

## **8. ANEXOS**

### **8.1 Instrumentación:**

La purificación de los productos finales por cromatografía en columna se ha llevado a cabo empleando una bomba de media presión, modelo Buchi C-605 con controlador C-615 y columnas Buchi de diferentes diámetros, siempre con soporte de silicagel (tamaño de partícula 0,04-0,06 mm).

Los puntos de fusión se han medido en un Gallenkamp, en capilares abiertos.

Los espectros de IR se han llevado a cabo en un espectrofotómetro Perkin Elmer 1600FT en pastilla de KBr. Las vibraciones se expresan en número de ondas ( $\text{cm}^{-1}$ ).

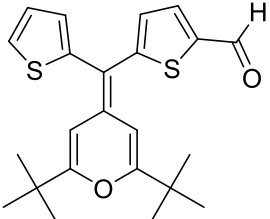
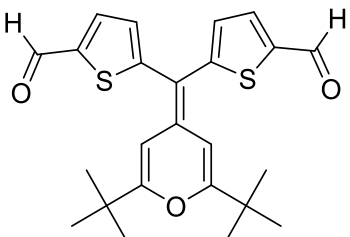
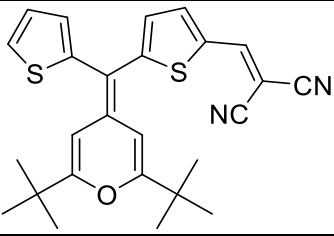
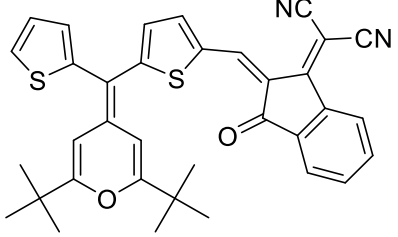
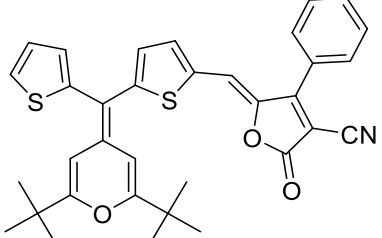
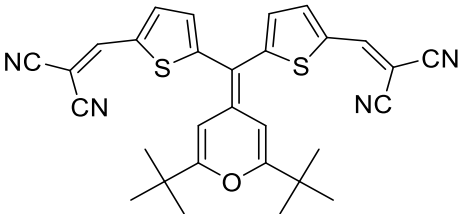
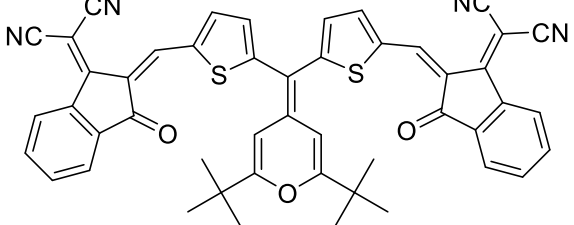
Los espectros de resonancia magnética nuclear (RMN) se han registrado en un Bruker ARX-300, a 75 MHz para  $^{13}\text{C}$ -RMN; o en un Bruker AV-400, a 400 MHz para  $^1\text{H}$ -RMN y a 100 MHz para  $^{13}\text{C}$ -RMN. Los desplazamientos químicos ( $\delta$ ) se expresan en ppm.

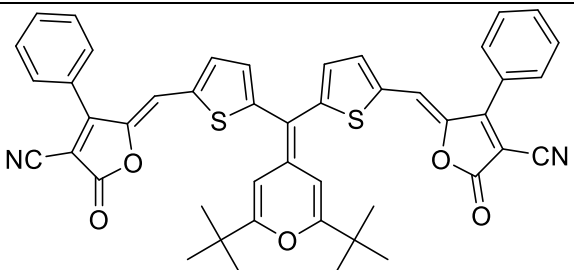
Los espectros de masas  $\text{ESI}^+$  se registraron en un Bruker MicroTOF-Q, usando formiato de sodio como referencia externa, para los espectros de masa exacta.

Los espectros de absorción UV-vis se han realizado en un espectrofotómetro UNICAM UV4. Los resultados se expresan en longitud de onda (nm).

Los experimentos de Voltamperometría Cíclica (VC) se han llevado a cabo en un potenciostato  $\mu$ -Autolab type III, utilizando como electrodo de trabajo uno de grafito, electrodo de referencia  $\text{Ag}/\text{AgCl}$ , un electrodo de platino como referencia,  $\text{Bu}_4\text{NPF}_6$  0,1 M en  $\text{CH}_2\text{Cl}_2$  como electrolito soporte y velocidad de barrido 100 mV/s.

## 8.2. Espectros y gráficas

Molécula	Referencia	Espectro	Figura	Pág.
	<b>1-CHO</b>	<sup>1</sup> H-RMN <sup>13</sup> C-RMN EM (HR-ESI <sup>+</sup> ) IR	8.1 8.2 8.3 8.4	29 29 30 30
	<b>2-CHO</b>	<sup>1</sup> H-RMN <sup>13</sup> C-RMN EM (HR-ESI <sup>+</sup> ) IR	8.5 8.6 8.7 8.8	31 31 32 32
	<b>1A</b>	<sup>1</sup> H-RMN <sup>13</sup> C-RMN EM (HR-ESI <sup>+</sup> ) IR UV-Vis VC	8.9 8.10 8.11 8.12 8.13 8.14	33 33 34 34 35 36
	<b>1B</b>	<sup>1</sup> H-RMN <sup>13</sup> C-RMN EM (HR-ESI <sup>+</sup> ) IR UV-Vis VC	8.15 8.16 8.17 8.18 8.19 8.20	36 37 37 38 39 39
	<b>1C</b>	<sup>1</sup> H-RMN <sup>13</sup> C-RMN EM (HR-ESI <sup>+</sup> ) IR UV-Vis VC	8.21 8.22 8.23 8.24 8.25 8.26	40 40 41 41 42 43
	<b>2A</b>	<sup>1</sup> H-RMN <sup>13</sup> C-RMN EM (HR-ESI <sup>+</sup> ) IR UV-Vis VC	8.27 8.28 8.29 8.30 8.31 8.32	43 44 44 45 46 46
	<b>2B</b>	<sup>1</sup> H-RMN <sup>13</sup> C-RMN EM (HR-ESI <sup>+</sup> ) IR UV-Vis	8.33 8.34 8.35 8.36 8.37 8.38 8.39	47 47 48 48 49 50 50

		VC	8.40	51
	<b>2C</b>	<sup>1</sup> H-RMN <sup>13</sup> C-RMN EM (HR-ESI <sup>+</sup> ) IR UV-Vis VC	8.41 8.42 8.43 8.44 8.45 8.46	51 52 52 53 54 54

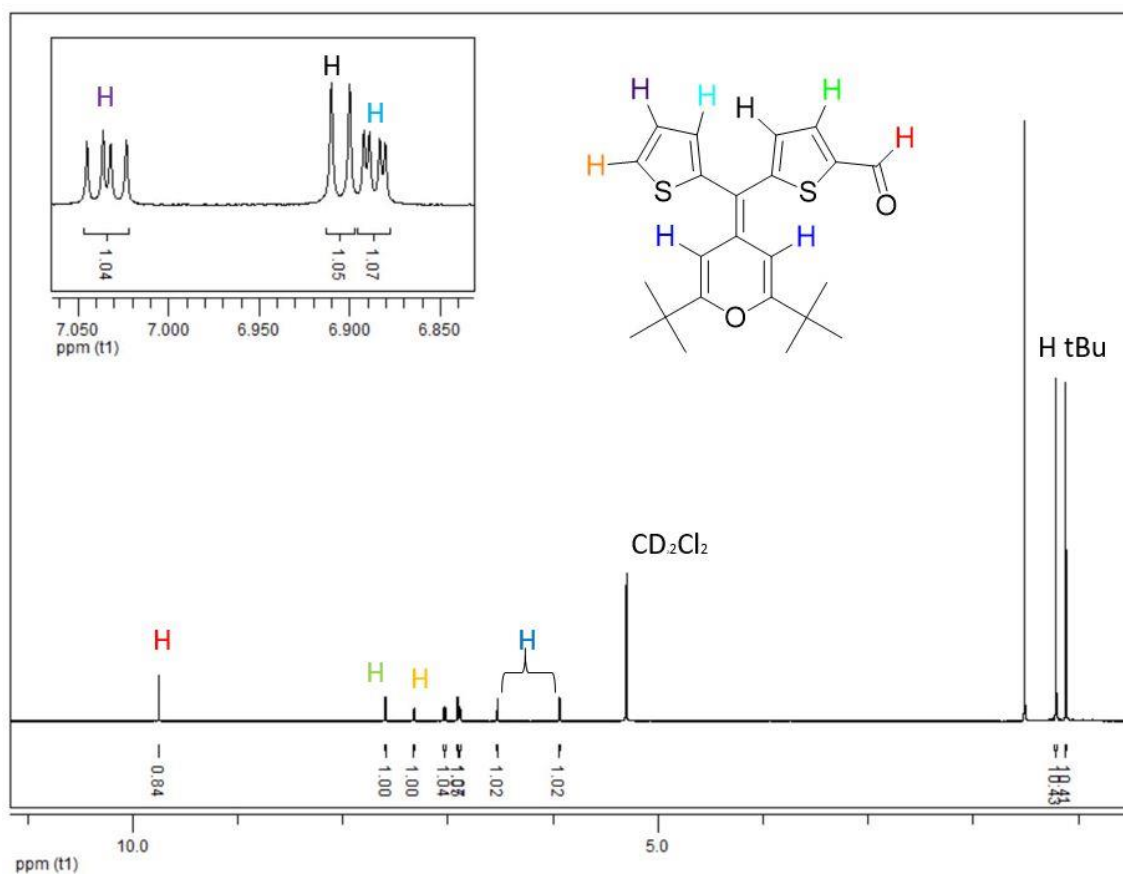


Figura 8.1. Espectro de  $^1\text{H-RMN}$  de **1-CHO**.

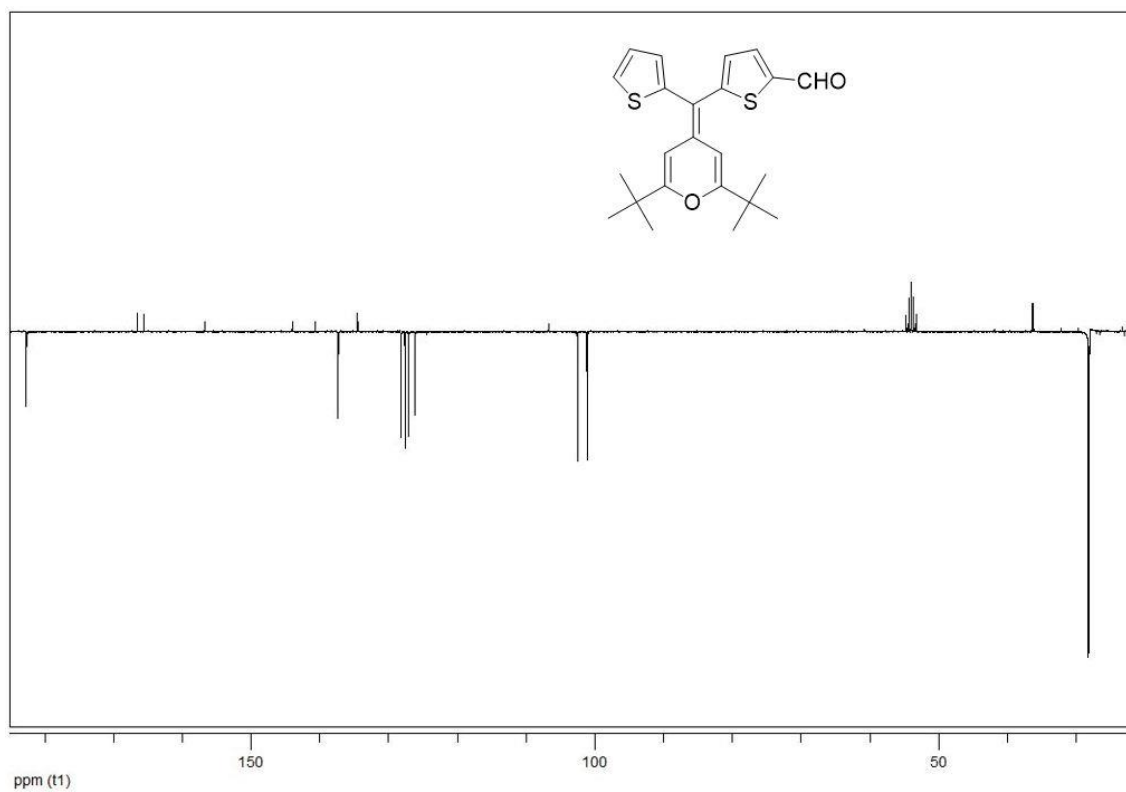


Figura 8.2. Espectro de  $^{13}\text{C-RMN}$  (APT) de **1-CHO**.

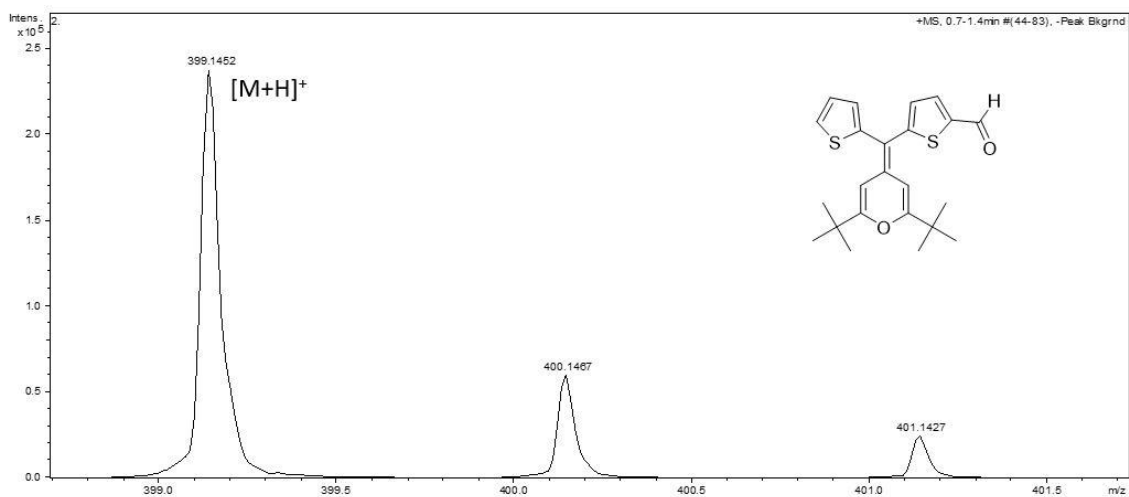


Figura 8.3. Espectro EM (HR-ESI<sup>+</sup>) de 1-CHO.

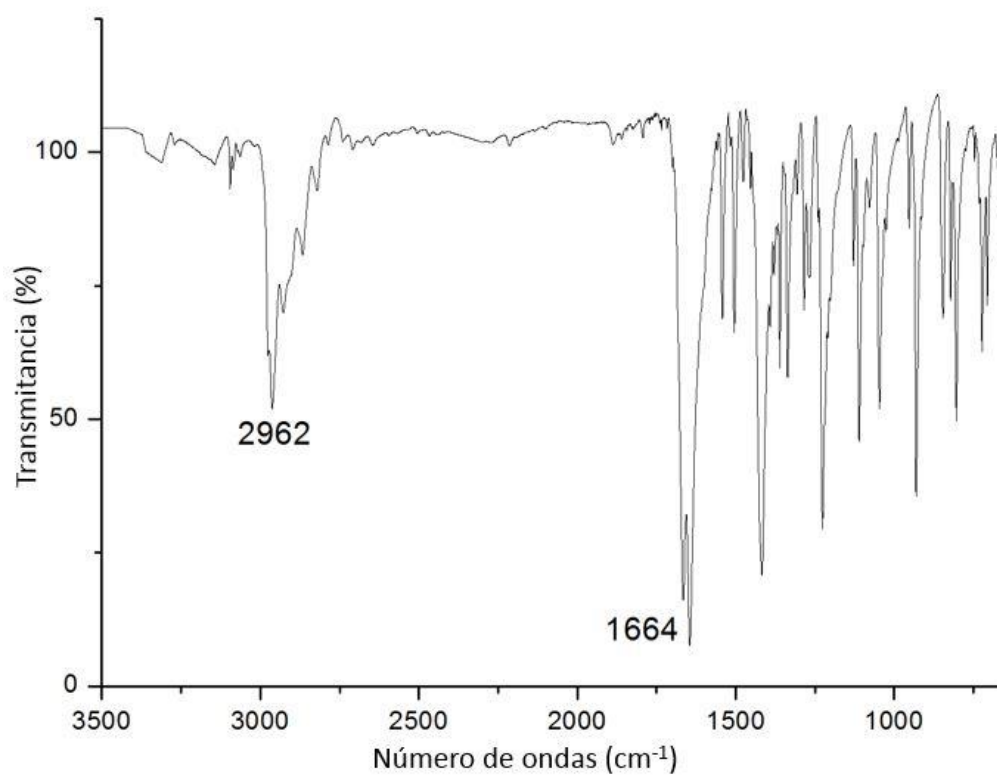


Figura 8.4. Espectro de IR de 1-CHO.

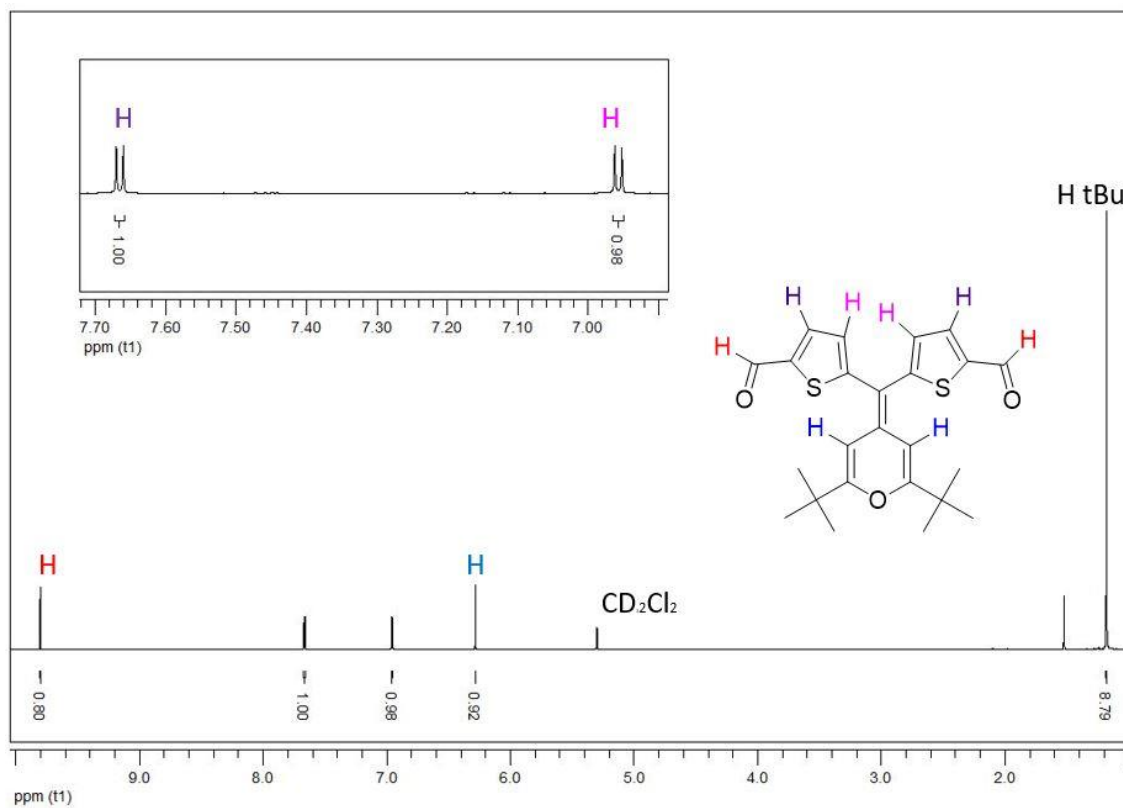


Figura 8.5. Espectro de  $^1\text{H-RMN}$  de 2-CHO.

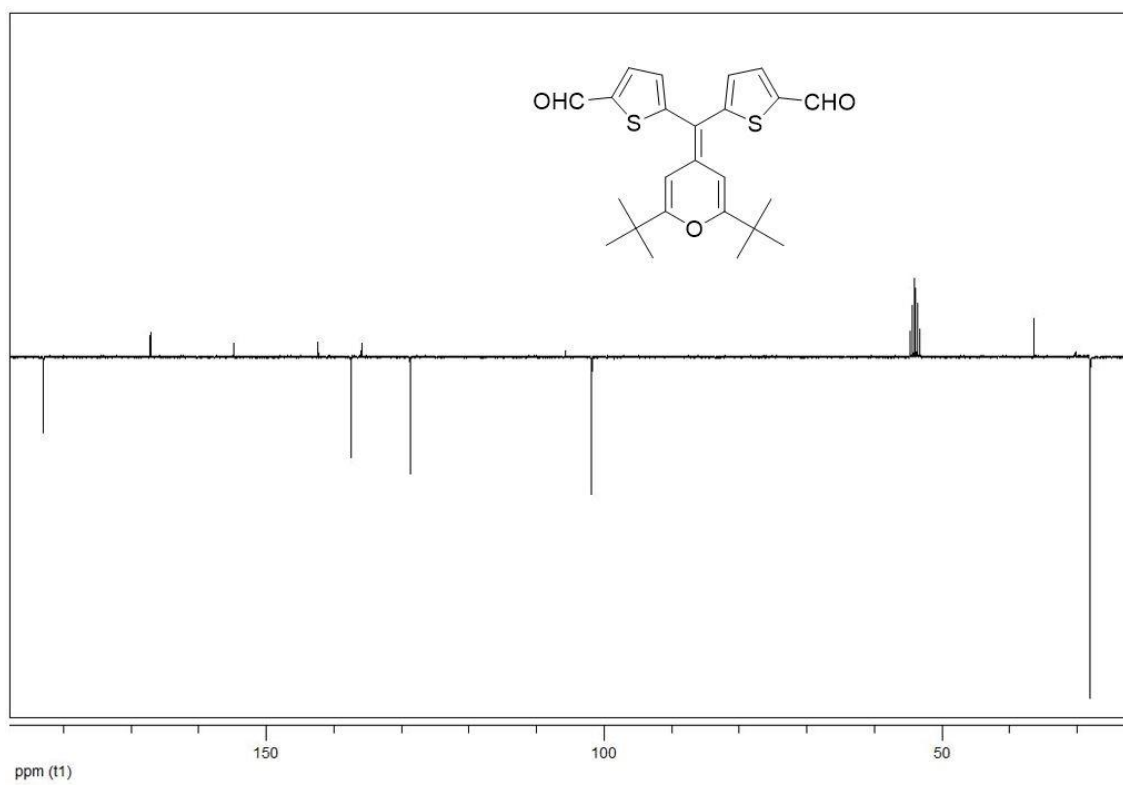


Figura 8.6. Espectro de  $^{13}\text{C-RMN}$  (APT) de 2-CHO.

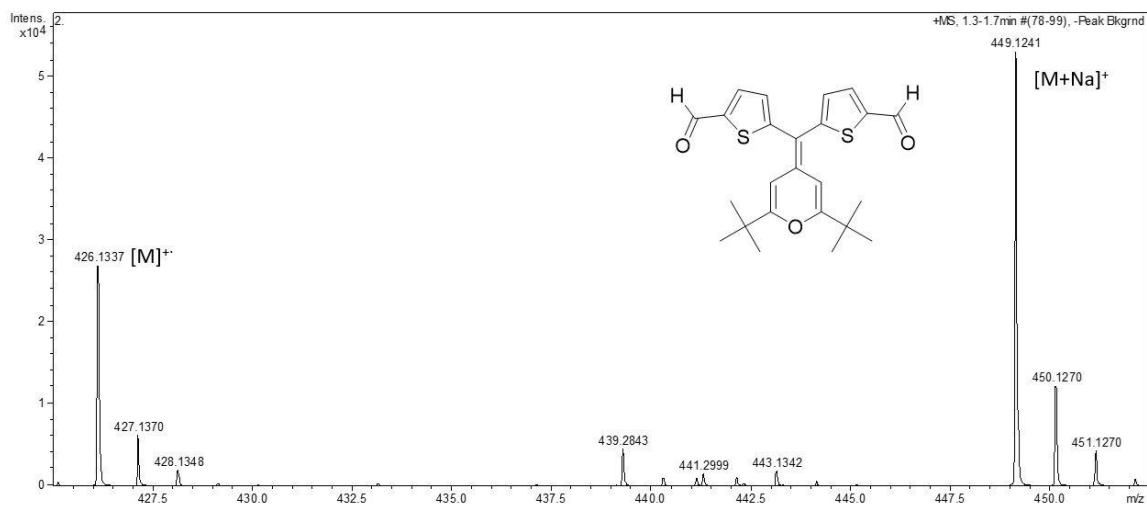
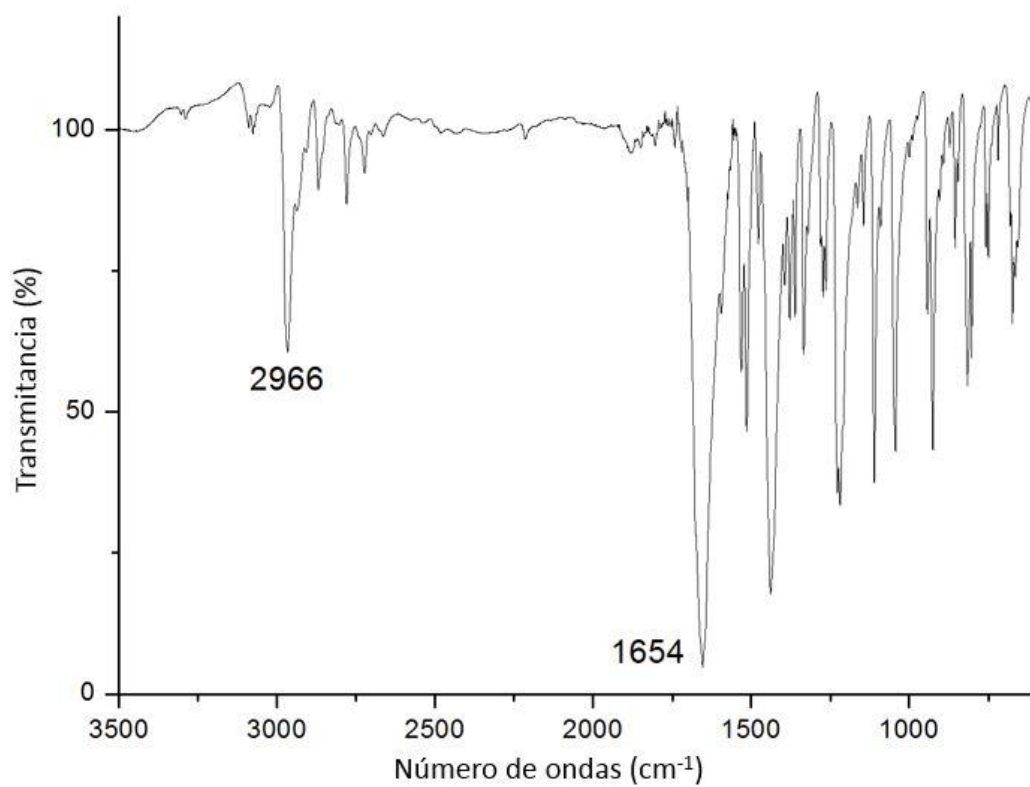
Figura 8.7. Espectro EM (HR-ESI<sup>+</sup>) de 2-CHO.

Figura 8.8. Espectro de IR de 2-CHO.

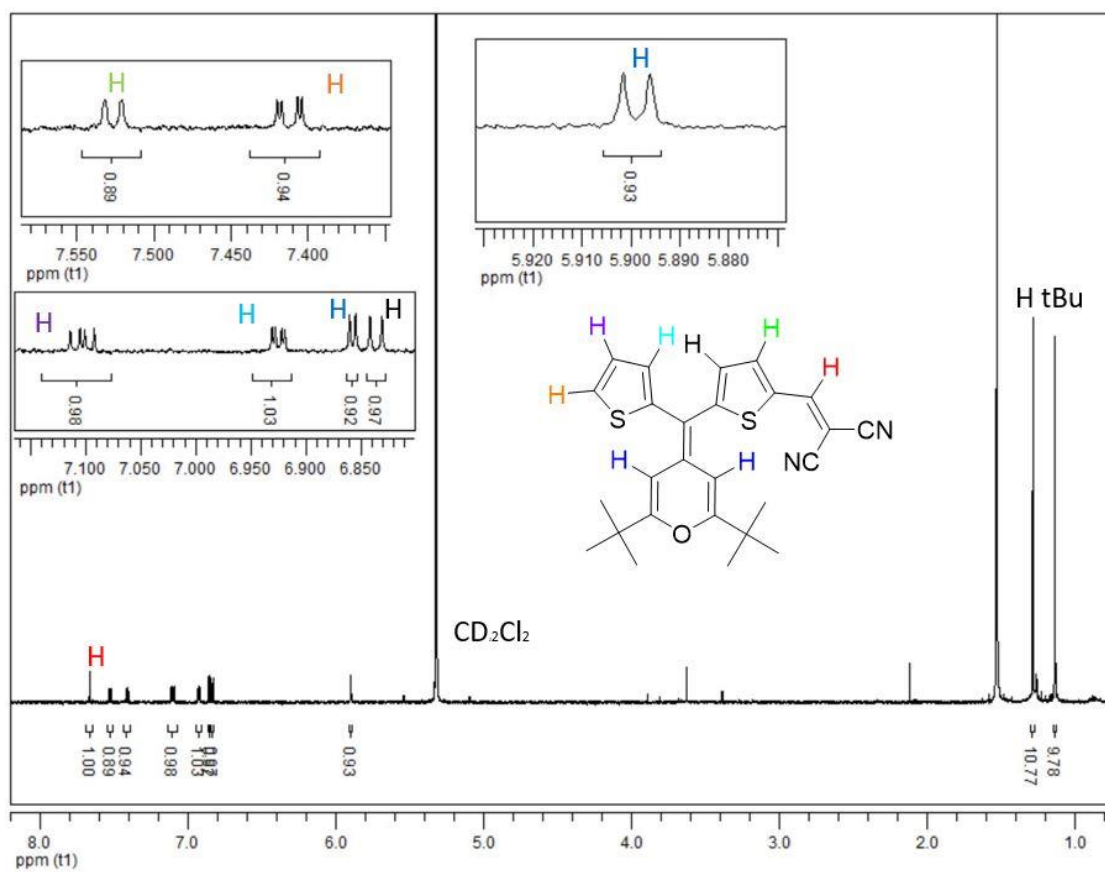


Figura 8.9. Espectro de  $^1\text{H-RMN}$  de **1A**.

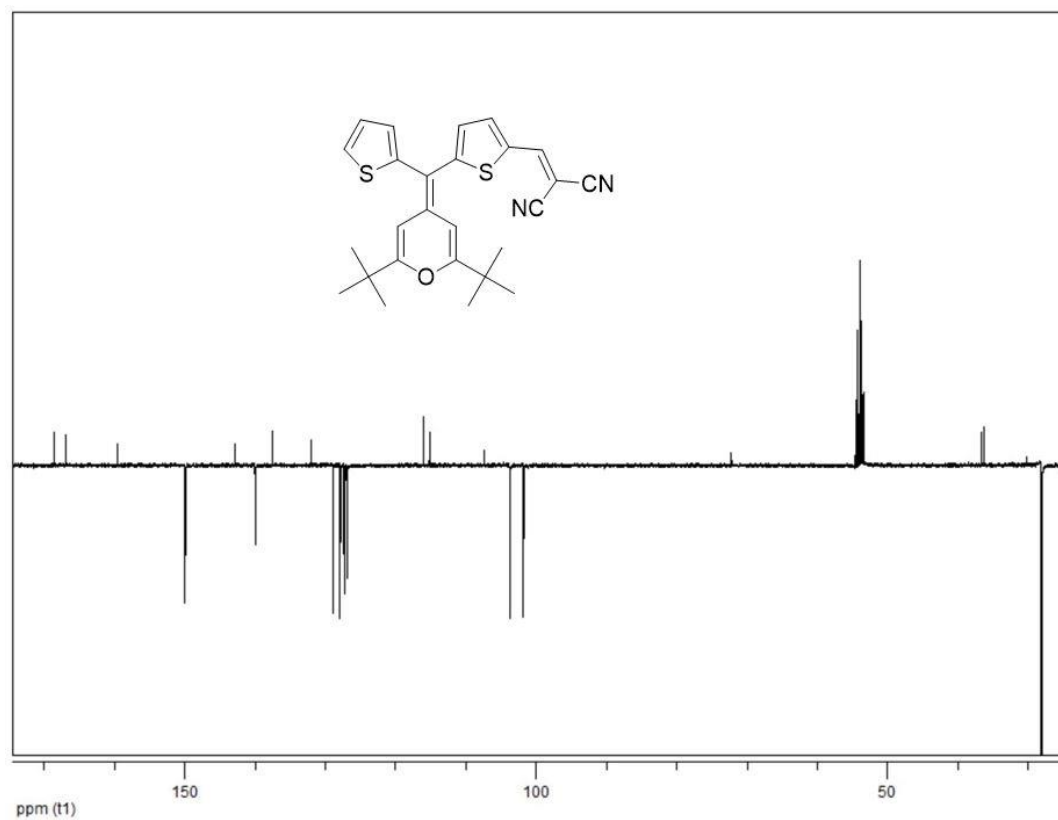


Figura 8.10. Espectro de  $^{13}\text{C-RMN}$  (APT) de **1A**.



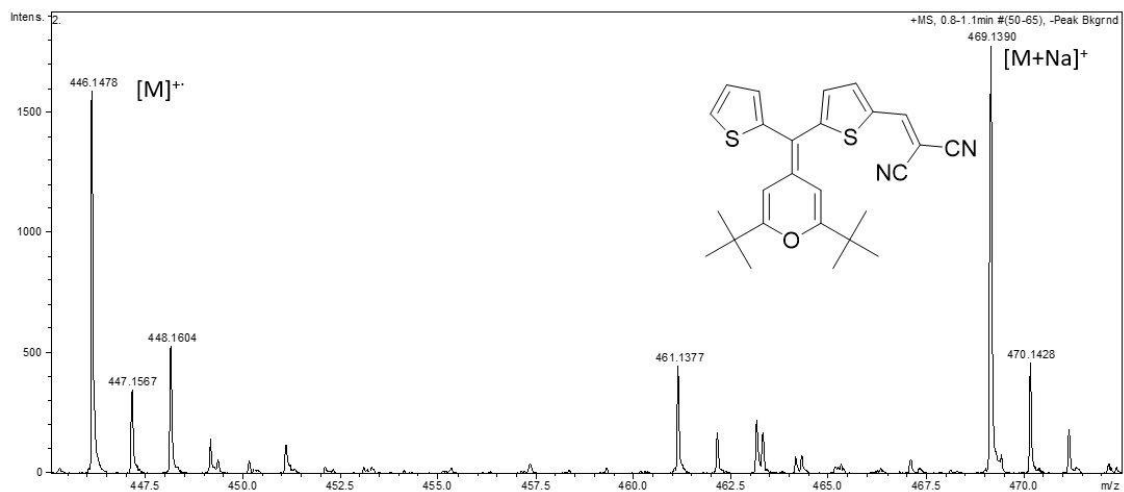


Figura 8.11. Espectro EM (HR-ESI<sup>+</sup>) de **1A**.

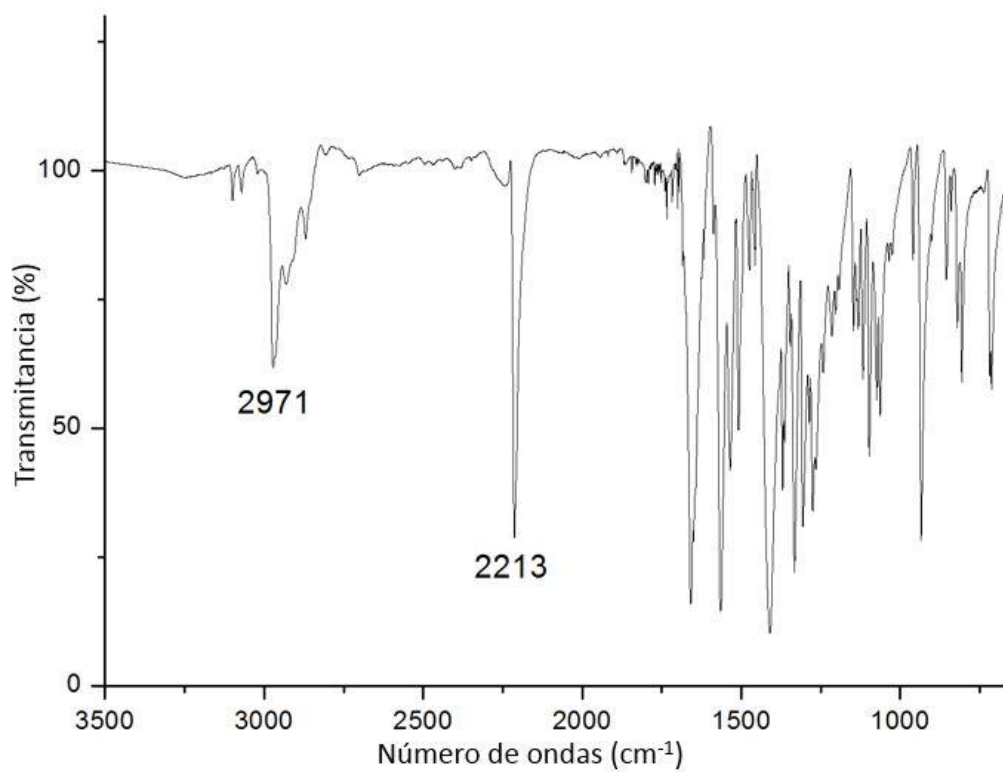
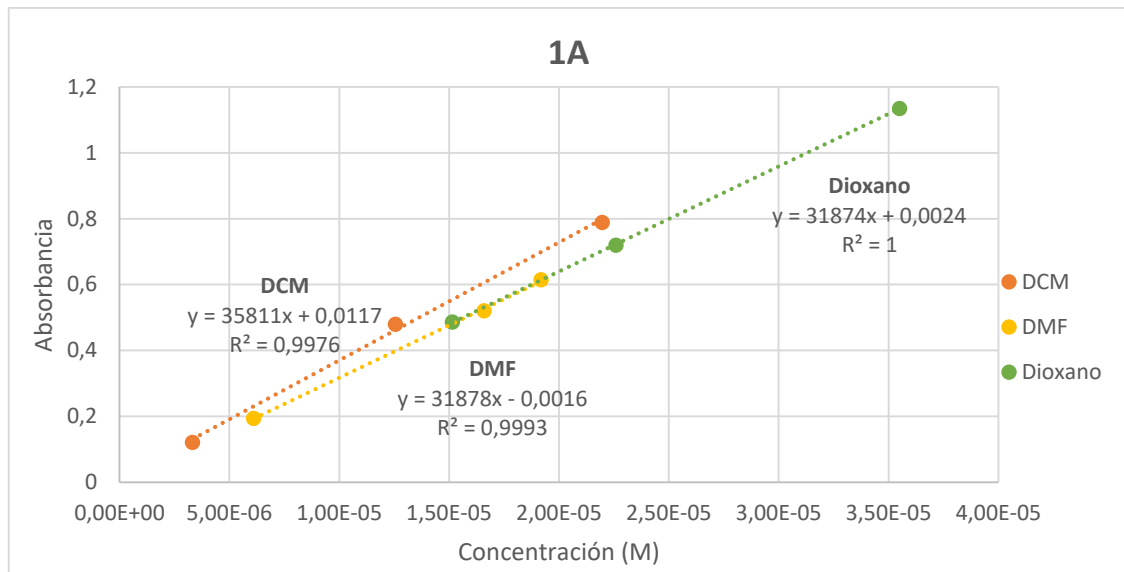
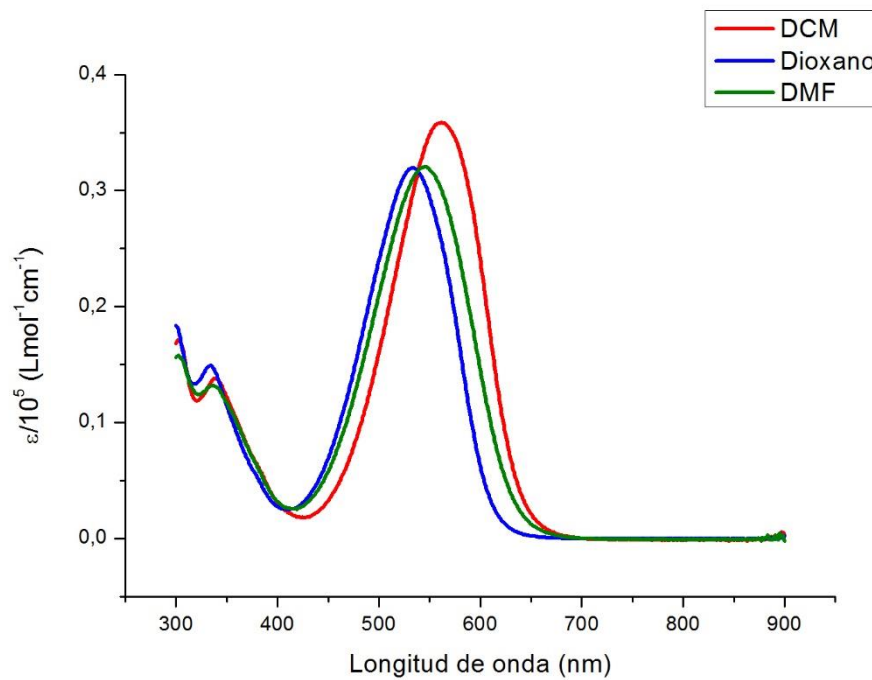


Figura 8.12. Espectro de IR de **1A**.

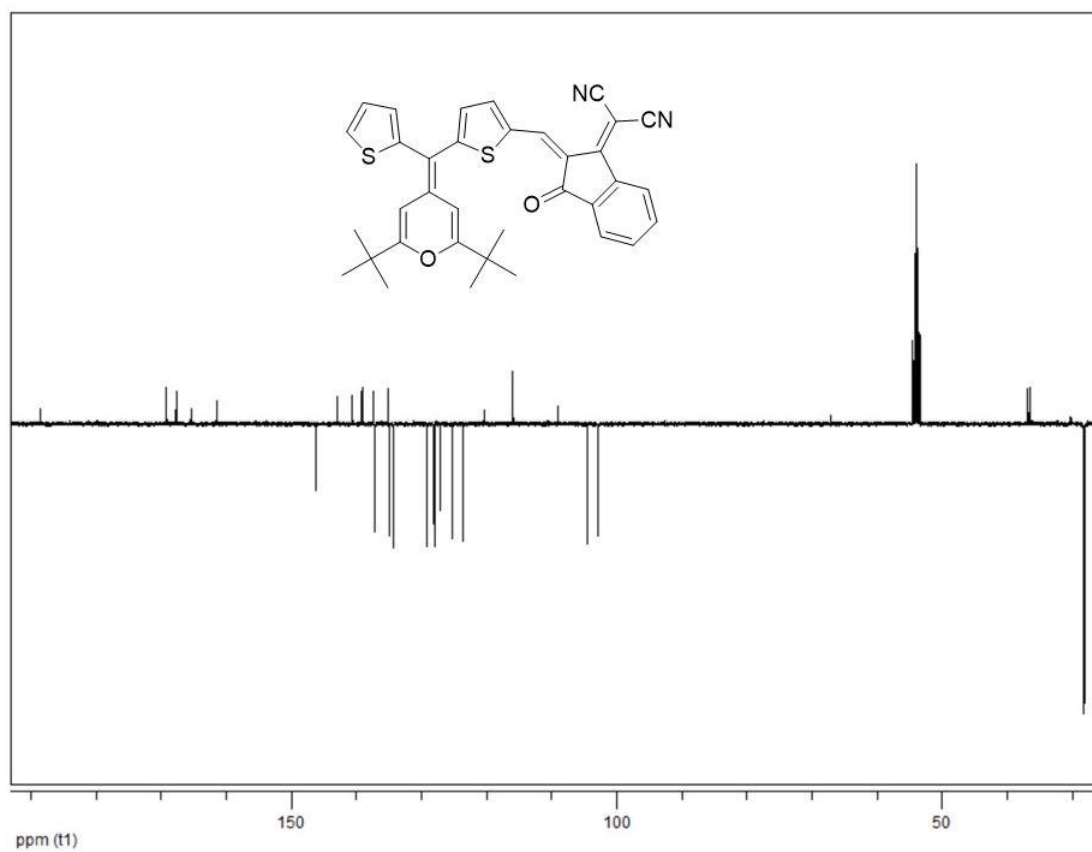
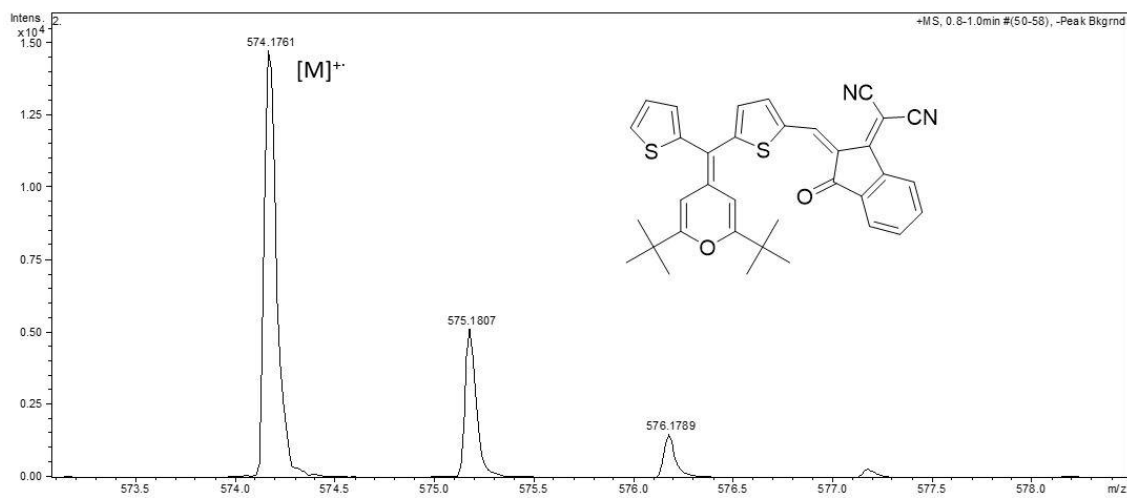


**Gráfica 8.1.** Recta de calibrado para **1A**.



**Figura 8.13.** Espectro de UV-Visible de **1A**



Figura 8.16. Espectro de  $^{13}\text{C}$ -RMN (APT) de 1B.Figura 8.17. Espectro EM (HR-ESI $^+$ ) de 1B.

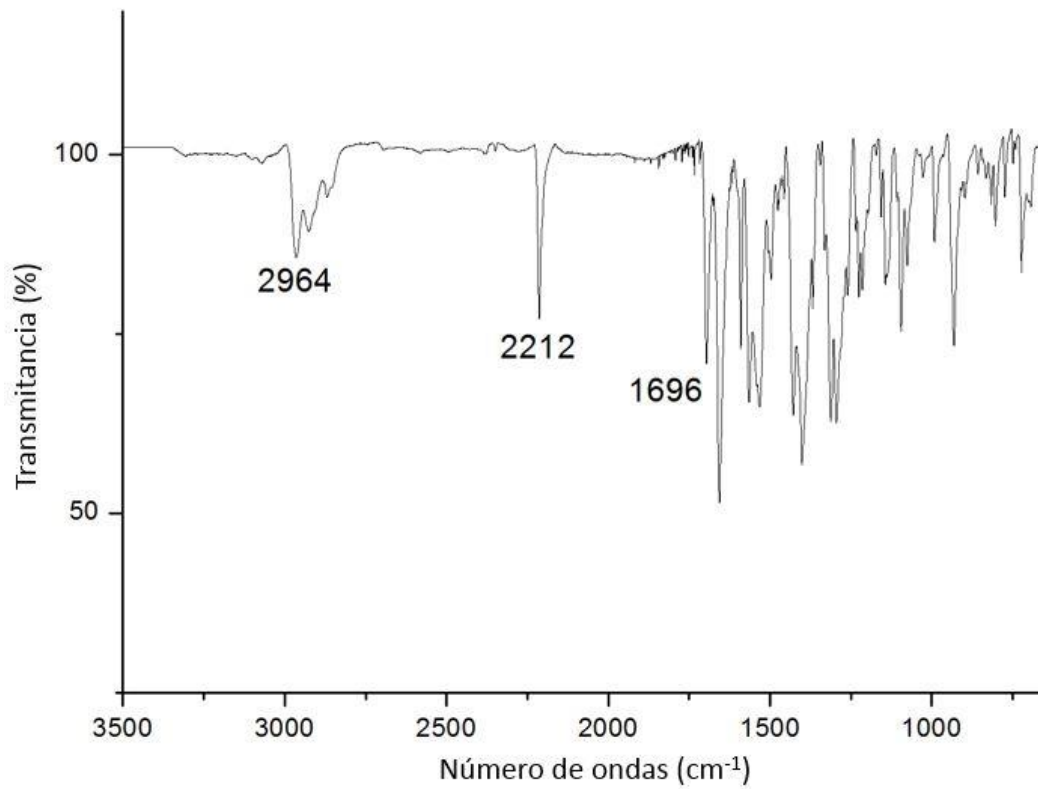
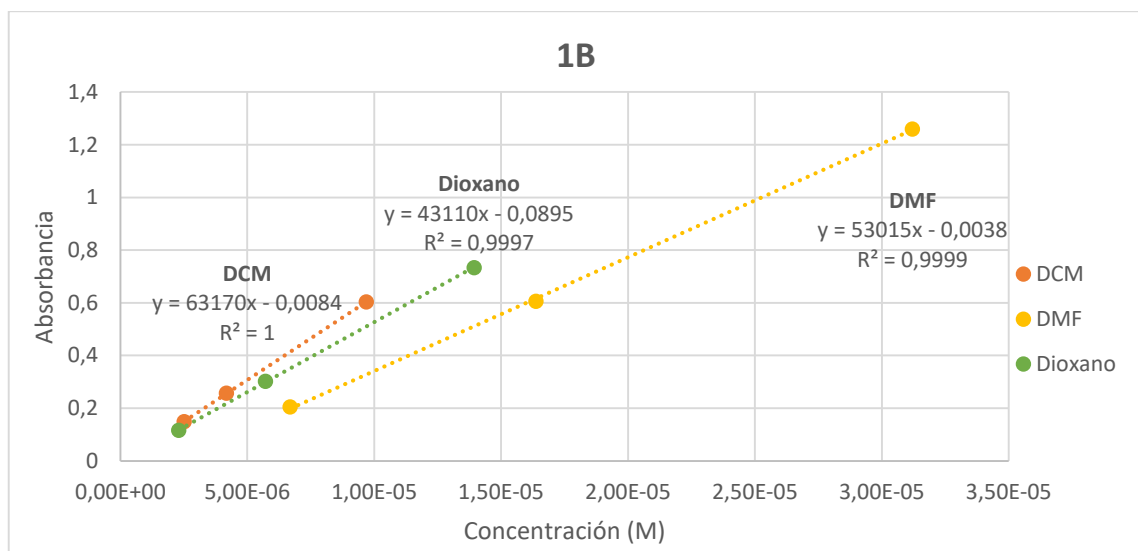


Figura 8.18. Espectro de IR de 1B.



Gráfica 8.2. Recta de calibrado para 1B.

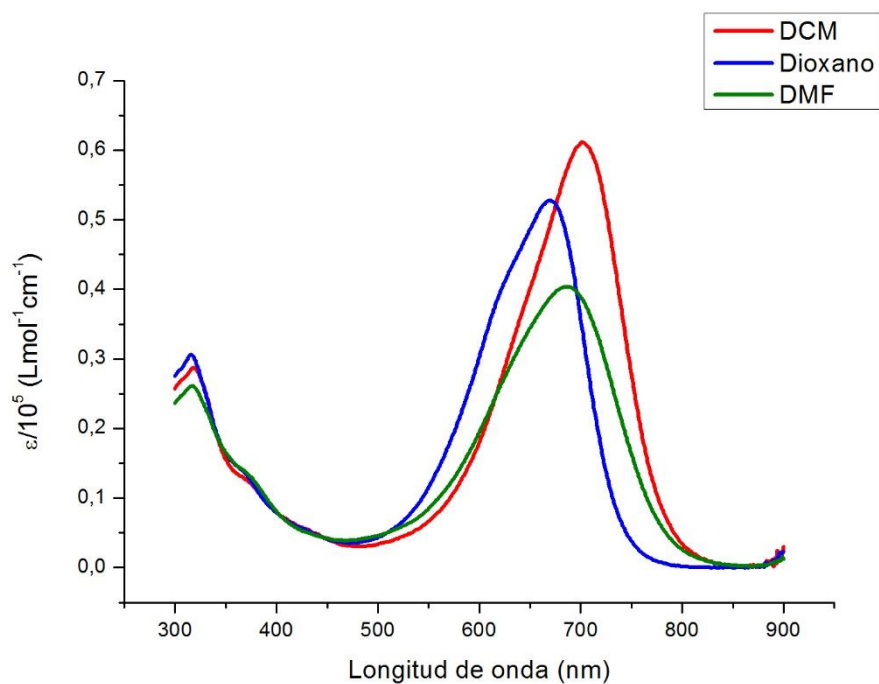


Figura 8.19. Espectro de UV-Visible de 1B.

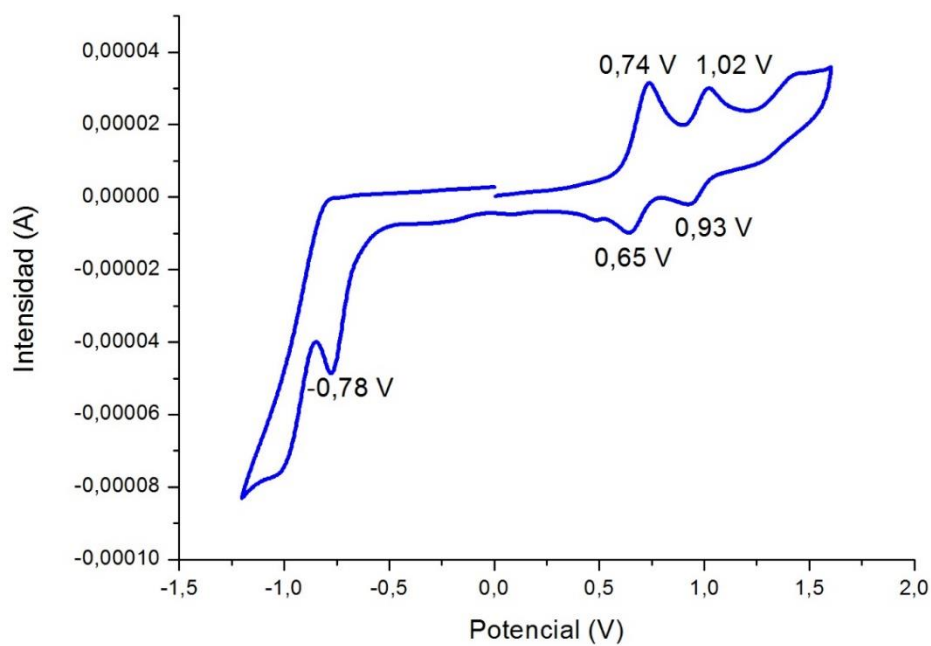
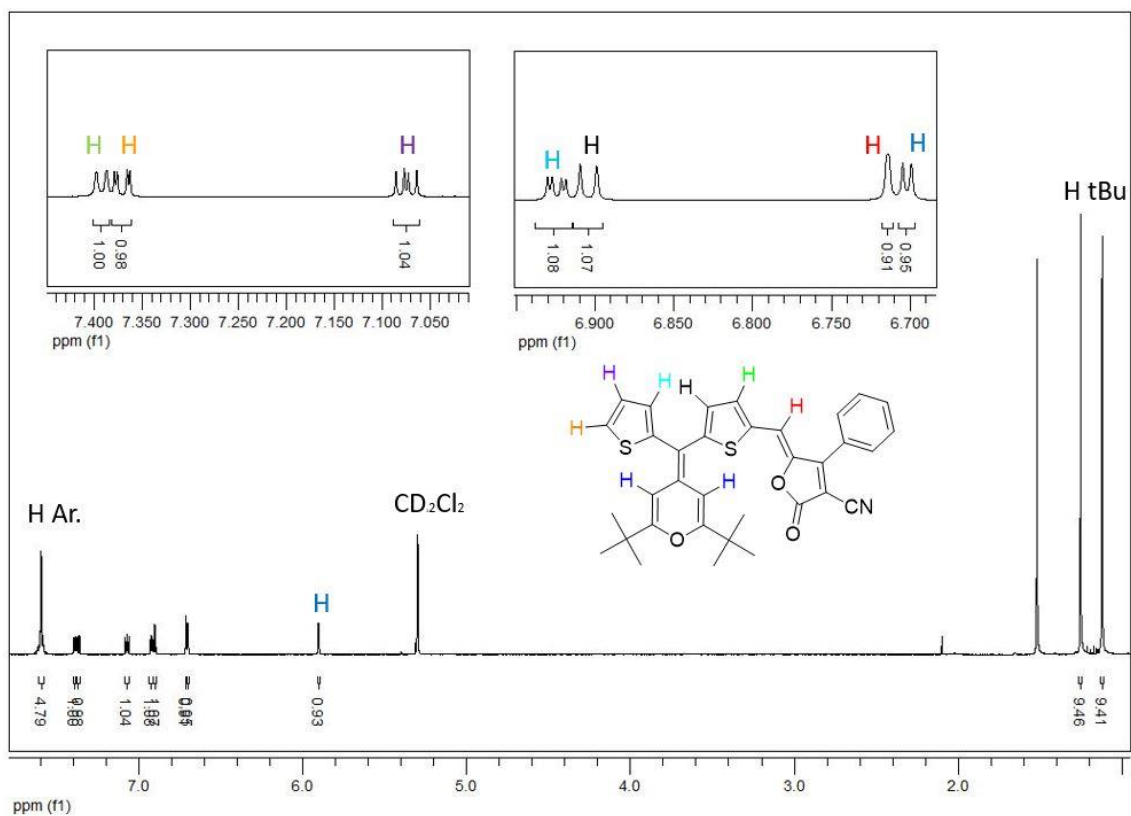
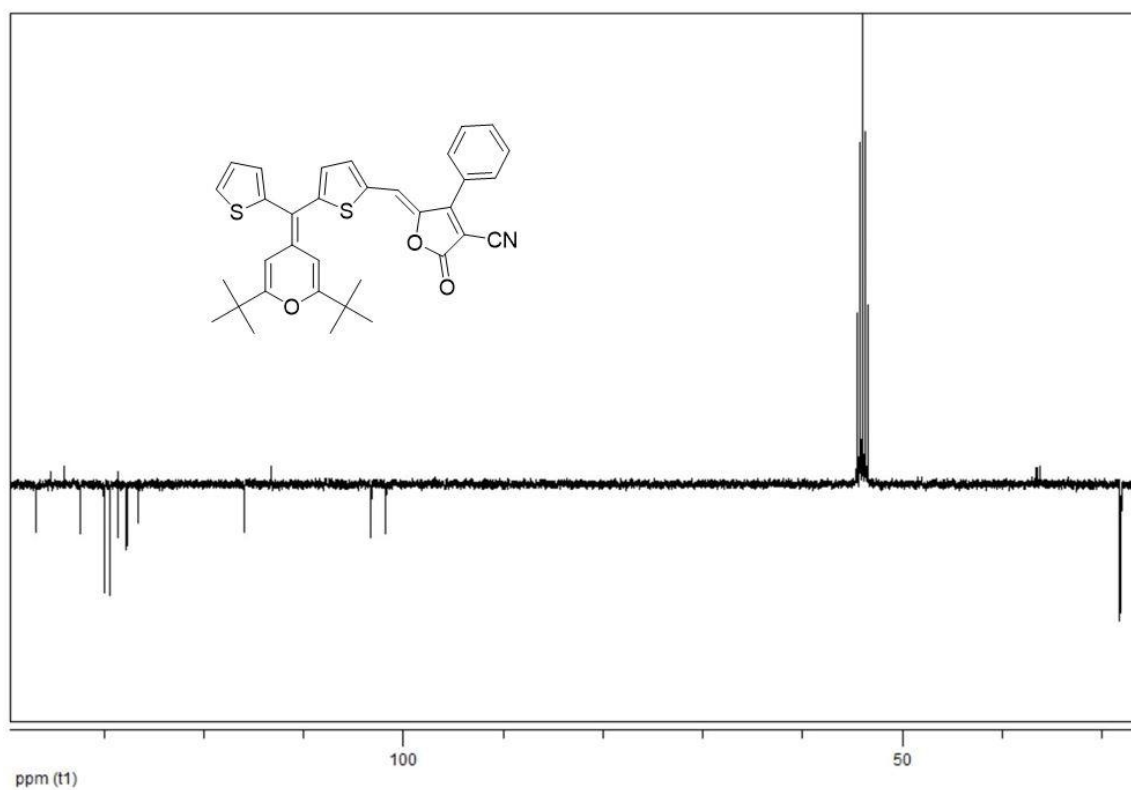


Figura 8.20. Voltamperograma de 1B.

Figura 8.21. Espectro de  $^1\text{H-RMN}$  de **1C**.Figura 8.22. Espectro de  $^{13}\text{C-RMN}$  (APT) de **1C**.

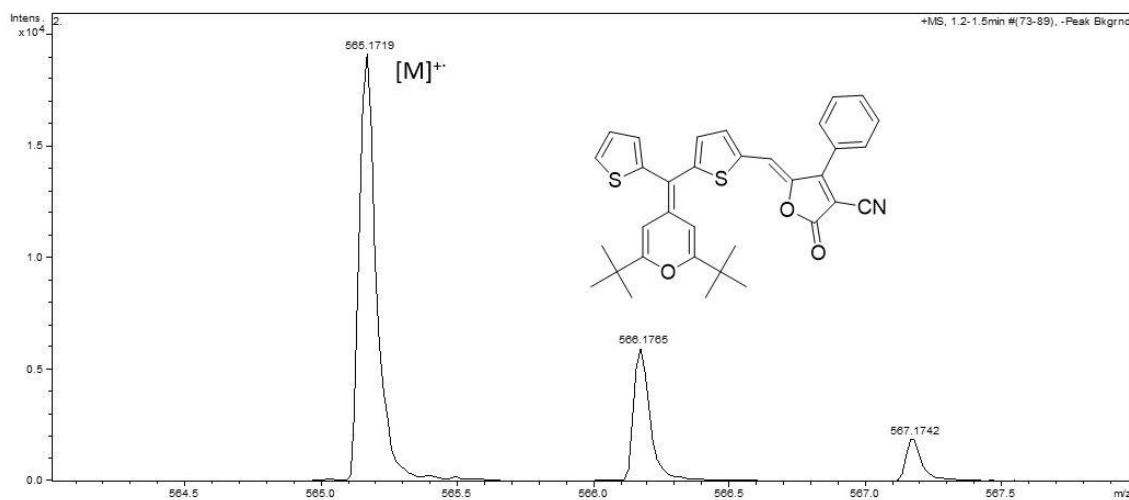


Figura 8.23. Espectro EM (HR-ESI<sup>+</sup>) de 1C.

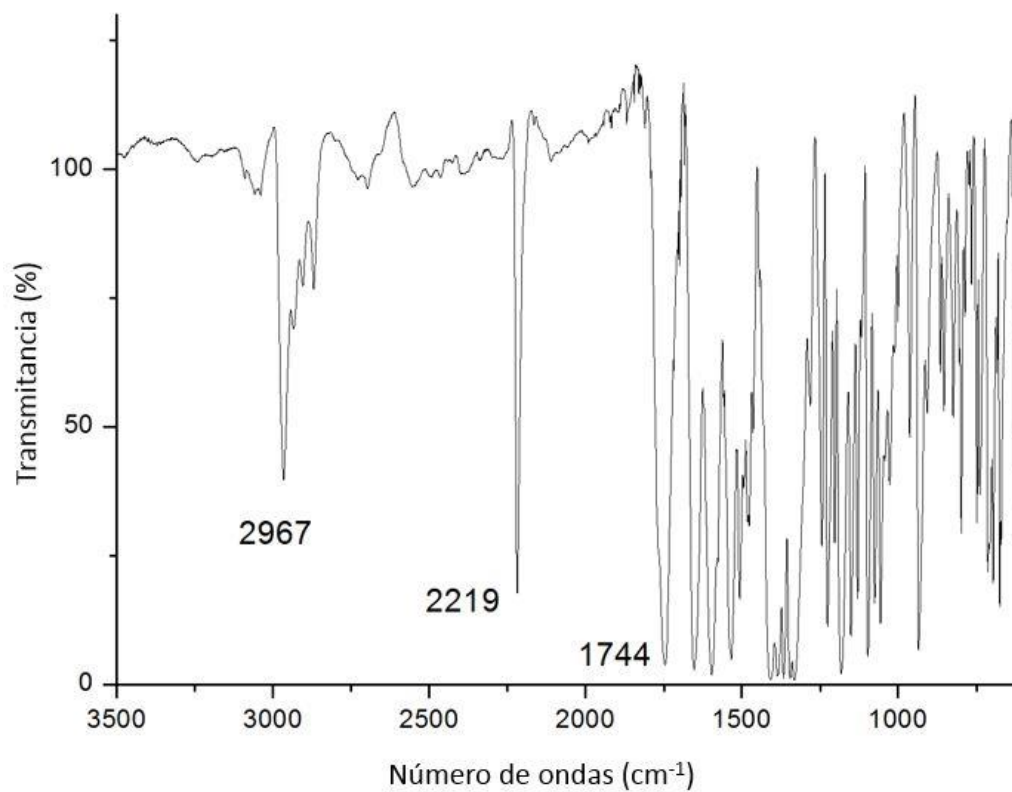
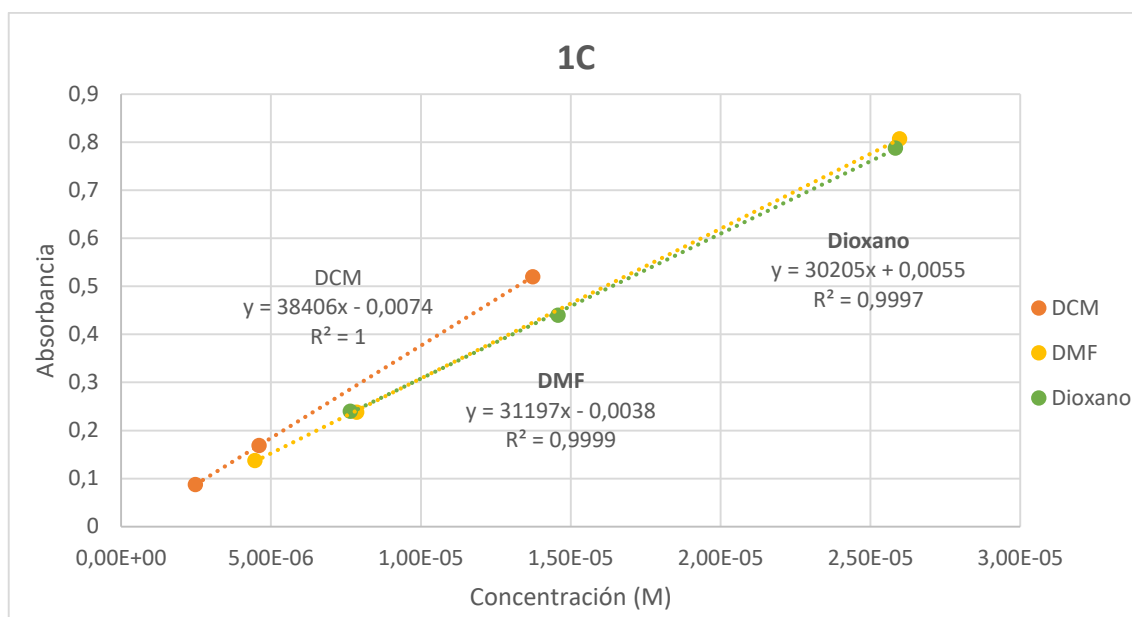
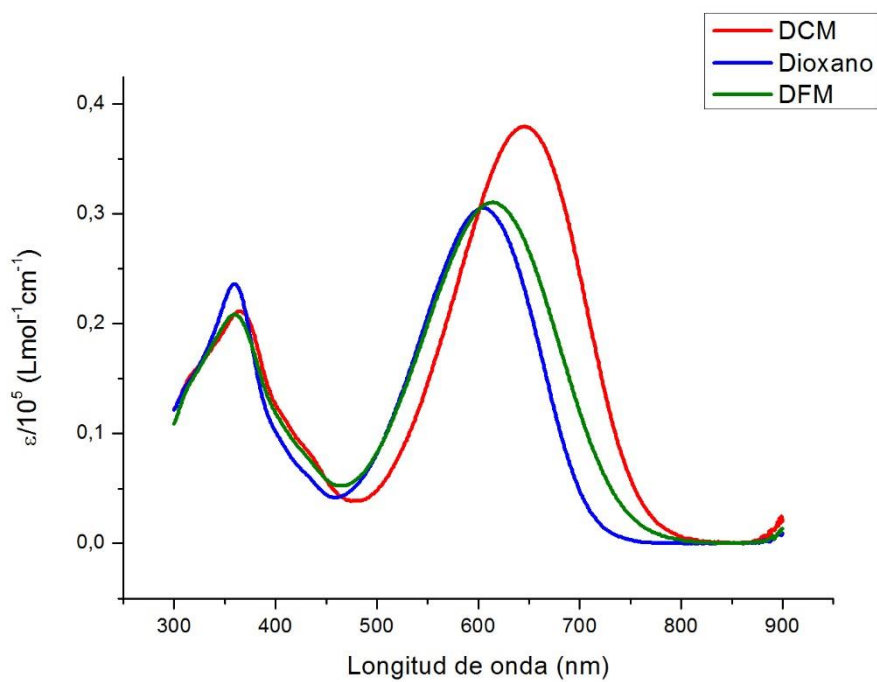


Figura 8.24. Espectro de IR de 1C.





**Gráfica 8.3.** Recta de calibrado para **1C**.



**Figura 8.25.** Espectro de UV-Visible de **1C**.

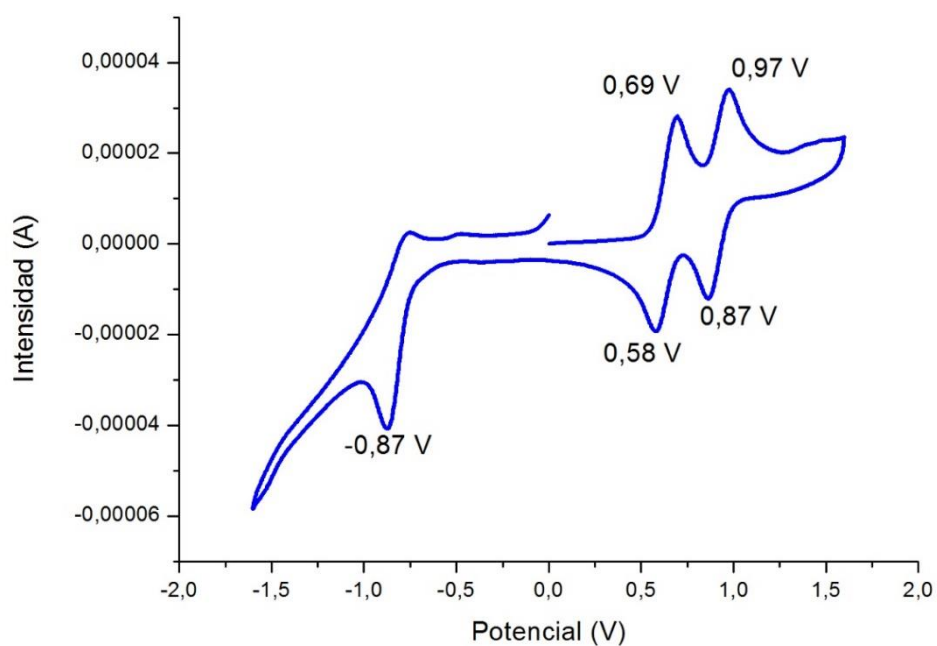


Figura 8.26. Voltamperograma de 1C.

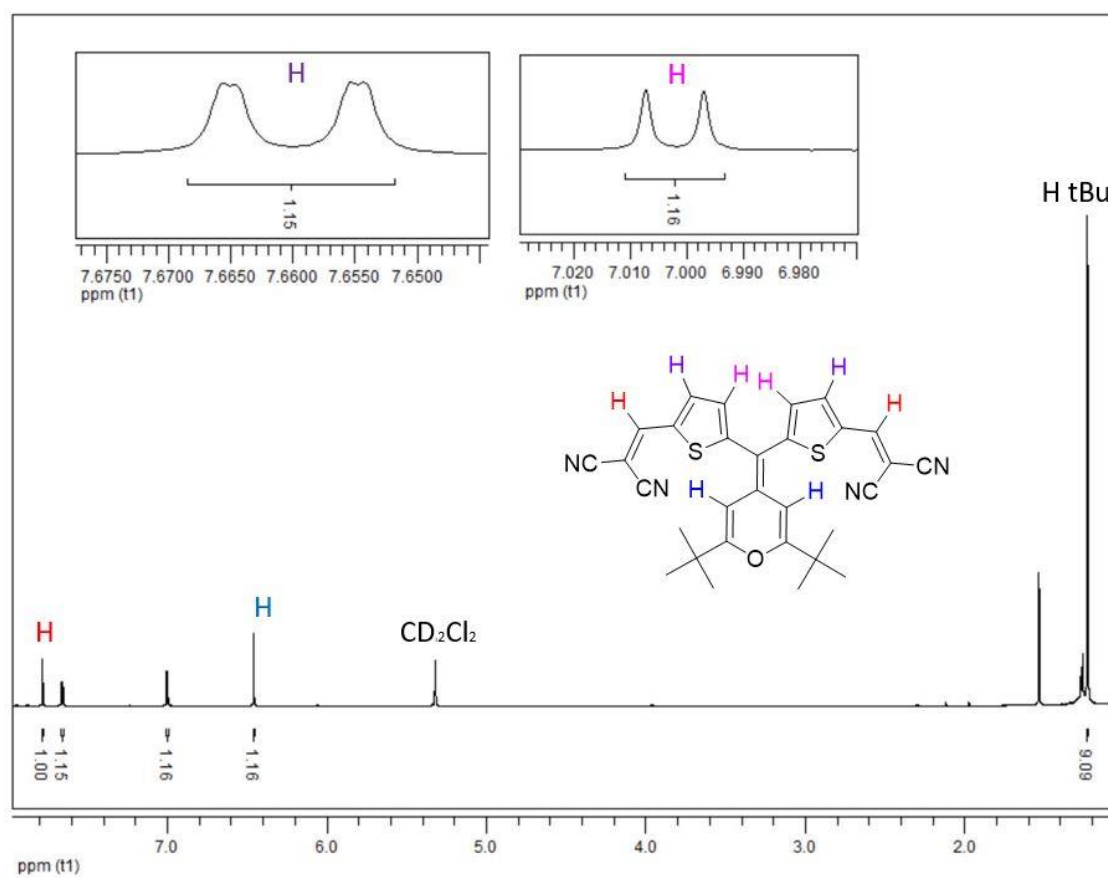


Figura 8.27. Espectro de  $^1\text{H}$ -RMN de 2A.



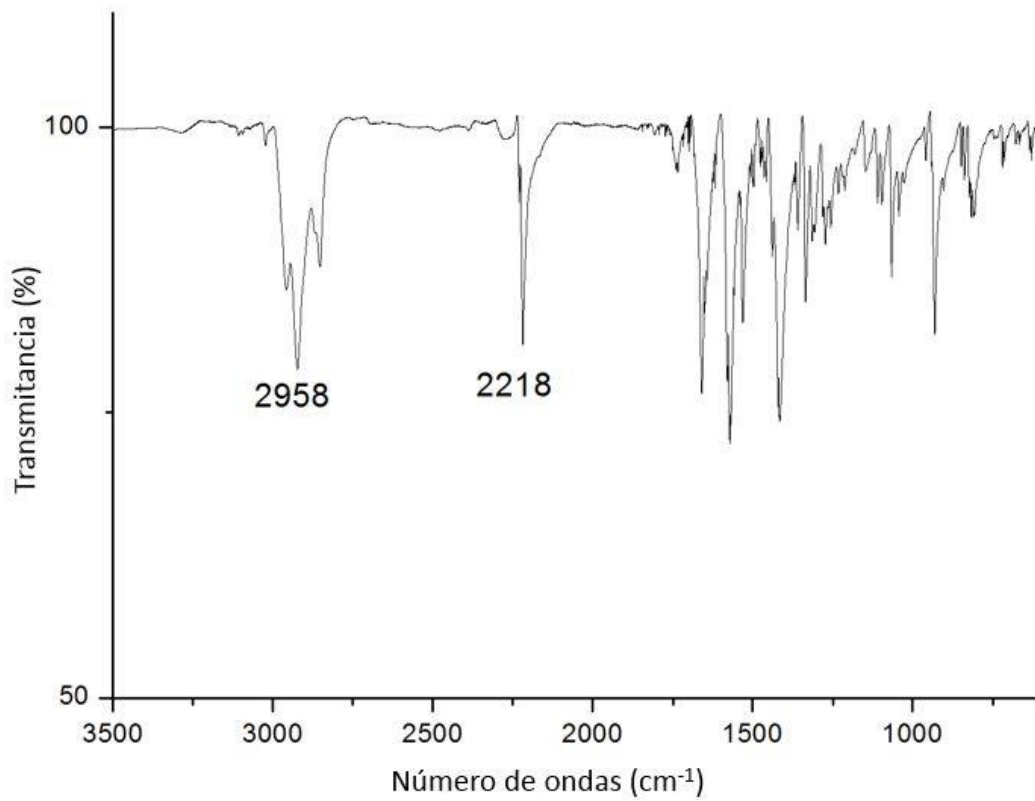
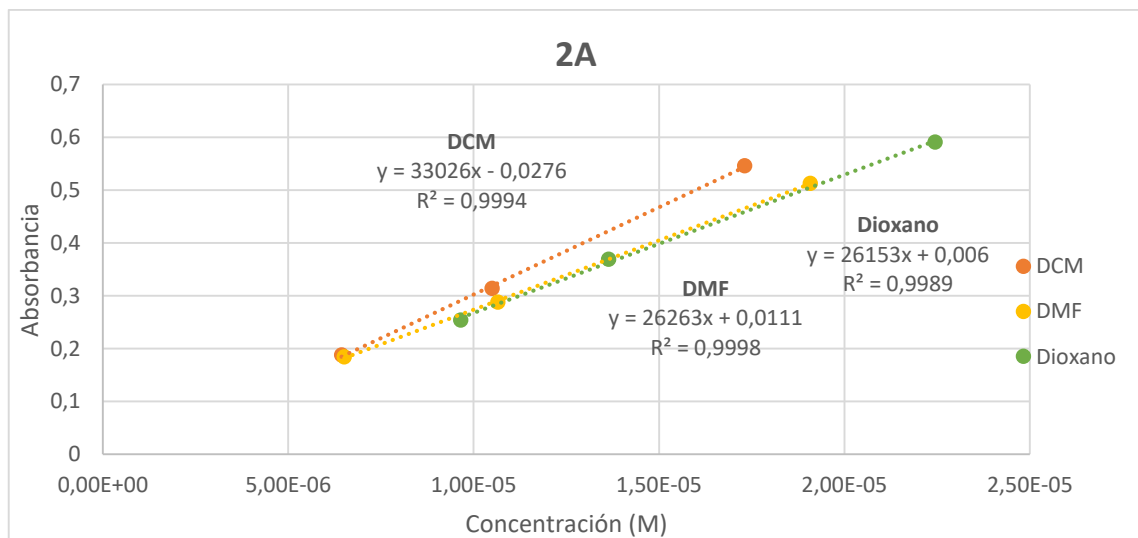


Figura 8.30. Espectro de IR de 2A.



Gráfica 8.4. Recta de calibrado para 2A.

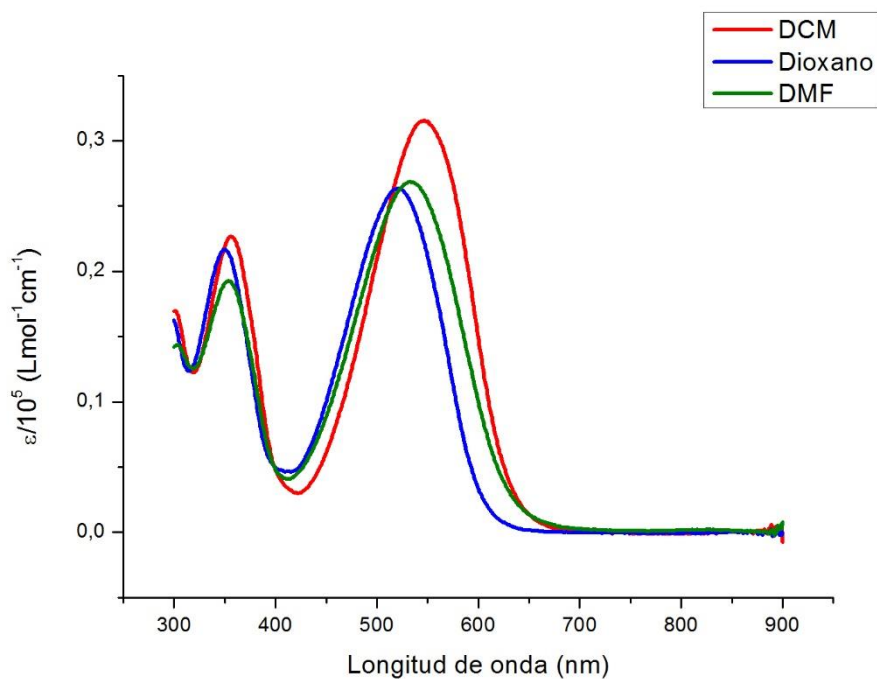


Figura 8.31. Espectro de UV-Visible de 2A.

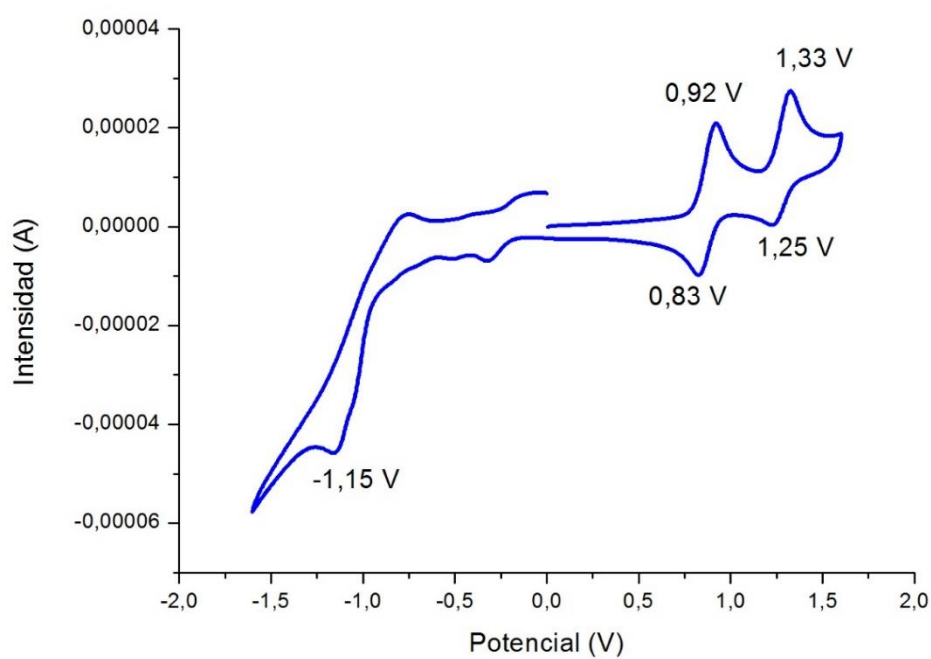


Figura 8.32. Voltamperograma de 2A.

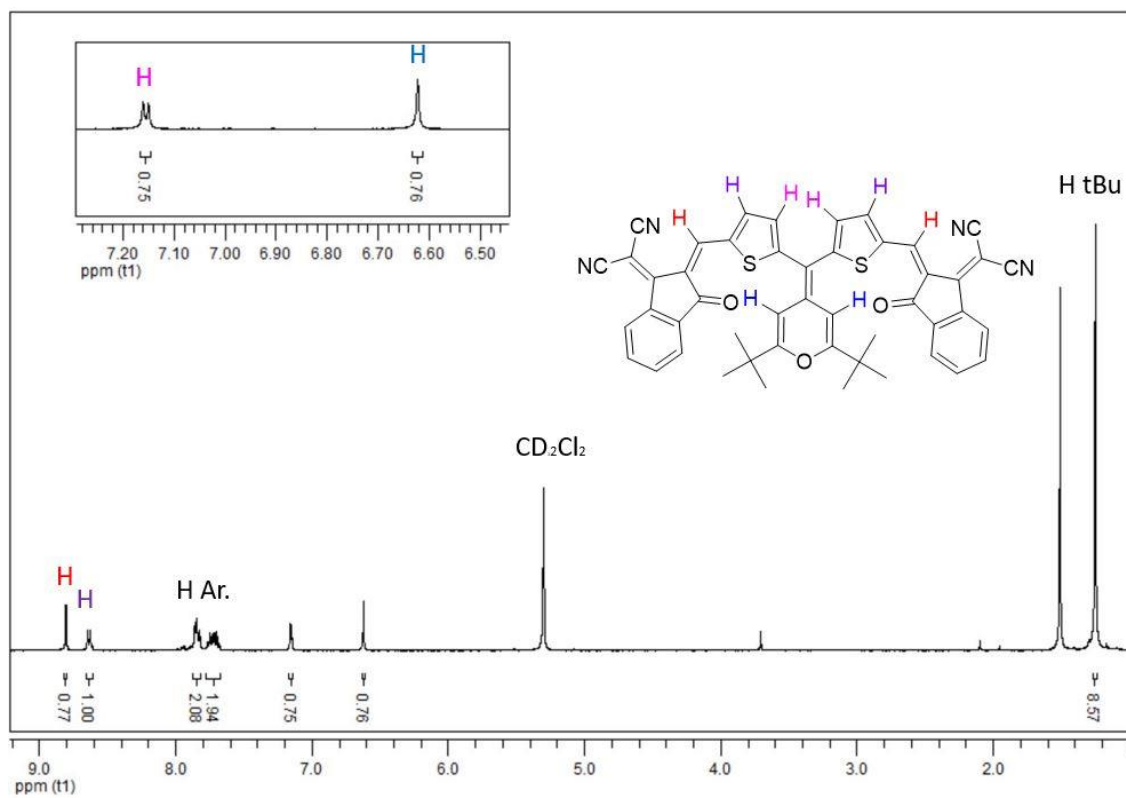


Figura 8.33. Espectro de  $^1\text{H-RMN}$  de **2B**.

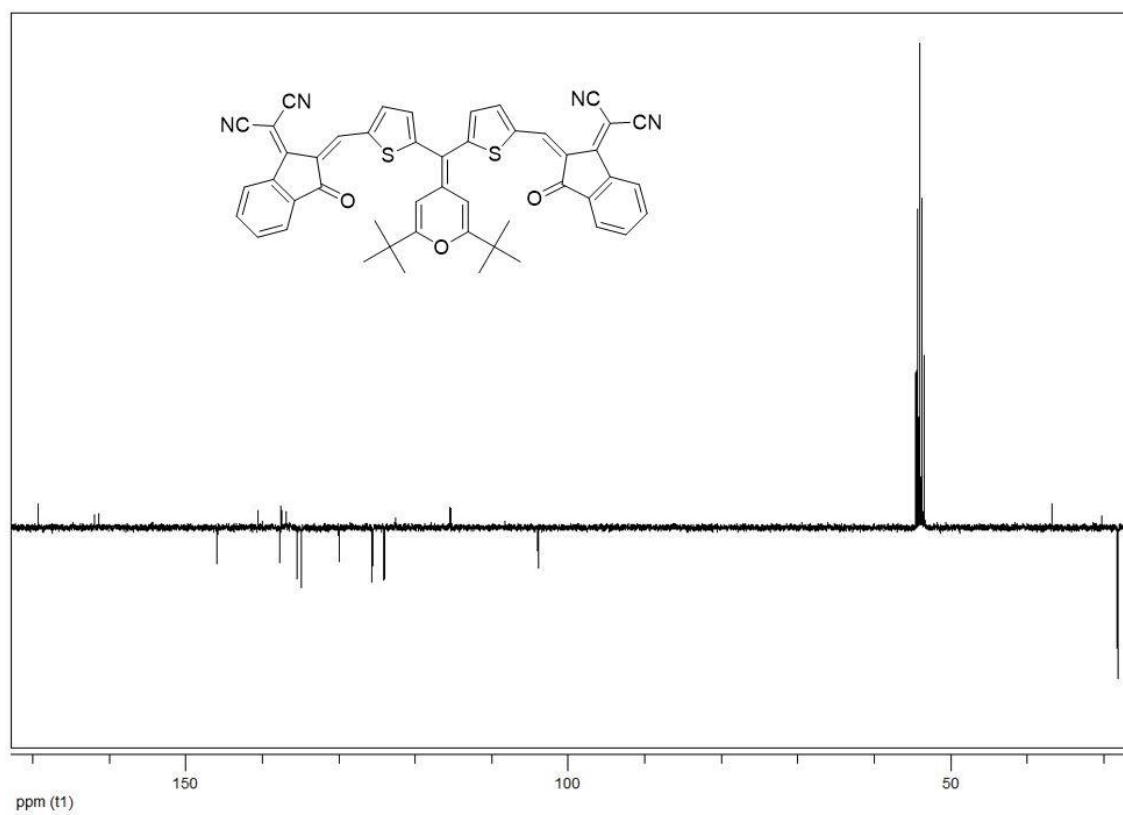


Figura 8.34. Espectro de  $^{13}\text{C-RMN}$  (APT) de **2B**.

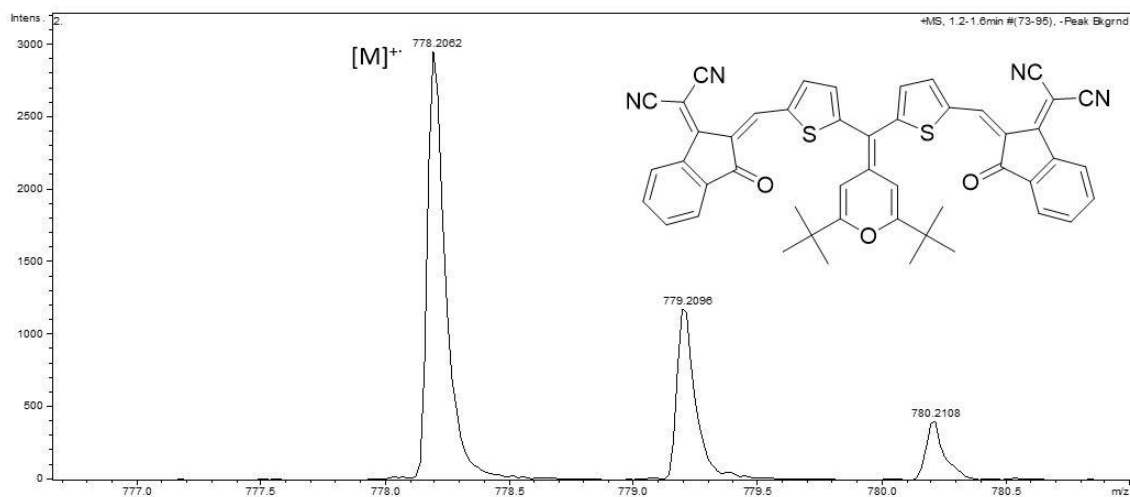


Figura 8.35. Espectro EM (HR-ESI<sup>+</sup>) de 2B.

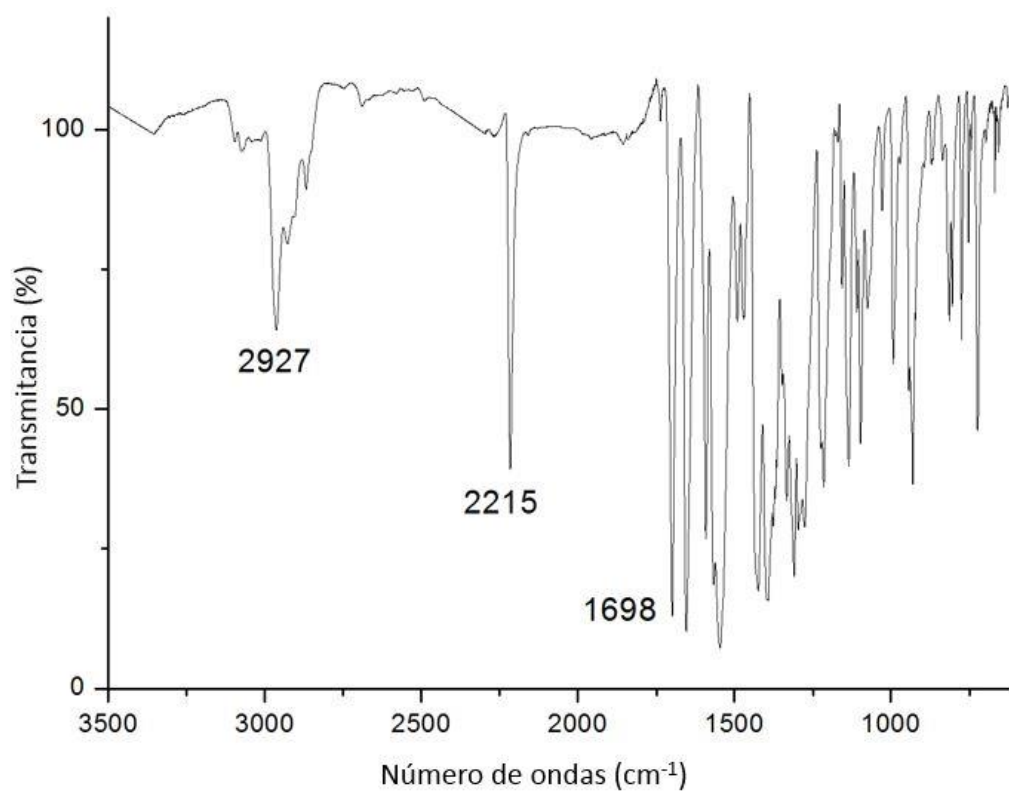
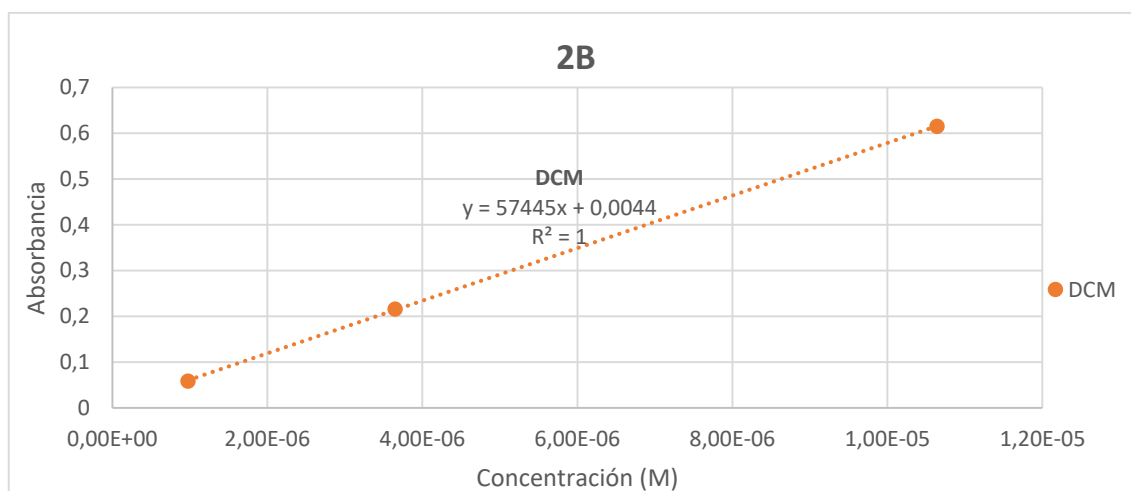


Figura 8.36. Espectro de IR de 2B.



Gráfica 8.5. Recta de calibrado para 2B.

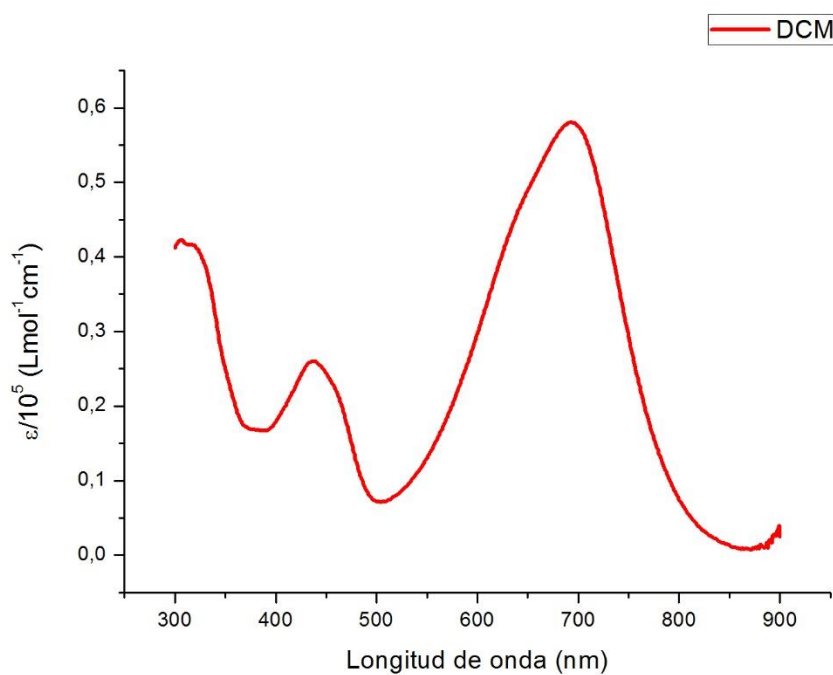


Figura 8.37. Espectro de UV-Visible de 2B en DCM.



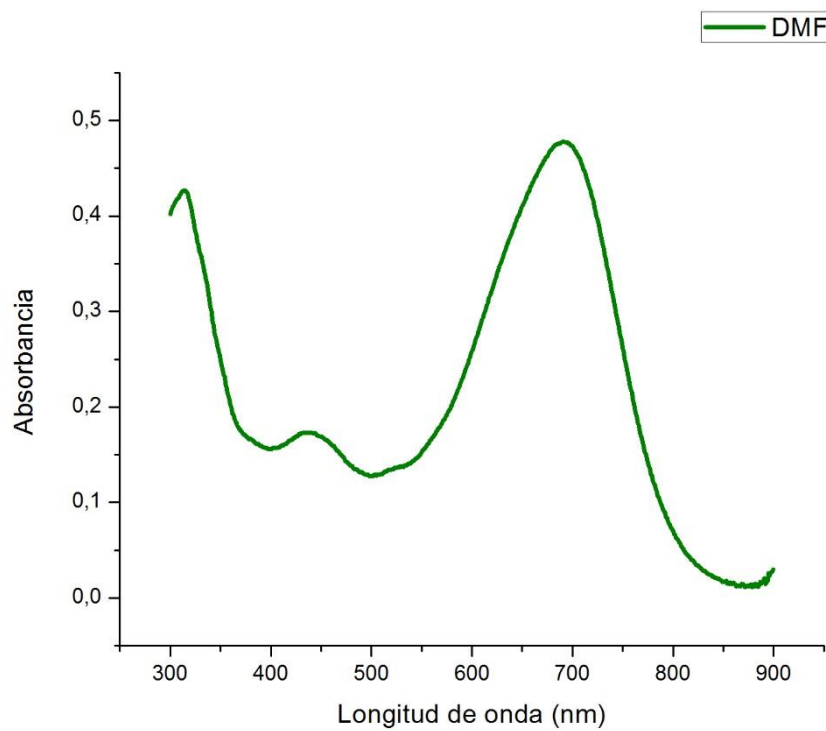


Figura 8.38. Espectro de UV-Visible de **2B** en DMF.

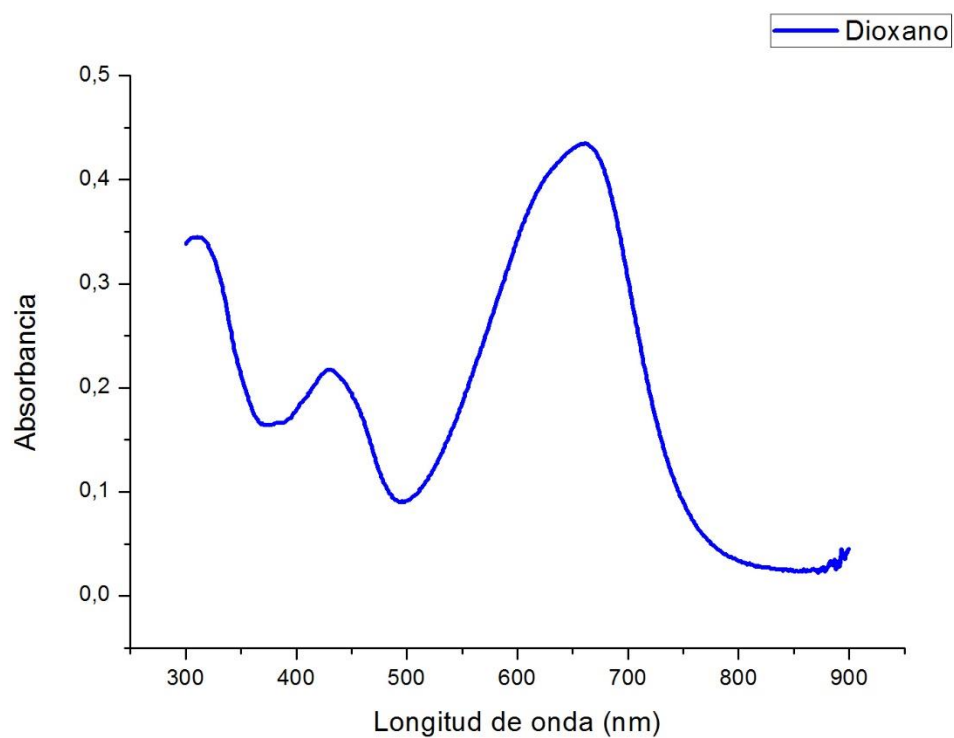


Figura 8.39. Espectro de UV-Visible de **2B** en dioxano.

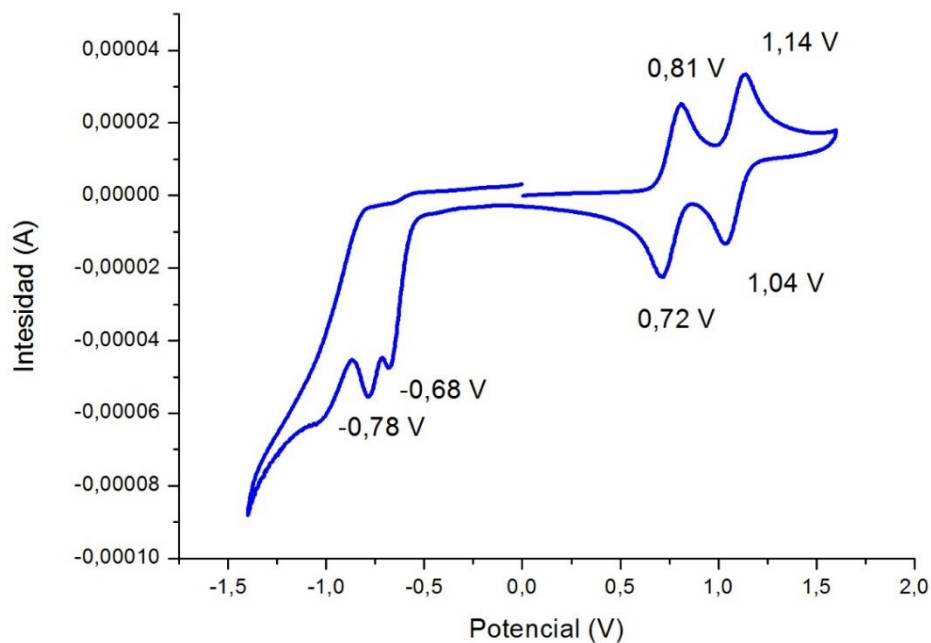


Figura 8.40. Voltamperograma de **2B**.

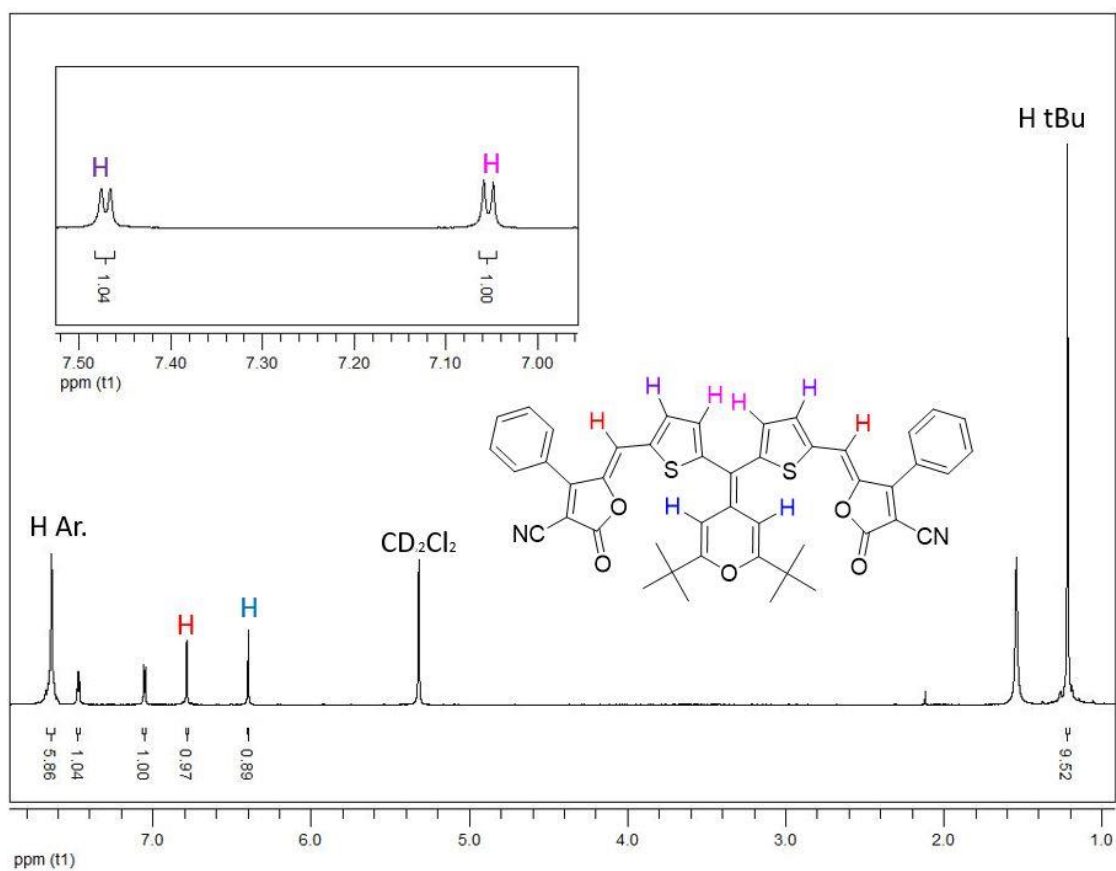
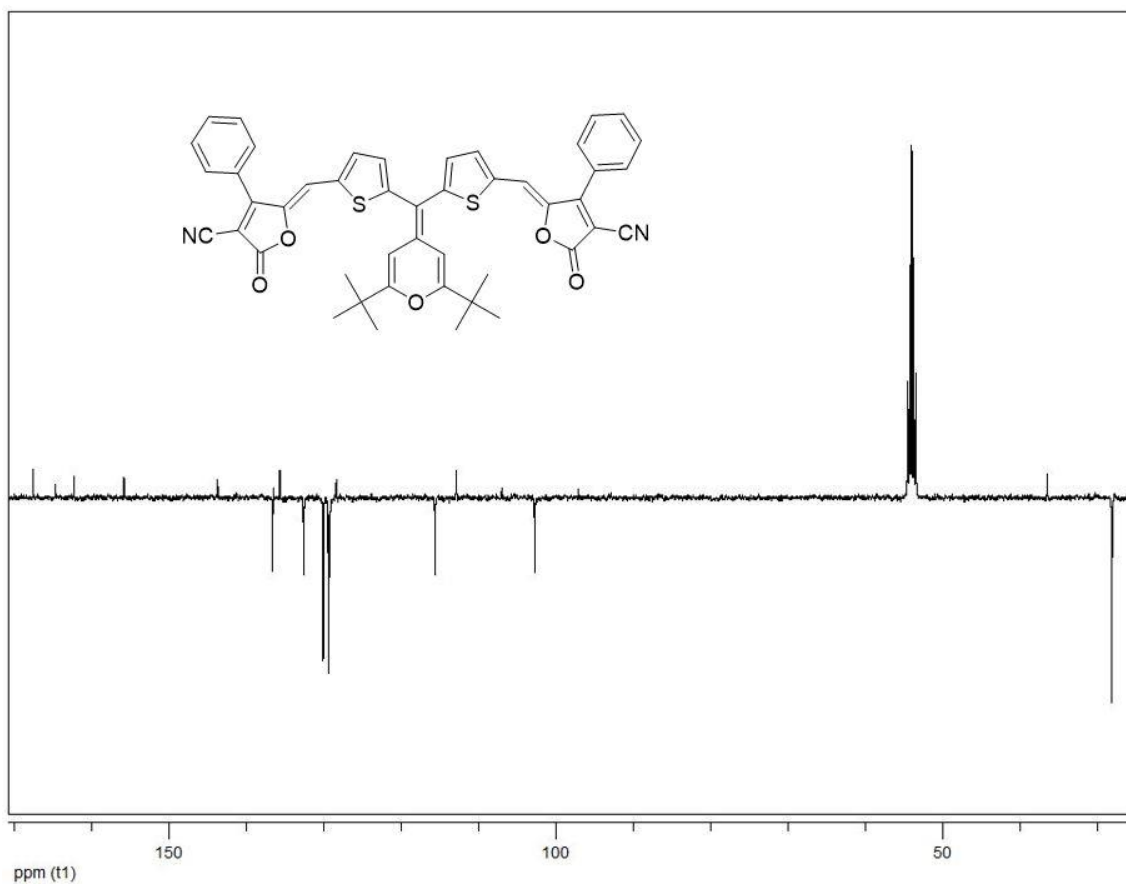
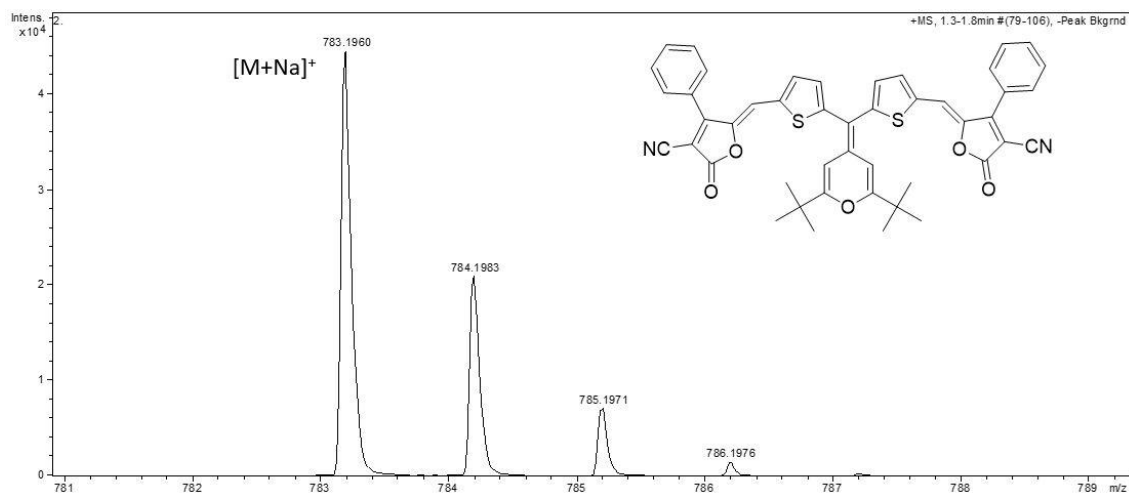


Figura 8.41. Espectro de  $^1\text{H}$ -RMN de **2C**.

Figura 8.42. Espectro de  $^{13}\text{C}$ -RMN (APT) de **2C**.Figura 8.43. Espectro EM (HR-ESI<sup>+</sup>) de **2C**.

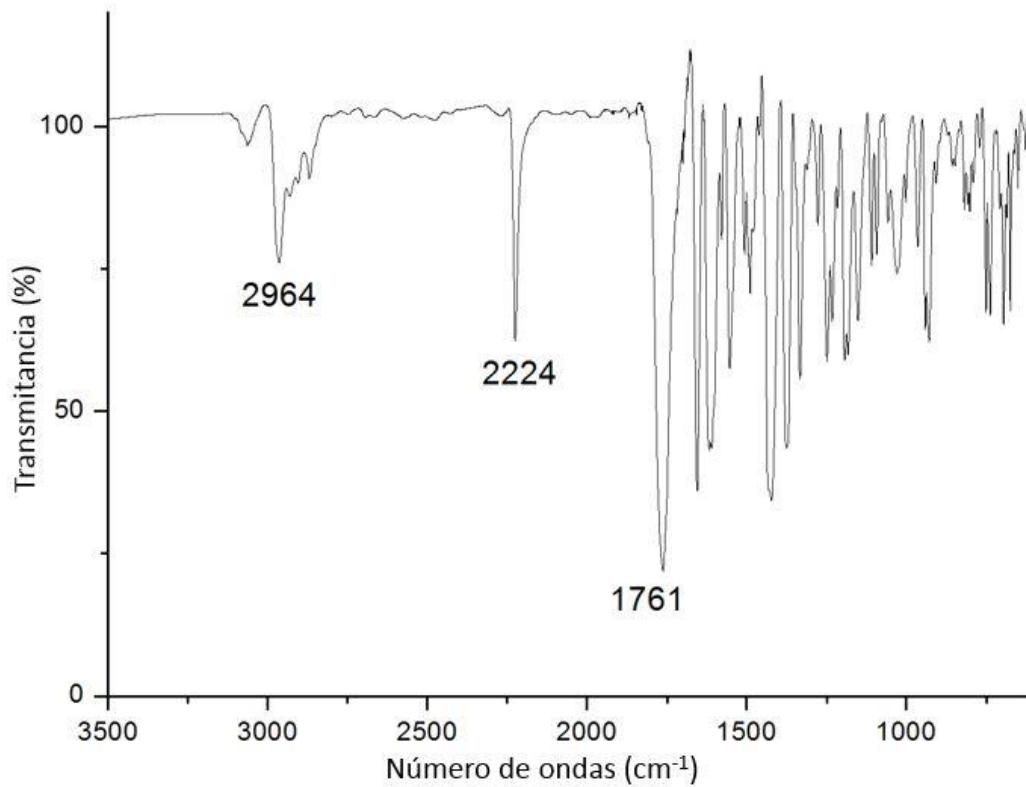
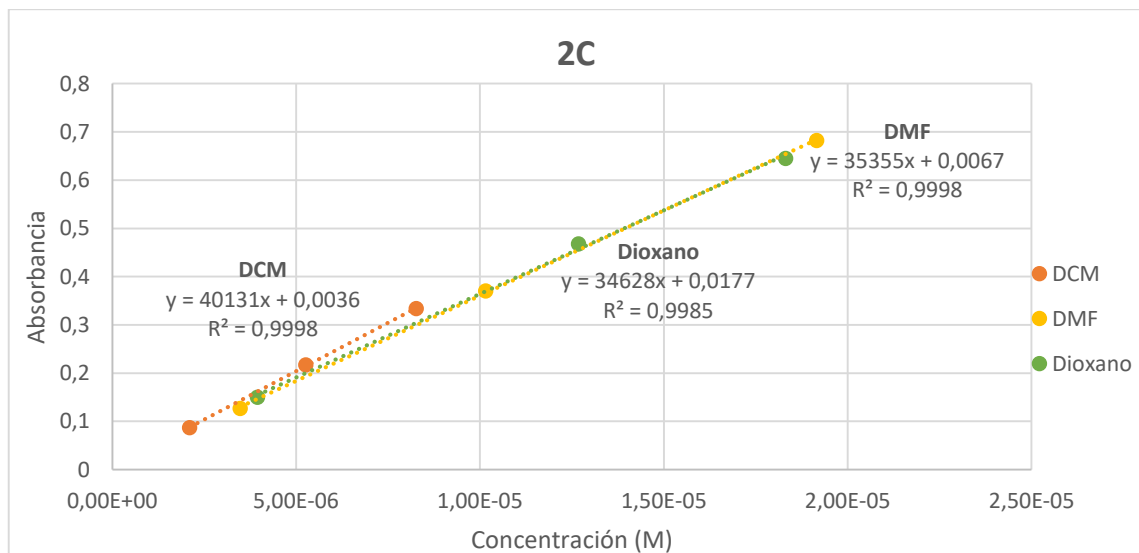


Figura 8.44. Espectro de IR de 2C.



Gráfica 8.6. Recta de calibrado para 2C.

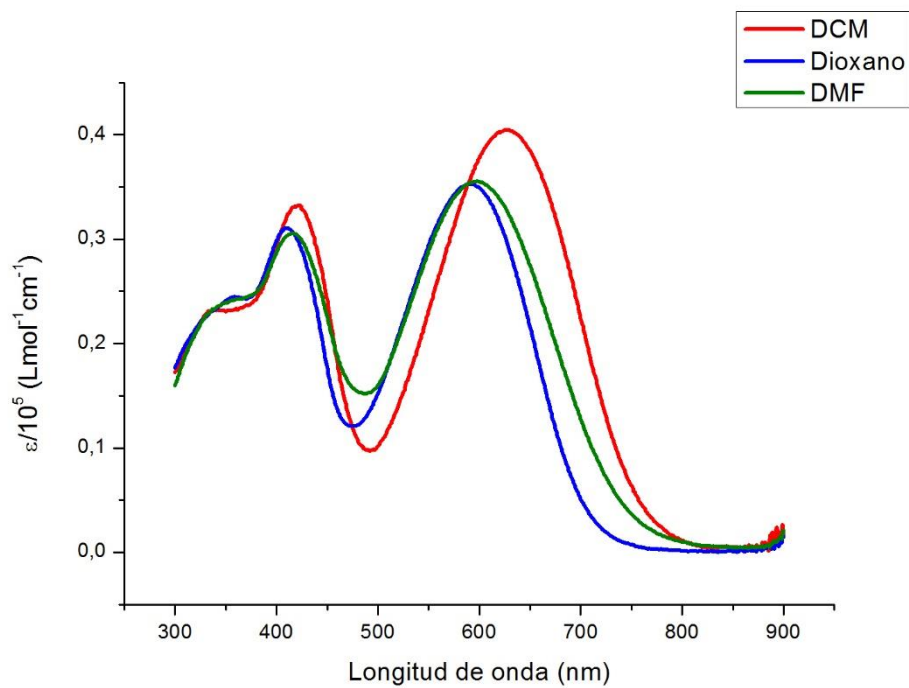


Figura 8.45. Espectro de UV-Visible de 2C.

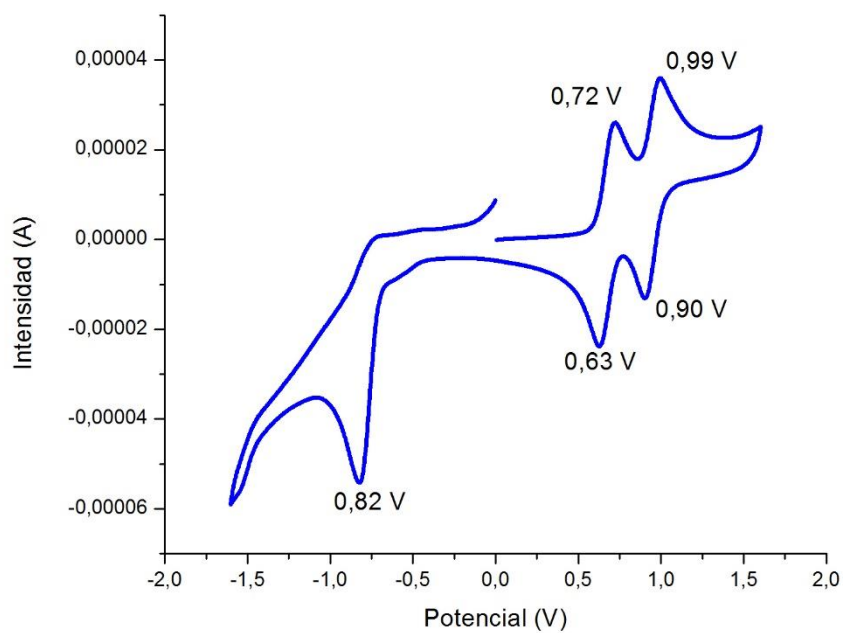


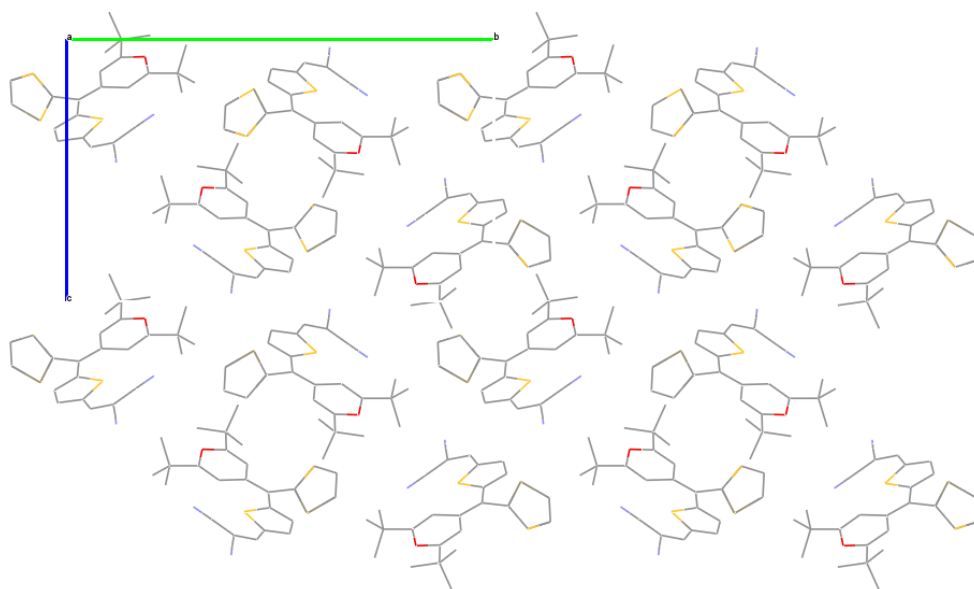
Figura 8.46. Voltamperograma de 2C.

**8.3. Rayos X**

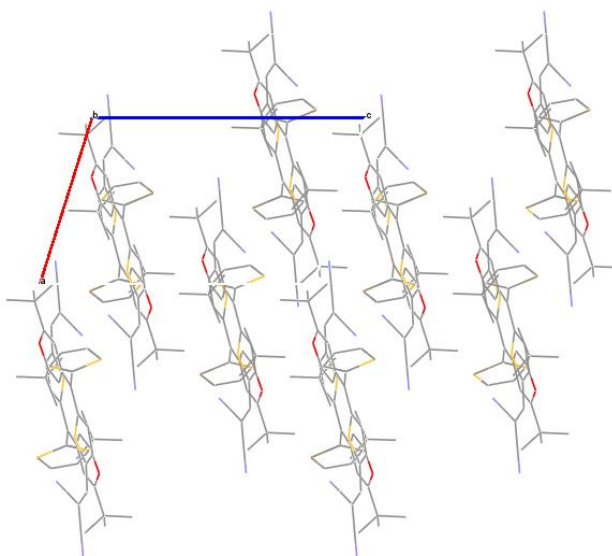
Por razones de espacio, se han omitido las tablas de datos de coordenadas atómicas, distancias y ángulos de enlace.

**Crystal data and structure refinement for 1A**

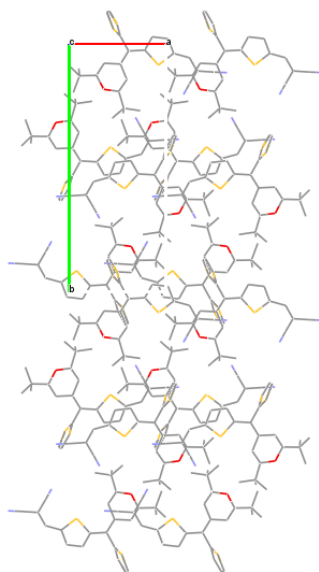
Empirical formula	C <sub>26</sub> H <sub>26</sub> N <sub>2</sub> O <sub>2</sub>
Formula weight	446.61
Temperature	150(1) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P 2 <sub>1</sub> /n
Unit cell dimensions	a = 8.6195(3) Å    alpha = 90 deg. b = 21.1663(6) Å    beta = 107.473(3) deg. c = 13.5680(4) Å    gamma = 90 deg.
Volume	2361.17(13) Å <sup>3</sup>
Z, Calculated density	4, 1.256 Mg/m <sup>3</sup>
Absorption coefficient	0.246 mm <sup>-1</sup>
F(000)	944
Crystal size	0.520 x 0.430 x 0.150 mm
Theta range for data collection	3.289 to 24.999 deg.
Limiting indices	-10 ≤ h ≤ 10, -25 ≤ k ≤ 25, -16 ≤ l ≤ 16
Reflections collected / unique	32987 / 4148 [R(int) = 0.0300]
Completeness to theta = 25.000	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.00000 and 0.94680
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	4148 / 0 / 289
Goodness-of-fit on F <sup>2</sup>	1.031
Final R indices [I > 2σ(I)]	R <sub>1</sub> = 0.0354, wR <sub>2</sub> = 0.0852
R indices (all data)	R <sub>1</sub> = 0.0418, wR <sub>2</sub> = 0.0893
Extinction coefficient	n/a
Largest diff. peak and hole	0.326 and -0.232 e.Å <sup>-3</sup>



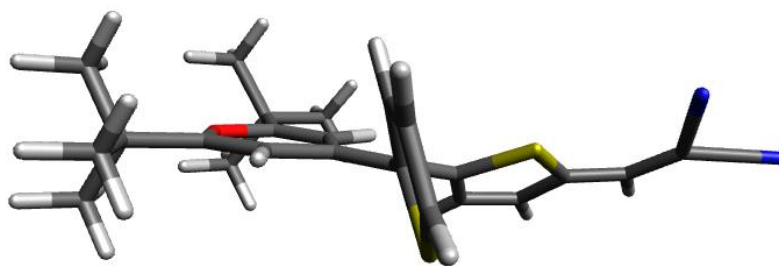
**Figura 8.47.** Empaquetamiento de **1A** a lo largo del eje a.



**Figura 8.48.** Empaquetamiento de **1A** a lo largo del eje b.

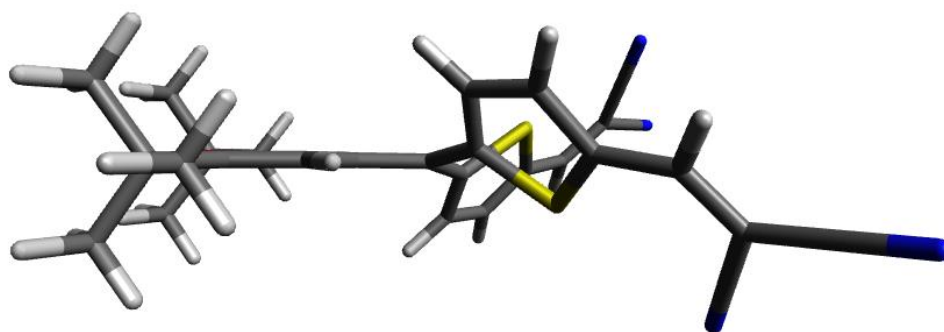


**Figura 8.49.** Empaquetamiento de **1A** a lo largo del eje c.



**Figura 8.50.** Estructura molecular calculada (método CPCM M062x/6-31G\*) del compuesto **1A** en DCM.

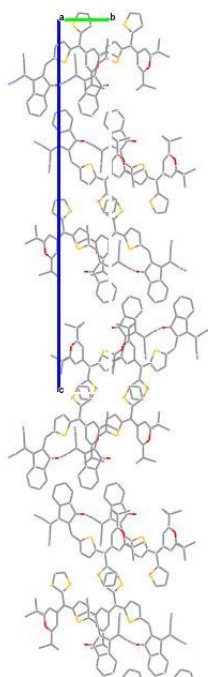




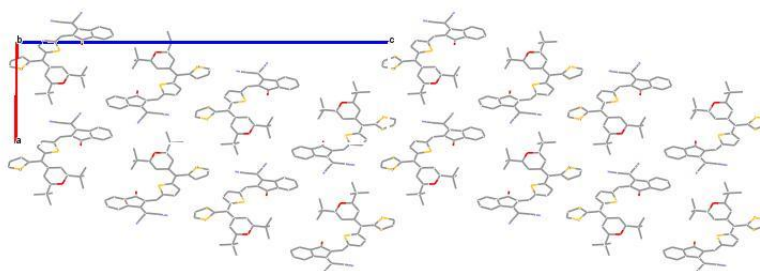
**Figura 8.51.** Estructura molecular calculada (método CPCM M062x/6-31G\*) del compuesto **2A** en DCM

Crystal data and structure refinement for **1B**

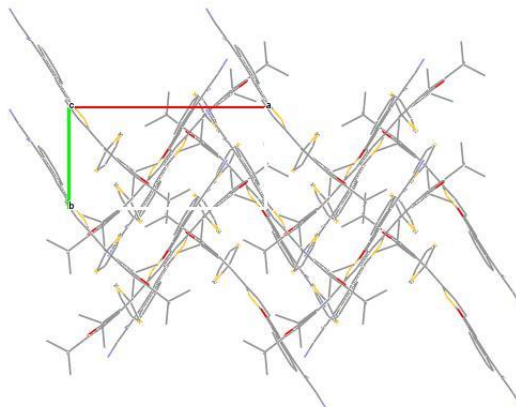
Empirical formula	C <sub>35</sub> H <sub>30</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>
Formula weight	574.73
Temperature	150(1) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P 2 <sub>1</sub> /n
Unit cell dimensions	a = 11.728(2) Å    alpha = 90 deg. b = 6.0061(12) Å    beta = 90.647(19) deg. c = 43.409(7) Å    gamma = 90 deg.
Volume	3057.5(9) Å <sup>3</sup>
Z, Calculated density	4, 1.249 Mg/m <sup>3</sup>
Absorption coefficient	0.208 mm <sup>-1</sup>
F(000)	1208
Crystal size	0.510 x 0.050 x 0.030 mm
Theta range for data collection	3.424 to 24.998 deg.
Limiting indices	-13<=h<=13, -6<=k<=7, -51<=l<=50
Reflections collected / unique	17317 / 5113 [R(int) = 0.3068]
Completeness to theta = 25.000	95.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.00000 and 0.60273
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	5113 / 51 / 379
Goodness-of-fit on F <sup>2</sup>	0.917
Final R indices [I>2sigma(I)]	R1 = 0.1060, wR2 = 0.1244
R indices (all data)	R1 = 0.3623, wR2 = 0.2063
Extinction coefficient	n/a
Largest diff. peak and hole	0.326 and -0.320 e.Å <sup>-3</sup>



**Figura 8.52.** Empaquetamiento de **1B** a lo largo del eje a.



**Figura 8.53.** Empaquetamiento de **1B** a lo largo del eje b.



**Figura 8.54.** Empaquetamiento de **1B** a lo largo del eje c.