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# Supporting Information

## Stereoselective synthesis of dispiro heterocycles by [3+2] cycloaddition of azomethine ylides with a thiazolo[3,2-a]indole derivative

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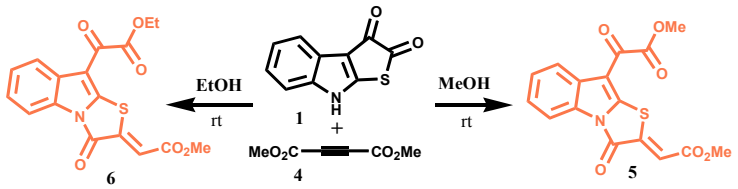
**Experimental Section**

**General remarks:** NMR spectra were recorded on a Bruker AVANCE III HD 400 MHz spectrometer with TMS as an internal standard. IR spectra were recorded on an Agilient Cary 630 FTIR spectrometer. DMSO-$d_6$ or $CDCl_3$ were used as the solvents for NMR analysis. Chemical shifts are given in ppm and coupling constants (*J*) are given in Hz. The purity of the samples was ascertained using PE 2400 series II CHNS/O Analyser. Dry glasswares were used for experiments. The chemicals used were purchased from Sigma-Aldrich and Spectrochem Pvt. Ltd and were used without further purification. Melting points were recorded on an electrothermal digital melting point apparatus from Analab Scientific instruments Pvt. Ltd. Commercial grade solvents were used. Thin layer chromatography was performed on silica gel coated on aluminium sheets and was monitored using UV light of wavelength 254 nm. Column chromatography was performed on 100-200 mesh silica gel. Compounds were eluted by a mixture of hexane and ethyl acetate.

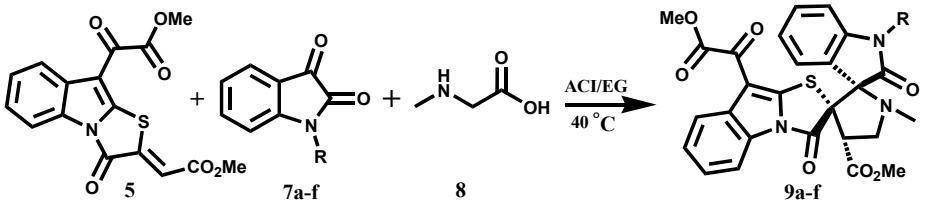



**General Procedure for the Reaction of Thieno[2,3-b]indole-2,3-dione and Dimethyl acetylenedicarboxylate in Methanol/Ethanol**: Thieno[2,3-b]indole-2,3-dione **1** (1 mmol) was dissolved in methanol/ethanol (8 ml) in a 100 ml round bottomed flask and was stirred for 15 minutes. To this stirred solution, dimethyl acetylenedicarboxylate **4** (1 mmol) was added and stirring was continued for 4 h. The reaction was monitored by TLC and precipitate formed was filtered and dried appropriately to obtain the pure product **5**/**6**.

**(2-Methoxycarbonylmethylene-3-oxo-2,3-dihydro thiazolo[3,2-b]indole-9 yl)-oxo-acetic acid methyl ester (5):** Thieno[2,3-b]indole-2,3-dione **1** (203 mg, 1 mmol) was reacted with dimethyl

acetylenedicarboxylate **4** (142 mg, 1 mmol) in 8 mL methanol following the general procedure mentioned above. The compound **5** was obtained as an orange powder (338 mg, 98%); mp 139-141°C; IR (KBr, v, $cm^{-1}$): 3110, 3167, 2934, 1721, 1710, 1690, 1680, 1624, 1545, 1300, 790. $^{1}H$ NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.28-8.26 (m, 1H), 8.15-8.13 (m, 1H), 7.44-7.36 (m, 2H), 7.15 (s, 1H), 4.03 (s, 3H), 3.93 (s, 3H). $^{13}C$ NMR (100 MHz, $CDCl_3$) (δ, ppm): 175.7, 165.7, 162.7, 160.0, 149.6, 145.1, 132.6, 131.6, 126.9, 126.1, 122.8, 121.2, 113.2, 112.9, 53.5, 52.9. Anal. calcd (%) for $C_{16}H_{11}NO_6S$: C, 55.65; H, 3.21; N, 4.06; S, 9.29. Found: C, 55.63; H, 3.20; N, 4.04; S, 9.28.

**(2-Ethoxycarbonylmethylene-3-oxo-2,3-dihydro-thiazolo[3,2-a]indol-9-yl)-oxo-acetic acid ethyl ester (6):** Thieno[2,3-b]indole-2,3-dione **1 (**203 mg,1 mmol) was reacted with dimethyl acetylenedicarboxylate **4** (142 mg, 1 mmol) in 8 mL ethanol following the general procedure mentioned above. Compound **6** was obtained as a yellow powder (362 mg, 97%); mp 150-151°C; IR (v, $cm^{-1}$): 3120, 3100, 2835, 1725, 1716, 1690, 1677, 1600, 1445, 1313, 1280, 820. $^{1}H$ NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.28-8.26 (m, 1H), 8.14-8.12 (m, 1H), 7.42-7.38 (m, 2H), 7.14 (s, 1H), 4.50 (q, J = 7.2 Hz, 2H), 4.07 (s, 3H), 1.47 (t, J = 6.8 Hz, 3H). $^{13}C$ NMR (100 MHz, $CDCl_3$) (δ, ppm): δ 176.9, 165.5, 162.6, 160.0, 147.6, 145.1, 137.0, 132.5, 131.5, 126.8, 126.0, 123.0, 113.1, 112.9, 60.9, 53.5, 21.5. Anal. calcd (%) for $C_{17}H_{13}NO_6S$: C, 56.82; H, 3.65; N, 3.90; S, 8.92. Found: C, 56.80; H, 3.66; N, 3.91; S, 8.90.




**General Procedure for the Cycloaddition of Thiazolo[3,2-b]indole derivative, Isatin and α-amino acids**: A mixture of (2-Methoxycarbonylmethylene-3-oxo-2,3-dihydro thiazolo[3,2-

b]indole-9 yl)-oxo-acetic acid methyl ester **5** (0.3 mmol), isatin (**7a-f)** (0.3 mmol) and α-amino acid (**8**/**10**/**12**) (0.4 mmol) were stirred in 2 ml ACI/EG eutectic solvent under 40 °C for 2h. Completion of the reaction was monitored by TLC and solvent was removed by vacuum distillation. The residue was then subjected to recrystallization in methanol to obtain pure products. The spectral data for the compounds are given below.

**9a:** The dipolarophile **5** (103 mg, 0.3 mmol), isatin **7a** (44 mg, 0.3 mmol) and sarcosine **8** (36 mg, 0.4 mmol) were reacted together as per the general procedure given above. Compound **9a** was obtained as a white powder (151 mg, 97%); mp 258-260 °C; IR (v, $cm^{-1}$): 3461, 3262, 2946, 2868, 1732, 1710, 1680, 1620, 1445, 1318, 711. $^{1}H$ NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.12 (d, J = 8 Hz, 1H), 7.98 (t, J = 6.8 Hz, 2H), 7.75 (d, J = 7.6 Hz, 1H), 7.32 (t, J = 8.0 Hz, 1H), 7.26 (s, NH, 1H), 7.24-7.18 (m, 1H), 6.96 (t, J = 7.6 Hz, 1H), 6.78 (d, J = 7.6 Hz, 1H), 5.23 (dd, J = 10.8 Hz, 7.2 Hz, 1H), 4.04 (s, 3H), 3.86 (dd, J = 9.2 Hz, 7.2 Hz, 1H), 3.61 (t, J = 10.4 Hz, 1H), 3.44 (s, 3H). $^{13}C$ NMR (100 MHz, $CDCl_3$) (δ, ppm): δ 176.9, 175.2, 169.4, 167.0, 163.2, 151.3, 141.8, 132.1, 131.1, 130.9, 128.6, 126.4, 125.0, 122.5, 122.2, 121.7, 113.0, 111.2, 110.2, 78.2, 74.6, 53.3, 52.4, 50.9, 50.3, 34.8. Anal. calcd (%) for $C_{26}H_{21}N_3O_7S$: C, 60.11; H, 4.07; N, 8.09; S, 6.17. Found: C, 60.01; H, 4.01; N, 8.04; S, 6.19. The COSY, HSQC, DEPT-135 and HMBC spectra are provided in the spectral section. The 2D $^{1}H$-$^{1}H$ COSY spectrum reveals the correlation between the protons at δ 5.23 ppm with that at δ 3.86 and 3.61 ppm indicating the strong coupling between the pyrrolidine ring protons. The HMBC spectrum shows correlation of the ester carbonyl with the protons at δ 5.23 ppm, and also with the methylene protons at δ 3.86 and 3.61 ppm. The spiro carbons at 74.6 and 78.2 ppm show strong correlations with protons at δ 5.23 ppm and with the methylene protons respectively.

**9b**: Dipolarophile **5** (103 mg, 0.3 mmol), N-methyl isatin **7b** (48 mg, 0.3 mmol) and sarcosine **8** (35.6 mg, 0.4 mmol) were reacted as per the general procedure. Compound **9b** was obtained as a white powder (152 mg, 95%); mp 265-266 °C; IR (v, $cm^{-1}$): 3400, 3132, 2934, 2860, 1728, 1712, 1680, 1689, 1456, 1300, 890, 700. $^{1}H$ NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.22 (d, J = 7.6 Hz, 1H), 8.15-8.11 (m, 2H), 7.85 (d, J = 7.2 Hz, 1H), 7.45-7.44 (m, 1H), 7.44-7.26 (m, 2H), 7.12 (t, J = 7.6 Hz, 1H), 6.85 (d, J = 7.2 Hz, 1H), 5.25-5.20 (m, 1H), 4.11 (s, 3H), 3.98-3.94 (m, 1H), 3.65-3.45 (m, 1H), 3.43 (s, 3H), 3.30 (s, 3H), 2.21 (s, 3H). $^{13}C$ NMR (100 MHz, $CDCl_3$) (δ, ppm): δ 177.4, 171.0, 167.7, 166.3, 165.6, 152.2, 135.5, 133.2, 132.3, 131.7, 130.9, 129.3, 129.2, 129.1, 129.0, 128.9, 128.8, 112.6, 111.8, 71.8, 71.2, 57.8, 53.3, 52.9, 52.8, 47.3, 31.9. Anal. calcd (%) for $C_{27}H_{23}N_3O_7S$: C, 60.78; H, 4.34; N, 7.88; S, 6.01. Found: C, 60.77; H, 4.31; N, 7.87; S, 6.00.

**9c**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), N-ethyl isatin **7c** (52.5 mg, 0.3 mmol) and sarcosine **8** (36 mg, 0.4 mmol). White powder (154 mg, 94%); mp 271-273 °C; IR (v, $cm^{-1}$): 3368, 3160, 2932, 2867, 1725, 1713, 1687, 1681, 1412, 1332, 870, 743. $^{1}H$ NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.13 (d, J = 8.0 Hz, 1H), 7.99 (d, J = 8.0 Hz, 1H), 7.75 (d, J = 7.2 Hz, 1H), 7.35-7.22 (m, 3H), 6.95 (t, J = 7.6 Hz, 1H), 6.73 (q, J = 7.6 Hz, 1H), 5.25 (dd, J = 11.2 Hz, 7.2 Hz, 1H), 4.03 (s, 3H), 3.94-3.82 (m, 2H), 3.66-3.61 (m, 2H), 3.43 (s, 3H), 2.17 (s, 3H), 1.27 (t, J = 7.2 Hz, 3H). $^{13}C$ NMR (100 MHz, $CDCl_3$) (δ, ppm): 176.9, 173.3, 170.2, 169.5, 166.7, 151.4, 144.2, 143.8, 130.8, 128.2, 126.3 (2C), 126.1, 125.2, 124.9, 121.9, 121.7, 112.9, 112.7, 74.7, 71.7, 53.9, 53.3, 53.1, 52.4, 51.0, 35.2, 34.7. Anal. calcd (%) for $C_{28}H_{25}N_3O_7S$: C, 61.42; H, 4.60; N, 7.67; S, 5.86. Found: C, 61.40; H, 4.61; N, 7.69; S, 5.87.

**9d**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), N-propyl isatin **7d** (57 mg, 0.3 mmol) and sarcosine **8** (36 mg, 0.4 mmol). White powder

(151 mg, 90%); mp 277-279 °C; IR (v, $cm^{-1}$): 3378, 3190, 2965, 2897, 1720, 1716, 1685, 1680, 1518, 1339, 812, 787. $^{1}H$ NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.13 (d, J = 8.0 Hz, 1H), 7.99 (d, J = 8.0 Hz, 1H), 7.76 (d, J = 6.8 Hz, 1H), 7.35-7.28 (m, 1H), 7.26-7.21 (m, 2H), 6.96 (t, J = 6.8 Hz, 1H), 6.74 (d, J = 7.6 Hz, 1H), 5.26 (dd, J = 10.4 Hz, 6.8 Hz, 1H), 4.03 (s, 3H), 3.87-3.78 (m, 2H), 3.65-3.54 (m, 2H), 3.43 (s, 3H), 2.16 (s, 3H), 1.31-1.24 (m, 2H), 1.01 (t, J = 7.2 Hz, 3H). $^{13}C$ NMR (100 MHz, $CDCl_3$) (δ, ppm): δ 176.9, 173.5, 169.4, 166.7, 163.2, 151.4, 144.4, 132.4, 132.1, 131.1, 130.8, 128.2, 126.3, 124.9, 122.2, 121.9, 121.6, 111.1, 108.5, 79.0, 74.6, 53.3, 52.3, 51.0, 50.2, 41.8, 36.0, 34.8, 34.6. Anal. calcd (%) for $C_{29}H_{27}N_3O_7S$: C, 62.02; H, 4.85; N, 7.48; S, 5.71. Found: C, 62.00; H, 4.81; N, 7.43; S, 5.79.

**9e**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), N-allyl isatin **7e** (56 mg, 0.3 mmol) and sarcosine **8** (36 mg, 0.4 mmol). White powder (158 mg, 94%); mp 275-277 °C. IR (v, $cm^{-1}$): 3468, 3156, 2952, 2900, 1723, 1718, 1685, 1691, 1489, 1334, 867, 749. $^{1}H$ NMR (400 MHz, DMSO) (δ, ppm): 7.97 (d, J = 7.6, 1H), 7.91 (d, J = 7.2 Hz, 1H), 7.66 (d, J = 7.6 Hz, 1H), 7.47-7.34 (m, 2H), 7.29-7.25 (m,1H), 7.01-6.97 (m, 1H), 6.89 (d, J = 8.0 Hz, 1H), 5.88-5.79 (m, 1H), 5.21-5.10 (m, 3H), 4.47-4.42 (m, 1H), 4.25 (dd, J = 16.4 Hz, 5.2 Hz, 1H), 3.98 (s, 3H), 3.75-3.71 (m, 1H), 3.52 (t, J = 9.6, 1H), 3.40 (s, 3H), 2.10 (s, 3H). $^{13}C$ NMR (100 MHz, DMSO) (δ, ppm): 177.5, 172.7, 169.5, 166.7, 163.3, 148.8 (2C), 144.1, 142.6, 131.8, 131.7, 131.5, 130.7, 127.8, 127.0, 125.8, 122.6, 121.6, 121.5, 110.7, 110.2, 77.9, 75.0, 53.8, 52.8, 51.1, 50.0, 42.1, 35.0. Anal. calcd (%) for $C_{29}H_{25}N_3O_7S$: C, 62.24; H, 4.50; N, 7.51; S, 5.73. Found: C, 62.20; H, 4.55; N, 7.48; S, 5.79.

**9f**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mm0l), N-benzyl isatin **7f** (71 mg, 0.3 mmol) and sarcosine **8** (36 mg, 0.4 mmol). White powder (170 mg, 93%). mp 281-283 °C. IR (v, $cm^{-1}$): 3543, 3234, 2897, 2800, 1724, 1720, 1697, 1687,

1546, 1444, 907, 649. $^{1}$H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.13 (d, J = 7.6 Hz, 1H), 8.02 (d, J = 8 Hz, 1H), 7.77 (d, J = 7.6 Hz, 1H), 7.37-7.26 (m, 7H), 7.11 (t, J = 7.6 Hz, 1H), 6.93 (t, J = 7.6, 1H), 6.56 (d, J = 7.6 Hz, 1H), 5.30 (dd, J = 10.4 Hz, 7.2 Hz, 1H), 5.09 (d, J = 15.6 Hz, 1H), 4.79 (d, J = 15.6 Hz, 1H), 4.03 (s, 3H), 3.89 (q, J = 9.2 Hz, 1H), 3.65 (q, J = 10.4 Hz, 1H), 3.45 (s, 3H), 2.20 (s, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) (δ, ppm): 176.9, 173.7, 169.4, 166.9, 163.1, 151.3, 144.0, 135.2, 132.1, 131.2, 130.8, 128.7, 128.2 (2C), 127.7, 127.4 (2C), 126.3, 125.0, 122.5, 121.8, 121.7, 110.0, 109.6, 74.6, 71.8, 53.3, 53.0, 52.4, 51.0, 50.3, 34.8.; Anal. calcd (%) for $C_{33}H_{27}N_3O_7S$: C, 65.01; H, 4.46; N, 6.89; S, 5.26. Found: C, 65.04; H, 4.43; N, 6.84; S, 5.29.

**11aa**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), isatin **7a** (44 mg, 0.3 mmol) and proline **10a** (46 mg, 0.4 mmol). Pale yellow powder (154 mg, 94%). mp 266-268 °C. IR (v, $cm^{-1}$): 3413, 3298, 2956, 2830, 1724, 1722, 1691, 1680, 1500, 1445, 1324, 1200, 896, 640. $^{1}$H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.72 (s, 1H), 8.22-8.19 (m, 2H), 7.46.7.41 (m, 1H), 7.40-7.36 (m, 2H), 7.28-7.24 (m, 1H), 7.11-7.07 (m, 1H), 6.96 (d, J = 7.6 Hz, 1H), 4.75 (s, 1H)**,** 4.57 (q, J = 6.0 Hz, 1H), 4.03 (s, 3H), 3.33 (s, 3H), 2.77-2.71 (m, 2H), 2.33-2.31 (m, 1H ), 2.17-2.04 (m, 1H), 1.94-1.87 (m, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) (δ, ppm): 177.4, 171.8, 166.9, 161.8, 160.7, 147.0, 139.2, 138.5, 138.4, 125.7, 124.8, 124.7, 124.4, 124.0, 123.9, 123.5, 119.1, 115.9, 114.0, 71.1, 70.1, 58.9, 58.8, 56.5, 46.3, 30.3, 30.2, 29.7.; Anal. calcd (%) for $C_{28}H_{23}N_3O_7S$: C, 61.64; H, 4.25; N, 7.70; S, 5.88. Found: C, 61.65; H, 4.27; N, 7.71; S, 5.89.

**11ba**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), N-methyl isatin **7b** (48 mg, 0.3 mmol) and proline **10a** (46 mg, 0.4 mmol). Pale yellow powder (149 mg, 89%). mp 274-276 °C. IR (v, $cm^{-1}$): 3433, 3010, 2901, 2815, 1727, 1722, 1695, 1680, 1565, 1475, 1367, 12120, 813, 723, 701. $^{1}$H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.22 (d, J

= 6.4 Hz, 2H), 8.19-7.92 (m, 3H), 7.45-7.26 (m, 3H), 4.99 (s, 1H), 4.75 (d, J = 7.2 Hz, 1H), 4.04 (s, 3H), 3.43 (s, 3H), 3.31 (s, 3H), 2.72-2.63 (m, 2H), 2.10-2.05 (m, 2H), 1.88-1.63 (m, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) (δ, ppm): 177.4, 171.0, 167.7, 166.3, 165.6, 152.2, 135.5, 133.2, 132.3, 131.7, 130.9, 129.3, 129.2, 129.1, 129.0, 128.9, 128.8, 112.6, 111.8, 71.8, 71.2, 57.8, 53.3, 52.9, 52.8, 47.3, 31.9. Anal. calcd (%) for $C_{29}H_{25}N_3O_7S$: C, 62.24; H, 4.50; N, 7.51; S, 5.73. Found: C, 62.25; H, 4.51; N, 7.53; S, 5.70.

**11ca**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), N-ethyl isatin **7c** (53 mg, 0.3 mmol) and proline **10a** (46 mg, 0.4 mmol). Pale yellow powder (146 mg, 85%). mp 283-285 °C. IR (v, $cm^{-1}$): 3453, 3043, 2943, 2832, 1726, 1717, 1698, 1670, 1632, 1512, 1423, 1243, 843. $^1$H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.13 (d, J = 6.8 Hz, 1H), 7.55 (d, J = 7.6 Hz, 1H), 7.44-7.40 (m, 2H), 7.39-7.30 (m, 1H), 7.29-7.13 (m, 1H), 7.11-7.09 (m, 1H), 6.90 (d, J = 8 Hz, 1H), 4.68 (s, 1H), 4.05 (d, J = 8.8 Hz, 1H), 3.99 (s, 3H), 3.46 (q, J = 7.2 Hz, 2H), 3.37 (s, 3H), 2.73-2.64 (m, 1H), 2.31-2.03 (m, 1H), 1.34-1.23 (m, 4H), 1.04 (t, J = 6.4 Hz, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) (δ, ppm): 177.0, 174.6, 168.4, 167.7, 166.6, 149.3, 148.3, 134.3, 131.9, 131.8, 131.6, 127.7, 127.5, 126.6, 126.2, 123.8, 121.4, 112.5, 111.6, 79.3, 70.5, 56.0, 50.3, 45.0, 41.6, 41.3, 35.2, 27.9, 27.7, 26.5. Anal. calcd (%) for $C_{30}H_{27}N_3O_7S$: C, 62.82; H, 4.74; N, 7.33; S, 5.59. Found: C, 62.80; H, 4.73; N, 7.34; S, 5.58.

**11ea**: Prepared according to the general procedure by using thiazolo derivative **5** (65 mg, 0.3 mmol), N-allyl isatin **7e** (67 mg, 0.3 mmol) and proline **10a** (26 mg, 0.4 mmol). Pale yellow powder (156 mg, 89%). mp 289-291 °C. IR (v, $cm^{-1}$): 3156, 3190, 2978, 2800, 1720, 1710, 1690, 1675, 1612, 1567, 1523, 1273, 1123, 980. $^1$H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.04 (d, J = 6.8 Hz, 1H), 7.88 (d, J = 7.6 Hz, 1H), 7.80 (d, J = 6.8 Hz, 1H), 7.35-7.21 (m, 2H), 6.99-6.95 (m, 2H), 6.63 (d, J = 8.4 Hz, 1H), 5.69-5.62 (m, 1H), 5.28-5.23 (m, 2H), 4.56 (s, 1H), 4.19-4.11 (m, 1H),

3.82 (s, 3H), 3.34 (s, 3H), 2.81-2.71 (m, 2H), 1.94-1.88 (m, 3H), 1.64-1.50 (m, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) (δ, ppm): 177.5, 175.7, 171.5, 166.4, 163.2, 143.4, 138.9 (2C), 138.5, 138.1, 135.7, 135.4, 135.2, 132.1, 132.0, 130.2, 129.6, 128.9, 128.8, 113.0, 112.9, 74.6, 72.6, 52.6, 52.4, 50.5, 47.2, 44.4, 31.9, 31.3. Anal. calcd (%) for $C_{31}H_{27}N_3O_7S$: C, 63.58; H, 4.65; N, 7.18; S, 5.48. Found: C, 63.59; H, 4.63; N, 7.16; S, 5.49.

**11fa**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), N-benzyl isatin **7f** (71 mg, 0.3 mmol) and proline **10a** (46 mg, 0.4 mmol). Pale yellow powder (171 mg, 90%). mp 299-301°C. IR (v, $cm^{-1}$): 3100, 3012, 2967, 2900, 2834, 1728, 1717, 1697, 1678, 1601, 1590, 1267, 1100, 956. $^1$H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.27-8.21 (m, 1H), 8.08 (d, J = 7.6 Hz, 1H), 7.73 (d, J = 6.8 Hz, 1H), 7.67-7.40 (m, 3H), 7.39-7.21 (m, 3H), 7.19-7.03 (m, 3H), 6.66 (t, J = 6 Hz, 1H), 5.08 (s, 1H), 4.90-4.61 (m, 1H), 4.04-4.01 (m, 2H), 3.96 (s, 3H), 3.32 (s, 3H), 2.70-2.68 (m, 1H), 2.36-2.21 (m, 2H), 1.92-1.89 (m, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) (δ, ppm): 177.5, 176.2, 173.6, 167.4, 167.2, 151.6, 151.5, 136.5, 136.2, 134.5, 134.4, 131.0, 129.7, 129.4, 129.3, 129.2, 129.1, 129.0, 128.8, 128.7, 128.0, 127.8 (2C), 116.3, 115.9, 78.5, 75.0, 50.5, 49.9, 49.4, 48.9, 46.8, 37.4, 36.7, 36.5. Anal. calcd (%) for $C_{35}H_{29}N_3O_7S$: C, 66.13; H, 4.60; N, 6.61; S, 5.04. Found: C, 66.10; H, 4.62; N, 6.62; S, 5.02.

**11ab**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), isatin **7a** (44 mg, 0.3 mmol) and thioproline **10b** (53 mg, 0.4 mmol). Pale yellow powder (160 mg, 95%). mp 268-271°C. IR (v, $cm^{-1}$): 3156, 3010, 2907, 2965, 2884, 1720, 1715, 1690, 1688, 1643, 1556, 1212, 1167, 790. $^1$H NMR (400 MHz, DMSO) (δ, ppm): 7.98-7.94 (m, 2H), 7.65 (d, J = 7.6 Hz, 1H), 7.44-7.36 (m, 2H), 7.20-7.16 (m, 1H), 7.11 (s, NH), 6.94-6.90 (m, 1H), 6.74 (d, J = 8.0 Hz, 1H), 5.11 (d, J = 8.4 Hz, 1H), 4.39-4.37 (m, 1H), 3.98 (s, 3H), 3.49 (s, 3H), 3.29-3.20 (m, 2H), 3.16-3.05 (m, 2H). $^{13}$C NMR (100 MHz, DMSO) (δ, ppm): 177.4, 173.8,

168.8, 165.1, 163.2, 153.8, 149.6, 140.7, 131.7, 130.7, 127.8, 127.1, 125.9, 123.6, 123.1, 122.6, 122.5, 116.0, 112.8, 78.8, 76.7, 53.8, 53.2, 53.1, 50.7, 41.9, 36.0. 32.5. Anal. calcd (%) for $C_{27}H_{21}N_3O_7S_2$: C, 57.54; H, 3.76; N, 7.46; S, 11.38. Found: C, 57.55; H, 3.73; N, 7.43; S, 11.35.

**11bb**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), N-methyl isatin **7b** (48 mg, 0.3 mmol) and thioproline **10b** (53 mg, 0.4 mmol). Pale yellow powder (158 mg, 91%). mp 272-274 °C. IR (v, $cm^{-1}$): 3113, 3078, 2943, 2900, 2867, 1718, 1710, 1689, 1679, 1600, 1500, 1456, 1100, 890. $^1H$ NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.14 (d, J = 8.0 Hz, 1H), 7.98 (d, J = 8.0 Hz, 1H), 7.79 (d, J = 6.8 Hz, 1H), 7.36-7.21 (m, 3H), 6.98-6.93 (m, 1H), 6.74 (d, J = 6.8 Hz, 1H), 5.71 (d, J = 7.2 Hz, 1H), 4.59-4.51 (m, 1H), 3.95 (s, 3H), 3.62 (d, J = 8.0 Hz, 1H), 3.51 (s, 3H), 3.32 (d, J = 8.0 Hz, 2H), 3.29 (s, 3H), 3.04-3.02 (m, 1H). $^{13}C$ NMR (100 MHz, $CDCl_3$) (δ, ppm): 177.8, 175.1, 170.0, 166.4, 164.1, 150.4, 139.2, 132.1, 131.2, 131.1, 125.0, 124.0, 123.4, 123.0, 122.4, 121.8, 115.9, 114.0, 112.9, 78.8, 68.3, 60.3, 53.4, 53.3, 52.0, 50.0, 36.2, 35.2. Anal. calcd (%) for $C_{28}H_{23}N_3O_7S_2$: C, 58.22; H, 4.01; N, 7.27; S, 11.10. Found: C, 58.20; H, 4.00; N, 7.29; S, 11.09.

**11cb**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), N-ethyl isatin **7c** (53 mg, 0.3 mmol) and thioproline **10b** (53 mg, 0.4 mmol). Pale yellow powder (165 mg, 93%). mp 282-285 °C. IR (v, $cm^{-1}$): 3190, 3097, 2900, 287900, 2867, 1722, 1709, 1686, 1674, 1667, 1567, 1400, 1198, 985. $^1H$ NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.13 (d, J = 8.0 Hz, 1H), 7.98 (d, J = 8.0 Hz, 1H), 7.79 (d, J = 7.6 Hz, 1H), 7.35-7.21 (m, 3H), 6.99-6.95 (m, 1H), 6.73 (d, J = 7.6 Hz, 1H), 5.21 (d, J = 8.4 Hz, 1H), 4.54-4.49 (m, 1H), 4.04 (s, 3H), 3.99-3.93 (m, 1H), 3.80 (d, J = 8.0 Hz**,** 1H), 3.62 (q, J = 7.2 H, 1H), 3.52 (d, J = 8.4 Hz, 2H), 3.49 (s, 3H), 3.24 (q, J = 6.0 Hz, 1H), 1.32 (t, J = 7.2 Hz, 3H). $^{13}C$ NMR (100 MHz, $CDCl_3$) (δ, ppm): 176.8, 174.1, 168.6, 165.1, 163.1, 150.4, 143.1, 131.2, 131.1, 128.2, 126.4, 125.0, 124.0, 123.4,

123.0, 122.4, 121.8, 112.9, 111.4, 78.2, 68.2, 53.6, 53.3, 52.6, 50.9, 36.3, 35.2, 34.7, 33.8, 29.6. Anal. calcd (%) for $C_{29}H_{25}N_3O_7S_2$: C, 58.87; H, 4.26; N, 7.10; S, 10.84. Found: C, 58.85; H, 4.23; N, 7.11; S, 10.80.

**11db**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), N-propyl isatin **7d** (57 mg, 0.3 mmol) and thioproline **10b** (53 mg, 0.4 mmol). Pale yellow powder (162 mg, 89%). mp 287-289 °C. IR (v, $cm^{-1}$): 3190, 3097, 2900, 2879, 2867, 1722, 1709, 1686, 1674, 1667, 1567, 1400, 1198, 985. $^1H$ NMR (400 MHz, DMSO) (δ, ppm): 7.90 (d, J = 7.2 Hz, 1H), 7.82-7.79 (m, 1H), 7.62 (t, J = 6.4 Hz, 1H), 7.35-7.21 (m, 3H), 6.95-6.90 (m, 2H), 5.04 (d, J = 8.4 Hz, 1H), 4.35--4.31 (m, 1H), 3.92 (s, 3H), 3.82-3.79 (m, 1H), 3.74-3.63 (m, 1H), 3.39 (s, 3H), 3.35-3.22 (m, 3H), 2.90-2.80 (m, 1H), 1.59-1.50 (m, 2H), 0.84 (t, J = 7.2 Hz, 3H). $^{13}C$ NMR (100 MHz, DMSO) (δ, ppm): $^{13}C$ NMR (100 MHz, DMSO) (δ, ppm): 177.4, 173.8, 168.8, 165.1, 163.2, 153.8, 140.7, 131.7, 130.7, 127.8, 127.1, 125.9, 123.6, 123.1, 122.6, 122.5, 121.7, 112.8, 110.9, 109.8, 78.8, 76.7, 53.8, 53.2, 53.1, 50.7, 41.9, 36.0, 29.8. Anal. calcd (%) for $C_{30}H_{27}N_3O_7S_2$: C, 59.49; H, 4.49; N, 6.94; S, 10.59. Found: C, 59.47; H, 4.47; N, 6.91; S, 9.58.

**11eb**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), N-allyl isatin **7d** (56 mg, 0.3 mmol) and thioproline **10b** (53 mg, 0.4 mmol). Pale yellow powder (163 mg, 90%). mp 292-295 °C. IR (v, $cm^{-1}$): 3154, 3012, 2954, 2819, 2800, 1727, 1713, 1686, 1670, 1669, 1500, 1467, 1154, 756. $^1H$ NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.12 (d, J = 8.1 Hz, 1H), 7.98 (d, J = 7.9 Hz, 1H), 7.80 (d, J = 6.8 Hz, 1H), 7.35-7.19 (m, 3H), 6.98 (t, J = 7.6 Hz, 1H), 6.70 (d, J = 7.6 Hz, 1H), 5.86-5.82 (m, 1H), 5.28-5.22 (m, 2H), 5.20 (d, J = 8.4 Hz, 1H), 4.57-4.49 (m, 2H), 4.23-4.19 (m, 1H), 4.03 (s, 3H), 3.81 (d, J = 8.4 Hz, 1H), 3.54-3.48 (m, 1H), 3.47 (s, 3H), 3.26-3.22 (m, 1H). $^{13}C$ NMR (100 MHz, $CDCl_3$) (δ, ppm): 175.7, 173.3, 167.5,

164.2, 162.0, 149.3, 142.2, 131.1, 130.2, 130.0, 129.9, 127.1, 125.4, 124.1, 121.7, 121.6, 120.7, 117.2, 111.9, 110.4, 108.4, 77.4, 67.1, 52.7, 52.3, 51.6, 49.9, 41.9, 40.8, 35.3. Anal. calcd (%) for $C_{30}H_{25}N_3O_7S_2$: C, 59.69; H, 4.17; N, 6.96; S, 10.62. Found: C, 59.67; H, 4.15; N, 6.95; S, 10.60.

**11fb**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), N-benzyl isatin **7f** (71 mg, 0.3 mmol) and thioproline **10b** (53 mg, 0.4 mmol). Pale yellow powder (177 mg, 90%). mp 284-286 °C. IR (v, $cm^{-1}$): 3160, 3009, 2989, 2810, 1726, 1710, 1687, 1671, 1660, 1567, 1247, 1136, 915. $^1H$ NMR (400 MHz, DMSO) (δ, ppm): 7.98-7.89 (m, 2H), 7.73 (d, J = 6.8 Hz, 1H), 7.42-7.34 (m, 6H), 7.31-7.29 (m, 1H), 7.28-7.15 (m, 1H), 6.99-6.95 (m, 1H), 6.75 (d, J = 8 Hz, 1H), 5.17 (d, J = 8.4 Hz, 1H), 5.11 (d, J = 15.6 Hz, 1H), 4.80 (d, J = 13.8 Hz, 1H), 4.47-4.41 (m, 1H), 3.99 (s, 3H), 3.83 (d, J = 8.4 Hz, 1H), 3.49 (s, 3H), 3.48-3.32 (m, 2H), 3.26-3.22 (m, 1H). $^{13}C$ NMR (100 MHz, DMSO) (δ, ppm): 177.4, 174.2, 168.8, 165.2, 163.2, 149.4, 143.6, 136.1, 131.7, 131.6, 130.8, 129.0, 128.0 (2C), 127.1, 125.9, 122.8, 122.7, 121.7, 112.9, 111.0, 110.3, 78.7, 77.0, 68.2, 53.8, 53.4, 53.2, 50.8, 44.0, 36.2. Anal. calcd (%) for $C_{34}H_{27}N_3O_7S_2$: C, 62.47; H, 4.16; N, 6.43; S, 9.81. Found: C, 62.45; H, 4.13; N, 6.41; S, 9.82.

**13aa**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), isatin **7a** (44 mg, 0.3 mmol) and tryptophan **12a** (82 mg, 0.4 mmol). Pale yellow powder (162 mg, 85%). mp 290-293 °C. IR (v, $cm^{-1}$): 3324, 3245, 3153, 3043, 2987, 2800, 1729, 1713, 1680, 1676, 1659, 1568, 1208, 1100, 978. $^1H$ NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.51 (br s, 1H), 8.18-8.10 (m, 2H), 7.36-6.68 (m, 11H), 6.67 (br s, 1H), 4.73 (d, J = 9.2 Hz, 1H), 4.59-4.54 (m, 1H), 3.98 (s, 3H), 3.86 (s, 3H), 3.73-3.34 (m, 2H), 2.30 (s, 1H). $^{13}C$ NMR (100 MHz, $CDCl_3$) (δ, ppm): 178.4, 177.6, 176.7, 170.7, 169.4, 151.1, 139.3, 136.2, 131.9, 131.2, 130.9, 127.9, 127.5, 126.4, 125.2, 124.6, 124.5, 124.0, 123.5, 123.3, 123.2 (2C), 122.9, 111.7, 111.1, 110.8, 109.7,

73.0, 71.0, 60.3, 56.8, 53.2, 52.8, 42.8. Anal. calcd (%) for $C_{34}H_{26}N_4O_7S$: C, 64.34; H, 4.13; N, 8.83; S, 5.05. Found: C, 64.30; H, 4.12; N, 8.86; S, 5.0. The NOESY correlation peak of the doublet at δ 4.73 ppm with the multiplet at δ 4.54-4.59 ppm indicates the *cis* geometry of adjacent protons of **13aa**.

**13ab**: Prepared according to the general procedure by using thiazolo derivative **5** (103 mg, 0.3 mmol), isatin **7a** (44 mg, 0.3 mmol) and phenylalanine **12b** (66 mg, 0.4 mmol). Pale yellow powder (145 mg, 81%). mp 274-276 °C. IR (v, $cm^{-1}$): 3345, 3312, 3200, 3112, 3000, 2965, 2898, 1728, 1710, 1689, 1671, 1650, 1500, 1265, 1176, 906. $^1H$ NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.14 (s, NH), 8.00 (d, J = 8.0 Hz, 1H), 7.64 (d, J = 6.8 Hz, 1H), 7.38-7.28 (m, 4H), 7.26-7.07 (m, 4H), 7.05-6.94 (m, 2H), 6.60 (d, J = 8.0 Hz, 1H), 5.06 (d, J = 9.2 Hz, 1H), 4.68-4.60 (m, 1H), 4.01 (s, 2H), 3.92 (s, 3H), 3.65 (s, 3H), 2.04 (s, NH). $^{13}C$ NMR (100 MHz, $CDCl_3$) (δ, ppm): 177.9, 173.9, 168.0, 165.0, 160.7, 156.5, 135.3, 134.4, 130.5, 130.4, 129.7, 129.2, 129.0, 128.5, 128.4, 128.3, 128.2, 127.8, 127.7, 127.3, 127.2, 127.0, 106.9, 104.0, 78.9, 70.5, 54.8, 54.3, 53.9, 53.3, 40.0. Anal. calcd (%) for $C_{32}H_{25}N_3O_7S$: C, 64.53; H, 4.23; N, 7.05; S, 5.38. Found: C, 64.50; H, 4.24; N, 7.04; S, 5.37.

## Proton and Carbon-13 NMR spectra

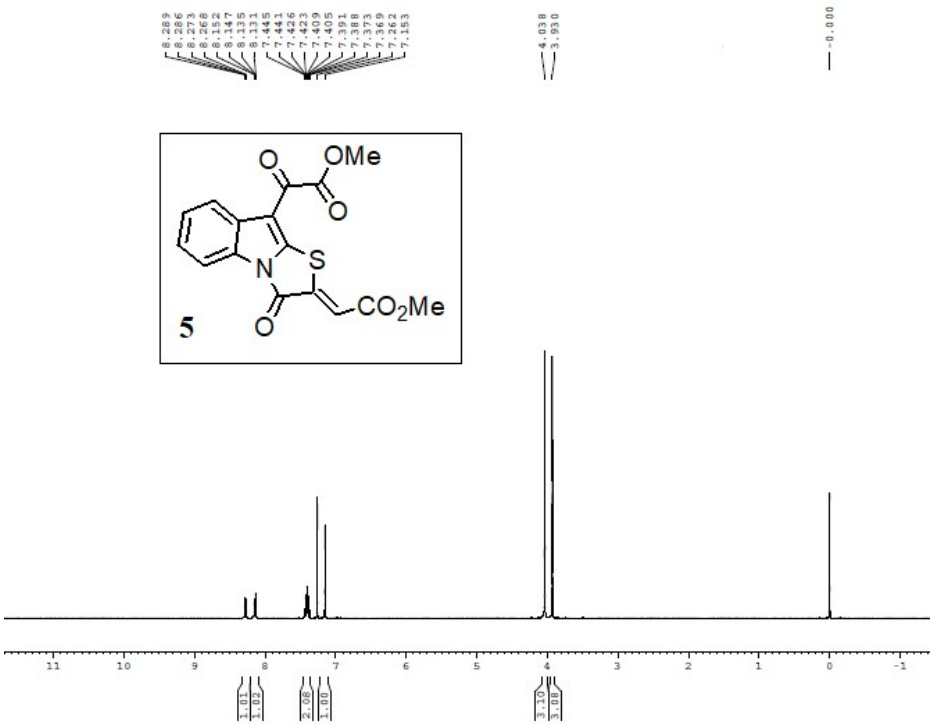



**Figure S1**. $^{1}H$ NMR of Compound **5** (400 MHz, $CDCl_3$)

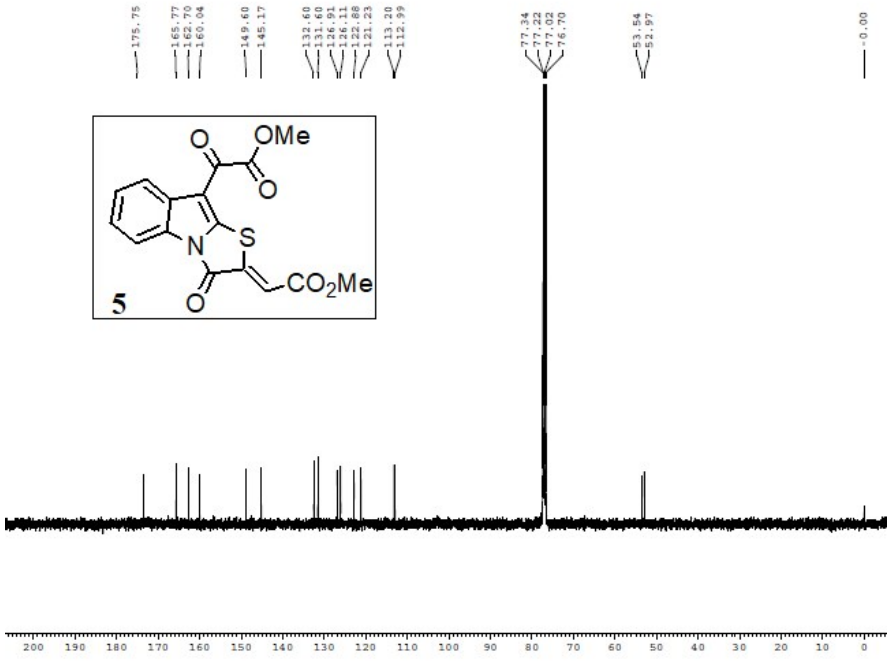



**Figure S2**. $^{13}C$ NMR of Compound **5** (100 MHz, $CDCl_3$)

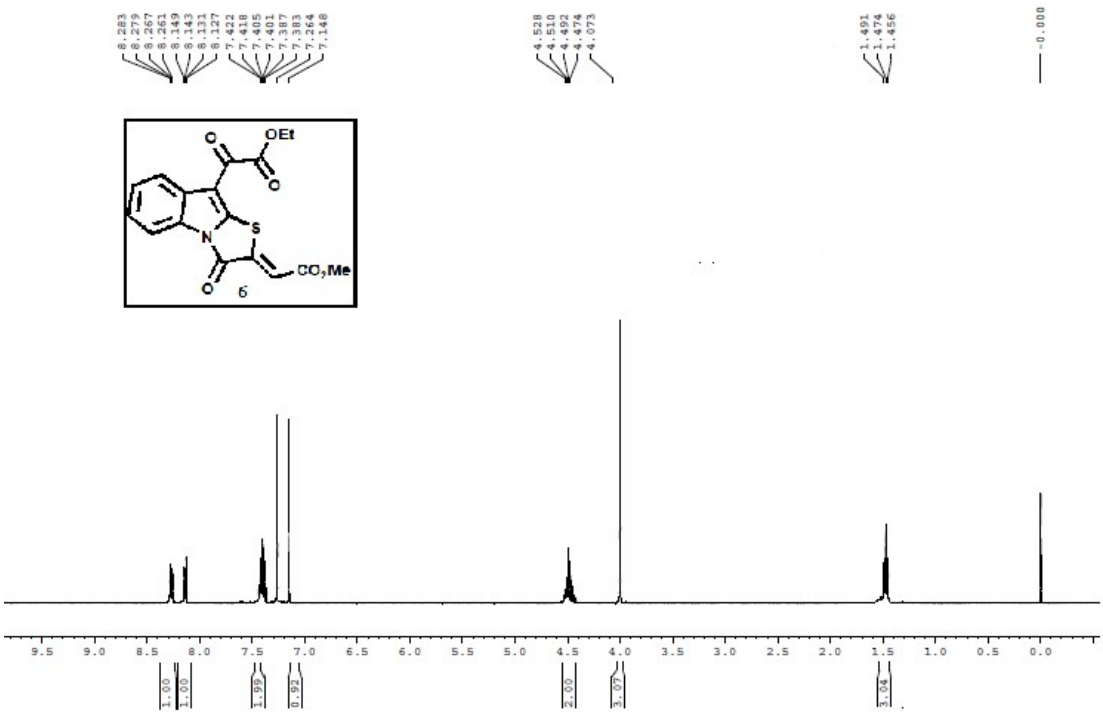



**Figure S3**. $^{1}$H NMR of Compound **6** (400 MHz, $CDCl_3$)

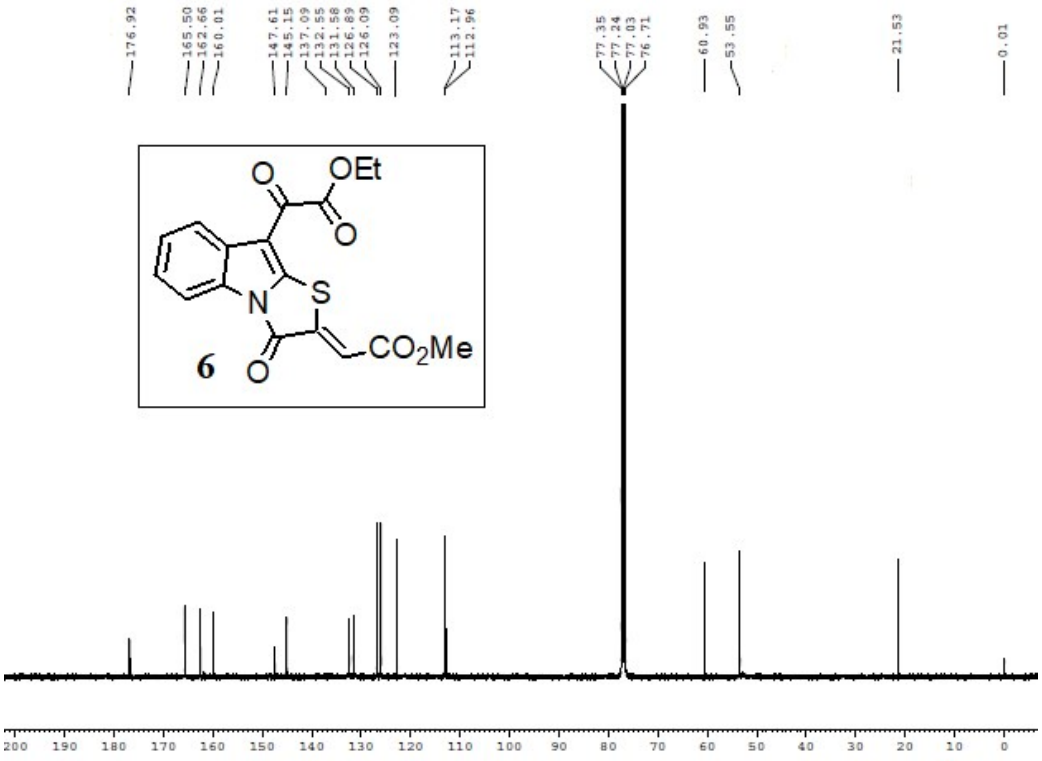



**Figure S4**. $^{13}$C NMR of Compound **6** (100 MHz, $CDCl_3$)

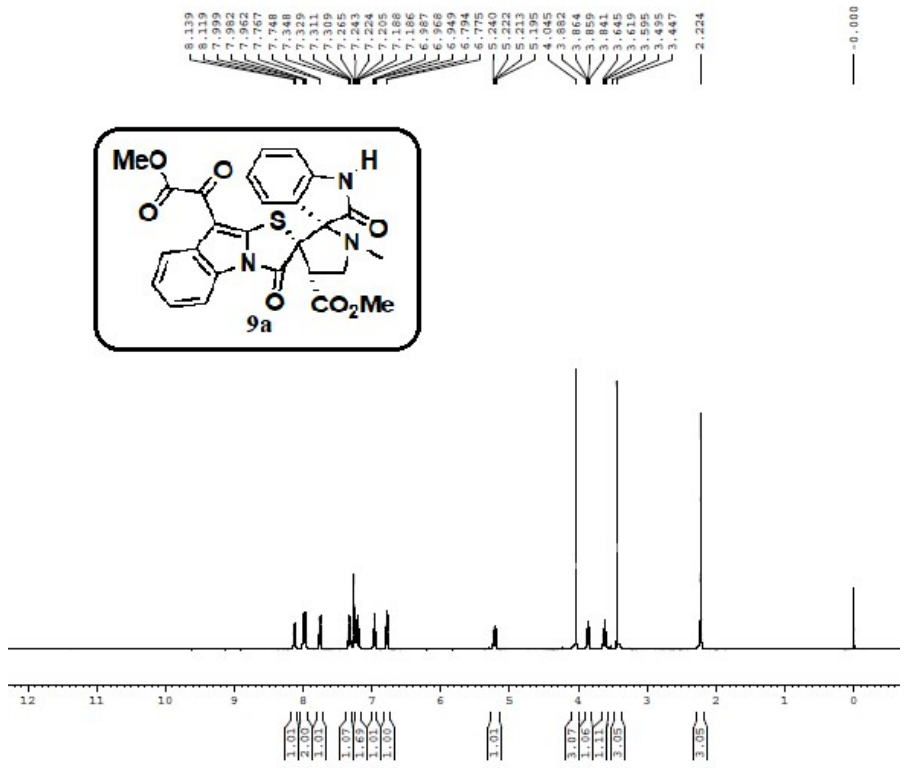



**Figure S5**. $^{1}H$ NMR of Compound **9a** (400 MHz, $CDCl_3$)

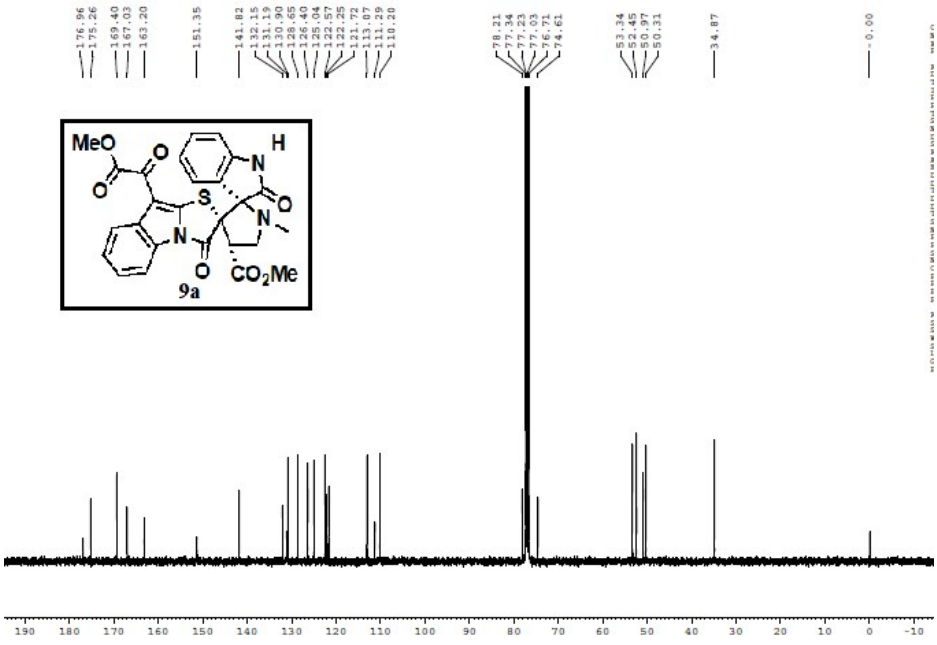
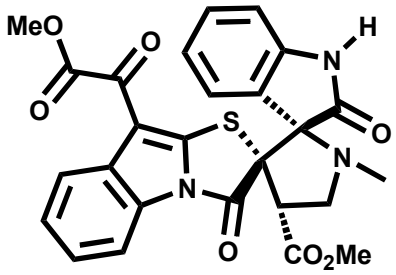



**Figure S6**. $^{13}C$ NMR of Compound **9a** (100 MHz, $CDCl_3$)




**9a**

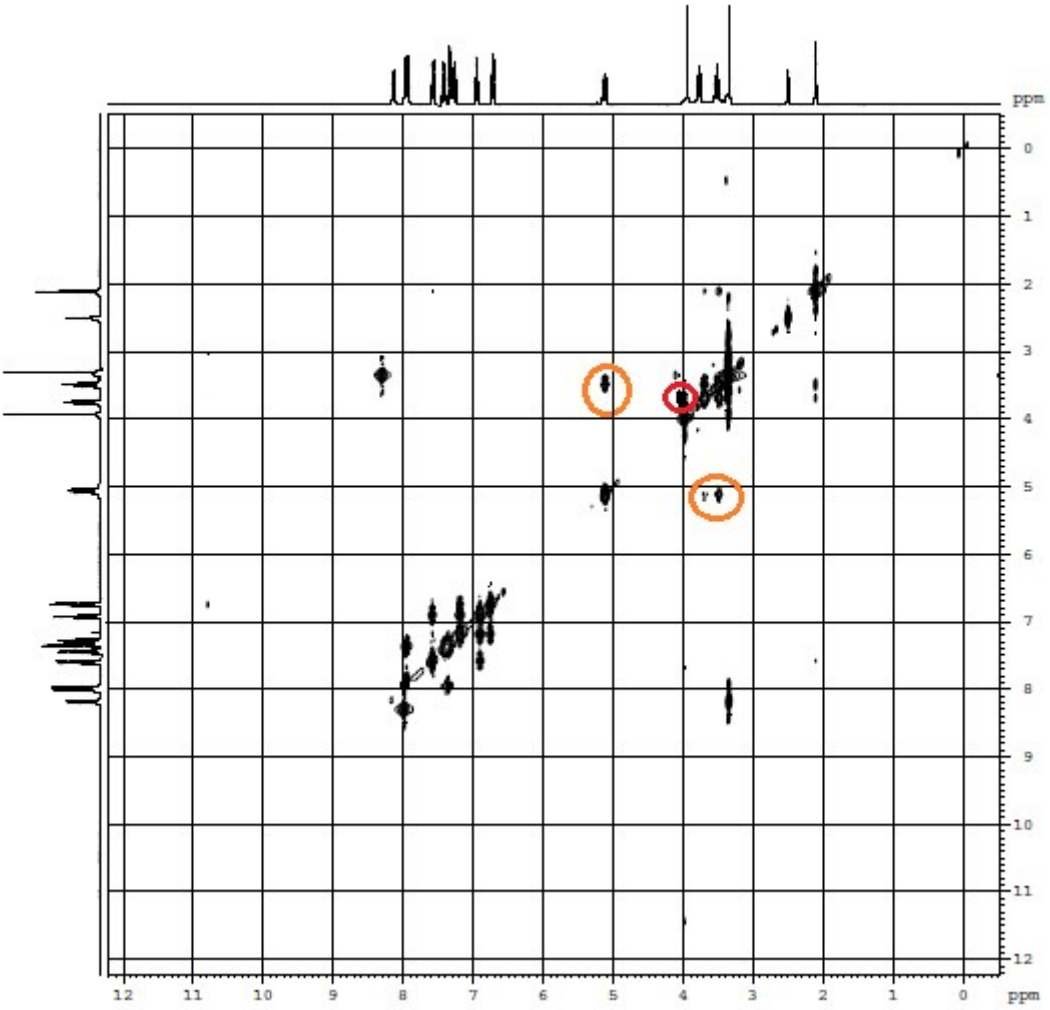
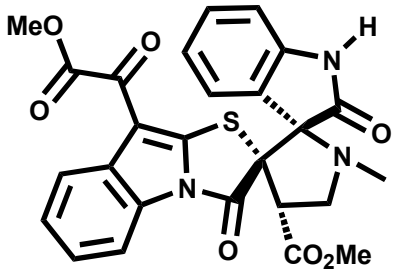



**Figure S7**. $^{1}$H-$^{1}$H NOESY spectrum of Compound **9a** (400 MHz, DMSO)




**9a**

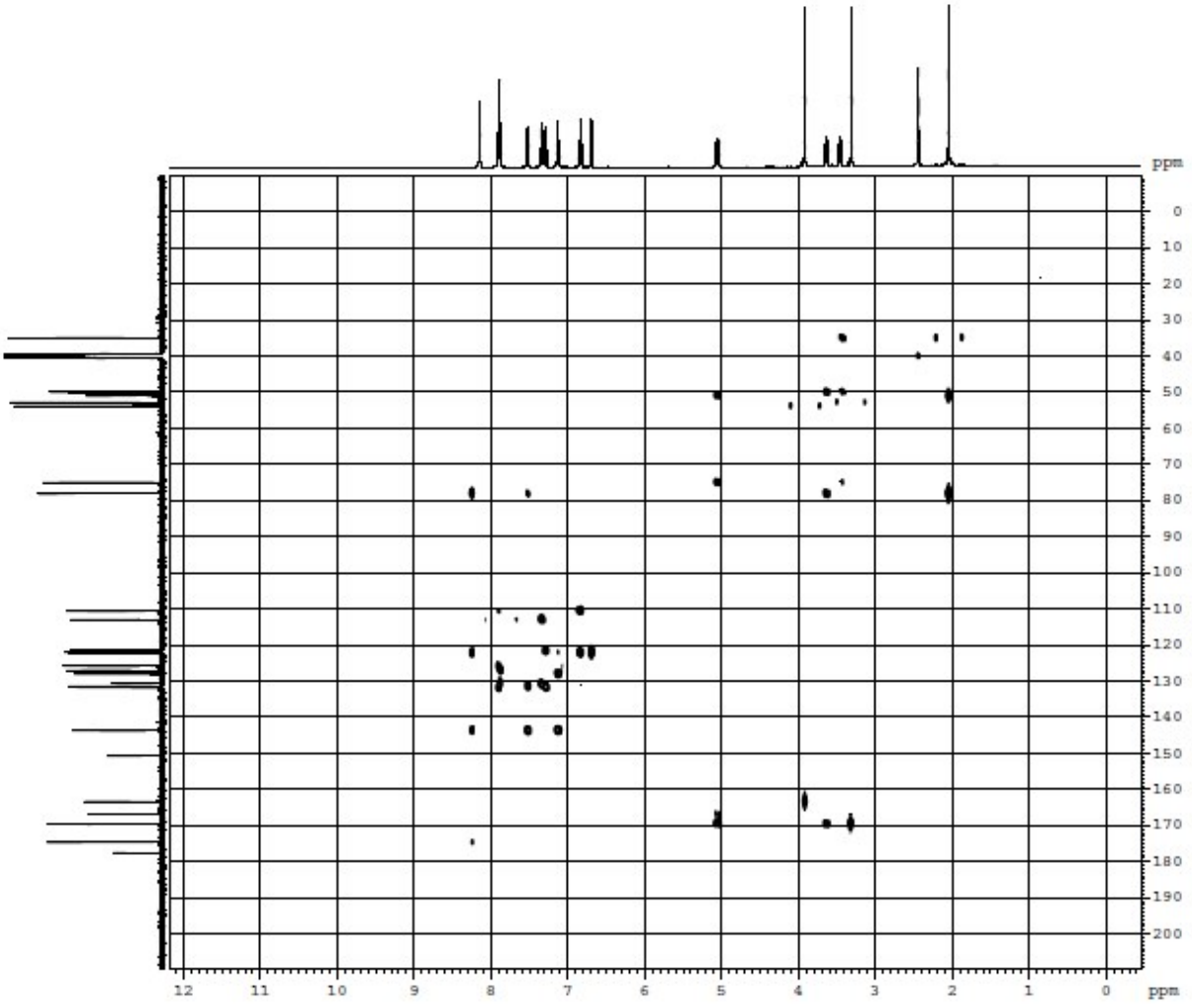
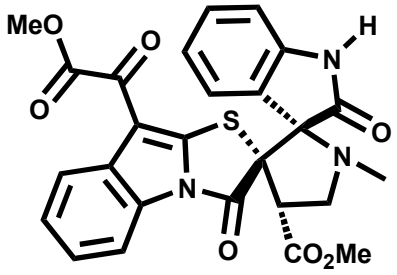



**Figure S8**. HMBC spectrum of Compound **9a** (DMSO)[1]




**9a**

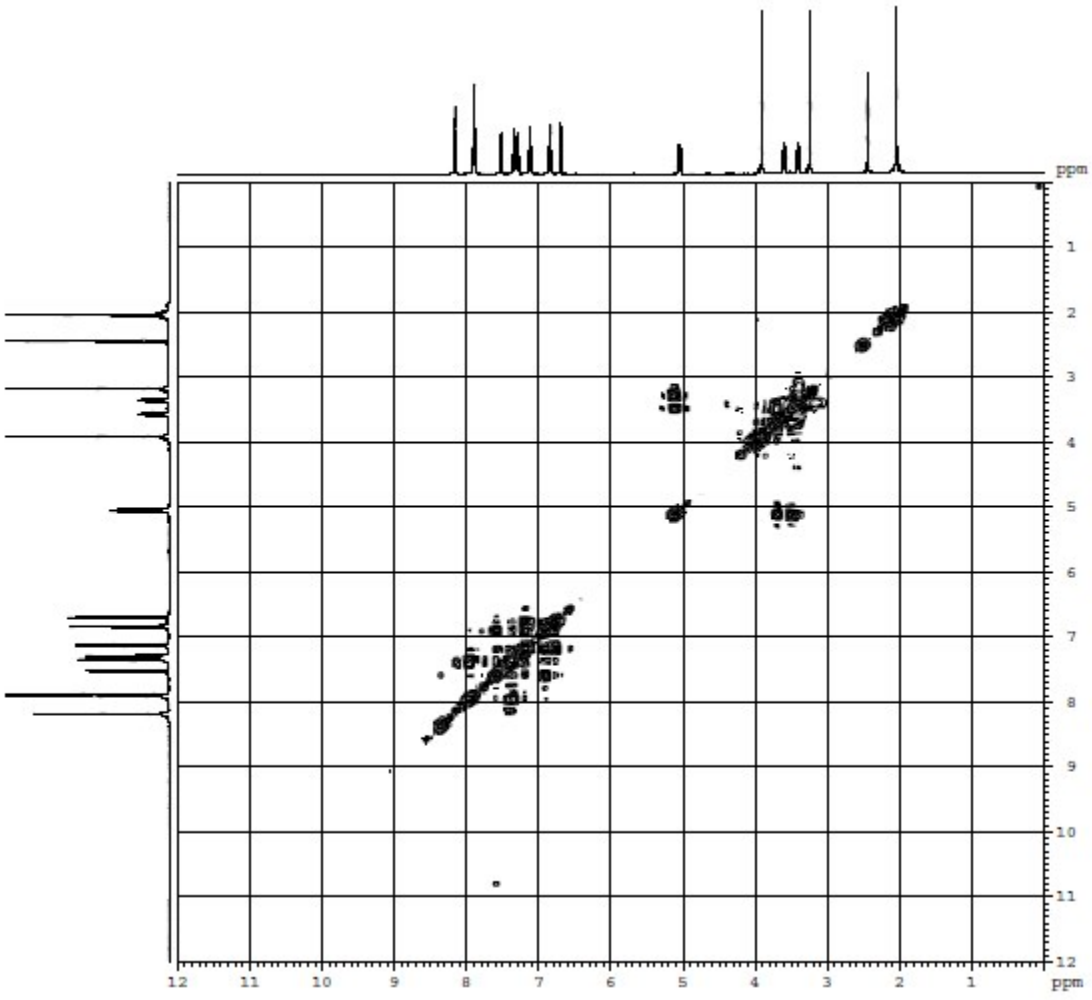



**Figure S9**. $^{1}$H-$^{1}$H COSY spectrum of Compound **9a** (400 MHz, DMSO)[1]

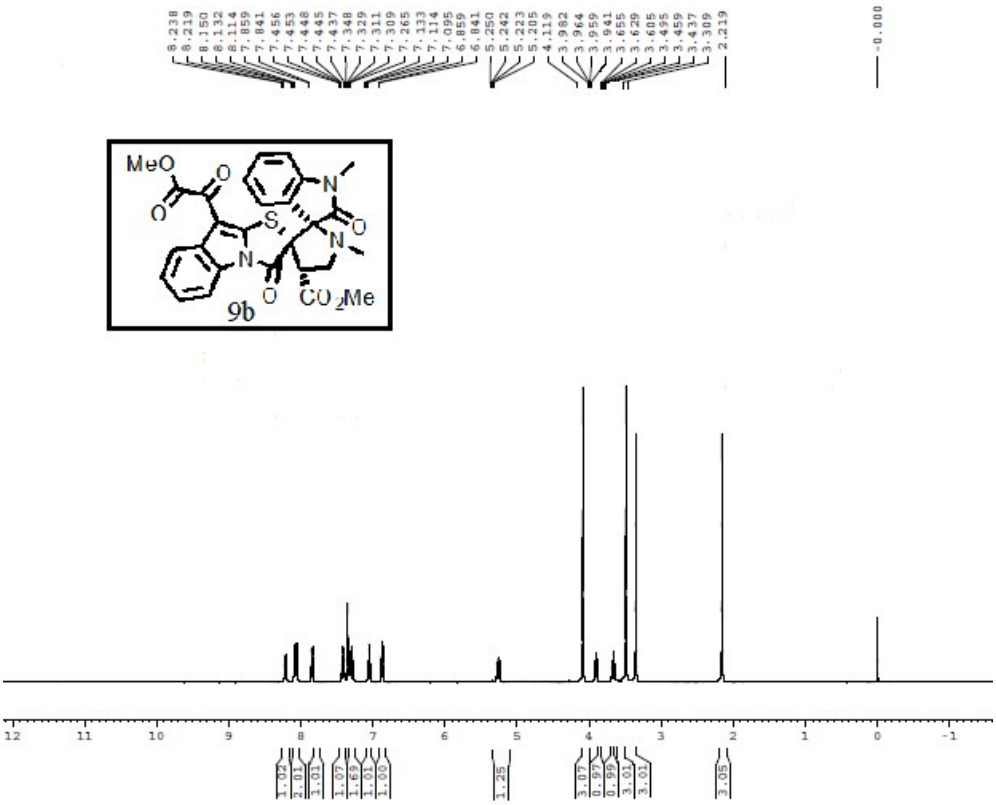



**Figure S10**. $^{1}H$ NMR of Compound **9b** (400 MHz, $CDCl_3$)

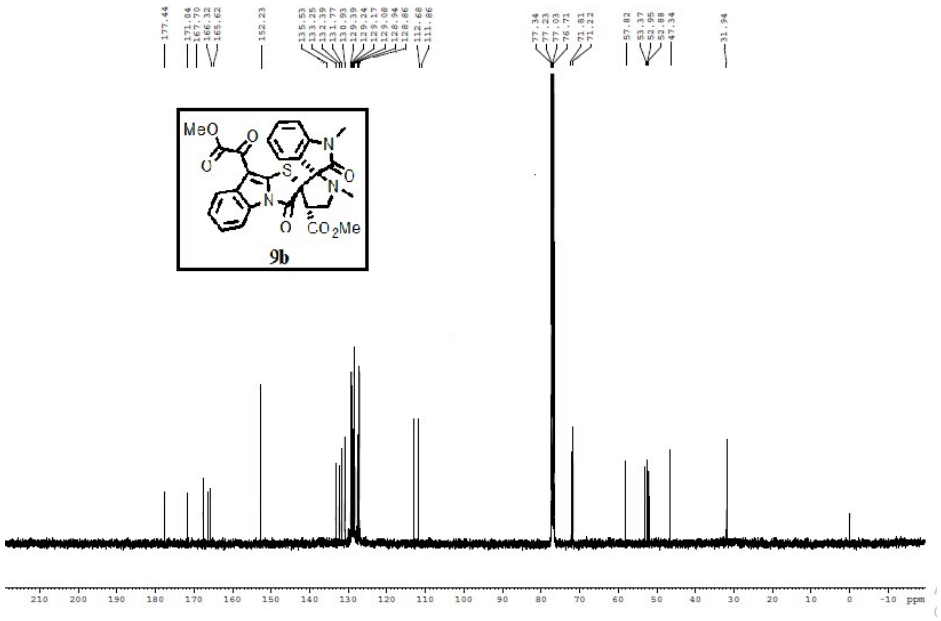



**Figure S11**. $^{13}C$ NMR of Compound **9b** (100 MHz, $CDCl_3$)

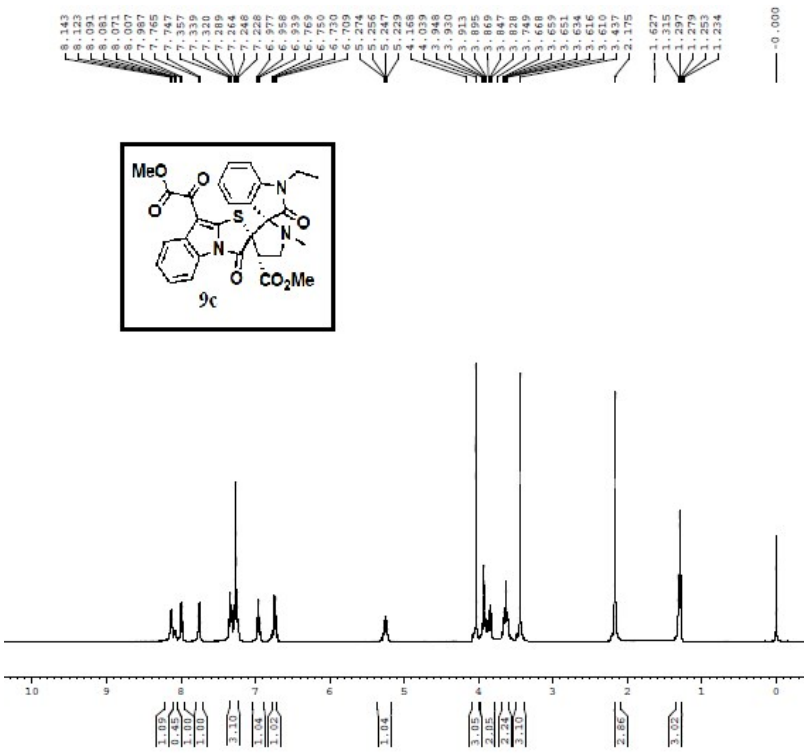



**Figure S12**. $^{1}H$ NMR of Compound **9c** (400 MHz, $CDCl_3$)

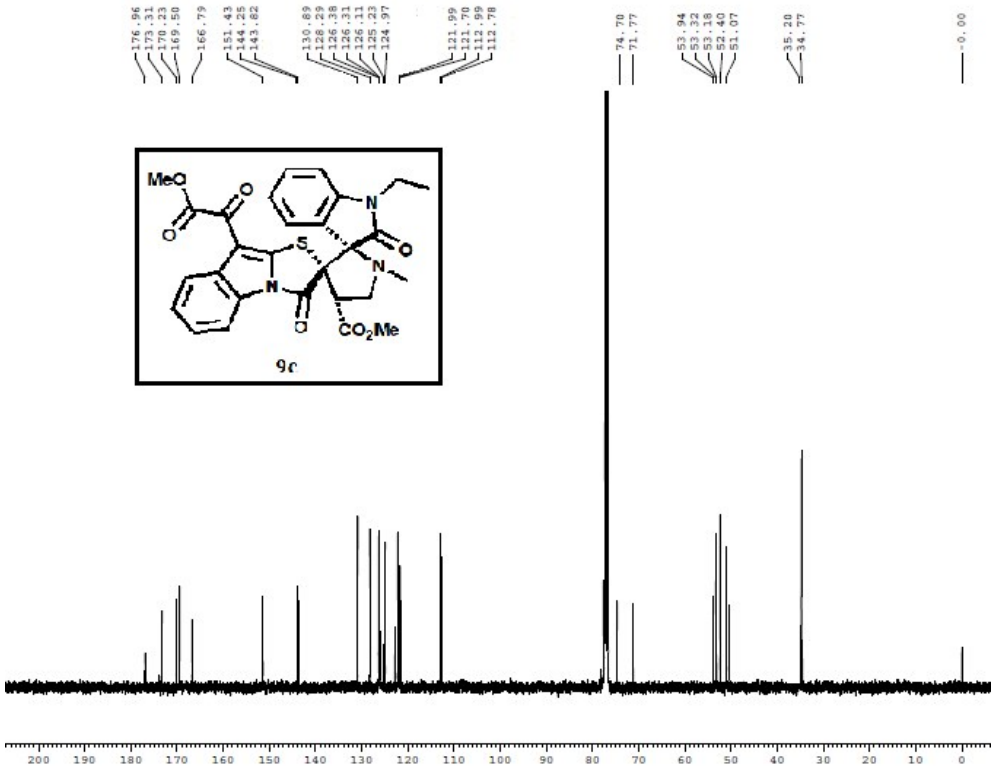


**Figure S13**. $^{13}C$ NMR of Compound **9c** (100 MHz, $CDCl_3$)

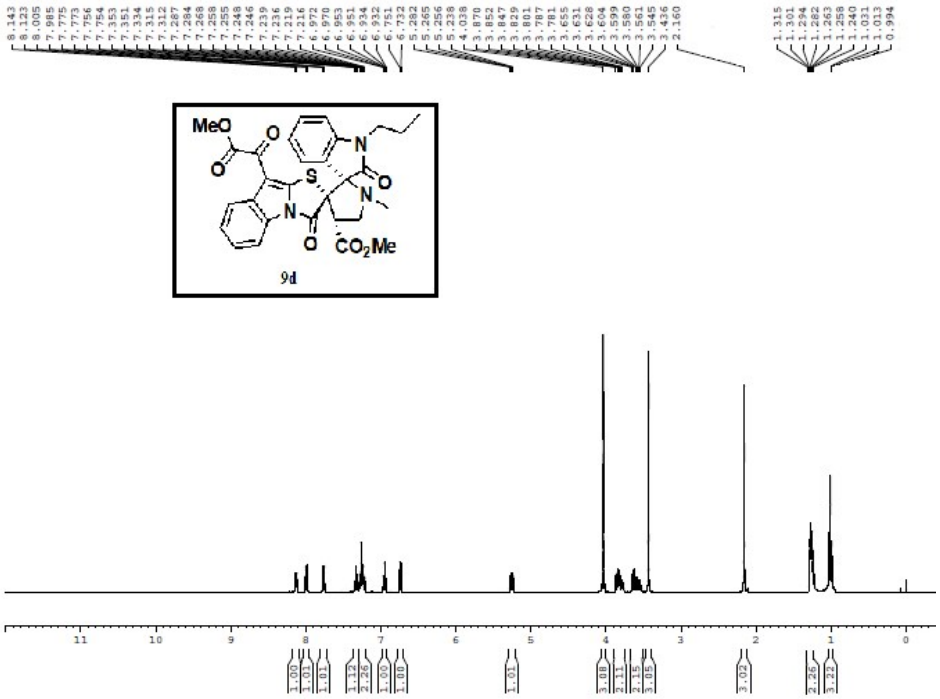



**Figure S14**. $^{1}H$ NMR of Compound **9d** (400 MHz, $CDCl_3$)

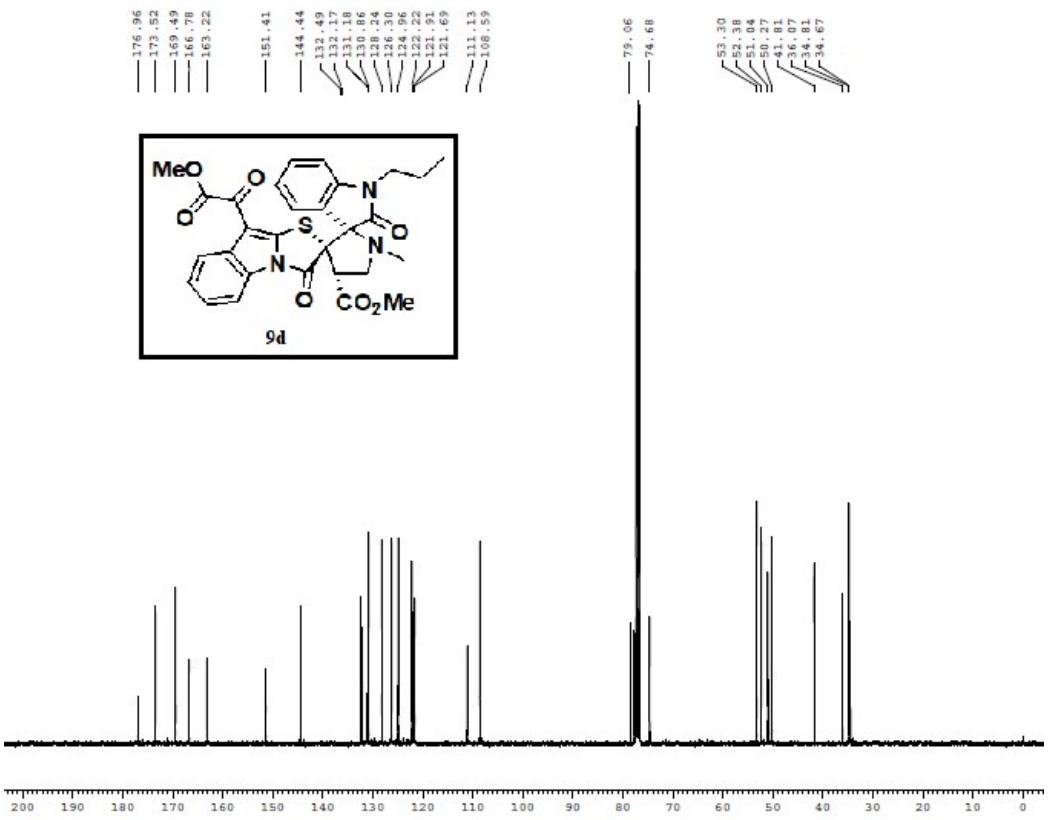



**Figure S15**. $^{13}C$ NMR of Compound **9d** (100 MHz, $CDCl_3$)

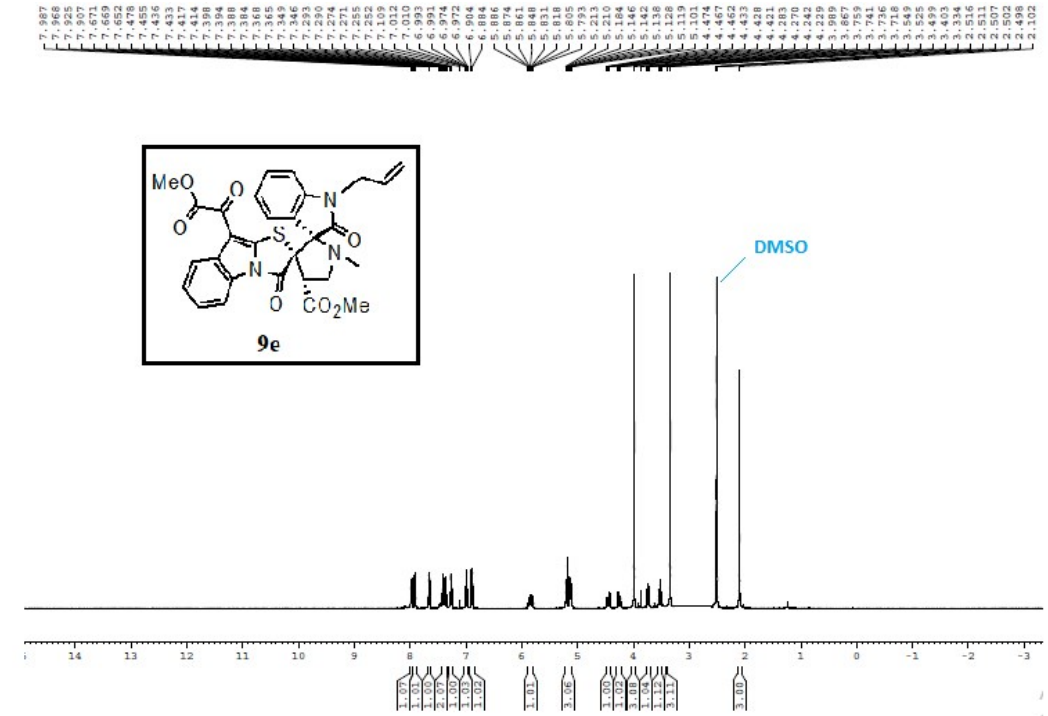


**Figure S16**. $^{1}$H NMR of Compound **9e** (400 MHz, DMSO)

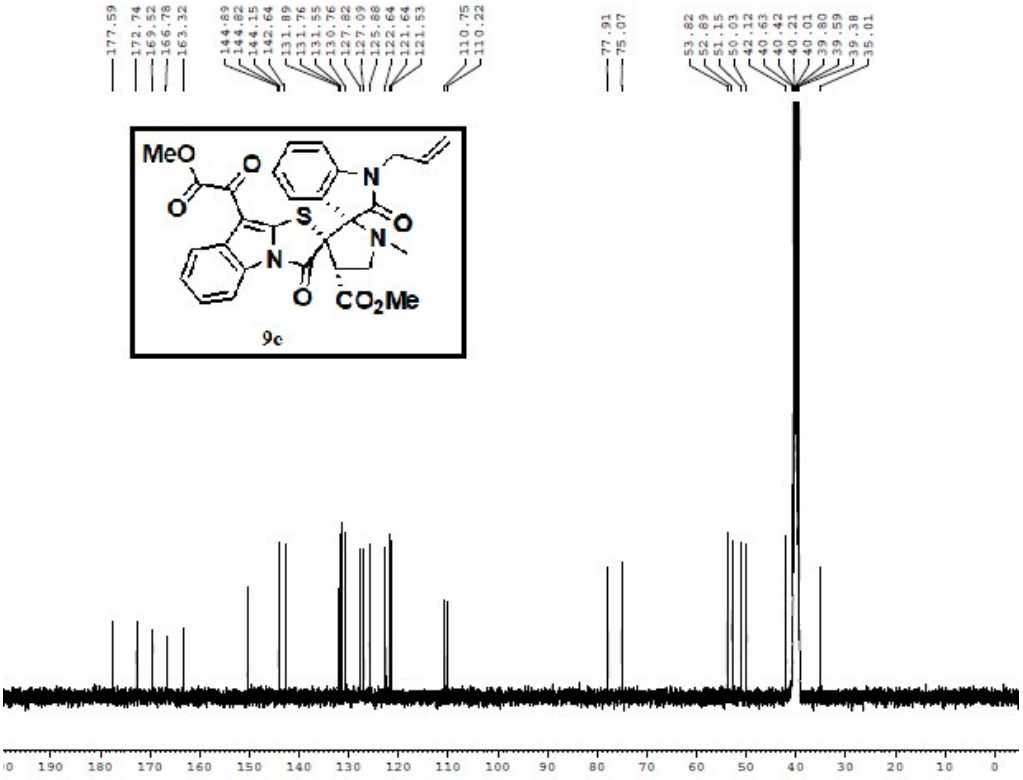



**Figure S17**. $^{13}$C NMR of Compound **9e** (100 MHz, DMSO)

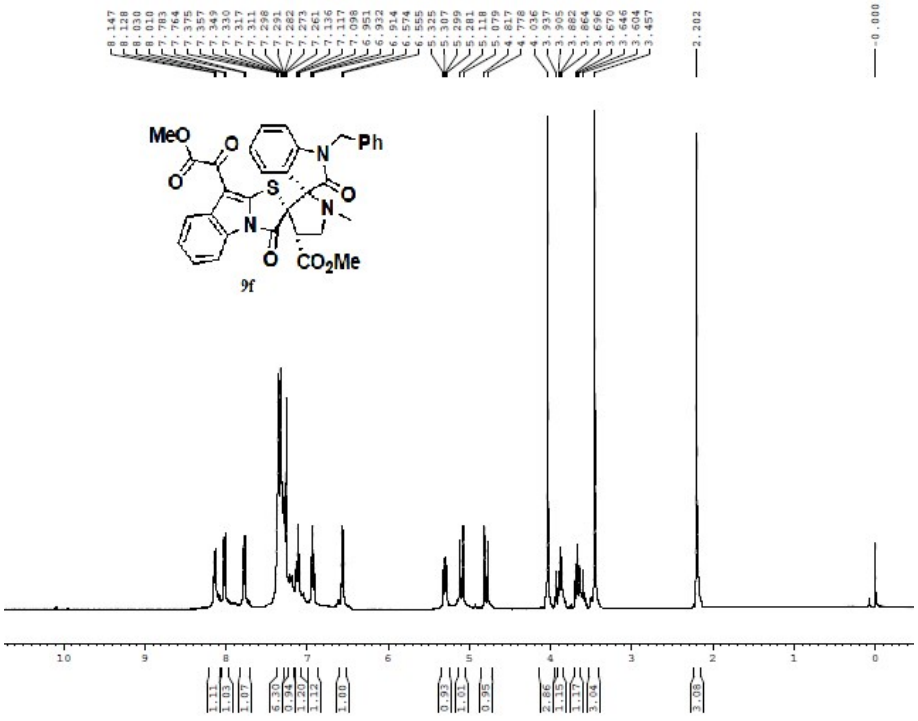


**Figure S18**. $^{1}H$ NMR of Compound **9f** (400 MHz, $CDCl_3$)

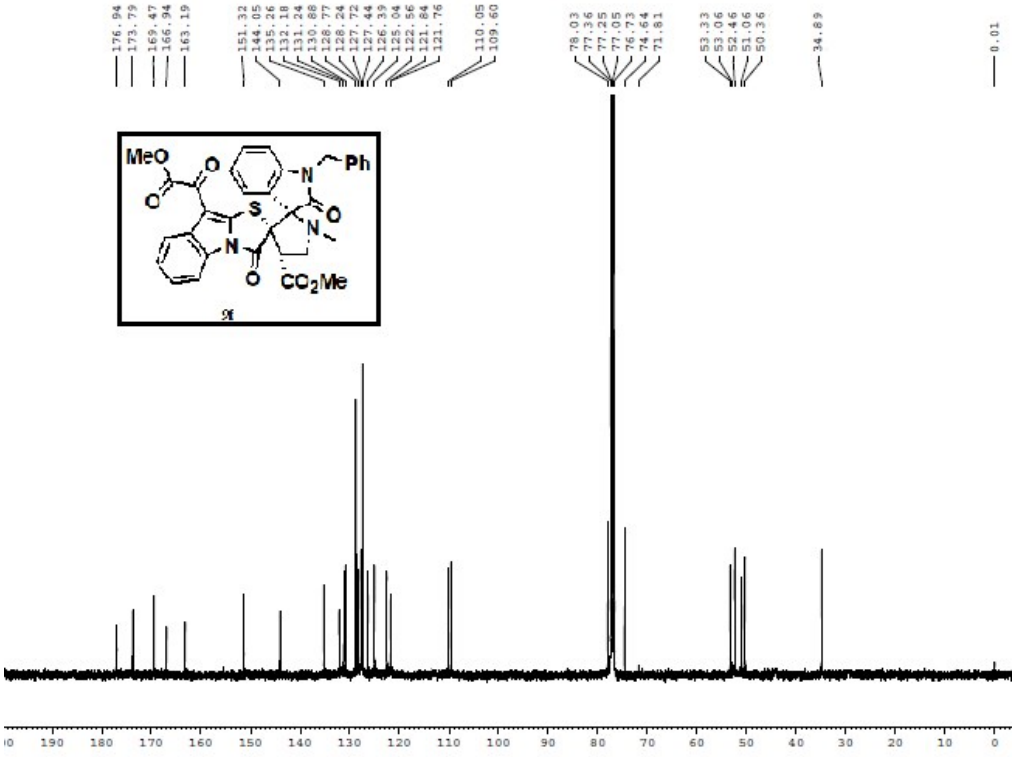



**Figure S19**. $^{13}C$ NMR of Compound **9f** (100 MHz, $CDCl_3$)

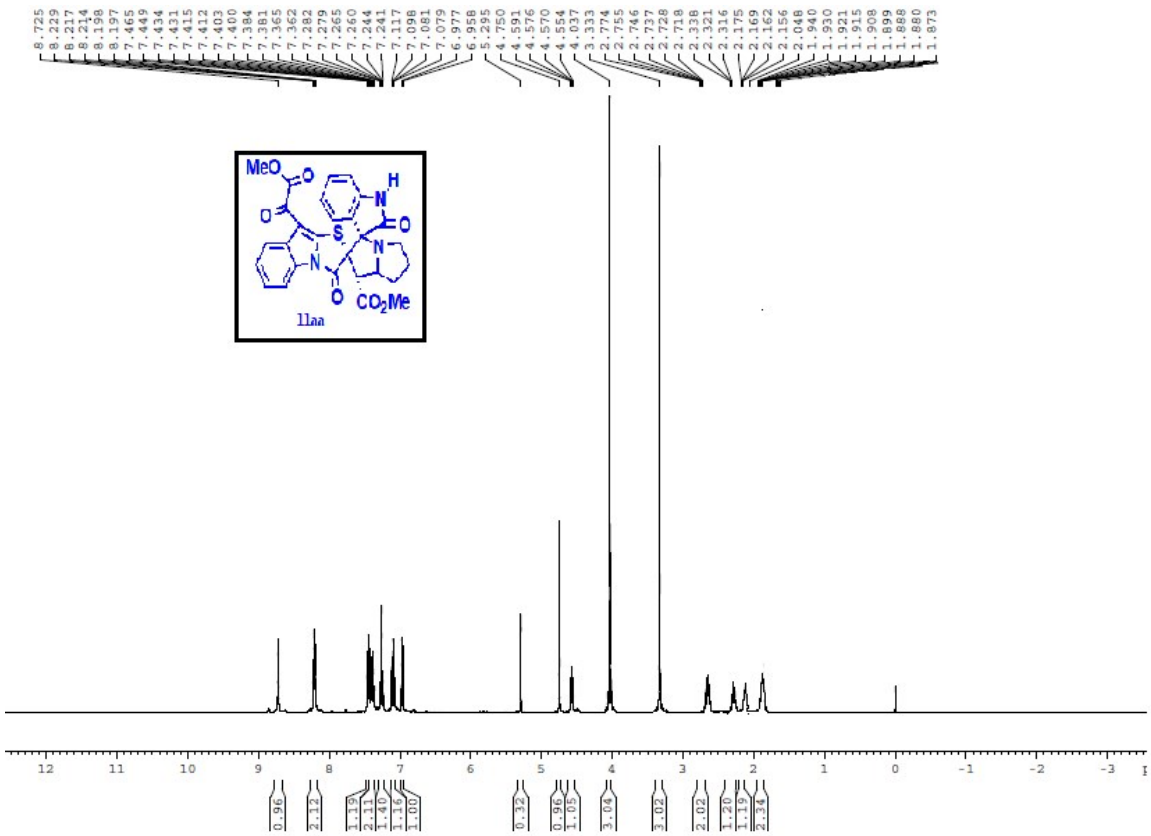



**Figure S20**. $^{1}$H NMR of Compound **11aa** (400 MHz, $CDCl_3$)

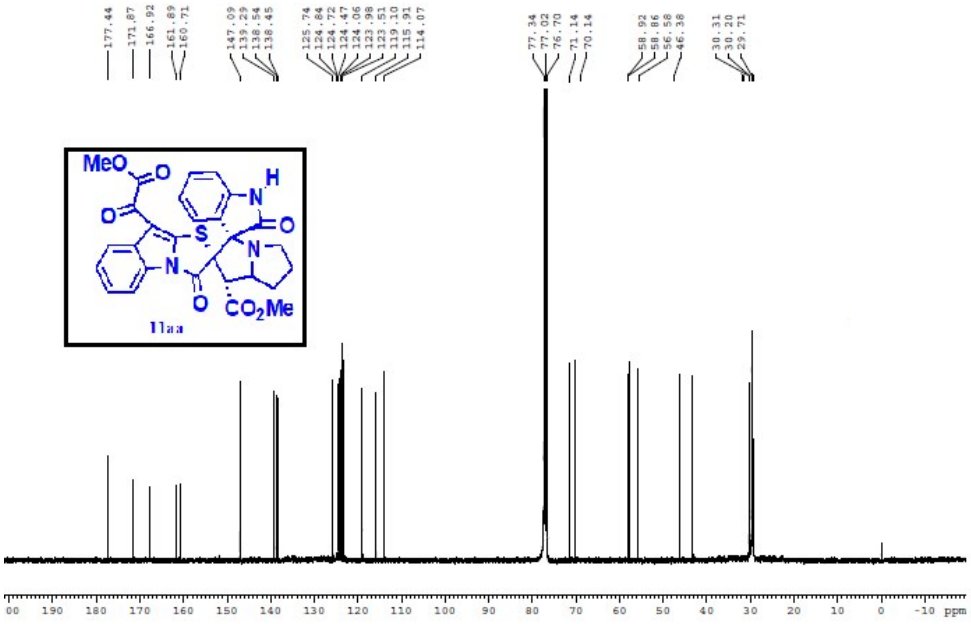
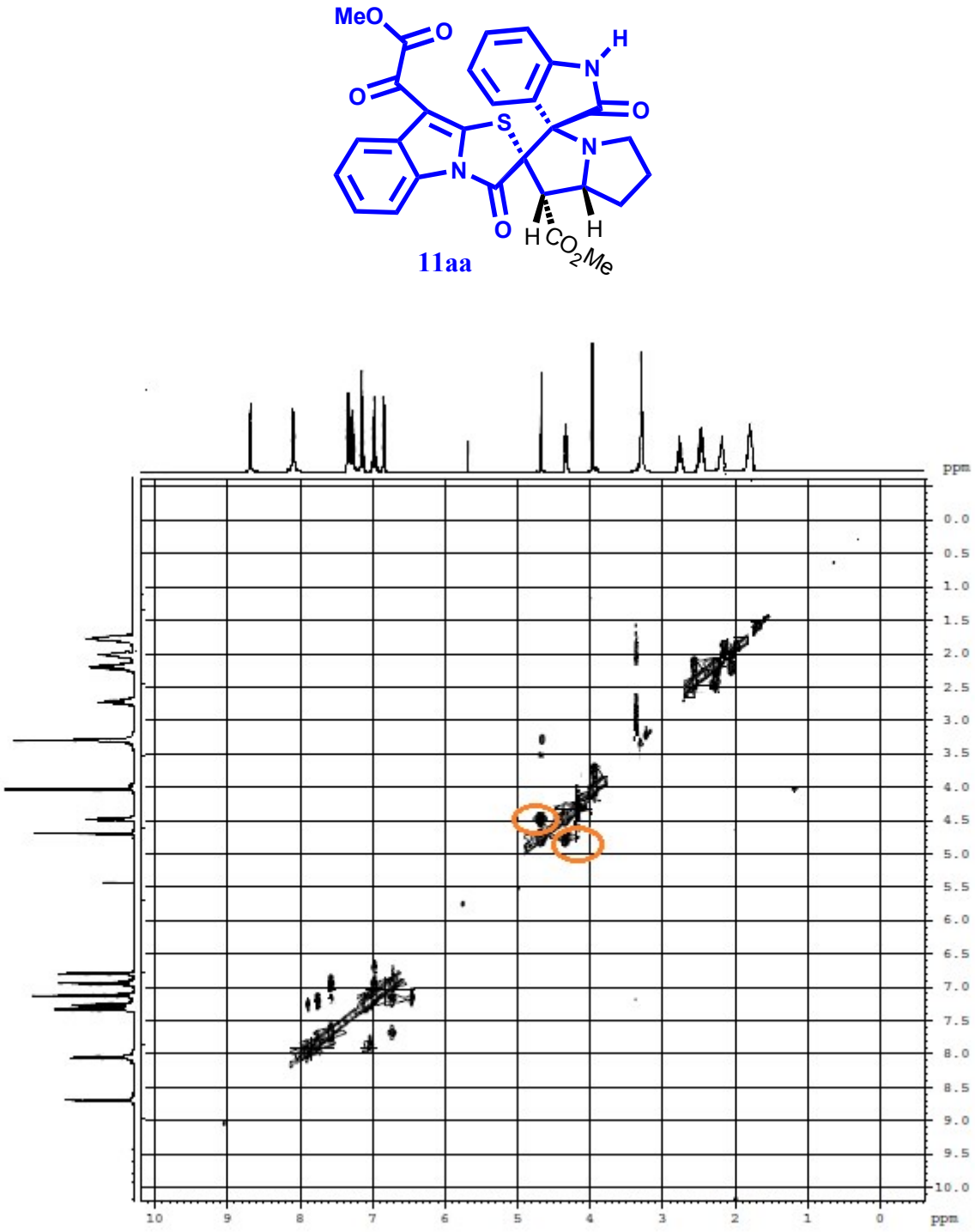



**Figure S21**. $^{13}$C NMR of Compound **11aa** (100 MHz, $CDCl_3$)




**11aa**

**Figure S22**. $^{1}$H-$^{1}$H NOESY of Compound **11aa** (100 MHz, $CDCl_3$)

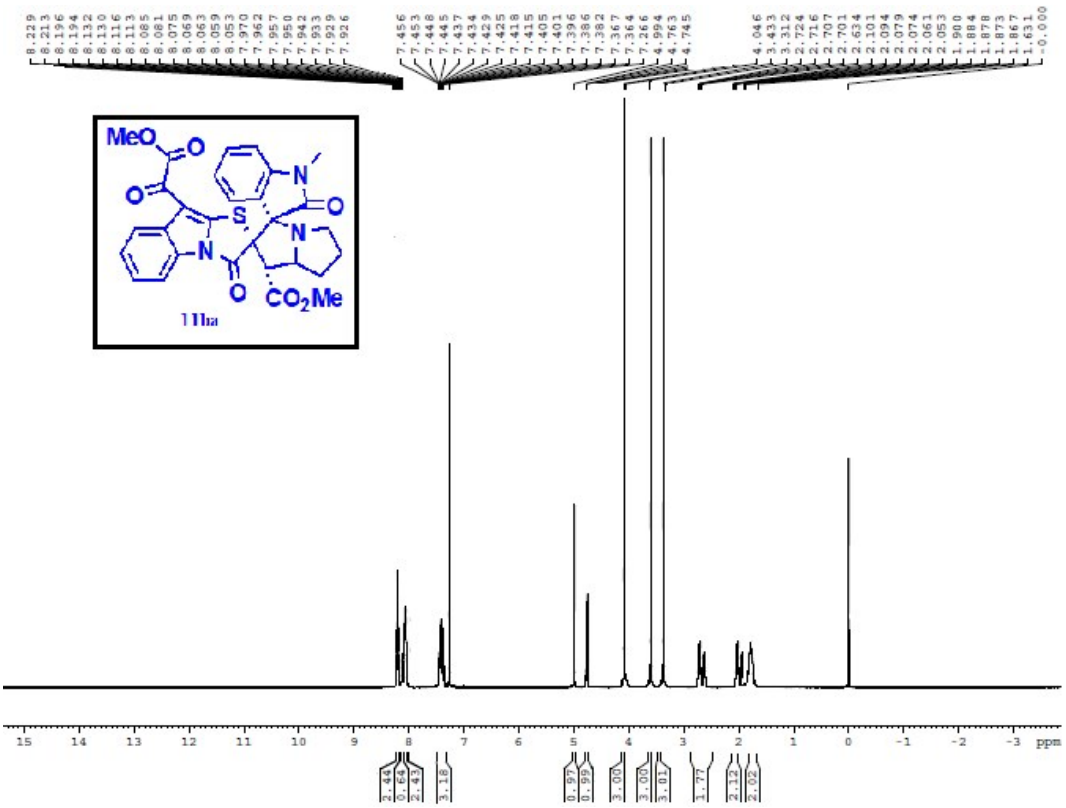



**Figure S23**. $^{1}$H NMR of Compound **11ba** (400 MHz, $CDCl_3$)

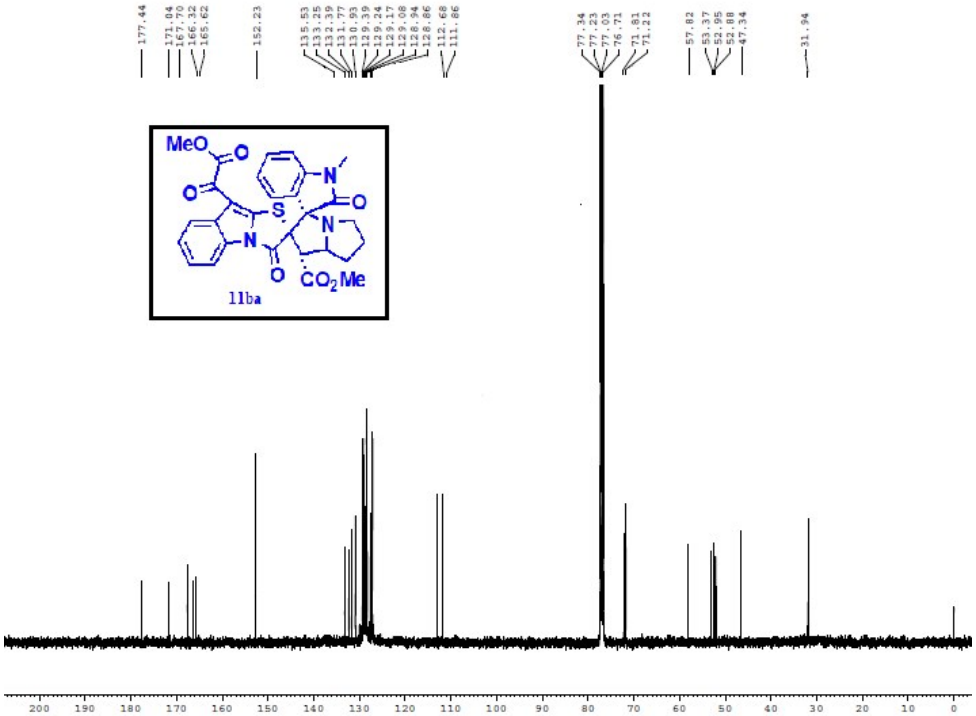



**Figure S24**. $^{13}$C NMR of Compound **11ba** (100 MHz, $CDCl_3$)

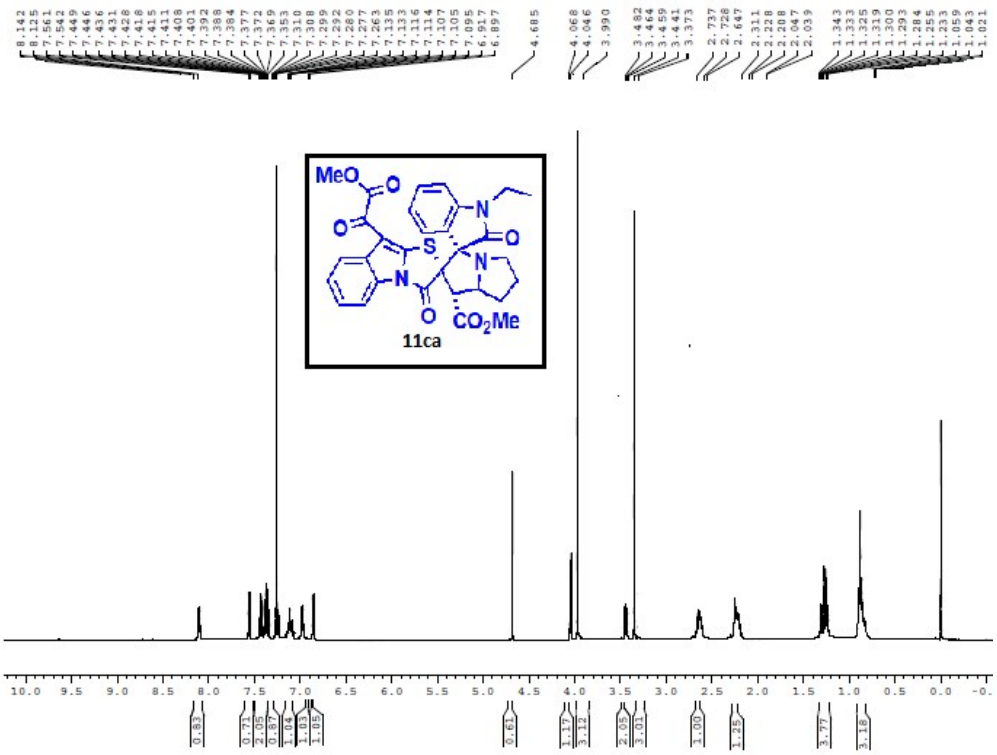



**Figure S25**. $^{1}$H NMR of Compound **11ca** (400 MHz, $CDCl_3$)

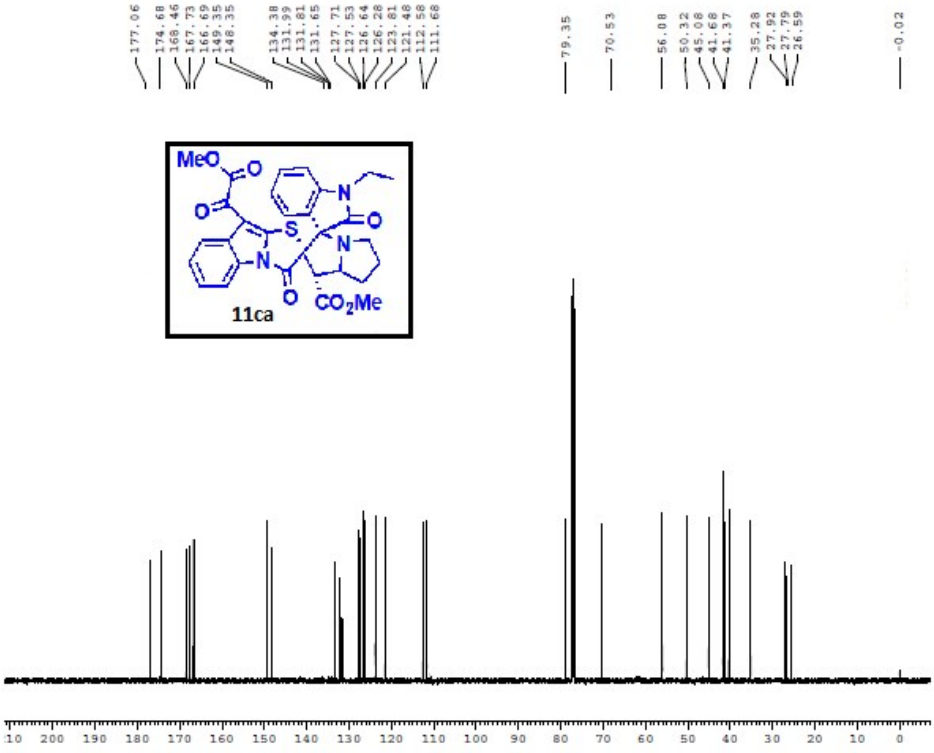



**Figure S26**. $^{13}$C NMR of Compound **11ca** (100 MHz, $CDCl_3$)

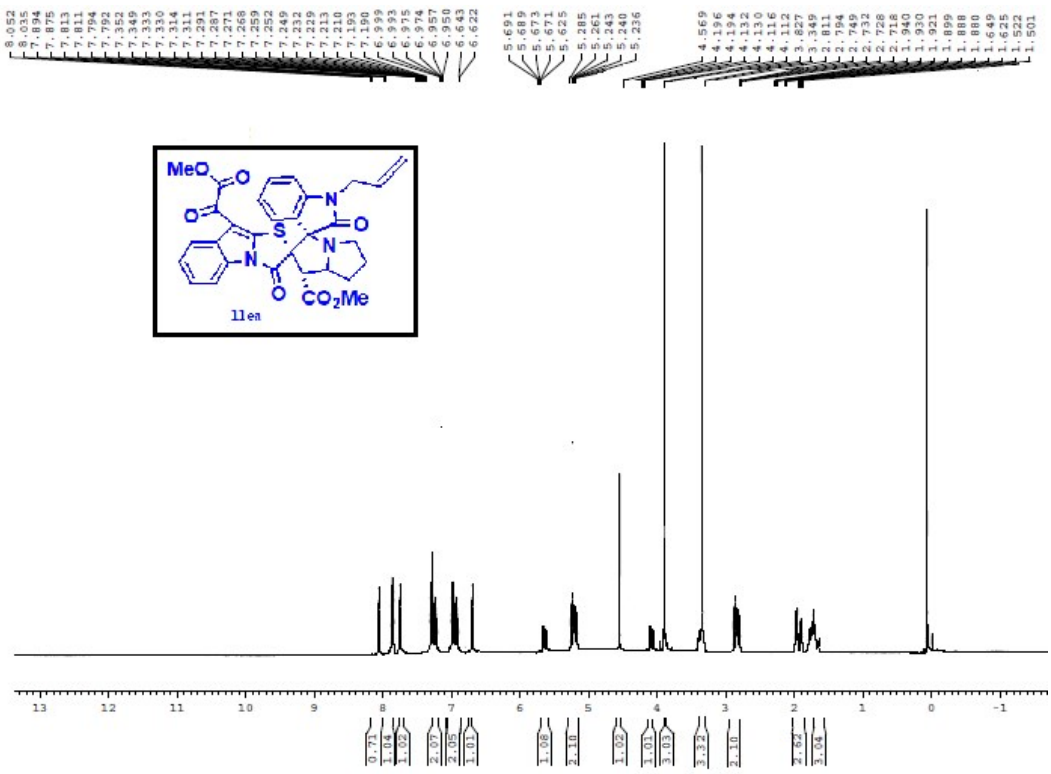



**Figure S27**. $^{1}$H NMR of Compound **11ea** (400 MHz, $CDCl_3$)

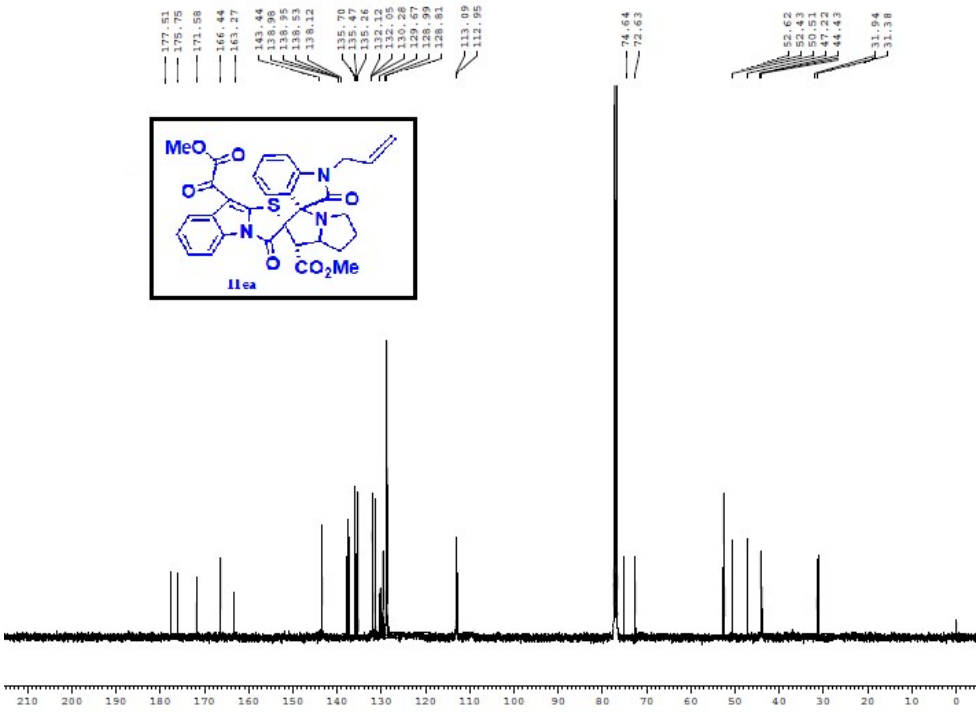



**Figure S28**. $^{13}$C NMR of Compound **11ea** (100 MHz, $CDCl_3$)

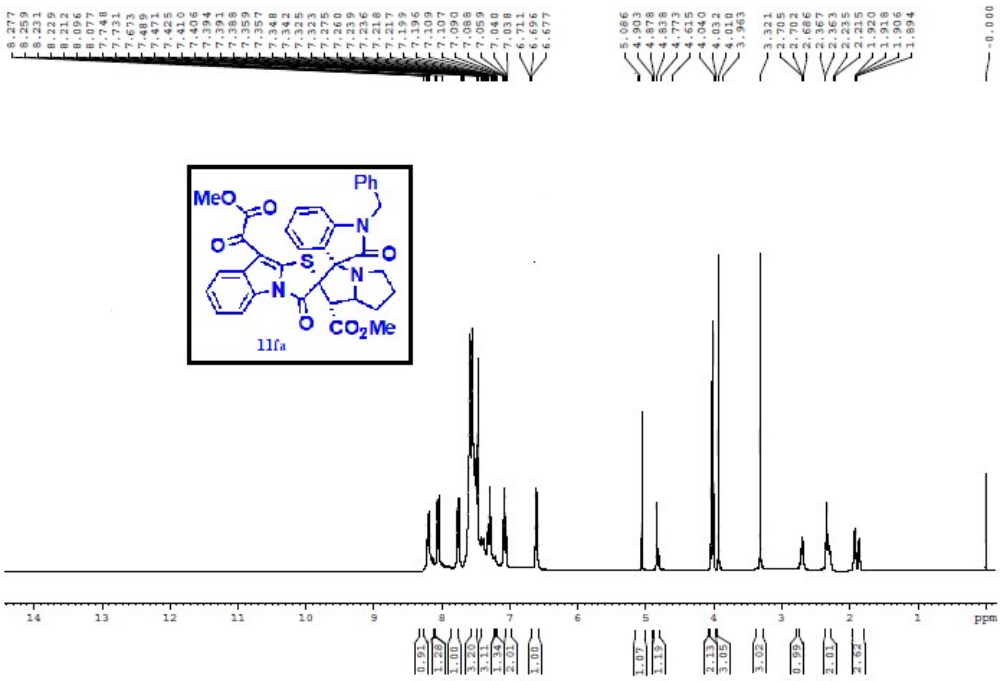



**Figure S29**. $^{1}$H NMR of Compound **11fa** (400 MHz, $CDCl_3$)

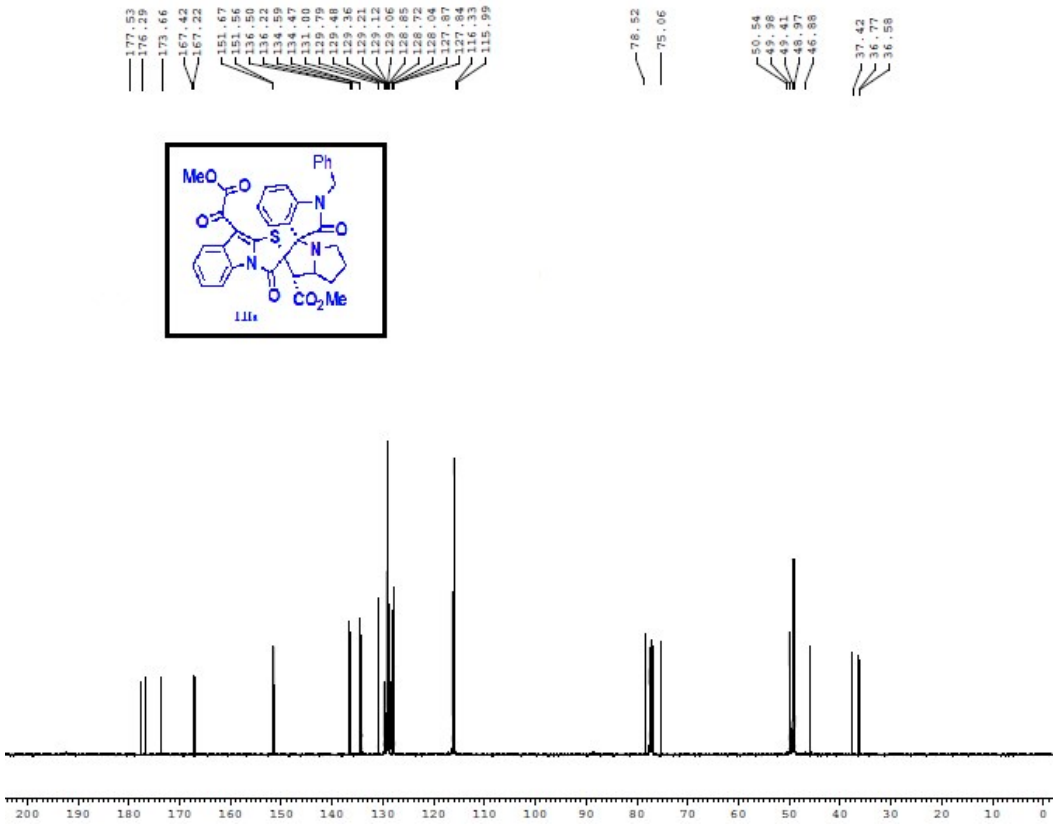



**Figure S30**. $^{13}$C NMR of Compound **11fa** (100 MHz, $CDCl_3$)

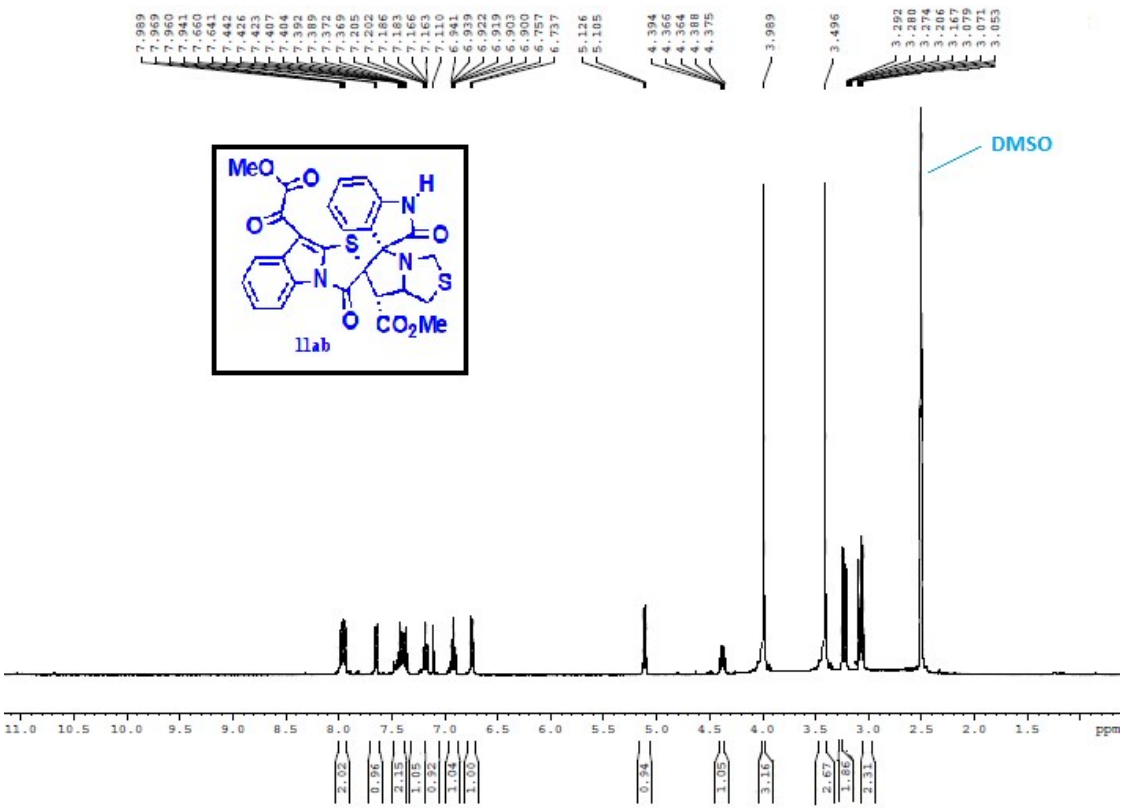



**Figure S31**. [1]H NMR of Compound **11ab** (400 MHz, DMSO)

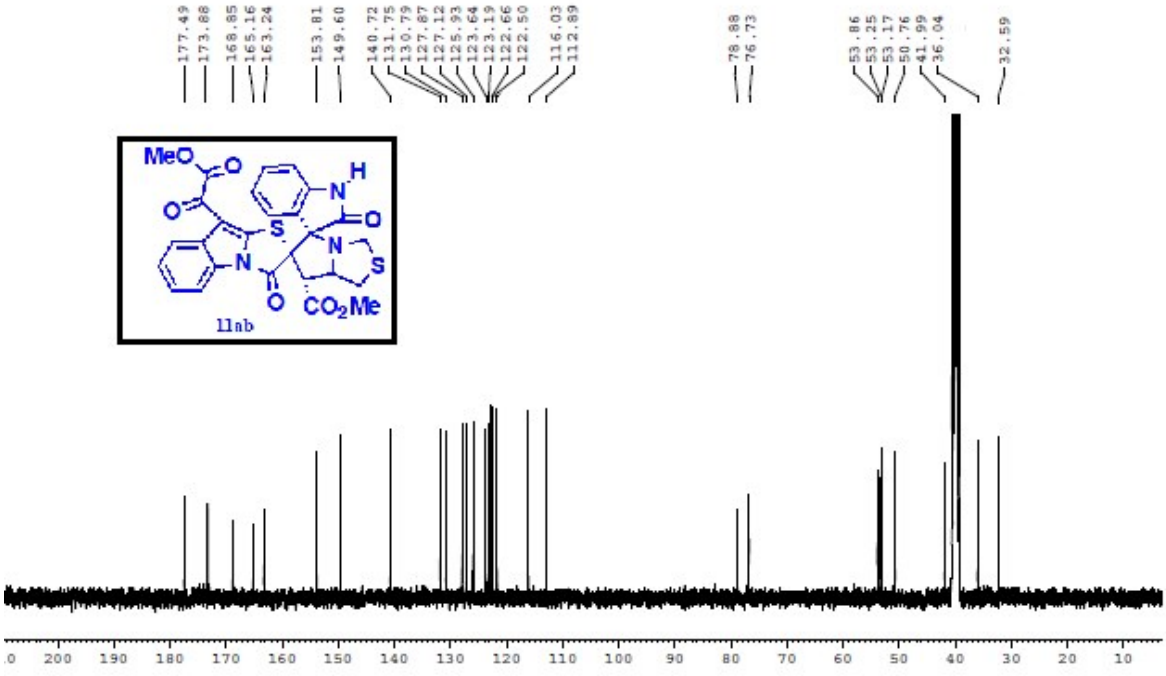



**Figure S32**. [13]C NMR of Compound **11ab** (100 MHz, DMSO)

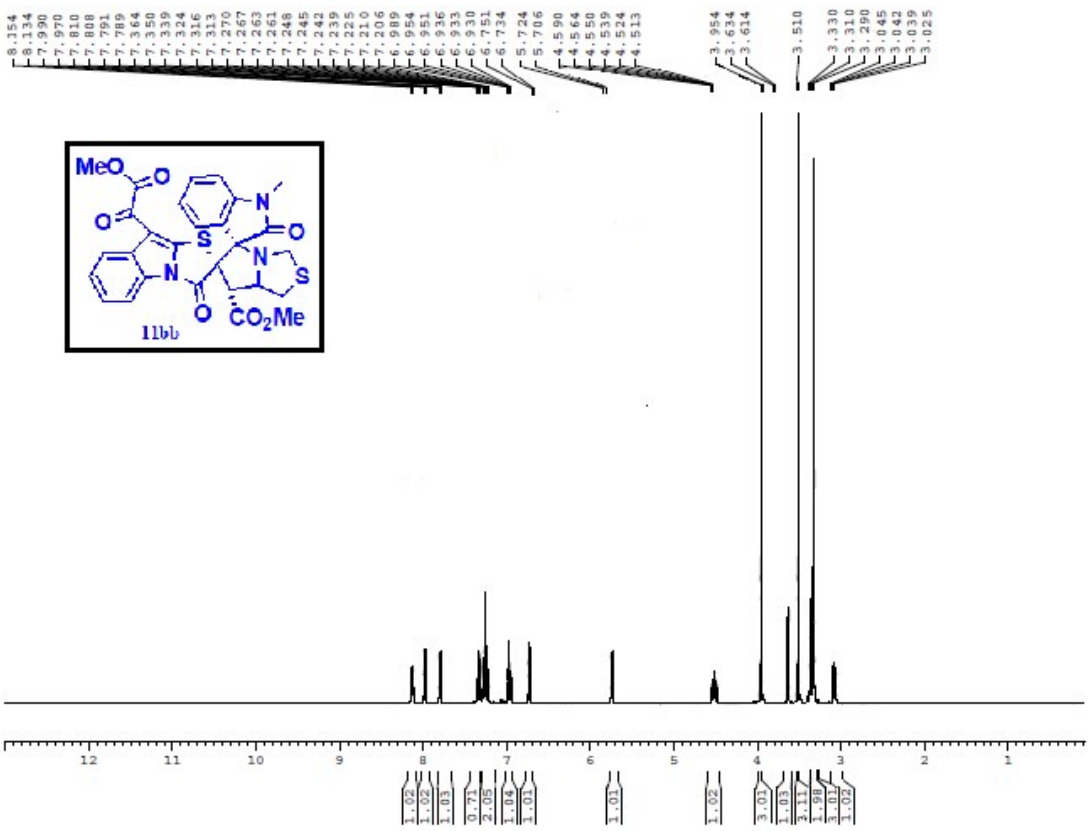



**Figure S33**. $^{1}H$ NMR of Compound **11bb** (400 MHz, $CDCl_3$)

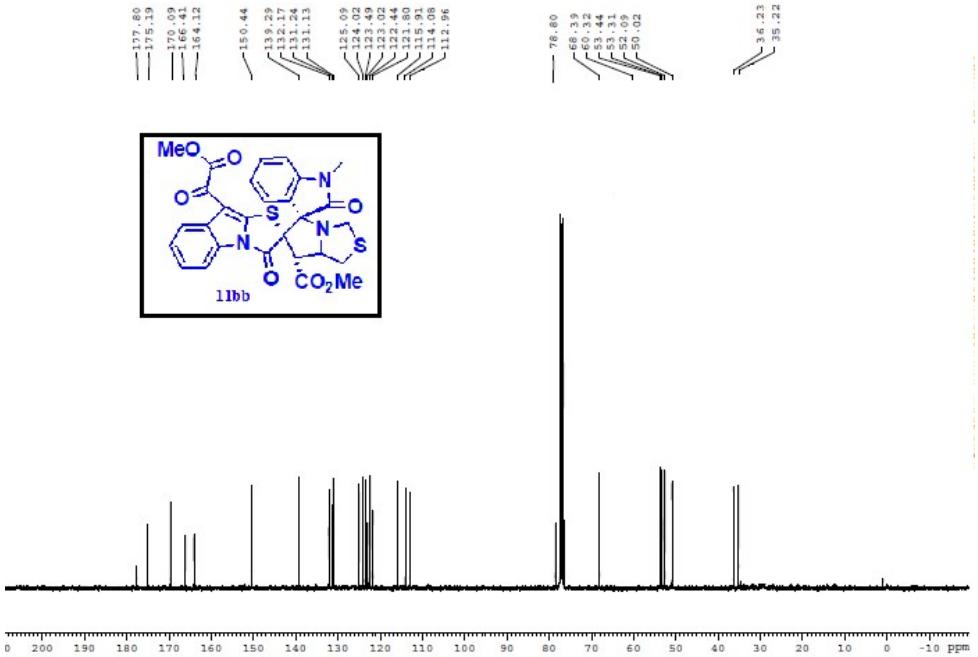



**Figure S34**. $^{13}C$ NMR of Compound **11ab** (100 MHz, $CDCl_3$)

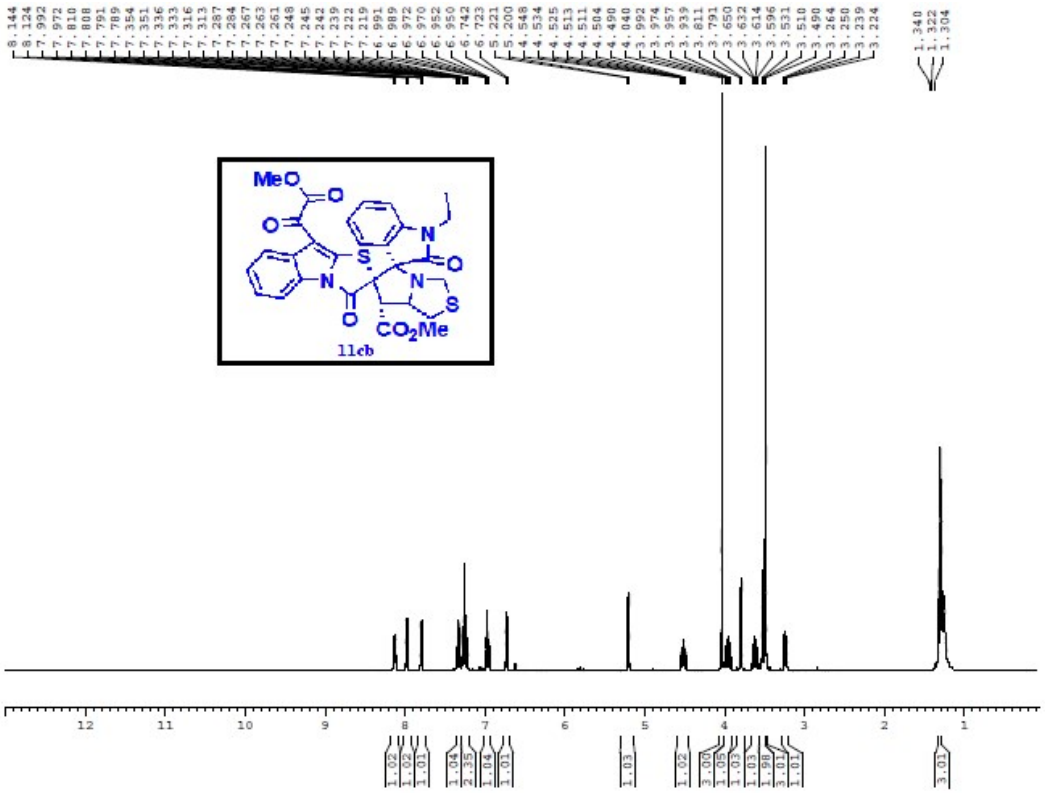



**Figure S35**. $^{1}H$ NMR of Compound **11cb** (400 MHz, $CDCl_3$)

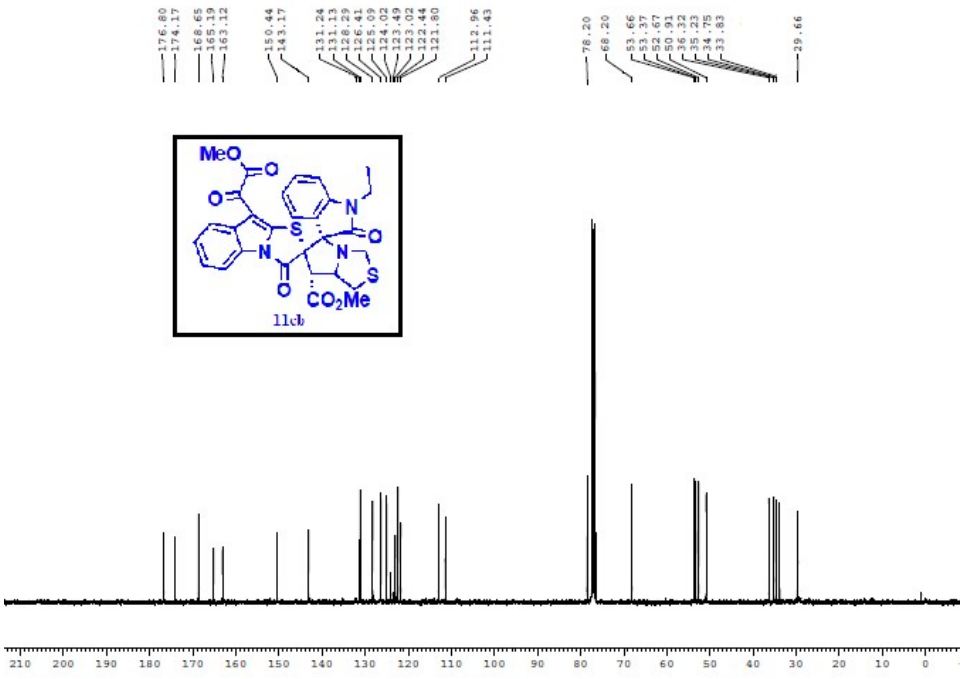



**Figure S36**. $^{13}C$ NMR of Compound **11cb** (100 MHz, $CDCl_3$)

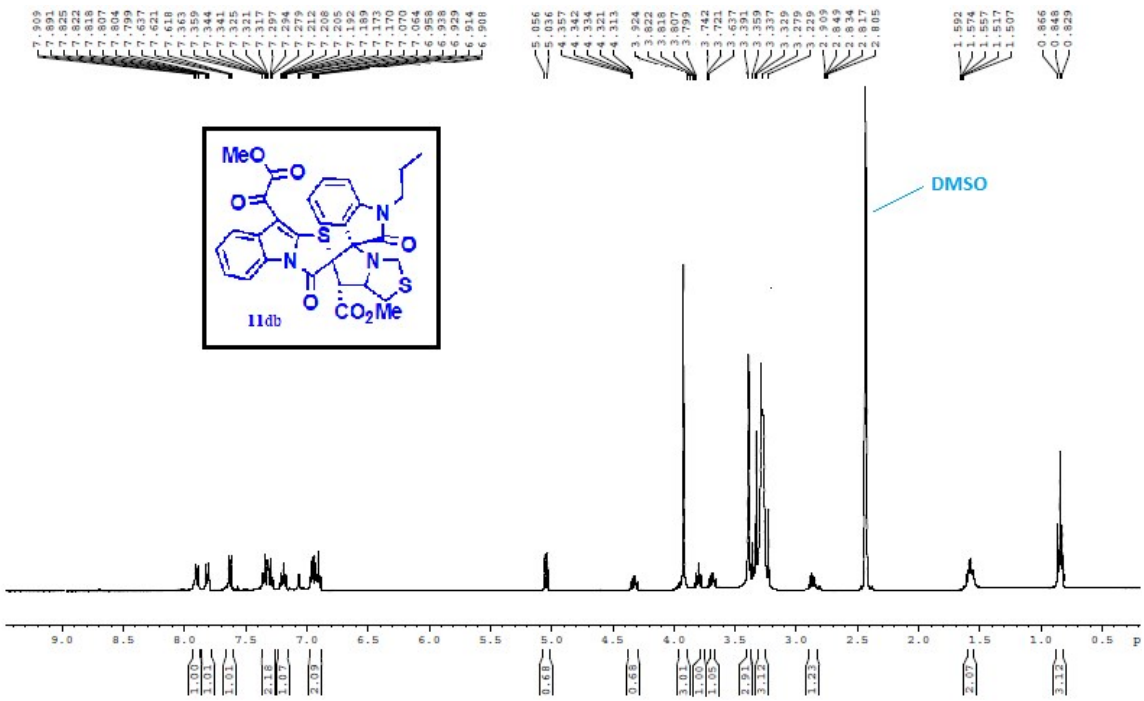



**Figure S37**. $^{1}$H NMR of Compound **11db** (400 MHz, DMSO)

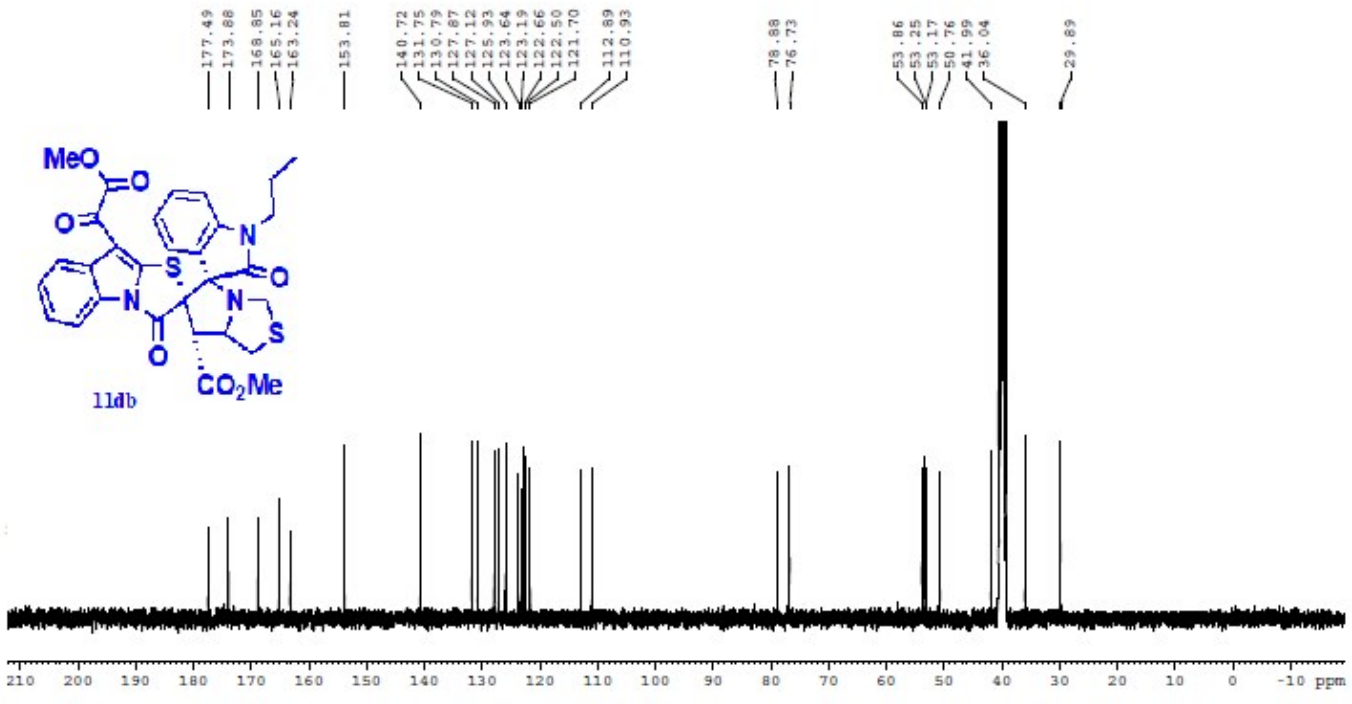



**Figure S38**. $^{13}$C NMR of Compound **11db** (100 MHz, DMSO)

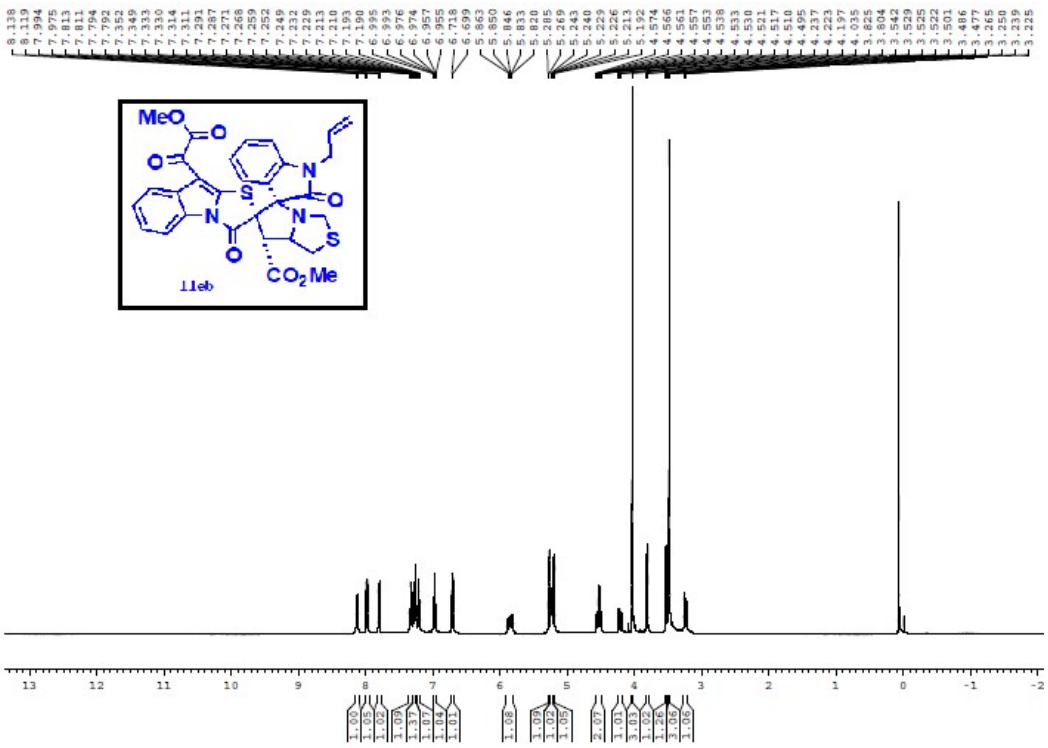



**Figure S39**. $^{1}H$ NMR of Compound **11eb** (400 MHz, $CDCl_3$)

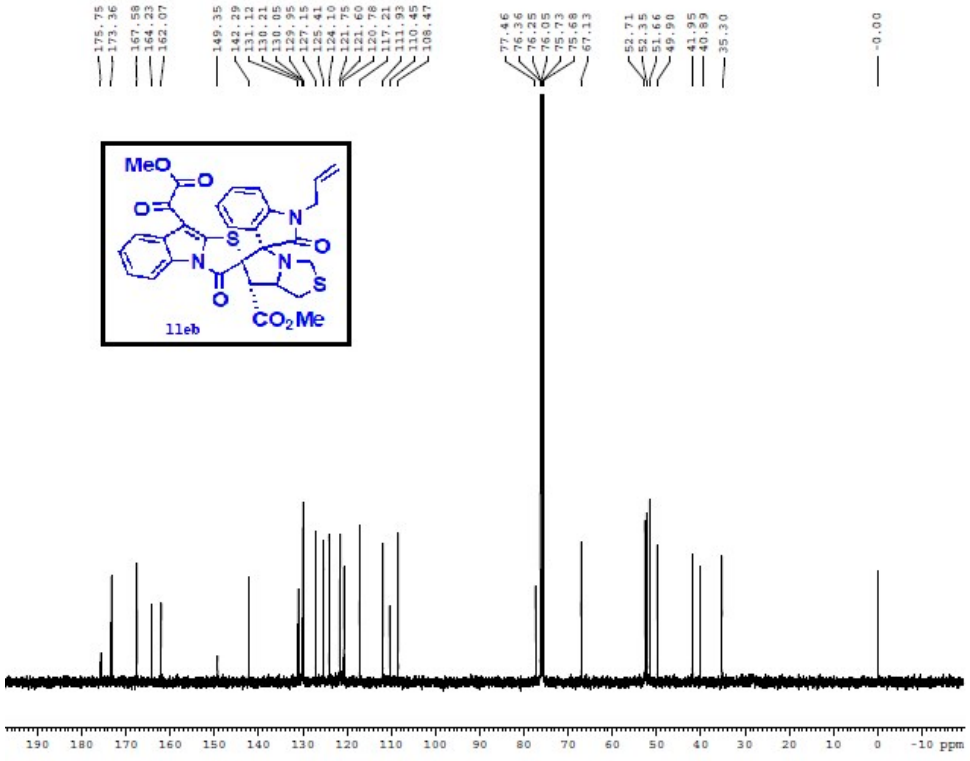
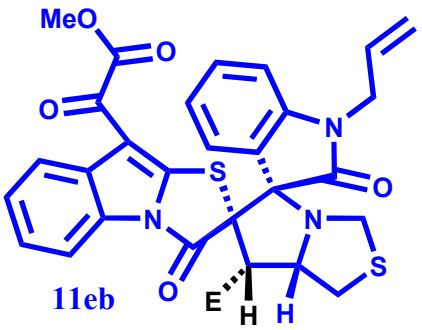



**Figure S40**. $^{13}C$ NMR of Compound **11eb** (100 MHz, $CDCl_3$)




**E = $CO_2Me$**

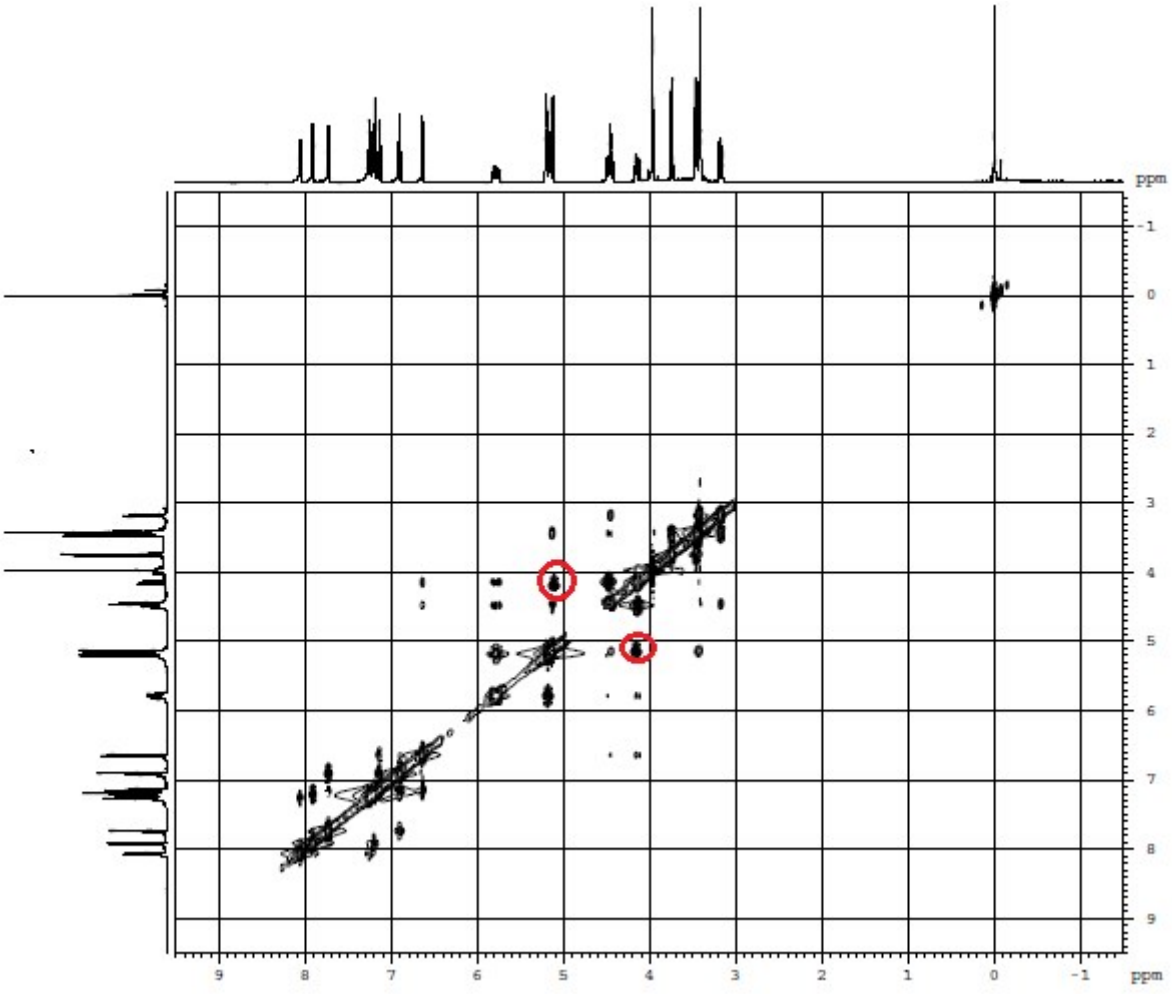
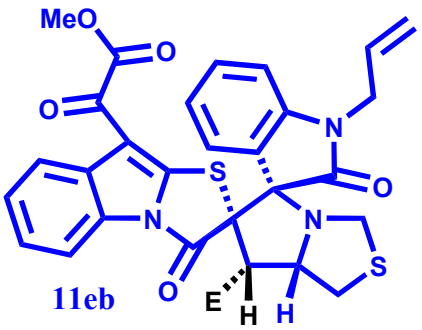



**Figure S41**. $^{1}H$-$^{1}H$ NOESY of Compound **11eb**




**E = $CO_2Me$**

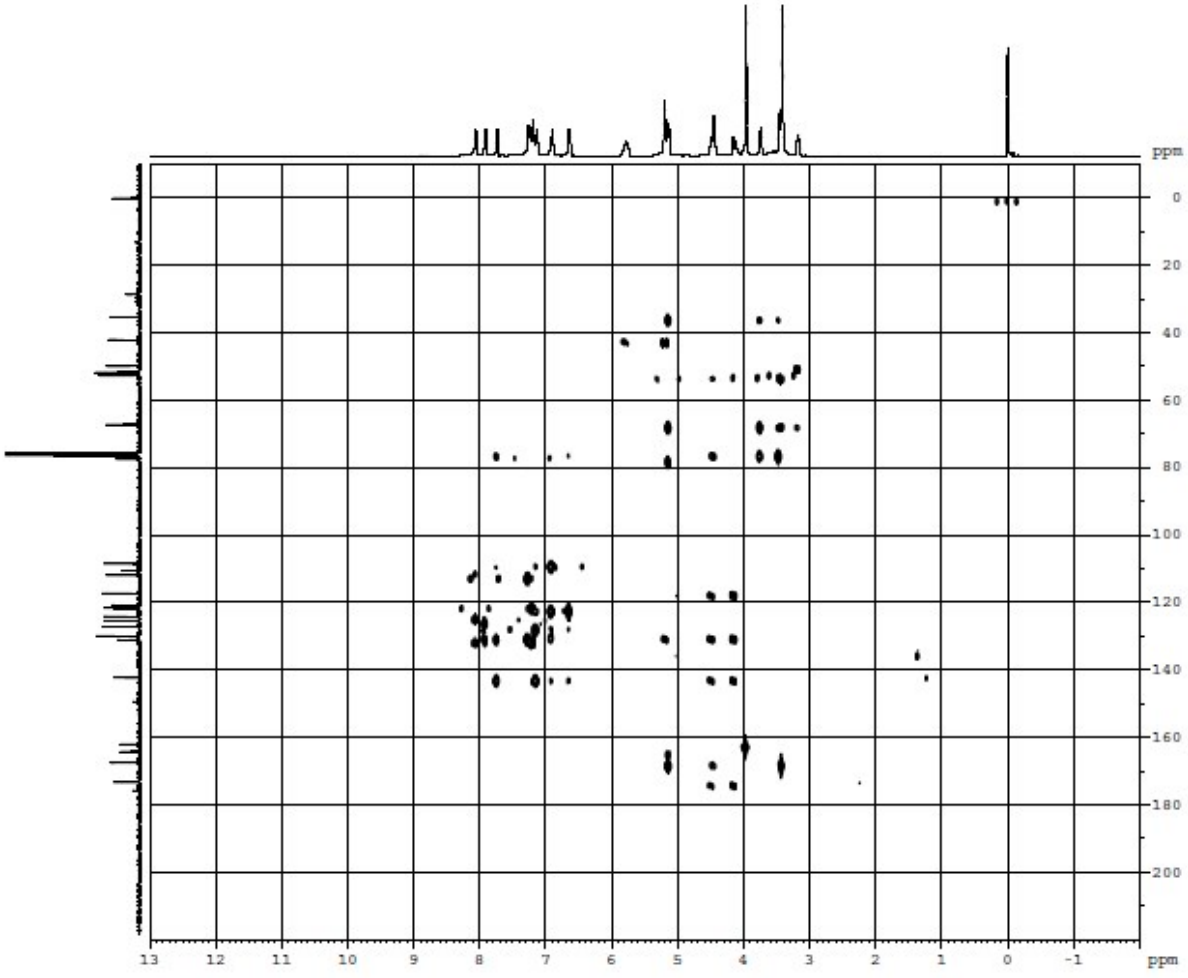
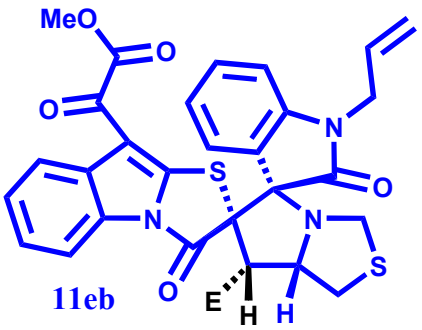



**Figure S42**. HMBC of Compound **11eb**




**E = $CO_2Me$**

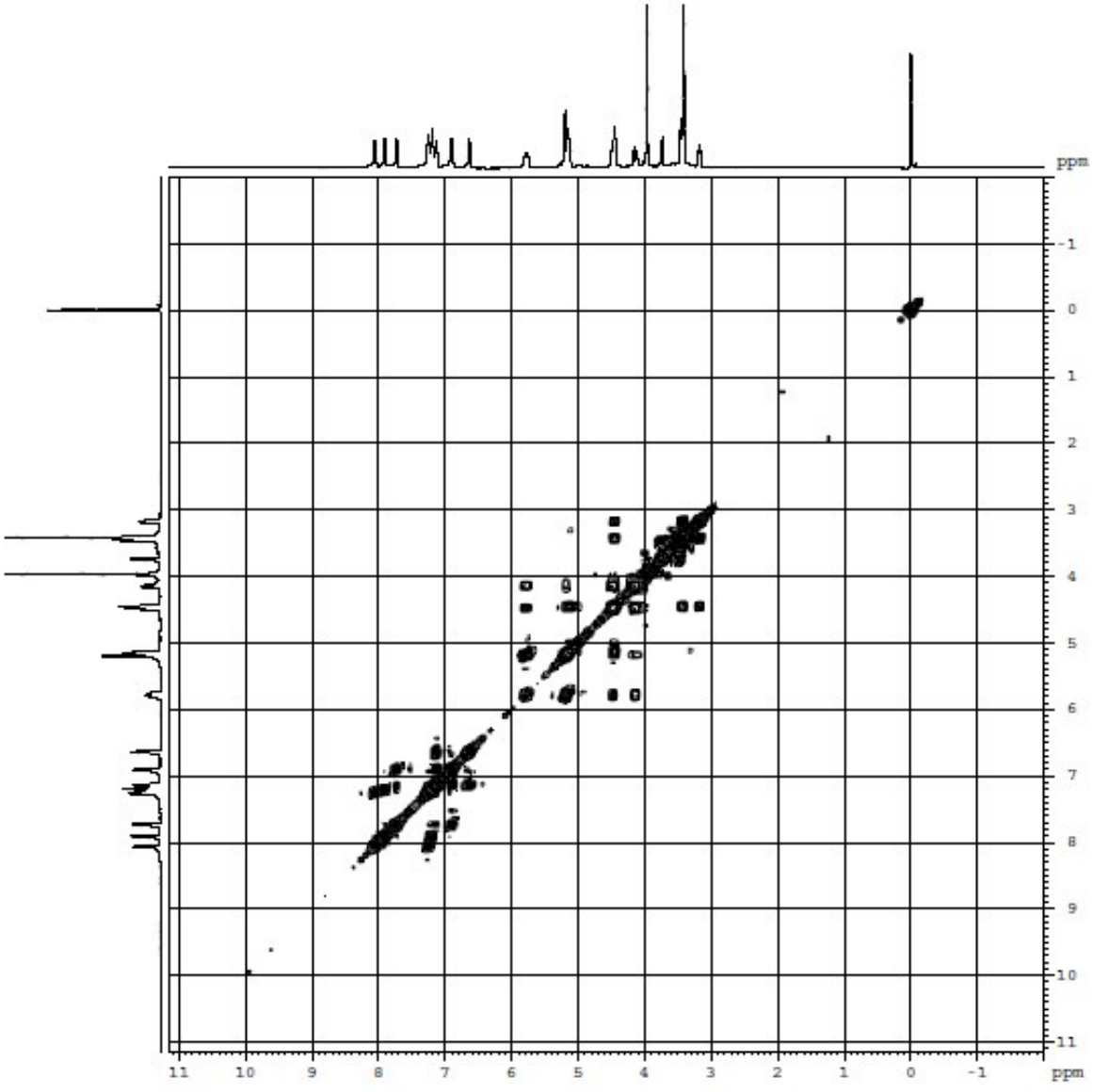



**Figure S43**. $^{1}H$-$^{1}H$ COSY of Compound **11eb**

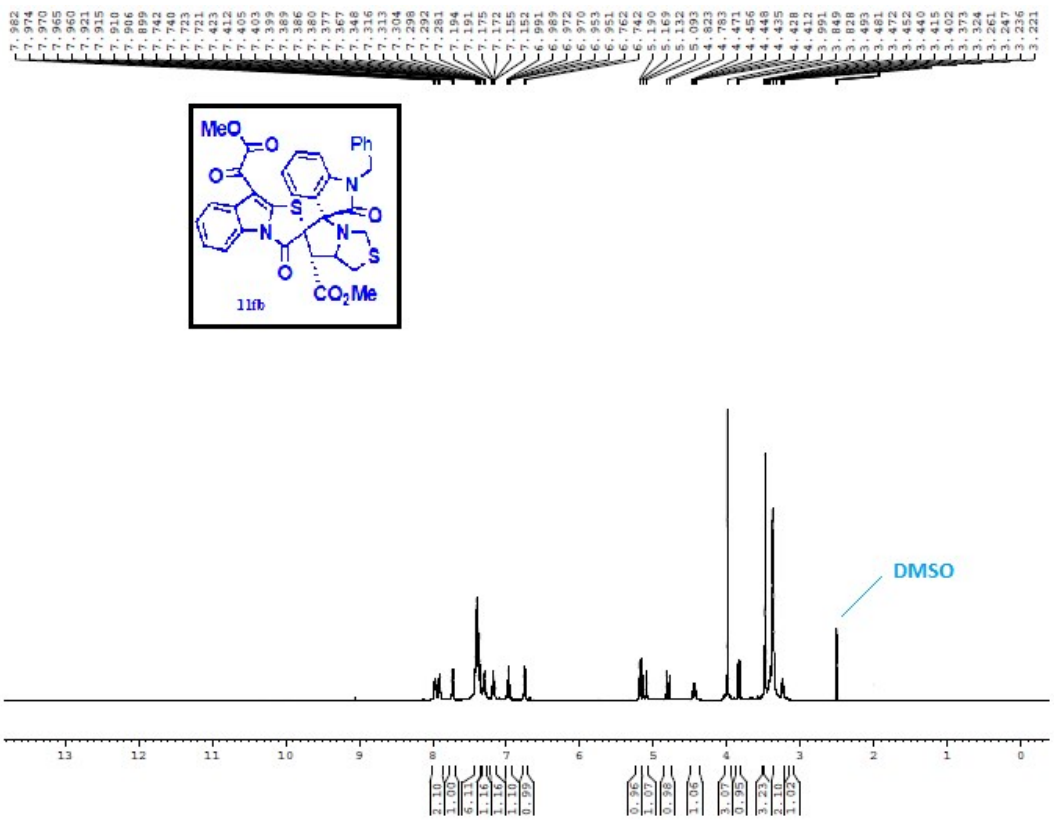



**Figure S44**. [1]H NMR of Compound **11fb** (400 MHz, DMSO)

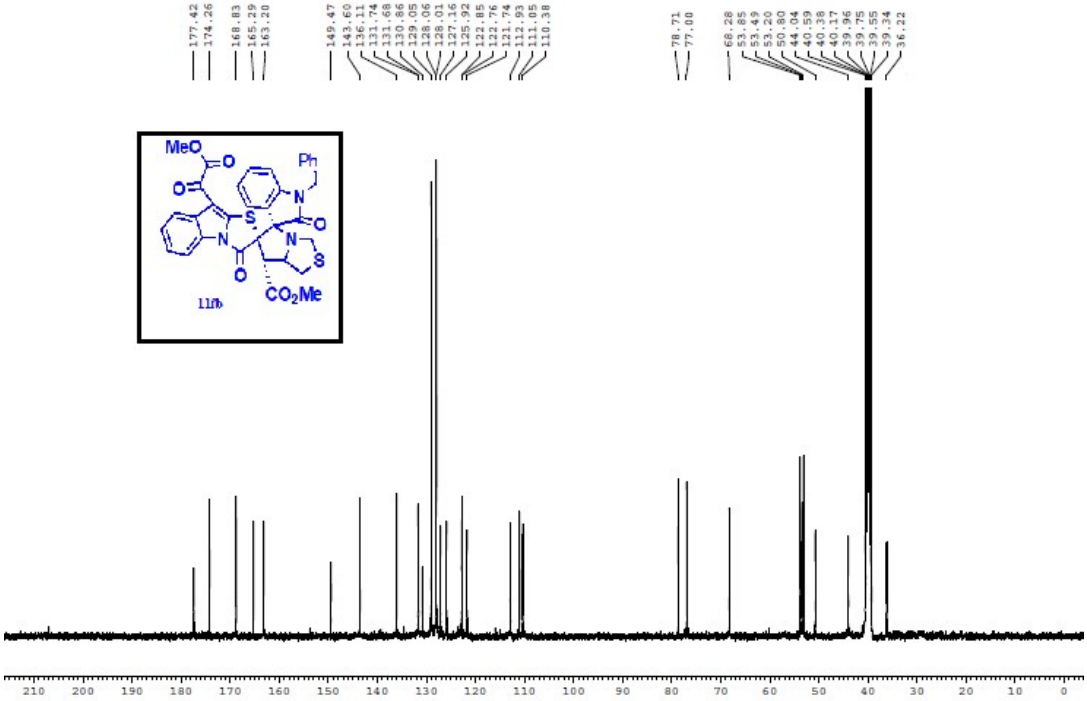



**Figure S45**. [13]C NMR of Compound **11eb** (100 MHz, $CDCl_3$)

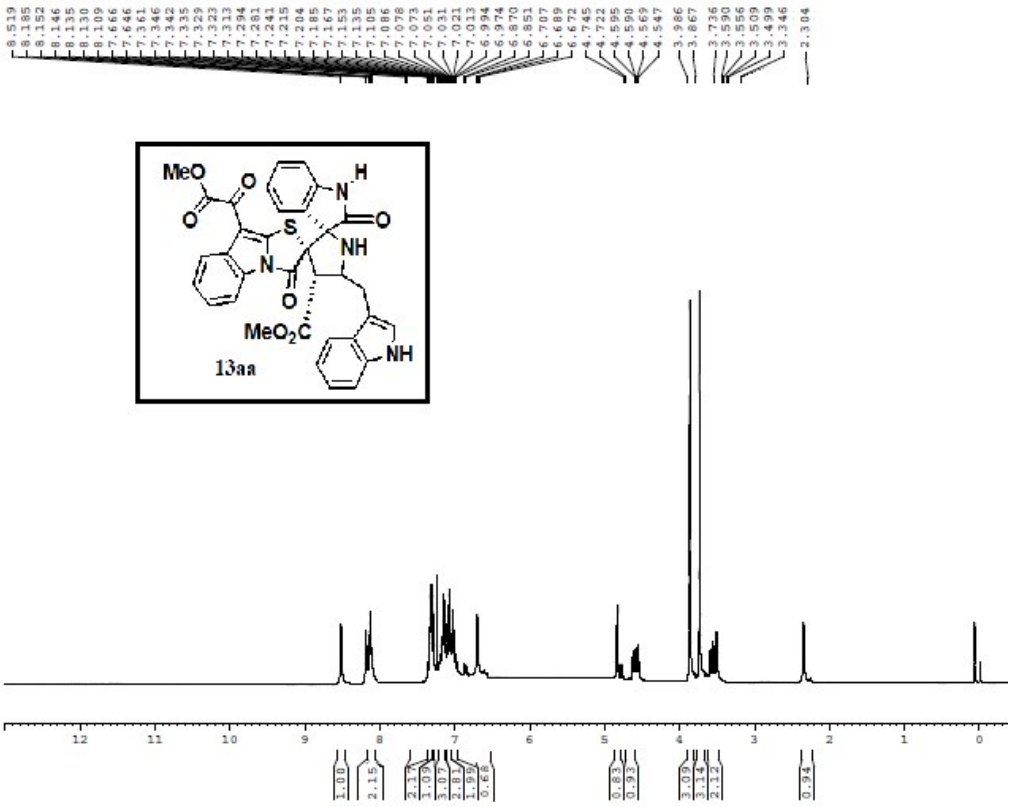



**Figure S46**. $^{1}H$ NMR of Compound **13aa** (400 MHz, $CDCl_3$)

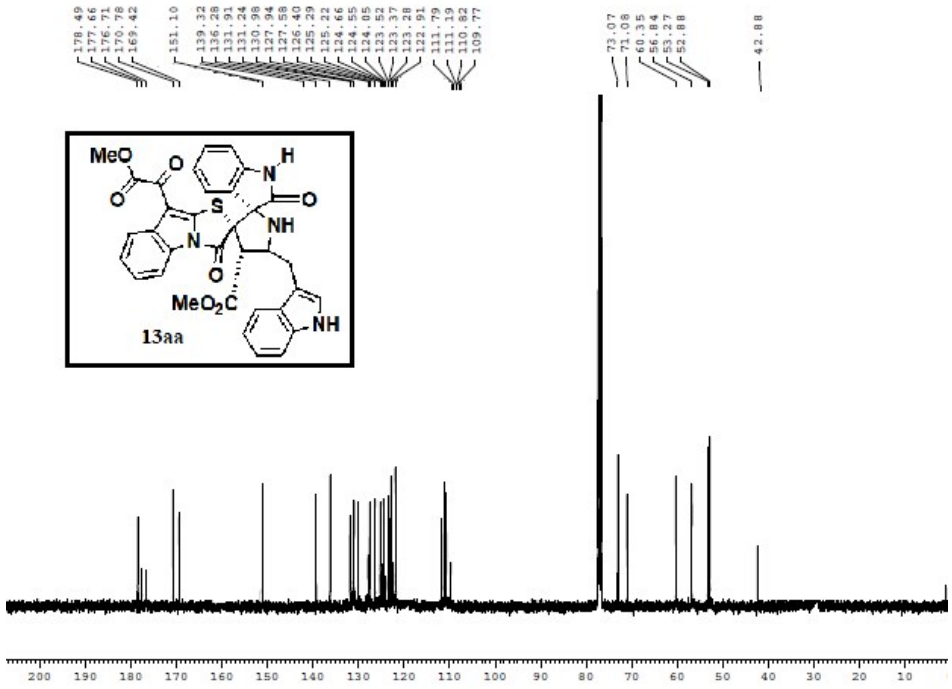
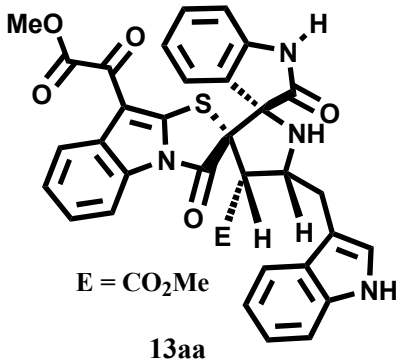



**Figure S47**. $^{13}C$ NMR of Compound **13aa** (100 MHz, $CDCl_3$)




**E = $CO_2Me$**

**13aa**

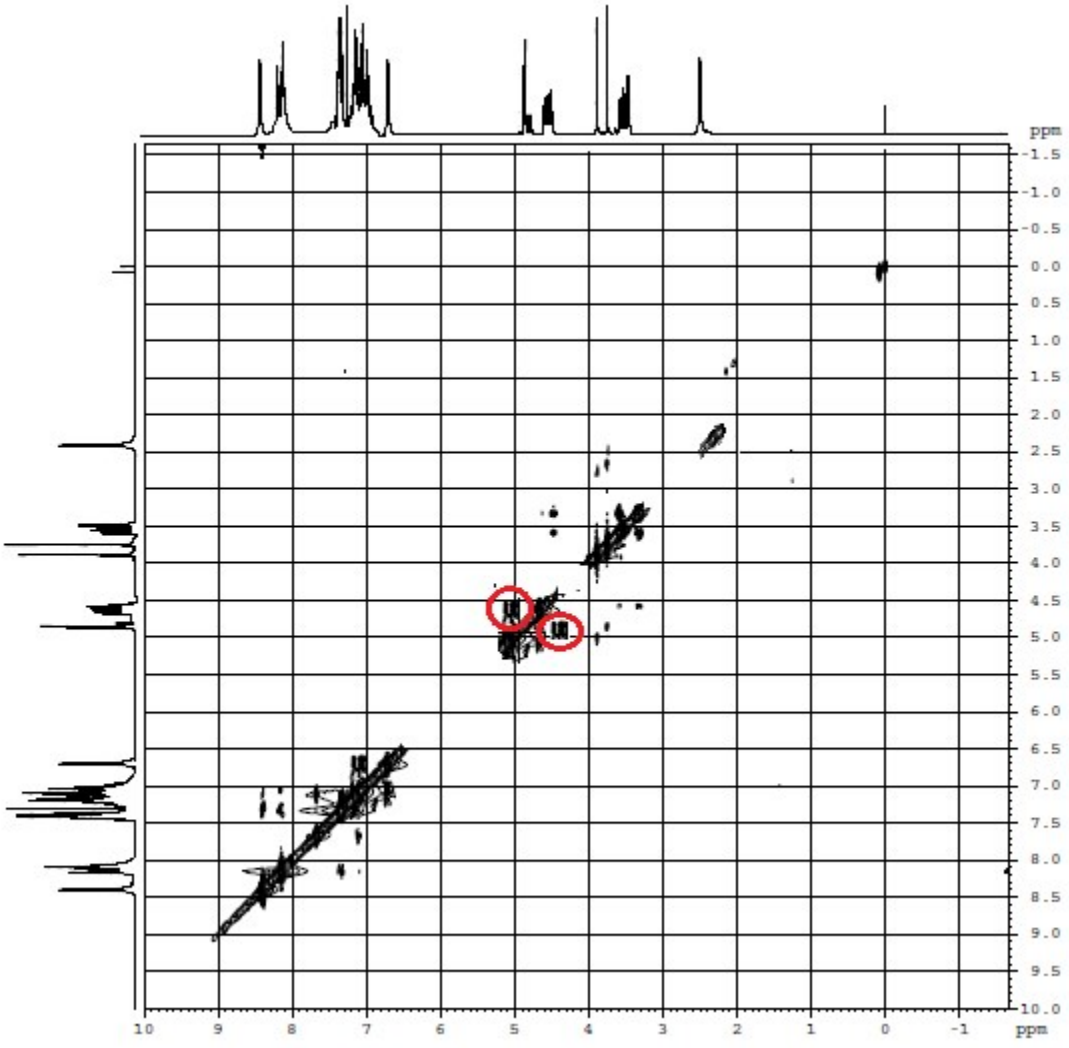
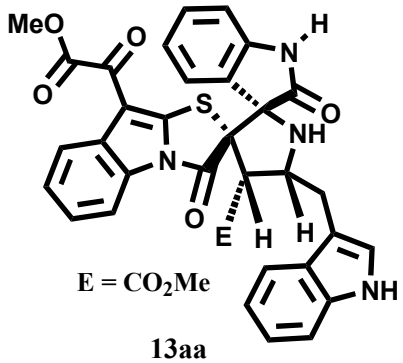



**Figure S48**. $^{1}$H-$^{1}$H NOESY of Compound **13aa**




**E = $CO_2Me$**

**13aa**

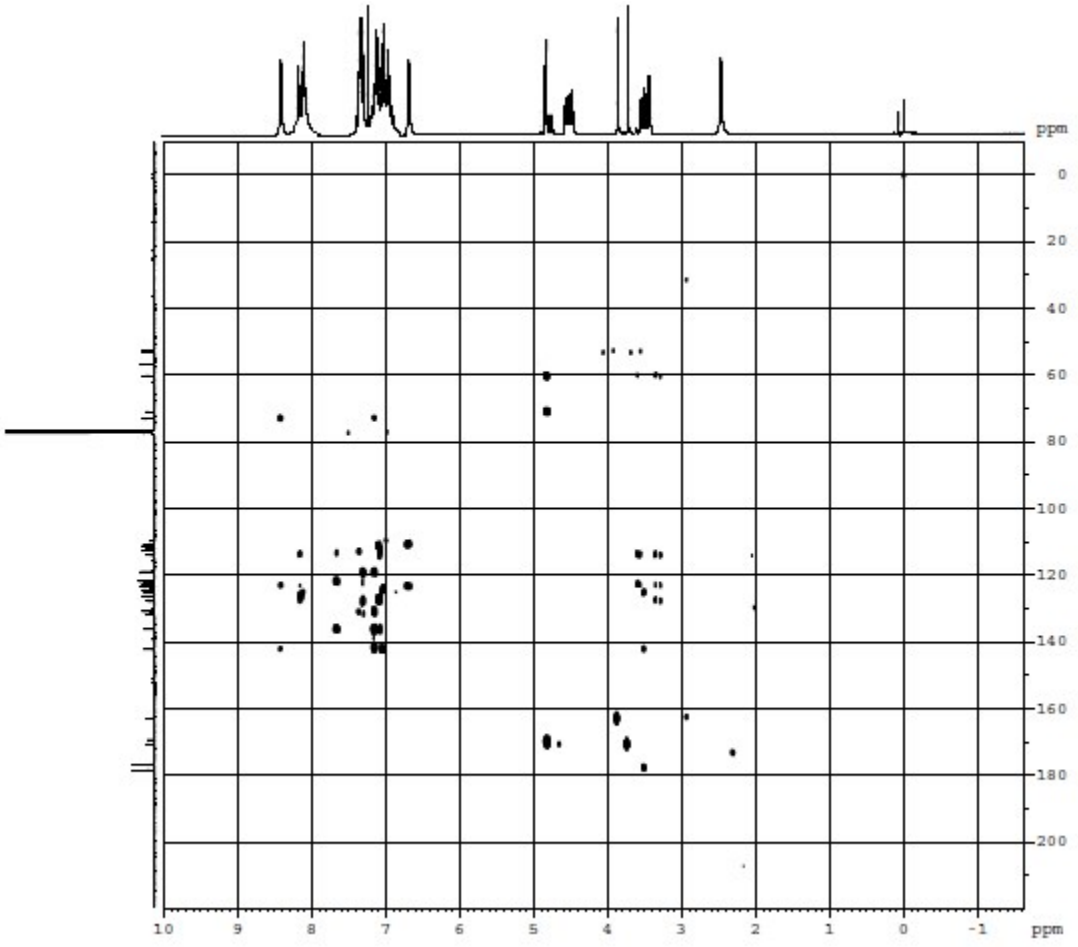
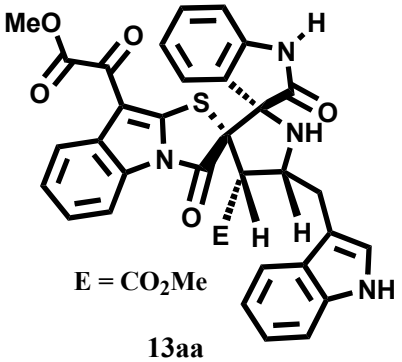



**Figure S49**. HMBC of Compound **13aa**




**E = $CO_2Me$**

**13aa**

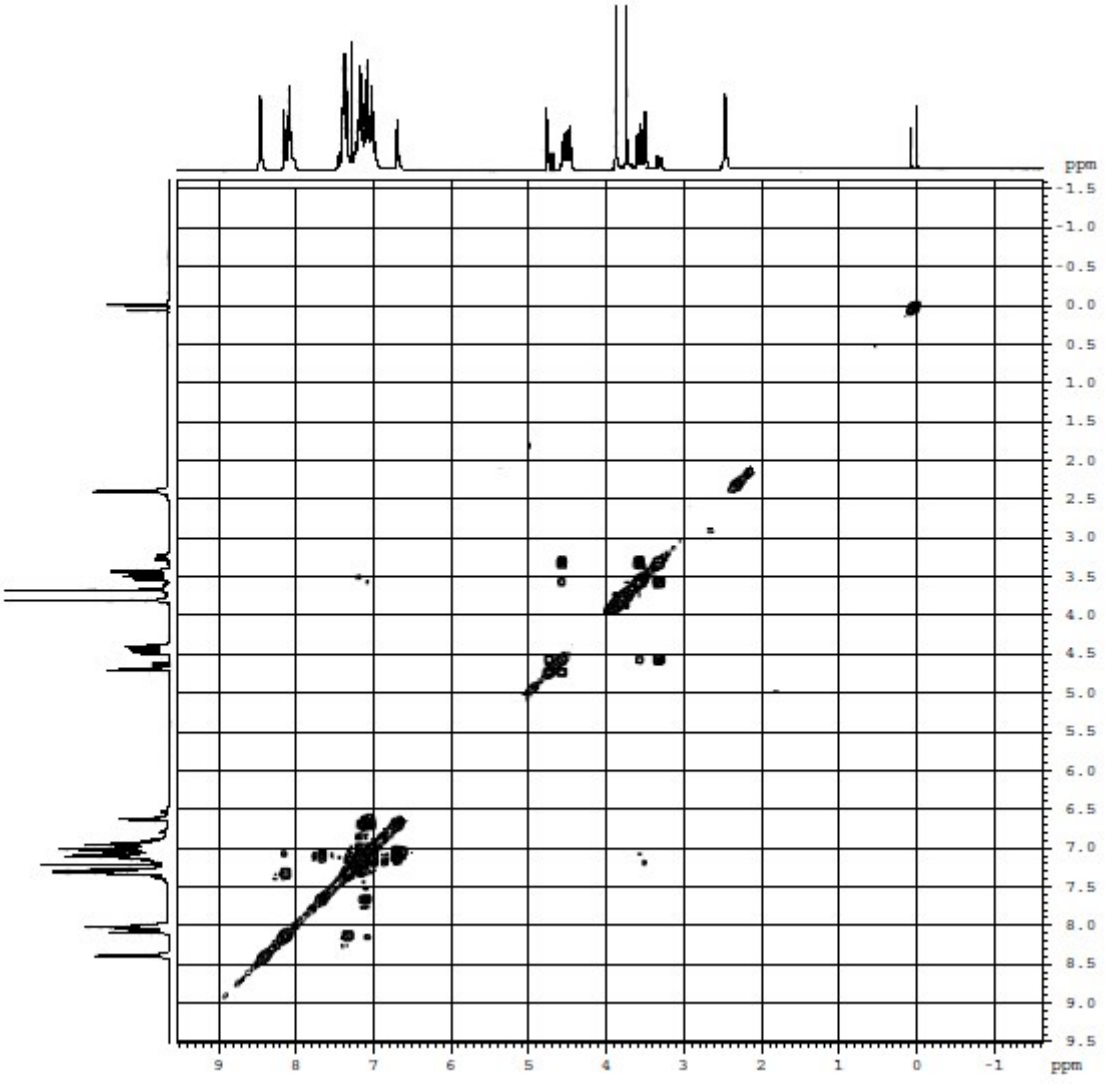



**Figure S50**. $^{1}$H-$^{1}$H COSY of Compound **13aa**[2]

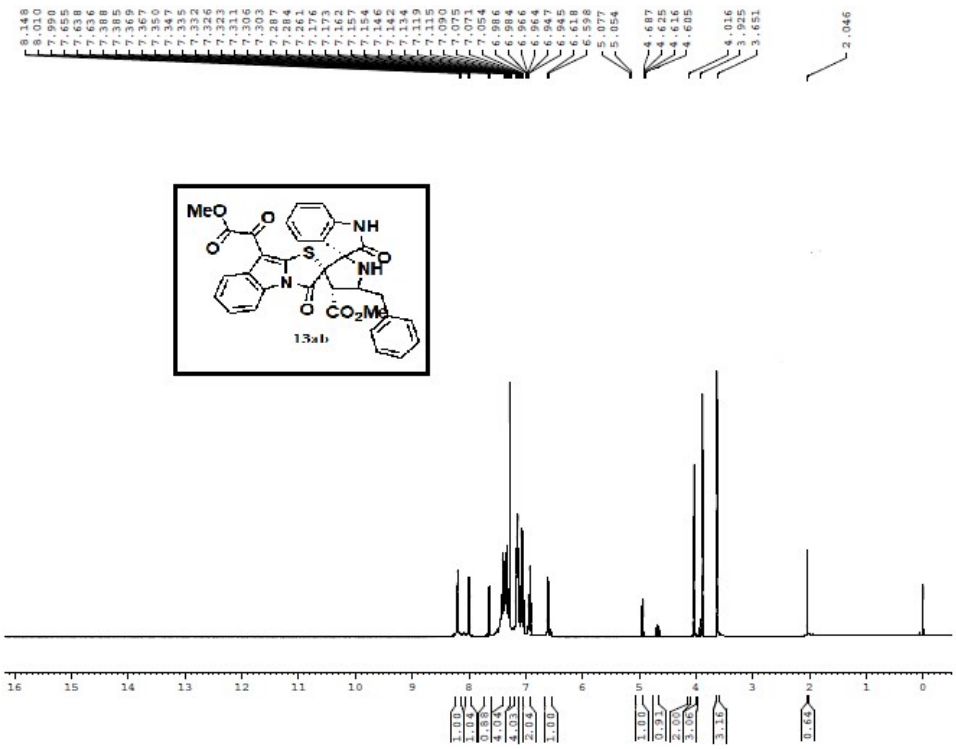



**Figure S51**. $^{1}H$ NMR of Compound **13ab** (400 MHz, $CDCl_3$)

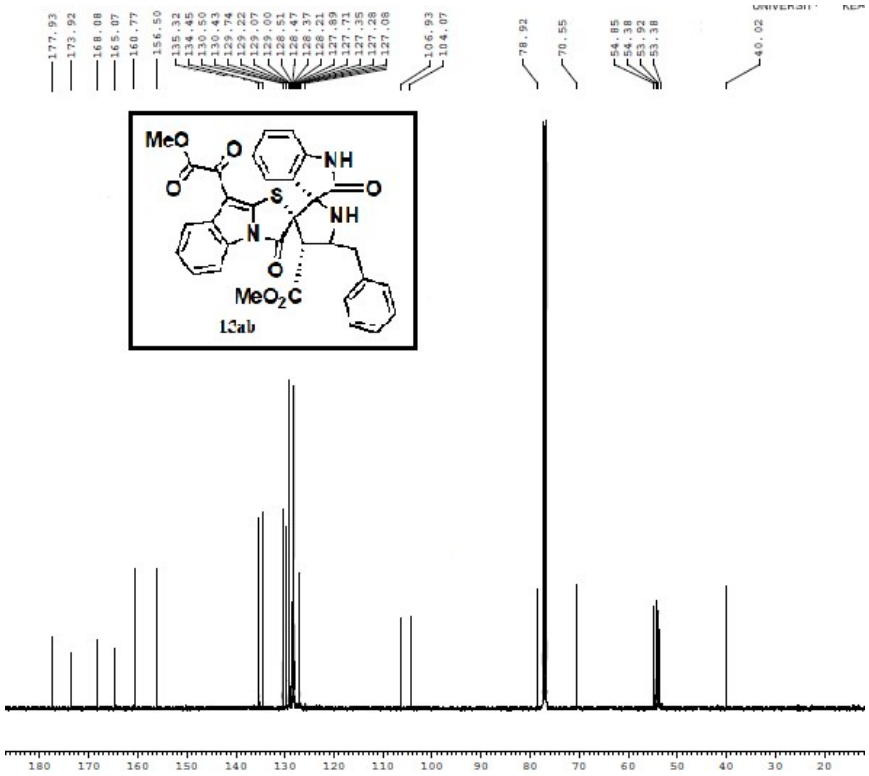



**Figure S52**. $^{13}C$ NMR of Compound **13ab** (100 MHz, $CDCl_3$)

**Table 1. Crystal data and structure refinement for 9a (S53)**

| | | |
|---|---|---|
| Identification code | shelx | |
| Empirical formula | C27 H25 N3 O8 S | |
| Formula weight | 551.56 | |
| Temperature | 296(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Triclinic | |
| Space group | P -1 | |
| Unit cell dimensions | a = 10.2438(16) Å | α= 100.773(5)°. |
| | b = 11.0264(18) Å | β= 92.253(5)°. |
| | c = 11.5014(17) Å | γ = 92.481(5)°. |
| Volume | 1273.5(3) $Å^3$ | |
| Z | 2 | |
| Density (calculated) | 1.438 $Mg/m^3$ | |
| Absorption coefficient | 0.185 $mm^{-1}$ | |
| F(000) | 576 | |
| Crystal size | 0.150 x 0.120 x 0.045 $mm^3$ | |
| Theta range for data collection | 2.623 to 24.999°. | |
| Index ranges | -12<=h<=12, -13<=k<=13, -13<=l<=13 | |
| Reflections collected | 29760 | |
| Independent reflections | 4481 [R(int) = 0.0563] | |
| Completeness to theta = 24.999° | 99.9 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.992 and 0.973 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 4481 / 0 / 357 | |
| Goodness-of-fit on $F^2$ | 1.017 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0430, wR2 = 0.0951 | |
| R indices (all data) | R1 = 0.0659, wR2 = 0.1093 | |
| Extinction coefficient | n/a | |
| Largest diff. peak and hole | 0.226 and -0.252 $e.Å^{-3}$ | |