

SÍNTESE DE 5-NITRO-ISATINA E 5-CLORO-ISATINA A PARTIR DA ISONITROSOACETANILIDA#

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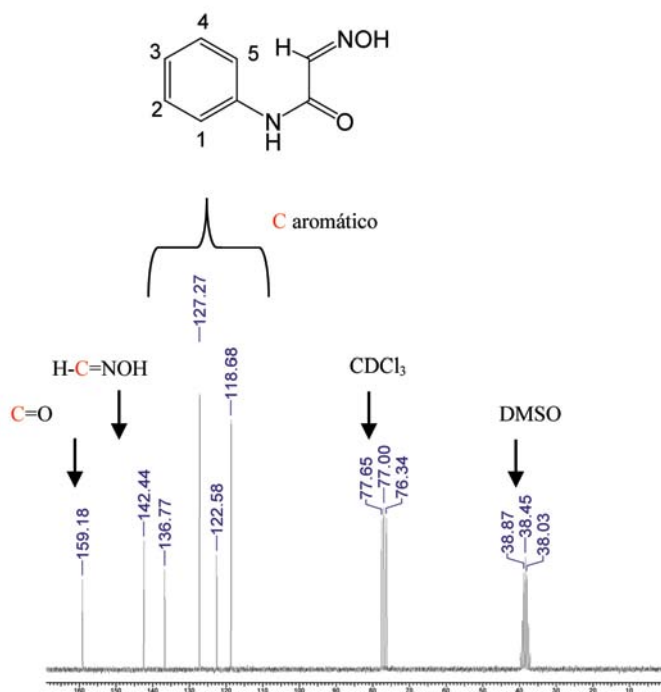


Figura 1S. Espectro de RMN ¹³C da isatina

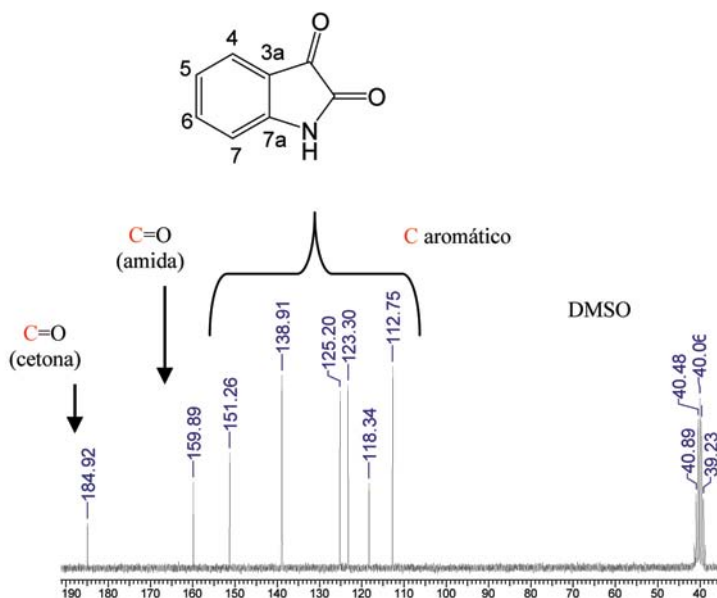


Figura 2S. Espectro de RMN ¹³C da isonitrosoacetanilida

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Artigo em homenagem ao Prof. Hans Viertler

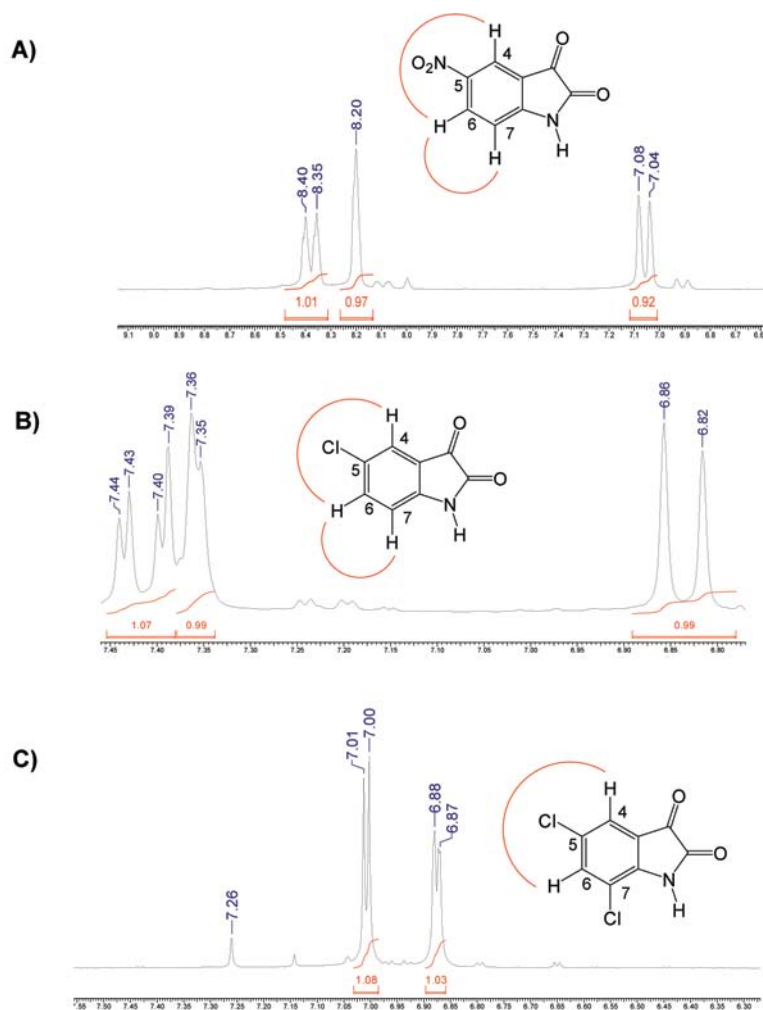


Figura 3S. Espectros de RMN ^1H com expansão da região que contém os sinais dos hidrogênios ligados ao anel aromático da 5-nitro (A), 5-cloro (B) e 5,7-dicloro (C)-isatina. As linhas em vermelho mostram o acoplamento entre os hidrogênios

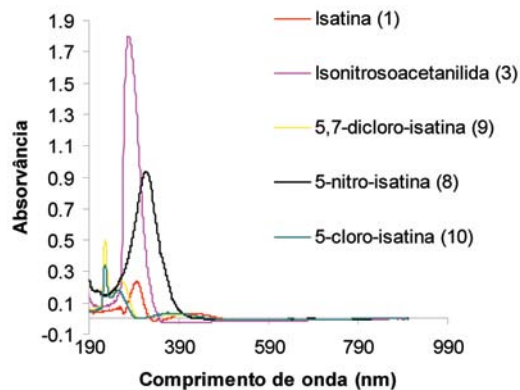


Figura 4S. Espectro de UV da isatina (1), isonitrosoacetanilida (3), 5-nitro-isatina (8), 5,7-dicloro-isatina (6) e 5-cloro-isatina (10)

Dados espectroscópicos

Isatina (1) - pf. 190-192 °C, *lit.*⁵ 189 – 192°C. IV (cm⁻¹): 3446, 3193, 3110, 1728, 1616, 1461, 1332, 771, 661. RMN ¹H (CDCl₃-DMSO-d₆, 200MHz) δ 7,48 (d, *J* = 8 Hz, 1H, H7), 6,89 (d, *J* = 8 Hz, 1H, H4), 7,57 (td, *J* = 2 e 8 Hz, 1H, H6), 7,04 (td, *J* = 2 e 8 Hz, 1H, H5), 11,0 (sl, 1H, NH). RMN ¹³C (CDCl₃-DMSO-d₆, 50 MHz) δ 112,7, 118,3, 123,3, 125,2, 138,9, 151,2, 159,8, 184,9.

Isonitrosoacetanilida (3) – pf. 173-175 °C, *lit.*⁵ 175 °C. IV (cm⁻¹): 3303, 3272, 3149, 1664, 1610, 1560, 1251, 1004, 904, 794, 694. RMN ¹H (CDCl₃-DMSO-d₆, 200 MHz) δ 7,05 (d, *J* = 8 Hz, 2H, H1 e H5), 6,67 (t, *J* = 8 Hz, 2H, H2 e H4), 6,45 (t, *J* = 8 Hz, 1H, H3), 9,08 (s, 1H, NH), 11,39 (sl, 1H, NOH). RMN ¹³C (CDCl₃-DMSO-d₆, 50 MHz) δ 159,1, 142,4, 136,7, 127,2, 122,5, 118,6.

5-nitro-isatina (8) - pf. 249-251 °C, *lit.*¹² 254-255 °C. IV (cm⁻¹): 3336, 1774, 1758, 1621, 1565, 1336, 748, 688. RMN ¹H (CDCl₃-

DMSO-d₆, 200 MHz) δ 7,08 (d, *J* = 8 Hz, 1H, H7), 8,20 (d, *J* = 2 Hz, 1H, H4), 8,37 (dd, *J* = 2 e 8 Hz, 1H, H6), 11,66 (sl, 1H, NH). RMN ¹³C (CDCl₃-DMSO-d₆, 50 MHz) δ 111,1, 115,9, 118,3, 131,5, 141,3, 153,7, 157,9, 180,9.

5,7-dicloro-isatina (9) - pf. 212-214 °C, *lit.*¹⁵ 213-217 °C. IV (cm⁻¹): 3473, 3174, 3110, 2925, 2854, 1758, 1726, 1612, 1457, 1292, 1172, 707, 688. RMN ¹H (CDCl₃-DMSO-d₆, 200 MHz) δ 7,13 (d, *J* = 1,7, 1H, H4), 7,01 (d, *J* = 1,7 Hz, 1H, H6), 10,7 (sl, 1H, NH) RMN ¹³C (CDCl₃-DMSO-d₆, 50 MHz) δ 116,9, 118,5, 121,8, 127,0, 135,5, 145,6, 157,8; 181,5.

5-cloro-isatina (10) - pf. 236-240 °C, *lit.*¹³ 244-245 °C. IV (cm⁻¹): 3477, 3417, 3095, 3070, 2987, 1747, 1702, 1618, 1452, 1276, 1168, 846, 705, 460. RMN ¹H (CDCl₃-DMSO-d₆, 200 MHz) δ 6,86 (d, *J* = 8,2 Hz, 1H, H7), 7,39 (s, 1H, H4), 7,46 (d, *J* = 8,2 Hz, 1H, H6), 11,1 (sl, 1H, NH). RMN ¹³C (CDCl₃-DMSO-d₆, 50 MHz) δ 112,7, 117,2, 123,1, 126,6, 136,3, 148,1, 157,7, 182,3.

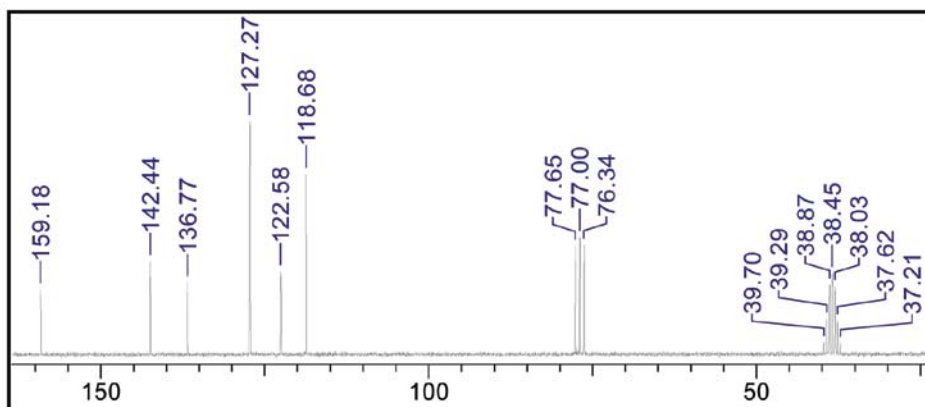


Figura 5S. Espectro de ¹³C RMN da isonitrosoacetanilida (3)

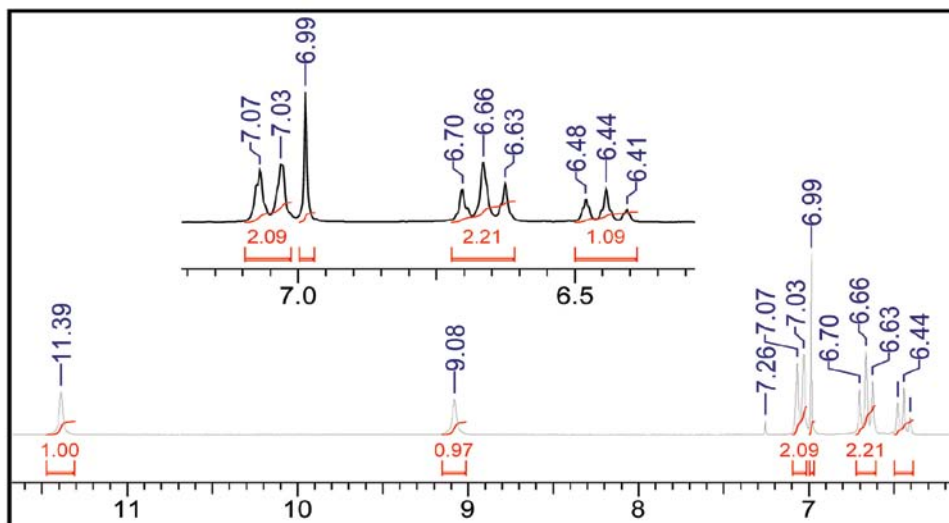


Figura 5S. Espectro de ¹H RMN da isonitrosoacetanilida (3)

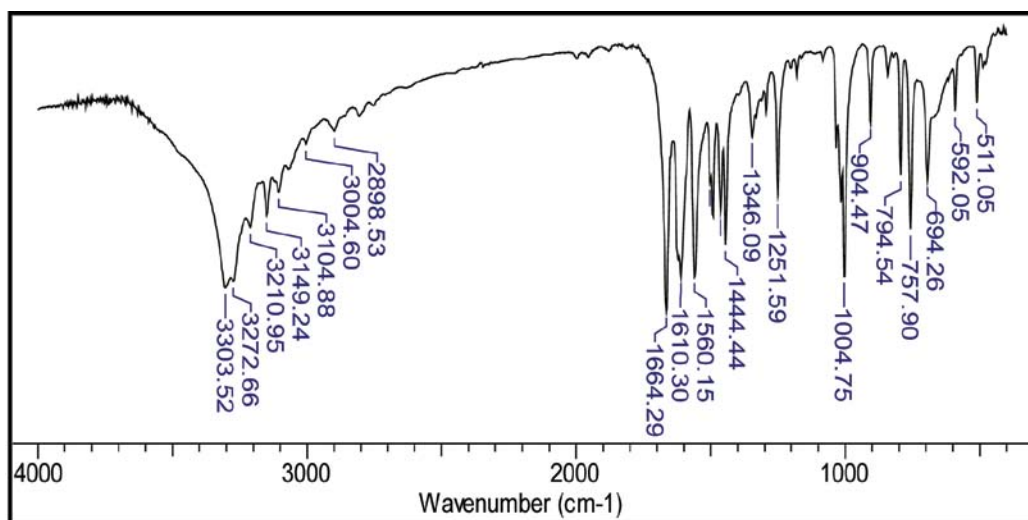


Figura 6S. Espectro de Infravermelho da isonitrosoacetanilida (3)

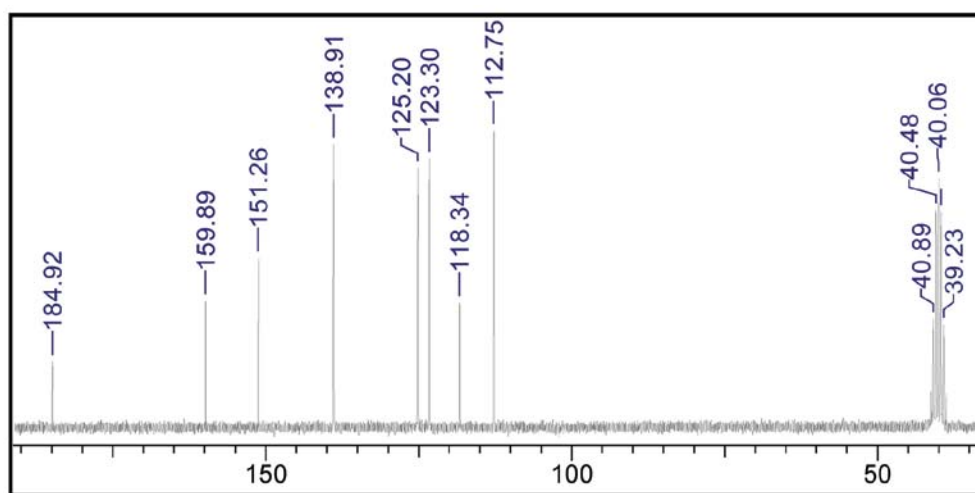


Figura 7S. Espectro de RMN ¹³C da isatina (1)

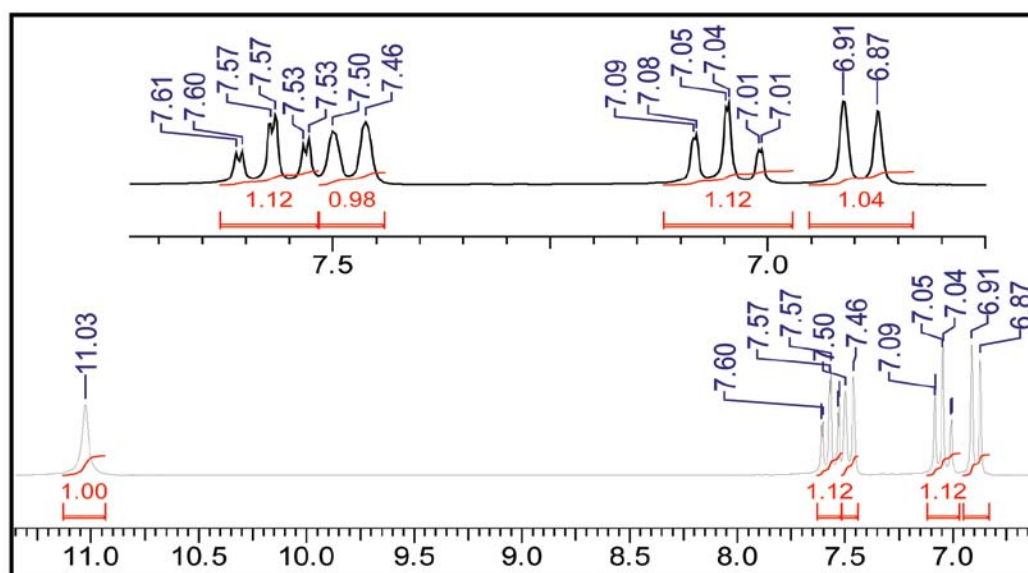


Figura 8S. Espectro de RMN ¹H da isatina (1)

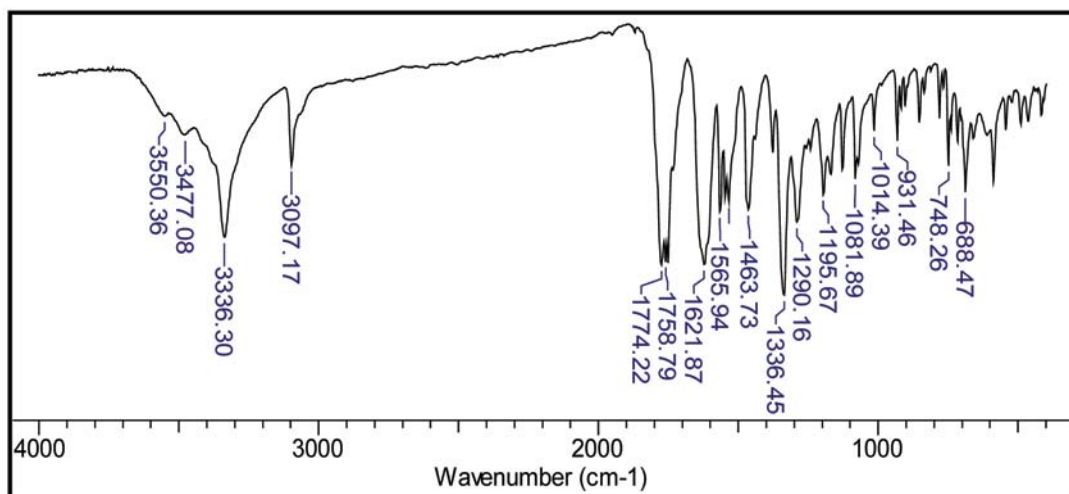


Figura 12S. Espectro de infravermelho da 5-nitro isatina (8)

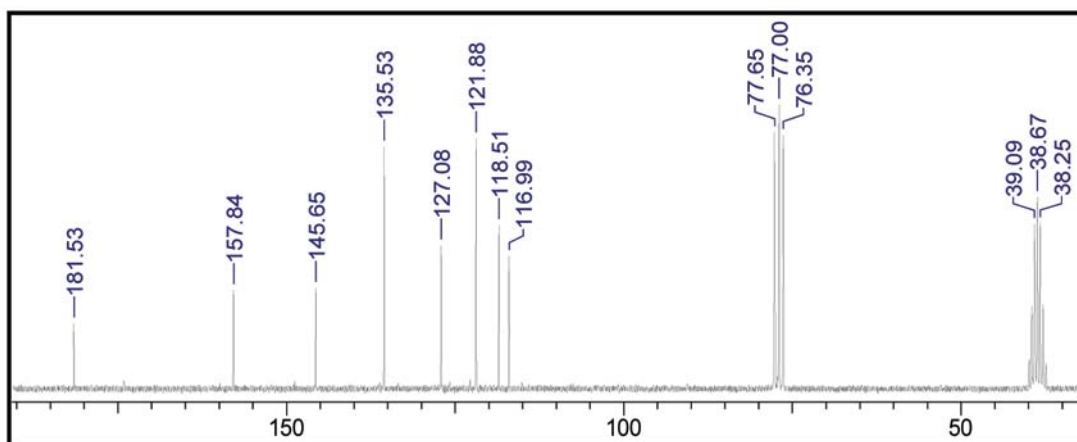


Figura 13S. Espectro de RMN ¹³C da 5,7-dicloro-isatina (9)

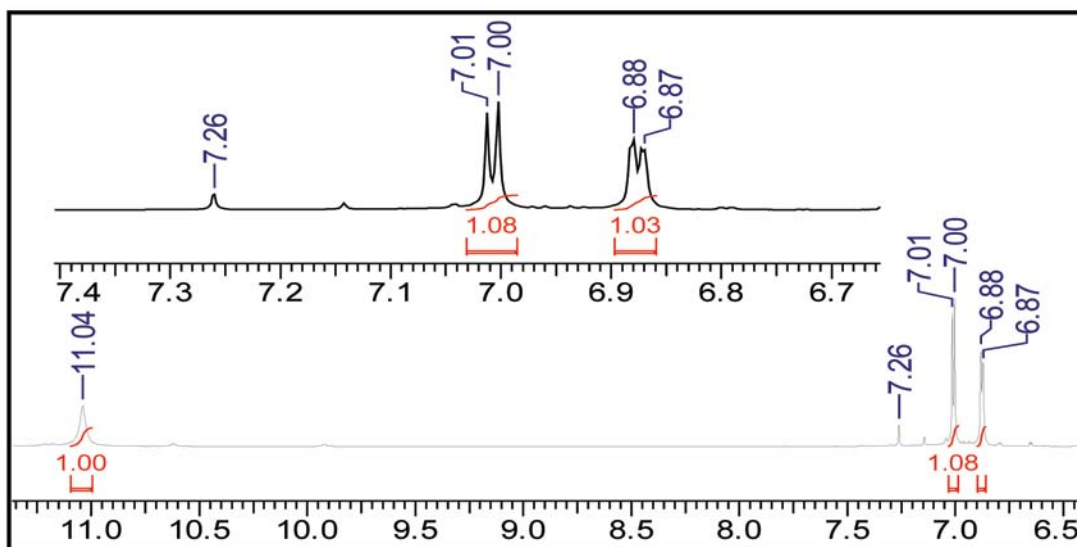


Figura 14S. Espectro de RMN ¹H da 5,7-dicloro-isatina (9)

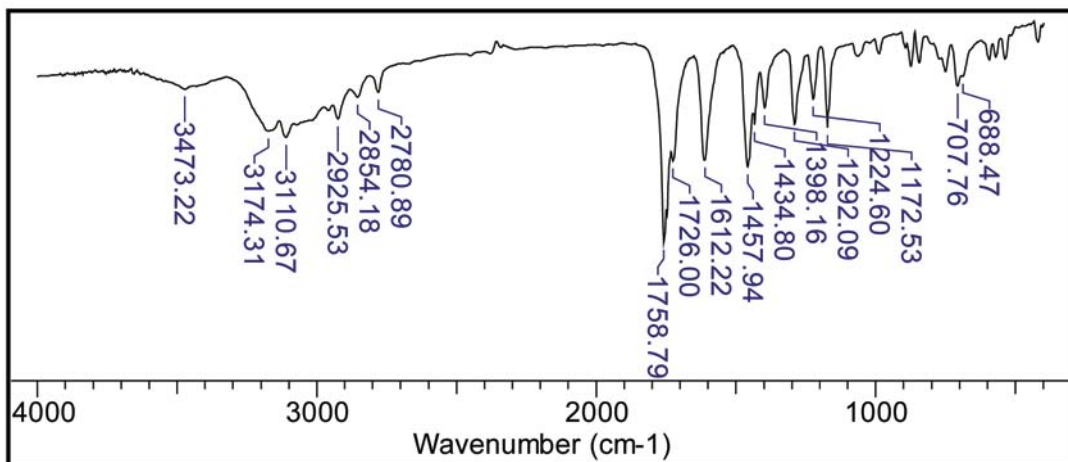


Figura 15S. Espectro de infravermelho da 5,7-dicloro-isatina (9)

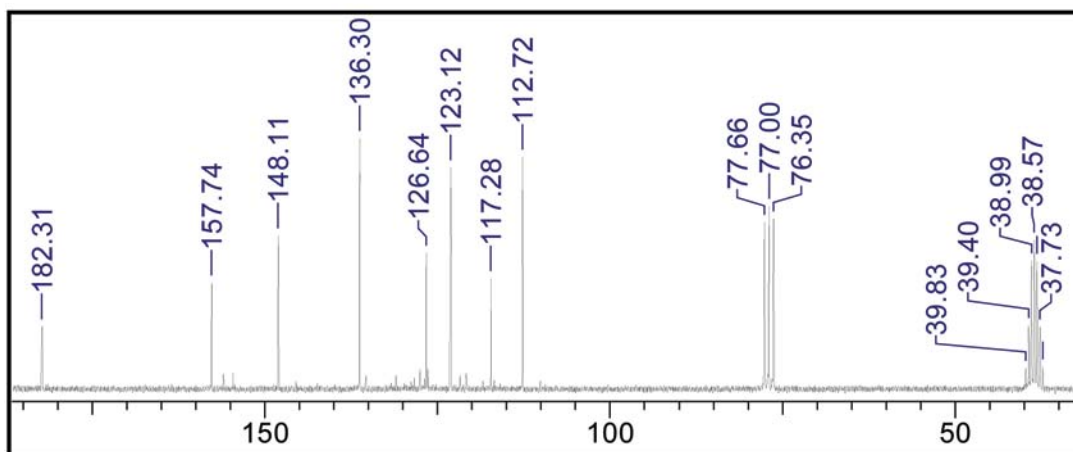


Figura 16S. Espectro de RMN ¹³C da 5-cloro-isatina (10)

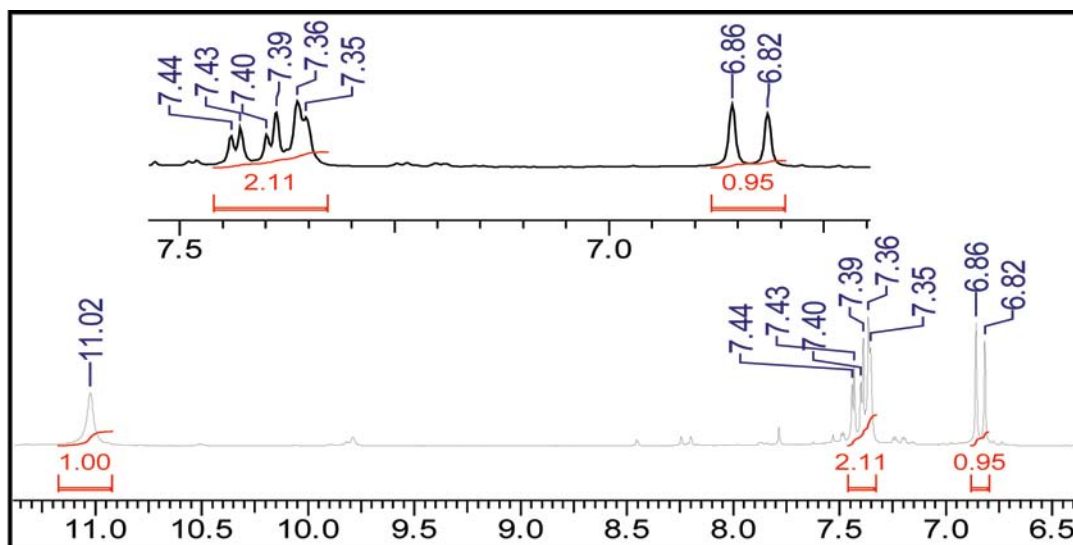


Figura 17S. Espectro de RMN ¹H da 5-cloro-isatina (10)

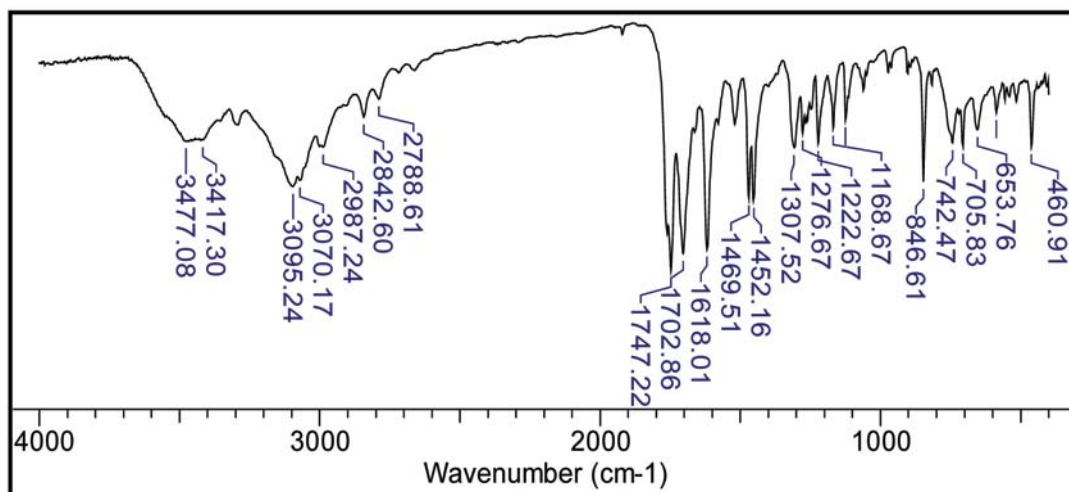


Figura 18S. Espectro de infravermelho da 5-cloro-isatina (10)