



SUPPLEMENTARY MATERIAL TO

Synthesis of sulfonamides bearing 1,3,5-triarylpyrazoline and 4-thiazolidinone moieties as novel antimicrobial agents

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GENERAL PROCEDURE FOR THE SYNTHESIS OF CHALCONES (1a–i)

To a stirred solution of acetophenones (0.215 mol) and an aldehyde (0.215 mol) in methanol (60 mL) was slowly added 100 mL of an aqueous sodium hydroxide solution (2.8 M) and mixed occasionally for 4 h at room temperature, monitoring by TLC. After completion of the reaction, the mixture was cooled overnight at 0 °C. The separated solid was filtered and washed with water (10 mL) and cooled ethanol (10 mL). The solid was dried under vacuum. It was purified by recrystallization from ethanol to afford the pure chalcone.

GENERAL PROCEDURE FOR THE SYNTHESIS OF PHENYLHYDRAZONES (3a–e)

To a stirred solution of 4-hydrazinylbenzenesulfonamide hydrochloride (2.5 mmol) and a benzaldehyde (2.5 mmol) in methanol (30 mL) was added one drop of acetic acid. The mixture was refluxed under stirring for 4 h with Dean–Stark equipment. The solvent was evaporated under vacuum and the residue recrystallized from an appropriate solvent to afford the pure phenylhydrazone.

CHARACTERIZATION DATA FOR CHALCONES (1a–i)

Benzalacetophenone (1a). Yield: 80.8 %; yellow powder; ¹H-NMR (500 MHz, acetone-*d*₆, δ / ppm): 8.16–8.14 (2H, *m*, CH), 7.89 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.86–7.83 (2H, *m*, CH), 7.81 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.67–7.64 (1H, *m*, CH), 7.59–7.55 (2H, *m*, CH), 7.49–7.45 (1H, *m*, CH).

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(2E)-3-(2-Hydroxyphenyl)-1-phenylprop-2-en-1-one (**1b**). Yield 82.3 %. orange powder; ¹H-NMR (500 MHz, acetone-*d*₆, δ / ppm): 8.23–8.19 (1H, *d*, *J* 15.5 Hz, CO–CH=CH), 8.17–8.14 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 8.09–8.06 (2H, *m*, CH), 7.57–7.54 (1H, *m*, CH), 7.52–7.46 (3H, *m*, CH), 7.07–7.00 (2H, *m*, CH), 6.48–6.45 (1H, *m*, CH).

(2E)-3-(4-Methylphenyl)-1-phenylprop-2-en-1-one (**1c**). Yield: 81.0 %. orange powder; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 7.77 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 8.01–7.97 (2H, *m*, CH), 7.86–7.57 (2H, *m*, CH), 7.52–7.50 (1H, *m*, CH), 7.48–7.44 (2H, *m*, CH), 7.42 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.16–7.12 (1H, *m*, CH), 2.34 (3H, *s*, CH₃).

(2E)-3-(4-Methoxyphenyl)-1-phenylprop-2-en-1-one (**1d**). Orange powder. Yield: 81.0 %; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 8.00–7.98 (2H, *m*, CH), 7.78 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.88–7.55 (2H, *m*, CH), 7.54–7.52 (1H, *m*, CH), 7.48–7.45 (2H, *m*, CH), 7.41 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 6.92–6.89 (2H, *m*, CH), 3.80 (3H, *s*, OCH₃).

(2E)-1-(4-Fluorophenyl)-3-(4-methylphenyl)prop-2-en-1-one (**1e**). Yield: 76.0 %; pale yellow powder; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 8.05–8.01 (2H, *m*, CH), 7.79 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.53 (2H, *d*, *J* = 8.0 Hz, CH), 7.46 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.21–7.20 (2H, *m*, CH), 7.17–7.12 (2H, *m*, CH), 2.37 (3H, *s*, CH₃).

(2E)-1-(4-Fluorophenyl)-3-(4-methoxyphenyl)prop-2-en-1-one (**1f**). Yield: 82.0 %; pale yellow powder; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 8.03–8.00 (2H, *m*, CH), 7.77 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.57 (2H, *d*, *J* = 8.5 Hz, CH), 7.37 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.15–7.11 (2H, *m*, CH), 6.92–6.89 (2H, *m*, CH), 3.81 (3H, *s*, OCH₃).

(2E)-1-(4-Methoxyphenyl)-3-phenylprop-2-en-1-one (**1g**). Yield: 78.0 %; pale yellow powder; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 8.15–8.13 (2H, *m*, CH), 7.84–7.82 (2H, *m*, CH), 7.80 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.69 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.08–7.06 (2H, *m*, CH), 7.02–7.00 (2H, *m*, CH), 3.81 (3H, *s*, OCH₃).

(2E)-1-(4-Methoxyphenyl)-3-(4-methylphenyl)prop-2-en-1-one (**1h**). Yield: 70.0 %; pale yellow powder; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 8.17–8.13 (2H, *m*, CH), 7.88 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.77–7.75 (2H, *d*, *J* = 8.0 Hz, CH), 7.69 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.27 (2H, *d*, *J* = 8.0 Hz, CH), 7.09–7.06 (2H, *m*, CH), 3.86 (3H, *s*, OCH₃), 2.35 (3H, *s*, CH₃).

(2E)-1,3-Bis(4-methoxyphenyl)prop-2-en-1-one (**1i**). Yield: 65.0 %; yellow powder; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 8.15–8.13 (2H, *m*, CH), 7.84–7.82 (2H, *m*, CH), 7.80 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.69 (1H, *d*, *J* = 15.5 Hz, CO–CH=CH), 7.08–7.06 (2H, *m*, CH), 7.02–7.00 (2H, *m*, CH), 3.86 (3H, *s*, OCH₃), 3.81 (3H, *s*, OCH₃).

SPECTRAL DATA OF DIHYDRO-1H-PYRAZOL-1-YLBENZENESULFONAMIDES
(2a–2i)

4-(3,5-Diphenyl-4,5-dihydro-1H-pyrazol-1-yl)benzenesulfonamide (2a). Yield: 61.0 %; pale yellow powder; m.p.: 209–210 °C; IR (KBr, ν_{\max} / cm^{-1}): 3389 (–NH₂), 3279 (NH₂), 3070, 3028 (C–H, Ar–H), 2875, 2617, 1958, 1881, 1755, 1592 (Ar–H), 1555, 1503, 1449 (Ar–H), 1397, 1328 (SO₂), 1247, 1153 (SO₂), 1096, 1025, 895, 869, 817, 757, 728, 694; ¹H-NMR (500 MHz, acetone-*d*₆, δ / ppm): 7.85–7.83 (2H, *m*, CH), 7.66 (2H, *d*, $J = 9.0$ Hz, CH), 7.46–7.40 (4H, *m*, CH), 7.39–7.34 (4H, *m*, CH), 7.31–7.28 (1H, *m*, CH), 7.16 (2H, *d*, $J = 9.0$ Hz, CH), 6.22 (2H, *s*, NH₂), 5.64 (1H, *dd*, $J_1 = 12.0$ Hz & $J_2 = 5.5$ Hz, CH₂–CH), 4.09 (1H, *dd*, $J_1 = 17.5$ & $J_2 = 12.5$, CH₂), 3.27 (1H, *dd*, $J_1 = 17.5$ Hz & $J_2 = 6.0$ Hz, CH₂); ¹³C-NMR (125 MHz, acetone-*d*₆, δ / ppm): 149.5, 146.8, 142.1, 133.3, 132.4, 129.2, 128.6, 127.7, 127.4, 126.1, 125.8, 112.2, 63.3, 43.3; ESI-MS (*m/z*): Calcd. for C₂₁H₁₈SO₂N₃ ([M – H][–]: 376.1119). Found: 376.1124.

4-[5-(2-Hydroxyphenyl)-3-phenyl-4,5-dihydro-1H-pyrazol-1-yl]benzenesulfonamide (2b). Yield: 60.0 %; pale yellow powder; m.p.: 255–257 °C (lit.¹ 266–268 °C); IR (KBr, ν_{\max} / cm^{-1}): 3449 (OH), 3387 (NH₂), 3272 (NH₂), 1592 (Ar–H), 1504 (Ar–H), 1453 (Ar–H), 1400, 1335 (SO₂), 1303, 1215, 1145 (SO₂), 1093, 906, 874, 820, 751, 690; ¹H-NMR (500 MHz, acetone-*d*₆, δ / ppm): 7.84 (2H, *d*, $J = 7.5$ Hz, CH), 7.66 (2H, *d*, $J = 9.0$ Hz, CH), 7.45–7.36 (3H, *m*, CH), 7.13–7.09 (3H, *m*, CH), 6.98–6.95 (2H, *m*, CH), 6.76 (1H, *t*, $J = 7.5$ Hz, CH), 6.23 (2H, *s*, NH₂), 5.83 (1H, *dd*, $J_1 = 12.0$ Hz & $J_2 = 5.5$ Hz, CH₂–CH), 4.03 (1H, *dd*, $J_1 = 17.5$ Hz & $J_2 = 12.0$ Hz, CH₂), 3.22 (1H, *dd*, $J_1 = 17.5$ Hz & $J_2 = 5.5$ Hz, CH₂); ¹³C-NMR (125 MHz, acetone-*d*₆, δ / ppm): 154.1, 150.0, 146.8, 132.9, 132.6, 128.7, 128.6, 127.5, 127.4, 126.3, 126.0, 120.1, 115.7, 114.4, 111.9, 57.6, 41.9. ESI-MS (*m/z*): 392.1065 [M – H][–]; Calcd. for C₂₁H₁₈N₃O₃S ([M – H][–] = 392.1069).

4-[5-(4-Methylphenyl)-3-phenyl-4,5-dihydro-1H-pyrazol-1-yl]benzenesulfonamide (2c). Yield: 57.0 %; pale yellow powder; m.p.: 211–212 °C; IR (KBr, ν_{\max} / cm^{-1}): 3363 (NH₂), 3264 (NH₂), 3057, 3024, 2914, 1592 (Ar–H), 1506 (Ar–H), 1443 (Ar–H), 1401, 1337 (SO₂), 1238, 1152 (SO₂), 1095, 910, 871, 818, 755, 687; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 7.75 (2H, *d*, $J = 8.0$ Hz, CH), 7.71 (2H, *d*, $J_1 = 9.0$ Hz, CH), 7.43–7.37 (3H, *m*, CH), 7.14 (4H, *br s*, CH), 7.10 (2H, *d*, $J = 9.0$ Hz, –CH), 5.35 (1H, *dd*, $J_1 = 12.5$ Hz & $J_2 = 5.5$ Hz, CH₂–CH), 4.60 (2H, *s*, NH₂), 3.91 (1H, *dd*, $J_1 = 17.5$ Hz & $J_2 = 7.5$ Hz, CH₂), 3.22 (1H, *dd*, $J_1 = 17.5$ Hz & $J_2 = 5.5$, CH₂), 2.32 (3H, *s*, CH₃); ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 149.6, 147.4, 138.2, 137.9, 132.0, 130.2, 130.1, 129.4, 128.7, 128.1, 126.1, 125.6, 112.6, 63.4, 43.7, 21.0; ESI-MS (*m/z*): Calcd. for C₂₂H₂₂N₃O₂S ([M + H]⁺: 392.1432). Found: 392.1298.

4-[5-(4-Methoxyphenyl)-3-phenyl-4,5-dihydro-1H-pyrazol-1-yl]benzenesulfonamide (2d). Yield: 55.0 %; pale yellow powder; m.p.: 215–217 °C. IR (KBr,

ν_{\max} / cm^{-1}): 3387 (NH₂), 3263 (NH₂), 1592 (Ar-H), 1506 (Ar-H), 1450 (Ar-H), 1402, 1336 (SO₂), 1300, 1220, 1148 (SO₂), 1095, 908, 872, 820, 754, 690. ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 7.75 (2H, *dd*, $J_1 = 8.0$ Hz & $J_2 = 1.5$ Hz, CH), 7.69 (2H, *d*, $J = 9.0$ Hz, CH), 7.42–7.37 (3H, *m*, CH), 7.17–7.15 (2H, *m*, CH), 7.09 (2H, *d*, $J = 9.0$ Hz, CH), 6.86–6.84 (2H, *m*, CH), 5.33 (1H, *dd*, $J_1 = 12.0$ Hz & $J_2 = 5.5$ Hz, CH₂–CH), 4.70 (2H, *s*, NH₂), 3.89 (1H, *dd*, $J_1 = 17.0$ Hz & $J_2 = 12.0$ Hz, CH₂), 3.77 (3H, *s*, OCH₃), 3.20 (1H, *dd*, $J_1 = 17.0$ Hz & $J_2 = 5.5$ Hz, CH₂); ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 159.3, 149.5, 147.4, 133.2, 132.0, 130.3, 129.4, 128.7, 128.0, 126.9, 126.1, 114.8, 112.6, 63.1, 55.3, 43.7. ESI-MS (*m/z*): Calcd. for C₂₂H₂₀N₃O₃S ([M–H][–]): 406.1225. Found: 406.1229 [M – H][–].

4-[3-(4-Fluorophenyl)-5-(4-methylphenyl)-4,5-dihydro-1H-pyrazol-1-yl]-benzenesulfonamide (**2e**). Yield: 53.0 %; pale yellow needles; m.p.: 158–160 °C; IR (KBr, ν_{\max} / cm^{-1}): 3371 (NH₂), 3259 (NH₂), 1594 (Ar-H), 1502 (Ar-H), 1414 (Ar-H), 1396, 1338 (SO₂), 1309, 1226, 1154 (SO₂), 1094, 960, 871, 820, 748, 708; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 7.73–7.70 (2H, *m*, CH), 7.69 (2H, *d*, $J = 9.0$ Hz, CH), 7.13–7.11 (4H, *m*, CH), 7.09–7.06 (4H, *m*, CH), 5.34 (1H, *dd*, $J_1 = 12.0$ Hz & $J_2 = 6.0$ Hz, CH₂–CH), 4.67 (2H, *s*, NH₂), 3.88 (1H, *dd*, $J_1 = 17.0$ Hz & $J_2 = 12.0$ Hz, CH₂), 3.19 (1H, *dd*, $J_1 = 17.5$ Hz & $J_2 = 6.0$ Hz, CH₂), 2.32 (3H, *s*, CH₃); ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 163.5 (C–F $J = 250$ Hz), 147.9 (C–F $J = 151.3$ Hz), 138.0 (C–F $J = 27.5$ Hz), 130.3, 130.1, 128.3, 128.1, 128.0, 127.9, 125.6, 115.8 (C–F $J = 21.2$ Hz), 112.6, 63.5, 43.8, 21.1; ESI-MS (*m/z*): Calcd. for C₂₂H₁₉N₃O₂SF ([M – H][–]): 408.118. Found: 408.1182 [M – H][–].

4-(3-(4-Fluorophenyl)-5-(4-methoxyphenyl)-4,5-dihydro-1H-pyrazol-1-yl)-benzenesulfonamide (**2f**). Yield: 63.0 %; pale yellow needles; m.p.: 195–197 °C; IR (KBr, ν_{\max} / cm^{-1}): 3351 (–NH₂), 3218 (–NH₂), 1594 (Ar-H), 1504 (Ar-H), 1415 (Ar-H), 1397, 1330 (SO₂), 1295, 1247, 1152 (SO₂), 1096, 962, 867, 832, 736; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 7.73–7.70 (2H, *m*, CH), 7.69 (2H, *d*, $J = 8.5$ Hz, CH), 7.16 (2H, *d*, $J = 9.0$ Hz, CH), 7.11–7.06 (4H, *m*, CH), 6.86 (2H, *d*, $J = 8.5$ Hz, CH), 5.33 (1H, *dd*, $J_1 = 12.0$ Hz & $J_2 = 6.0$ Hz, CH₂–CH), 4.76 (2H, *s*, NH₂), 3.87 (1H, *dd*, $J_1 = 17.0$ Hz & $J_2 = 12.0$ Hz, CH₂), 3.77 (3H, *s*, OCH₃), 3.17 (1H, *dd*, $J_1 = 17.5$ Hz & $J_2 = 6.0$ Hz, CH₂); ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 163.5 (C–F, $J = 250$ Hz), 159.4, 147.9 (C–F, $J = 151.3$ Hz), 133.1, 130.4, 128.6, 128.04, 127.99, 127.9, 126.9, 115.8 (C–F, $J = 21.2$ Hz), 114.8, 112.6, 63.2, 55.3, 43.8; ESI-MS (*m/z*): Calcd. for C₂₂H₁₉N₃O₃SF ([M–H][–]): 424.1131. Found: 424.1129 [M – H][–].

4-[3-(4-Methoxyphenyl)-5-phenyl-4,5-dihydro-1H-pyrazol-1-yl]benzenesulfonamide (**2g**). Yield: 57.0 %; orange powder; m.p.: 202–203 °C; IR (KBr, ν_{\max} / cm^{-1}): 3351 (NH₂), 3218 (NH₂), 1594 (Ar-H), 1504 (Ar-H), 1415 (Ar-H), 1397, 1330 (SO₂), 1295, 1247, 1152 (SO₂), 1096, 962, 867, 832, 736;

$^1\text{H-NMR}$ (500 MHz, $\text{DMSO-}d_6$, δ / ppm): 7.74 (2H, *d*, $J = 9.0$ Hz, CH), 7.58 (2H, *d*, $J = 9.0$ Hz, CH), 7.36–7.33 (2H, *m*, CH), 7.27–7.24 (3H, *m*, CH), 7.05 (2H, *d*, $J = 9.0$ Hz, CH), 7.02 (2H, *d*, $J = 9.0$ Hz, CH), 6.99 (2H, *s*, NH_2), 5.60 (1H, *dd*, $J_1 = 12.0$ Hz & $J_2 = 5.0$ Hz, $\text{CH}_2\text{-CH}$), 3.97 (1H, *dd*, $J_1 = 17.5$ Hz & $J_2 = 12.0$ Hz, CH_2), 3.80 (3H, *s*, OCH_3), 3.17 (1H, *dd*, $J_1 = 17.5$ Hz & $J_2 = 5.5$ Hz, CH_2); $^{13}\text{C-NMR}$ (125 MHz, $\text{DMSO-}d_6$, δ / ppm): 160.3, 149.6, 146.0, 141.7, 132.6, 129.1, 127.7, 127.5, 127.1, 125.7, 124.3, 114.2, 111.7, 62.2, 55.3, 43.2; ESI-MS (m/z): Calcd. for $\text{C}_{22}\text{H}_{20}\text{N}_3\text{O}_3\text{S}$ ($[\text{M-H}]^-$): 406.1225). Found: 406.1228 $[\text{M-H}]^-$.

4-[3-(4-Methoxyphenyl)-5-(4-methylphenyl)-4,5-dihydro-1H-pyrazol-1-yl]-benzenesulfonamide (2h). Yield: 54.0 %; orange powder, m.p.: 210–212 °C; IR (KBr, ν_{max} / cm^{-1}): 3415 (NH_2), 3277 (NH_2), 1589 (Ar-H), 1504 (Ar-H), 1423 (Ar-H), 1400, 1331 (SO_2), 1307, 1246, 1156 (SO_2), 1096, 941, 863, 830, 734; $^1\text{H-NMR}$ (500 MHz, $\text{DMSO-}d_6$, δ / ppm): 7.74–7.72 (2H, *m*, CH), 7.57 (2H, *d*, $J = 9.0$ Hz, CH), 7.15–7.13 (4H, *m*, CH), 7.27–7.24 (3H, *m*, CH), 7.04–7.00 (4H, *m*, CH), 6.97 (2H, *s*, NH_2), 5.56 (1H, *dd*, $J_1 = 12.0$ Hz & $J_2 = 5.0$ Hz, $\text{CH}_2\text{-CH}$), 3.95 (1H, *dd*, $J_1 = 18.0$ Hz & $J_2 = 12.0$ Hz, CH_2), 3.80 (3H, *s*, OCH_3), 3.15 (1H, *dd*, $J_1 = 17.5$ Hz & $J_2 = 5.5$ Hz, CH_2), 2.24 (3H, *s*, CH_3); $^{13}\text{C-NMR}$ (125 MHz, $\text{DMSO-}d_6$, δ / ppm): 160.2, 149.6, 146.0, 138.7, 136.7, 132.5, 129.6, 127.6, 127.0, 125.6, 124.4, 114.2, 111.7, 62.0, 55.3, 43.2, 20.6; ESI-MS (m/z): Calcd. for $\text{C}_{23}\text{H}_{24}\text{N}_3\text{O}_3\text{S}$ ($[\text{M} + \text{H}]^+$): 422.1528). Found: 422.1382 $[\text{M} + \text{H}]^+$.

4-[3,5-Bis(4-methoxyphenyl)-4,5-dihydro-1H-pyrazol-1-yl]benzenesulfonamide (2i). Yield: 60.0 %; orange powder; m.p.: 213–215 °C; IR (KBr, ν_{max} / cm^{-1}): 3342 (NH_2), 3258 (NH_2), 1592 (Ar-H), 1505 (Ar-H), 1421 (Ar-H), 1399, 1339 (SO_2), 1309, 1242, 1154 (SO_2), 1095, 927, 871, 832, 744; $^1\text{H-NMR}$ (500 MHz, $\text{DMSO-}d_6$, δ / ppm): 7.74–7.72 (2H, *m*, CH), 7.57 (*d*, 2H, $J = 9.0$, CH), 7.18–7.16 (2H, *m*, CH), 7.27–7.24 (3H, *m*, CH), 7.05–7.00 (4H, *m*, CH), 6.98 (2H, *s*, NH_2), 6.90–6.88 (2H, *m*, CH), 5.55 (1H, *dd*, $J_1 = 12.0$ Hz & $J_2 = 5.0$, $\text{CH}_2\text{-CH}$), 3.93 (1H, *dd*, $J_1 = 18.0$ Hz & $J_2 = 12.0$ Hz, CH_2), 3.80 (3H, *s*, OCH_3), 3.70 (1H, *s*, OCH_3), 3.15 (1H, *dd*, $J_1 = 17.5$ Hz & $J_2 = 5.0$ Hz, CH_2); $^{13}\text{C-NMR}$ (125 MHz, $\text{DMSO-}d_6$, δ / ppm): 160.2, 158.5, 149.6, 146.0, 133.6, 132.5, 127.6, 127.0, 126.9, 124.4, 114.4, 114.2, 111.7, 61.7, 55.3, 55.0, 43.2; ESI-MS (m/z): Calcd. for $\text{C}_{23}\text{H}_{22}\text{N}_3\text{O}_4\text{S}$ ($[\text{M-H}]^-$): 436.1331). Found: 436.1327 $[\text{M-H}]^-$.

CHARACTERIZATION DATA FOR PHENYLHYDRAZONES (3a–e)

4-(2-Benzylidenehydrazinyl)benzenesulfonamide (3a). Recrystallization from EtOAc. Yield 63.0 %; pale yellow powder, m.p.: 174–175 °C; $^1\text{H-NMR}$ (500 MHz, $\text{DMSO-}d_6$, δ / ppm): 10.77 (1H, *s*, =N–NH–), 7.95 (1H, *s*, CH=N), 7.70 (4H, *t*, $J = 9.0$ Hz, CH), 7.43 (2H, *t*, $J = 7.5$ Hz, CH), 7.35 (1H, *t*, $J = 7.5$ Hz, CH), 7.16 (2H, *d*, $J = 8.5$ Hz, CH), 7.06 (2H, *s*, SO_2NH_2).

4-[2-(4-Methylbenzylidene)hydrazinyl]benzenesulfonamide (3b). Recrystallization from EtOH. Yield: 67.0 %; yellow powder; m.p.: 213–214 °C. ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 10.68 (1H, *s*, =N–NH–), 7.91 (1H, *s*, CH=N), 7.66 (2H, *d*, *J* = 9.0 Hz, CH), 7.59 (2H, *d*, *J* = 8.0 Hz, CH), 7.23 (2H, *d*, *J* = 8.0 Hz, CH), 7.14 (2H, *d*, *J* = 9.0 Hz, CH), 7.04 (2H, *s*, SO₂NH₂), 2.33 (3H, *s*, CH₃).

4-[2-(4-Methoxybenzylidene)hydrazinyl]benzenesulfonamide (3c). Recrystallization from EtOH. Yield: 74.0 %; yellow needles; m.p.: 225–226 °C; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 10.59 (1H, *s*, =N–NH–), 7.90 (1H, *s*, CH=N), 7.65 (4H, *t*, *J* = 8.5 Hz, CH), 7.12 (2H, *d*, *J* = 9.0 Hz, CH), 7.03 (2H, *s*, *J* = 8.0 Hz, SO₂NH₂), 6.99 (2H, *d*, *J* = 9.0 Hz, CH), 3.79 (3H, *s*, OCH₃).

4-[2-(2-Hydroxybenzylidene)hydrazinyl]benzenesulfonamide (3d). Recrystallization from 2-propanol. Yield 71.0 %; yellow needles, m.p.: 254–255 °C; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 10.78 (1H, *s*, OH), 10.23 (1H, *s*, =N–NH), 8.24 (1H, *s*, CH=N), 7.68–7.64 (3H, *m*, CH), 7.21–7.17 (1H, *m*, CH), 7.07–7.05 (4H, *m*, CH & SO₂NH₂), 6.90–6.86 (2H, *m*, CH).

4-[2-(4-Chlorobenzylidene)hydrazinyl]benzenesulfonamide (3e). Recrystallization from dichloromethane. Yield: 66.0 %; pale yellow needles; m.p.: 210–211 °C; ¹H-NMR (500 MHz, DMSO-*d*₆ / δ, ppm): 10.85 (1H, *s*, =N–NH), 7.93 (1H, *s*, CH=N), 7.72 (2H, *d*, *J* = 8.5 Hz, CH), 7.67 (2H, *d*, *J* = 8.5 Hz, CH), 7.47 (2H, *d*, *J* = 9.0 Hz, CH), 7.16 (2H, *d*, *J* = 8.5 Hz, CH) 7.07 (2H, *m*, CH).

SPECTRAL DATA FOR 4-THIAZOLIDINONE SULFONAMIDES (4a–4e)

4-[(4-Oxo-2-phenyl-3-thiazolidinyl)amino]benzenesulfonamide (4a). Yield: 62.0 %; white needles, m.p.: 179–181 °C (lit. 181 °C¹). IR (KBr, ν_{max} / cm⁻¹): 3265 (NH), 2925, 2854, 1714 (C=O), 1599, 1461, 1378, 1331 (SO₂), 1273, 1157 (SO₂), 1045, 833, 797, 698; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 8.67 (1H, *s*, NH), 7.59 (1H, *d*, *J* = 8.0 Hz, CH), 7.42–7.39 (2H, *m*, CH), 7.39–7.34 (3H, *m*, CH), 7.05 (2H, *s*, SO₂NH₂), 6.74 (2H, *d*, *J* = 8.5 Hz, CH), 5.88 (1H, *s*, S–CH), 3.95 (1H, *d*, *J* = 15.5 Hz, CH₂), 3.81 (1H, *d*, *J* = 15.5 Hz, CH₂); ¹³C-NMR (125 MHz, DMSO-*d*₆, δ / ppm): 169.1, 149.3, 134.2, 128.8, 128.7, 127.2, 111.3, 59.8, 28.9; ESI-MS (*m/z*): Calcd. for C₁₅H₁₆N₃O₃S₂ ([M + H]⁺: 350.0633). Found: 350.0487 [M + H]⁺.

4-[[2-(4-Methylphenyl)-4-oxo-3-thiazolidinyl]amino]benzenesulfonamide (4b). Yield: 52.0 %; pale yellow needles; m.p.: 173–175 °C (lit. 159 °C¹); IR (KBr, ν_{max} / cm⁻¹): 3264 (NH), 2924, 2854, 1690 (C=O), 1598, 1509, 1459, 1410, 1331 (SO₂), 1266, 1216, 1155 (SO₂), 1097, 1040, 902, 826, 704; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 8.63 (1H, *s*, NH), 7.60 (1H, *d*, *J* = 8.0 Hz, CH), 7.32 (2H, *br*, CH), 7.20 (2H, *d*, *J* = 8.0 Hz, CH), 7.05 (2H, *s*, SO₂NH₂), 6.75 (2H, *d*, *J* = 8.5 Hz, CH), 5.85 (1H, *s*, S–CH), 3.94 (1H, *d*, *J* = 15.5 Hz, CH₂), 3.82 (1H, *d*, *J* = 15.5 Hz, CH₂), 2.31 (3H, *s*, CH₃); ¹³C-NMR (125 MHz, DMSO-*d*₆, δ / ppm): 170.3, 149.3, 138.2, 134.1, 129.1, 127.1, 111.3, 59.7, 28.8,

20.7, 14.0; ESI-MS (m/z): Calcd. for $C_{16}H_{16}N_3O_3S_2$ ($[M-H]^-$): 362.0633). Found: 362.0636 $[M-H]^-$.

4-([2-(4-Methoxyphenyl)-4-oxo-3-thiazolidinyl]amino)benzenesulfonamide (4c). Yield: 49.0 %; yellow needles; m.p.: 186–187 °C; IR (KBr, ν_{max} / cm^{-1}): 3265 (NH), 3097, 2929, 2855, 2364, 1689 (C=O), 1598, 1512, 1462, 1407, 1332 (SO₂), 1249, 1155 (SO₂), 1098, 1027, 901, 829, 734, 708, 676; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 8.59 (1H, *s*, NH), 7.58 (1H, *d*, $J = 7.5$ Hz, CH), 7.36 (2H, *br*, CH), 7.04 (2H, *s*, SO₂NH₂), 6.93 (2H, *d*, $J = 8.0$ Hz, CH), 6.73 (2H, *d*, $J = 9.0$ Hz, CH), 5.83 (1H, *s*, S-CH), 3.93 (1H, *d*, $J = 16.0$ Hz, CH₂), 3.81 (1H, *d*, $J = 16.0$ Hz, CH₂), 3.75 (3H, *s*, OCH₃); ¹³C-NMR (125 MHz, DMSO-*d*₆, δ / ppm): 168.8, 159.6, 149.4, 134.1, 129.1, 127.1, 126.7, 113.9, 111.2, 59.7, 55.2, 28.9; ESI-MS (m/z): Calcd. for $C_{16}H_{14}N_3O_4S_2$ ($[M-H]^-$): 378.0582). Found: 378.0580 $[M-H]^-$.

4-([2-(2-Hydroxyphenyl)-4-oxo-3-thiazolidinyl]amino)benzenesulfonamide (4d). Yield 55.0 %; yellow needles; m.p.: 209–210 °C; IR (KBr, ν_{max} / cm^{-1}): 3266 (NH), 3099, 2986, 2929, 2602, 1908, 1682 (C=O), 1598, 1502, 1459, 1398, 1329 (SO₂), 1153 (SO₂), 1098, 1042, 900, 885, 829, 757, 708, 682; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 9.95 (1H, *s*, OH), 8.70 (1H, *s*, NH), 7.62 (2H, *d*, $J = 7.5$ Hz, CH), 7.22–7.21 (1H, *m*, CH), 7.18 (1H, *dt*, $J_1 = 8.0$ & $J_2 = 1.5$ Hz, CH), 7.07 (2H, *s*, SO₂NH₂), 6.84 (2H, *d*, $J = 8.0$ Hz, CH), 6.79 (2H, *d*, $J = 9.0$ Hz, CH), 5.99 (1H, *s*, S-CH), 3.84 (1H, *d*, $J = 15.5$ Hz, CH₂), 3.74 (1H, *d*, $J = 16.0$ Hz, CH₂); ¹³C-NMR (125 MHz, DMSO-*d*₆, δ / ppm): 169.6, 155.1, 149.5, 134.2, 129.4, 127.3, 119.1, 111.5, 111.2, 59.7, 28.8; ESI-MS (m/z): calcd. for $C_{15}H_{14}N_3O_4S_2$ ($[M-H]^-$): 364.0425). Found: 364.0421 $[M-H]^-$.

4-([2-(4-Chlorophenyl)-4-oxo-3-thiazolidinyl]amino)benzenesulfonamide (4e). Yield: 43.0 %. pale yellow needles; m.p. 178–181 °C (lit. 163 °C²⁷); IR (KBr, ν_{max} / cm^{-1}): 3281 (NH), 3095, 2925, 2854, 2364, 2342, 1873, 1693 (C=O), 1598, 1493, 1411, 1388, 1330 (SO₂), 1264, 1220, 1155 (SO₂), 1094, 1014, 902, 828, 790, 760, 739, 707; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 8.68 (1H, *s*, NH), 7.79 (2H, *d*, $J = 8.0$, CH), 7.45–7.43 (*m*, 4H, CH), 7.06 (*s*, 2H, SO₂NH₂), 6.73 (*d*, 2H, $J = 8.5$ Hz, CH), 5.90 (1H, *s*, S-CH), 3.93 (1H, *d*, $J = 15.5$ Hz, CH₂), 3.82 (1H, *d*, $J = 16.0$ Hz, CH₂); ¹³C-NMR (125 MHz, DMSO-*d*₆, δ / ppm): 170.3, 169.0, 149.9, 149.2, 134.3, 133.3, 128.7, 127.2, 111.4, 59.8, 28.8; ESI-MS (m/z): Calcd. for $C_{15}H_{13}N_3O_3S_2Cl$ ($[M-H]^-$): 382.0086. Found: 382.0085 $[M-H]^-$.

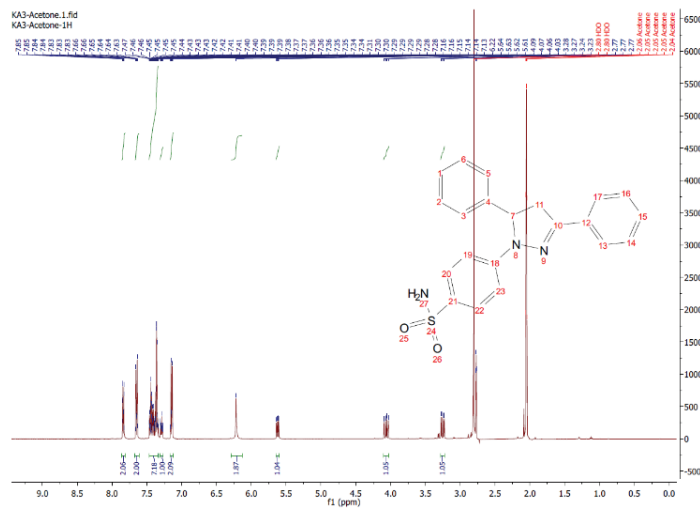


Fig. S-1. ¹H-NMR Spectrum of compound 2a (Acetone-*d*₆).

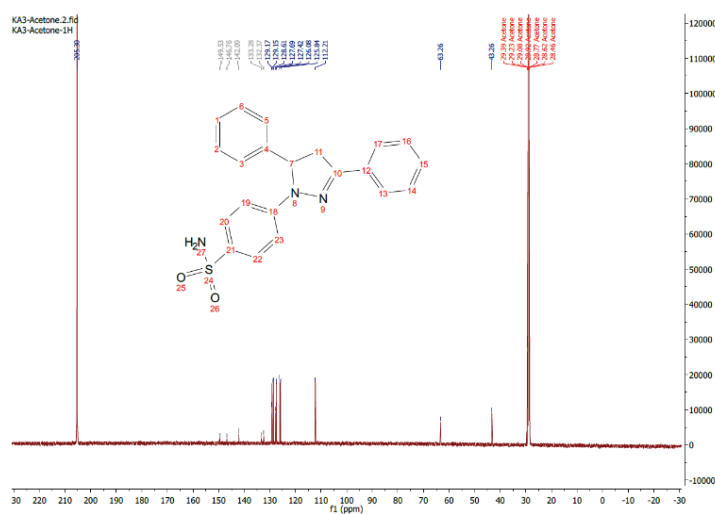


Fig. S-2. ¹³C-NMR Spectrum of compound 2a (Acetone-*d*₆).

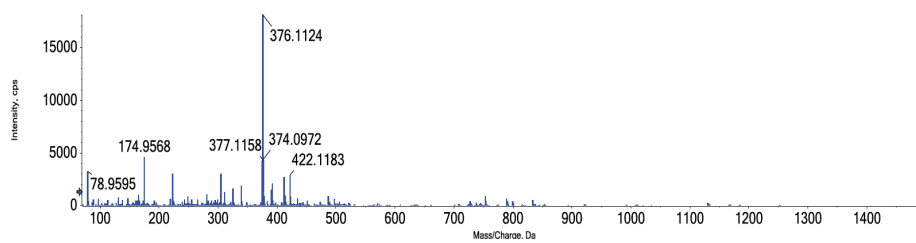
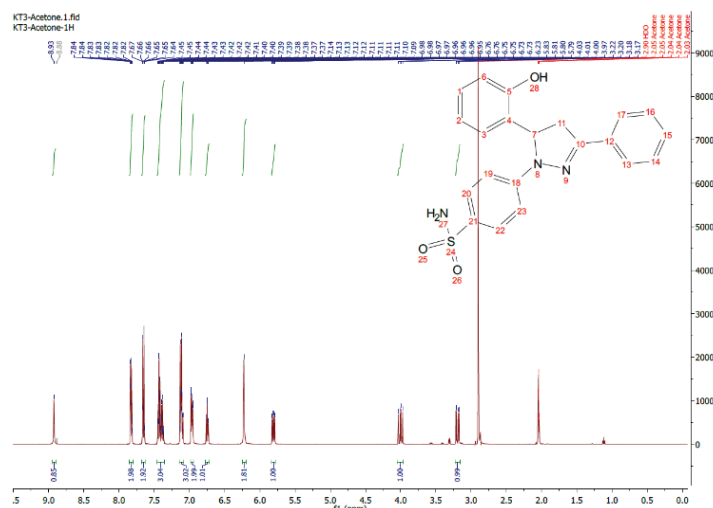
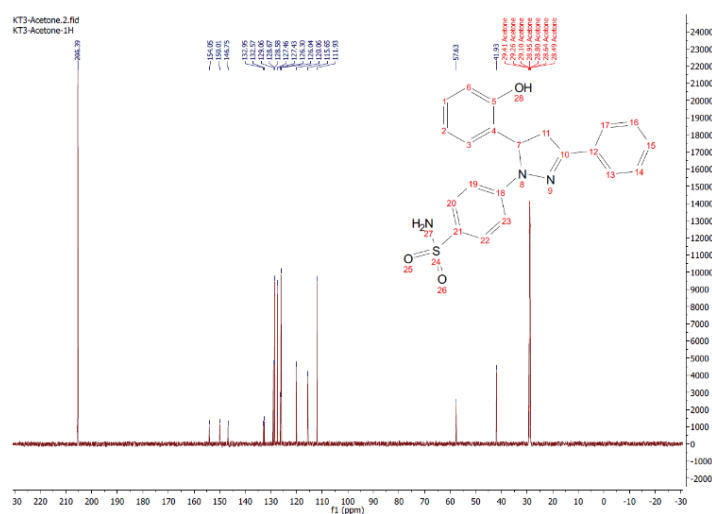
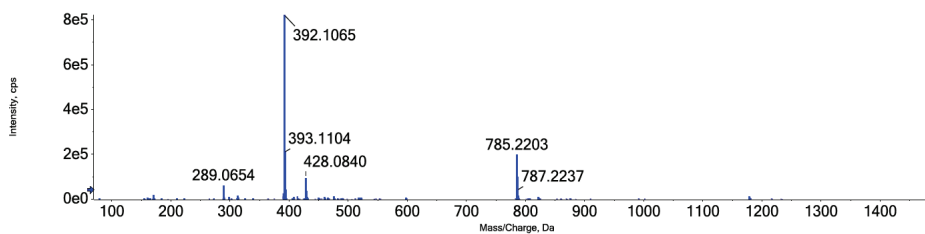


Fig. S-3. HR-MS Spectrum of compound 2a.

Fig. S-4. ^1H -NMR Spectrum of compound **2b** (Acetone- d_6).Fig. S-5. ^{13}C -NMR Spectrum of compound **2b** (Acetone- d_6).Fig. S-6. HR-MS Spectrum of compound **2b**.

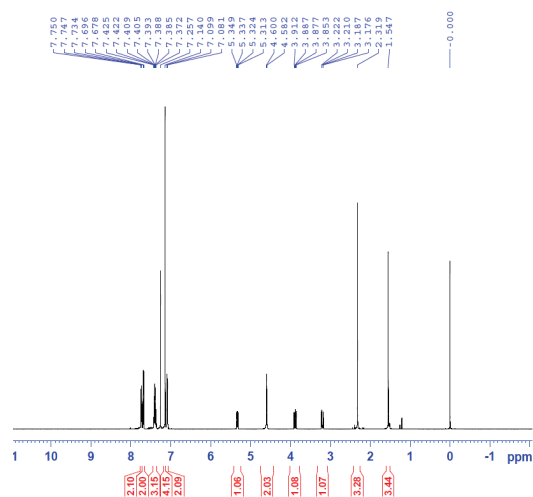


Fig. S-7. ¹H-NMR Spectrum of compound 2c (CDCl₃).

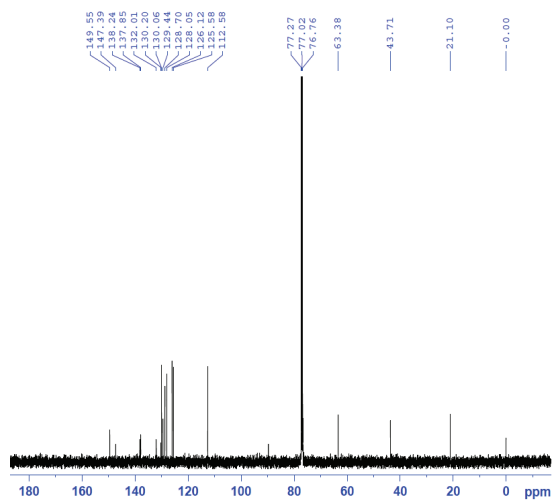


Fig. S-8. ¹³C-NMR Spectrum of compound 2c (CDCl₃).

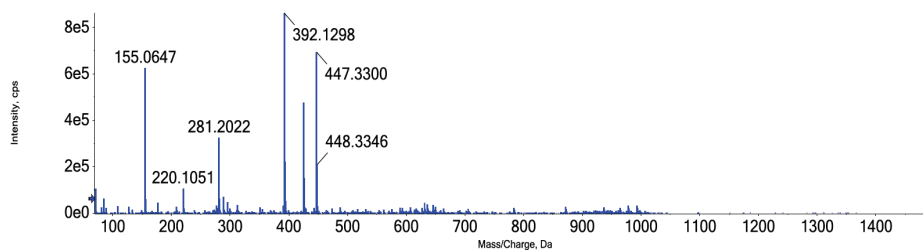
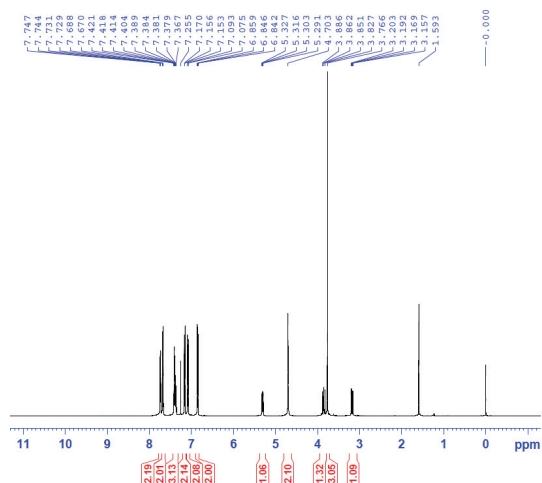
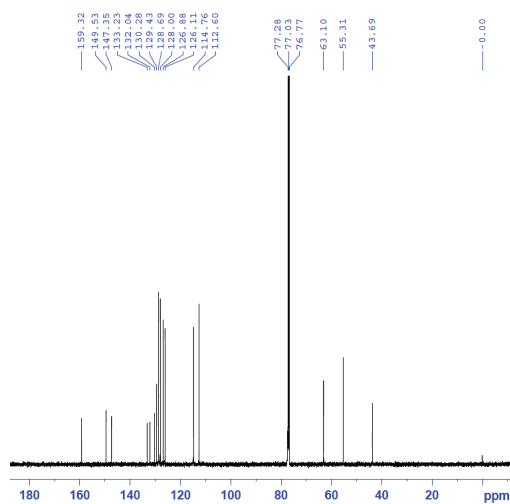
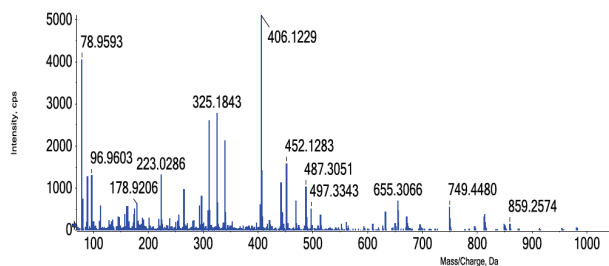


Fig. S-9. HR-MS Spectrum of compound 2c.

Fig. S-10. $^1\text{H-NMR}$ Spectrum of compound **2d** (CDCl_3).Fig. S-11. $^{13}\text{C-NMR}$ Spectrum of compound **2d** (CDCl_3).Fig. S-12. HR-MS Spectrum of compound **2d**.

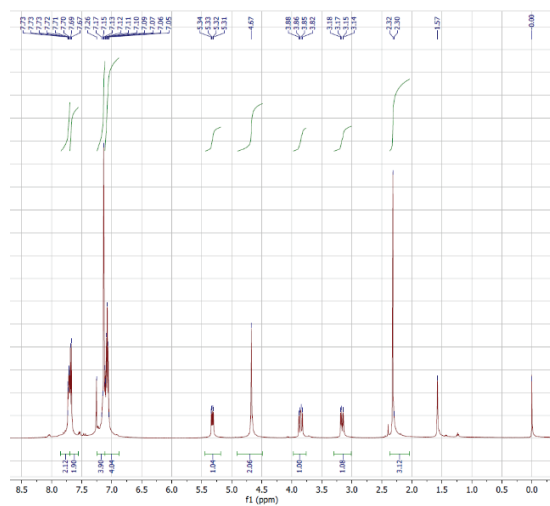


Fig. S-13. ¹H-NMR Spectrum of compound **2e** (CDCl₃).

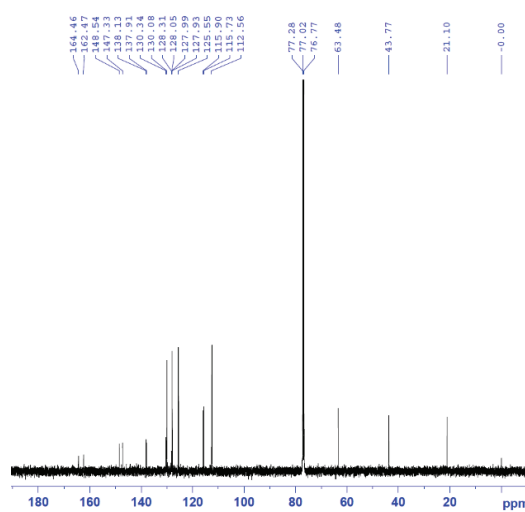


Fig. S-14. ¹³C-NMR Spectrum of compound **2e** (CDCl₃).

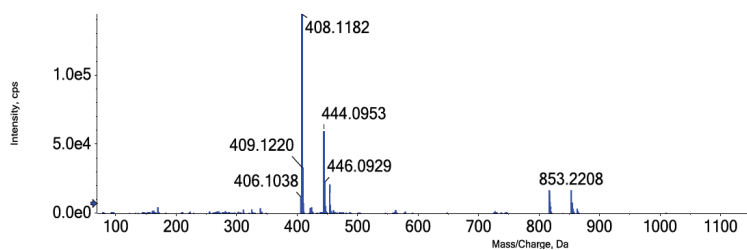
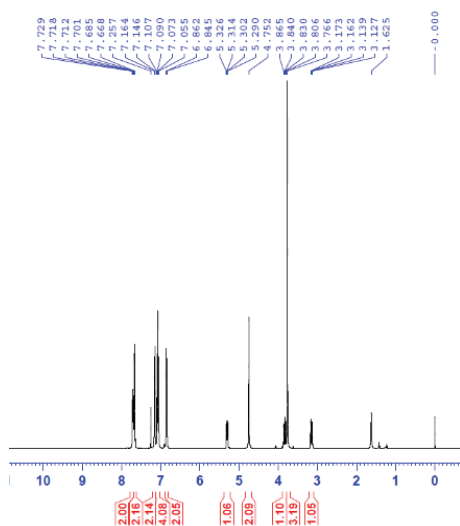
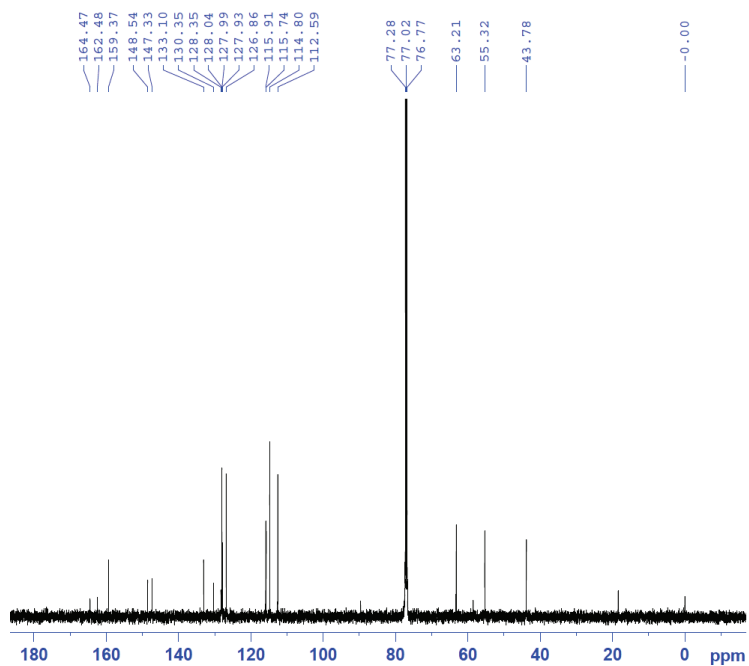
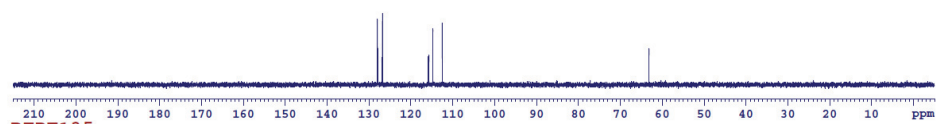


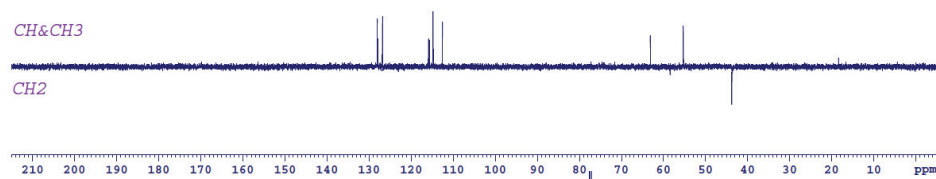
Fig. S-15. HR-MS Spectrum of compound **2e**.

Fig. S-16. ¹H-NMR Spectrum of compound **2f** (CDCl₃).Fig. S-17. ¹³C-NMR Spectrum of compound **2f** (CDCl₃).

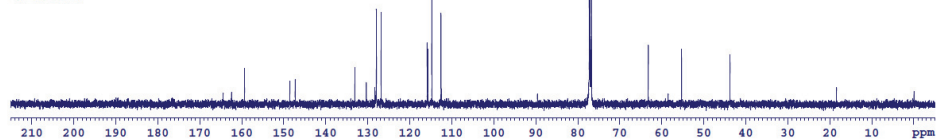
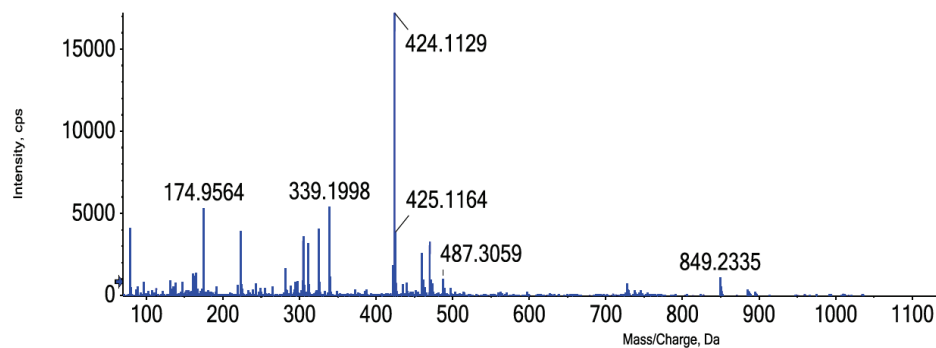
DEPT90

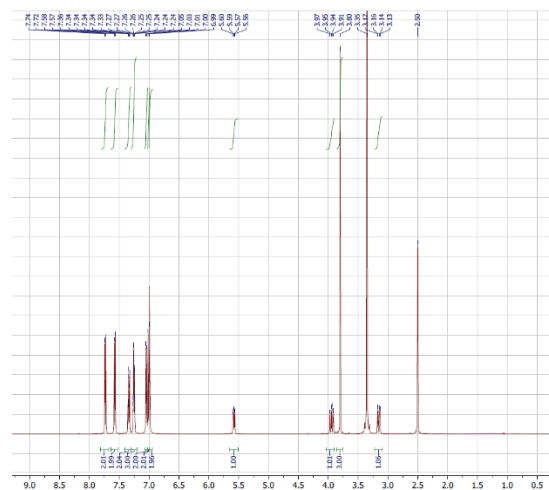
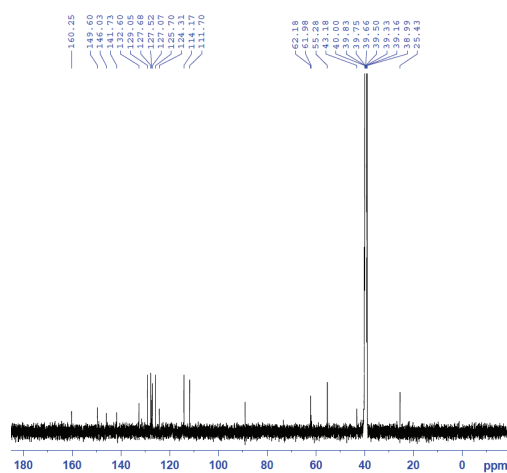
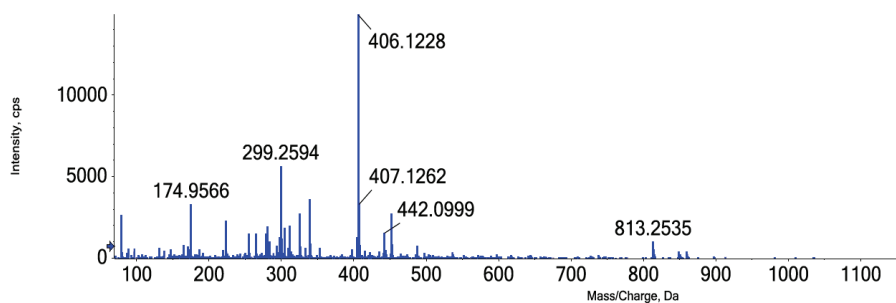


DEPT135



C13CPD

Fig. S-18. DEPT Spectra of compound **2f** (CDCl₃).Fig. S-19. HR-MS Spectrum of compound **2f**.

Fig. S-20. ¹H-NMR Spectrum of compound **2g** (DMSO).Fig. S-21. ¹³C-NMR Spectrum of compound **2g** (DMSO).Fig. S-22. HR-MS Spectrum of compound **2g**.

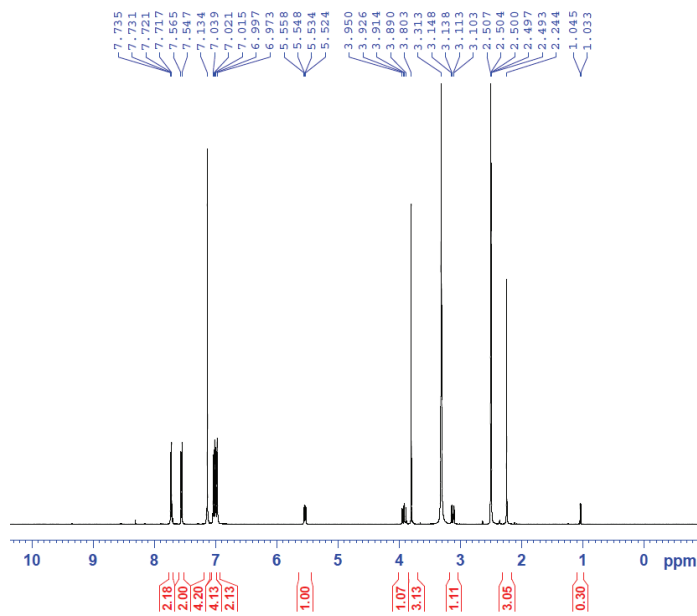


Fig. S-23. ¹H-Spectrum of compound **2h** (DMSO).

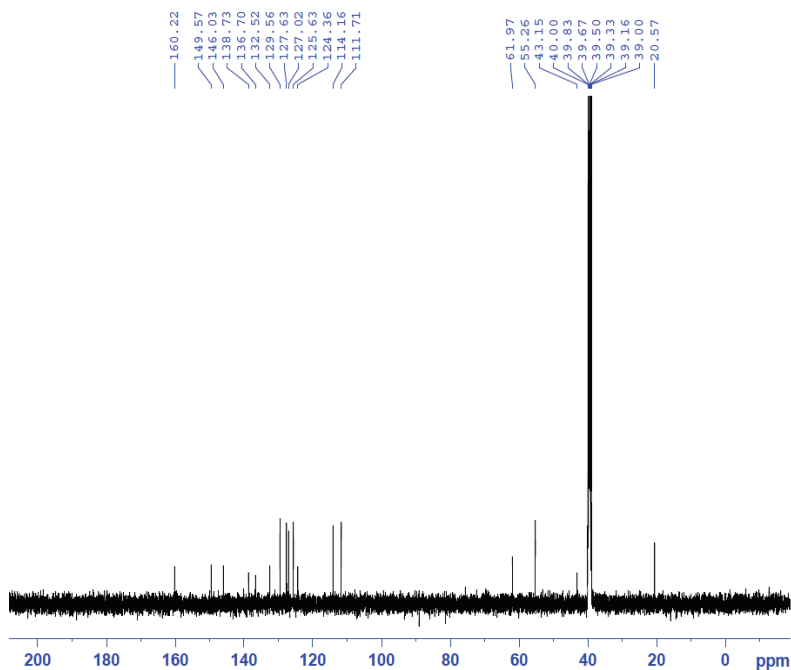
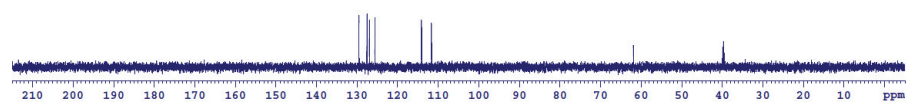


Fig. S-24. ¹³C-NMR Spectrum of compound **2h** (DMSO).

DEPT90

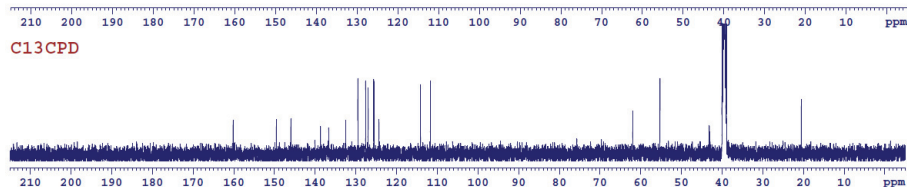


DEPT135

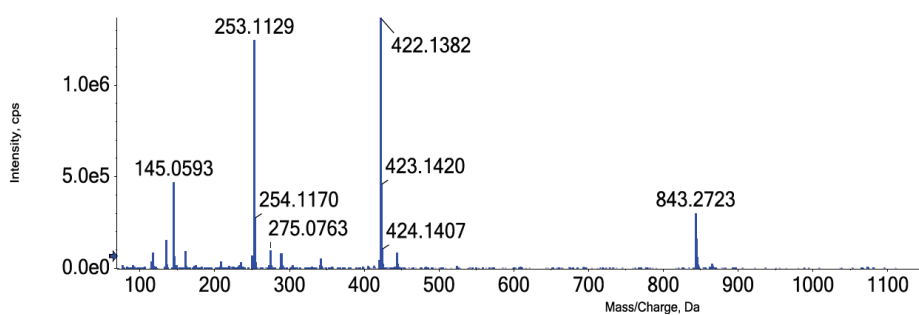


CH&CH3

CH2



C13CPD

Fig. S-24. DEPT Spectra of compound **2h** (DMSO).Fig. S-25. HR-MS spectrum of compound **2h**.

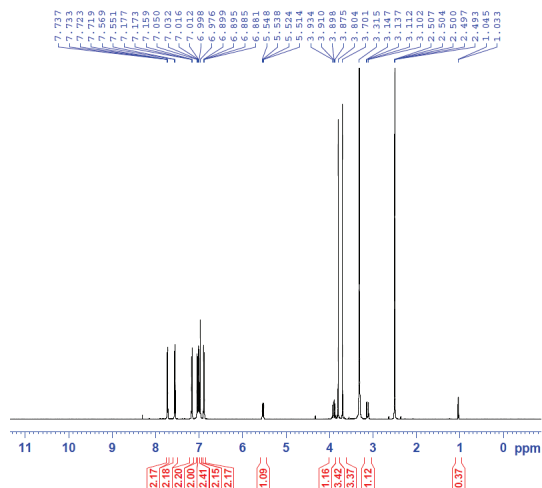


Fig. S-26. ¹H-NMR Spectrum of compound **2i** (DMSO).

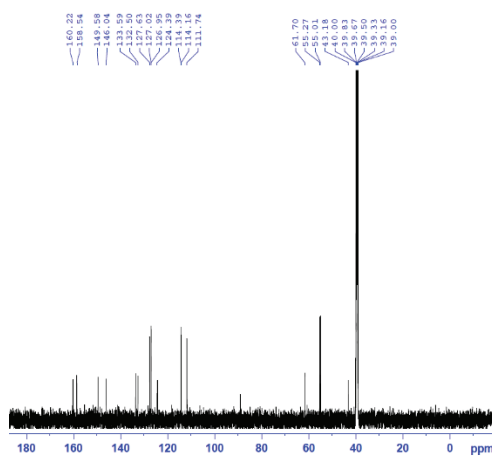


Fig. S-27. ¹³C-NMR Spectrum of compound **2i** (DMSO).

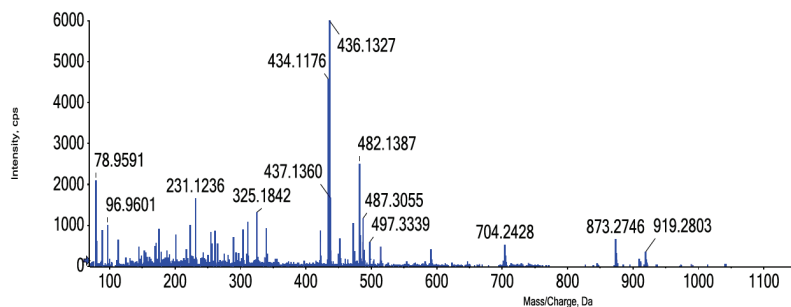
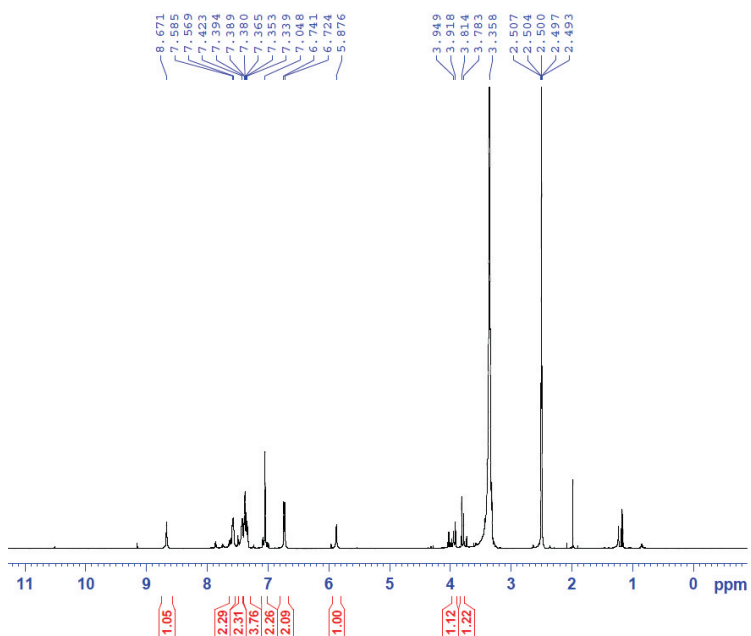
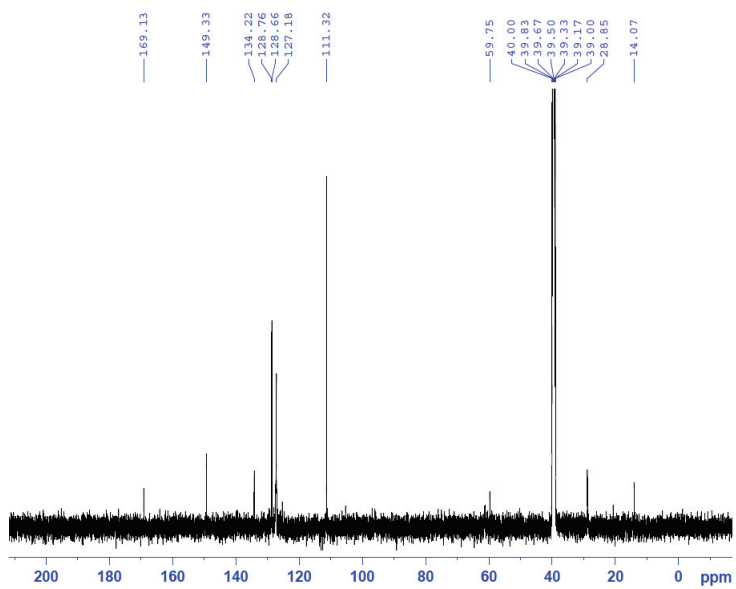


Fig. S-28. HR-MS spectrum of compound **2i**.

Fig. S-29. ¹H-NMR Spectrum of compound 4a (DMSO).Fig. S-30. ¹³C-NMR Spectrum of compound 4a (DMSO).

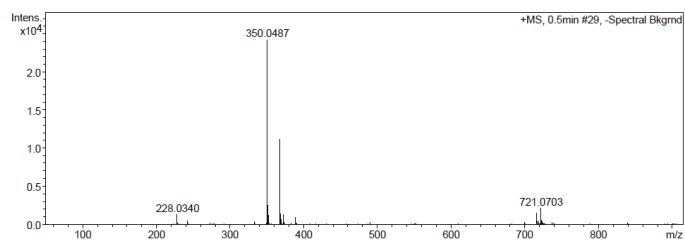


Fig. S-31. HR-MS spectrum of compound **4a**.

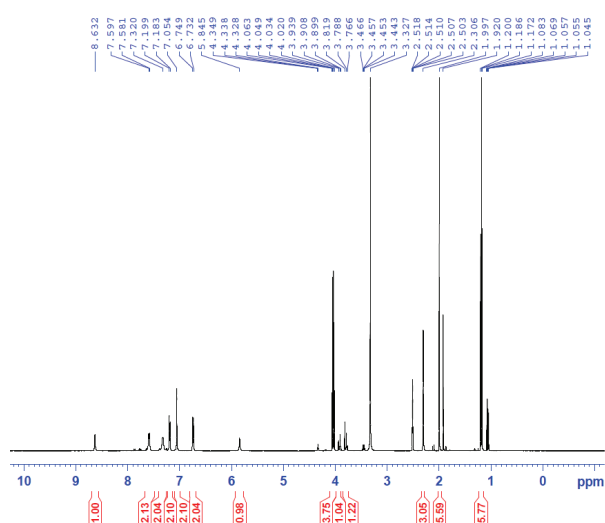


Fig. S-32. ¹H-NMR Spectrum of compound **4b** (DMSO).

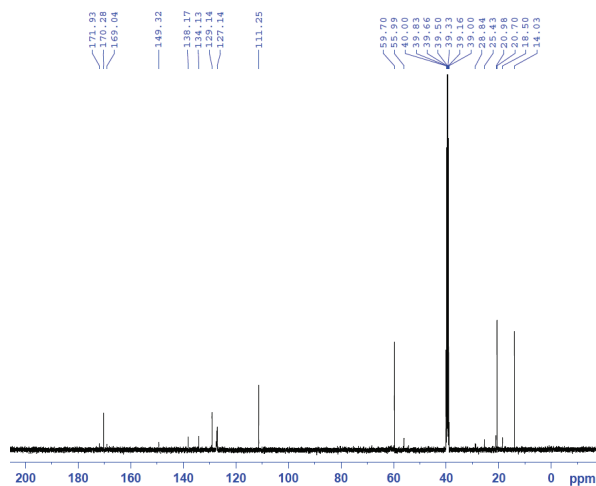
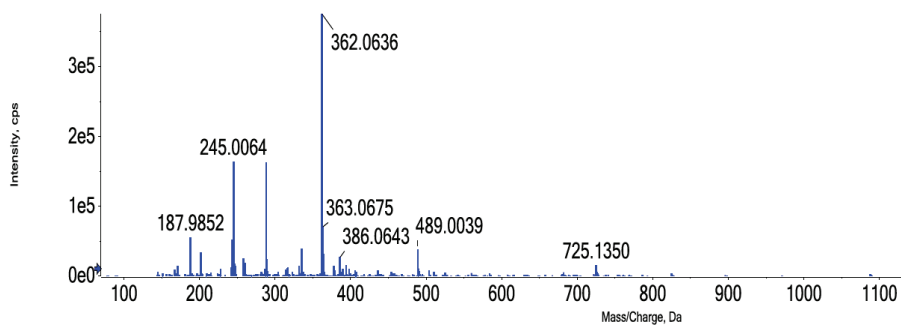
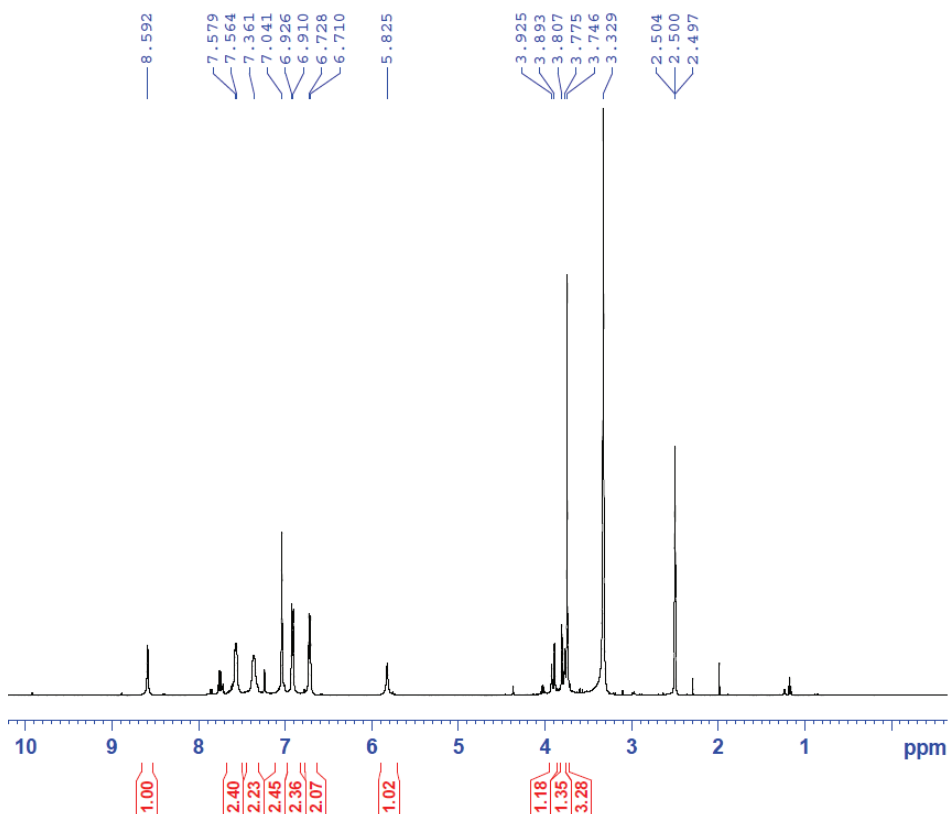


Fig. S-33. ¹³C-NMR Spectrum of compound **4b** (DMSO).

Fig. S-34. HR-MS spectrum of compound **4b**.Fig. S-35. ¹H-NMR Spectrum of compound **4c** (DMSO).

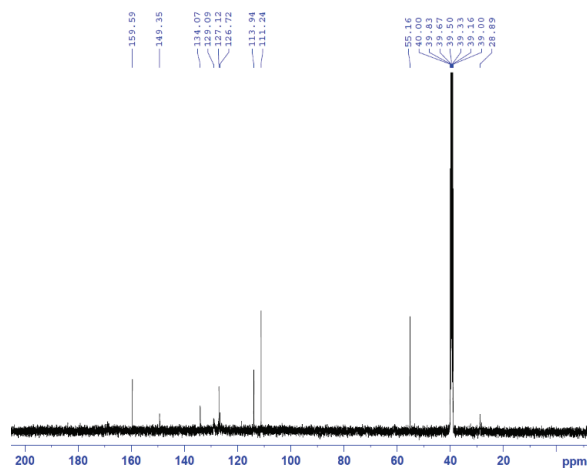


Fig. S36. ^{13}C -NMR Spectrum of compound **4c** (DMSO).

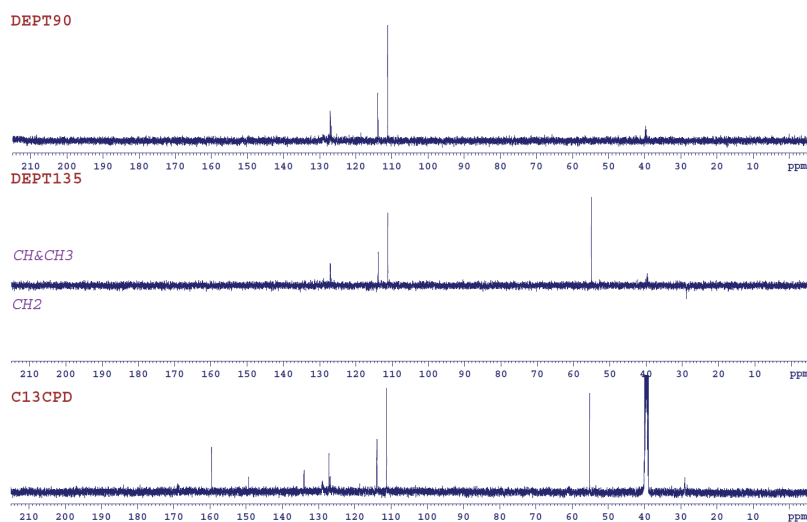


Fig. S-37. DEPT Spectra of compound **4c** (DMSO).

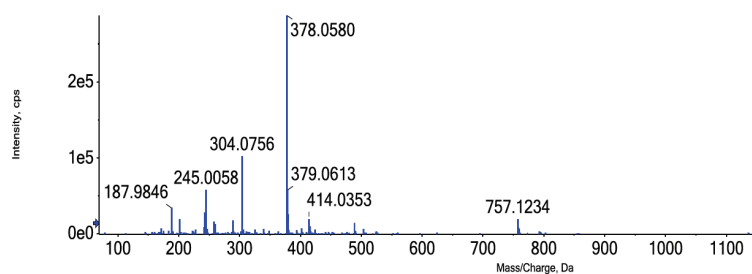
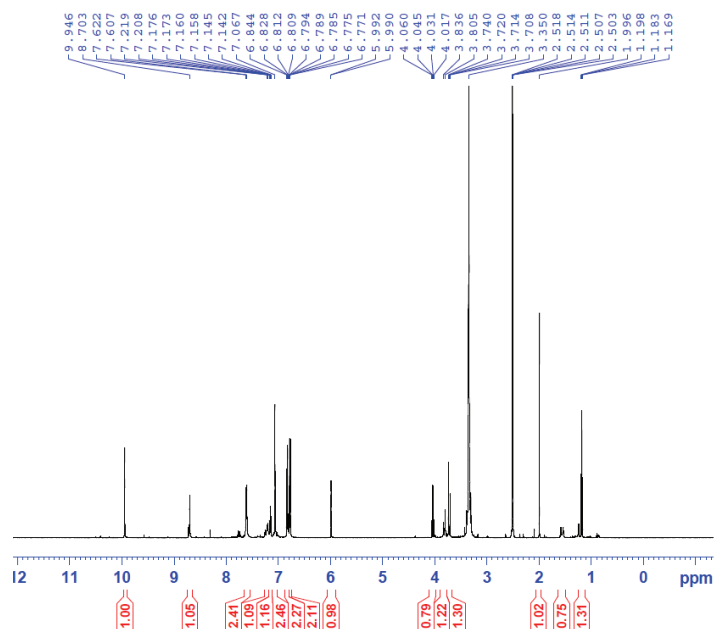
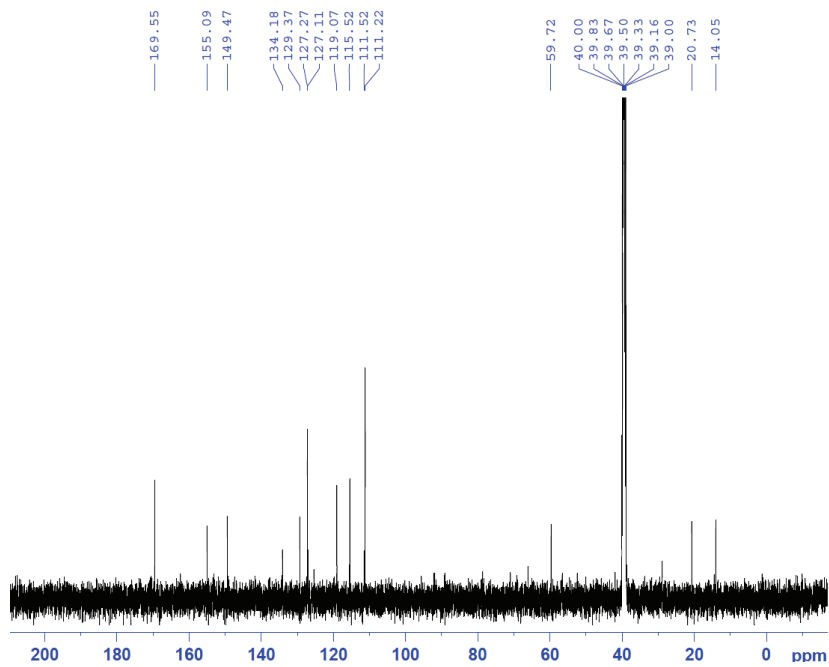
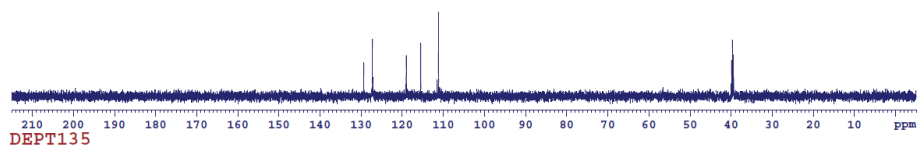


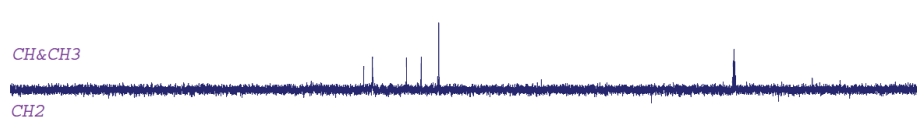
Fig. S-38. HR-MS spectrum of compound **4c**.

Fig. S-39. ¹H-NMR Spectrum of compound **4d** (DMSO).Fig. S-40. ¹³C-NMR Spectrum of compound **4d** (DMSO).

DEPT90

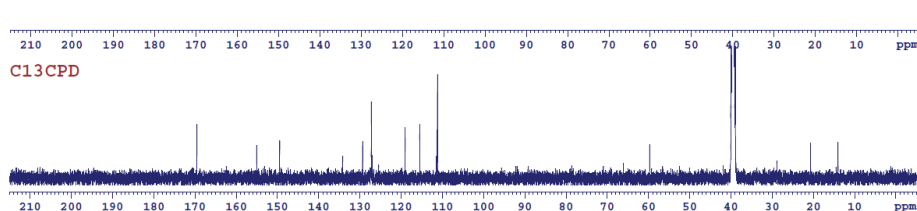


DEPT135

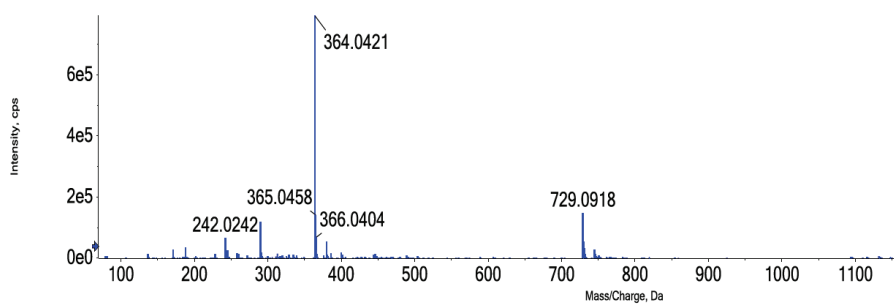


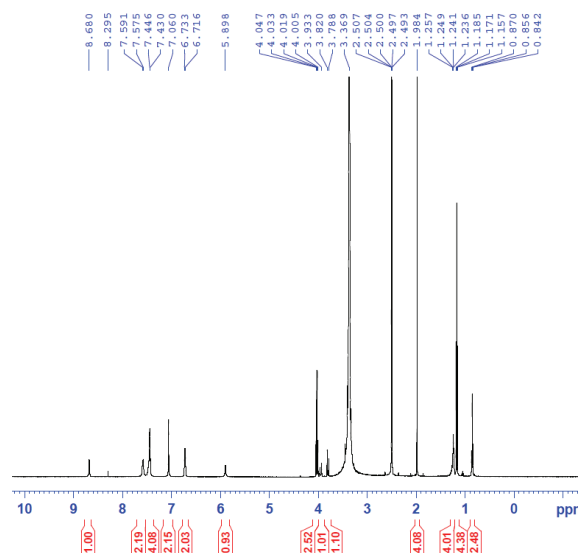
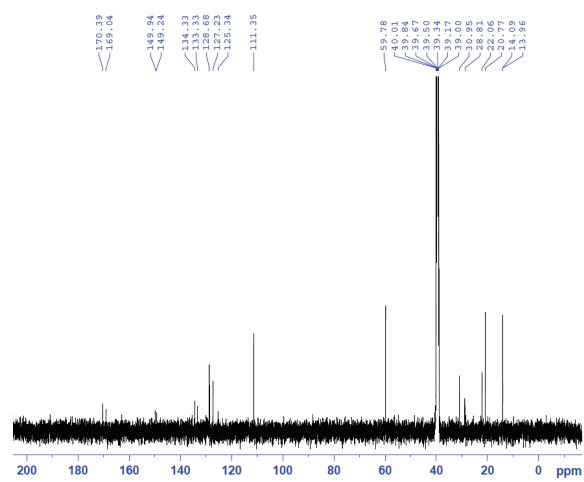
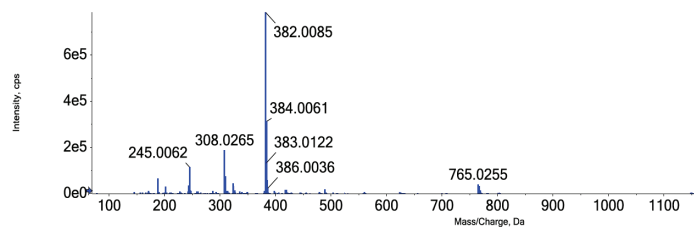
CH&CH3

CH2



C13CPD

Fig. S-41. DEPT Spectra of compound **4d** (DMSO).Fig. S-42. HR-MS spectrum of compound **4d**.

Fig. S-43. ^1H -NMR Spectrum of compound **4e** (DMSO).Fig. S-44. ^{13}C -NMR Spectrum of compound **4e** (DMSO).Fig. S-45. HR-MS spectrum of compound **4e** (DMSO).

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