

## **Fe<sub>3</sub>O<sub>4</sub>@NH<sub>2</sub>@Oxalic Acid: A Convenient Catalyst for Synthesis of Pyrrolinone Derivatives**

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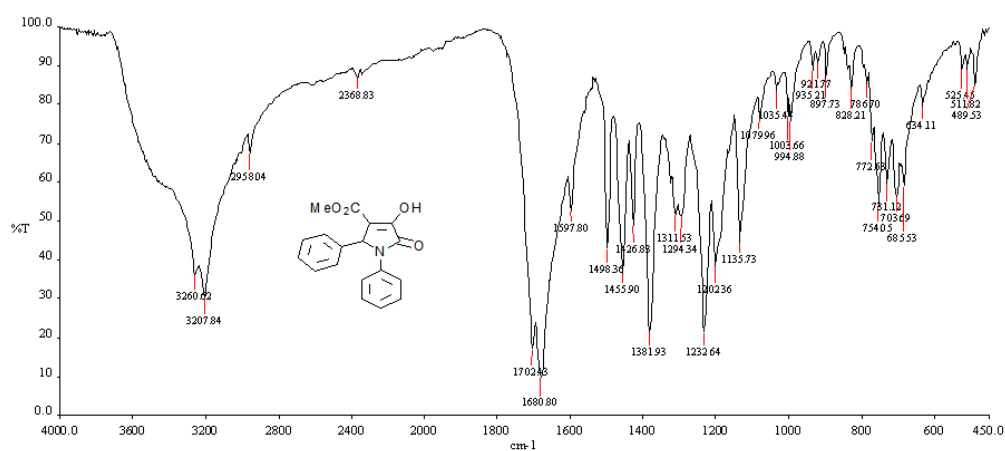
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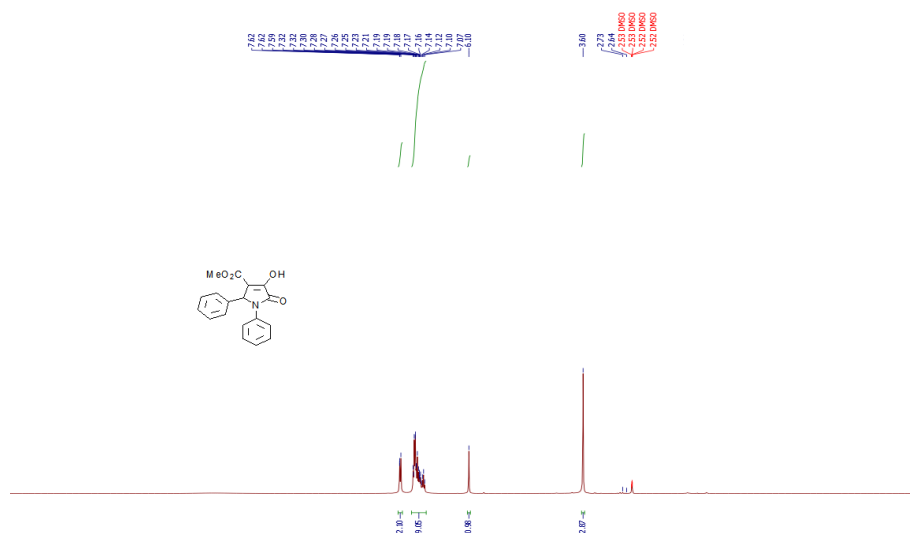
## **Supplementary Information**

**1,5-diphenyl-3-hydroxy-4-methoxycarbonyl-pyrrolin-2-one (1a)**

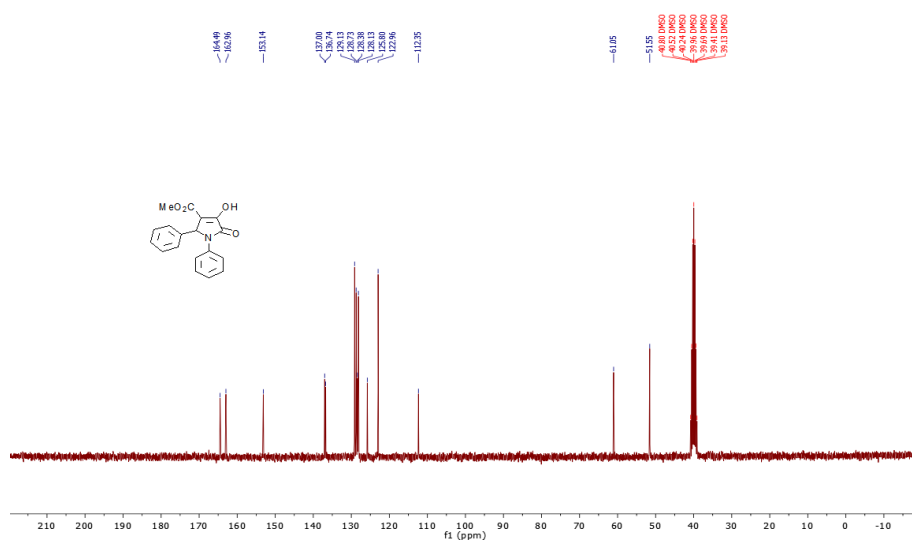
White powder, 8 hours, yields: 95 %, mp 182-183 °C (180-182 [20]), IR (KBr)( $\text{cm}^{-1}$ ): 3260 (OH), 2958, 1702 (C=O), 1680 (C=O), 1498, 1382, 1232, 1002;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  3.60 (s, 3H,  $\text{CH}_3$ ), 6.10 (s, 1H, CH), 7.07–7.62 (m, 10 H, Ar and br s, 1H, OH);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  50.55, 61.05, 112.35, 122.96, 125.80, 128.13, 128.38, 128.73, 129.13, 136.74, 137.00, 153.14, 162.96 (C=O), 164.49 (C=O). Anal. Calcd for  $\text{C}_{18}\text{H}_{15}\text{NO}_4$ : C, 69.89; H, 4.89; N, 4.53. Found: C, 69.92; H, 4.81; N, 4.48.



**Fig. S1.** FT-IR (KBr,  $\text{cm}^{-1}$ ) spectrum of compound **1a**.



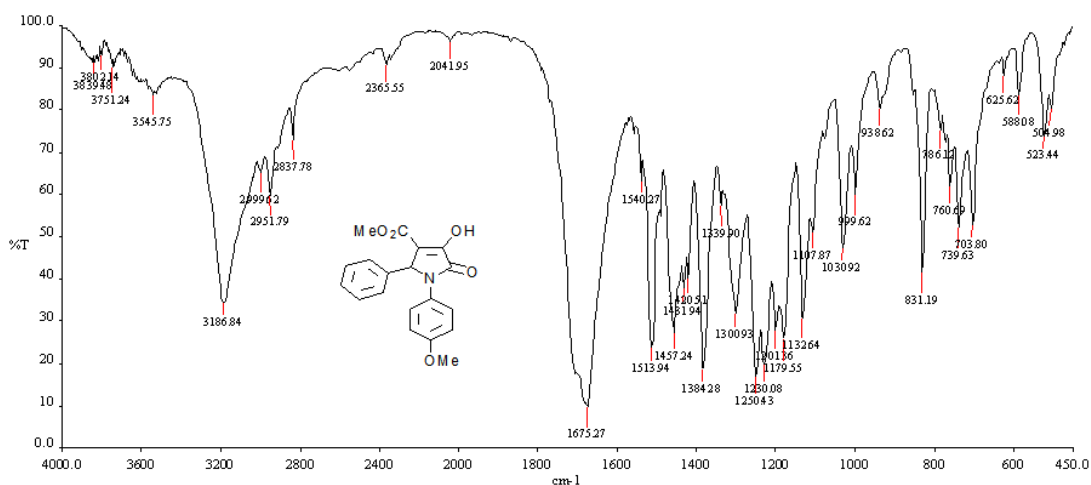
**Fig. S2.**  $^1\text{H}$ -NMR (400 MHz,  $\text{CDCl}_3$ ,  $\delta/\text{ppm}$ ) spectrum of compound **1a**.



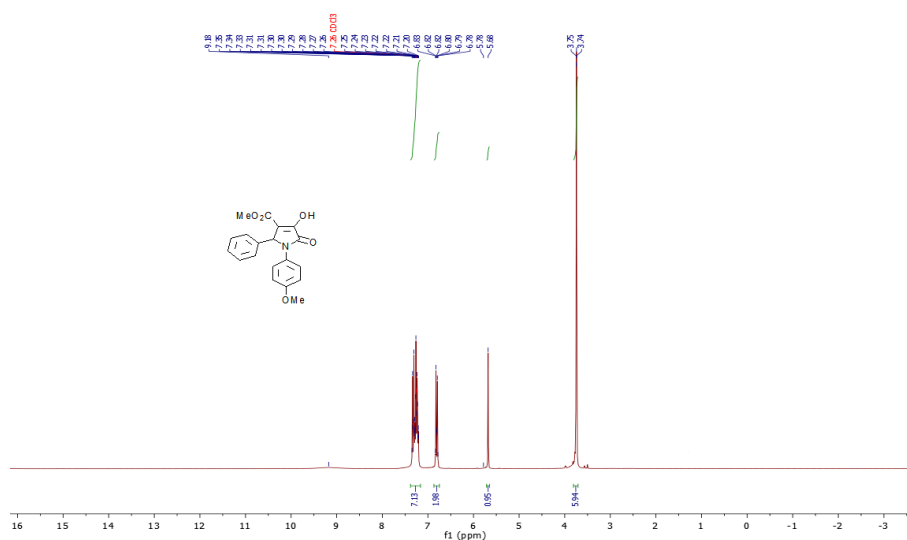
**Fig. S3.**  $^{13}\text{C}$ -NMR (100 MHz,  $\text{CDCl}_3$ ,  $\delta/\text{ppm}$ ) spectrum of compound **1a**.

**1-(4-methoxyphenyl)-5-phenyl-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (2a)**

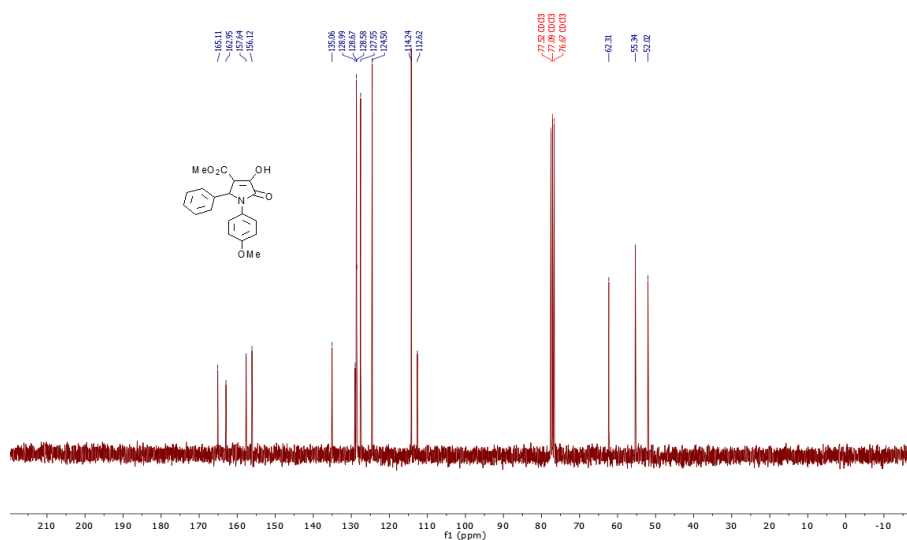
The cream powder, 6 hours, yields: 90 %, mp 151-152 °C (152-154 [30]). IR (KBr)( $\text{cm}^{-1}$ ): 3186 (OH), 2951, 1702 (C=O), 1675 (C=O), 1513, 1457, 1384, 1300, 1250, 831;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  3.74 (s, 6H,  $2 \times \text{OCH}_3$ ), 5.68 (s, 1H, CH), 6.78–7.35 (m, 9 H, Ar), 9.18 (br s, 1H, OH).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$  52.04, 55.34, 62.31, 112.62, 114.24, 127.55, 128.58, 128.67, 128.99, 135.06, 156.12, 157.64, 162.95 (C=O), 165.11 (C=O). Anal. Calcd for  $\text{C}_{19}\text{H}_{17}\text{NO}_5$ : C, 67.25; H, 5.05; N, 4.13. Found: C, 67.42; H, 5.15; N, 4.05.



**Fig. S4.** FT-IR (KBr,  $\text{cm}^{-1}$ ) spectrum of compound **2a**.



**Fig. S5.**  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ,  $\delta/\text{ppm}$ ) spectrum of compound **2a**.



**Fig. S6.**  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ ,  $\delta/\text{ppm}$ ) spectrum of compound **2a**.

**1-(4-methoxyphenyl)-5-(3-bromophenyl)-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (3a)**

The cream powder, 6 hours, yields: 95 %, mp 172–173 °C (170–171 °C [35]). IR (KBr)( $\text{cm}^{-1}$ ): 3198 (OH), 2954, 1707 (C=O), 1683 (C=O), 1514, 1385, 1254, 1132.  $^1\text{H NMR}$  ( $\text{CDCl}_3$ )  $\delta$  3.76 (s, 3H,  $\text{OCH}_3$ ), 3.78 (s, 3H,  $\text{OCH}_3$ ), 5.63 (s, 1H, CH), 6.81–7.40 (m, 8H, Ar), 9.15 (br s, 1H, OH).  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ )  $\delta$  52.15, 55.39, 61.60, 112.20, 114.40, 122.62, 124.48, 126.31, 128.64, 130.25, 130.49, 131.81, 137.54, 156.27, 157.82, 162.78 (C=O), 164.86 (C=O). Anal. Calcd. for  $\text{C}_{19}\text{H}_{16}\text{BrNO}_5$ : C, 54.56; H, 3.86; Br, 19.10; N, 3.35. Found: C, 54.82; H, 3.65; Br, 19.29; N, 3.51.

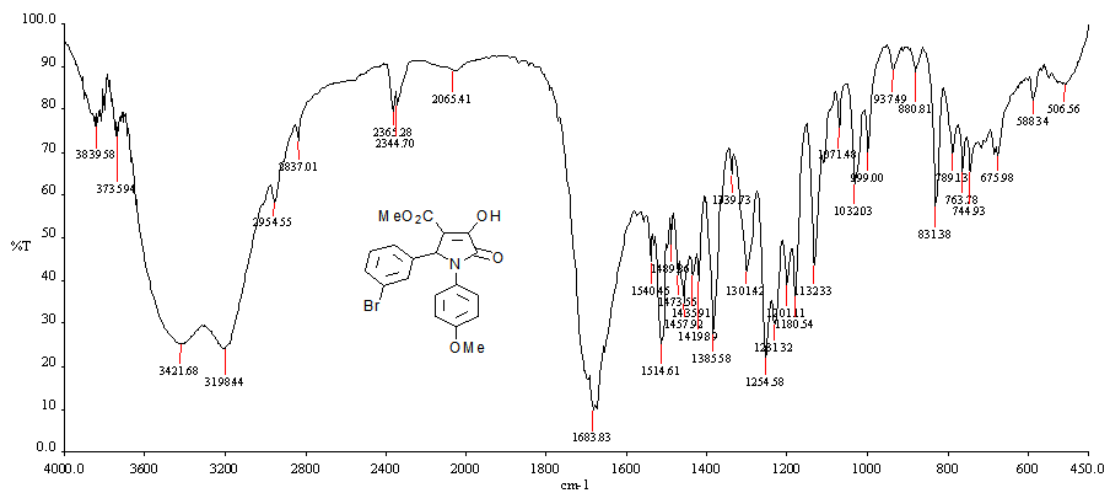


Fig. S7. FT-IR (KBr, cm<sup>-1</sup>) spectrum of compound 3a.

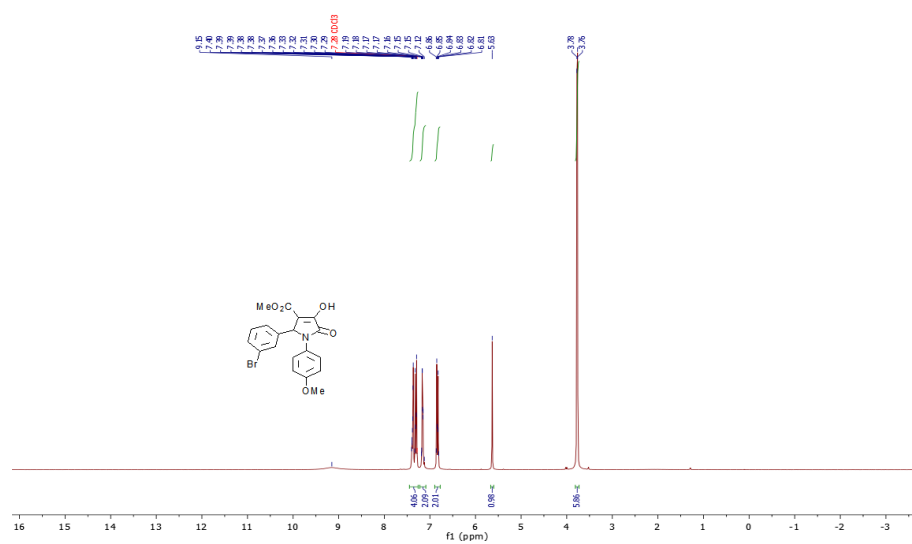
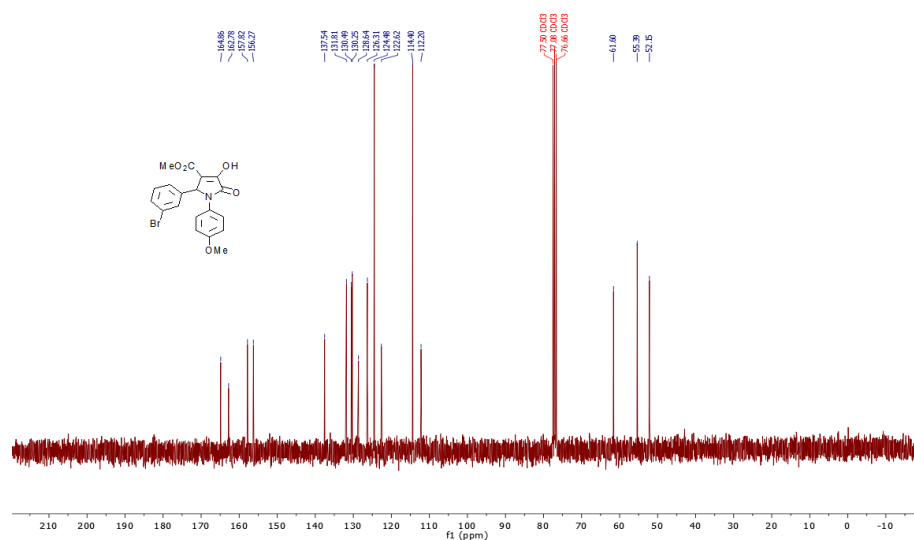


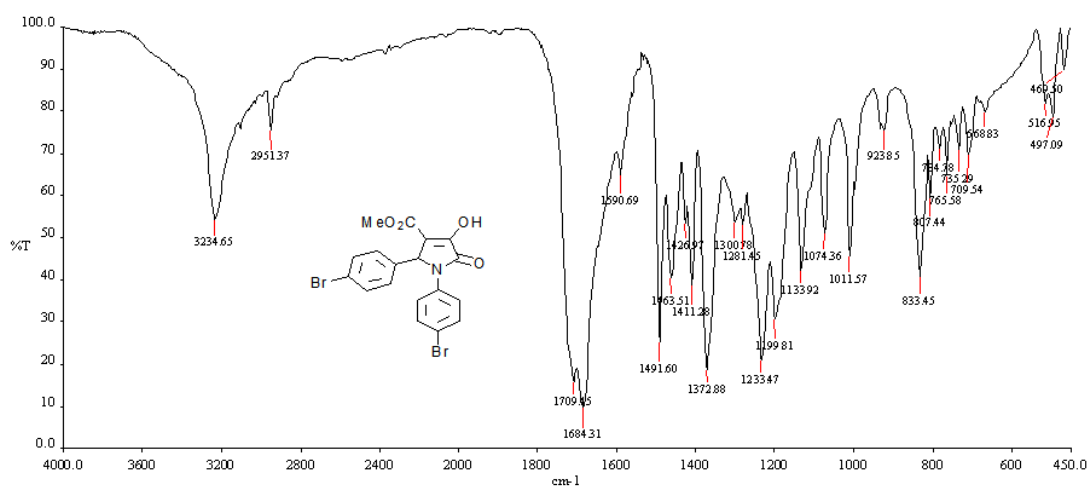
Fig. S8. <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>, δ/ppm) spectrum of compound 3a.



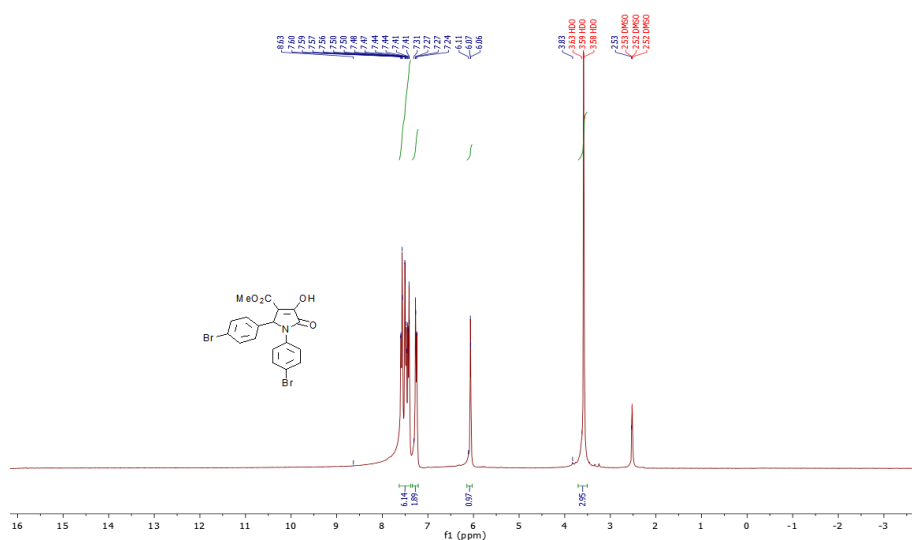
**Fig. S9.**  $^{13}\text{C}$ -NMR (100 MHz,  $\text{CDCl}_3$ ,  $\delta/\text{ppm}$ ) spectrum of compound **3a**.

**1-(4-bromophenyl)-5-(4-bromophenyl)-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (4a)**

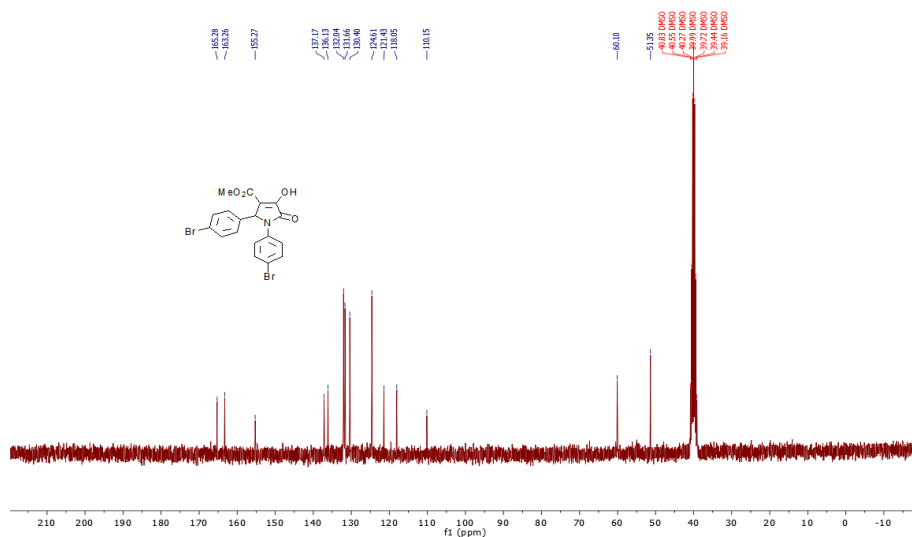
The white powder, 10 hours, yields: 93 %, mp 217-219 °C (218-220 [34b]). IR (KBr)( $\text{cm}^{-1}$ ): 3234 (OH), 2951, 1709 (C=O), 1684 (C=O), 1491, 1372, 1233, 1133.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  3.59 (s, 3H,  $\text{OCH}_3$ ), 6.07(s, 1H, CH), 7.24–8.63 (m, 8 H, Ar).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$  51.35, 60.10, 110.15, 118.05, 121.43, 124.61, 130.40, 131.66, 132.04, 136.13, 137.17, 155.27, 163.26 (C=O), 165.28 (C=O). Anal. Calcd for  $\text{C}_{18}\text{H}_{13}\text{Br}_2\text{NO}_4$ : C, 46.28; H, 2.81, N, 3.00. Found: C, 46.33; H, 2.75; N, 2.95.



**Fig. S10.** FT-IR (KBr,  $\text{cm}^{-1}$ ) spectrum of compound **4a**.



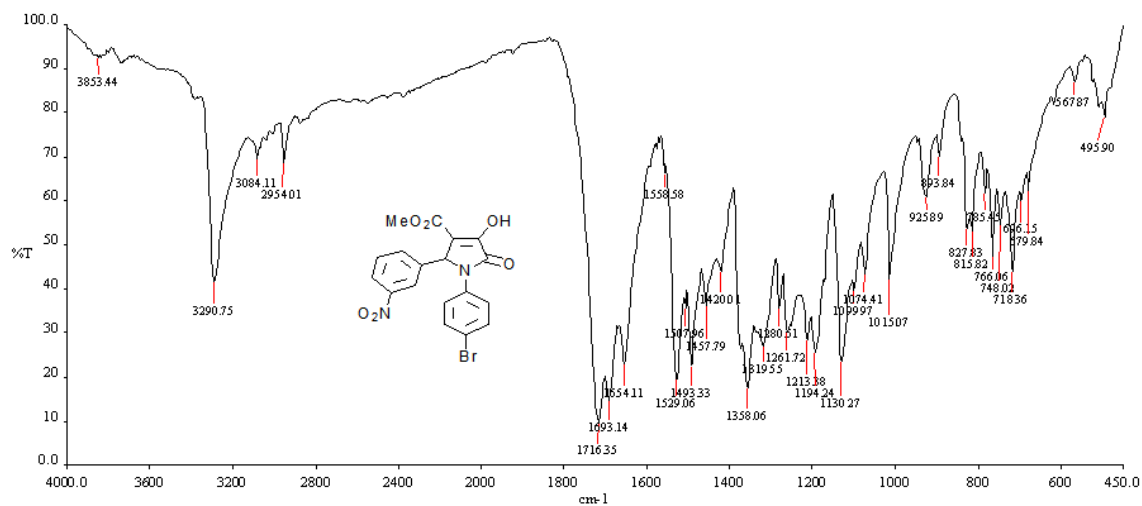
**Fig. S11.**  $^1\text{H-NMR}$  (400 MHz,  $d_6$ -DMSO,  $\delta/\text{ppm}$ ) spectrum of compound **4a**.



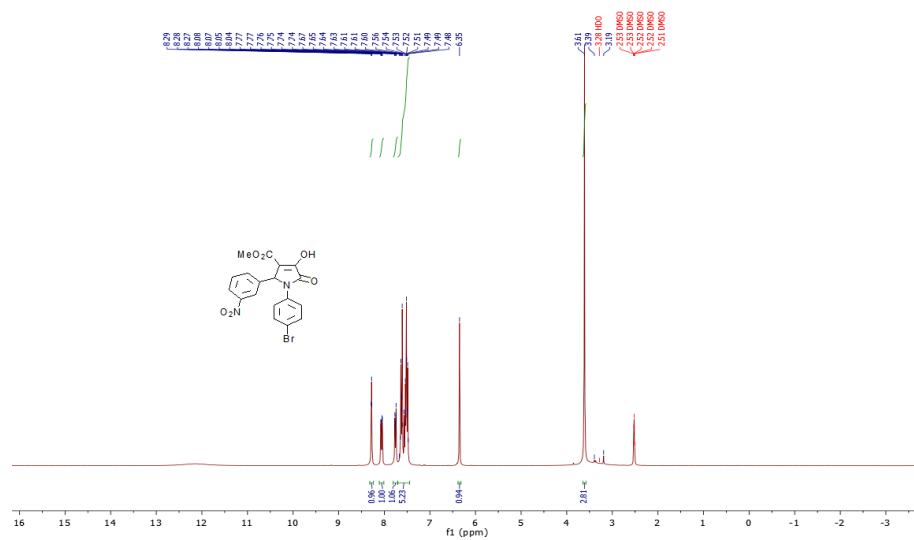
**Fig. S12.**  $^{13}\text{C-NMR}$  (100 MHz,  $d_6$ -DMSO,  $\delta/\text{ppm}$ ) spectrum of compound **4a**.

**1-(4-bromophenyl)-5-(3-nitrophenyl)-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (5a)**

White solid, 10 hours, Yield: 0.39 g (91 %). m.p. 171-173 °C (170-171 °C [34a]). FT-IR (KBr):  $\nu_{\text{max}}(\text{cm}^{-1})$ : 3290 (OH), 2952, 1716 (C=O), 1693 (C=O), 1529 (NO<sub>2</sub>), 1493, 1358 (NO<sub>2</sub>), 1213, 1190;  $^1\text{H-NMR}$  (250.13 MHz, CDCl<sub>3</sub>)  $\delta\text{H}$ : 3.61 (s, 3H, OCH<sub>3</sub>), 6.35 (s, 1H, CH), 7.48–8.29 (m, 8 H, Ar). The  $^{13}\text{C-NMR}$  (100.51 MHz,  $d_6$ -DMSO)  $\delta\text{C}$ : 51.74, 59.88, 111.61, 118.44, 123.57, 123.69, 124.76, 130.47, 132.22, 134.44, 135.68, 139.47, 148.07, 153.52, 162.84 (C=O), 164.45 (C=O). Anal. Calcd for C<sub>18</sub>H<sub>13</sub>BrN<sub>2</sub>O<sub>6</sub>: C, 49.90; H, 3.02; Br, 18.44; N, 6.47. Found: C, 49.80; H, 3.05; Br, 18.35; N, 6.48.

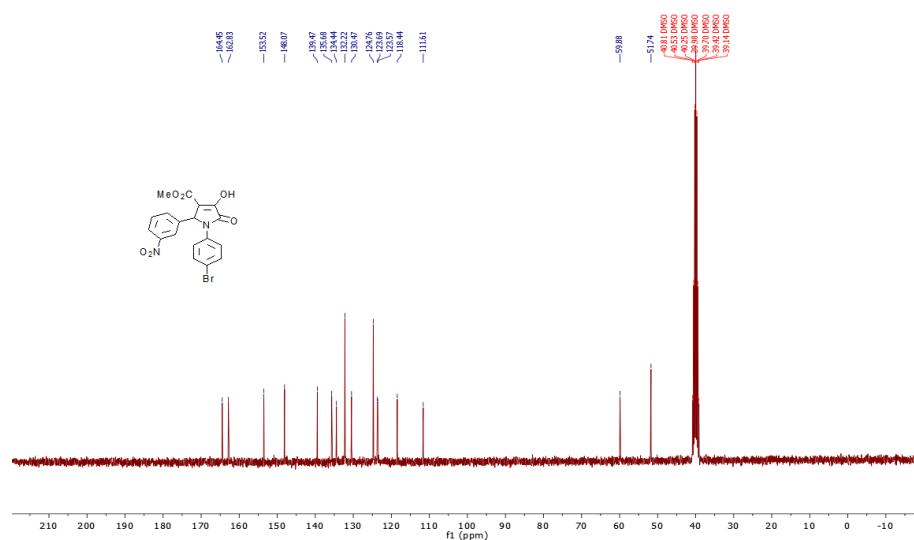


**Fig. S13.** FT-IR (KBr, cm<sup>-1</sup>) spectrum of compound **5a**.



**Fig. S14.** <sup>1</sup>H-NMR (400 MHz, d<sub>6</sub>-DMSO, δ/ppm) spectrum of compound **5a**.

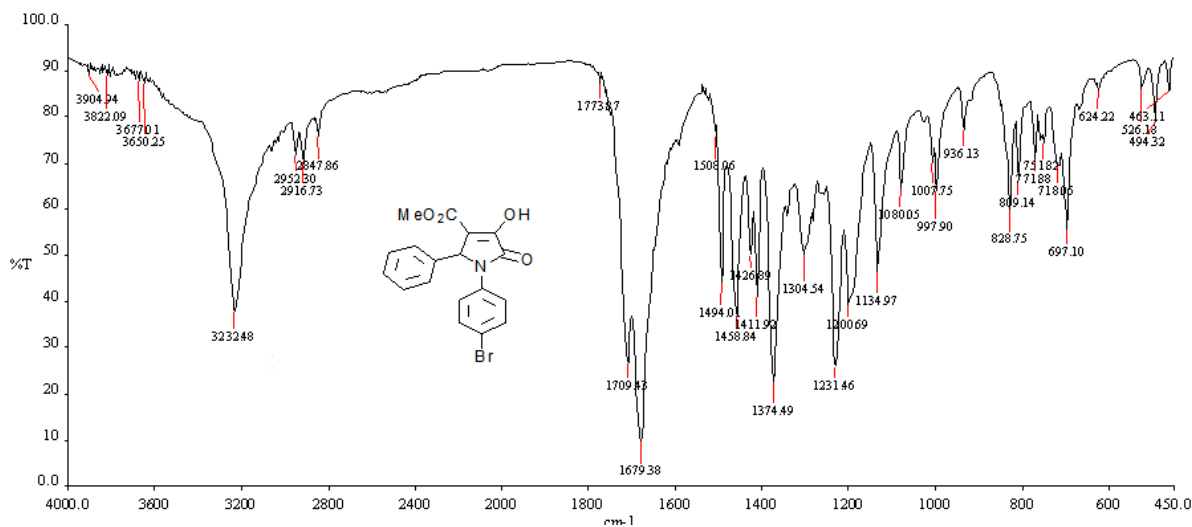




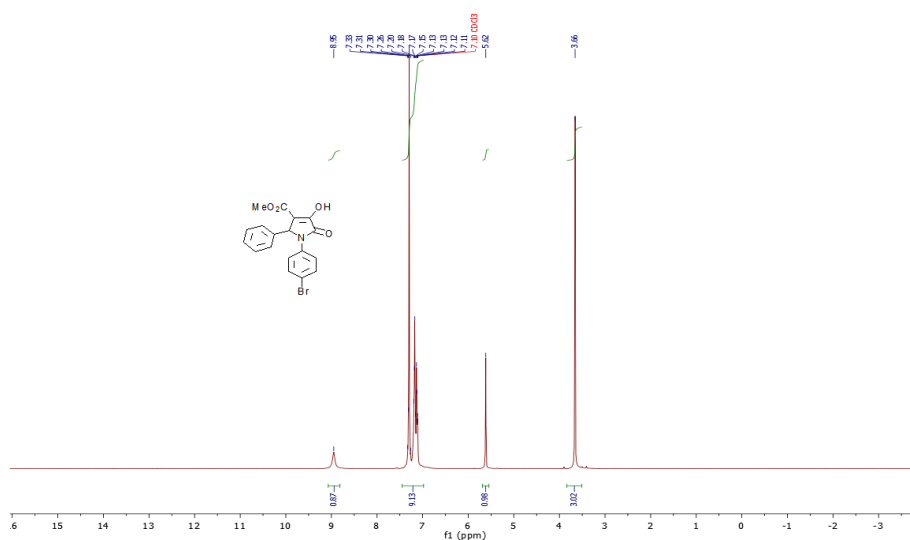
**Fig. S15.**  $^{13}\text{C}$ -NMR (100 MHz,  $d_6$ -DMSO,  $\delta$ /ppm) spectrum of compound **5a**.

**1-(4-bromophenyl)-5-(phenyl)-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (6a)**

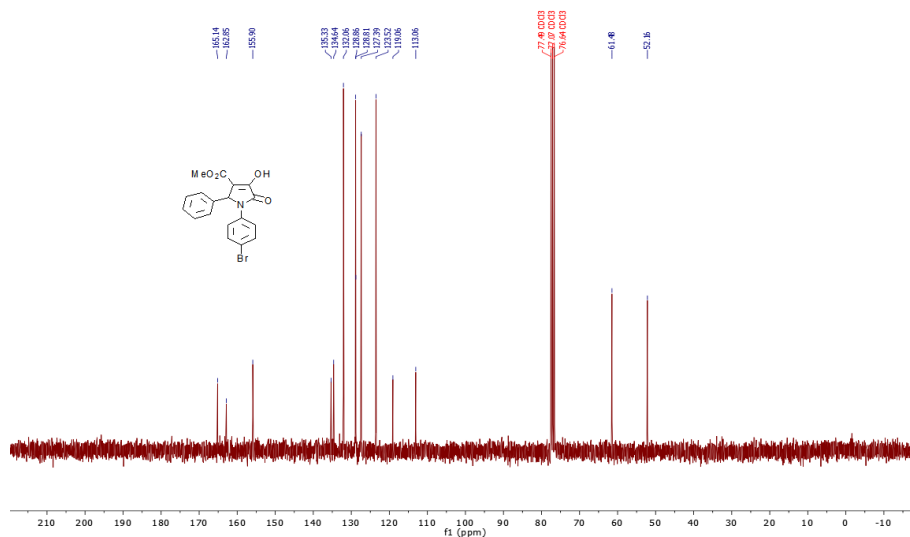
White powder, 10 hours, yields: 94 %, mp 172-173 °C (170-171 °C [35]). IR (KBr)( $\text{cm}^{-1}$ ): 3232 (OH), 2952, 1709 (C=O), 1679 (C=O), 1494, 1374, 1231, 1134;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  3.66 (s, 3H,  $\text{OCH}_3$ ), 5.62 (s, 1H, CH), 7.11–7.33 (m, 9 H, Ar).  $^{13}\text{C}$ -NMR ( $\text{CDCl}_3$ )  $\delta$  52.16, 61.48, 113.06, 119.06, 123.52, 127.39, 128.81, 132.06, 134.64, 135.33, 155.90, 162.85 (C=O), 165.14 (C=O). Anal. Calcd. for  $\text{C}_{18}\text{H}_{14}\text{BrNO}_4$ : C, 55.69; H, 3.63; Br, 20.58; N, 3.61. Found: C, 55.89; H, 3.73; Br, 20.88; N, 3.44.



**Fig. S16.** FT-IR (KBr,  $\text{cm}^{-1}$ ) spectrum of compound **6a**.



**Fig. S17.**  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ,  $\delta/\text{ppm}$ ) spectrum of compound **6a**.



**Fig. S18.**  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ ,  $\delta/\text{ppm}$ ) spectrum of compound **6a**.

**1-(4-methoxyphenyl)-5-(2-chlorophenyl)-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (7a)**

The brown powder, 6 hours, yields: 90 %, mp 192–193 °C (192–194 [34a]). IR (KBr)( $\text{cm}^{-1}$ ): 3198 (OH), 2954, 1701 (C=O), 1684 (C=O), 1533, 1351, 1250, 1029.  $^1\text{H NMR}$  ( $\text{CDCl}_3$ )  $\delta$  3.73 (s, 6H,  $\text{OCH}_3$ ), 6.41 (s, 1H, CH), 6.77–7.41 (m, 8 H, Ar), 9.11 (br s, 1H, OH).  $^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ )  $\delta$  52.07, 55.33, 57.13, 112.45, 114.27, 123.80, 126.94, 127.47, 128.83, 129.70, 132.70, 134.99, 156.73, 157.61, 162.84 (C=O), 165.03 (C=O). Anal. Calcd. for  $\text{C}_{19}\text{H}_{16}\text{ClNO}_5$ : C, 61.05; H, 4.31; Cl, 9.48; N, 3.75. Found: C, 61.22; H, 4.43; Cl, 9.26; N, 3.98.

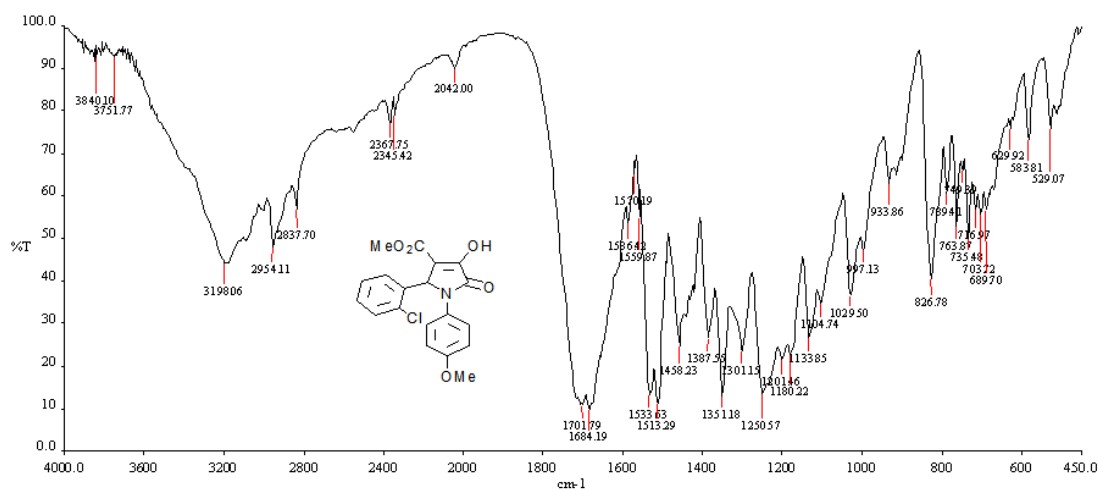


Fig. S19. FT-IR (KBr, cm<sup>-1</sup>) spectrum of compound 7a.

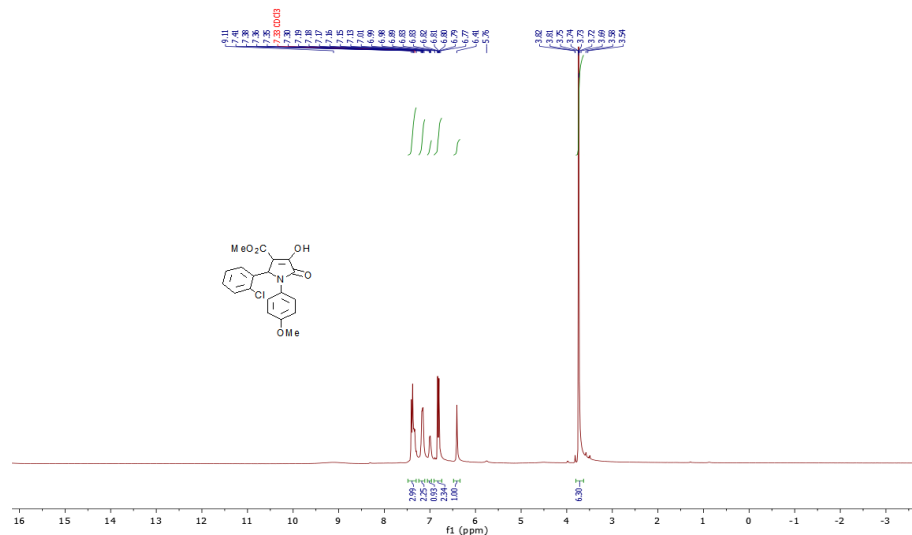
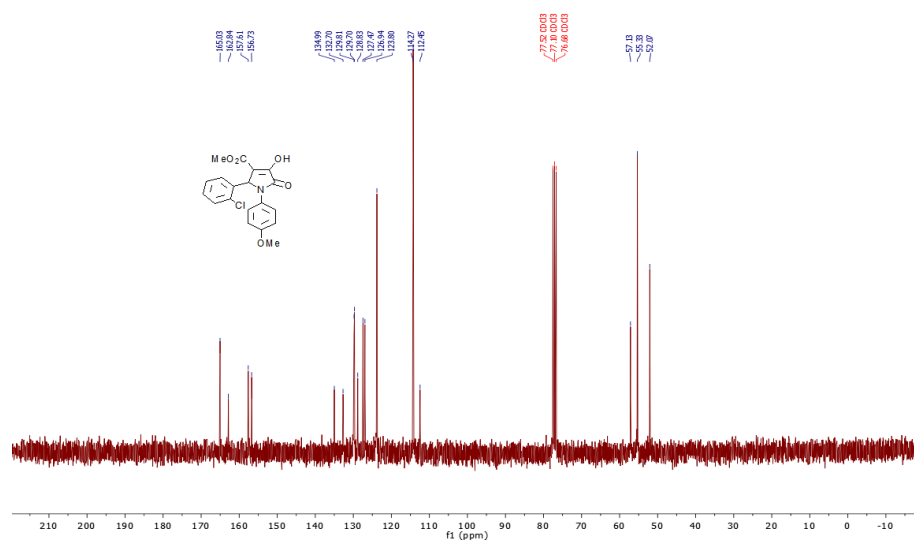


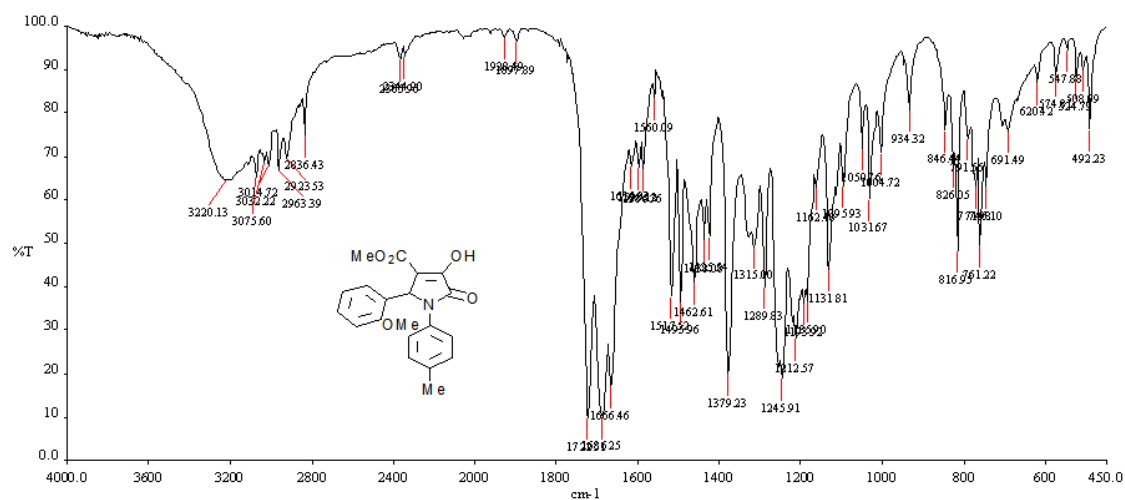
Fig. S20. <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>, δ/ppm) spectrum of compound 7a.



**Fig. S21.**  $^{13}\text{C}$ -NMR (100 MHz,  $\text{CDCl}_3$ ,  $\delta/\text{ppm}$ ) spectrum of compound **7a**.

**1-(4-methylphenyl)-5-(2-methoxyphenyl)-3-hydroxy-4-methoxycarbonyl-3-pyrrolin-2-one (**8a**)**

White solid, 8 hours, Yield: 0.31 g (92 %). m.p. 172-173 °C (173-175 °C [34b]). FT-IR (KBr):  $\nu_{\text{max}}(\text{cm}^{-1})$ : 3220 (OH), 2963, 1720 (C=O), 1686 (C=O), 1513, 1379, 1245, 1131;  $^1\text{H}$ -NMR (250.13 MHz,  $\text{CDCl}_3$ )  $\delta\text{H}$ : 2.28 (s, 3H,  $\text{CH}_3$ ), 3.73 (s, 3H,  $\text{OCH}_3$ ), 3.90 (s, 3H,  $\text{OCH}_3$ ), 6.29 (s, 1H, CH), 6.82–7.46 (m, 8 H, Ar), 9.14 (br s, 1H, OH). The  $^{13}\text{C}$ -NMR (62.90 MHz,  $\text{CDCl}_3$ )  $\delta\text{C}$ : 20.82, 51.97, 55.92, 111.40, 120.94, 121.88, 123.00, 129.37, 129.65, 133.85, 135.36, 156.48, 157.86, 163.11 (C=O), 165.31 (C=O). Anal. Calcd. for  $\text{C}_{20}\text{H}_{19}\text{NO}_5$ : C, 67.98; H, 5.42; N, 3.96. Found: C, 68.02; H, 5.48; N, 3.98.



**Fig. S22.** FT-IR (KBr,  $\text{cm}^{-1}$ ) spectrum of compound **8a**.

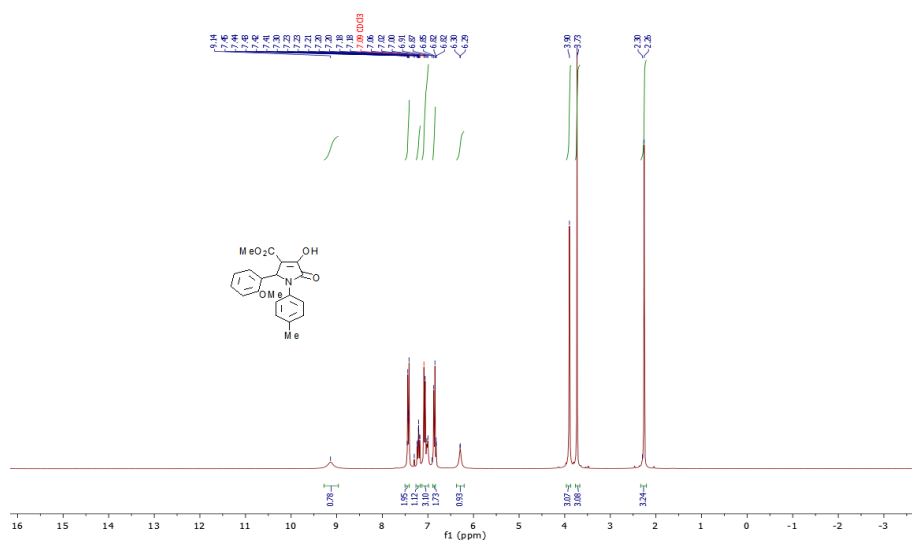


Fig. S23. <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>, δ/ppm) spectrum of compound 8a.

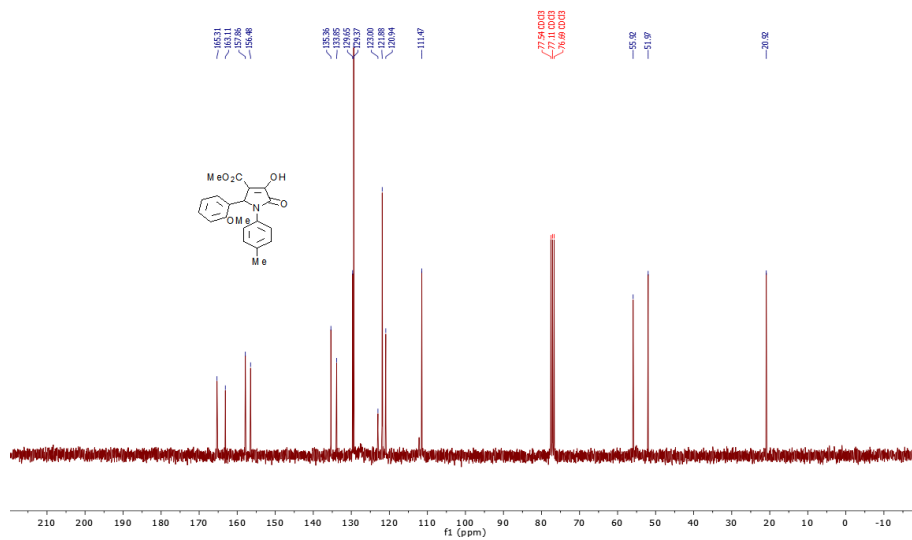
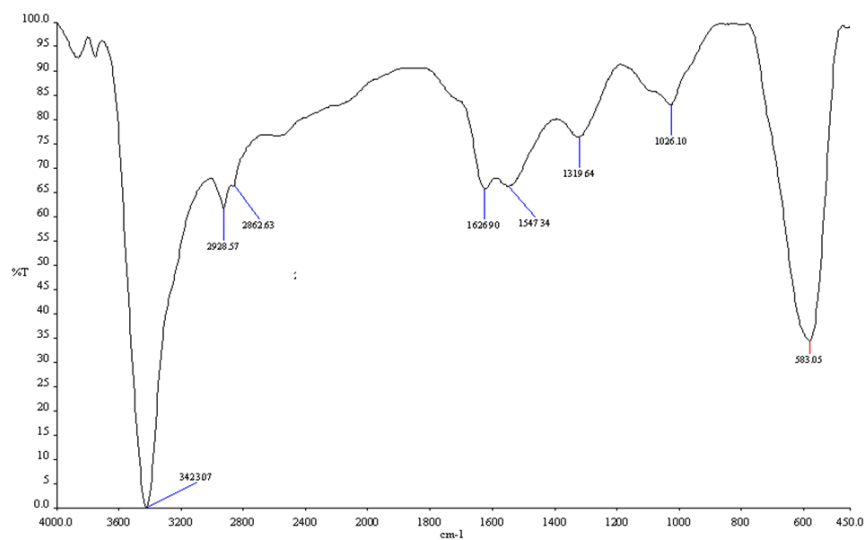
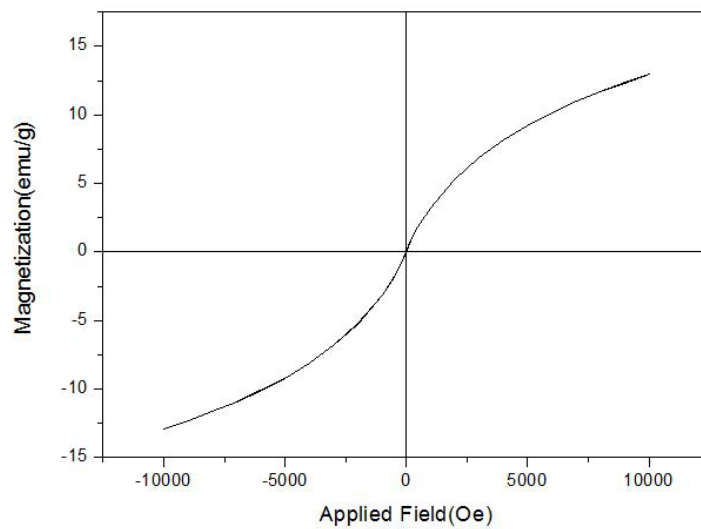


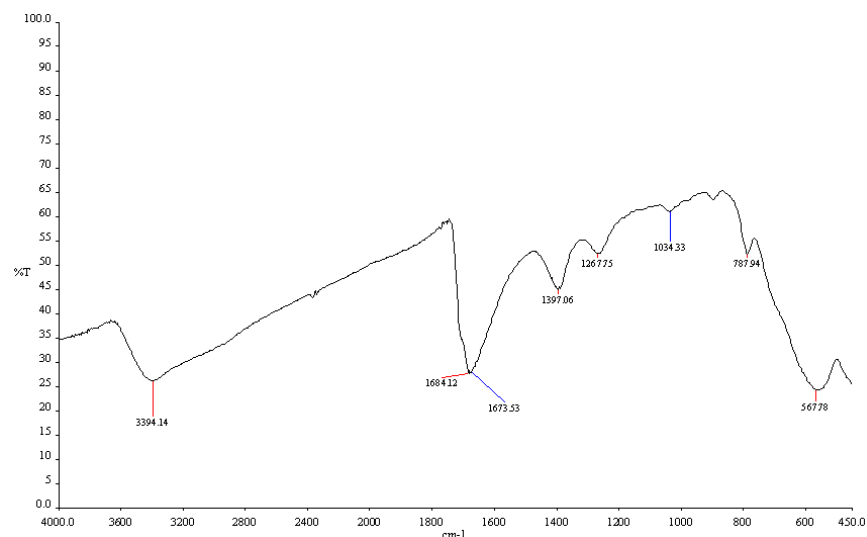
Fig. S24. <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>, δ/ppm) spectrum of compound 8a.



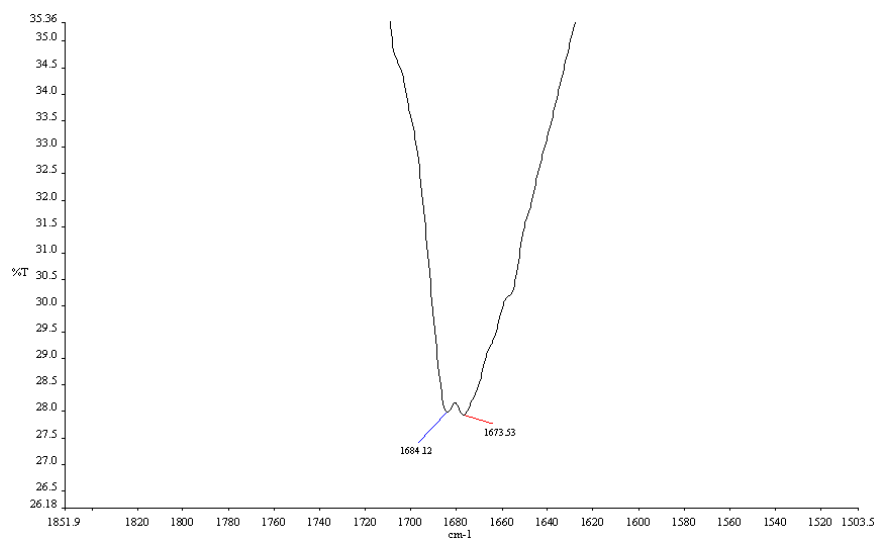
**Fig. S25.** FT-IR (KBr) spectrum of the Fe<sub>3</sub>O<sub>4</sub>@NH<sub>2</sub>.



**Fig. S26.** VSM spectrum of the Fe<sub>3</sub>O<sub>4</sub>@NH<sub>2</sub>



**Fig. S27.** FT-IR spectrum of the Fe<sub>3</sub>O<sub>4</sub>@NH<sub>2</sub>@Oxalic acid.



**Fig. S28.** FT-IR spectrum of the Fe<sub>3</sub>O<sub>4</sub>@NH<sub>2</sub>@Oxalic acid (carbonyl group expanded).

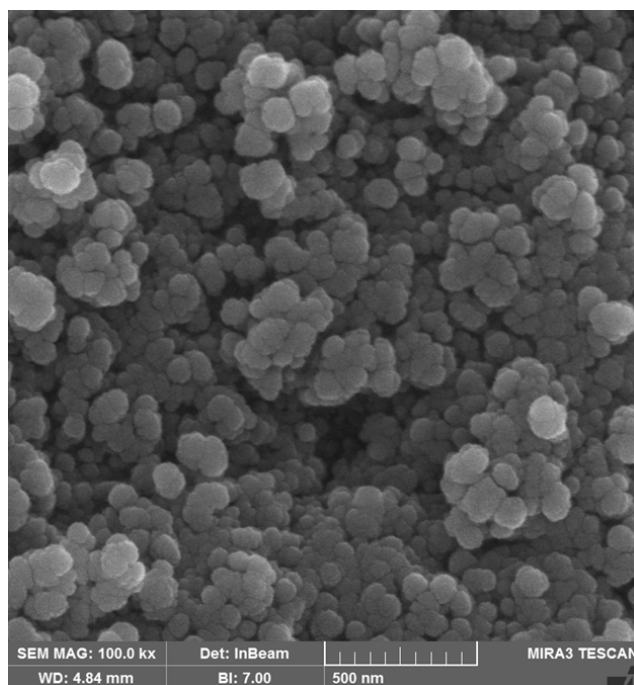


Fig. S29. SEM spectrum of the  $\text{Fe}_3\text{O}_4@\text{NH}_2@\text{Oxalic acid}$ .

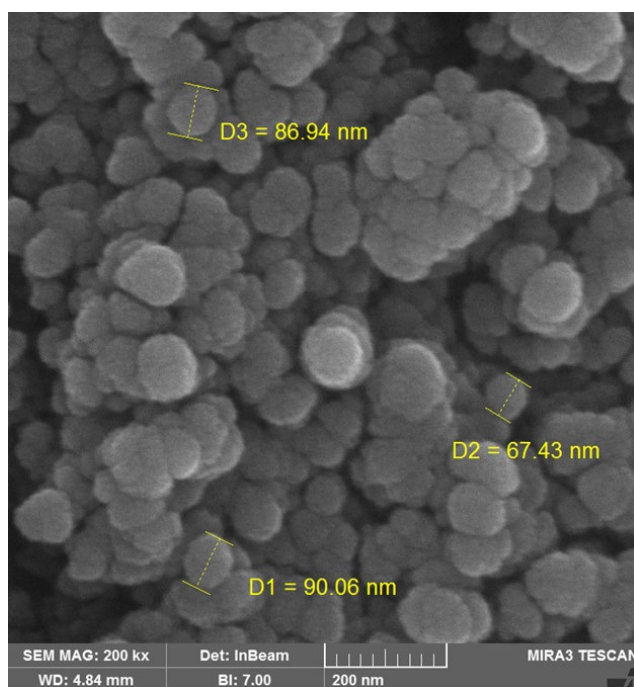
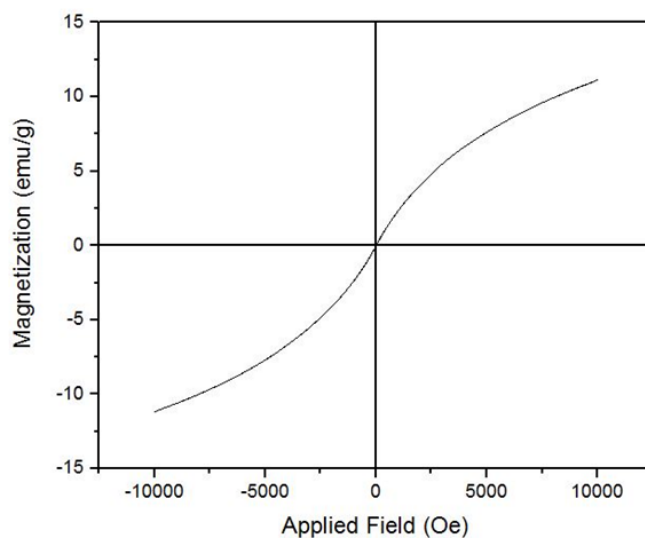
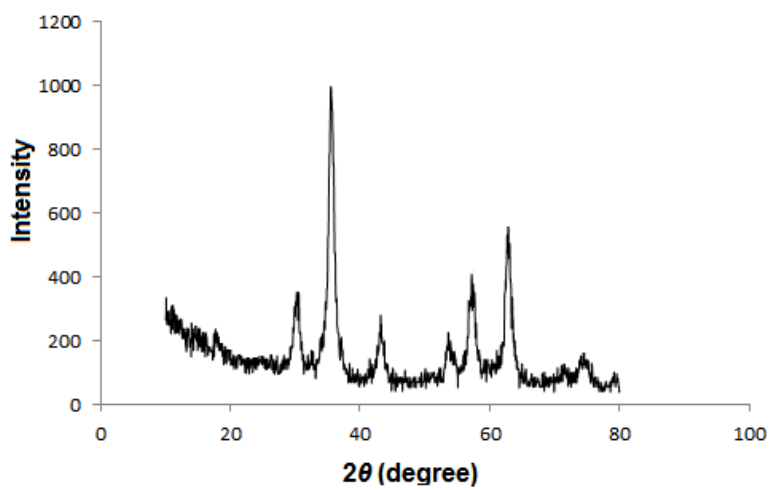


Fig. S30. SEM spectrum of the  $\text{Fe}_3\text{O}_4@\text{NH}_2@\text{Oxalic acid}$ .





**Fig. S31.** VSM spectrum of the  $\text{Fe}_3\text{O}_4@\text{NH}_2@\text{Oxalic acid}$ .



**Fig. S32.** XRD spectrum of the  $\text{Fe}_3\text{O}_4@\text{NH}_2@\text{Oxalic acid}$

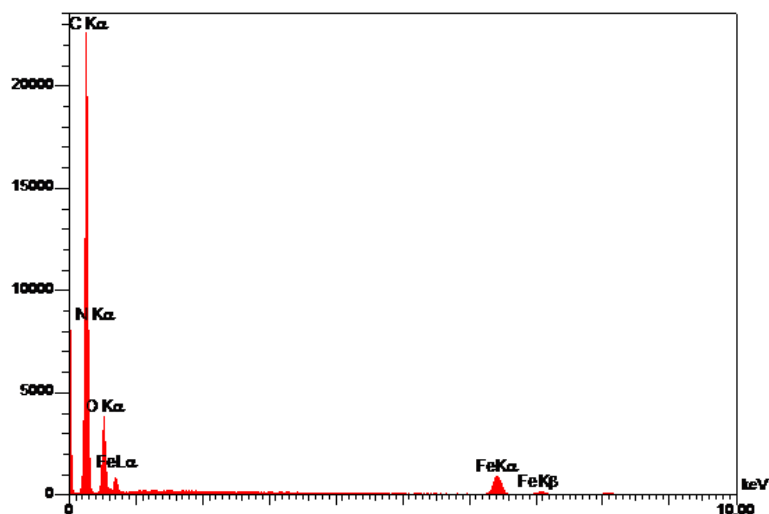


Fig. S33. EDAX spectrum of the Fe<sub>3</sub>O<sub>4</sub>@NH<sub>2</sub>@Oxalic acid.

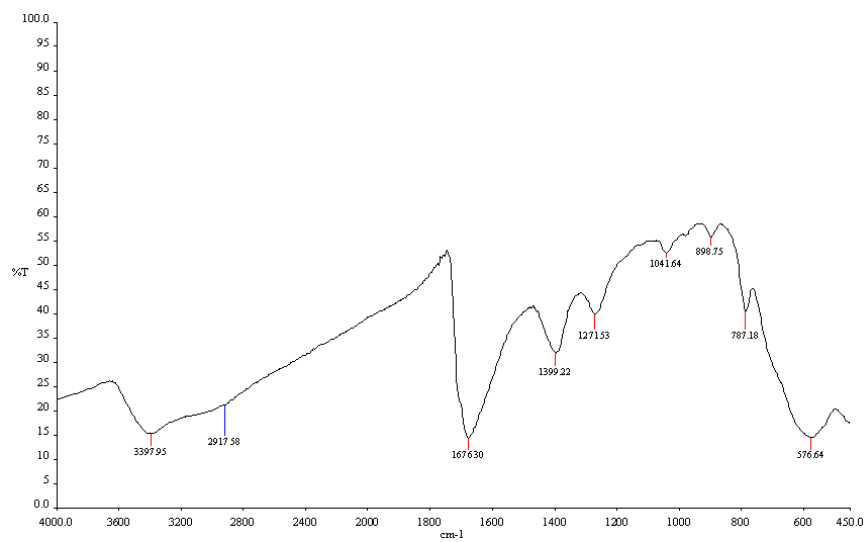


Fig. S34. FT-IR spectrum of the Fe<sub>3</sub>O<sub>4</sub>@NH<sub>2</sub>@Oxalic acid (Recovered).