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Supplementary material

SUPPLEMENTARY MATERIAL TO

Synthesis and characterization of Fe_3O_4/PEG-400/oxalic acid magnetic nanoparticles as a heterogeneous catalyst for the synthesis of pyrrolin-2-ones derivatives

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J. Serb. Chem. Soc. 86 (11) (2021) 1039–1052

*1-(4-Boromophenyl)-5-(3-boromophenyl)-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (Table II, entry **8**)*

Limon solid; yield: 0.44 g (95 %); m.p. 230–231 °C; FT-IR (KBr, ν_{max} / cm^{-1}): 3309 (OH), 2952, 1717 (C=O), 1689 (C=O), 1490, 1374, 1212, 1011; ^{1}H-NMR (250.13 MHz, $CDCl_3$, δ / ppm): 3.70 (3H, *s*, OCH_3), 5.64 (1H, *s*, CH), 6.14–7.39 (8H, *m*, Ar), 8.93 (1H, *brs*, OH); ^{13}C-NMR (62.90 MHz, $CDCl_3$, δ / ppm) 52.27, 60.66, 102.92, 112.69, 123.42, 126.05, 130.33, 132.05, 132.23, 135.02, 137.06, 152.39, 156.23, 160.93 (C=O); Mass (*m*/*z*): 469 (M^++4), 467.1 (M^++2), 465.1 (M^+), 267.0, 208 (100%), 157.1, 129.1, 101.1.

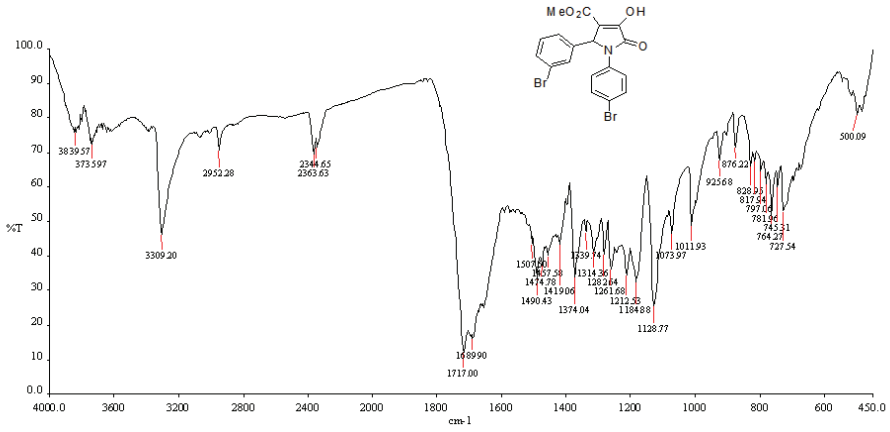

Fig. S-1. FT-IR (KBr, cm^{-1}) spectrum of compound **8**.

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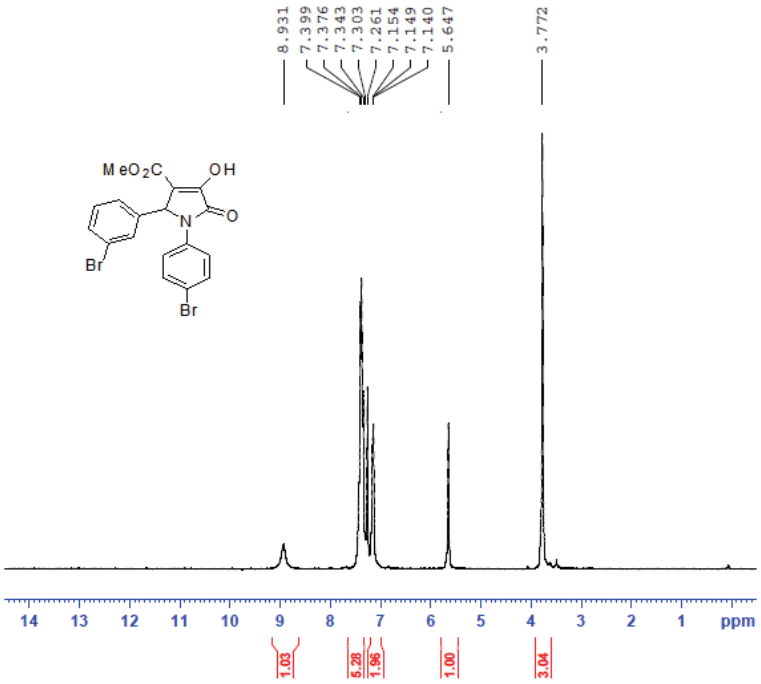

Fig. S-2. ^{1}H-NMR spectrum of compound **8** (250 MHz, $CDCl_3$, δ / ppm).

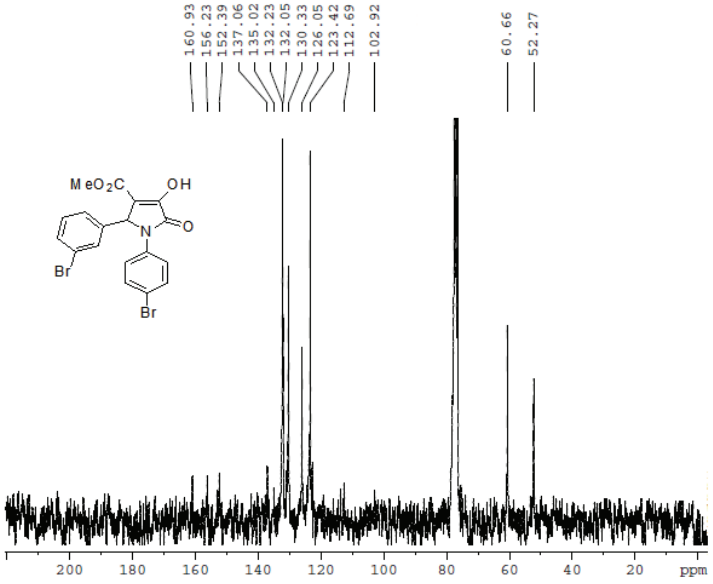

Fig. S-3. ^{13}C-NMR spectrum of compound **8** (63 MHz, $CDCl_3$, δ / ppm).

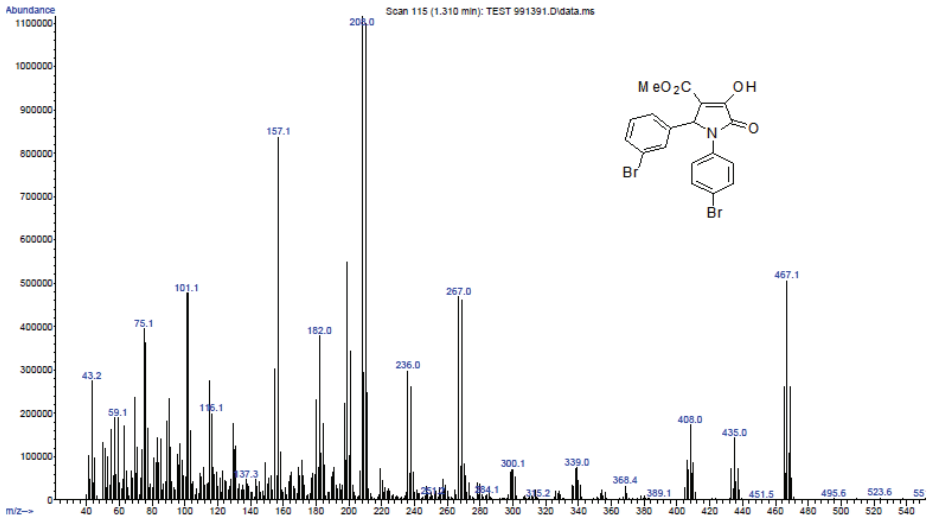

Fig. S-4. Mass spectrum of compound **8**.

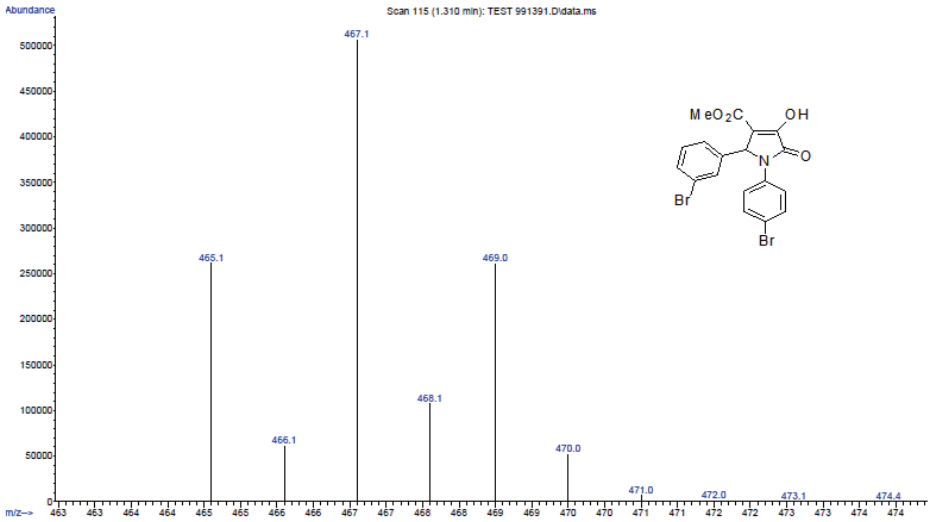

Fig. S-5. Mass spectrum of compound **8**.

*1-(4-Boromophenyl)-5-(4-methoxyphenyl)-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (Table II, entry **9**)*

White yellow solid; Yield: 0.38 g (92 %); m.p.: 165–167 °C; FT-IR (KBr, ν_{max} / cm^{-1})): 3214 (OH), 2952, 1710 (C=O), 1683(C=O), 1495, 1374, 1229, 1031; ^{1}H-NMR (MHz: 250.13, $CDCl_3$, δ / ppm): 3.74 (6H, *s*, 2×OCH_3), 5.64 (1H, *s*, CH), 6.76–7.87 (8H, *m*, Ar), 8.9 (1H, *brs*, OH); ^{13}C-NMR (MHz: 62.90, $CDCl_3$, δ / ppm): 52.10, 55.16, 60.99, 113.06, 114.22, 118.99, 123.62, 126.21, 128.53, 132.00, 135.30, 155.69, 159.70, 162.77 (C=O), 165.16 (C=O); Mass (*m*/*z*): 419.1 (M^++2), 417.2 (M^+), 289.1, 219.2 (100 %), 151.1, 134.2, 97.2.

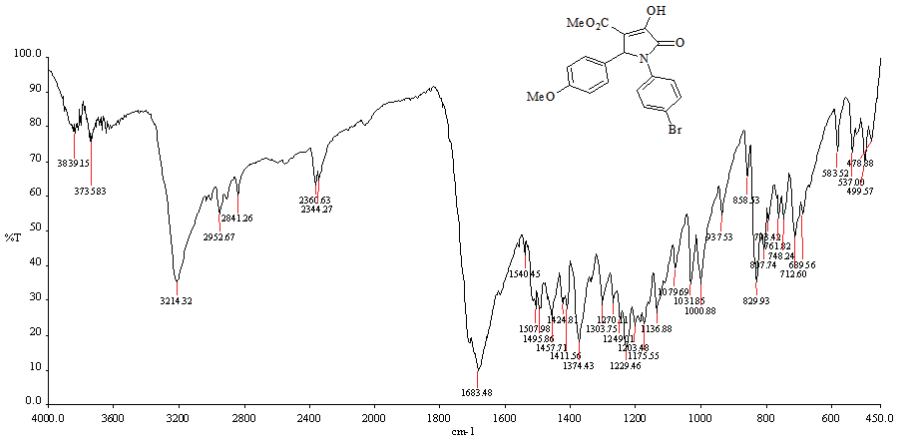

Fig. S-6. FT-IR (KBr, cm^{-1}) spectrum of compound **9**.

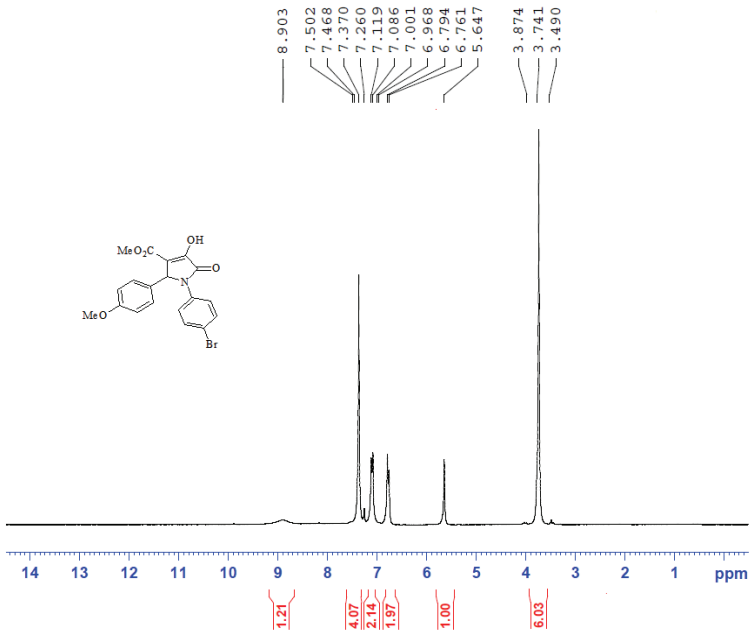

Fig. S-7. ^{1}H-NMR spectrum of compound **9** (250 MHz, $CDCl_3$, δ / ppm).

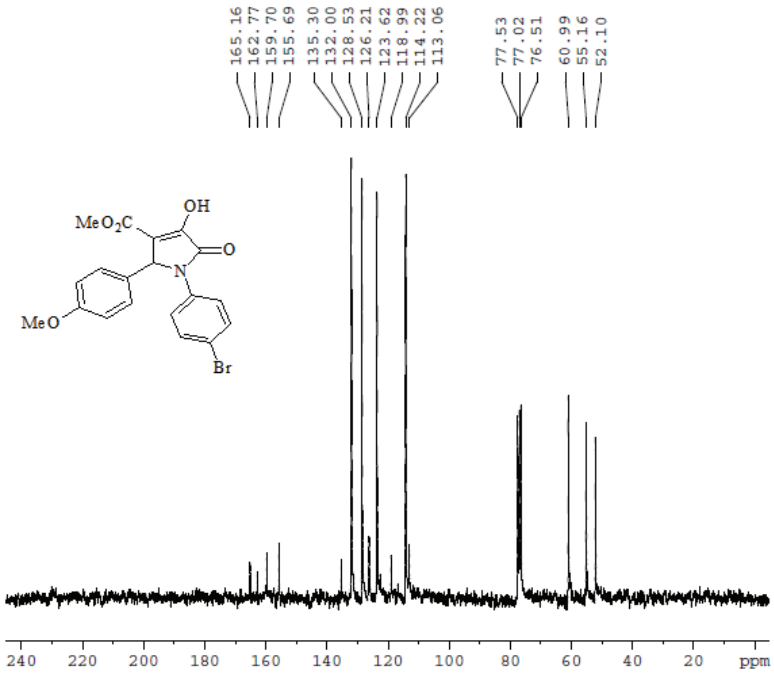

Fig. S-8. ^{13}C-NMR spectrum of compound **9** (63 MHz, $CDCl_3$, δ / ppm).

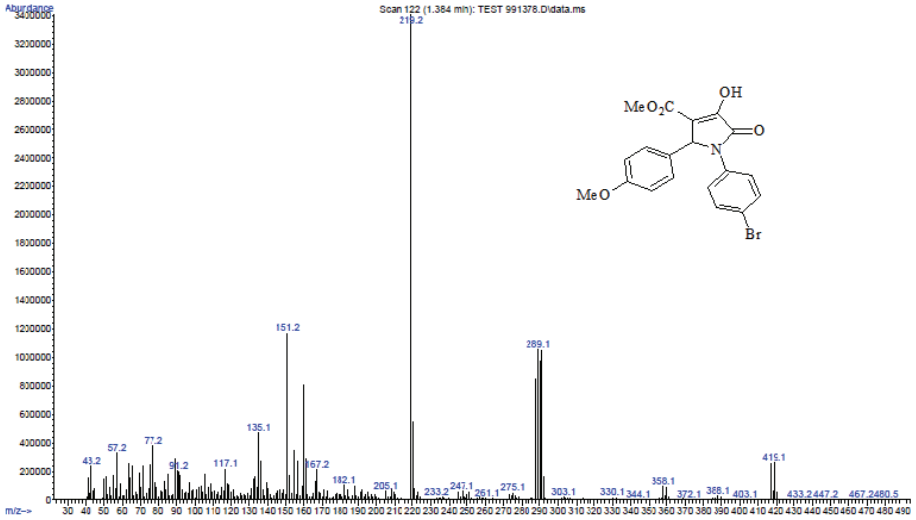

Fig. S-9. Mass spectrum of compound **9**.

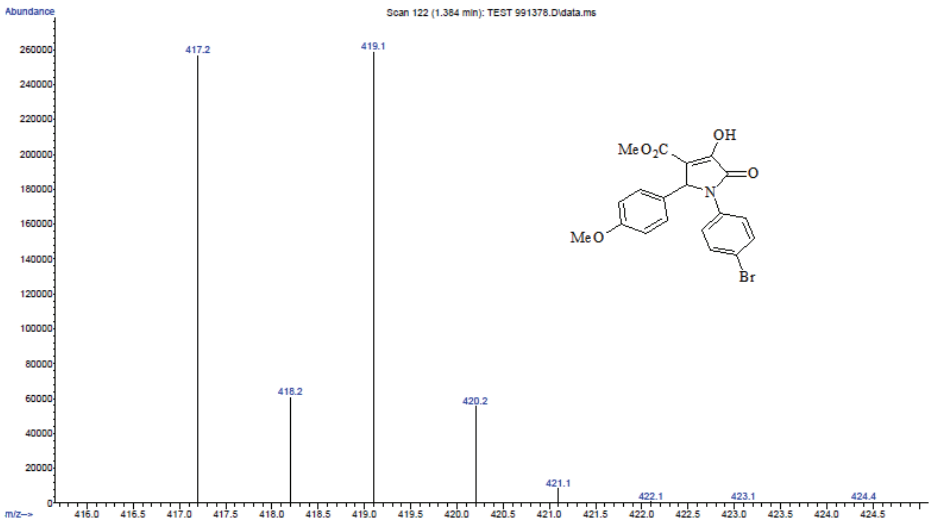

Fig. S-10. Mass spectrum of compound **9**.

*1-(4-Methoxyphenyl)-5-(2-methoxyphenyl)-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (Table II, entry **10**)*

White solid, Yield: 0.34 g (94 %); m.p.: 210–212 °C; FT-IR (KBr, ν_{max} / cm^{-1})): 3273 (OH), 2954, 1723 (C=O), 1687 (C=O), 1495, 1372, 1247, 1131; ^{1}H-NMR (250.13 MHz, $CDCl_3$, δ / ppm): 3.70 (3H, *s*, OCH_3), 3.88 (3H, *s*, OCH_3), 6.23 (1H, *s*, CH), 6.79–7.46 (m, 8H, Ar), 8.93 (brs, 1H, OH). The ^{13}C-NMR (62.90 MHz, $CDCl_3$, δ / ppm): 30.89, 52.03, 55.87, 111.48, 118.60, 121.08, 122.99, 127.52, 129.91, 131.82, 135.58, 156.58, 162.50 (C=O); Mass (*m*/*z*): 369.2 (M^+), 337.2, 219.1 (100 %), 151.2, 131.1, 83.2, 57.2.

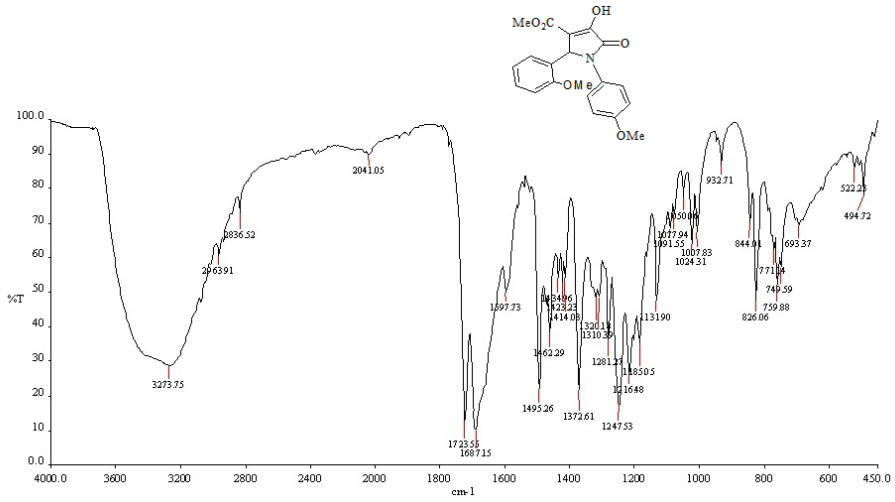

Fig. S-11. FT-IR (KBr / cm^{-1}) spectrum of compound **10**.

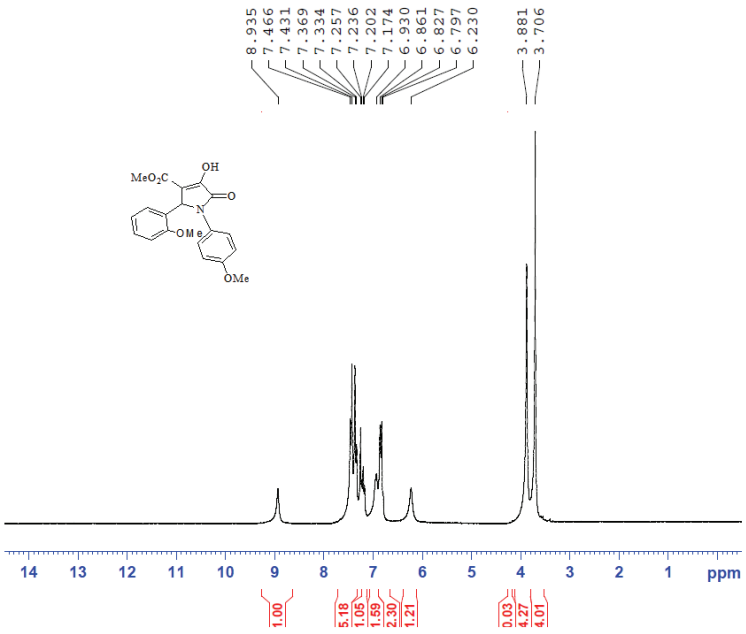

Fig. S-12. ^{1}H-NMR spectrum of compound **10** (250 MHz, $CDCl_3$, δ / ppm).

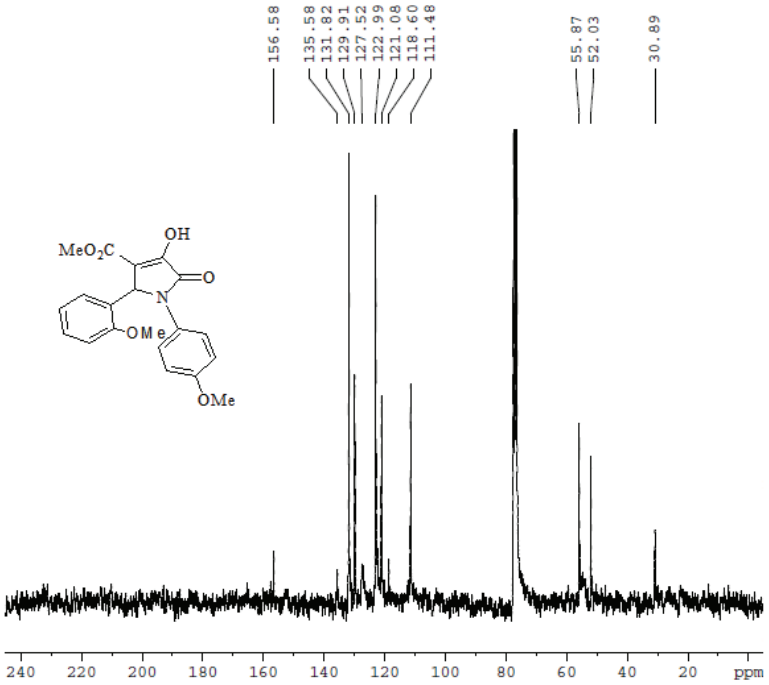

Fig. S-13. ^{13}C-NMR spectrum of compound **10** (63 MHz, $CDCl_3$, δ / ppm).

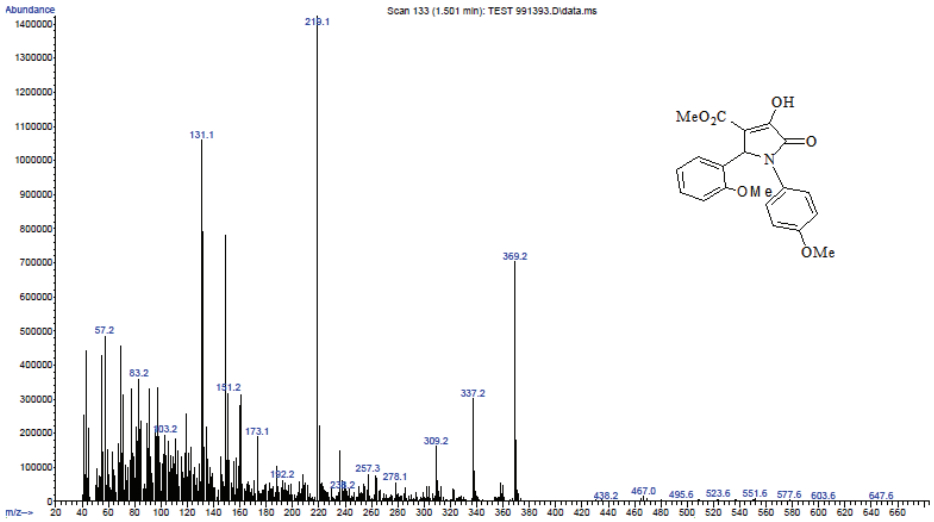

Fig. S-14. Mass spectrum of compound **10**.

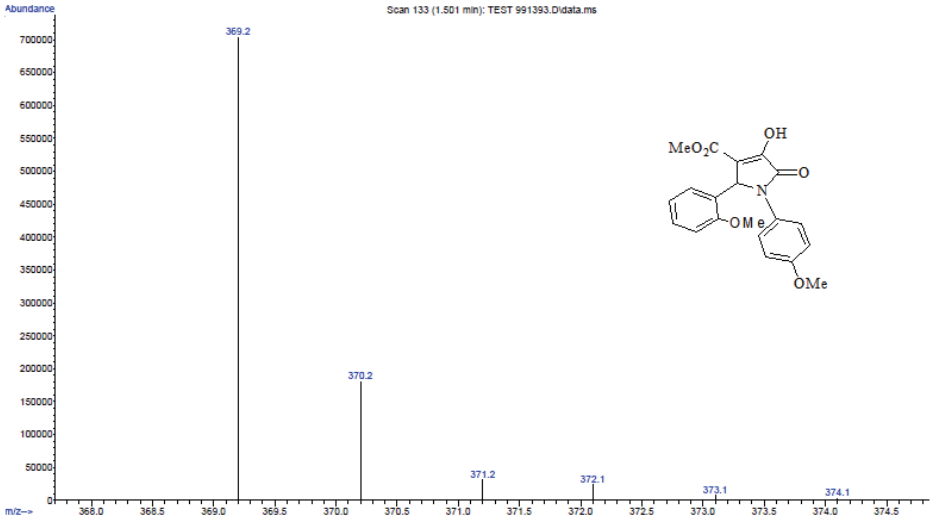

Fig. S-15. Mass spectrum of compound **10**.

*1-(4-Boromophenyl)-5-(2-cholorophenyl)-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (Table II, entry **11**)*

White solid, Yield: 0.40 g (95%). m.p.: 193–194 °C. FT-IR (KBr, ν_{max} /cm^{-1}): 3309 (OH), 2954, 1714 (C=O), 1683 (C=O), 1490, 1363, 1234, 1009; ^{1}H-NMR (250.13 MHz, CDC_{l3}, δ / ppm): 3.72 (3H, *s*, OCH_3), 6.38 (1H, *s*, CH), 6.70–7.40 (8H, *m*, Ar); ^{13}C-NMR (62.90 MHz, $CDCl_3$ δ / ppm): 52.18, 56.38, 112.79, 119.06, 122.94, 126.73, 127.65, 129.92, 132.13, 135.07, 156.69, 162.64 (C=O), 165.15 (C=O), Mass (*m*/*z*): 425.1 (M^++4), 423.1 (M^++2), 421.1 (M^+), 391.0, 223.1, 164.1 (100%), 136.1, 101.1, 75.1.

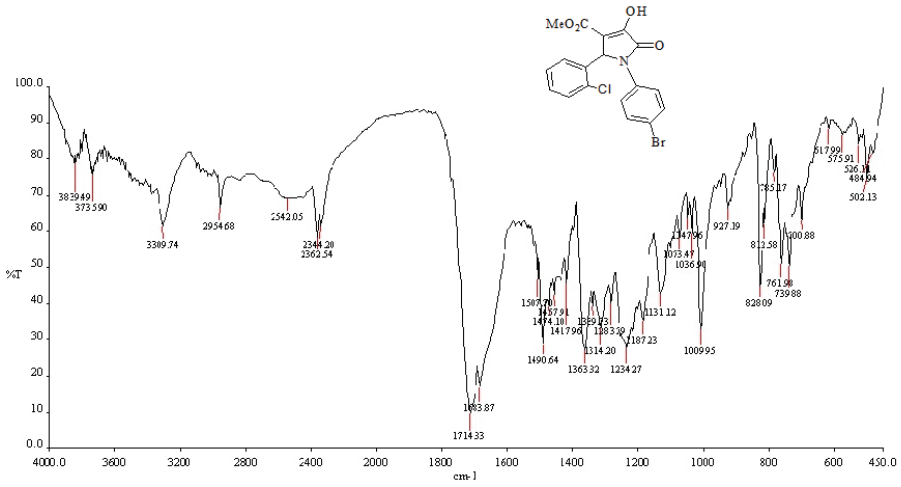

Fig. S-16. FT-IR (KBr, cm^{-1}) spectrum of compound **11**.

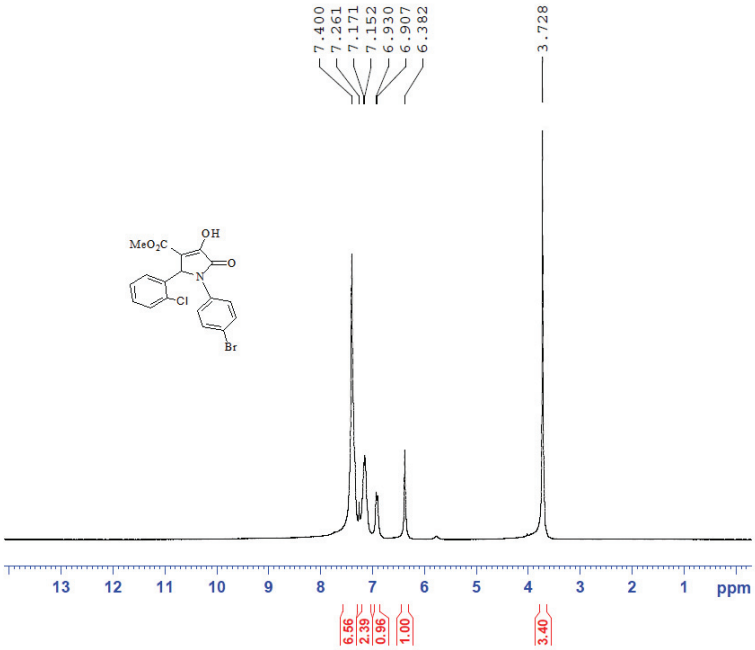

Fig. S-17. ^{1}H-NMR spectrum of compound **11** (250 MHz, $CDCl_3$, δ / ppm).

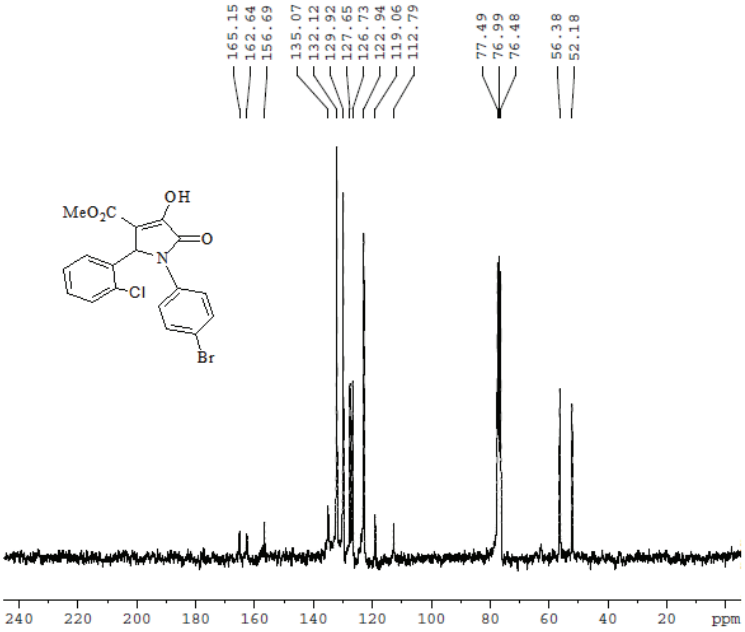

Fig. S-18. ^{13}C-NMR spectrum of compound **11** (63 MHz, $CDCl_3$, δ / ppm).

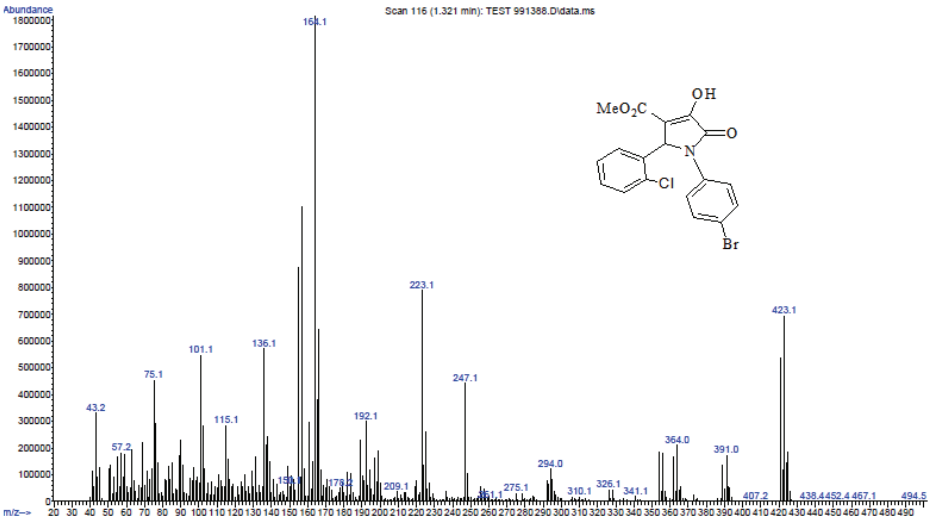

Fig. S-19. Mass spectrum of compound **11**.

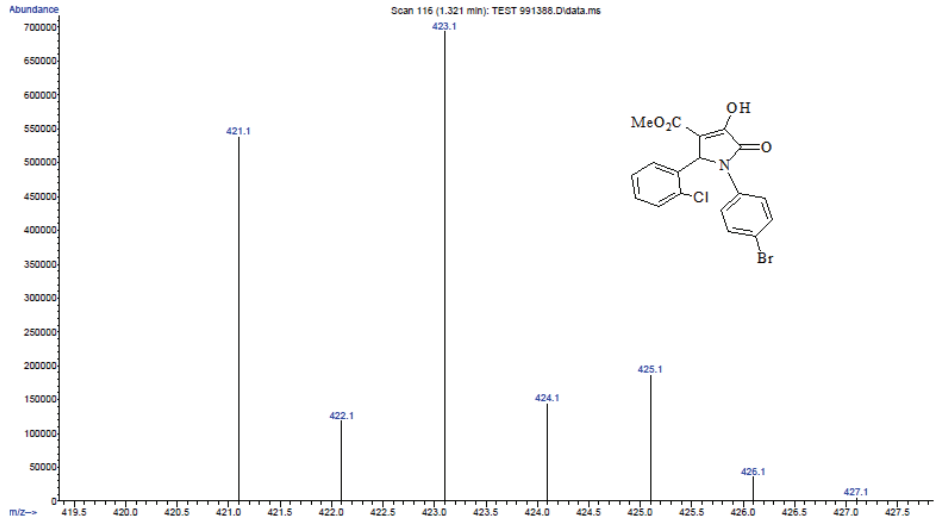

Fig. S-20. Mass spectrum of compound **11**.

*1-(4-Methylphenyl)-5-(2-methoxyphenyl)-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (Table 2, entry **12**)*

White solid; Yield: 0.31 g (90 %); m.p.: 173–175 °C; FT-IR (KBr, ν_{max} / cm^{-1}): 3220 (OH), 2963, 1720 (C=O), 1686 (C=O), 1513, 1379, 1245, 1131; ^{1}H-NMR ($CDCl_3$, 250.13 MHz, δ / ppm): 2.22 (3H, *s*, CH_3), 3.70 (3H, *s*, OCH_3), 3.86 (3H, *s*, OCH_3), 6.25 (1H, *s*, CH), 6.78–7.40 (8H, *m*, Ar), 9.10 (1H, brs, OH); ^{13}C-NMR (62.90 MHz, $CDCl_3$, δ / ppm): 20.88, 51.96, 55.70, 55.86, 111.40, 120.91, 121.84, 122.93, 127.52, 129.35, 129.65, 133.81, 135.34, 156.67, 157.81, 162.96 (C=O), 165.42 (C=O). Mass (m/z): 353.2 (M^+), 321.2, 294.1, 278.1, 238.2, 219.1 (100%), 188.0, 160.1 133.1, 91.2, 65.2.

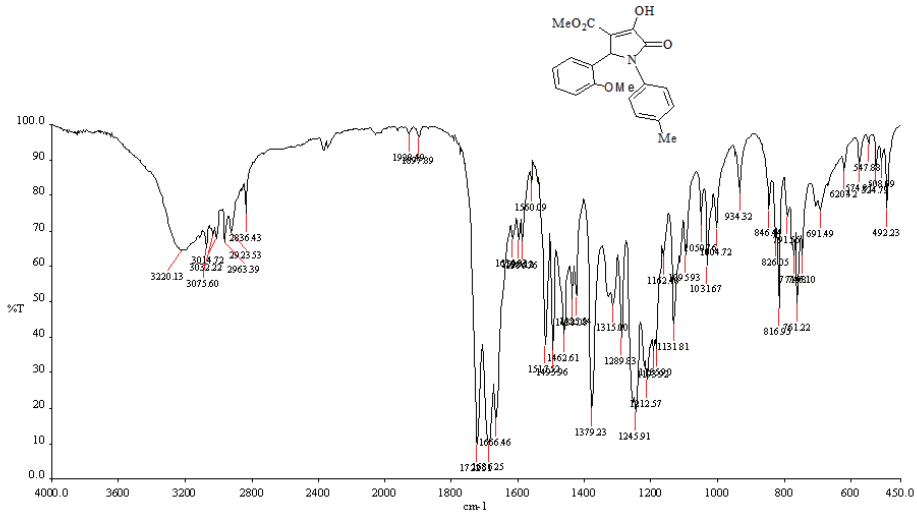

Fig. S-21. FT-IR (KBr, cm^{-1}) spectrum of compound **12**.

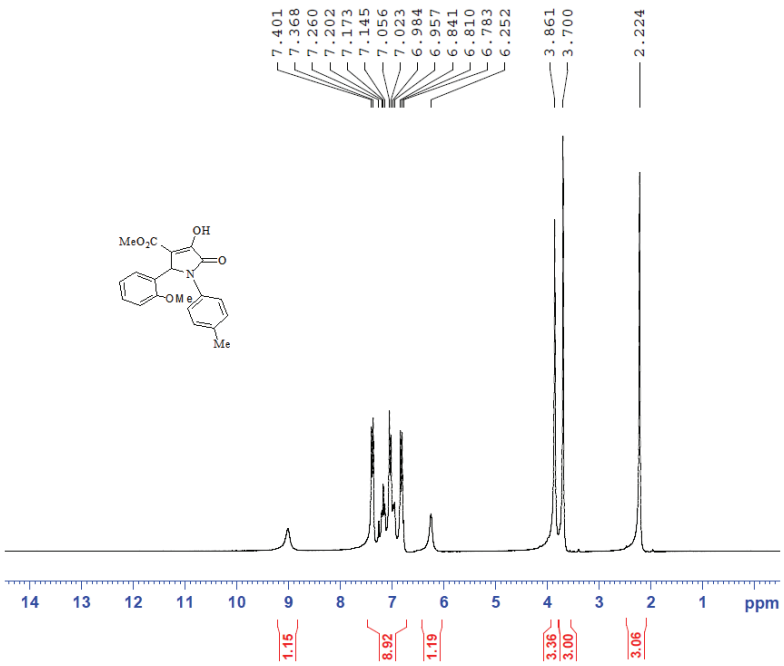

Fig. S-22. ^{1}H-NMR spectrum of compound **12** (250 MHz, $CDCl_3$, δ / ppm).

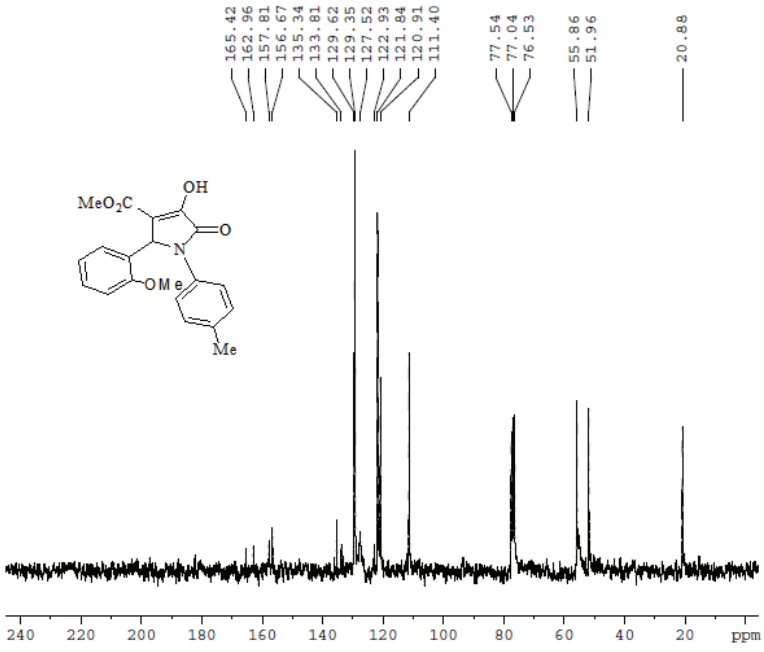

Fig. S-23. ^{13}C-NMR spectrum of compound **12** (63 MHz, $CDCl_3$, δ / ppm).

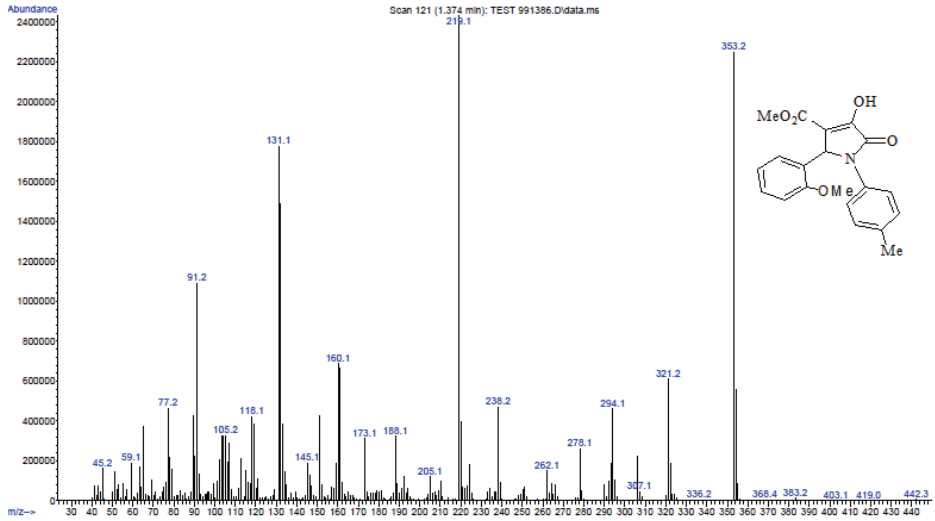

Fig. S-24. Mass spectrum of compound **12**.

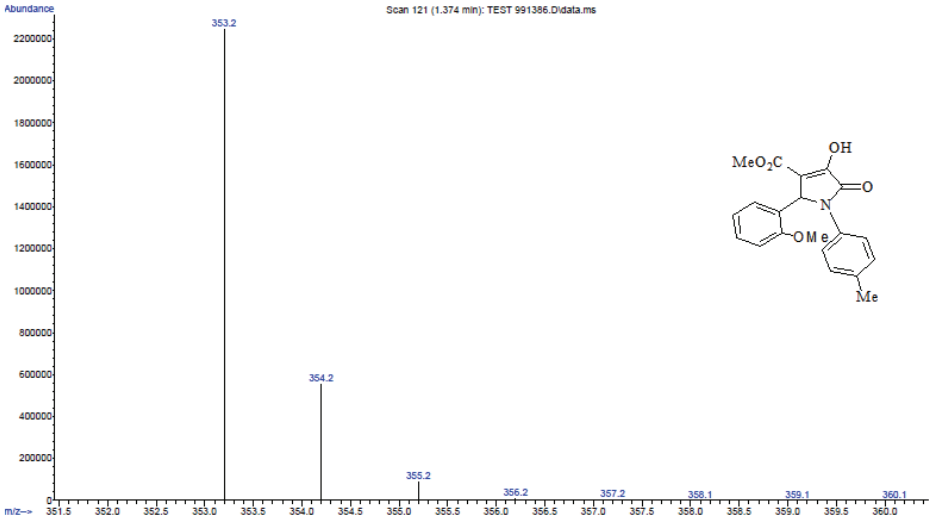

Fig. S-25. Mass spectrum of compound **12**.

*1-(4-Methylphenyl)-5-(3-boromophenyl)-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (Table II, entry **13**)*

Yellow solid; Yield: 0.36 g (91 %); m.p.: 184–186 °C; FT-IR (KBr, ν_{max} / cm^{-1}): 3322 (OH), 2953, 1720 (C=O), 1686 (C=O), 1513, 1351, 1262, 1127; ^{1}H-NMR (500 MHz, DMSO-d_6, δ / ppm): 2.21 (3H, *s*, CH_3), 3.62 (3H, *s*, OCH_3), 6.08 (1H, *s*, CH), 7.05–7.53 (8H, *m*, Ar), 11.92 (1H, *brs*, OH); ^{13}C-NMR (MHz, 100.51, DMSO-d_6, δ / ppm): 20.90, 51.64, 60.34, 111.62, 122.95, 127.01, 129.71, 130.94, 131.17, 131.35, 133.96, 135.33, 140.01, 153.47, 162.91 (C=O), 164.19 (C=O); Mass (*m*/*z*): 403.1 (M^++2), 401.2 (M^+), 369.1, 342.1, 269.0, 236.0, 208.0, 180.0, 157.1, 138.1 (100%), 115.2, 91.2, 65.2.

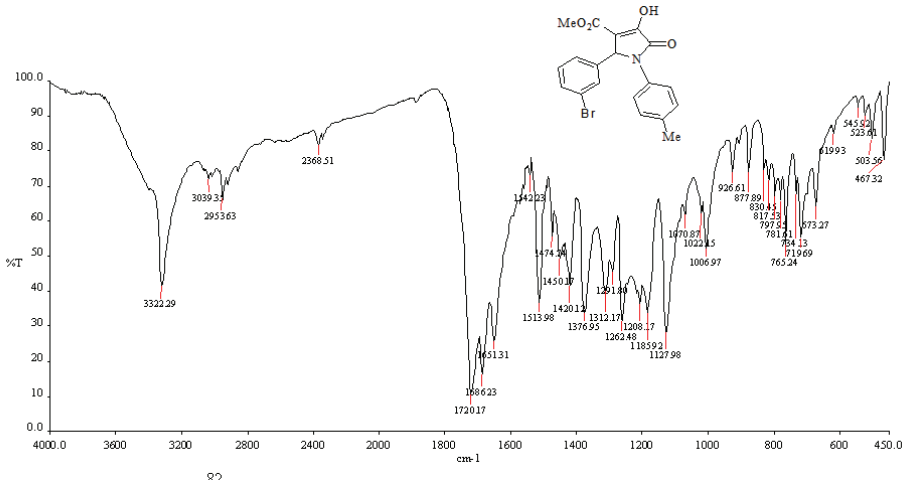

Fig. S-26. FT-IR (KBr, cm^{-1}) spectrum of compound **13**.

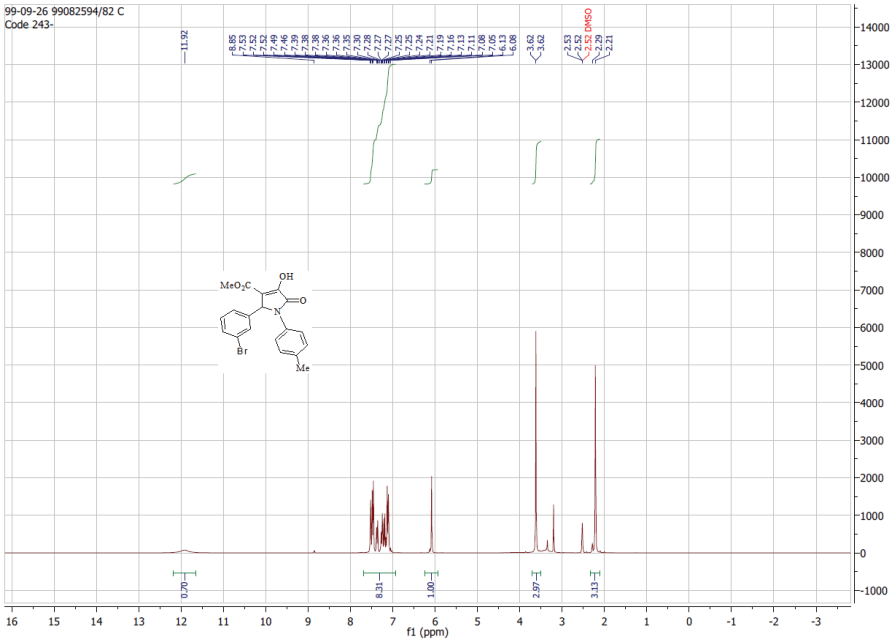

Fig. S-27. ^{1}H-NMR spectrum of compound **13** (500 MHz, DMSO-d_6, δ / ppm).

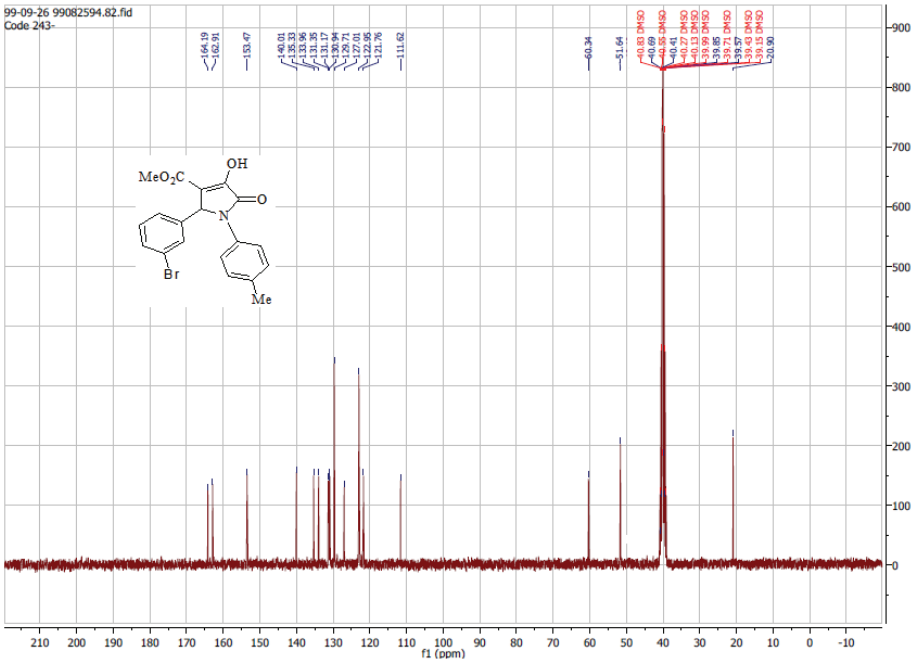

Fig. S-28. ^{13}C-NMR spectrum of compound **13** (100 MHz, DMSO-d_6, δ / ppm).

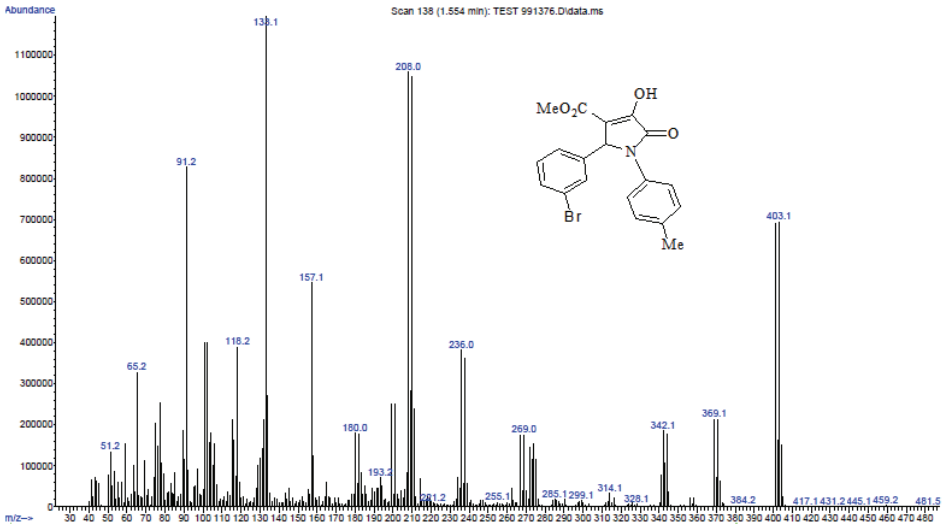

Fig. S-29. Mass spectrum of compound **13**.

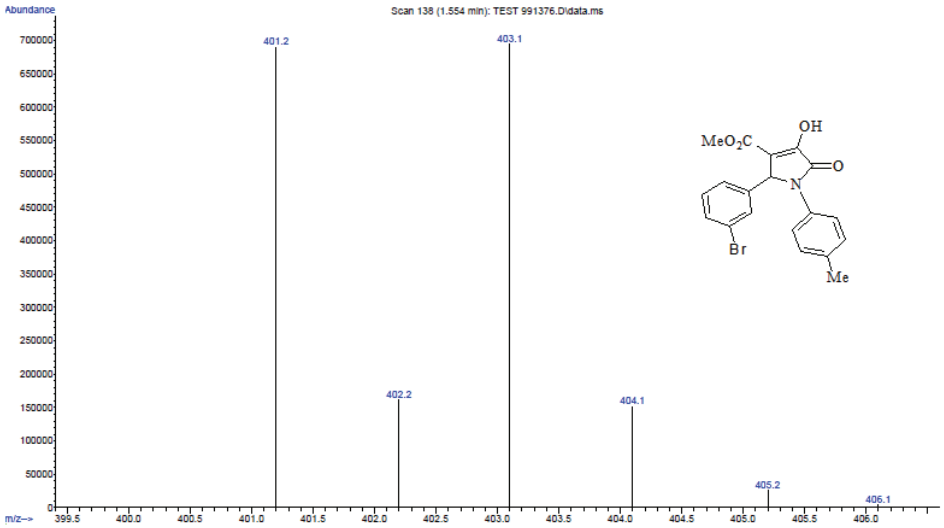

Fig. S-30. Mass spectrum of compound **13**.

*1-(4- Bromophenyl)-5-(3-nitrophenyl)-3-hydroxy-4-methoxycarbonyl-3-pyrrolin--2-one (Table II, entry **14**)*

White solid; Yield: 0.39 g (90 %); m.p.: 220–222 °C. FT-IR (KBr, ν_{max} / cm^{-1}): 3290 (OH), 2952, 1716 (C=O), 1693 (C=O), 1529 (NO_2), 1493, 1358 (NO_2), 1213, 1190; ^{1}H-NMR (250.13 MHz, $CDCl_3$, δ / ppm): 3.76 (3H, *s*, OCH_3), 5.82 (1H, *s*, CH), 7.21–8.1 (8H, *m*, Ar), 8.90 (1H, *s*, OH); ^{13}C-NMR (MHz 100.51, DMSO-d_6, δ / ppm): 51.73, 59.76, 111.63, 118.45, 123.58, 124.75, 130.46, 132.20, 134.43, 135.67, 139.44, 148.08, 153.48, 162.82 (C=O), 164.45 (C=O); Mass (*m*/*z*): 434.1 (M^++2), 432.1 (M^+), 402.1, 373.1, 306.1, 234.1, 218.1, 197, 175.1 (100%), 157.1, 129.1, 101.1, 76.1.

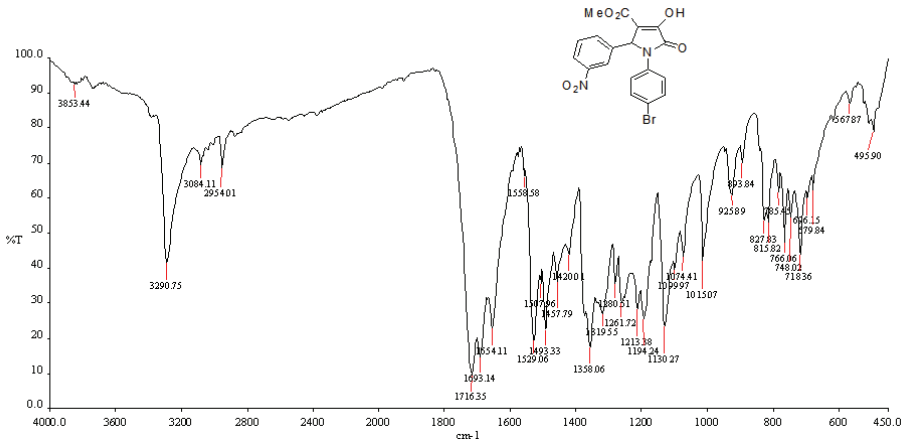

Fig. S-31. FT-IR (KBr, cm^{-1}) spectrum of compound **14**.

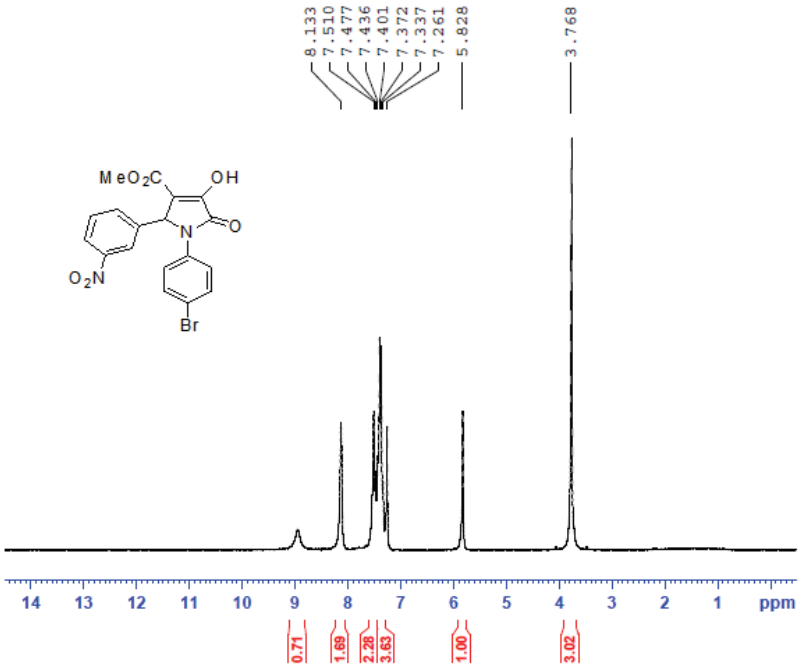

Fig. S-32. ^{1}H-NMR spectrum of compound **14** (250 MHz, $CDCl_3$, δ / ppm).

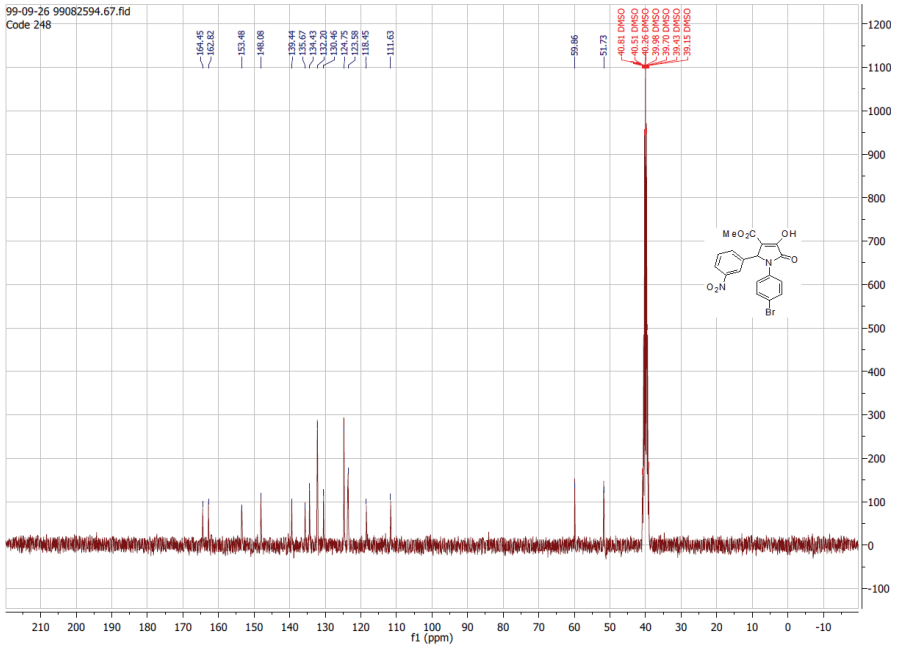

Fig. S-33. ^{13}C-NMR spectrum of compound **14** (100 MHz, DMSO-d_6, δ / ppm).

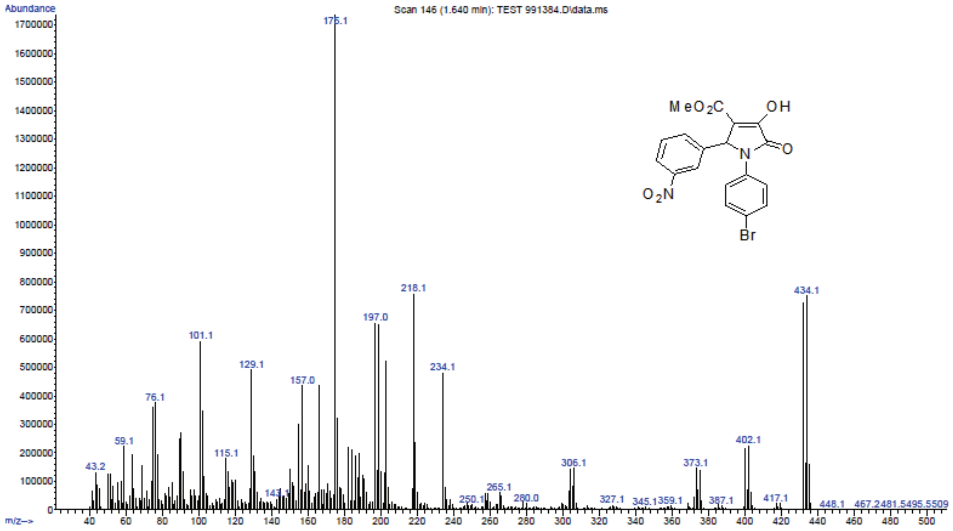

Fig. S-39. Mass spectrum of compound **14**.

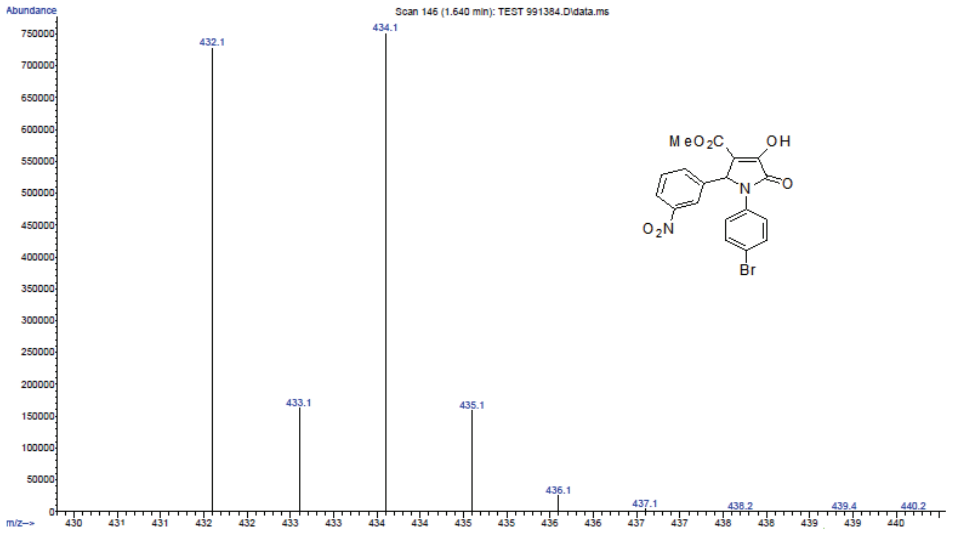

Fig. S-40. Mass spectrum of compound **14**.