Supplementary Information

Improved Method for Microwave-Assisted Synthesis of Benzodiazepine-2,5-diones from Isatoic Anhydrides Mediated by Glacial Acetic Acid

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Spectroscopic data for benzodiazepine-2,5-diones (**3**-**14**)

3,4-Dihydro-1*H*-benzo[*e*][1,4]diazepine-2,5-dione (**3**)

Pale brown solid (62% yield), mp 316-318 °C; IR (KBr) ν / cm^{-1} 3163, 3040, 1661, 1688; ^{1}H NMR (200 MHz, DMSO-d_6) δ 3.58 (d, 2H, *J* 5.6 Hz, CH_2), 7.10 (brd, 1H, *J* 8.0 Hz, H-9), 7.21 (ddd, 1H, *J* 8.0, 8.0, 1.2 Hz, H-7), 7.51 (ddd, 1H, *J* 8.0, 8.0, 1.6 Hz, H-8), 7.75 (dd, 1H, *J* 8.0, 1.6 Hz, H-6), 8.57 (t, 1H, *J* 5.6 Hz, NH-4), 10.37 (s, 1H, NH-1); ^{13}C NMR (50 MHz, DMSO-d_6) δ 49.5, 126.0, 129.0, 130.6, 135.9, 137.4, 142.2, 173.2, 176.2; EIMS *m/z* (rel. int. %): $[M]^+$ 176 (100), 147 (51), 119 (54), 92 (34). The spectroscopic data are in agreement with those reported in the literature.[1-3]

3-Methyl-3,4-dihydro-1*H*-benzo[*e*][1,4]diazepine-2,5-dione (**4**)

Pink solid (65% yield), mp 328-330 °C; IR (KBr) ν / cm^{-1} 3149, 3043, 1691, 1670; $[\alpha]^D_{20}$ = +7 (*c* 0.14, MeOH); ^{1}H NMR (200 MHz, DMSO-d_6) δ 1.22 (d, 3H, *J* 6.6 Hz, CH_3), 3.78 (qd, 1H, *J* 6.6, 5.4 Hz, CH), 7.09 (dd, 1H, *J* 8.0, 1.2 Hz, H-9), 7.17 (ddd, 1H, *J* 8.0, 8.0, 1.2 Hz, H-7), 7.51 (ddd, 1H, *J* 8.0, 8.0, 1.8 Hz, H-8), 7.73 (dd, 1H, *J* 8.0, 1.8 Hz, H-6), 8.44 (d, 1H, *J* 5.4 Hz, NH-4), 10.38 (s, 1H, NH-1); ^{13}C NMR (50 MHz, DMSO-d_6) δ 13.9, 47.3, 121.0, 124.0, 126.3, 130.5, 132.3, 136.8, 167.8, 172.3; EIMS *m/z* (rel. int. %): $[M]^+$ 190 (88), 162 (7), 147 (100), 120 (74), 92 (54). The spectroscopic data are in agreement with those reported in the literature.[1,3]

2,3-Dihydro-1*H*-benzo[*e*]pyrrolo[1,2-*a*][1,4]diazepine-5,11(10*H*,11a*H*)-dione (**5**)

Pale brown solid (68% yield), mp 198-201 °C; IR (KBr) ν / cm^{-1} 3220, 1693, 1650; ^{1}H NMR (200 MHz, $CDCl_3$) δ 2.08-2.01 (m, 3H), 2.82-2.73 (m, 1H), 3.67-3.58 (m, 1H), 3.88-3.78 (m, 1H), 4.09 (d, 1H, *J* 5.8 Hz, H-3), 7.02 (dd, 1H, *J* 8.0, 1.2 Hz, H-9), 7.28 (ddd, 1H, *J* 8.0, 8.0, 1.2 Hz, H-7), 7.49 (ddd, 1H, *J* 8.0, 8.0, 1.6 Hz, H-8), 8.01 (dd, 1H, *J* 8.0, 1.6 Hz, H-6), 8.67 (brs, 1H, NH-1); ^{13}C NMR (50 MHz, $CDCl_3$) δ 23.7, 26.5, 47.6, 56.9, 121.2, 125.4, 127.3, 131.4, 132.7, 135.4, 165.7, 171.6; EIMS *m/z* (rel. int. %): $[M]^+$ 216 (80), 187 (43), 160 (51), 146 (18), 137 (36), 119 (100), 92 (47). The spectroscopic data are in agreement with those reported in the literature.[4,5]

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3-Benzyl-3,4-dihydro-1*H*-benzo[*e*][1,4]diazepine-2,5-dione (**6**)

Pale brown solid (71% yield), mp 246-248 °C; IR (KBr) ν / cm^{-1} 3149, 3058, 1681, 1653; $[\alpha]^{D}_{20}$ = +9 (*c* 0.11, MeOH); ^{1}H NMR (200 MHz, DMSO-d_6) δ 2.78-2.95 (m, 1H), 3.14 (dd, 2H, *J* 14.0, 9.3 Hz, CH_2), 7.30-7.13 (m, 7H), 7.51 (ddd, 1H, *J* 7.8, 7.8, 1.4 Hz, H-8), 7.67 (dd, 1H, *J* 7.8, 1.2 Hz, H-6), 8.56 (d, 1H, *J* 6.2 Hz, NH-4), 10.48 (s, 1H, NH-1); ^{13}C NMR (50 MHz, DMSO-d_6) δ 38.7, 59.2, 126.4, 129.4, 131.7, 133.6, 134.8, 135.8, 137.7, 142.1, 143.3, 173.1, 176.7; EIMS *m/z* (rel. int. %): $[M]^+$ 266 (34), 223 (17), 175 (100), 148 (79), 120 (57), 92 (41). The spectroscopic data are in agreement with those reported in the literature.[1,3]

3-Isobutyl-3,4-dihydro-1*H*-benzo[*e*][1,4]diazepine-2,5-dione (**7**)

Pale yellow solid (69% yield), mp 241-243 °C; IR (KBr) ν / cm^{-1} 3154, 3063, 1682, 1653; $[\alpha]^{D}_{20}$ = +160 (*c* 0.15, MeOH); ^{1}H NMR (200 MHz, DMSO-d_6) δ 0.77 (d, 3H, *J* 6.6 Hz, CH_3), 0.85 (d, 3H, *J* 6.2 Hz, CH_3), 1.72-1.52 (m, 2H, CH_2), 3.78 (psq, 1H, *J* 7.4 Hz, CH), 7.08 (dd, 1H, *J* 8.0, 1.2 Hz, H-9), 7.22 (ddd, 1H *J* 8.0, 7.4, 1.0 Hz, H-7), 7.52 (ddd, 1H, *J* 8.0, 7.4, 1.4 Hz, H-8), 7.73 (dd, 1H, *J* 8.0, 1.8 Hz, H-6), 8.47 (d, 1H, *J* 5.8 Hz, NH-4), 10.41 (brs, 1H, NH-1); ^{13}C NMR (50 MHz, DMSO-d_6) δ 21.6, 22.9, 23.9, 36.2, 50.3, 121.0, 124.0, 126.3, 130.4, 132.3, 136.8, 167.8, 171.7; EIMS *m/z* (rel. int. %): $[M]^+$ 232 (7), 189 (11), 176 (51), 147 (100), 120 (53), 92 (33). The spectroscopic data are in agreement with those reported in the literature.[1]

3-Isopropyl-3,4-dihydro-1*H*-benzo[*e*][1,4]diazepine-2,5-dione (**8**)

Pale brown solid (61% yield), mp 208-210 °C; IR (KBr) ν / cm^{-1} 3178, 3064, 1678, 1657; $[\alpha]^{D}_{20}$ = –45 (*c* 0.11, MeOH); ^{1}H NMR (200 MHz, DMSO-d_6) δ 0.88 (d, 3H, *J* 6.6 Hz, CH_3), 0.93 (d, 3H, *J* 6.6 Hz, CH_3), 2.01-1.86 (m, 1H, CH), 3.22 (dd, 1H, *J* 10.2, 7.0 Hz, CH), 7.08 (dd, 1H, *J* 8.0, 0.8 Hz, H-9), 7.20 (ddd, 1H, *J* 7.8, 7.8, 1.2 Hz, H-7), 7.50 (ddd, 1H, *J* 8.8, 8.0, 1.4 Hz, H-8), 7.73 (dd, 1H, *J* 7.6, 2.0 Hz, H-6), 8.57 (d, 1H, *J* 7.0 Hz, NH-4), 10.37 (brs, 1H, NH-1); ^{13}C NMR (50 MHz, DMSO-d_6) δ 18.9, 19.8, 26.4, 120.8, 123.9, 126.4, 130.4, 132.3, 136.6, 167.5, 171.7; EIMS *m/z* (rel. int. %): $[M]^+$ 218 (38), 203 (7), 176 (8), 147 (100), 120 (65), 92 (43).

7-Iodo-3,4-dihydro-1*H*-benzo[*e*][1,4]diazepine-2,5-dione (**9**)

Pale yellow solid (68% yield), mp 278-280 °C; IR (KBr) ν / cm^{-1} 3058, 2925, 1723, 1671; ^{1}H NMR (200 MHz, DMSO-d_6) δ 3.61 (d, 2H, *J* 5.8 Hz, CH_2), 6.91 (d, 1H, *J* 8.5 Hz, H-9), 7.82 (dd, 1H, *J* 8.5, 2.2 Hz, H-8), 8.00 (d, 1H, *J* 2.2 Hz, H-6), 8.63 (t, 1H, *J* 5.8 Hz, NH-4), 10.43 (s, 1H, NH-1); ^{13}C NMR (50 MHz, DMSO-d_6) δ 49.4, 92.8, 128.3, 132.6, 142.1, 144.1, 145.8, 171.9, 176.0; EIMS *m/z* (rel. int. %): $[M]^+$ 302 (100), 273 (41), 245 (39), 217 (12).

7-Iodo-3-methyl-3,4-dihydro-1*H*-benzo[*e*][1,4]diazepine-2,5-dione (**10**)

Pale brown solid (70% yield), mp 318-320 °C; IR (KBr) ν / cm^{-1} 3429, 3265, 3066, 1762, 1667; $[\alpha]^{D}_{20}$ = –254 (*c* 0.13, MeOH); ^{1}H NMR (400 MHz, CD_3OD) δ 1.40 (d, *J* 6.8 Hz, 3H), 3.94 (c, *J* 6.8 Hz, 1H), 6.90 (d, *J* 8.5, Hz, 1H), 7.84 (dd, *J* 8.5, 2.1 Hz, 1H), 8.14 (d, *J* 2.1 Hz, 1H); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.49 (d, 3H, *J* 6.8 Hz, CH_3), 3.92 (cd, 1H, *J* 6.8, 5.2 Hz, CH), 6.10 (d, 1H, *J* 4.1 Hz, NH-4),

6.74 (d, 1H, *J* 8.4 Hz, H-9), 7.79 (dd, 1H, *J* 8.4, 2.1 Hz, H-8), 7.85 (s, 1H, NH-1), 8.29 (d, 1H, *J* 2.1 Hz, H-6); ^{13}C NMR (100 MHz, CD_3OD) δ 12.5, 47.7, 86.9, 122.8, 127.7, 136.5, 138.9, 141.3, 168.0, 172.6; EIMS *m/z* (rel. int. %): $[M]^+$ 316 (100), 273 (93), 245 (50), 218 (18), 111 (14).

7-Iodo-2,3-dihydro-1*H*-benzo[*e*]pyrrolo[1,2-*a*][1,4]diazepine-5,11(10*H*,11a*H*)-dione (**11**)

Pale brown solid (69% yield), mp 208-210 °C; IR (KBr) ν / cm^{-1} 3216, 3129, 3053, 2926, 1689, 1620; $[\alpha]^D_{20}$ = –3 (*c* 0.13, MeOH); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.04-2.01 (m, 3H, CH_3), 2.78-2.75 (m, 1H, CH), 3.64-3.57 (m, 1H, CH), 3.82-3.78 (m, 1H, CH), 4.05 (d, 1H, *J* 6.2 Hz, H-3), 6.70 (d, 1H, *J* 8.4 Hz, H-9), 7.63 (s, 1H, NH-1), 7.75 (dd, 1H, *J* 8.4, 2.1 Hz, H-8), 8.83 (d, 1H, *J* 2.1 Hz, H-6); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.5, 26.3, 47.5, 56.7, 88.7, 122.7, 130.9, 134.8, 139.9, 141.2, 163.8, 170.6; EIMS *m/z* (rel. int. %): $[M]^+$ 342 (100), 313 (34), 286 (36), 245 (32), 218 (7), 217 (11), 70 (68).

3-Benzyl-7-iodo-3,4-dihydro-1*H*-benzo[*e*][1,4]diazepine-2,5-dione (**12**)

Pale brown solid (65% yield), mp 146-148 °C; IR (KBr) ν / cm^{-1} 3200, 3060, 2925 1686, 1652; $[\alpha]^D_{20}$ = –71 (*c* 0.14, MeOH); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.05 (dd, 2H, *J* 14.6, 8.5 Hz, CH_2), 3.42 (dd, 2H, *J* 14.6, 5.9 Hz, CH_2), 3.94 (dc, 1H, *J* 8.4, 5.8 Hz, CH), 6.77 (d, 1H, *J* 8.5 Hz, H-9), 7.26-7.16 (m, 5H, Ph), 7.69 (dd, 1H, *J* 8.5, 2.1 Hz, H-8), 8.04 (d, 1H, *J* 5.5 Hz, NH-4), 8.10 (d, 1H, *J* 2.1 Hz, H-6), 9.86 (s, 1H, NH-1); ^{13}C NMR (100 MHz, $CDCl_3$) δ 33.3, 59.4, 87.5, 122.2, 126.1, 126.3, 127.8 (2C), 128.4 (2C), 134.7, 135.4, 138.6, 140.8, 167.1, 170.7; EIMS *m/z* (rel. int. %): $[M]^+$ 392 (16), 301 (71), 274 (54), 207 (39), 91 (100).

7-Iodo-3-isobutyl-3,4-dihydro-1*H*-benzo[*e*][1,4]diazepine-2,5-dione (**13**)

Pale yellow solid (70% yield), mp 190-192 °C; IR (KBr) ν / cm^{-1} 3210, 3063, 2955, 1686, 1653; $[\alpha]^D_{20}$ = –191 (*c* 0.11, MeOH); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.90 (d, 3H, *J* 6.4 Hz, CH_3), 0.97 (d, 3H, *J* 6.5 Hz, CH_3), 1.67-1.60 (m, 1H, CH), 1.85-1.77 (m, 1H, CH), 1.92-1.87 (m, 1H, CH), 3.78 (dc, 1H, *J* 8.8, 5.5 Hz, H-3), 6.28 (d, 1H, *J* 5.3 Hz, NH-4), 6.76 (d, 1H, *J* 8.4 Hz, H-9), 7.80 (dd, 1H, *J* 8.4, 2.1 Hz, H-8), 8.05 (s, 1H, NH-1), 8.29 (d, 1H, *J* 2.1 Hz, H-6); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.8, 22.9, 24.4, 37.2, 50.5, 88.7, 122.6, 127.3, 135.4, 140.0, 141.9, 167.1, 171.3; EIMS *m/z* (rel. int. %): $[M]^+$ 358 (31), 315 (9), 302 (62), 273 (100), 276 (31), 218 (10), 146 (7), 86 (18).

7-Iodo-3-isopropyl-3,4-dihydro-1*H*-benzo[*e*][1,4]diazepine-2,5-dione (**14**)

Pale brown solid (69% yield), mp 256-258 °C; IR (KBr) ν / cm^{-1} 3199, 3058, 2960, 2926, 1682, 1651; $[\alpha]^D_{20}$ = –9 (*c* 0.11, MeOH); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.06 (d, 3H, *J* 6.6 Hz, CH_3), 1.09 (d, 3H, *J* 6.7 Hz, CH_3), 2.24 (oct, 1H, *J* 6.9 Hz, CH), 3.46 (dd, 1H, *J* 7.2, 6.9 Hz, H-3), 6.56 (d, 1H, *J* 5.6 Hz, NH-4), 6.76 (d, 1H, *J* 8.4 Hz, H-9), 7.79 (dd, 1H, *J* 8.4, 2.0 Hz, H-8), 8.22 (s, 1H, NH-1), 8.28 (d, 1H, *J* 2.0 Hz, H-6); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.4, 19.9, 27.0, 49.8, 88.5, 122.5, 127.4, 135.4, 139.9, 141.8, 167.1, 170.8; EIMS *m/z* (rel. int. %): $[M]^+$ 344 (51), 302 (5), 273 (100), 246 (26), 218 (10).

References

1. Jadidi, K.; Aryan, R.; Mehrdad, M.; Lugger, T.; Hahn, E.; Weng, S.; *J. Mol. Struct.* **2004**, *692*, 37.
2. Boojamra, C.; Burow, K.; Thompson, L.; Ellman, J.; *J. Org. Chem.* **1997**, *62*, 1240.
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5. Nain, S.; Arya, M.; Gupta, V.; Sirohi, R.; Kishore, D.; *Int. J. Chem. Sci.* **2009**, *7*, 1255.

^{1}H NMR and ^{13}C NMR spectra of benzodiazepine-2,5-diones (**3**-**14**)

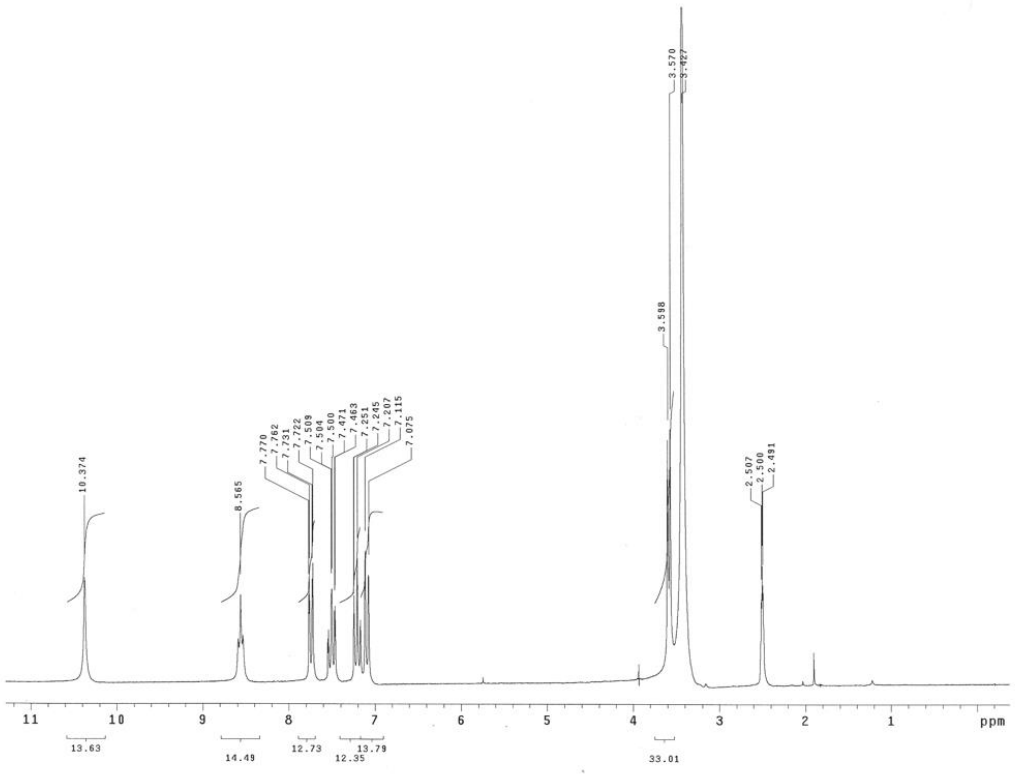

Figure S1. ^{1}H NMR spectrum (200 MHz, DMSO-d_6) of compound **3**.

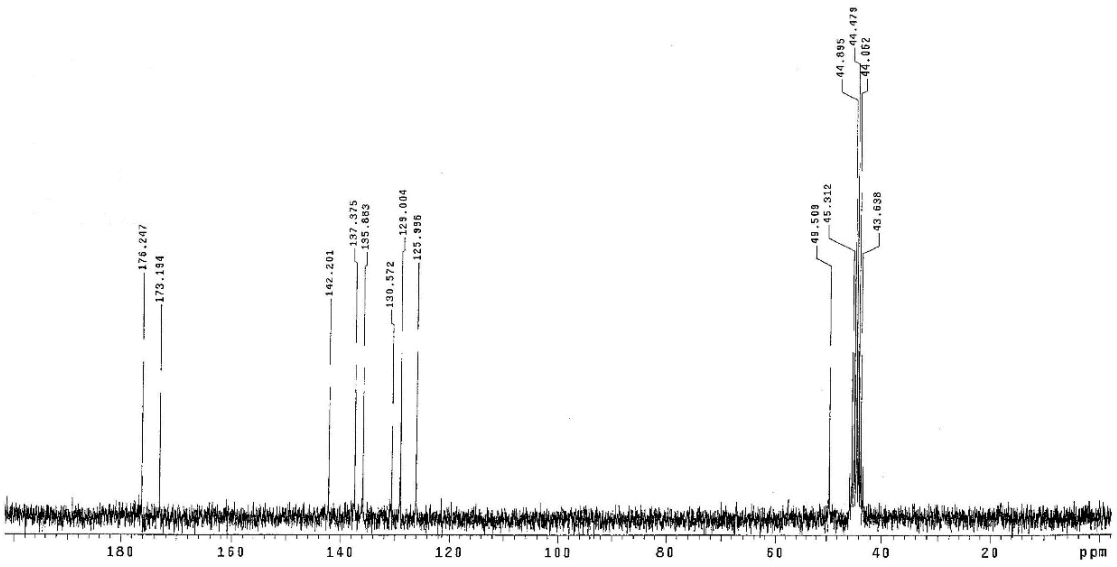

Figure S2. ^{13}C NMR spectrum (50 MHz, DMSO-d_6) of compound **3**.

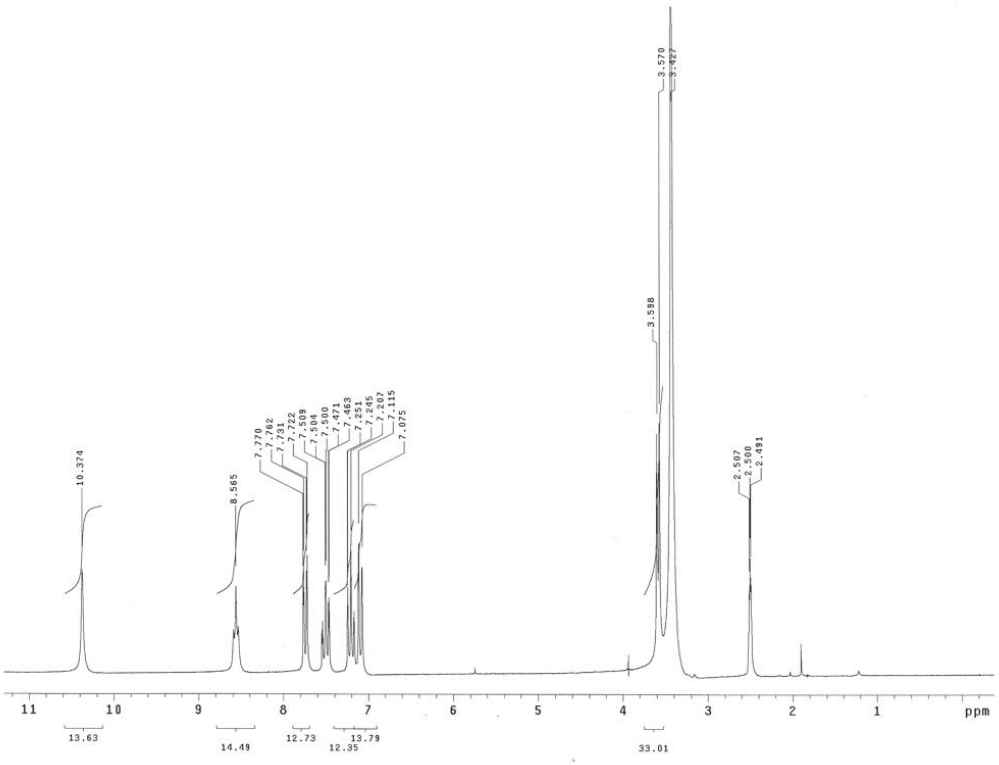

Figure S3. ^{1}H NMR spectrum (200 MHz, DMSO-d_6) of compound **4**.

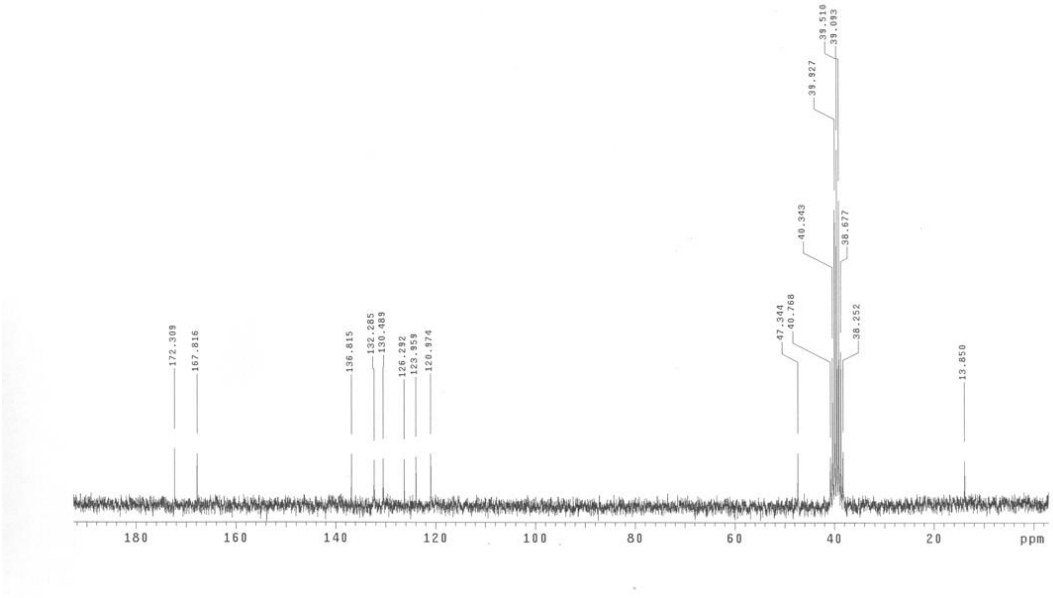

Figure S4. ^{13}C NMR spectrum (50 MHz, DMSO-d_6) of compound **4**.

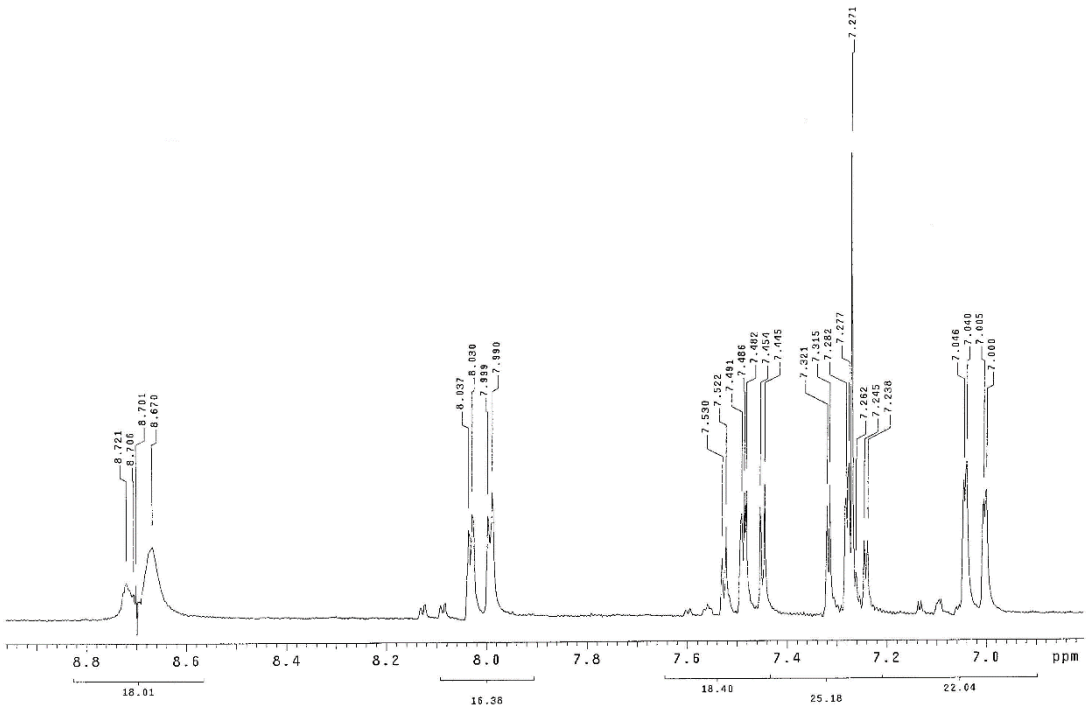

Figure S5. ^{1}H NMR spectrum (200 MHz, $CDCl_3$) of compound **5**.

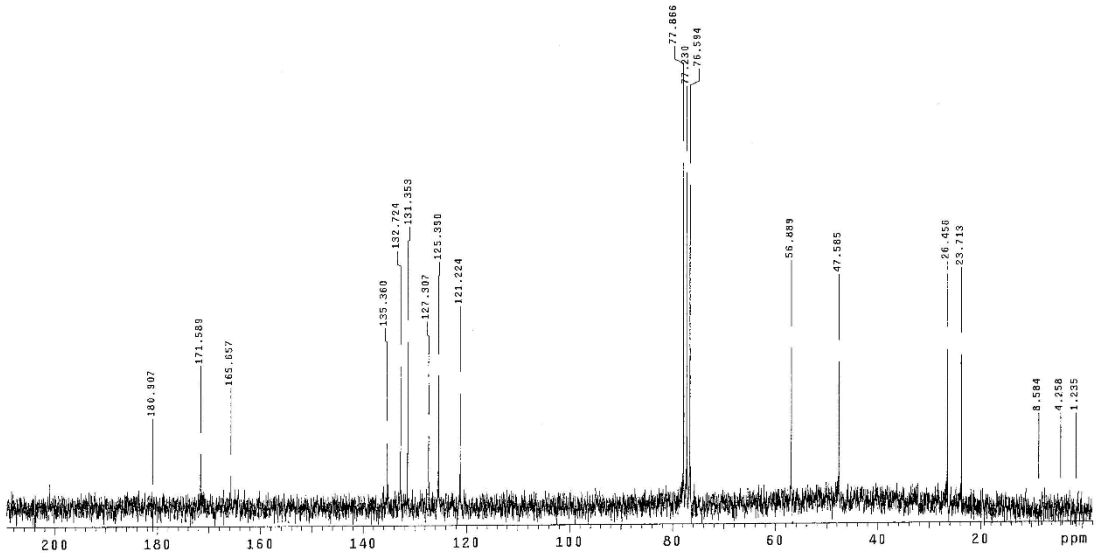

Figure S6. ^{13}C NMR spectrum (50 MHz, $CDCl_3$) of compound **5**.

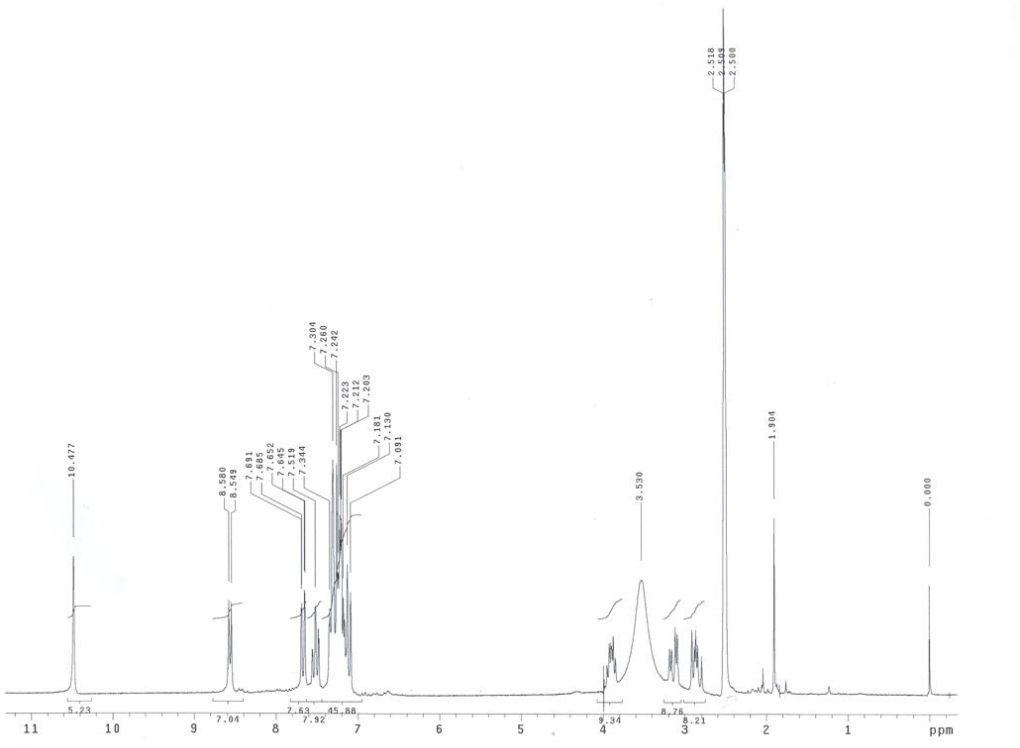

Figure S7. ^{1}H NMR spectrum (200 MHz, DMSO-d_6) of compound **4**.

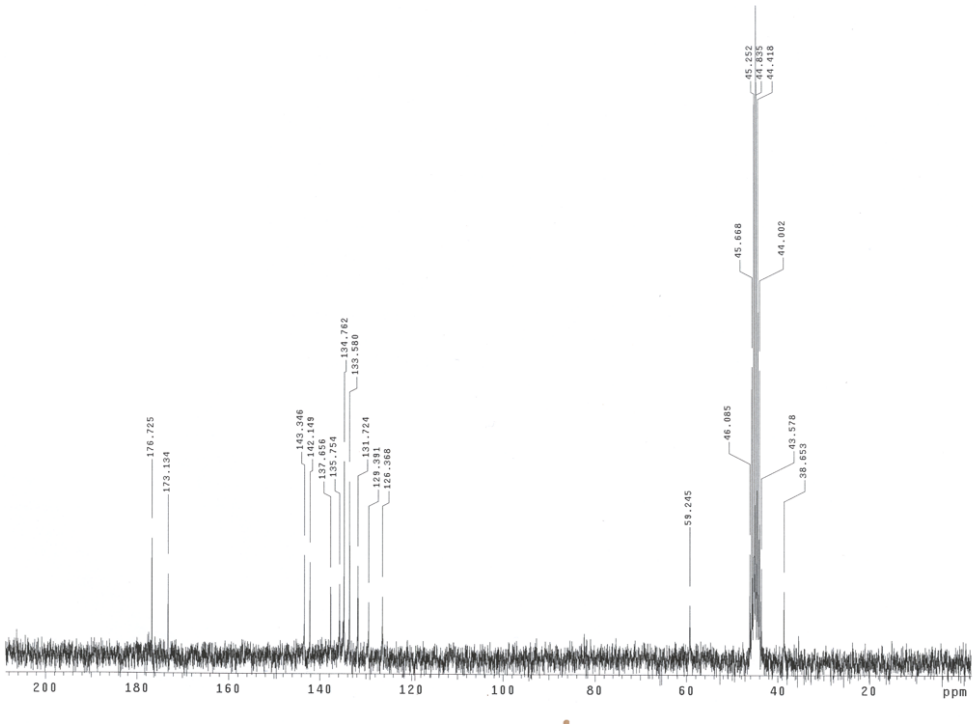

Figure S8. ^{13}C NMR spectrum (50 MHz, DMSO-d_6) of compound **6**.

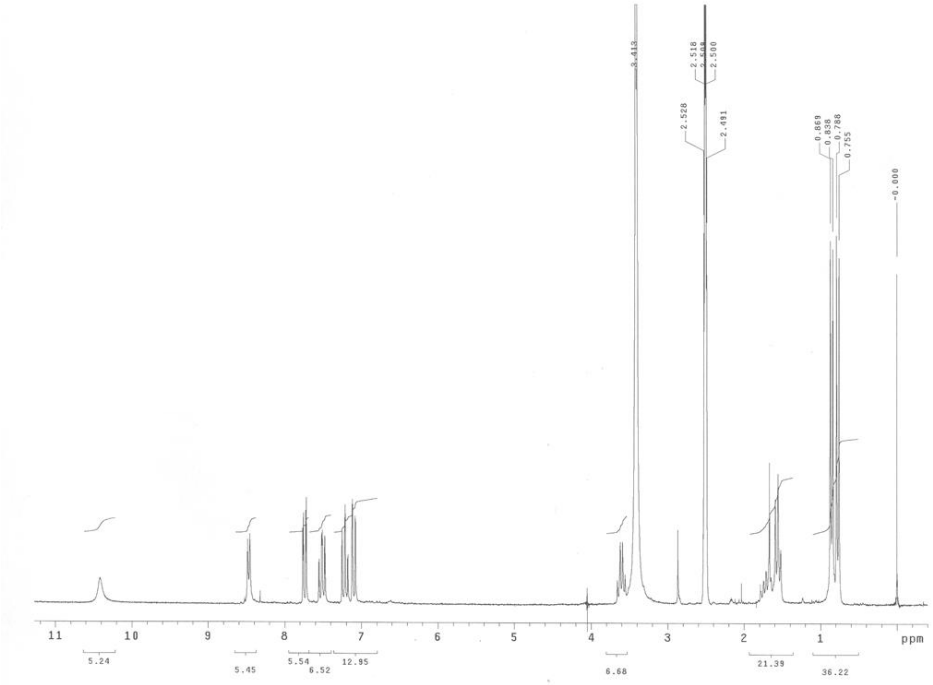

Figure S9. ^{1}H NMR spectrum (200 MHz, DMSO-d_6) of compound **7**.

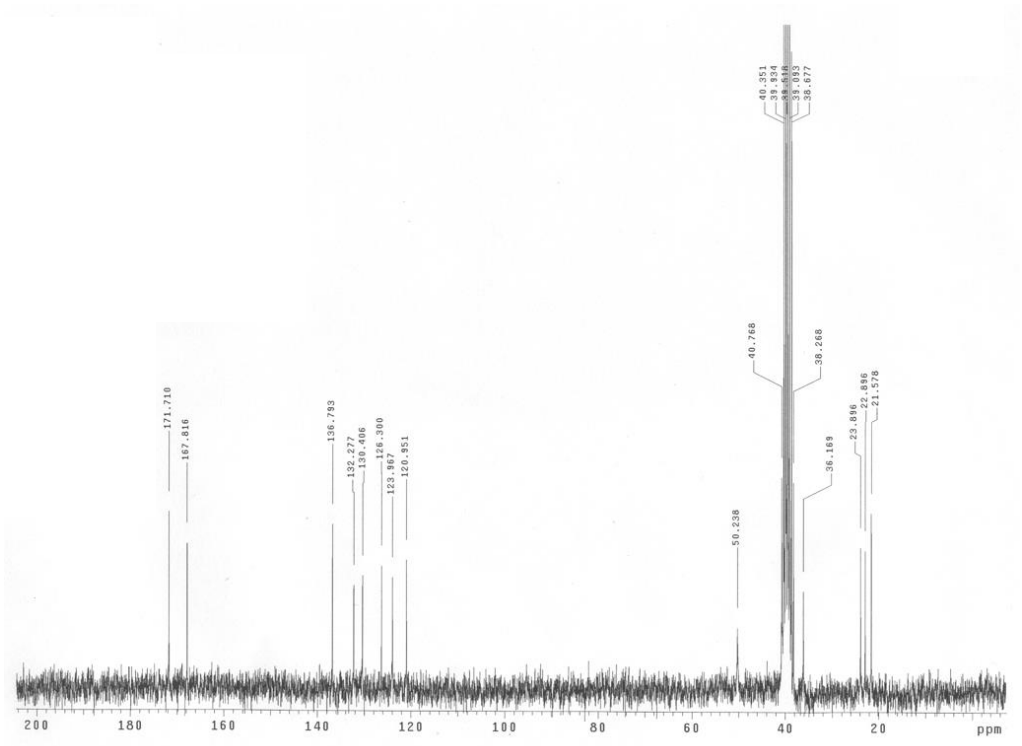

Figure S10. ^{13}C NMR spectrum (50 MHz, DMSO-d_6) of compound **7**.

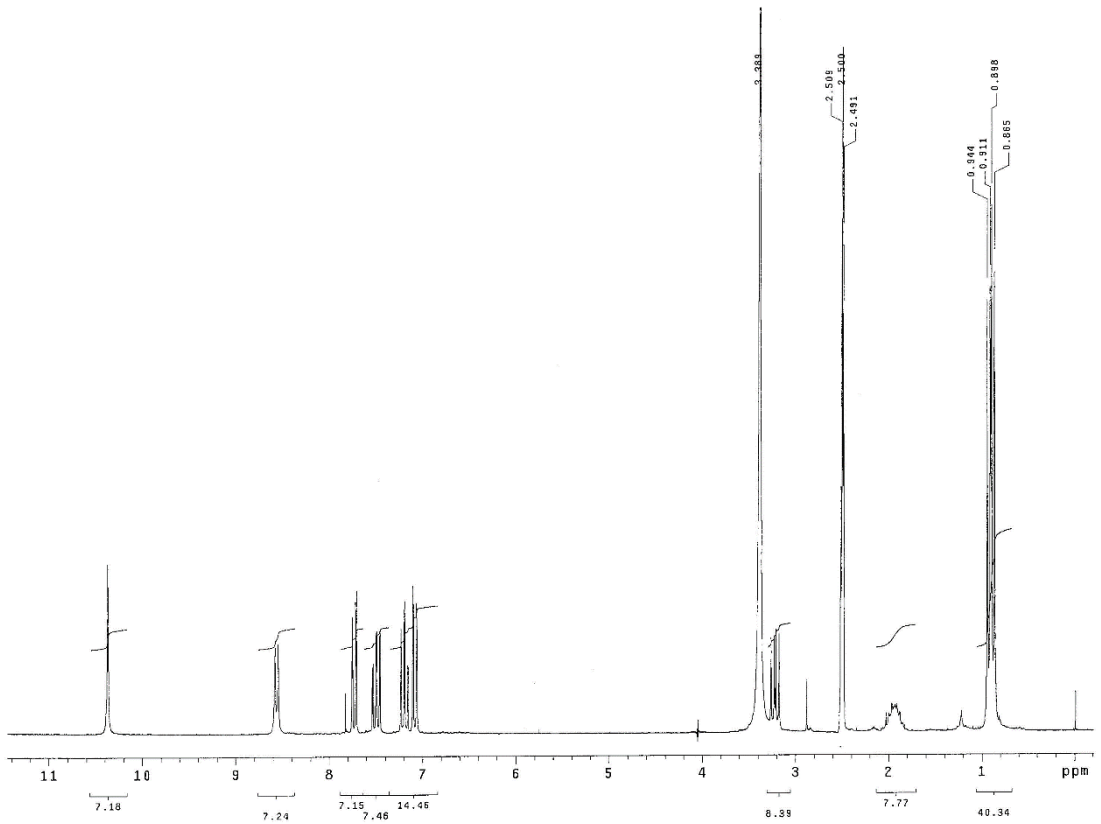

Figure S11. ^{1}H NMR spectrum (200 MHz, DMSO-d_6) of compound **8**.

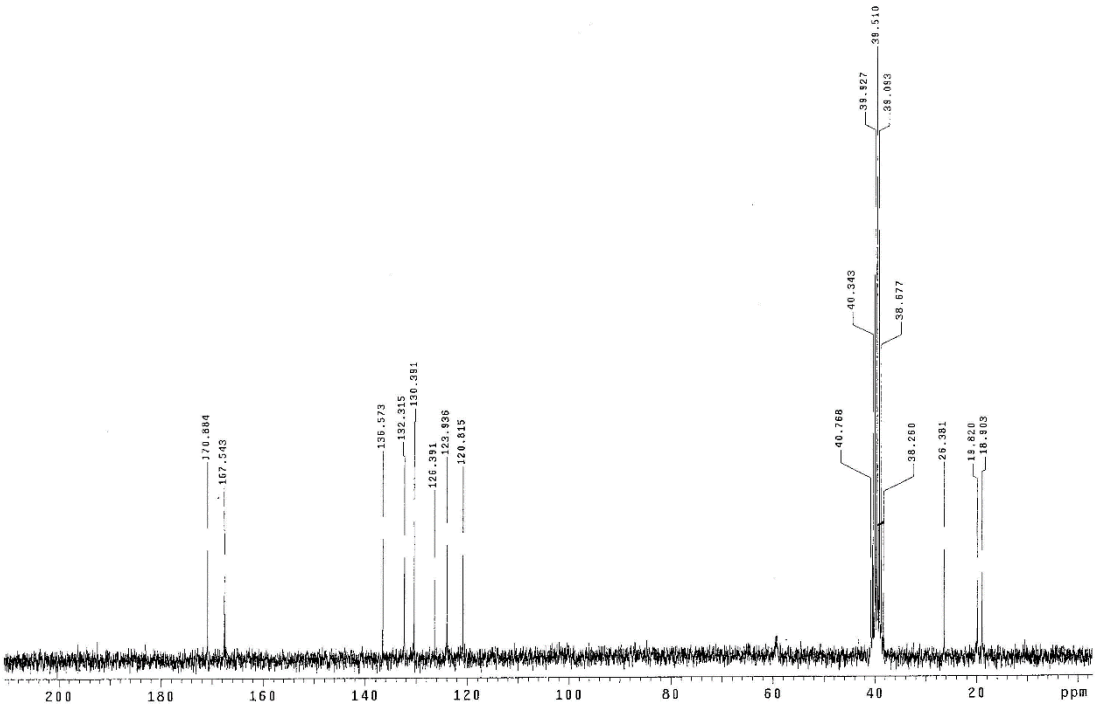

Figure S12. ^{13}C NMR spectrum (50 MHz, DMSO-d_6) of compound **8**.

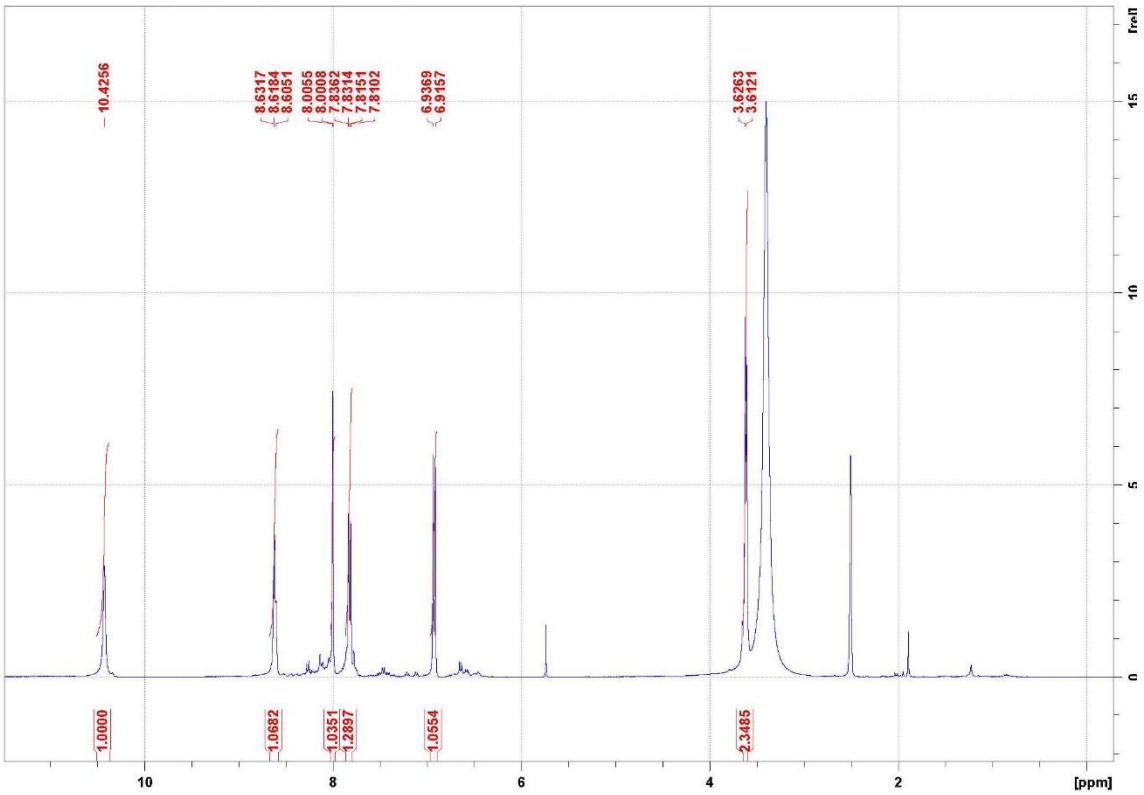

Figure S13. ^{1}H NMR spectrum (400 MHz, DMSO-d_6) of compound **9**.

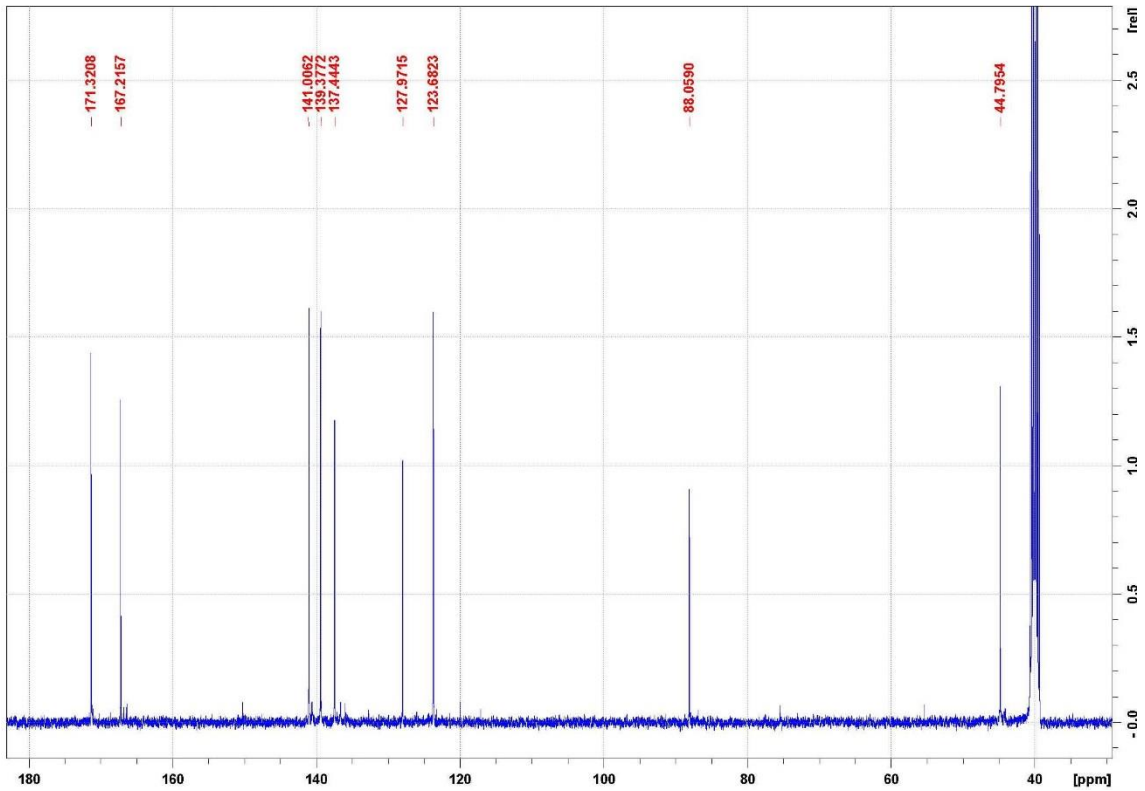

Figure S14. ^{13}C NMR spectrum (100 MHz, DMSO-d_6) of compound **9**.

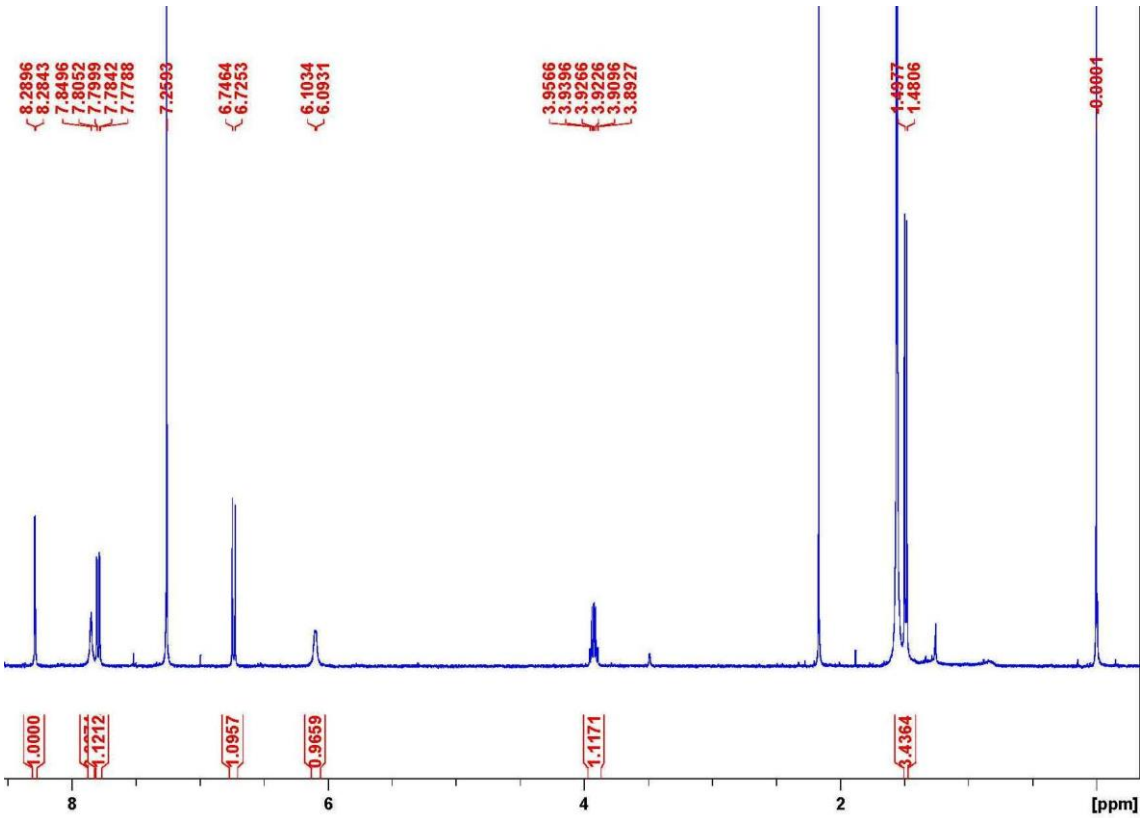

Figure S15. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of compound **10**.

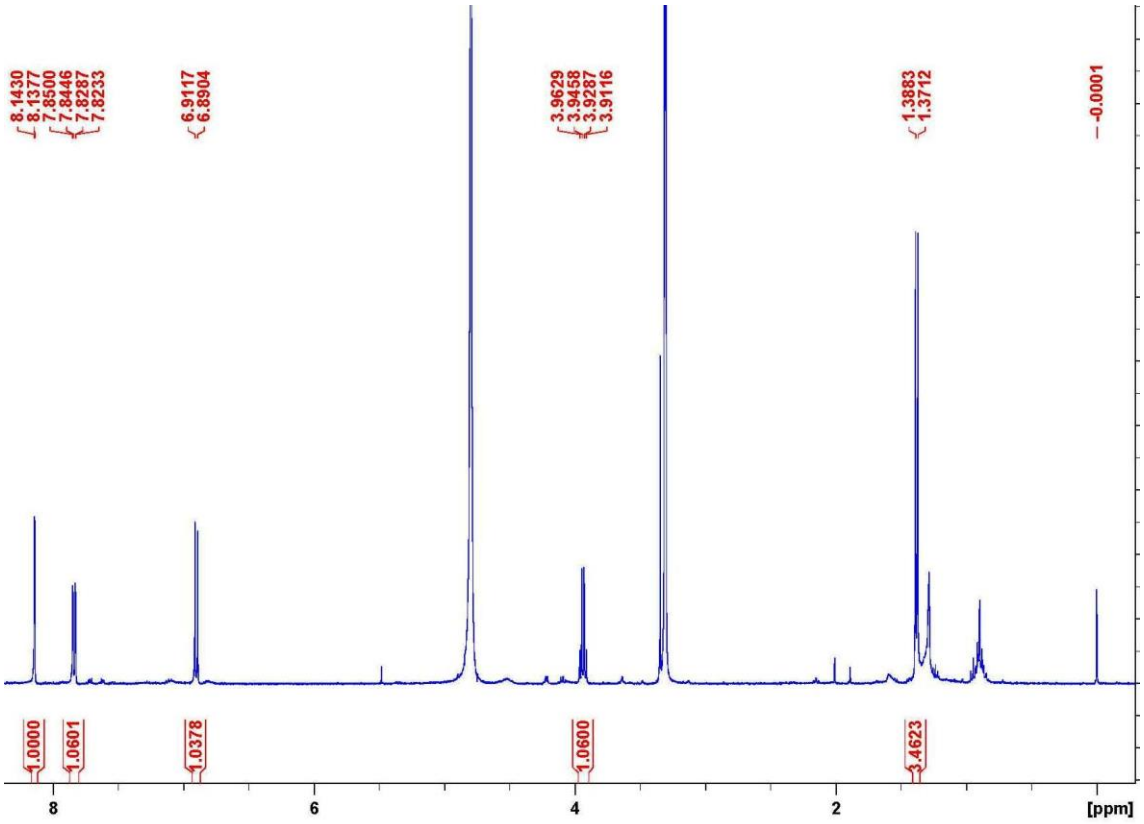

Figure S16. ^{1}H NMR spectrum (400 MHz, CD_3OD) of compound **10**.

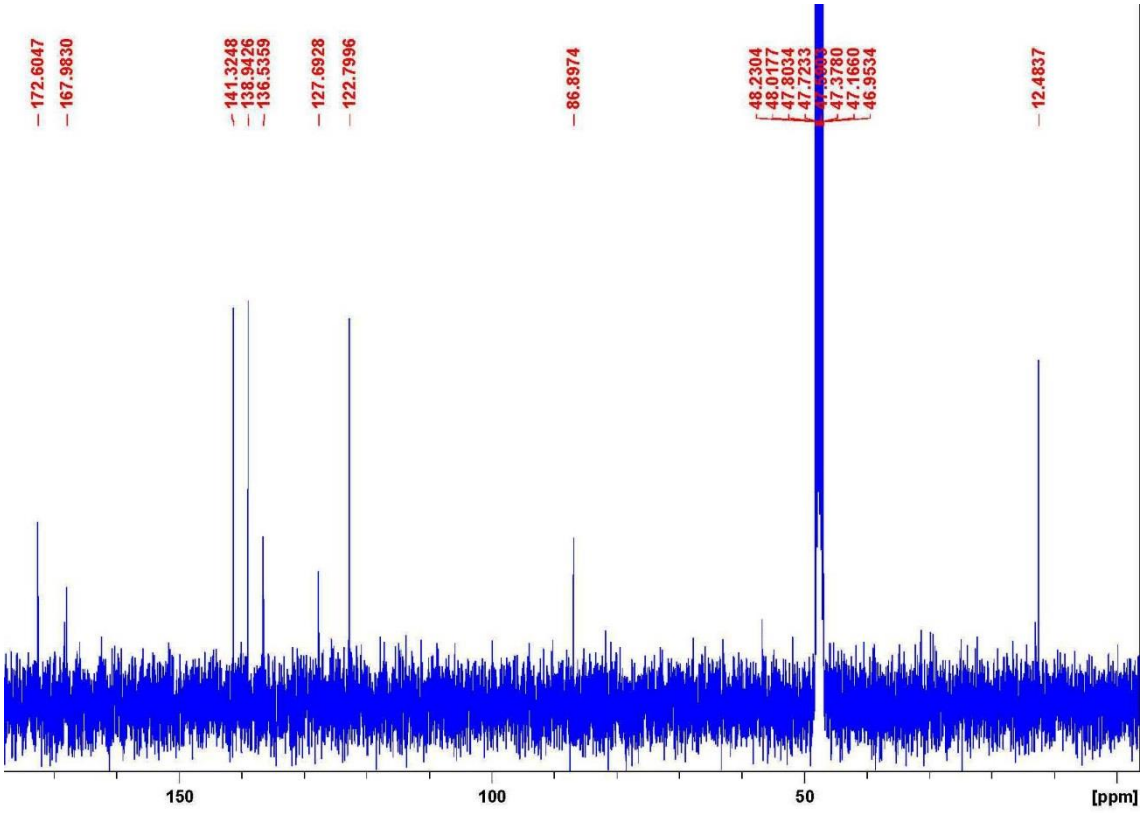

Figure S17. ^{13}C NMR spectrum (100 MHz, CD_3OD) of compound **11**.

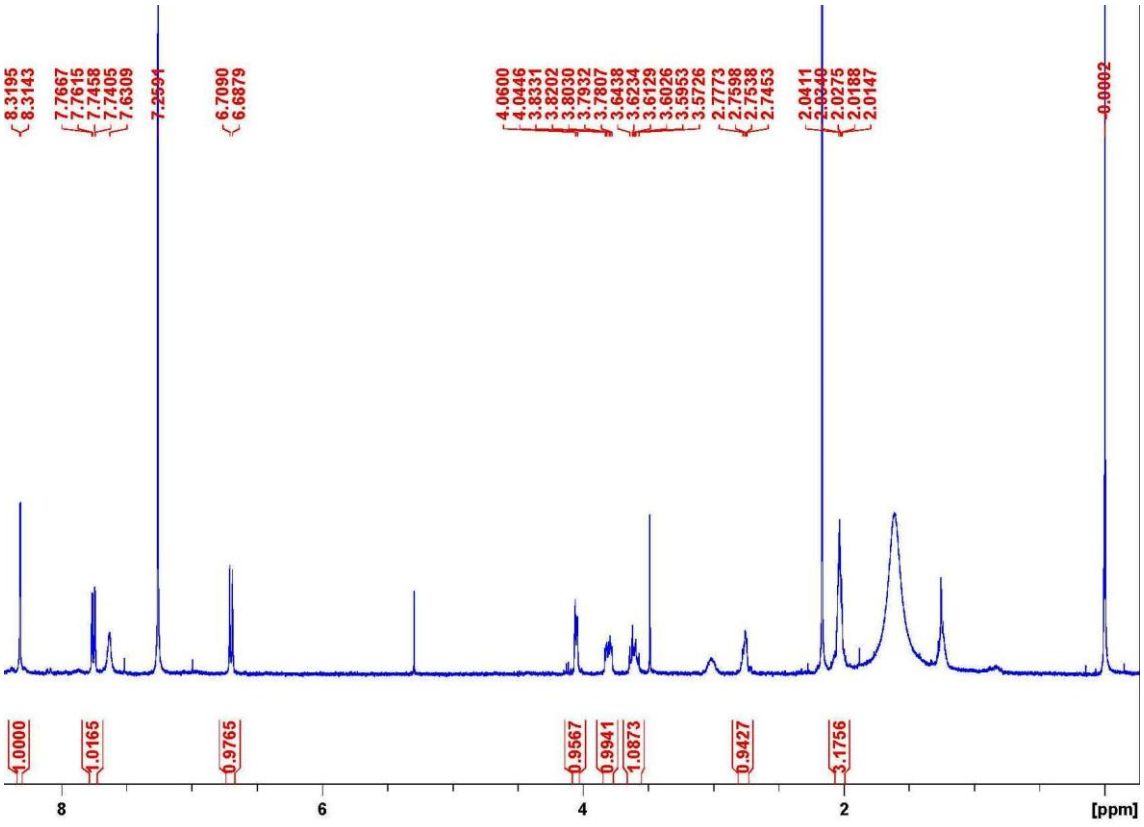

Figure S18. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of compound **11**.

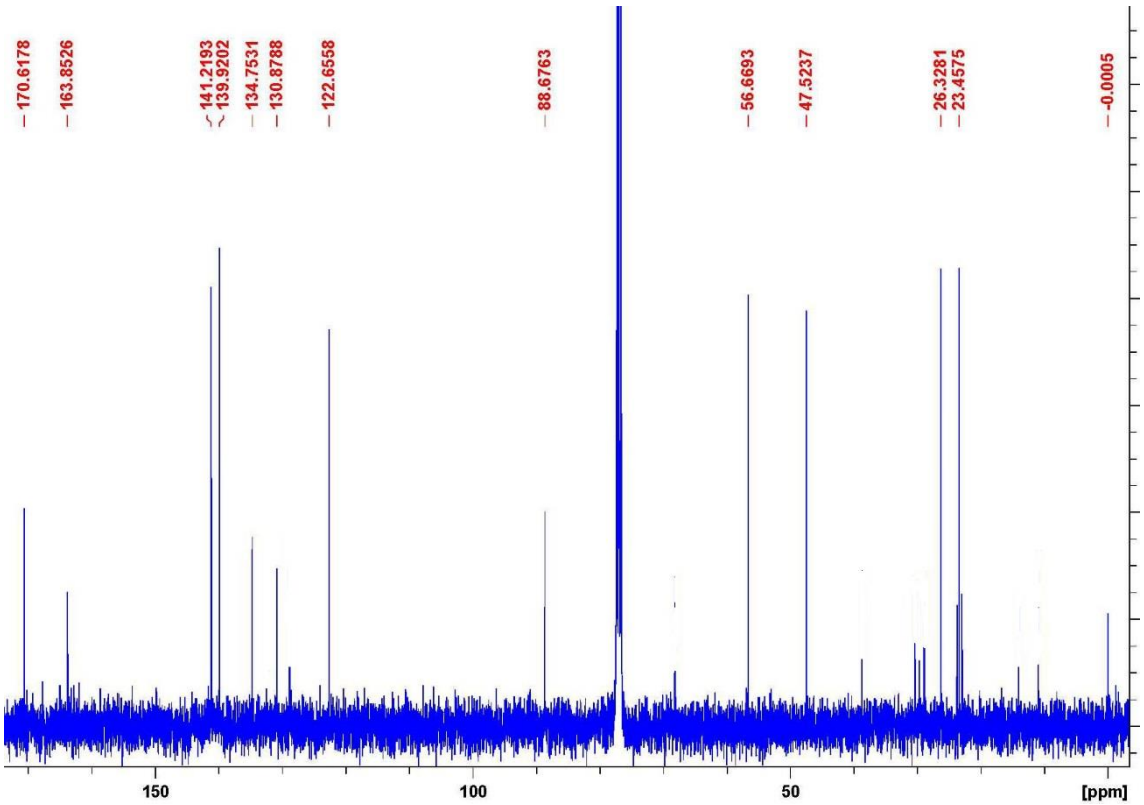

Figure S19. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of compound **11**.

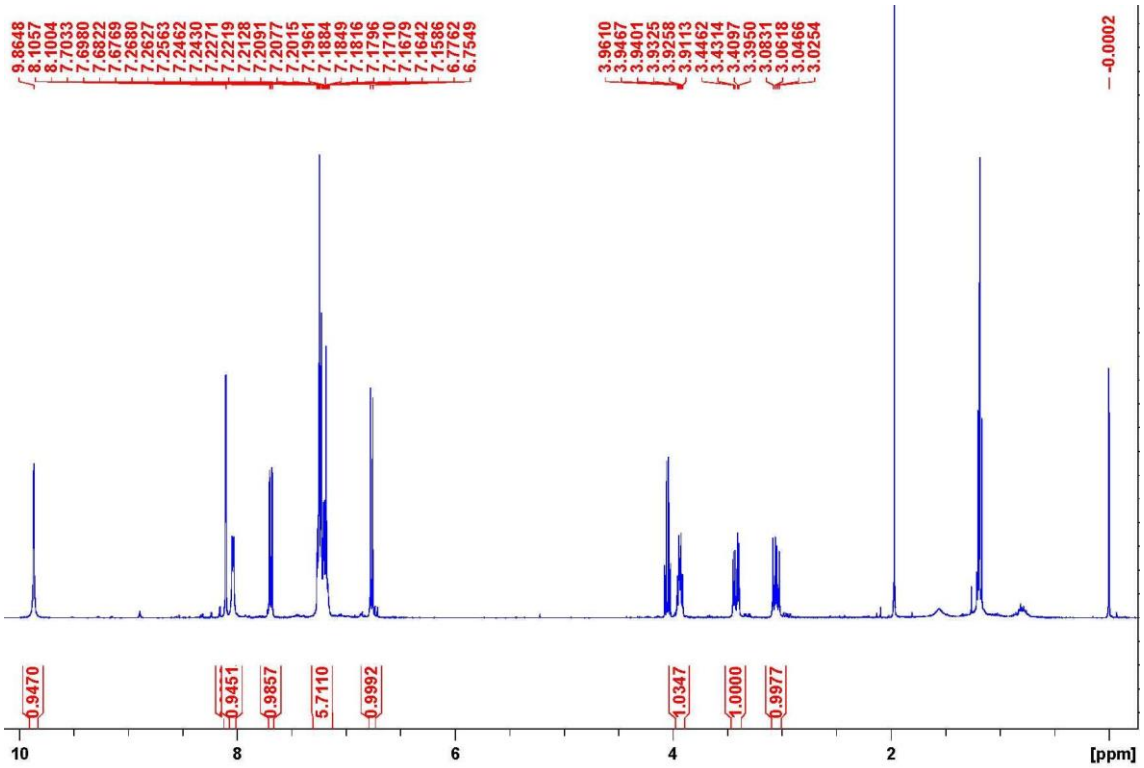

Figure S20. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of compound **12**.

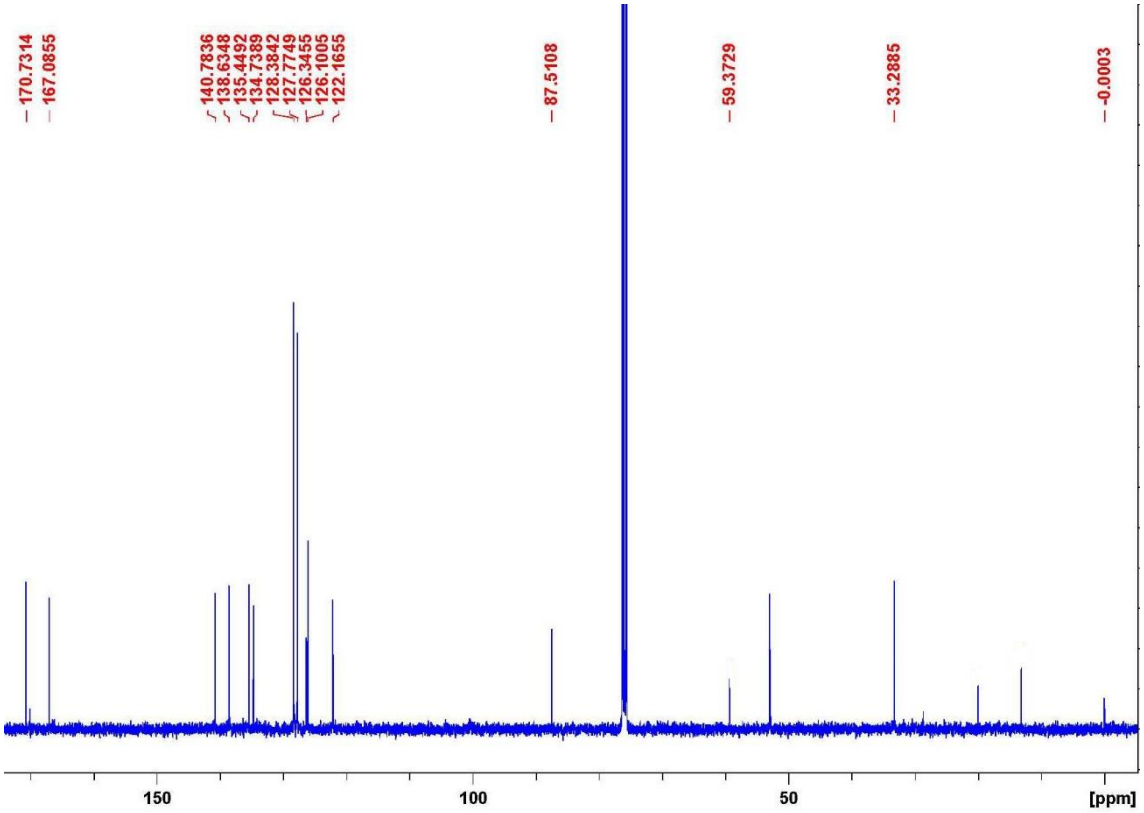

Figure S21. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of compound **12**.

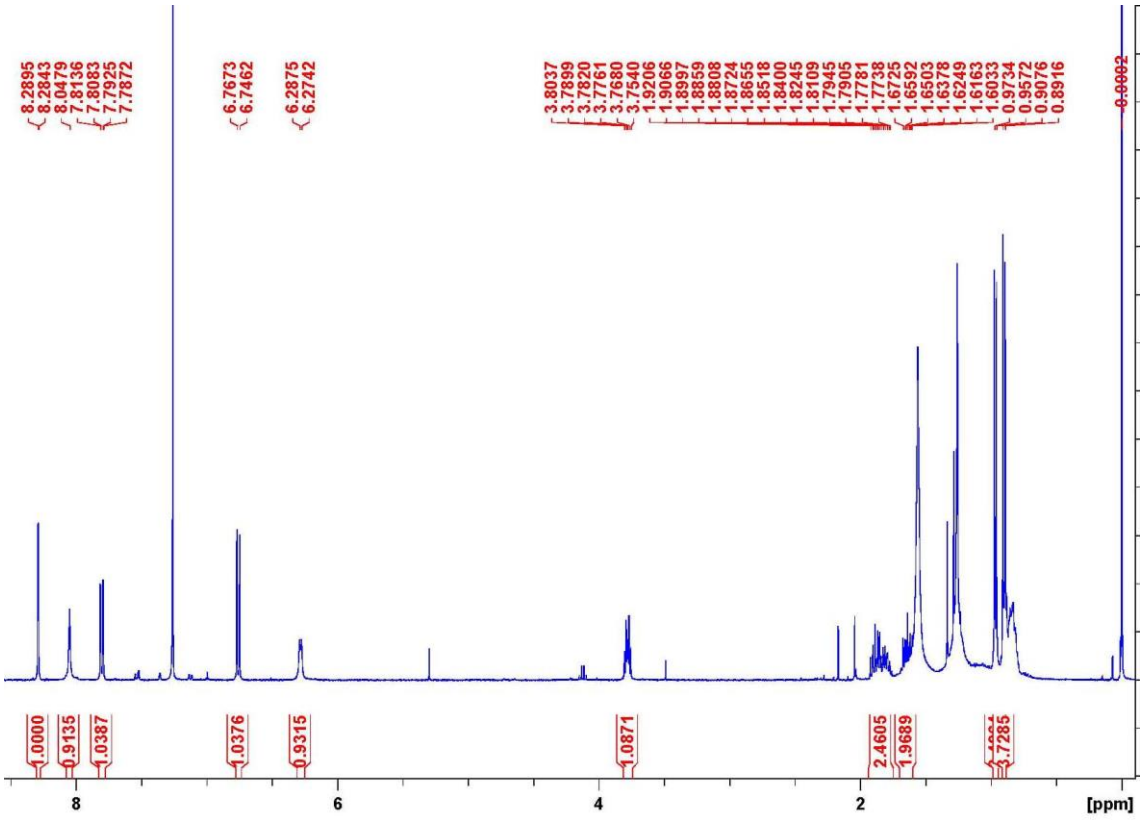

Figure S22. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of compound **13**.

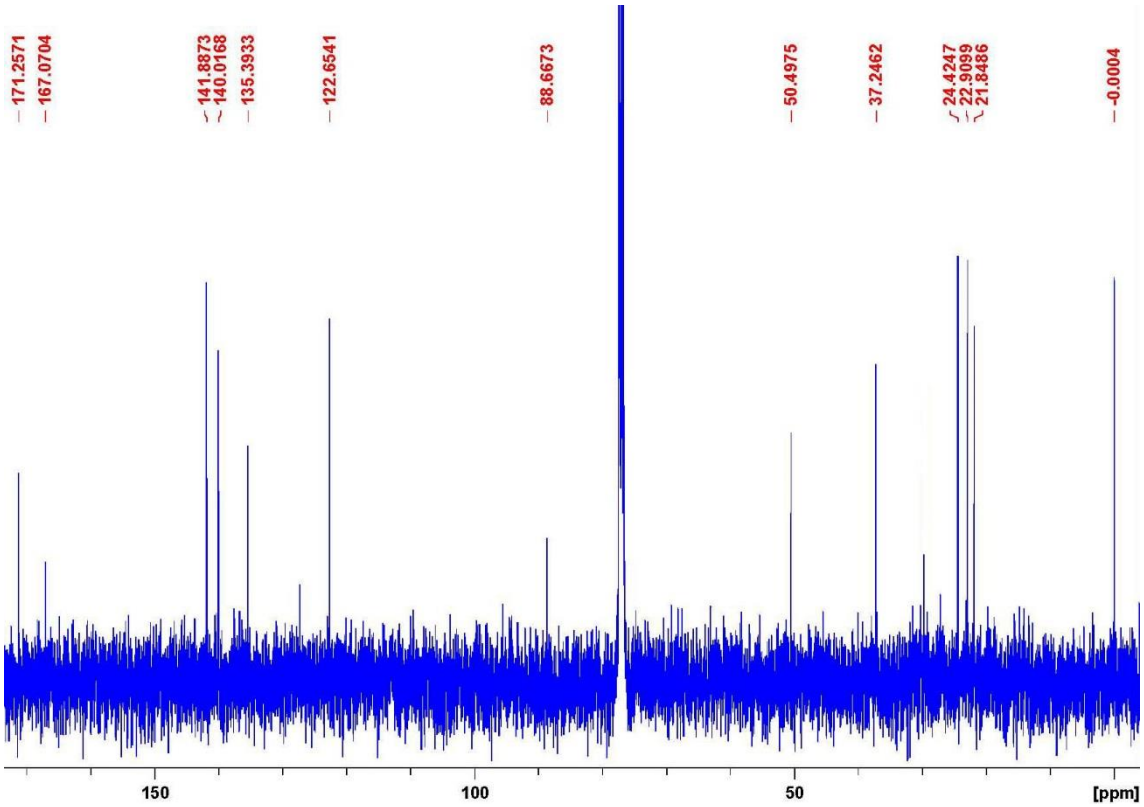

Figure S23. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of compound **13**.

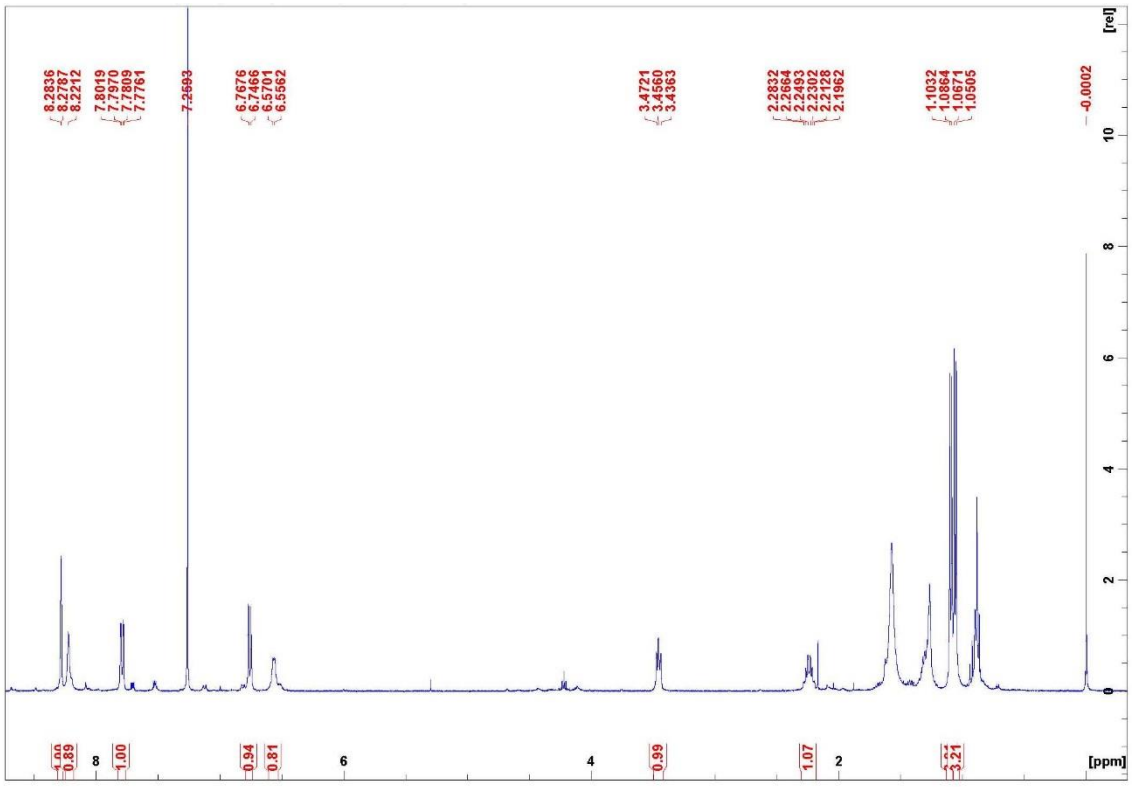

Figure S24. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of compound **14**.

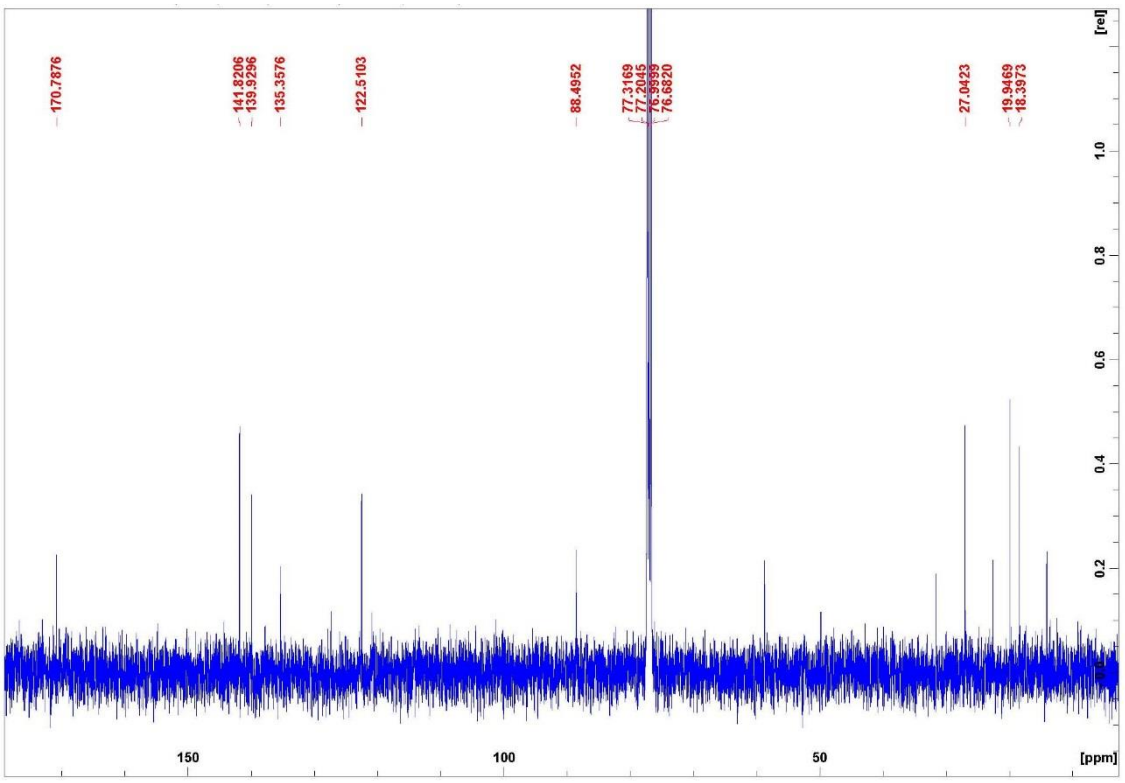

Figure S25. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of compound **14**.

Mass spectra (EI) of benzodiazepine-2,5-diones (**3**-**14**)

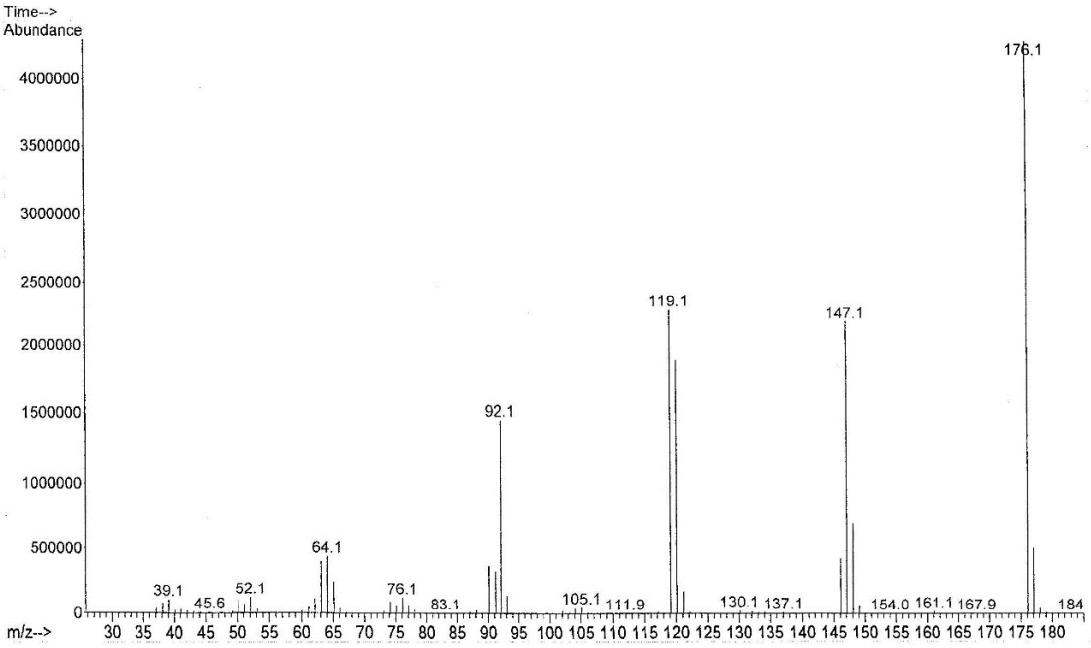

Figure S26. Mass spectrum of compound **3**.

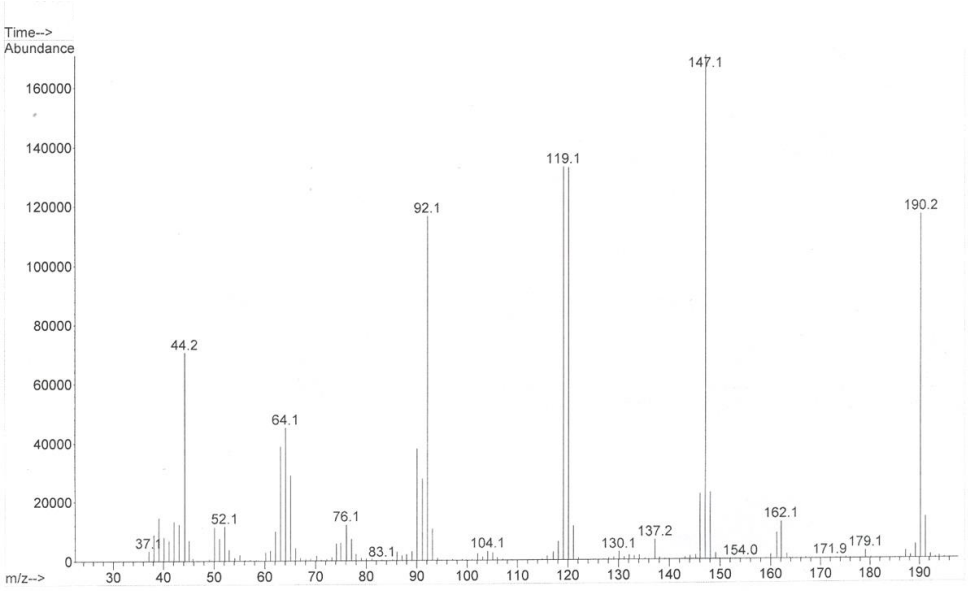

Figure S27. Mass spectrum of compound **4**.

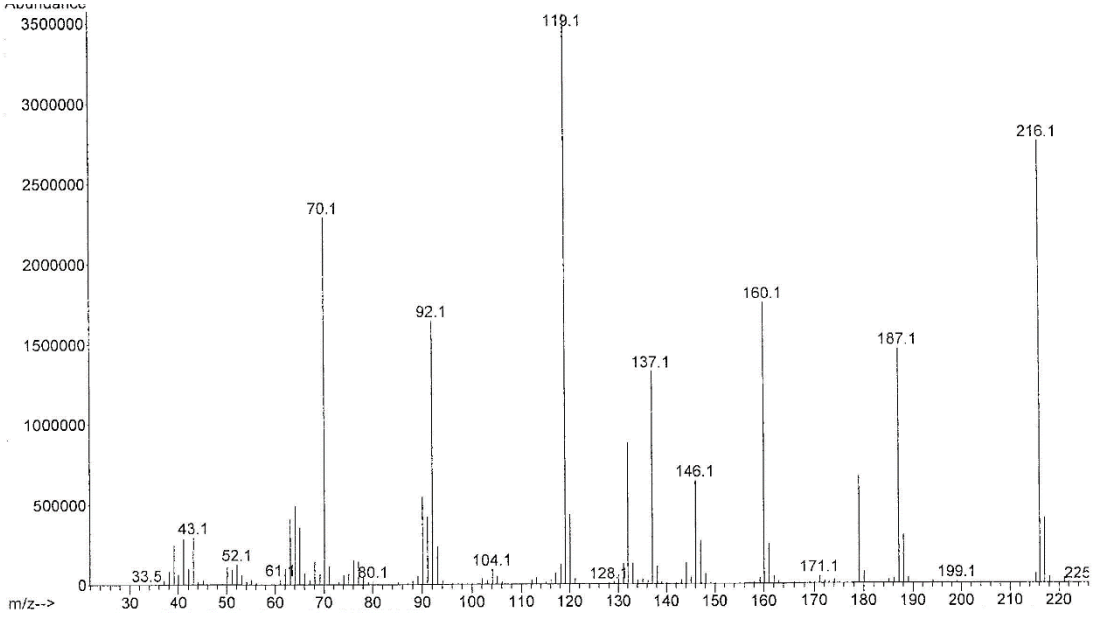

Figure S28. Mass spectrum of compound **5**.

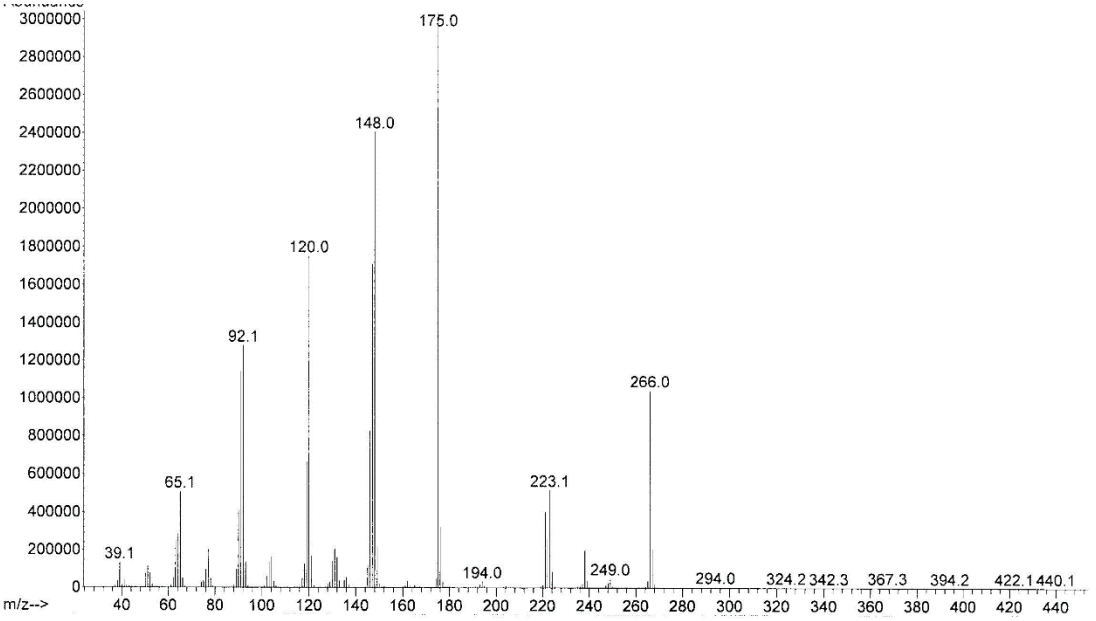

Figure S29. Mass spectrum of compound **6**.

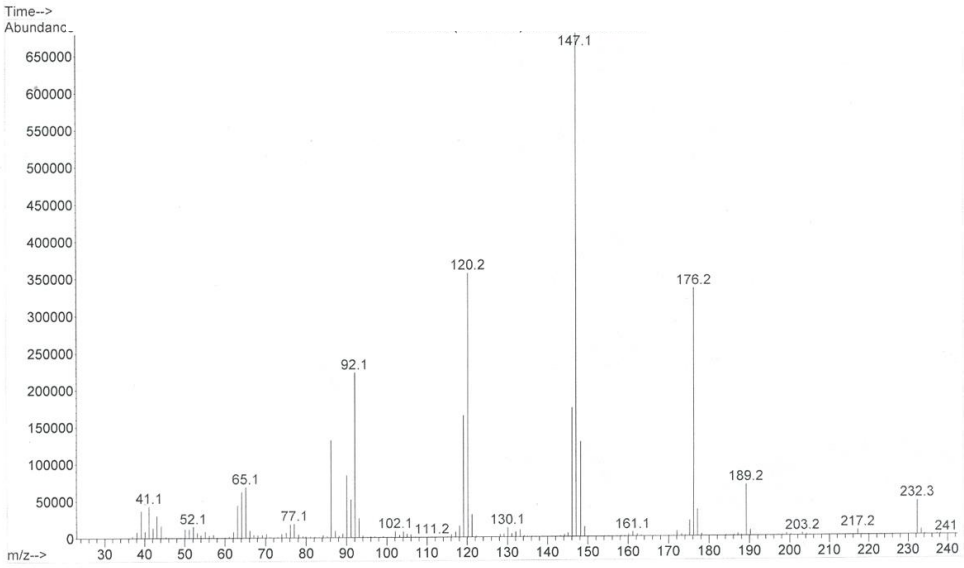

Figure S30. Mass spectrum of compound **7**.

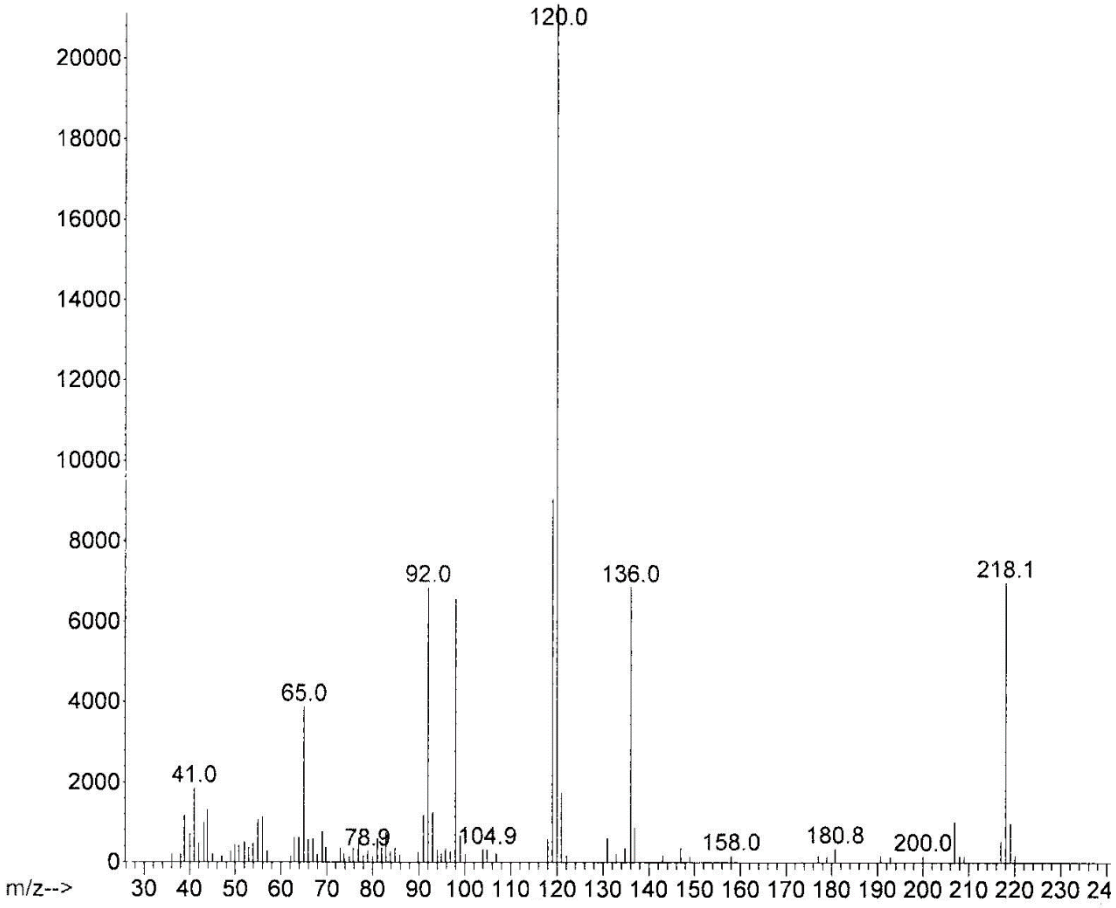

Figure S31. Mass spectrum of compound **8**.

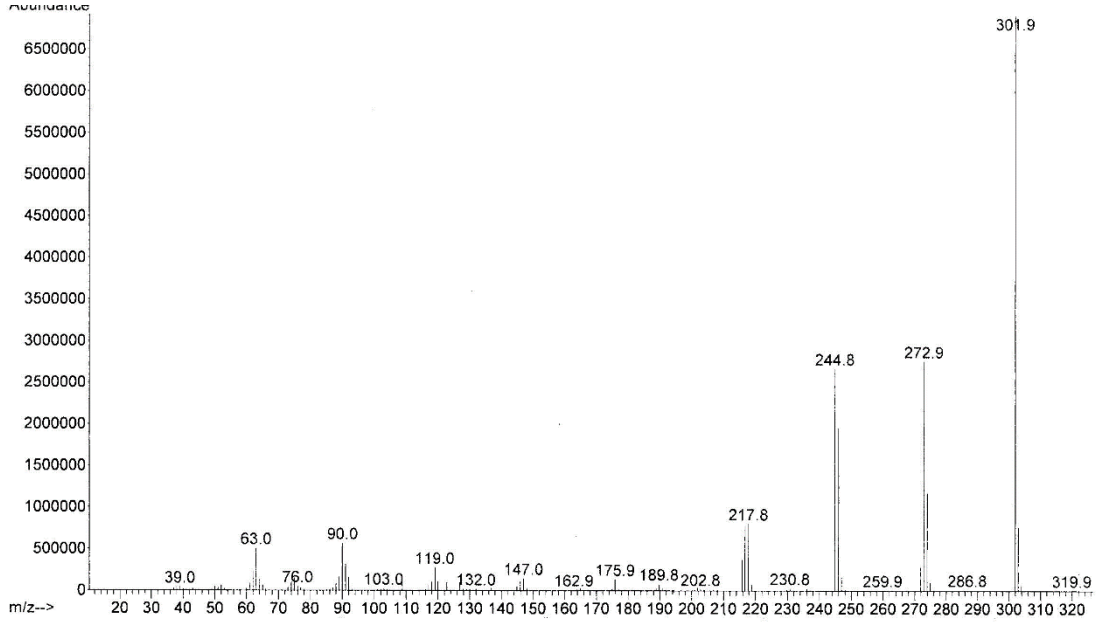

Figure S32. Mass spectrum of compound **9**.

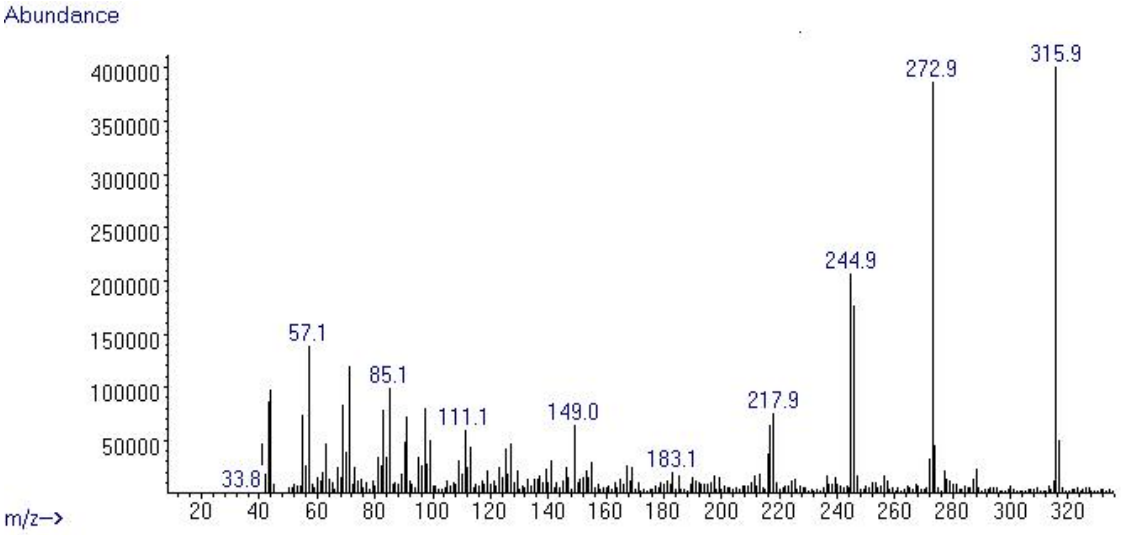

Figure S33. Mass spectrum of compound **10**.

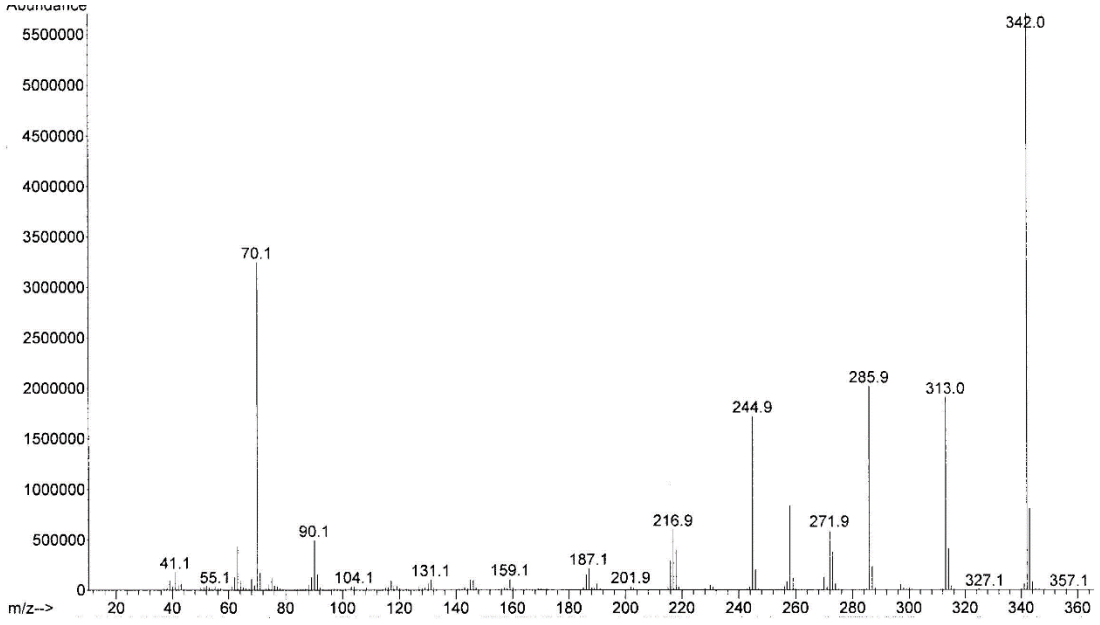

Figure S34. Mass spectrum of compound **11**.

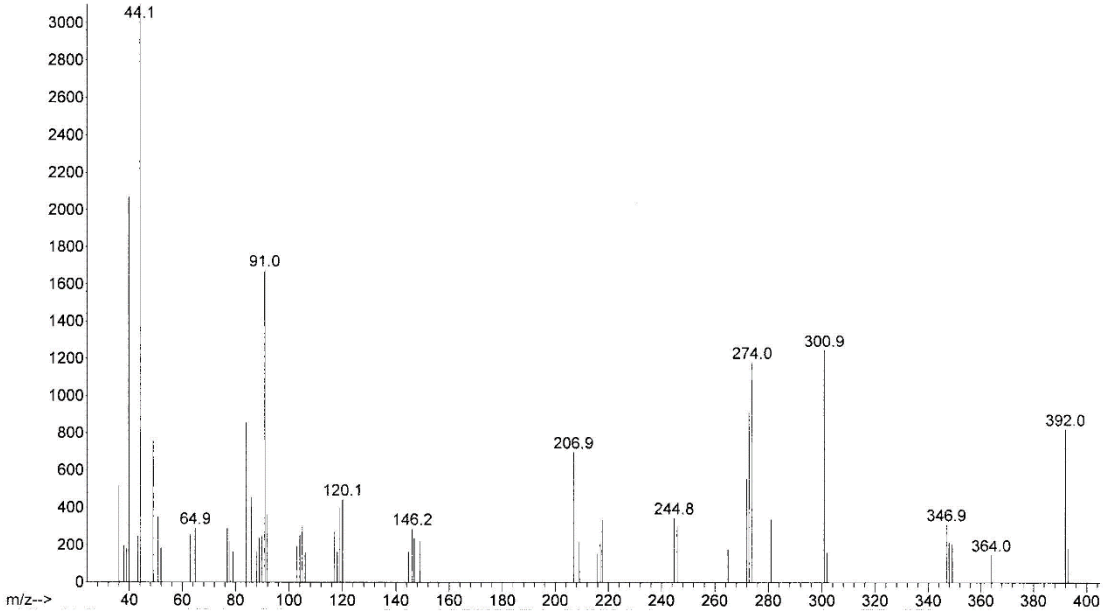

Figure S35. Mass spectrum of compound **12**.

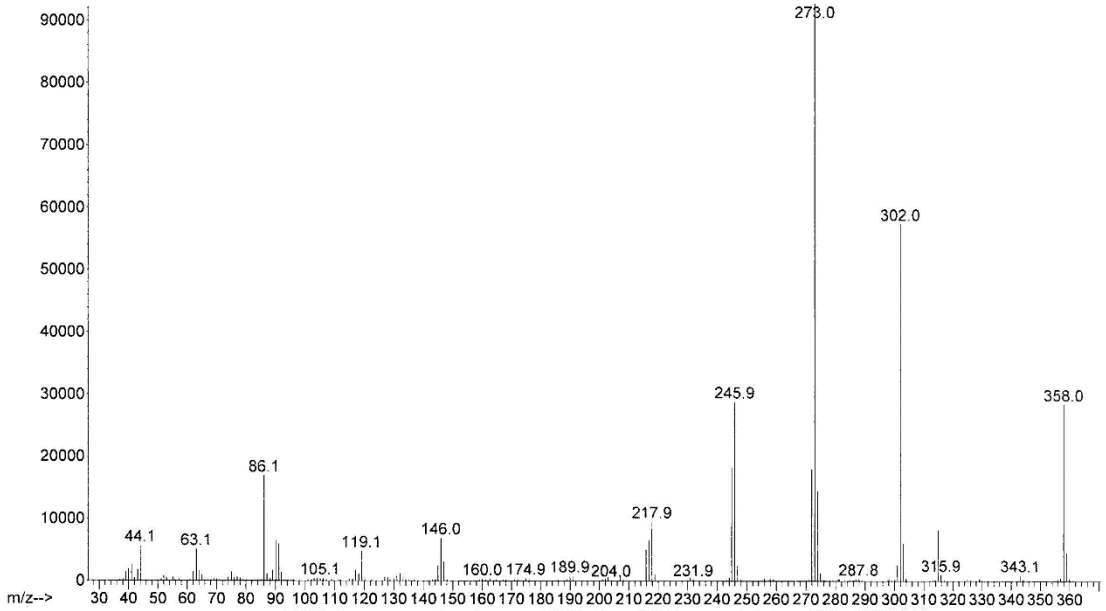

Figure S36. Mass spectrum of compound **13**.

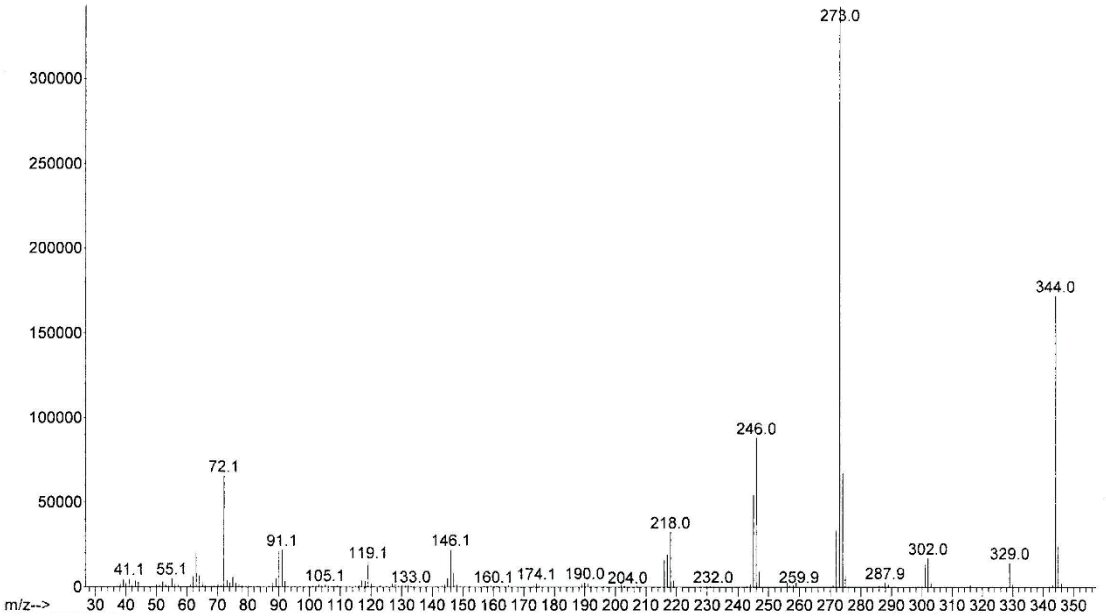

Figure S37. Mass spectrum of compound **14**.

Spectroscopic data for 6-iodoisatoic anhydride (**2**)

6-Iodo-1*H*-benzo[*d*][1,3]oxazine-2,4-dione (**2**)

Pale brown solid (76% yield), mp 196-198 °C; IR (KBr) ν / cm^{-1} 3169, 3083, 1758, 1703; ^{1}H NMR (400 MHz, DMSO-d_6) δ 6.96 (d, *J* 8.5, Hz, 1H), 8.01 (dd, *J* 8.5, 1.2 Hz, 1H), 8.13 (d, *J* 1.2 Hz, 1H), 11.63 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 86.4, 113.1, 118.2, 136.9, 141.6, 145.3, 147.3, 159.2; EIMS *m/z* (rel. int. %): $[M]^+$ 289 (52), 245 (100), 217 (22).

^{1}H NMR and ^{13}C NMR spectra of 6-iodoisatoic anhydride (**2**)

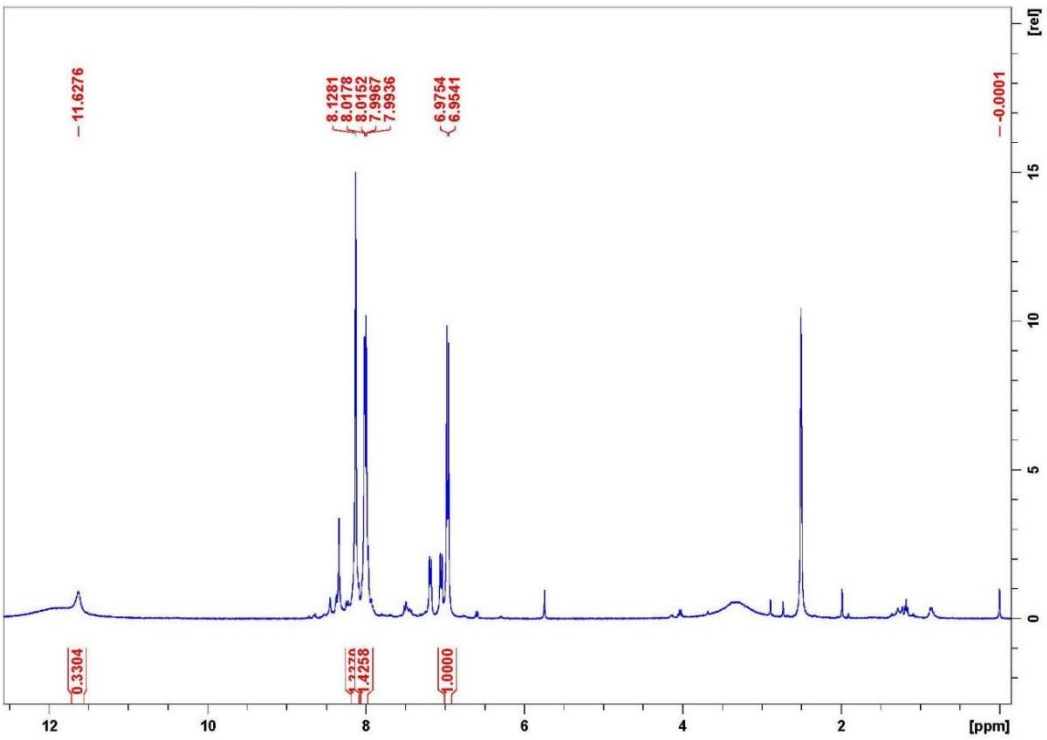

Figure S38. ^{1}H NMR spectrum (400 MHz, DMSO-d_6) of compound **2**.

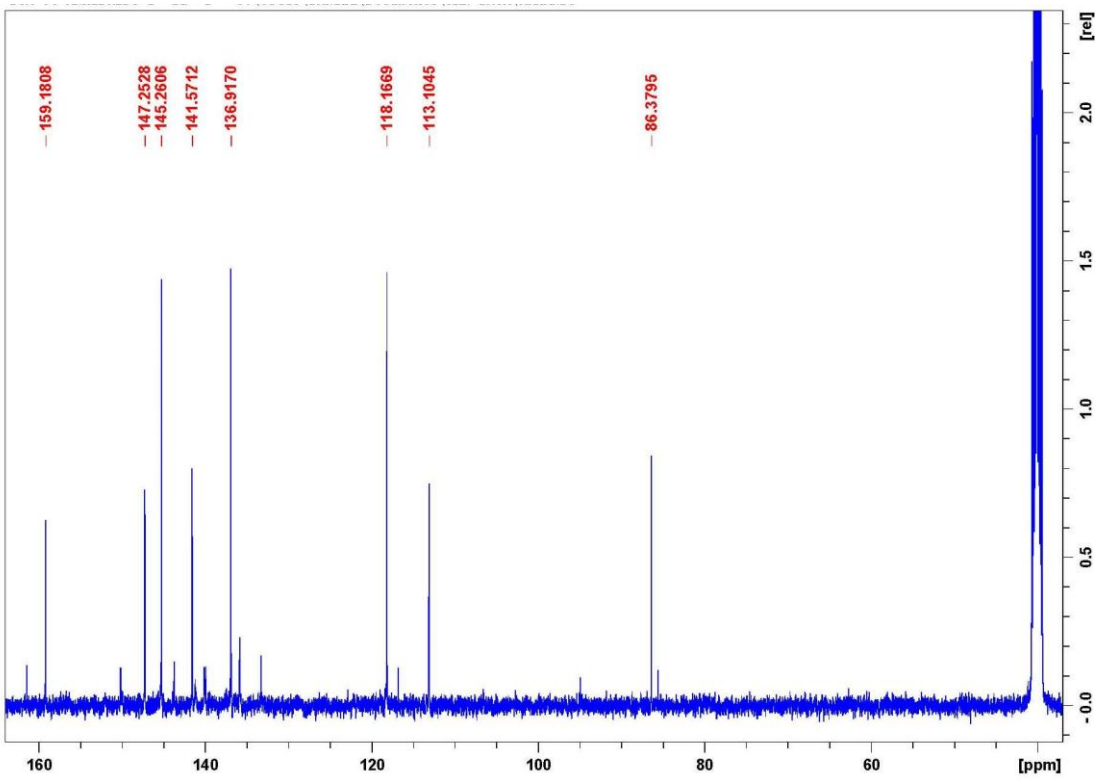

Figure S39. ^{13}C NMR spectrum (100 MHz, DMSO-d_6) of compound **2**.

Mass spectrum (EI) of 6-iodoisatoic anhydride (**2**)

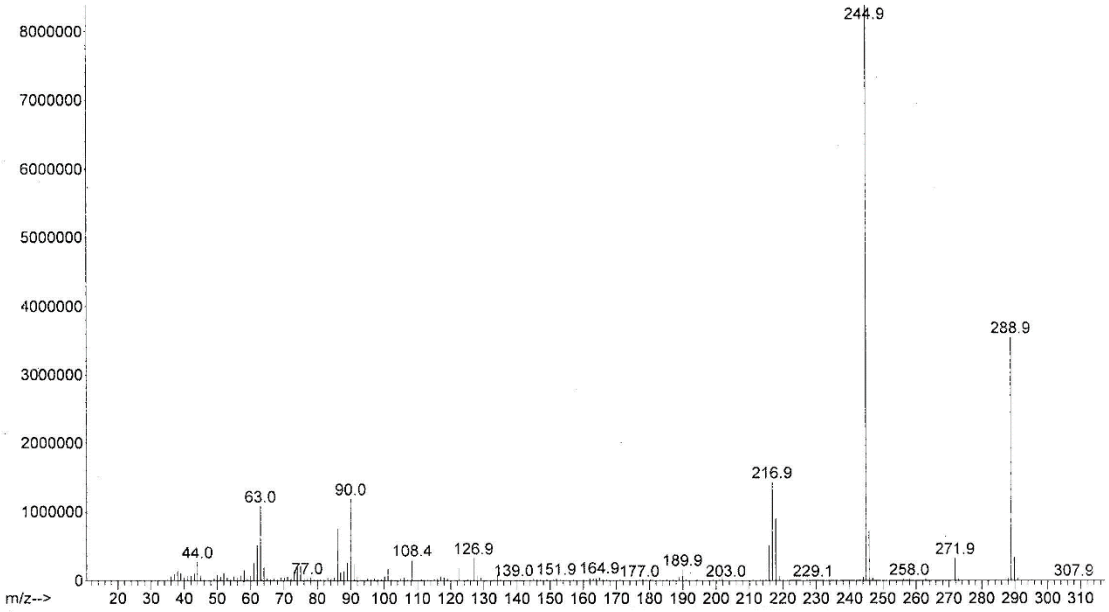

Figure S40. Mass spectrum of compound **2**.

^{1}H NMR and ^{13}C NMR spectra of isatoic anhydride (**1**) and the mixture of isatoic anhydride (**1**) and glacial acetic acid irradiated at 130 °C for 3 min

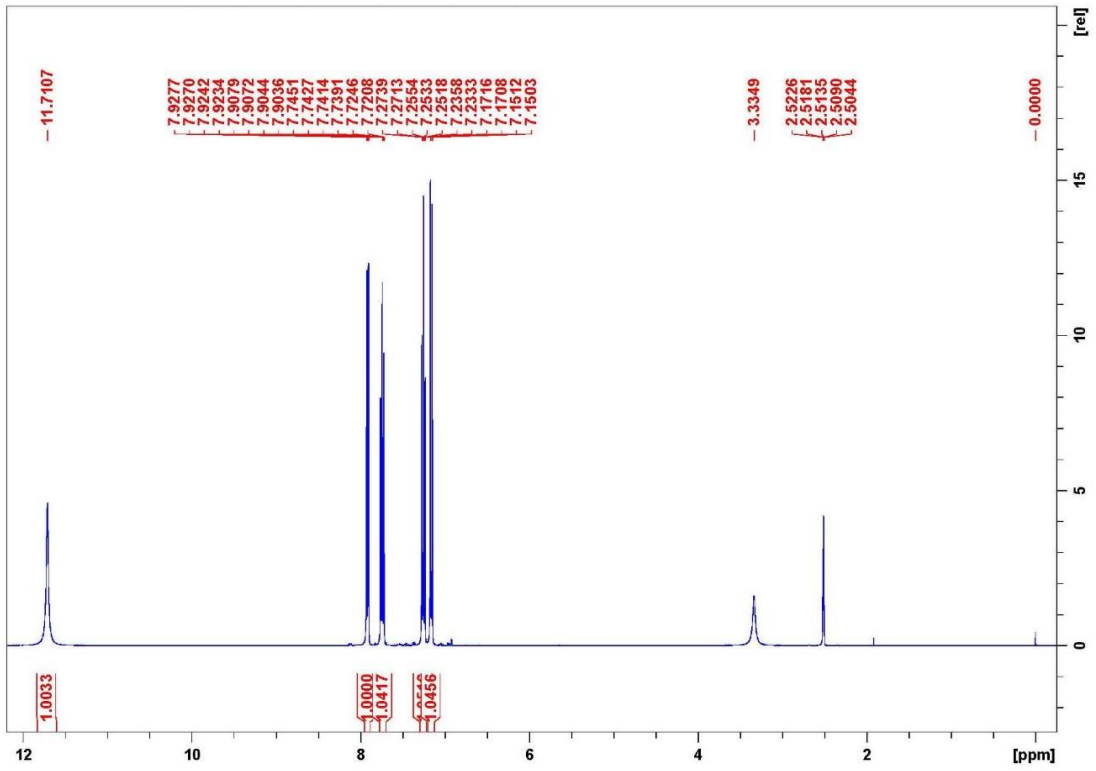

Figure S41. ^{1}H NMR spectrum (400 MHz, DMSO-d_6) of isatoic anhydride (**1**).

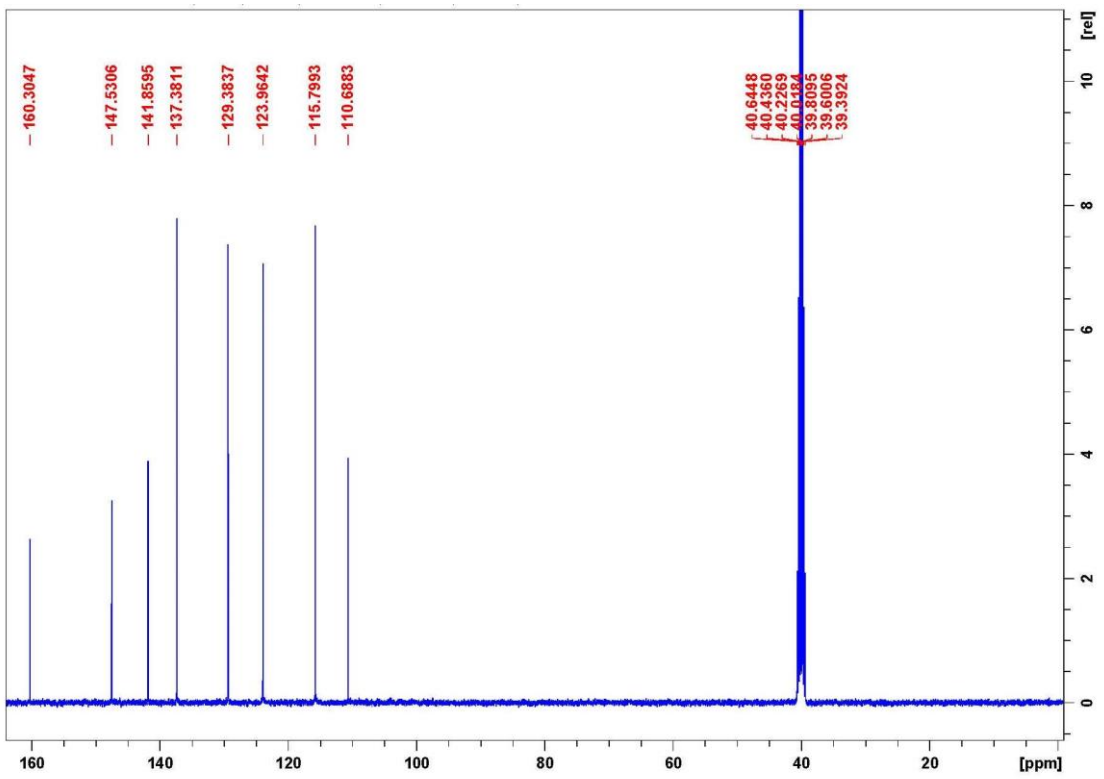

Figure S42. ^{13}C NMR spectrum (100 MHz, DMSO-d_6) of isatoic anhydride (**1**).

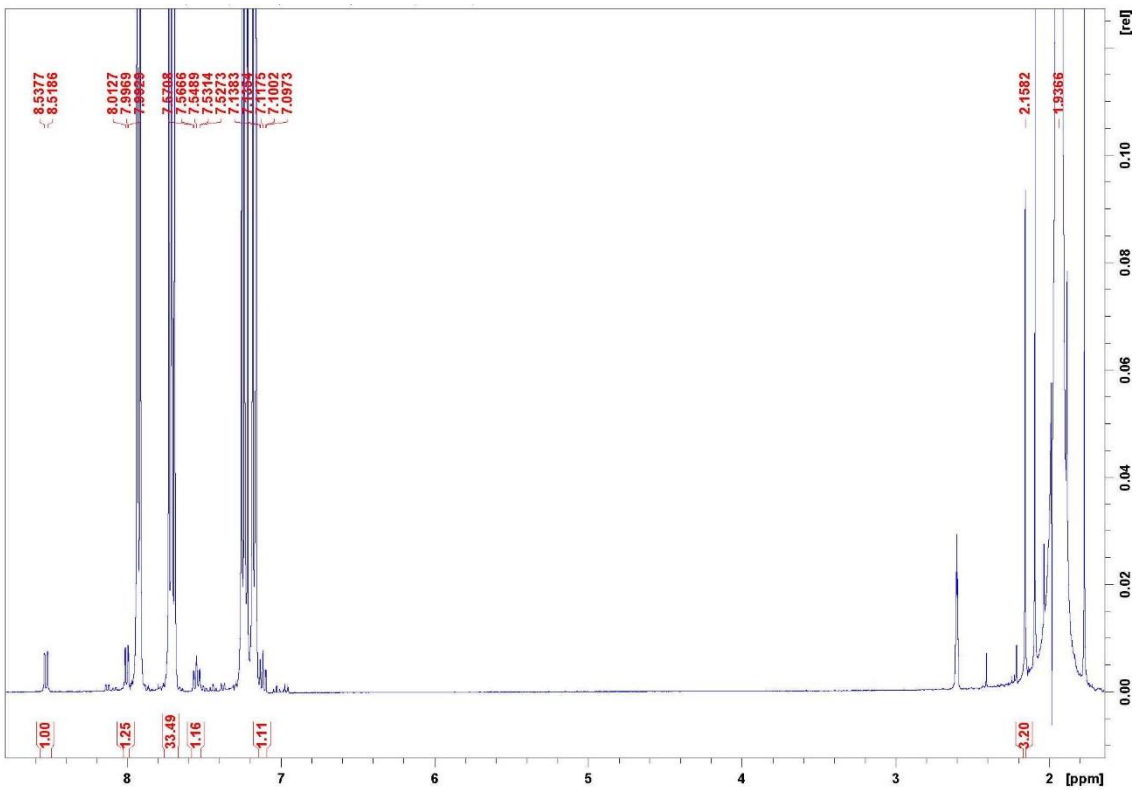

Figure S43. ^{1}H NMR spectrum (400 MHz, DMSO-d_6) of the mixture of isatoic anhydride (**1**) and glacial acetic acid irradiated at 130 °C for 3 min.

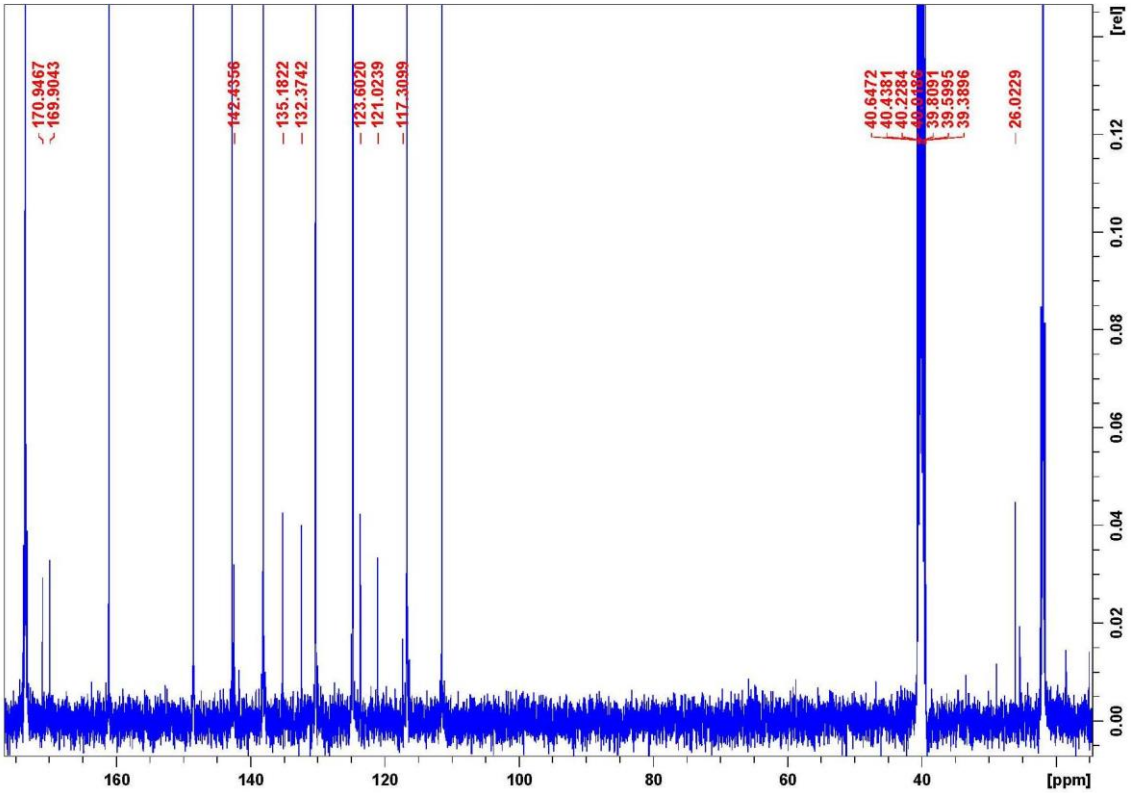

Figure S44. ^{13}C NMR spectrum (100 MHz, DMSO-d_6) of the mixture of isatoic anhydride (**1**) and glacial acetic acid irradiated at 130 °C for 3 min.