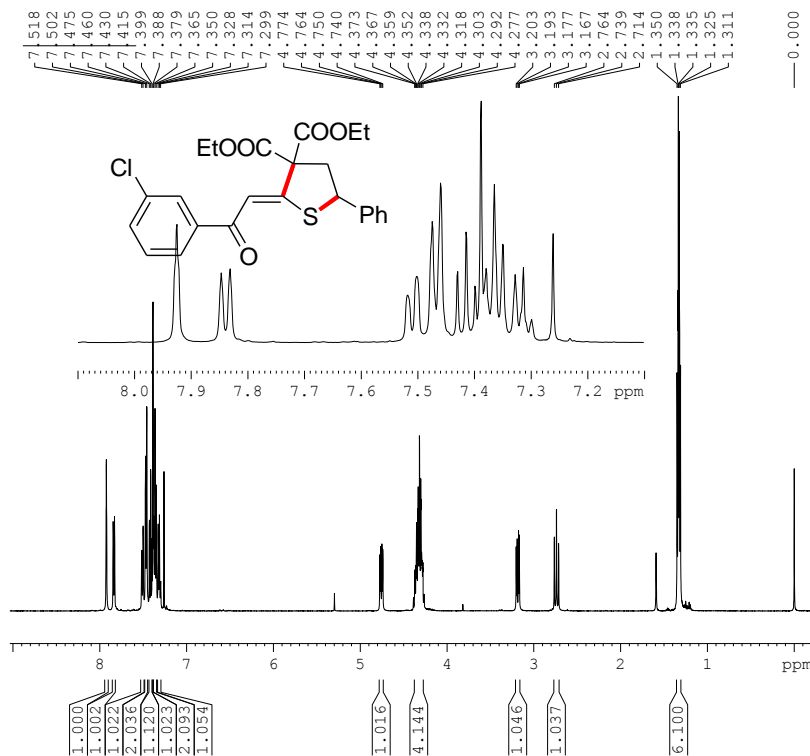


NAME WSW-2-c  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20131108  
 Time 16.04  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 740  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 1620  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 673.2 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 20

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326490 MHz  
 WDW EM  
 SSB 0  
 LB 12.00 Hz  
 GB 0  
 PC 4.00

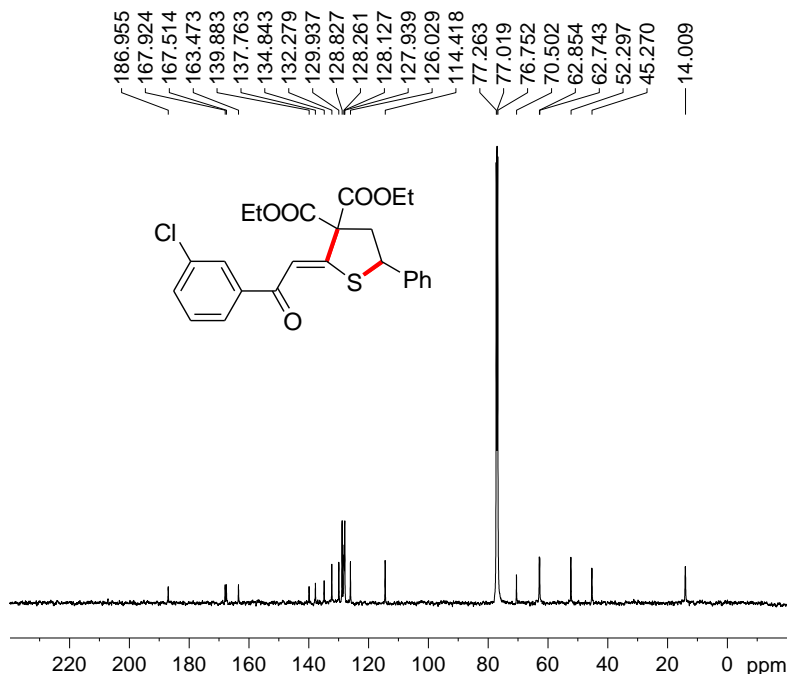
WSW-2-e 1H 1D 2013 12 06



NAME WSW-2-e  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20131206  
 Time\_ 15.34  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 228  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 294.1 K  
 D1 1.00000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300102 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

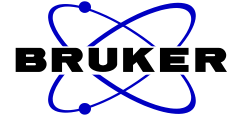
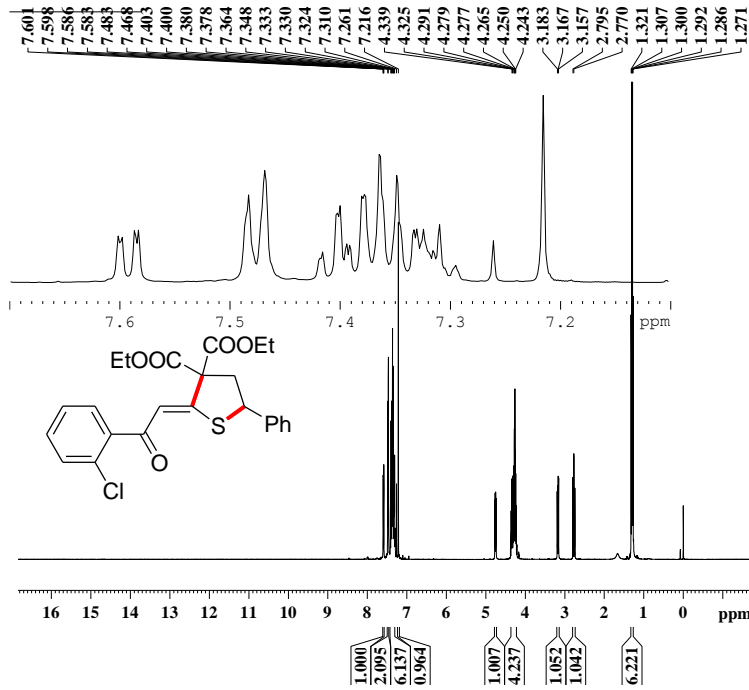
WSW-2-e 13C 2013 12 06



NAME WSW-2-e  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20131206  
 Time\_ 16.59  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 653  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 1290  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 294.3 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.899999998 sec  
 TDO 20

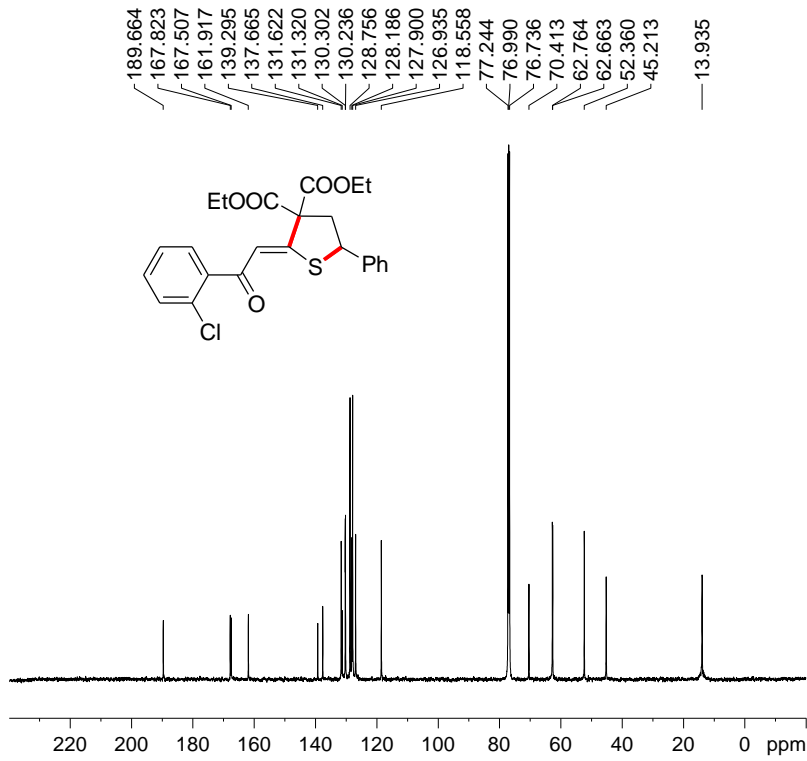
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326472 MHz  
 WDW EM  
 SSB 0  
 LB 12.00 Hz  
 GB 0  
 PC 1.00



NAME WSW-2-g  
 EXPNO 1  
 PROCNO 20140327  
 Date\_ 20140327  
 Time 15.51  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 1000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 203  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 296.3 K  
 D1 1.00000000 sec  
 TDO 1

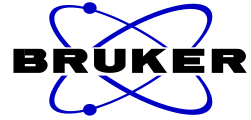
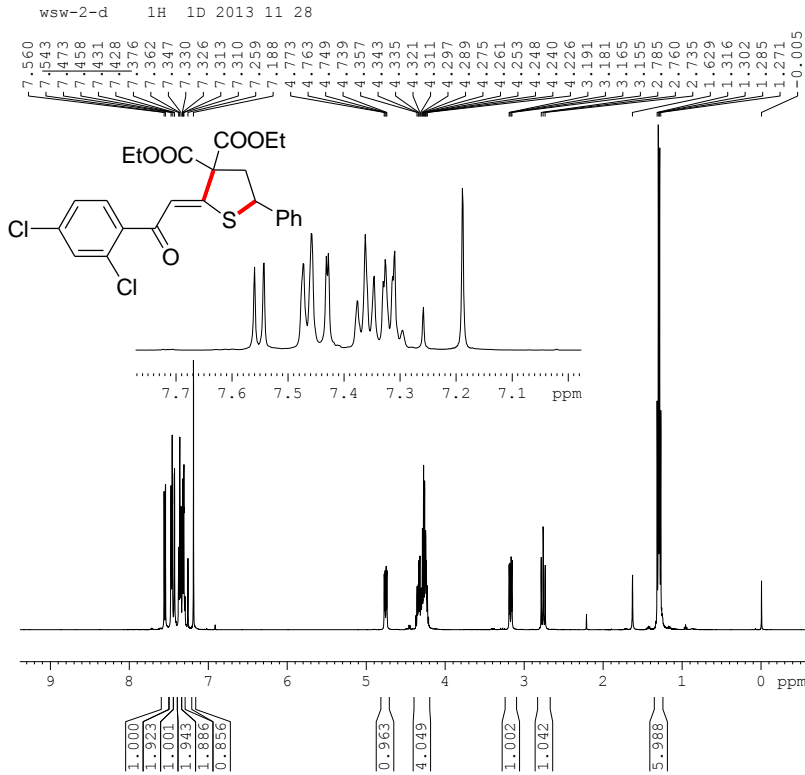
===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300103 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00



NAME WSW-2-g  
 EXPNO 2  
 PROCNO 20140328  
 Date\_ 20140328  
 Time 17.20  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 1142  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 2890  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 673.2 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TDO 20

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326539 MHz  
 WDW EM  
 SSB 0  
 LB 6.00 Hz  
 GB 0  
 PC 4.00



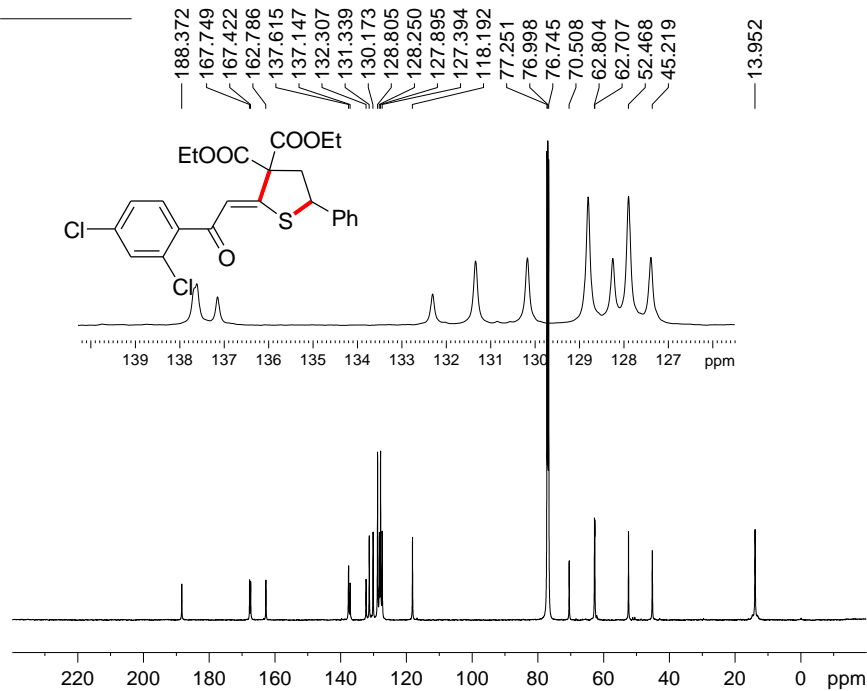
```

NAME          wsw-2-d
EXPNO         1
PROCNO        1
Date_         20131128
Time          16.27
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            16384
SOLVENT       CDCl3
NS            16
DS            1
SWH           10000.000 Hz
FIDRES        0.610352 Hz
AQ            0.8193000 sec
RG            181
DW            50.000 usec
DE            8.00 usec
TE            293.4 K
D1            1.00000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            13.00 usec
PL1           2.00 dB
SFO1          500.0338500 MHz
SI            16384
SF            500.0300097 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            4.00
  
```

wsw-2-d 13C 2013 11 29



```

NAME          wsw-2-d
EXPNO         2
PROCNO        1
Date_         20131129
Time          20.01
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16755
DS            2
SWH           32679.738 Hz
FIDRES        0.498653 Hz
AQ            1.0027661 sec
RG            1620
DW            15.300 usec
DE            6.00 usec
TE            295.1 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0           20
  
```

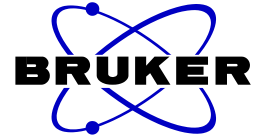
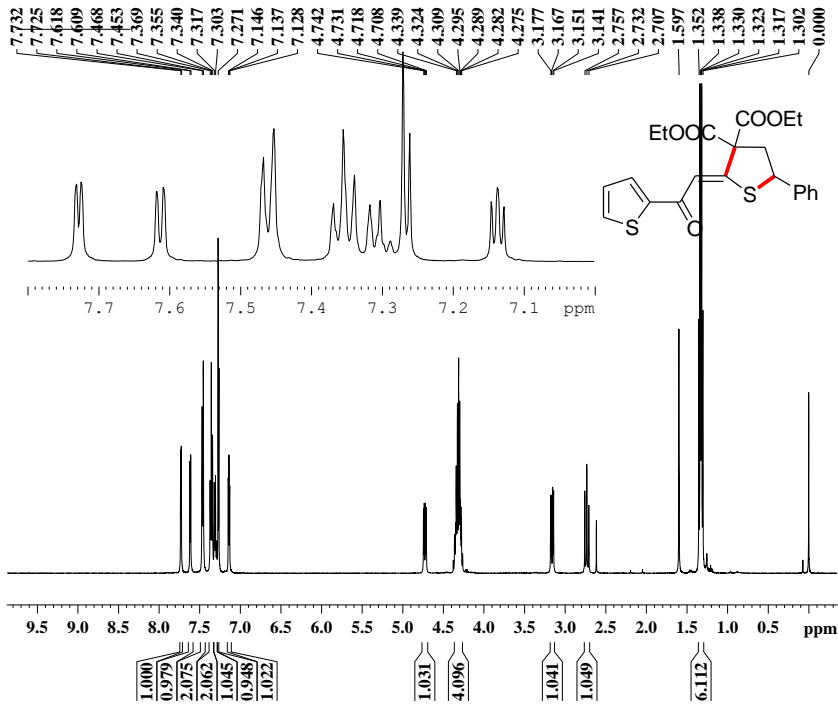
```

===== CHANNEL f1 =====
NUC1          13C
P1            12.20 usec
PL1           3.00 dB
SFO1          125.7464750 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.00 dB
PL12          17.70 dB
PL13          17.70 dB
SFO2          500.0355000 MHz
SI            32768
SF            125.7326490 MHz
WDW           EM
SSB           0
LB            12.00 Hz
GB            0
PC            4.00
  
```

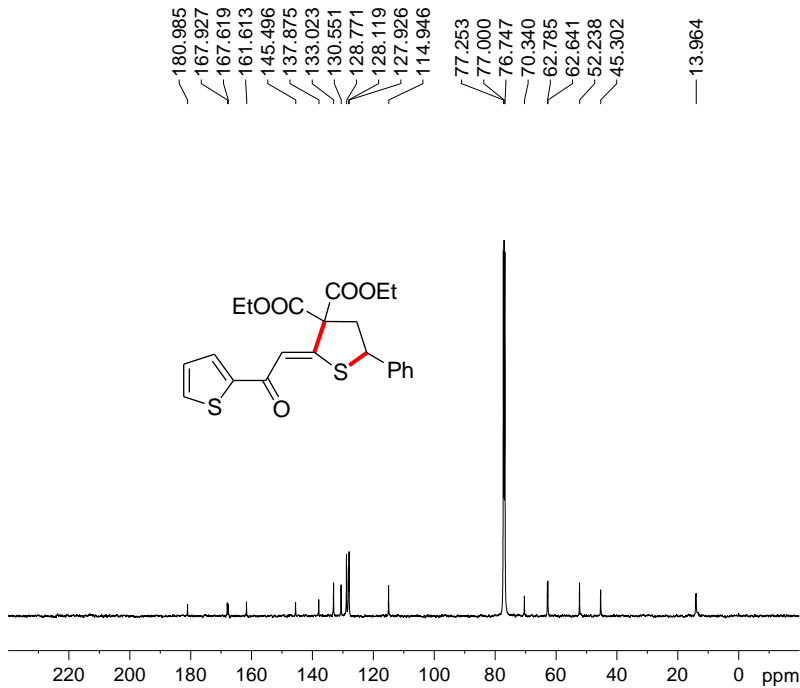
WSW140412 1H 1D 2014 04 15



NAME WSW140412  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20140415  
 Time 16.54  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 322  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 295.6 K  
 D1 1.00000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.038500 MHz  
 SI 16384  
 SF 500.0300101 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 4.00

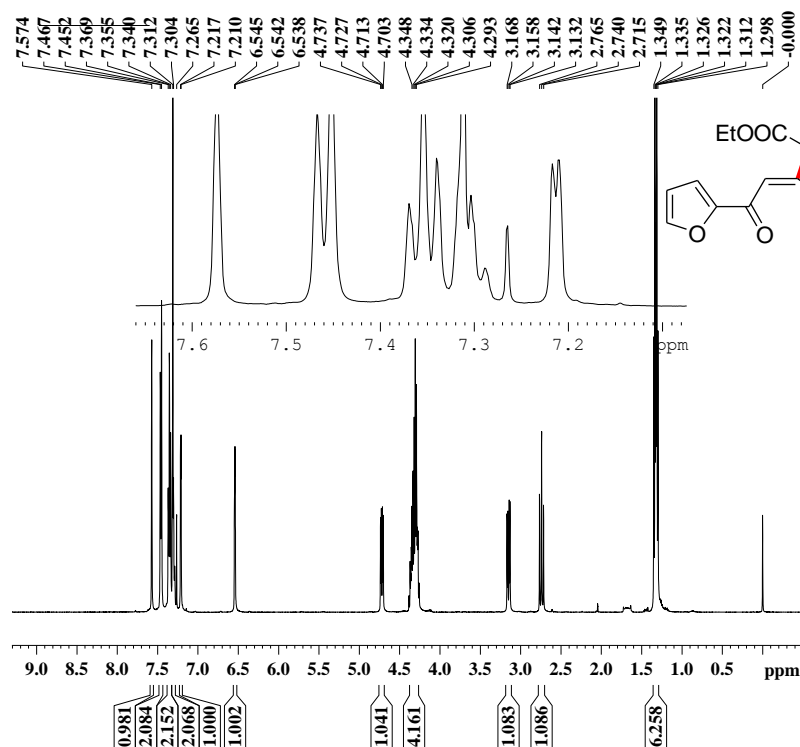
WSW140412 13C 1D 2014 04 17



NAME WSW140412  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20140417  
 Time 13.01  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 1579  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 1820  
 DW 15.300 usec  
 DE 8.00 usec  
 TE 297.1 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 20

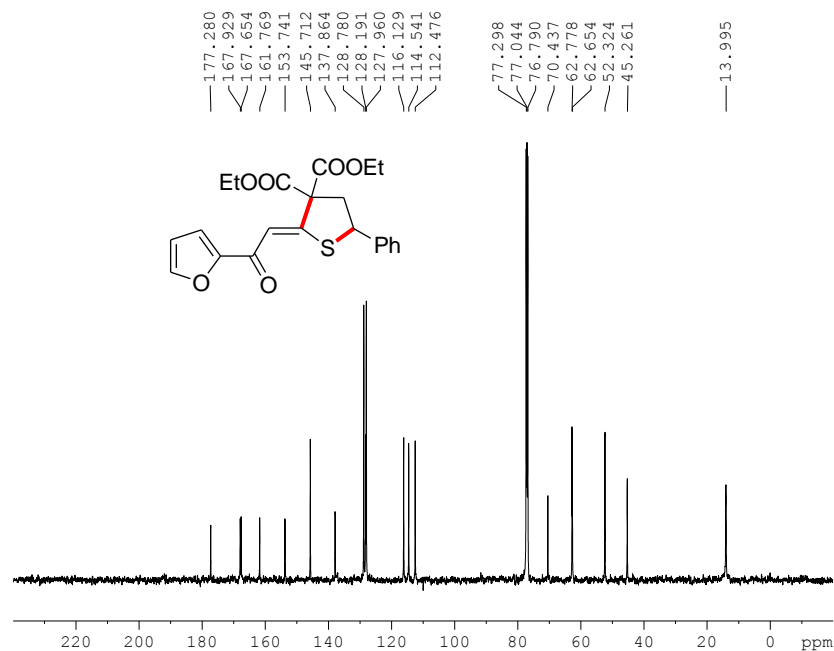
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326480 MHz  
 WDW EM  
 SSB 0  
 LB 12.00 Hz  
 GB 0  
 PC 2.00



NAME WSW140520-2  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20141215  
 Time 13.47  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 8  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 144  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 293.5 K  
 D1 1.00000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300079 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

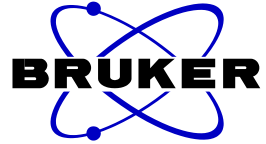
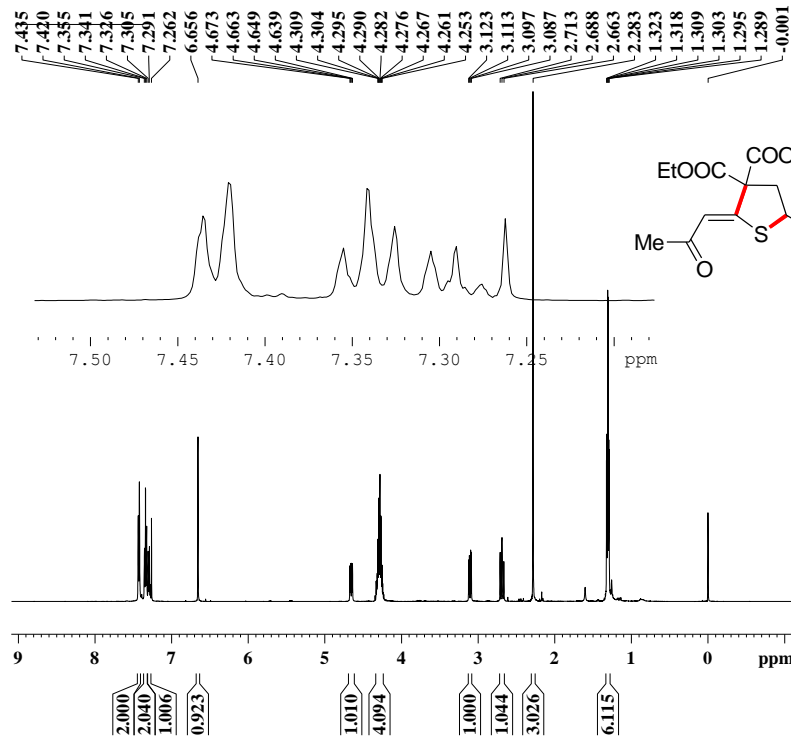


NAME WSW140520-2  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20141215  
 Time 18.39  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 363  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 287  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 294.3 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 20

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326472 MHz  
 WDW EM  
 SSB 0  
 LB 8.00 Hz  
 GB 0  
 PC 4.00

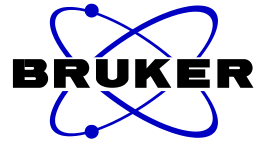
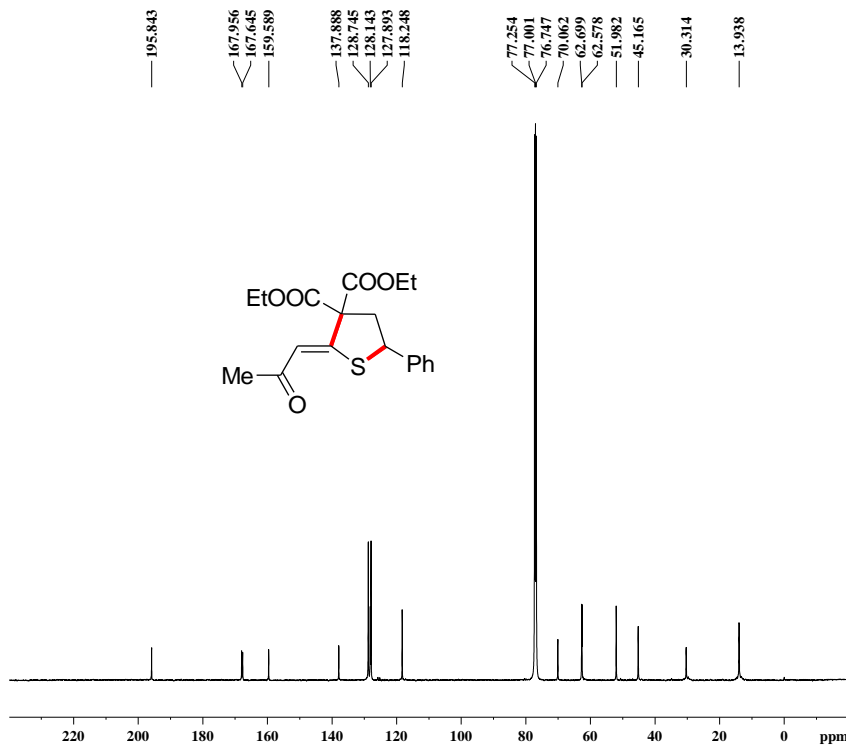
WSW-140915 1H ID 2014 09 17



NAME WSW-140915  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20140917  
 Time 14.58  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 228  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 673.2 K  
 D1 1.0000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300096 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

WSW140915 13C ID 2014 09 17

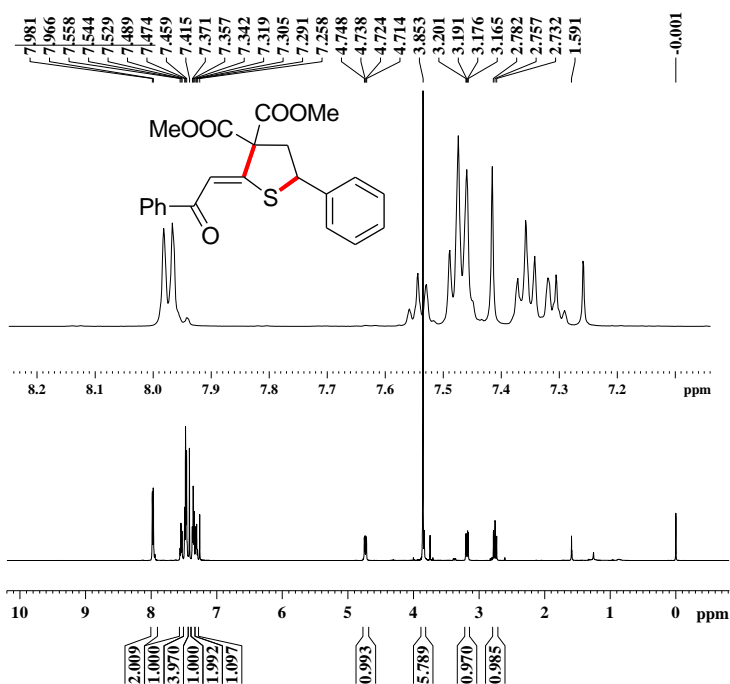


NAME WSW-140915  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20140917  
 Time 22.07  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 15174  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 287  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 673.2 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 20

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326472 MHz  
 WDW EM

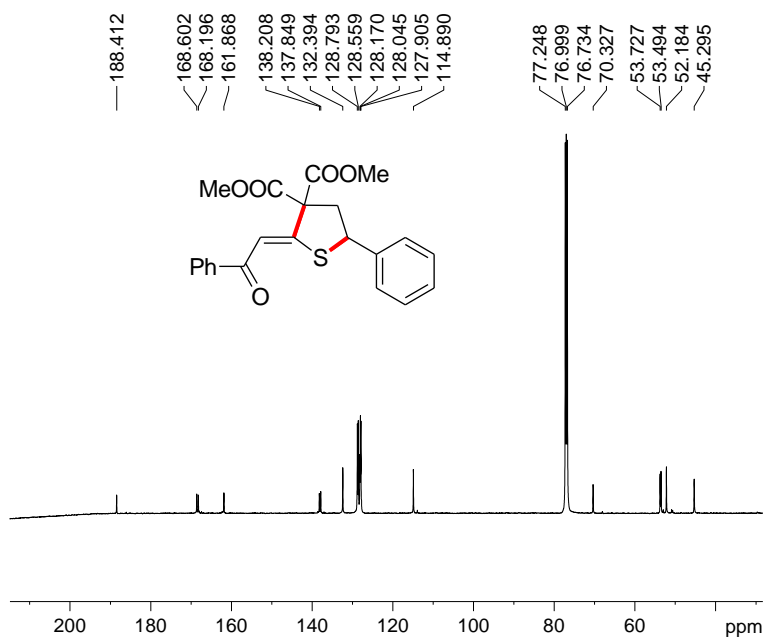
WSW140926 1H 1D 2014 10 13



NAME WSW140926  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20141013  
 Time 15.55  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 64  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 673.2 K  
 D1 1.00000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300113 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

WSW140926 13C 1D 2014 10 14



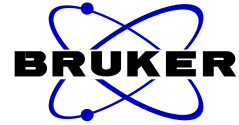
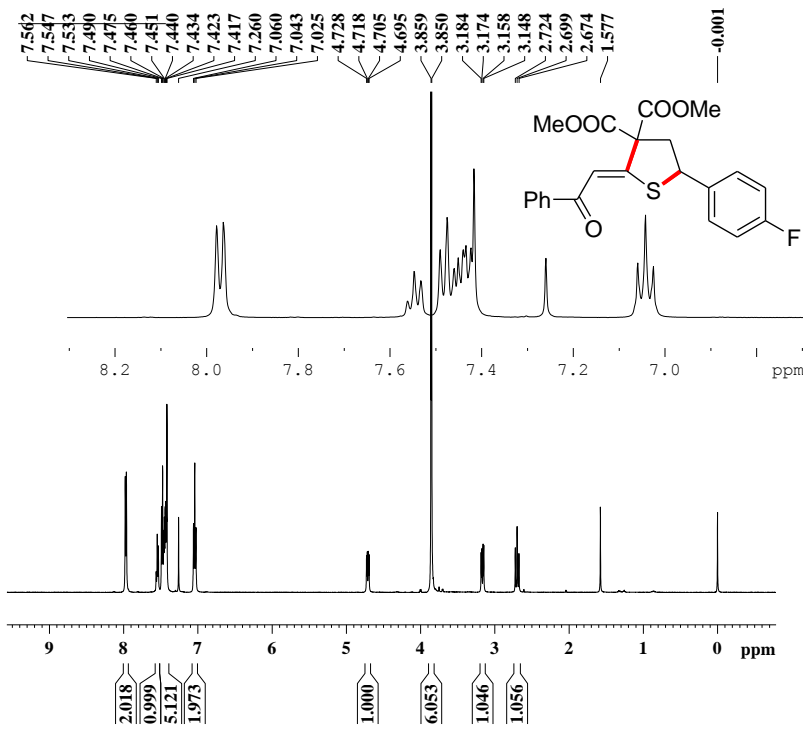
NAME WSW140926  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20141014  
 Time 18.00  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 15229  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 575  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 673.2 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 20

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326472 MHz  
 WDW EM  
 SSB 0  
 LB 8.00 Hz  
 GB 0  
 PC 4.00



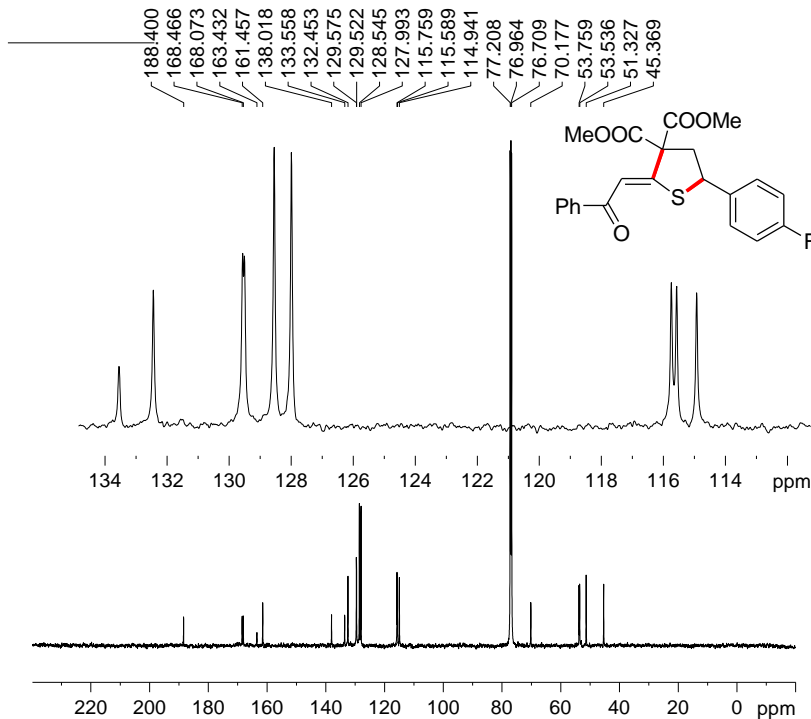
WSW140606 1H 1D 2014 06 17



NAME WSW140606  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20140617  
 Time 10.21  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 322  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 300.1 K  
 D1 1.00000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.038500 MHz  
 SI 16384  
 SF 500.0300110 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 4.00

WSW140606 13C 1D 2014 06 19

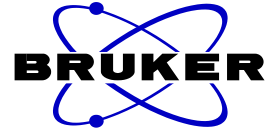
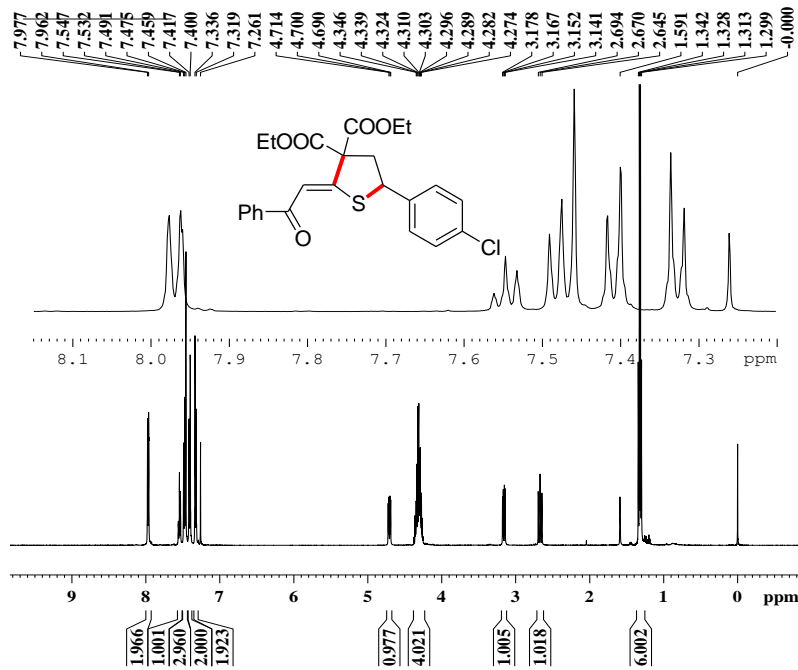


NAME WSW140606  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20140619  
 Time 18.33  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 1155  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 287  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 673.2 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 20

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326539 MHz  
 WDW EM  
 SSB 0  
 LB 8.00 Hz  
 GB 0  
 PC 4.00

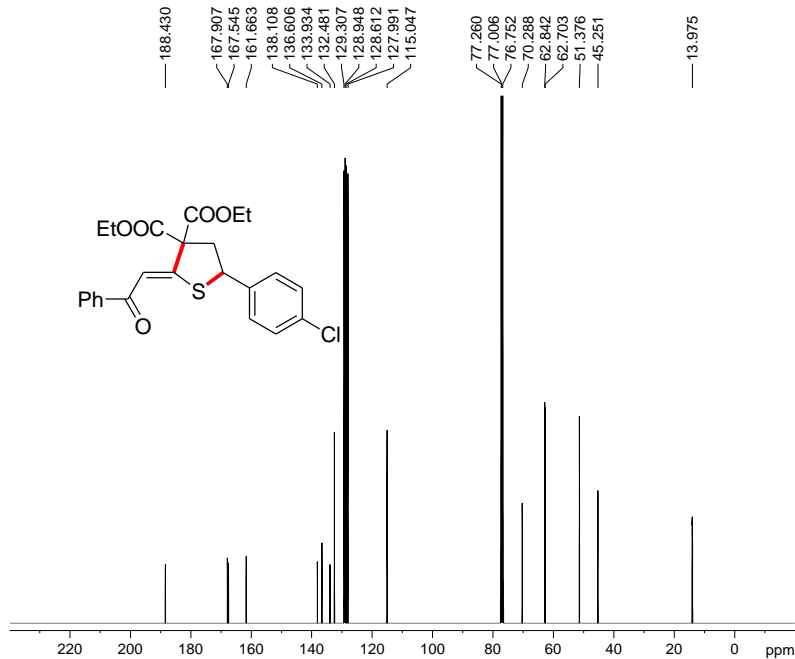
WSW141011 1H ID 2014 10 13



NAME WSW141011  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20141013  
 Time 16.03  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 287  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 673.2 K  
 D1 1.00000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300101 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

WSW141011 13C 1D 2014 10 16

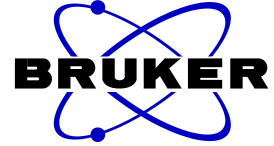
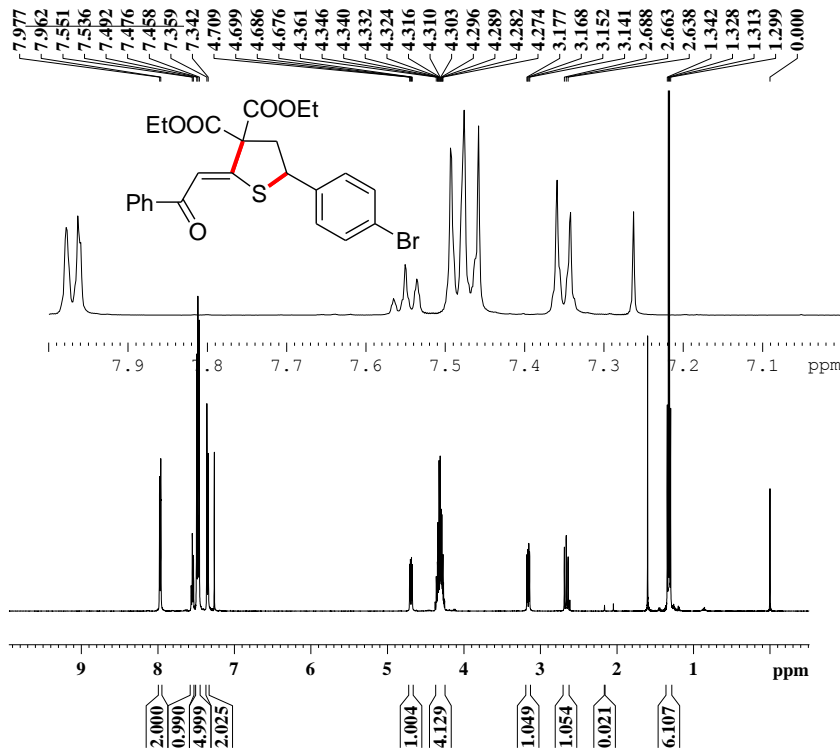


NAME WSW141011  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20141016  
 Time 10.57  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 5151  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 575  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 673.2 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 10

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326472 MHz  
 WDW EM  
 SSB 0  
 LB 8.00 Hz  
 GB 0  
 PC 4.00

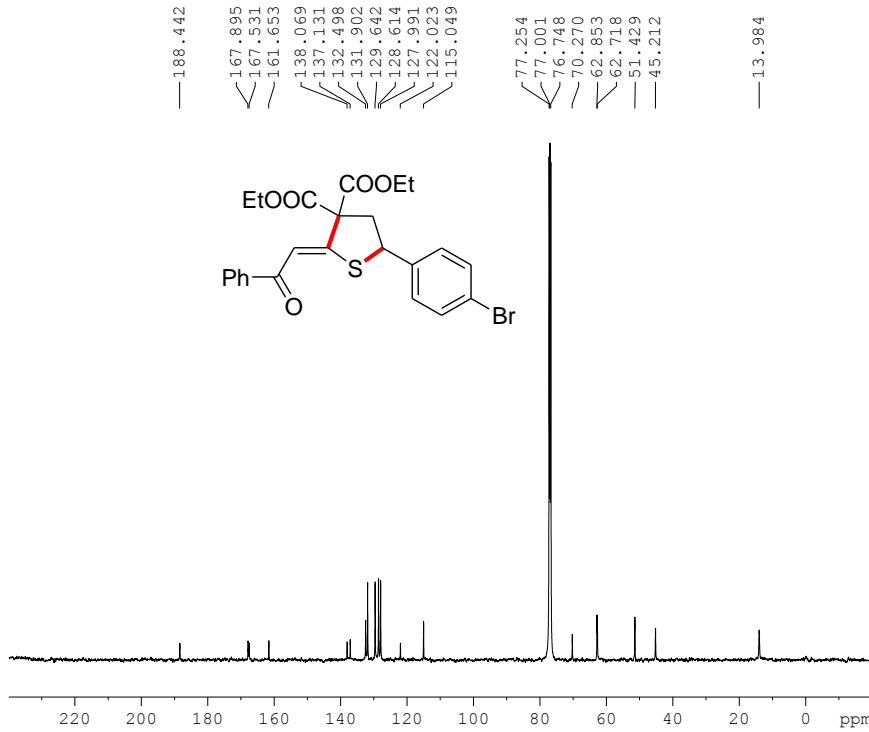
WSW-140430 1H 1D 2014 05 05



NAME WSW-140430  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20140505  
 Time 15.52  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 362  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 673.2 K  
 D1 1.00000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300095 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

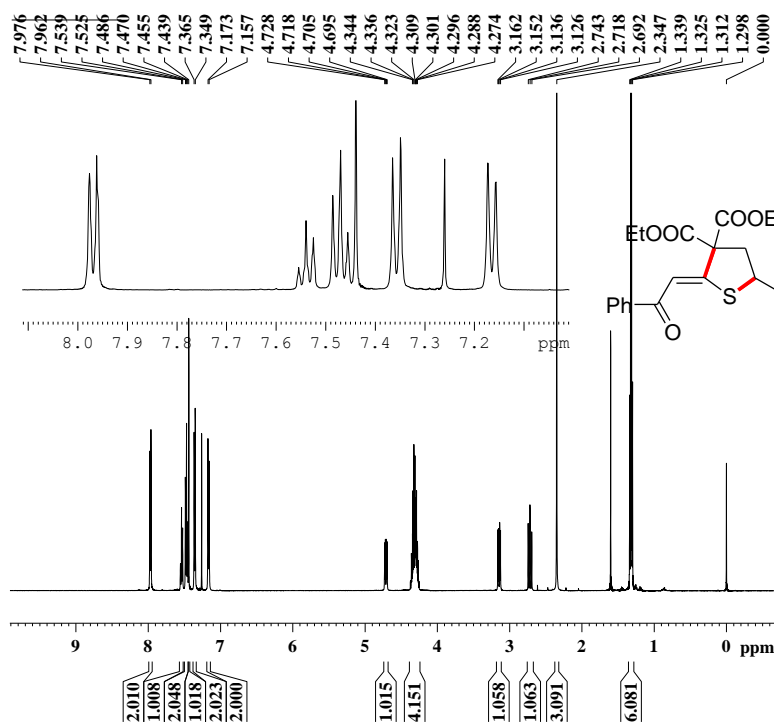
WSW-140430 13C 1D 2014 05 05



NAME WSW-140430  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20140505  
 Time 19.33  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 6536  
 SOLVENT CDCl3  
 NS 1342  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 1820  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 673.2 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 40

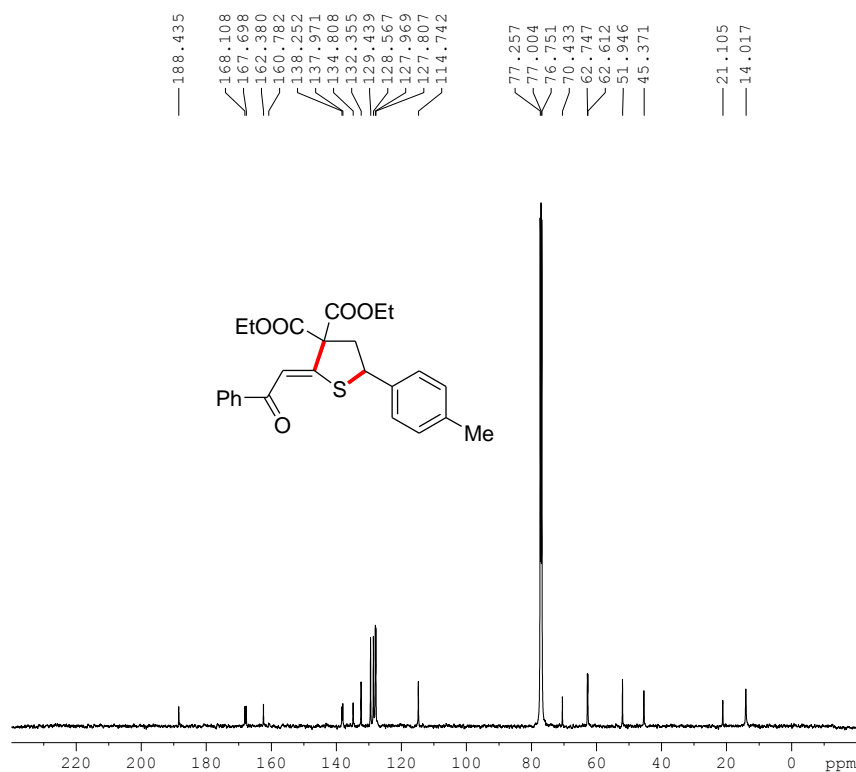
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326483 MHz  
 WDW EM  
 SSB 0  
 LB 12.00 Hz  
 GB 0  
 PC 1.00



NAME WSW-140424  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20140505  
 Time 15.43  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDC13  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 18  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 673.2 K  
 DI 1.00000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300108 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

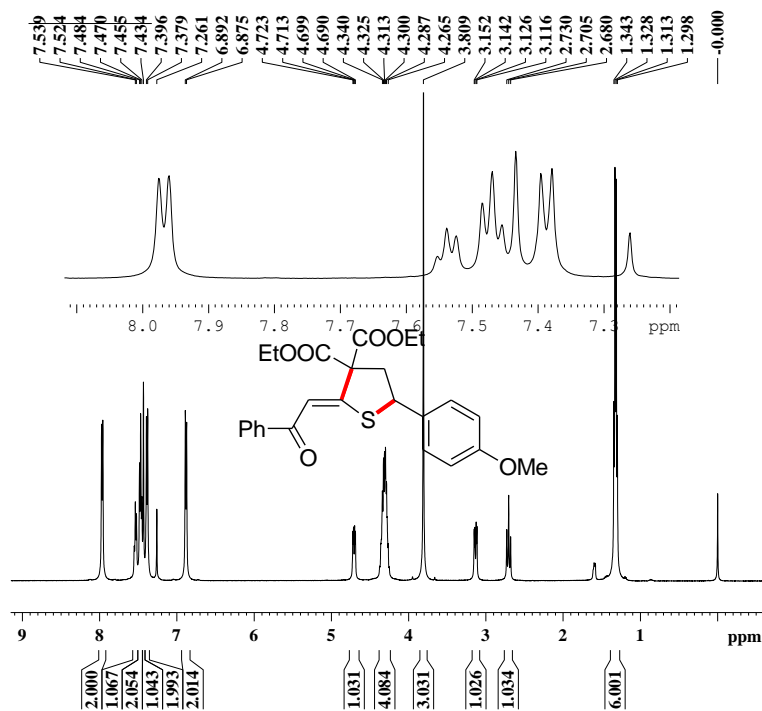


NAME WSW-140424  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20140505  
 Time 18.24  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 1250  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 1820  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 673.2 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 40

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326486 MHz  
 WDW EM  
 SSB 0  
 LB 12.00 Hz  
 GB 0  
 PC 1.00

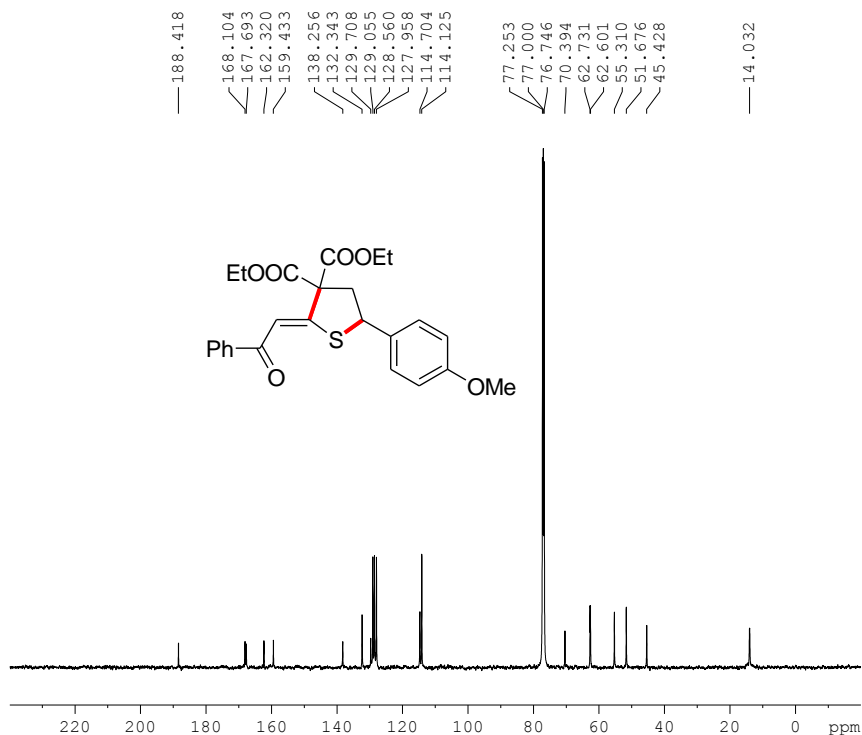
WSW141127 1H 1D 2014 11 27



NAME WSW141127  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20141127  
 Time 14.46  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 287  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 673.2 K  
 D1 1.00000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300099 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

WSW141127 13C 1D 2014 11 27

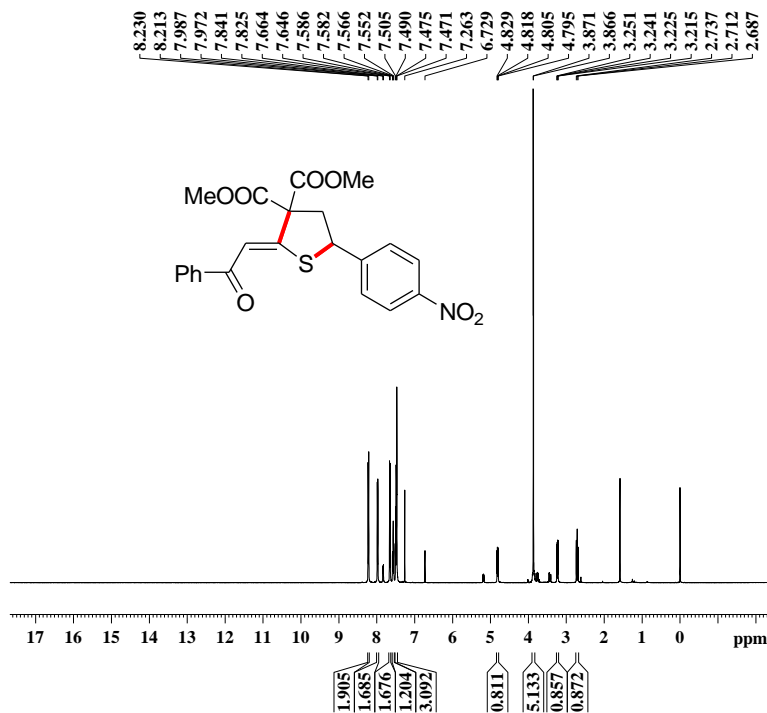


NAME WSW141127  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20141127  
 Time 16.47  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 1237  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 575  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 673.2 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 20

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326487 MHz  
 WDW EM  
 SSB 0  
 LB 10.00 Hz  
 GB 0  
 PC 4.00

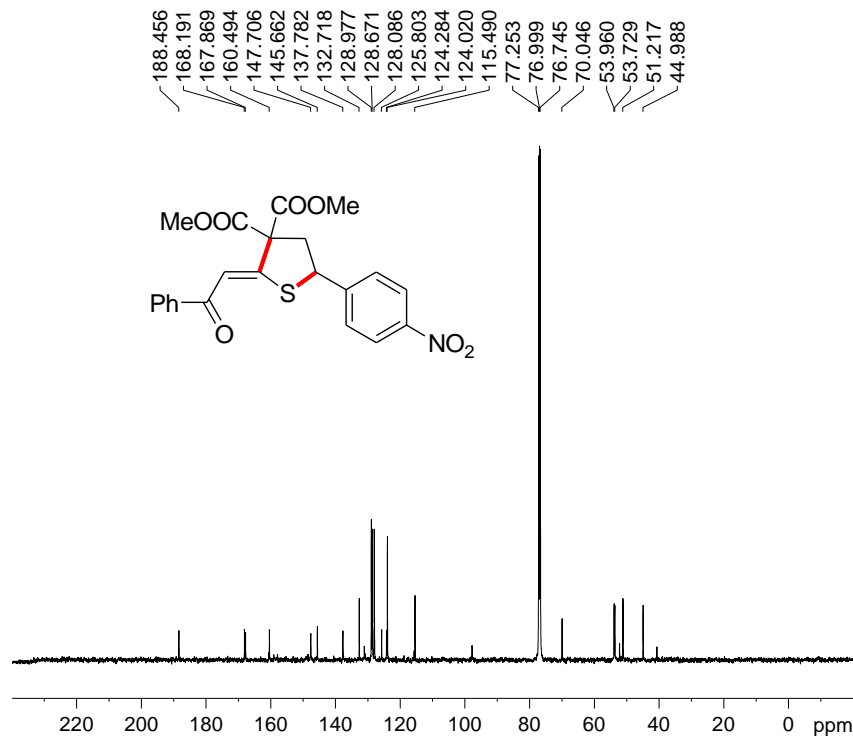
WSW140607 1H 1D 2014 06 17



NAME WSW140607  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20140617  
 Time 10.33  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 322  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 300.0 K  
 D1 1.0000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300093 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 4.00

WSW140607 13C 1D 2014 06 19

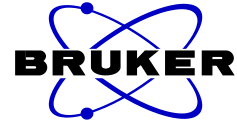
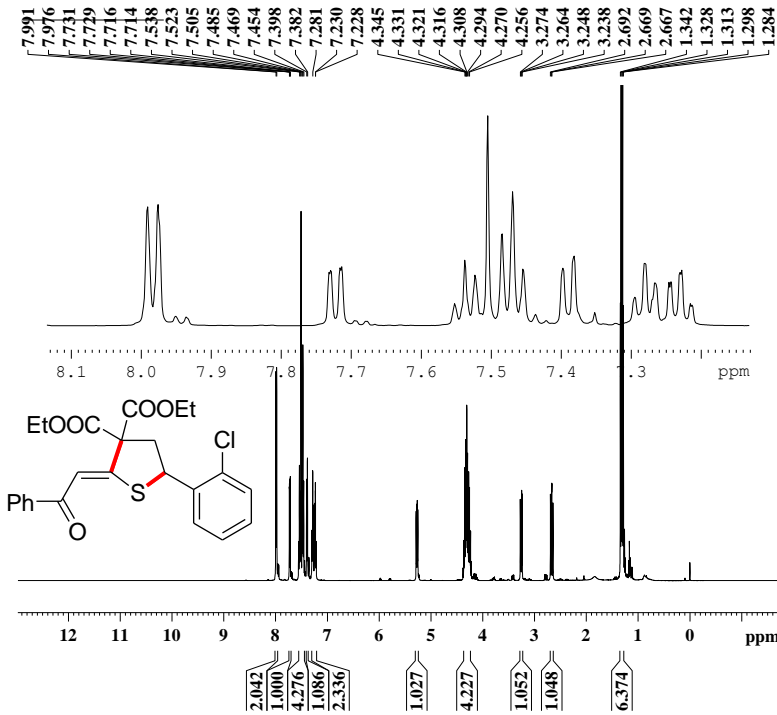


NAME WSW140607  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20140619  
 Time 21.03  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 879  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 287  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 299.8 K  
 D1 2.0000000 sec  
 d11 0.0300000 sec  
 DELTA 1.89999998 sec  
 TD0 20

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326479 MHz  
 WDW EM  
 SSB 0  
 LB 6.00 Hz  
 GB 0  
 PC 4.00

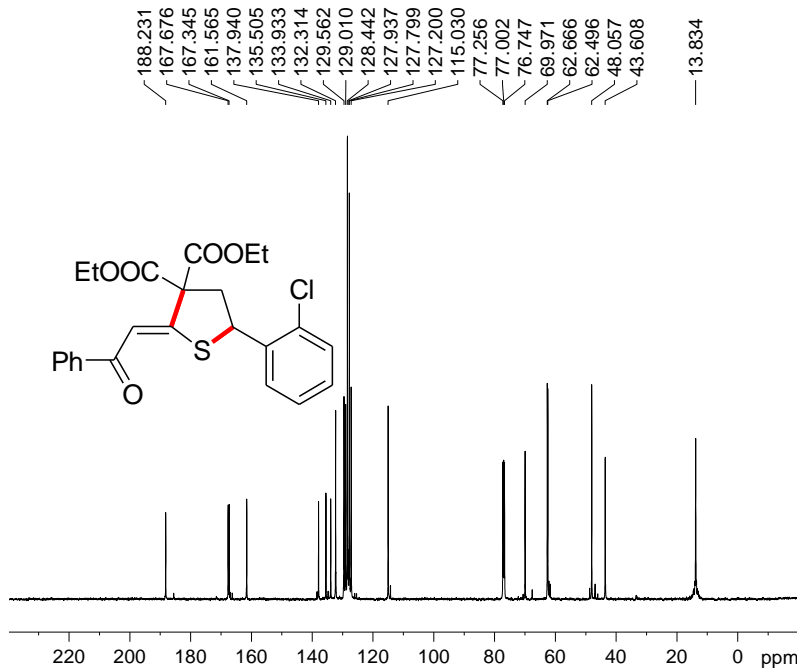
WSW140609 1H 1D 2014 06 17



NAME WSW140609  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20140617  
 Time 11.17  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 8  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 64  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300054 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 4.00

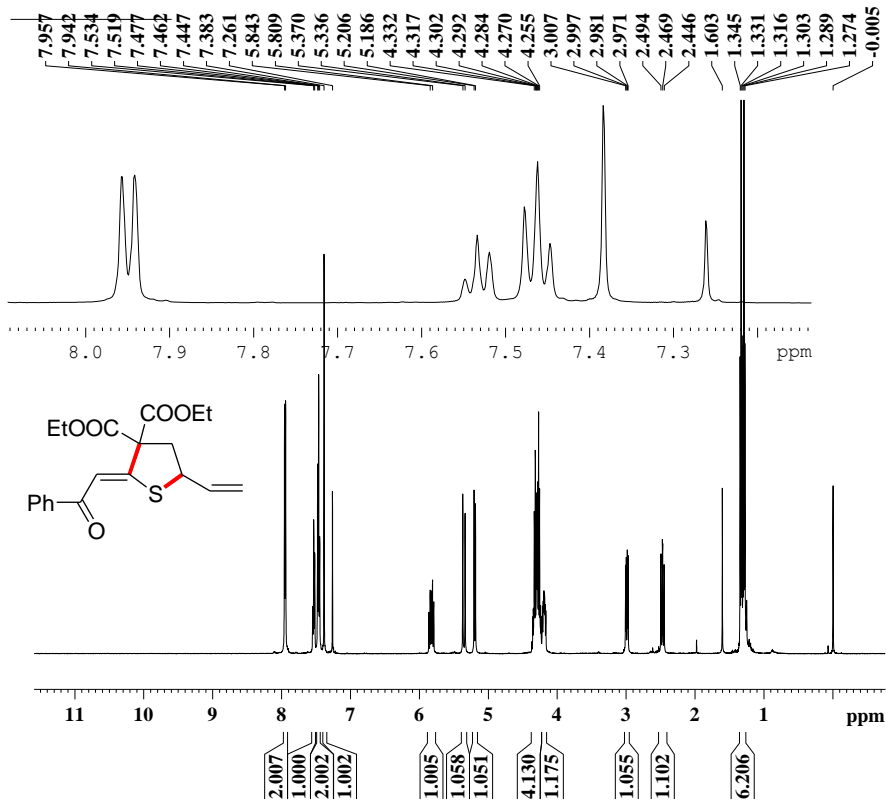
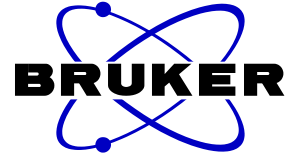
WSW140609 13C 1D 2014 06 23



NAME WSW140609  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20140623  
 Time 9.26  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 624  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 287  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 301.4 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 20

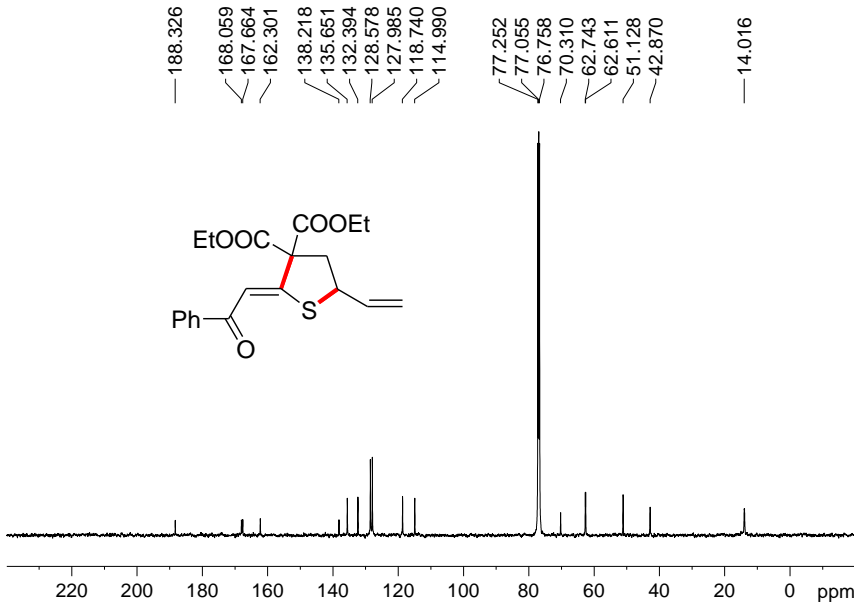
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326682 MHz  
 WDW EM  
 SSB 0  
 LB 6.00 Hz  
 GB 0  
 PC 4.00



NAME WSW141202  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20141203  
 Time 11.13  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 287  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 673.2 K  
 D1 1.00000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300099 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00



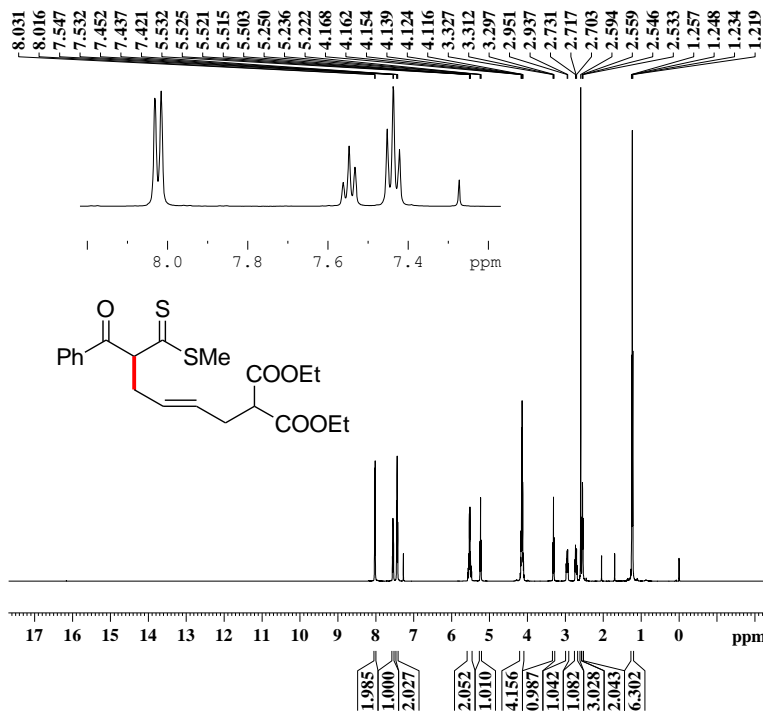
NAME WSW141202  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20141203  
 Time 18.48  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 725  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 4600  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 673.2 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 20

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326484 MHz  
 WDW EM  
 SSB 0  
 LB 10.00 Hz  
 GB 0  
 PC 1.00



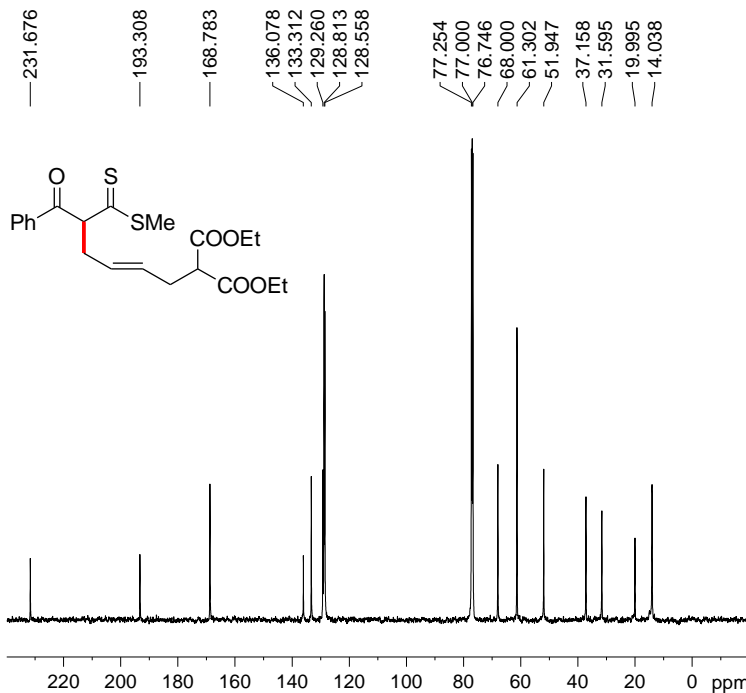
WSW141125 1H 1D 2014 12 01



NAME WSW141125  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20141201  
 Time 15.56  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 16384  
 SOLVENT CDCl3  
 NS 16  
 DS 1  
 SWH 10000.000 Hz  
 FIDRES 0.610352 Hz  
 AQ 0.8193000 sec  
 RG 144  
 DW 50.000 usec  
 DE 8.00 usec  
 TE 673.2 K  
 D1 1.00000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.00 usec  
 PL1 2.00 dB  
 SFO1 500.0338500 MHz  
 SI 16384  
 SF 500.0300040 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

WSW121125 13C 1D 2014 12 03



NAME WSW141125  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20141203  
 Time 18.10  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 536  
 DS 2  
 SWH 32679.738 Hz  
 FIDRES 0.498653 Hz  
 AQ 1.0027661 sec  
 RG 1820  
 DW 15.300 usec  
 DE 6.00 usec  
 TE 673.2 K  
 D1 2.00000000 sec  
 d11 0.03000000 sec  
 DELTA 1.89999998 sec  
 TD0 20

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 12.20 usec  
 PL1 3.00 dB  
 SFO1 125.7464750 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 2.00 dB  
 PL12 17.70 dB  
 PL13 17.70 dB  
 SFO2 500.0355000 MHz  
 SI 32768  
 SF 125.7326531 MHz  
 WDW EM  
 SSB 0  
 LB 10.00 Hz  
 GB 0  
 PC 4.00