

Supplementary Information

Synthesis of 3-Bromotetronamides via Amination of 3,4-Dibromofuran-2(5H)-one

Silvio Cunha,^{*,a,c} Caio C. Oliveira^{a,c} and José R. Sabino^b

^aInstituto de Química and ^cInstituto Nacional de Ciência e Tecnologia - INCT em Energia e Ambiente, Universidade Federal da Bahia, Campus de Ondina, 40170-290 Salvador-BA, Brazil

^bInstituto de Física, Universidade Federal de Goiás, CP 131, 74001-970 Goiânia-GO, Brazil

General information

Melting points were determined on a Microquímica MQAPF 301 hot plate apparatus and are uncorrected. Infrared spectra were recorded as KBr discs on a FT-IR BOMEM MB100 instrument. NMR spectra were obtained for ¹H at 300 MHz and for ¹³C at 75 MHz using a Varian Gemini 300 spectrometer. Chemical shifts are reported in ppm units downfield from reference (internal TMS).

Table S1. Physical data for compounds 3a-j

	3a: yellow solid, mp 128-129 °C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3260, 1766, 1029, 990; ¹ H NMR (CDCl ₃) δ 3.63-3.66 (m, 4H), 3.78-3.81 (m, 5H), 4.71 (s, 2H); ¹³ C NMR (CDCl ₃) δ 171.41, 159.26, 72.80, 67.52, 66.17, 47.10.		3f: brown solid, mp 100-105 °C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3300, 3072, 1727, 1632 1050; ¹ H NMR (CDCl ₃) δ 3.82 (s, 3H) 4.88 (s, 2H), 6.81-6.88 (m, 3H), 7.03-7.08 (m, 2H) 7.13 (sl, 1H); ¹³ C NMR (CDCl ₃) δ 169.50, 160.01, 150.30, 126.95, 126.06, 121.29, 119.42, 111.61, 78.41, 67.70, 56.14.
	3b: yellow solid, mp 165-167 °C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3204, 1745, 1637, 1179; ¹ H NMR (DMSO- <i>d</i> ₆) 4.97 (s, 1H), 7.09 (t, <i>J</i> 7.2 Hz, 1H), 7.17 (d, <i>J</i> 7.2 Hz, 2H) 7.28 (t, <i>J</i> 7.2 Hz, 2H), 9.47 (s, 1H), ¹³ C NMR (CDCl ₃) δ 170.22, 162.16, 138.73, 129.85, 125.74, 123.05, 74.66, 67.99.		3g: green solid, mp 152-154 °C (Condition A), or yellow solid, 135-138 °C (Condition B); IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3191, 3088, 2928, 1728, 1620, 1050; ¹ H NMR (CDCl ₃) δ 6.79 (s, 1H) 4.86 (s, 2H), 6.97 (d, <i>J</i> 8.7 Hz, 2H), 7.30 (d, <i>J</i> 8.7 Hz, 2H), 7.13 (s, 1H); ¹³ C NMR (DMSO- <i>d</i> ₆ /CDCl ₃) δ 170.17, 161.09, 137.25, 130.26, 129.48, 124.44, 75.74, 67.84.
	3c: brown solid, mp 146-148 °C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3229, 3072, 1730, 1627, 1049; ¹ H NMR (DMSO- <i>d</i> ₆) δ 2.19 (s, 3H) 4.91 (s, 2H), 7.07 (s, 4H), 9.36 (s, 1H); ¹³ C NMR (DMSO- <i>d</i> ₆) δ 170.23, 162.37, 136.10, 135.22, 130.25, 123.41, 73.98, 67.83, 21.04.		3h: green solid, mp 149-151 °C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3214, 3038, 1731, 1626, 1045; ¹ H NMR (DMSO- <i>d</i> ₆) δ 4.82 (s, 2H), 7.25-7.47 (m, 3H), 7.56-7.59 (m, 1H), 9.53 (s, 1H); ¹³ C NMR (DMSO- <i>d</i> ₆) δ 169.95, 161.314, 134.33, 130.17, 129.88, 129.22, 128.53, 127.87, 73.49, 67.30.
	3d: brown solid, mp 166-167 °C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3227, 3042, 2933; 2836, 1713, 1611, 1036; ¹ H NMR (DMSO- <i>d</i> ₆) δ 2.15 (s, 3H) 4.65 (s, 2H), 7.11-7.22 (m, 4H), 9.16 (s, 1H); ¹³ C NMR (DMSO- <i>d</i> ₆) δ 170.69, 162.91, 136.48, 135.23, 131.30, 127.92, 127.36, 127.17, 72.40, 67.57, 18.12.		3i: yellow solid, mp 166-169 °C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3456, 3360, 3230, 3028, 1701, 1616, 1042; ¹ H NMR (CDCl ₃) 3.90 (sl, 2H), 4.67 (s, 2H), 6.30 (sl, 1H), 6.77-6.85 (m, 2H), 7.05-7.08 (m, 1H), 7.16-7.21 (m, 1H); ¹³ C NMR (DMSO- <i>d</i> ₆) δ 170.86, 163.82, 154.24, 128.90, 128.45, 122.44, 116.75, 116.22, 72.27, 67.62.
	3e: green solid, mp 135-138 °C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3175, 3067, 1723, 1631, 1025; ¹ H NMR (CDCl ₃) δ 3.72 (s, 3H) 4.65 (s, 2H), 6.82 (d, <i>J</i> 10 Hz 2H), 6.85 (sl, 1H), 7.00 (d, <i>J</i> 10 Hz 2H); ¹³ C NMR (CDCl ₃) δ 170.17, 161.74, 158.58, 129.73, 125.40, 115.16, 76.00, 67.18, 55.75.		3j: brown solid, mp 151-155 °C; IR (KBr) $\nu_{\max}/\text{cm}^{-1}$: 3441, 3218, 1741, 1609, 1396, 1030; ¹ H NMR (DMSO- <i>d</i> ₆) δ 4.84 (s, 2H), 7.51-7.63 (m, 4H), 7.89-7.92 (dd, <i>J</i> 6.9 Hz and <i>J</i> 2.7 Hz, 1H), 7.98-8.02 (m, 2H); ¹³ C NMR (DMSO- <i>d</i> ₆) δ 170.18, 162.49, 133.80, 133.17, 129.43, 128.20, 127.32, 126.70, 126.63, 125.55, 123.76, 122.83, 72.65, 67.19.

*e-mail: silviadc@ufba.br

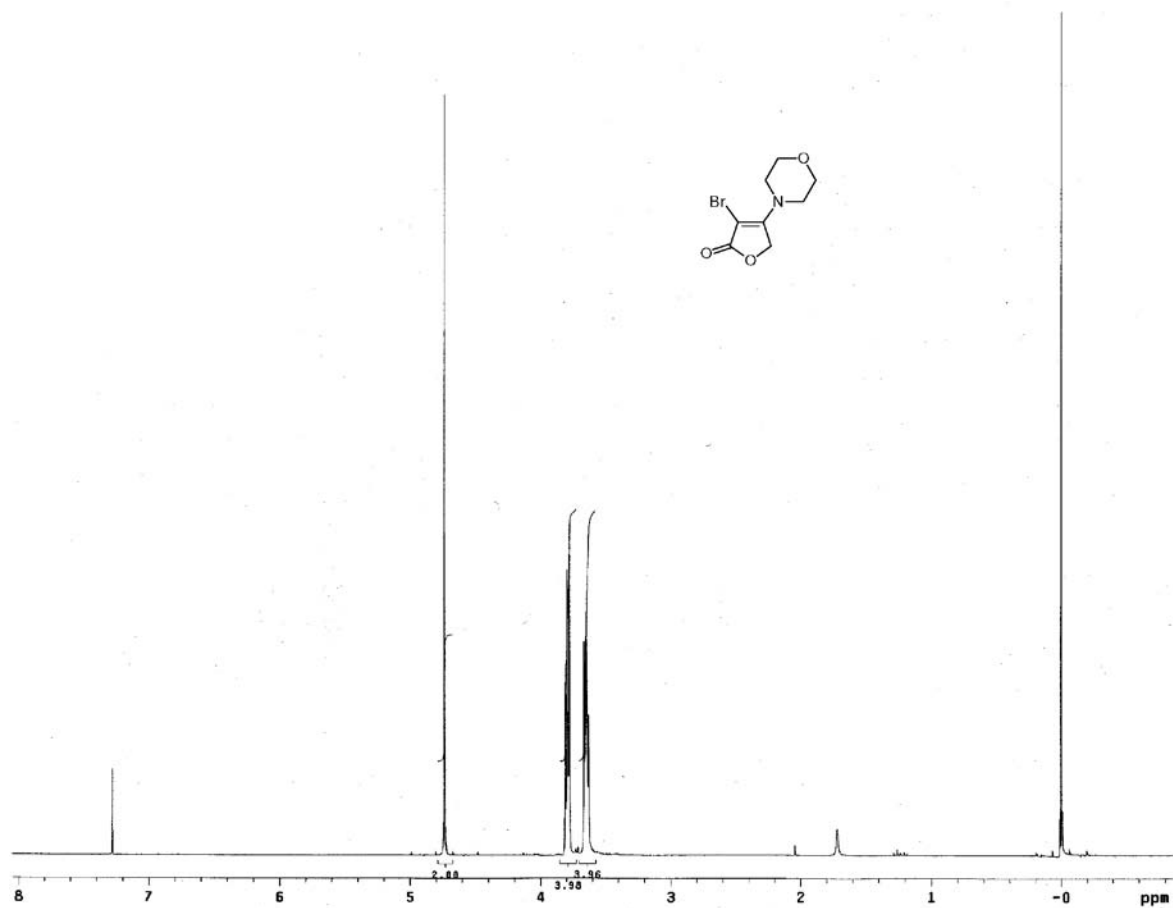


Figure S1. Full ¹H NMR spectrum of compound 3a (CDCl₃).

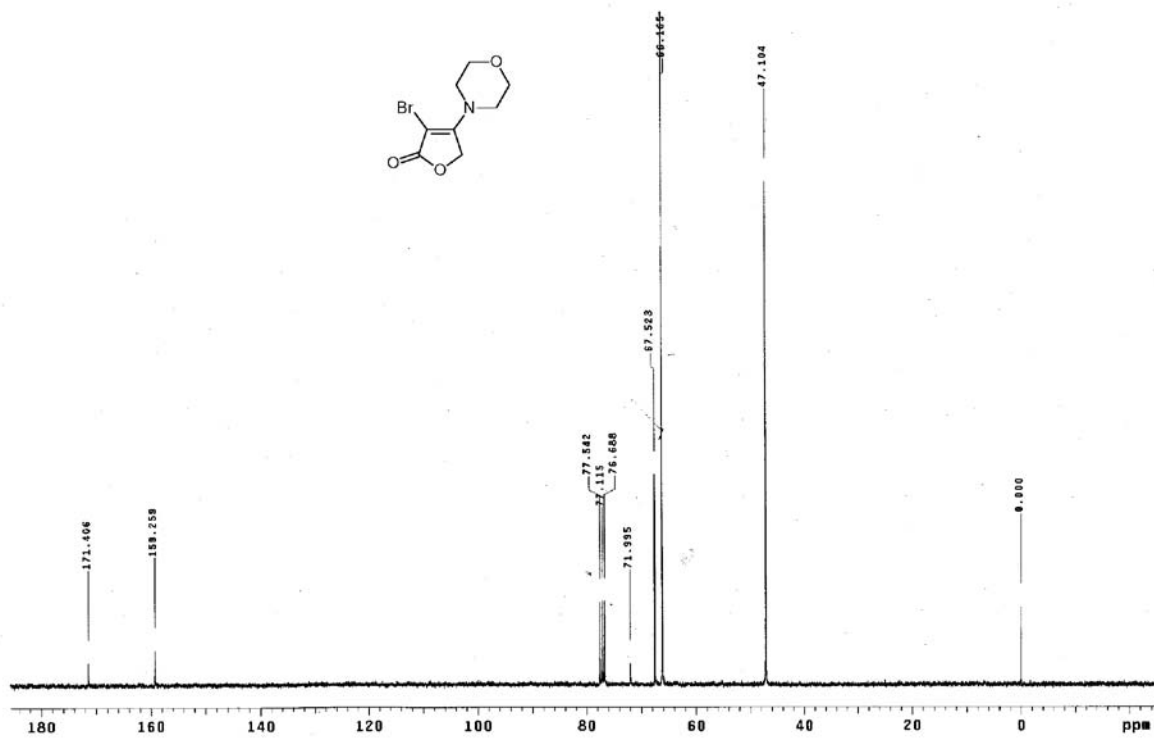


Figure S2. Full ¹³C NMR spectrum of compound 3a (CDCl₃).

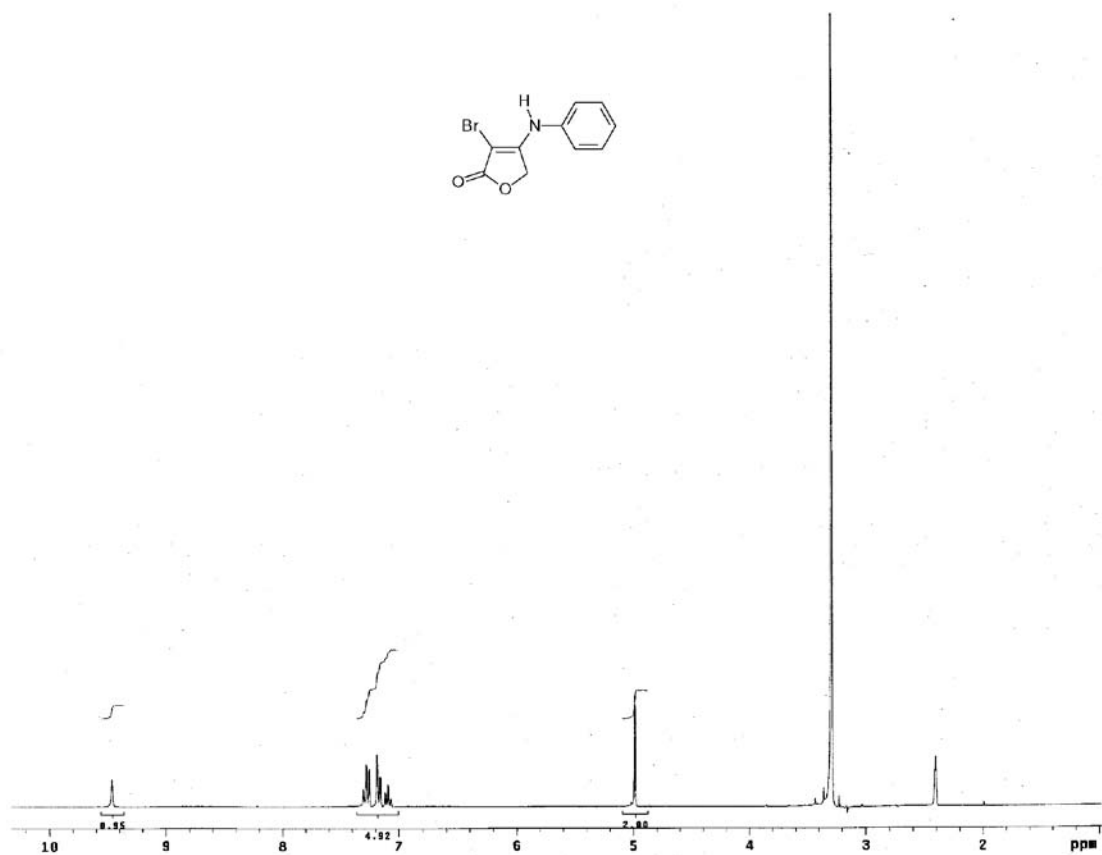


Figure S3. Full ^1H NMR spectrum of compound 3b (DMSO- d_6).

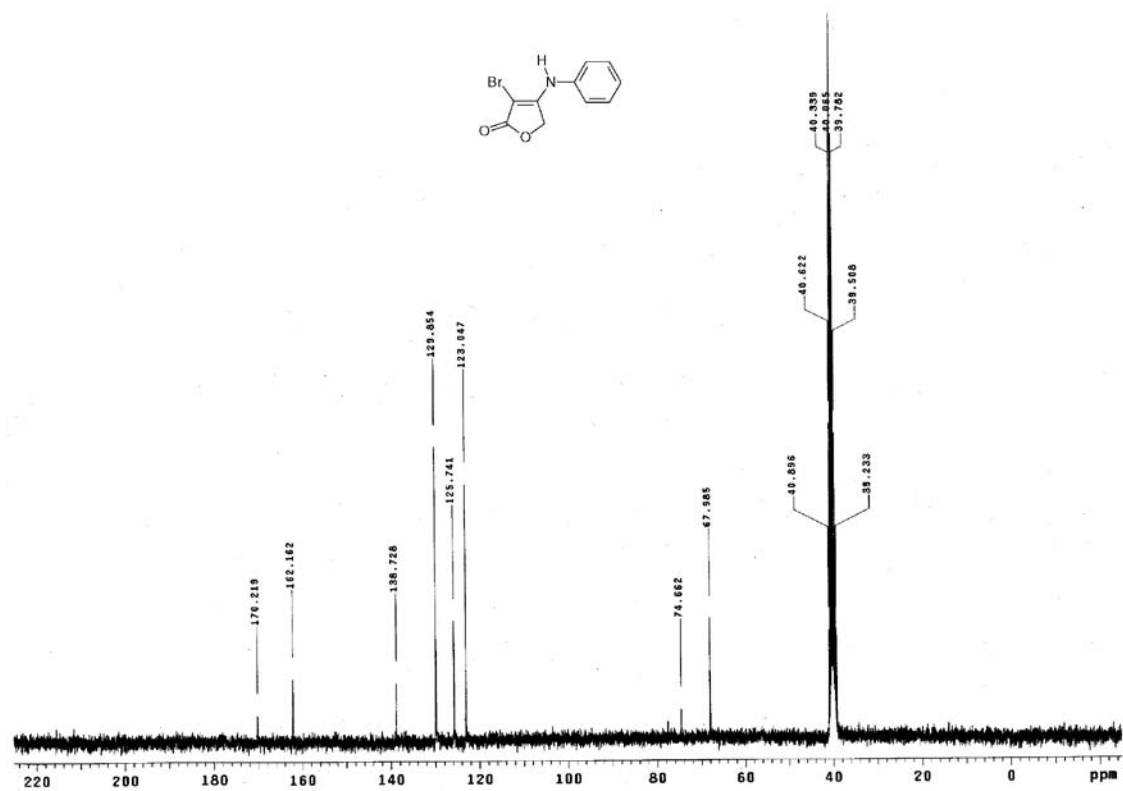
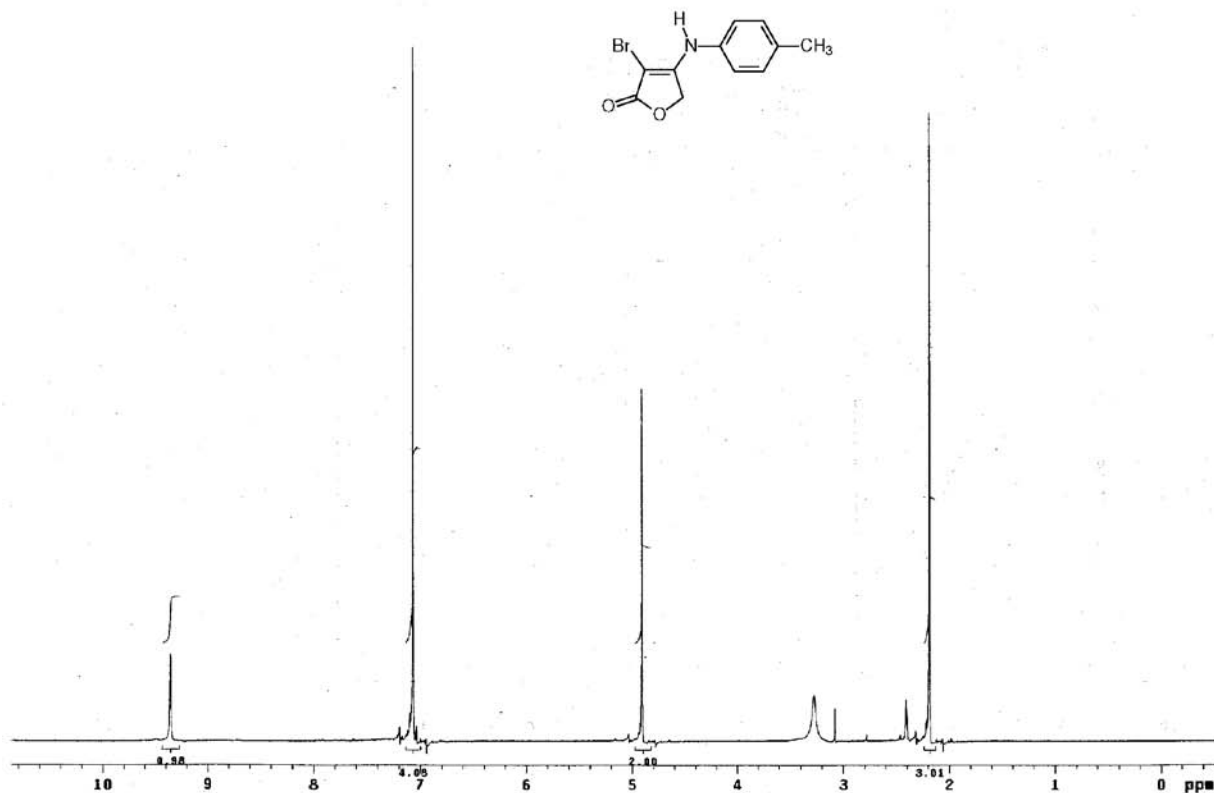
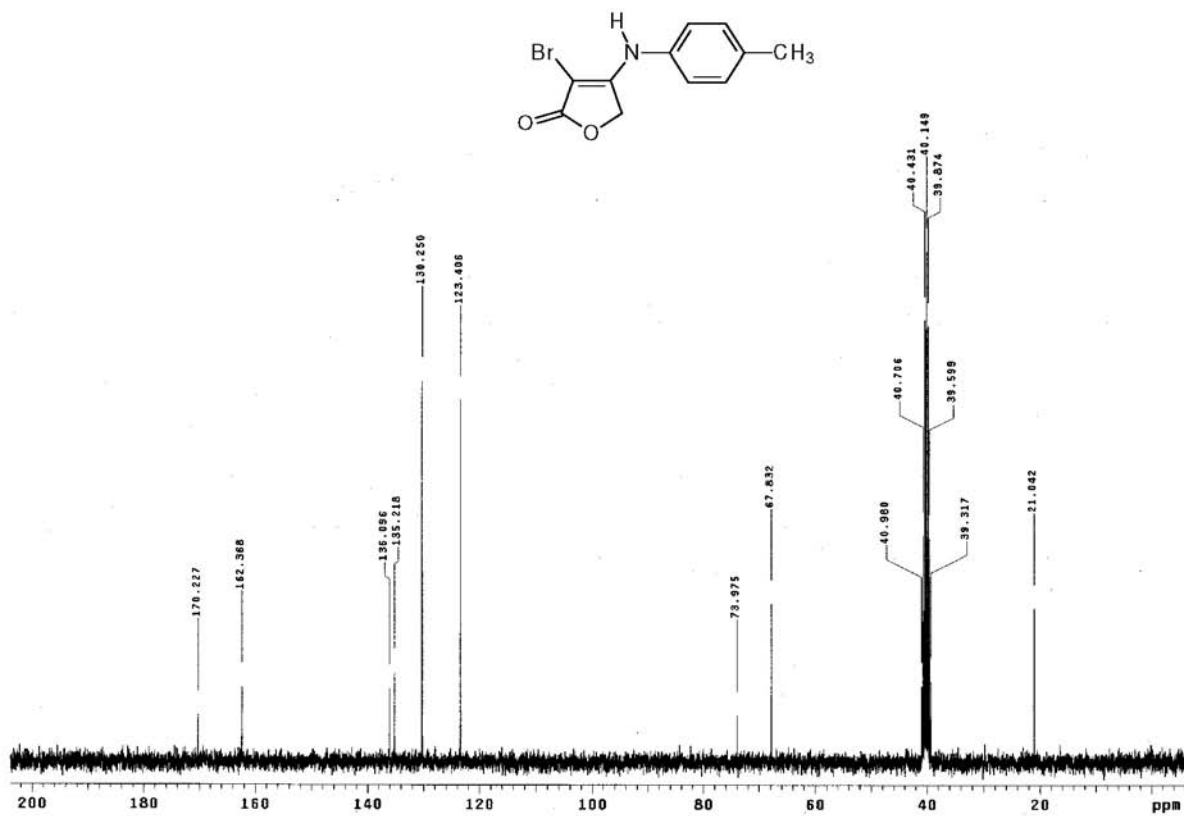


Figure S4. Full ^{13}C NMR spectrum of compound 3b (DMSO- d_6).

Figure S5. Full ¹H NMR spectrum of compound 3c (DMSO-*d*₆).Figure S6. Full ¹³C NMR spectrum of compound 3c (DMSO-*d*₆).

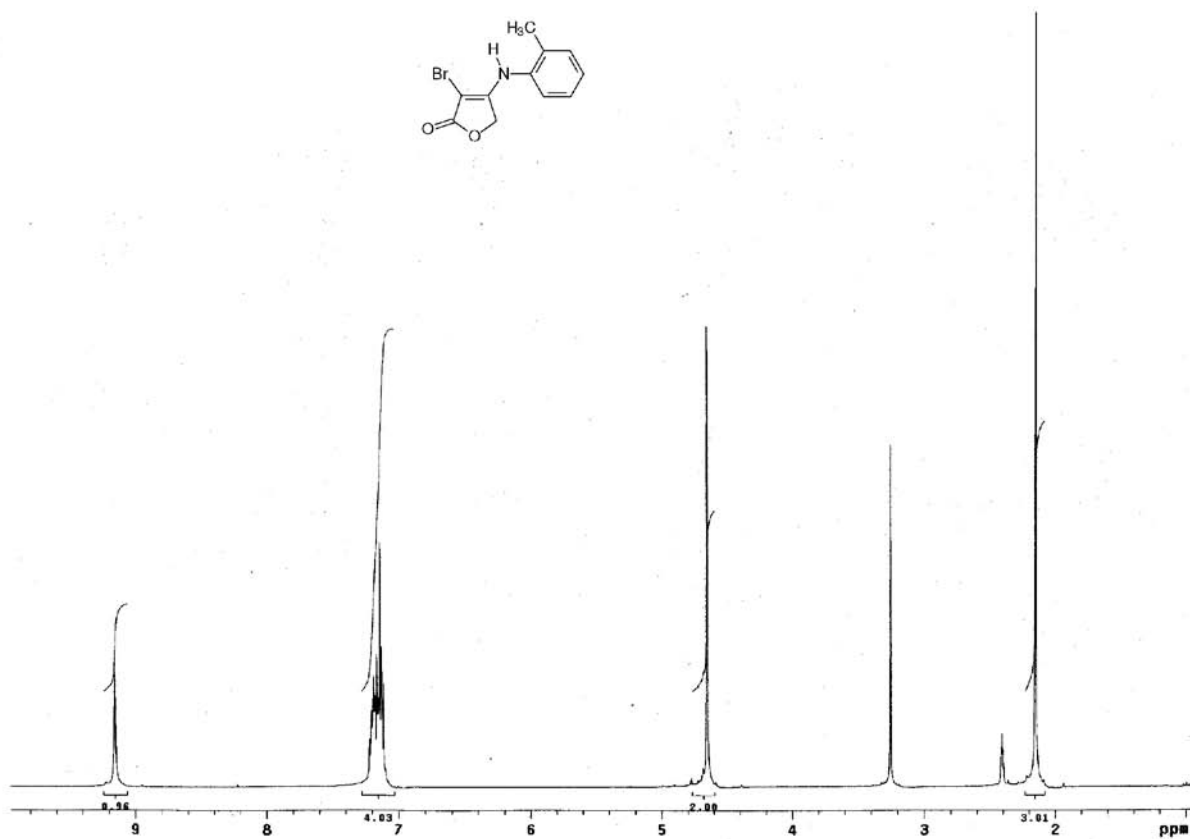


Figure S7. Full ^1H NMR spectrum of compound 3d ($\text{DMSO-}d_6$).

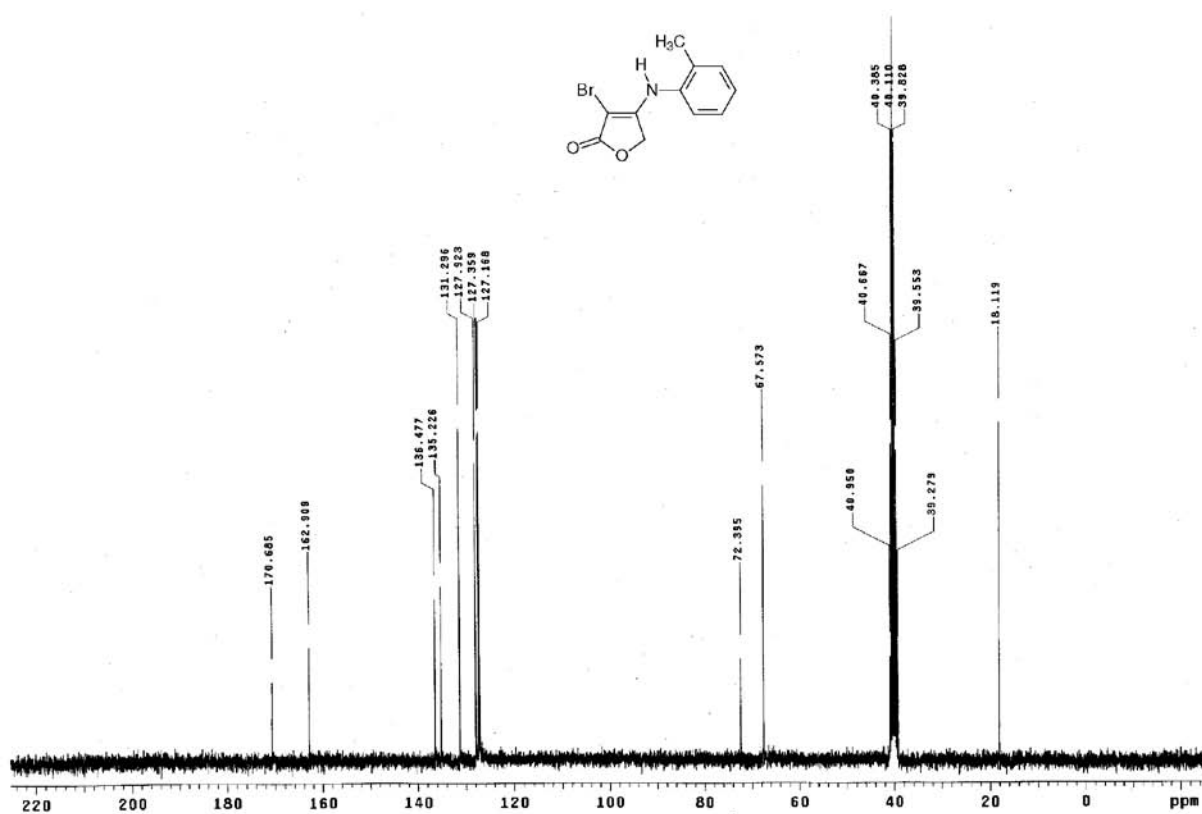
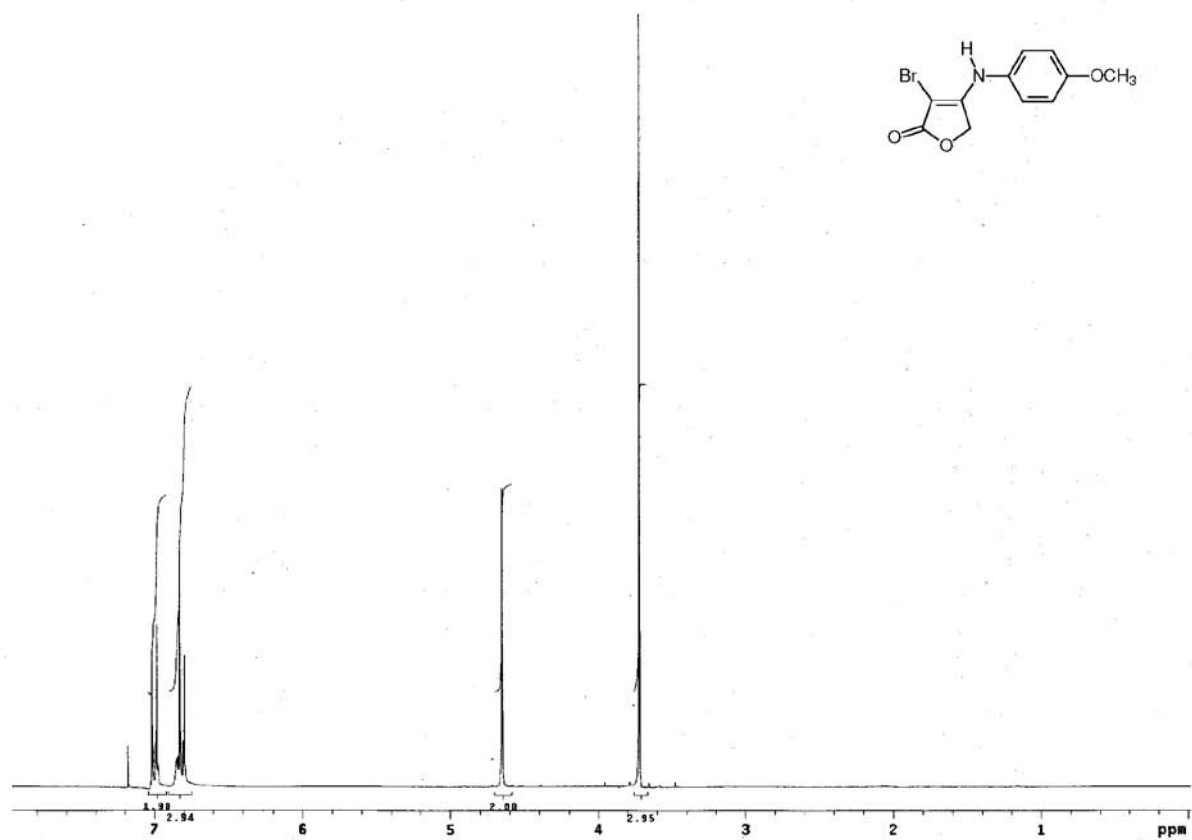
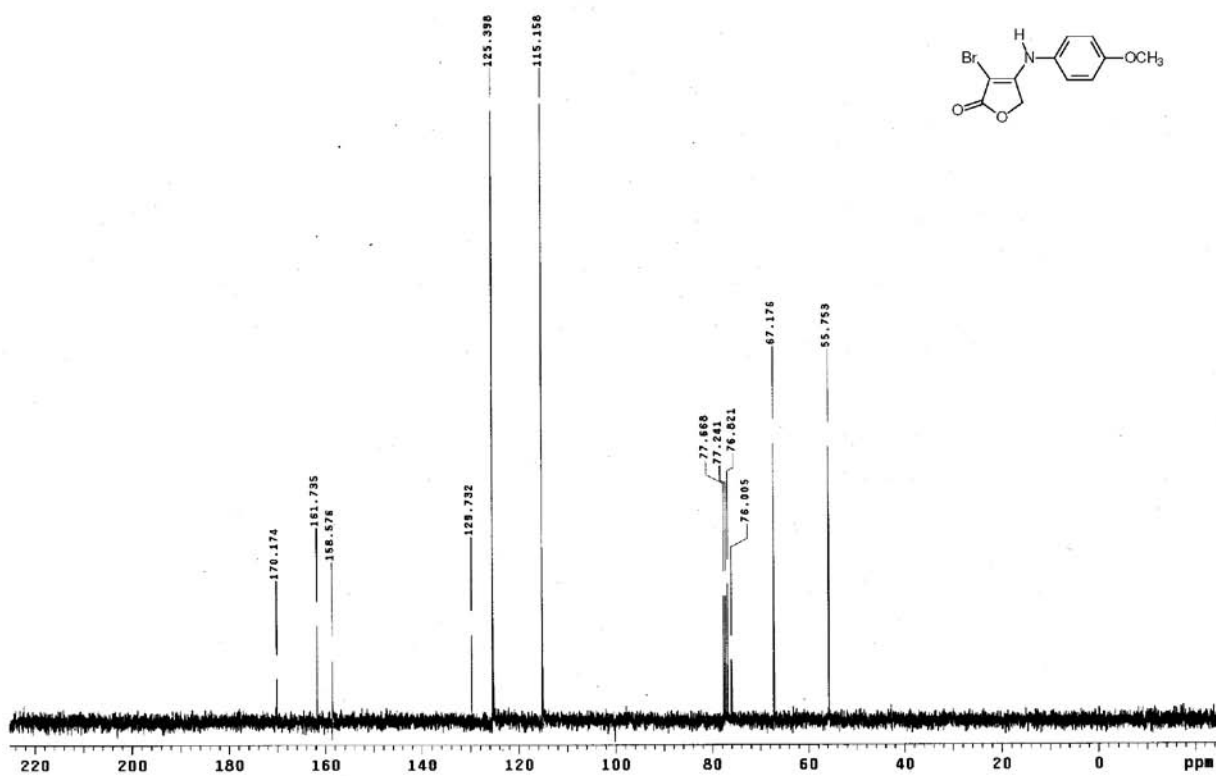


Figure S8. Full ^{13}C NMR spectrum of compound 3d ($\text{DMSO-}d_6$).

Figure S9. Full ¹H NMR spectrum of compound 3e (CDCl₃).Figure S10. Full ¹³C NMR spectrum of compound 3e (CDCl₃).

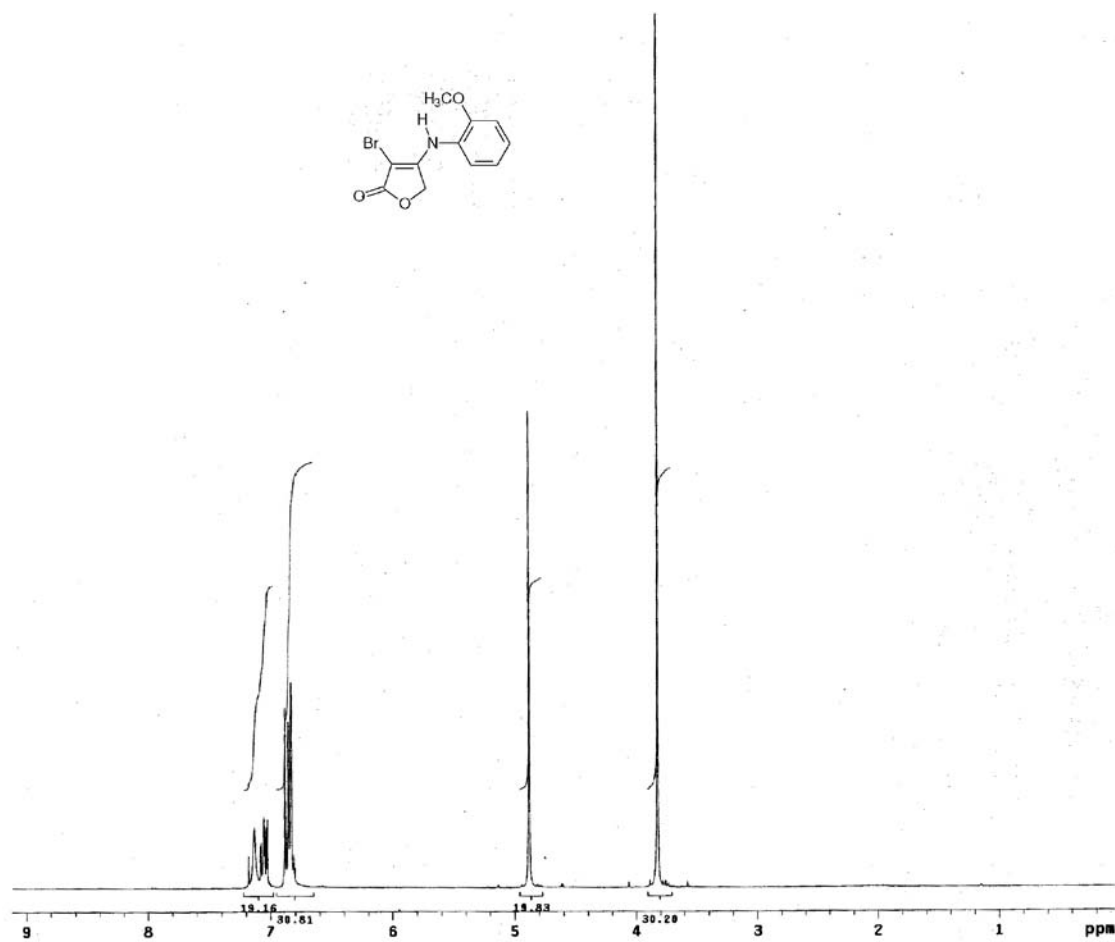


Figure S11. Full ¹H NMR spectrum of compound 3f (CDCl₃).

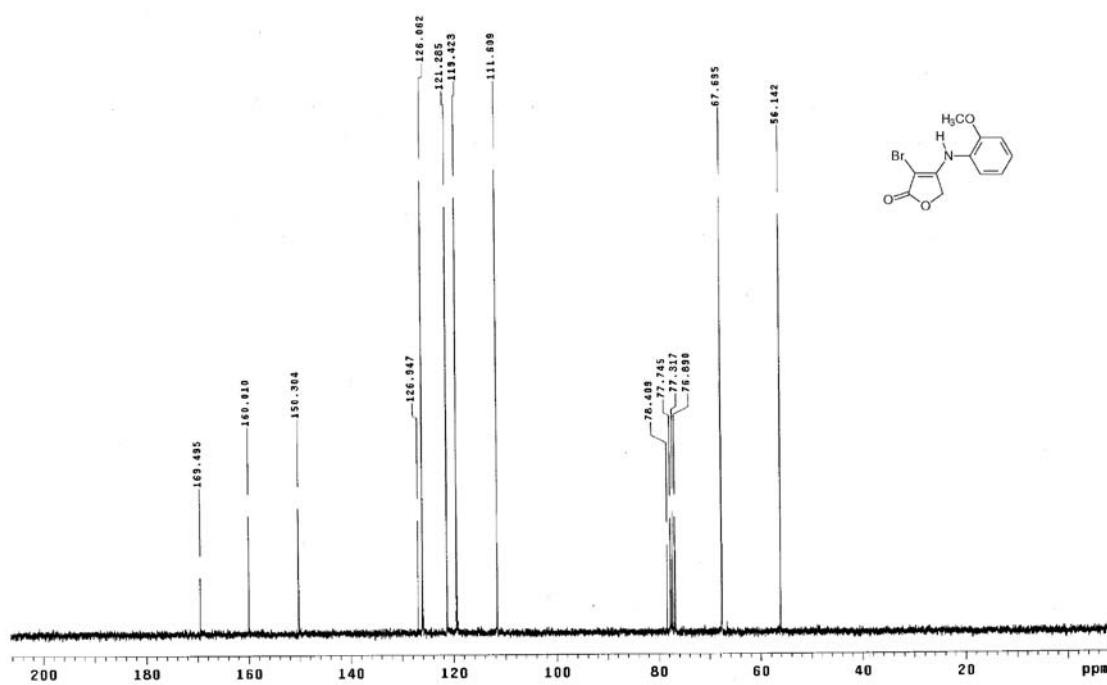


Figure S12. Full ¹³C NMR spectrum of compound 3f (CDCl₃).

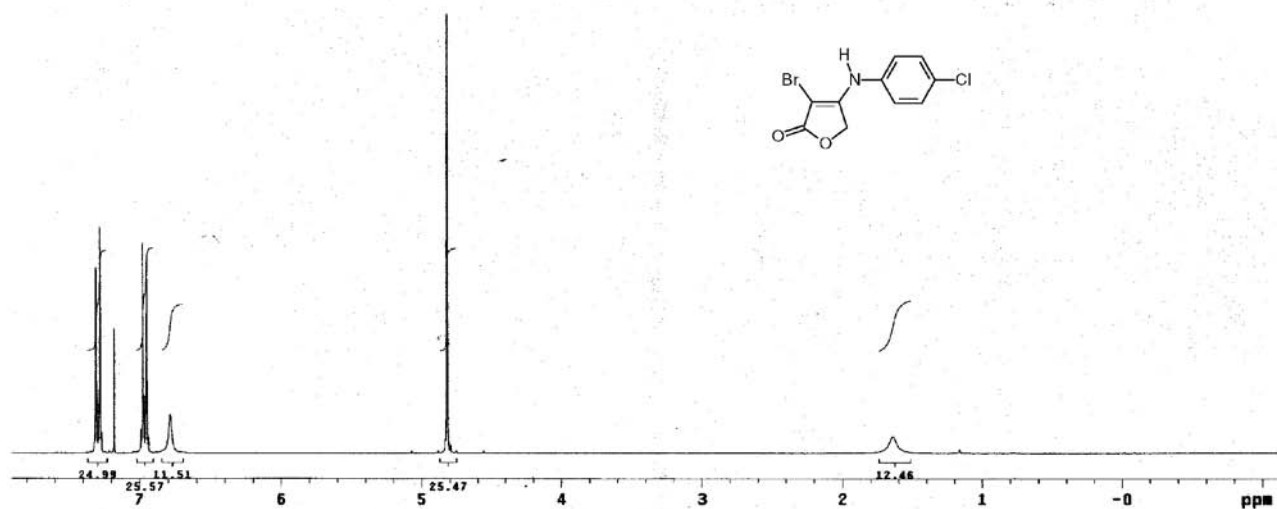


Figure S13. Full ¹H NMR spectrum of compound 3g (CDCl₃).

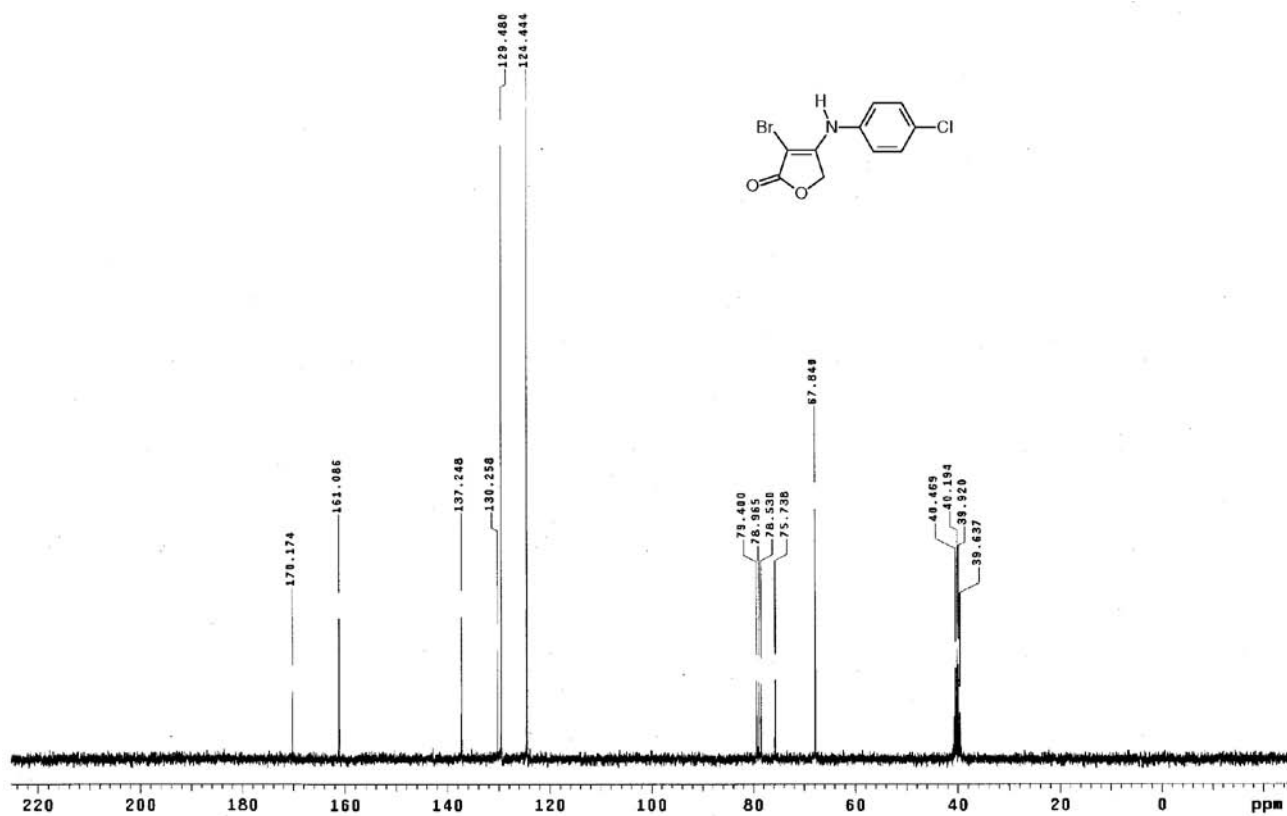


Figure S14. Full ¹³C NMR spectrum of compound 3g (CDCl₃/DMSO-*d*₆).

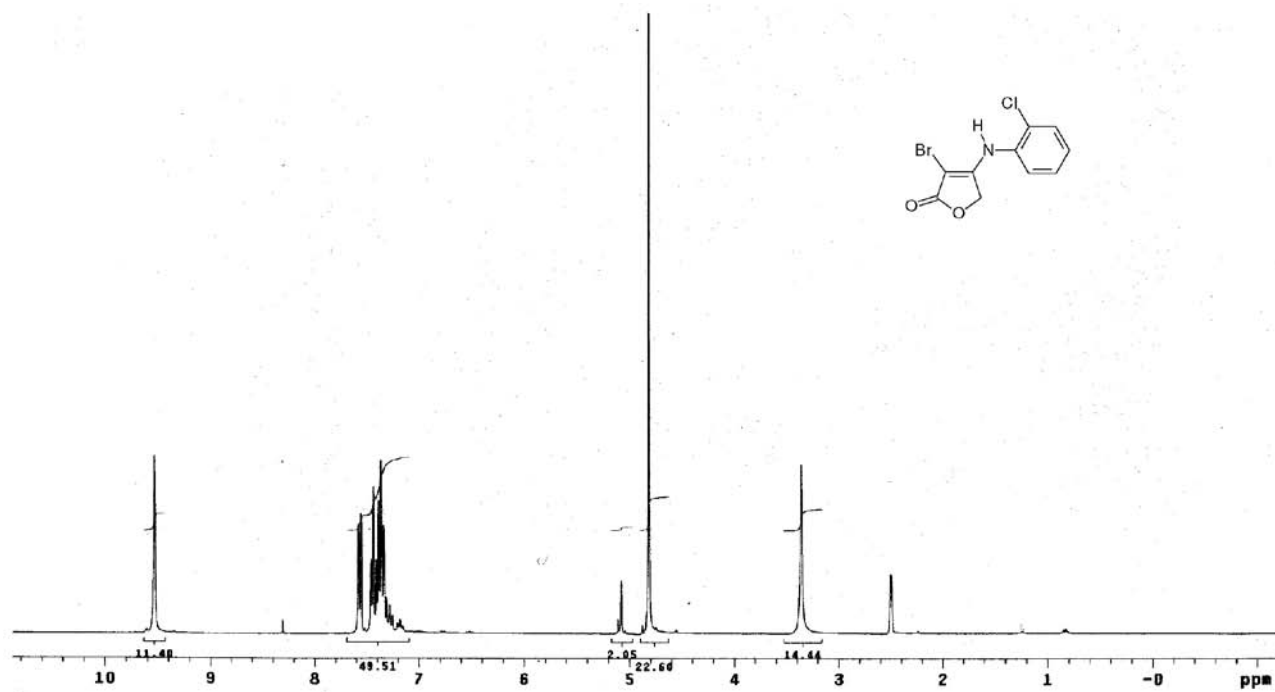


Figure S15. Full ¹H NMR spectrum of compound 3h (CDCl₃).

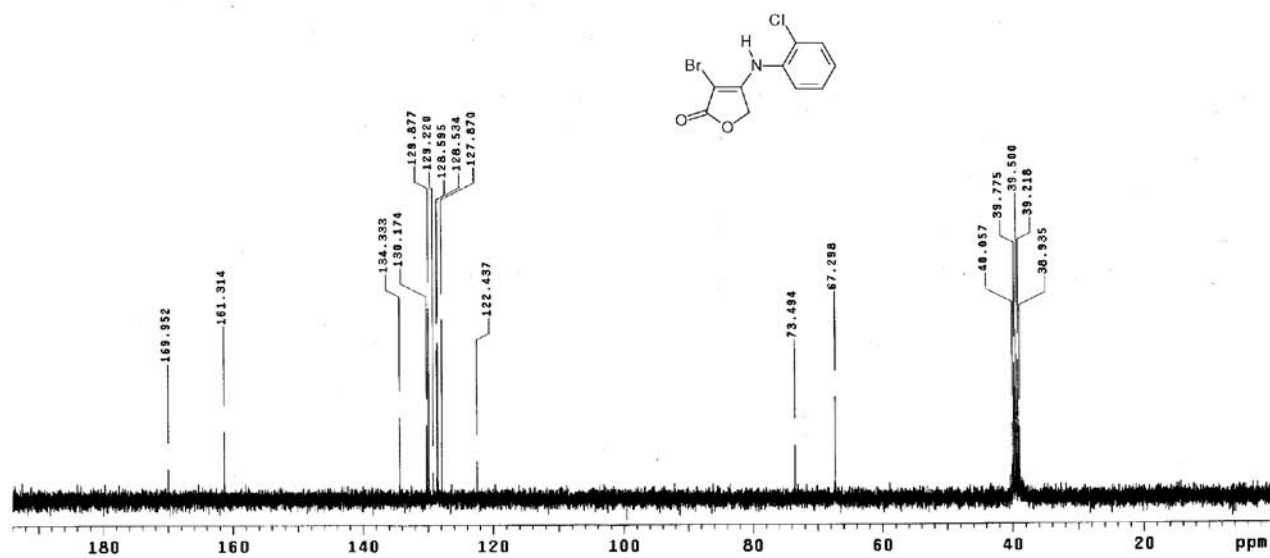


Figure S16. Full ¹³C NMR spectrum of compound 3h (CDCl₃).

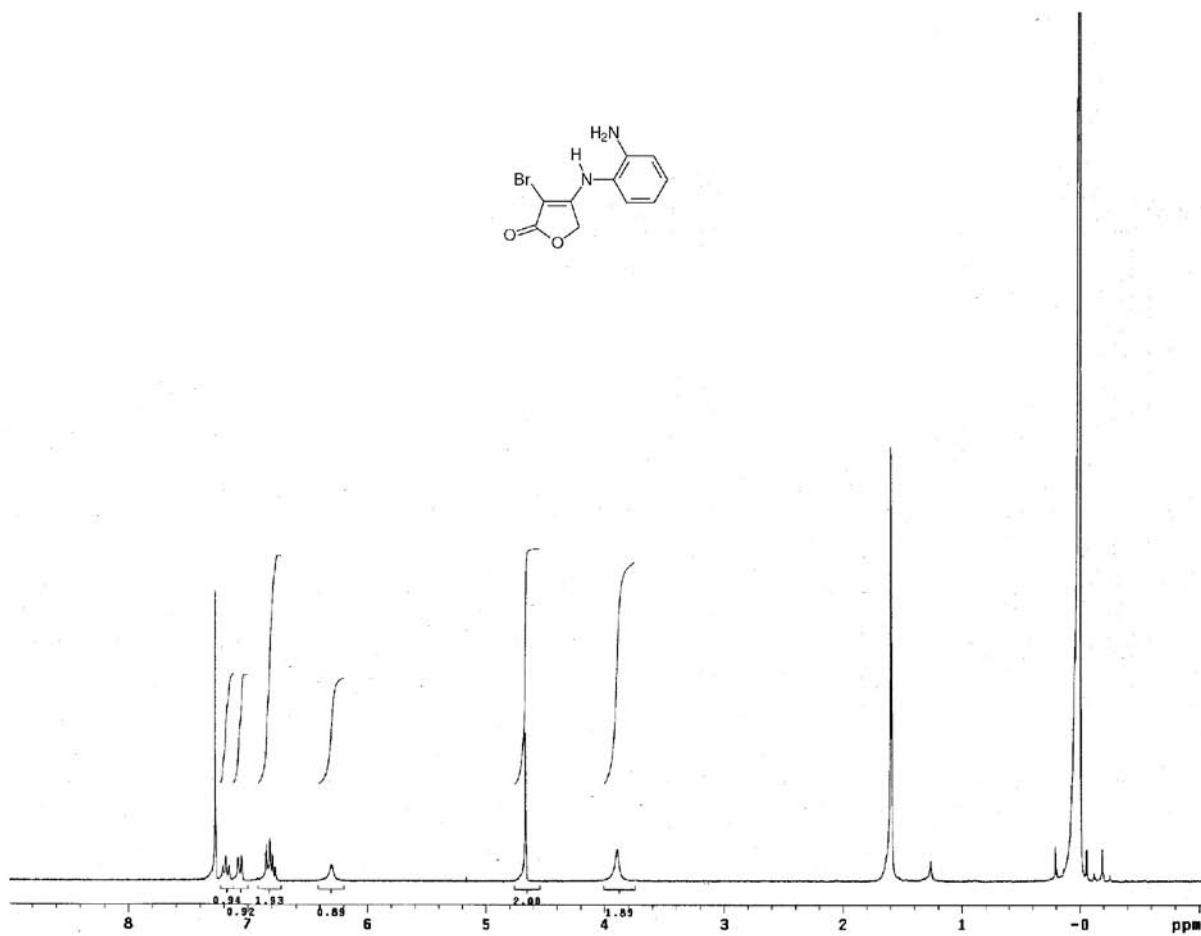


Figure S17. Full ¹H NMR spectrum of compound **3i** (CDCl₃).

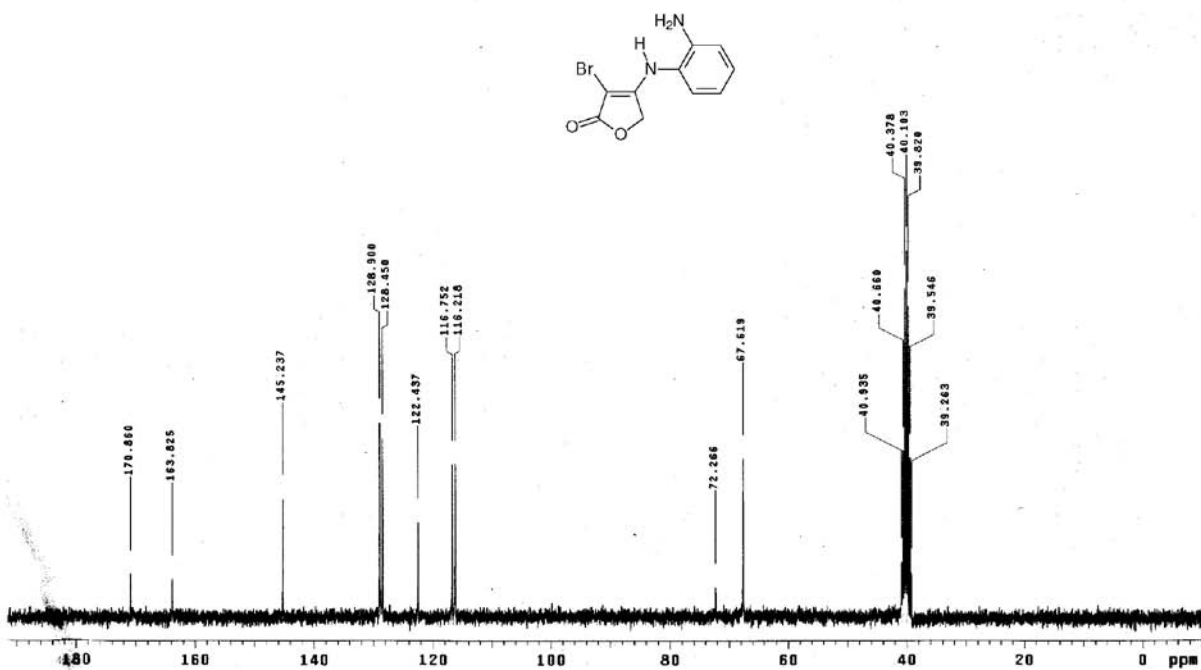
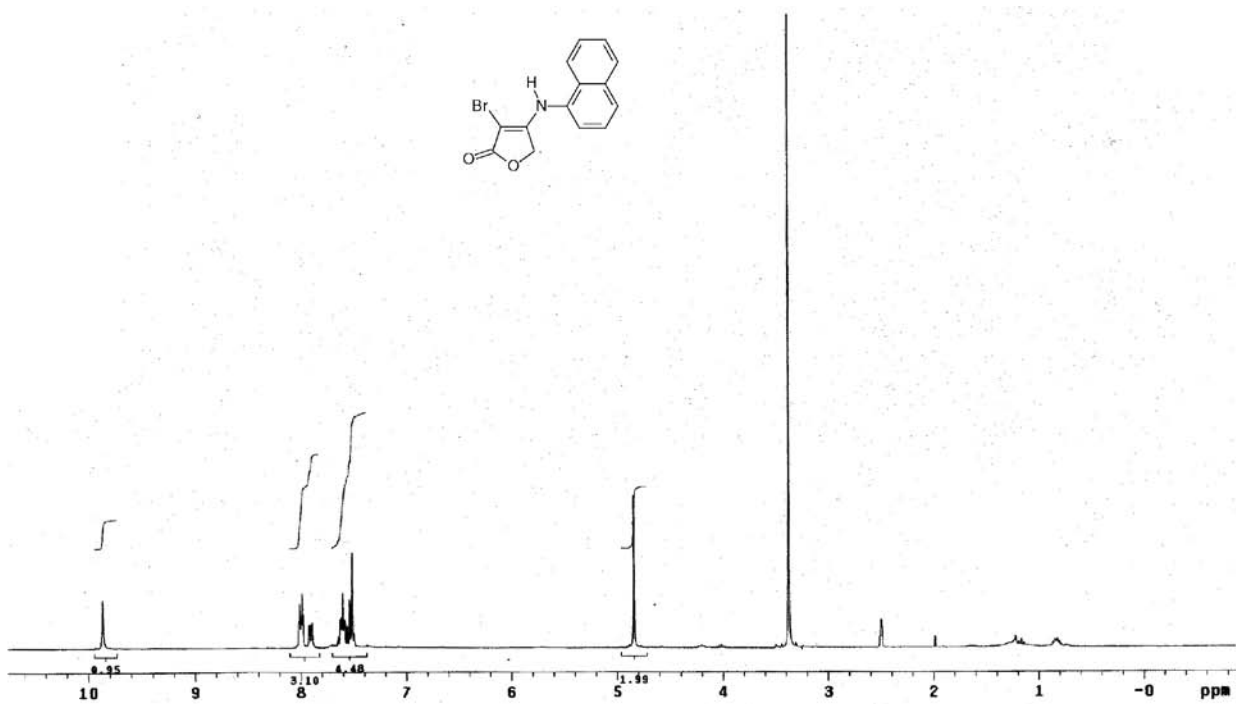
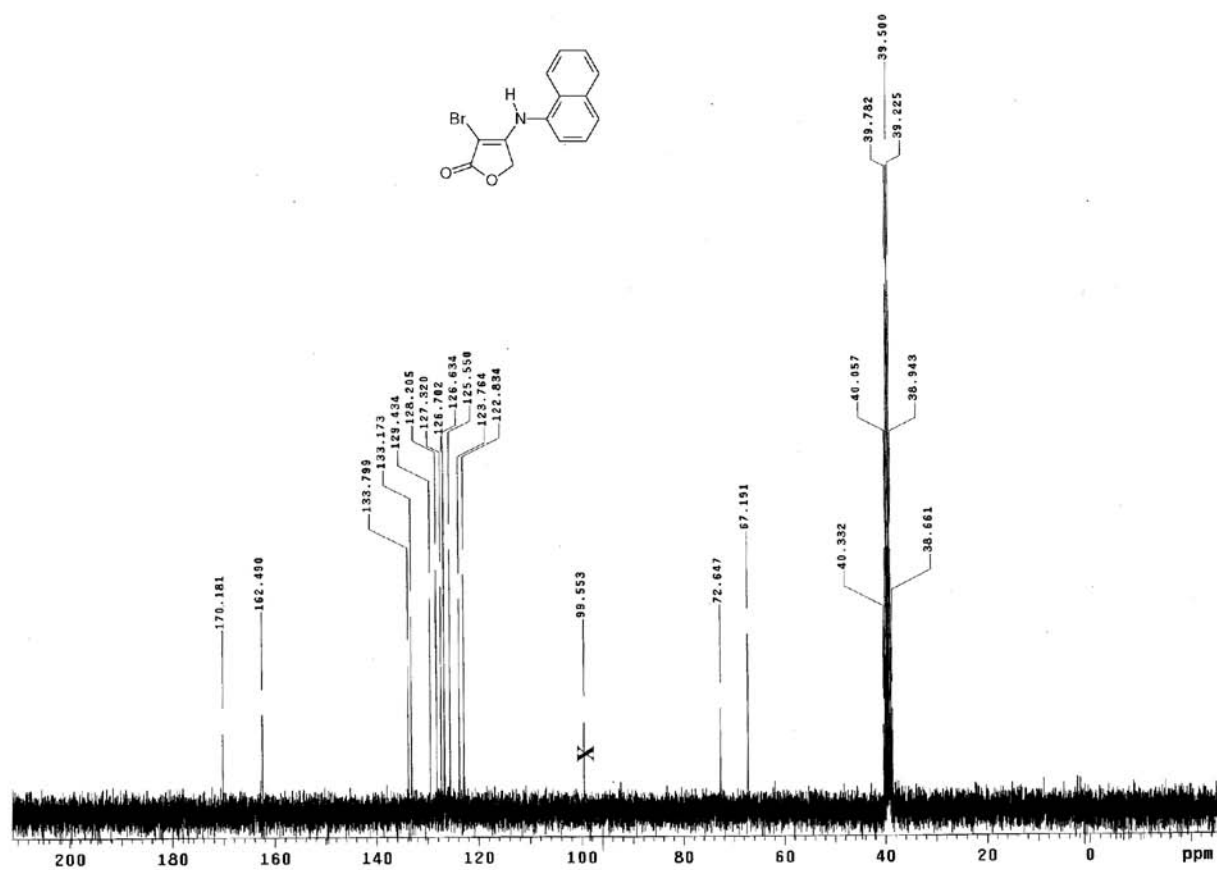


Figure S18. Full ¹³C NMR spectrum of compound **3i** (DMSO-*d*₆).

Figure S19. Full ^1H NMR spectrum of compound 3j (CDCl_3).Figure S20. Full ^{13}C NMR spectrum of compound 3j (CDCl_3).