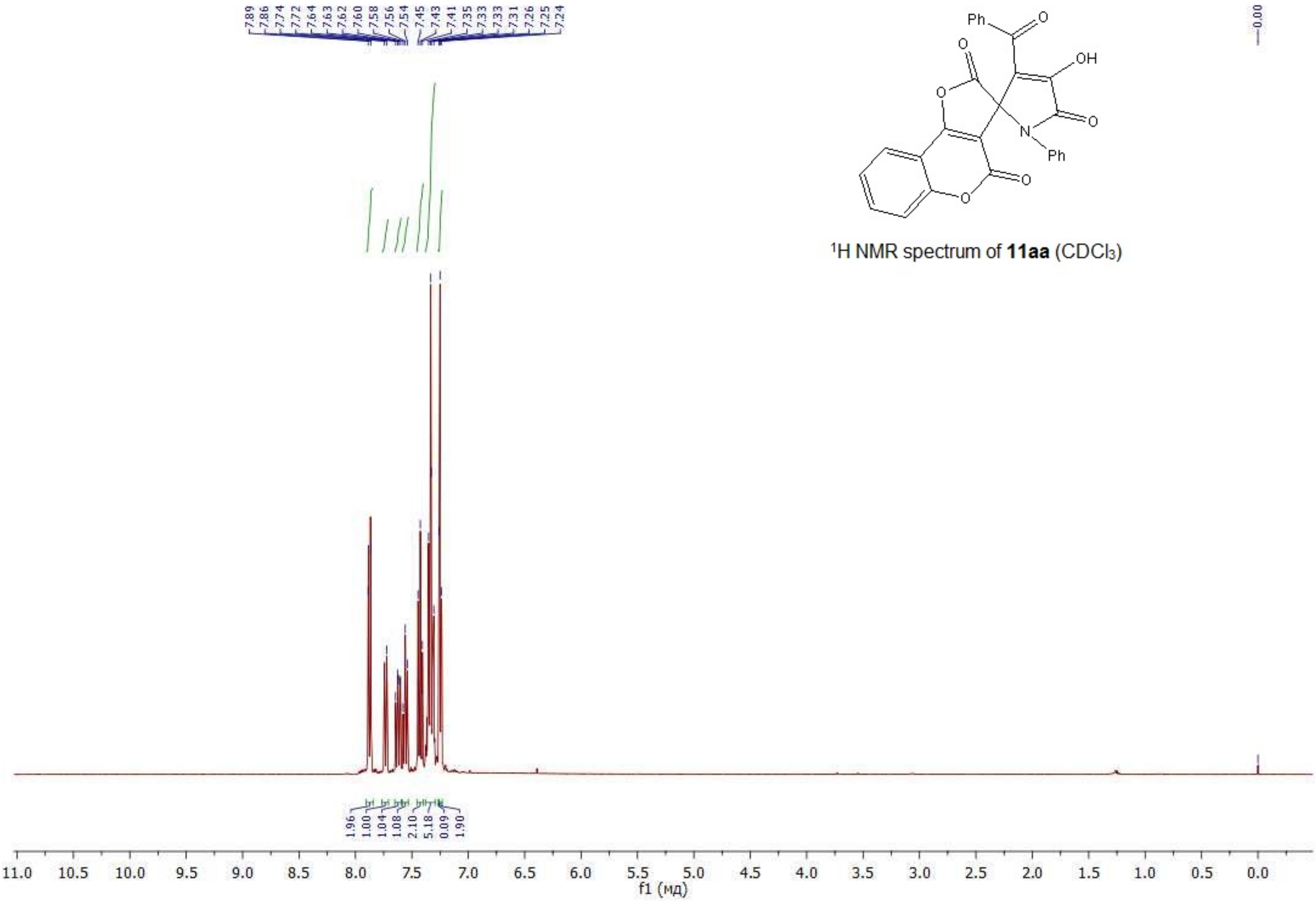
Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

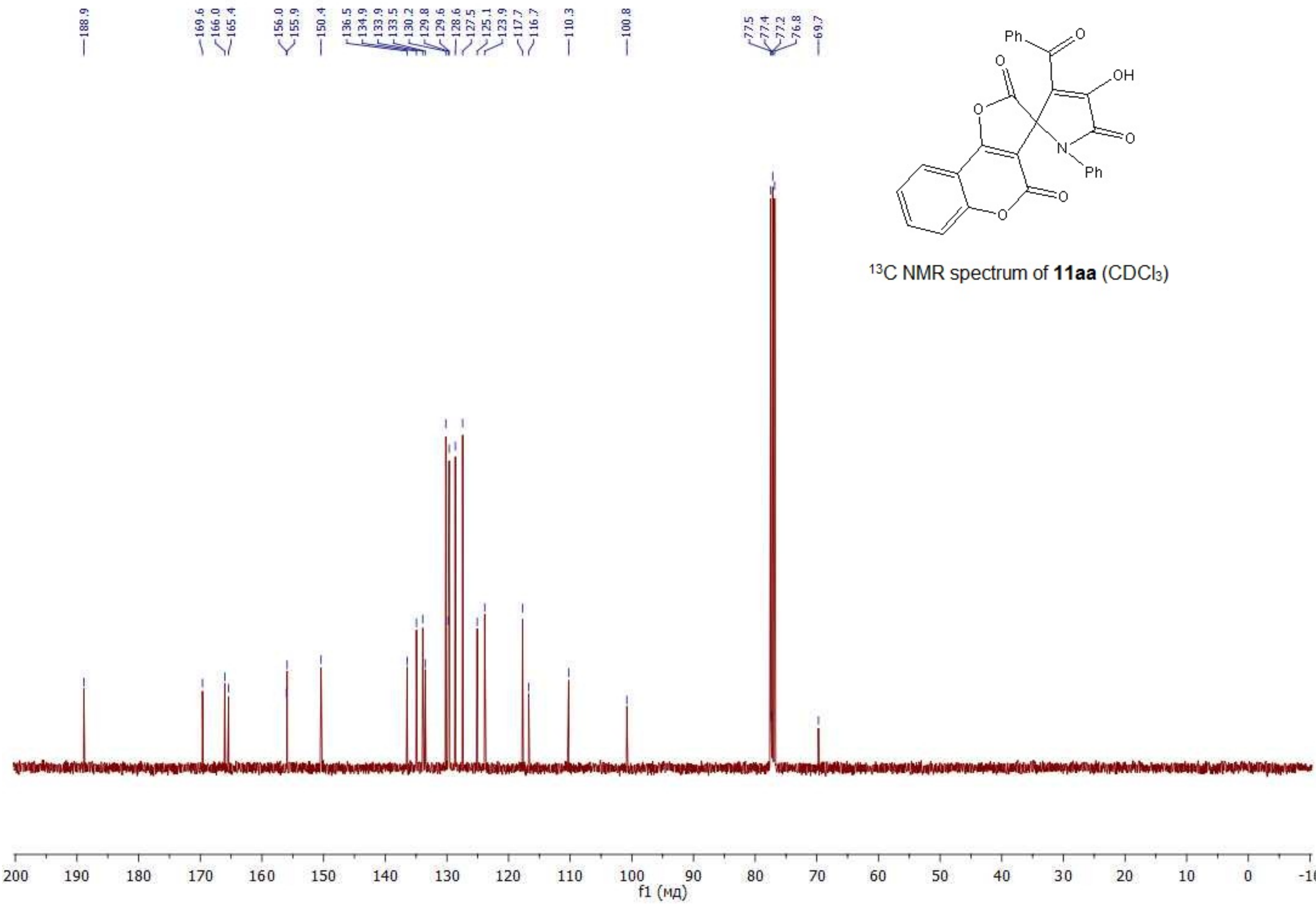
Spiro-condensation of 5-Methoxycarbonyl-1*H*-Pyrrole-2,3-diones with Cyclic Enoles to Form Spiro Substituted Furo[3,2-c]- coumarins and Quinolines

Alexey Yu. Dubovtsev,[a] Pavel S. Silaichev,[a] Mikhail A. Nazarov,[a] Maksim V. Dmitriev,[a] Andrey N. Maslivets,*[a] and Michael Rubin*[b,c]

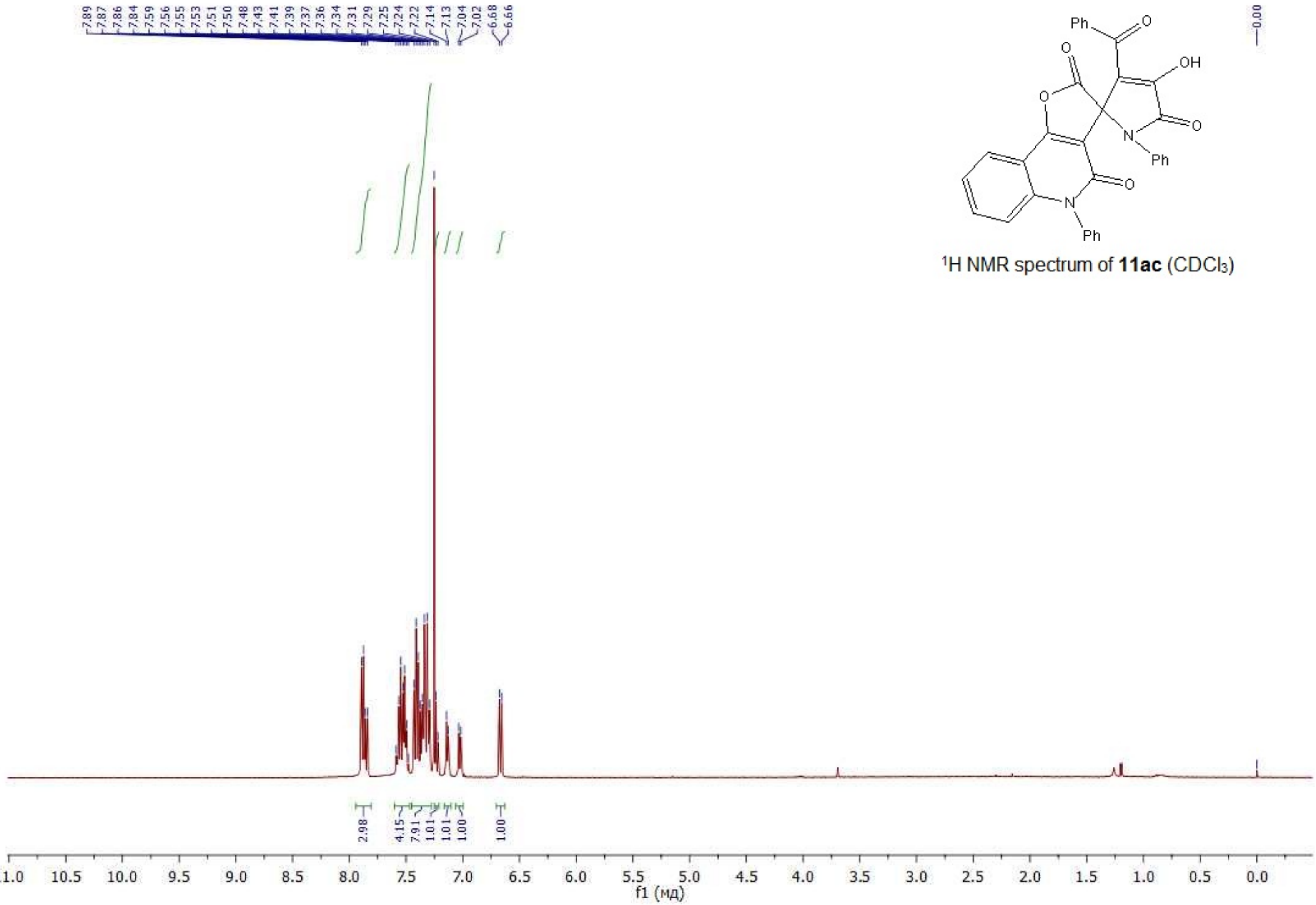
a. Department of Chemistry, Perm State University, ul. Bukireva 15, Perm 614990, Russia, E-mail: koh2@psu.ru
b. Department of Chemistry, North Caucasus Federal University, 1a Pushkin St., Stavropol 355009, Russian Federation
c. Department of Chemistry, University of Kansas, 1251 Wescoe Hall Dr., Lawrence, KS 66045-7582, USA. E-mail: mrubin@ku.edu; Fax: +1 (785) 864-5396; Tel: +1 (785) 864-5071



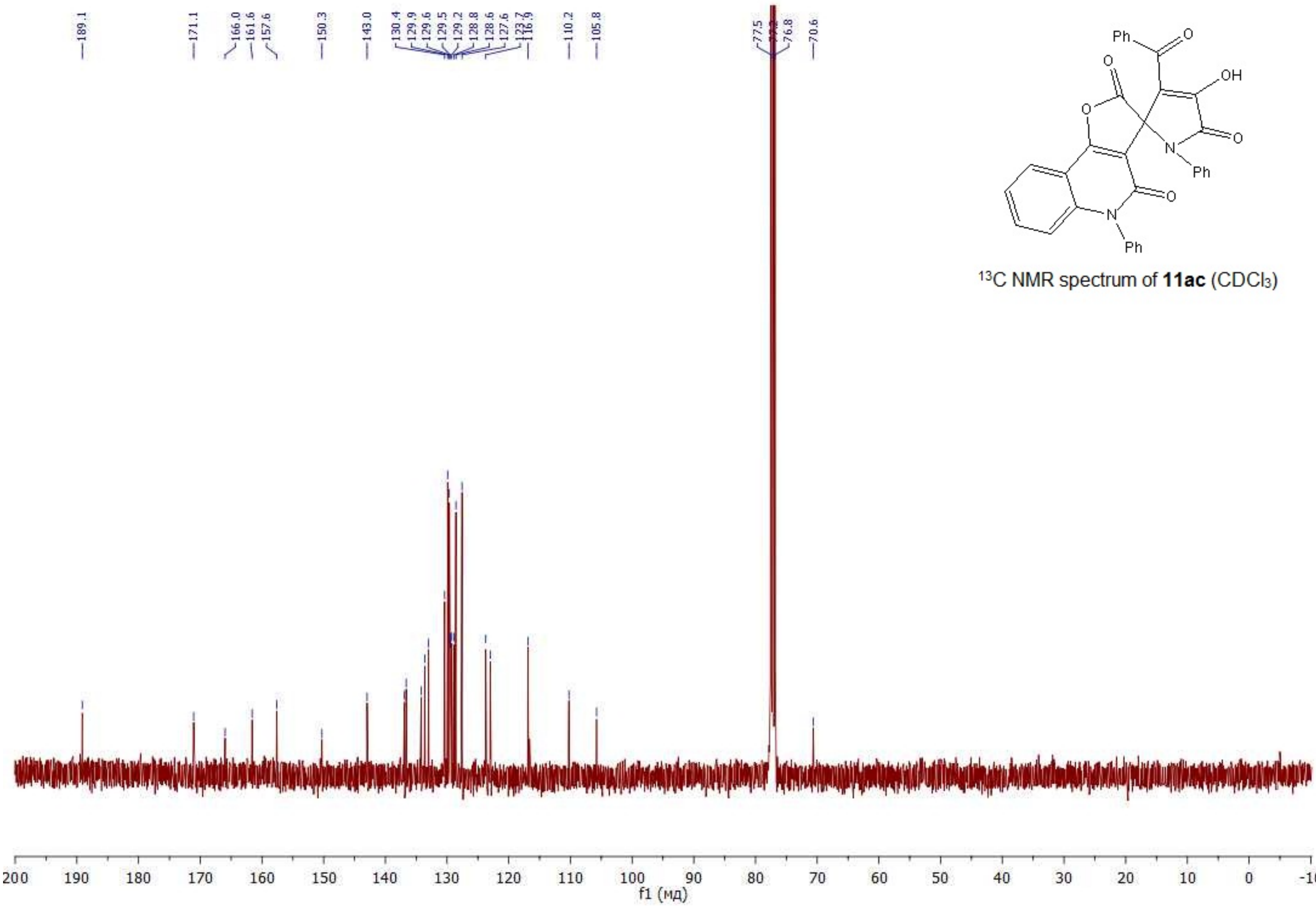
[1]H NMR spectrum of **11aa** ($CDCl_3$)



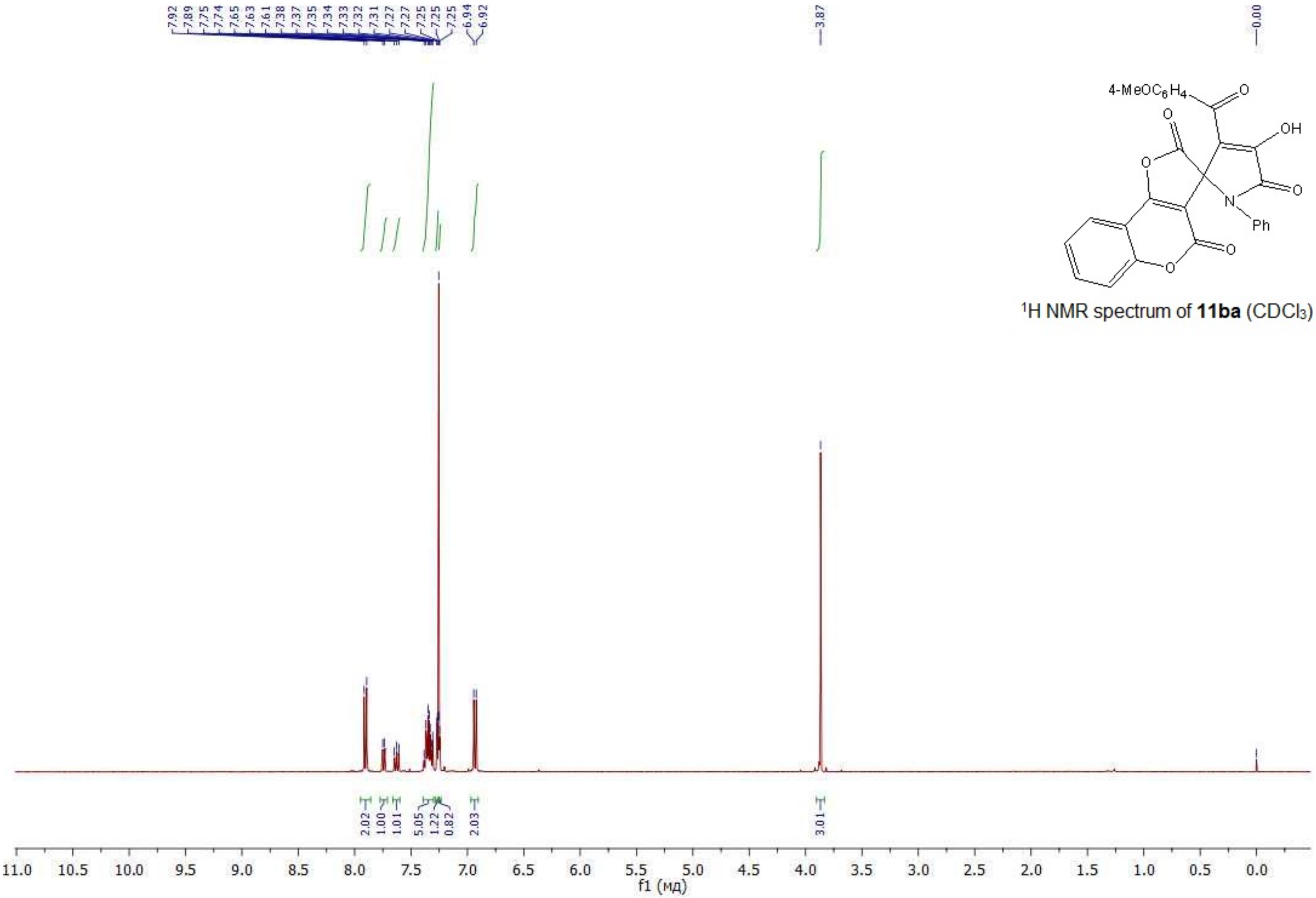
^{13}C NMR spectrum of **11aa** ($CDCl_3$)



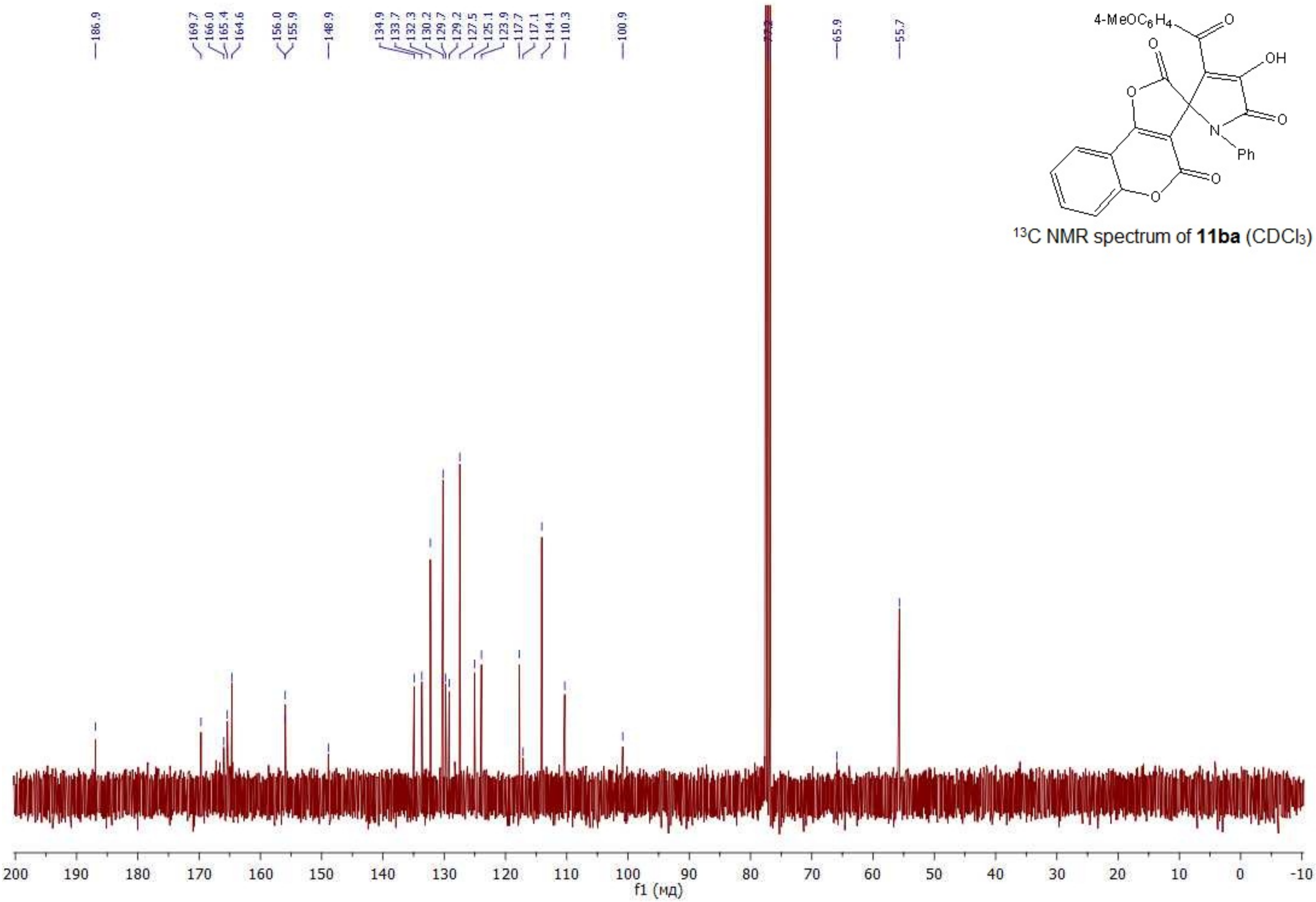
[1]H NMR spectrum of **11ac** ($CDCl_3$)



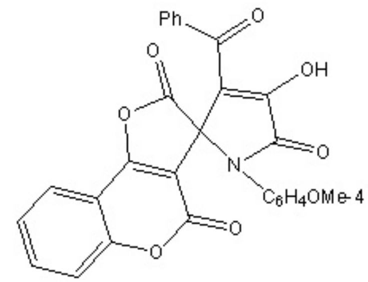
^{13}C NMR spectrum of **11ac** ($CDCl_3$)



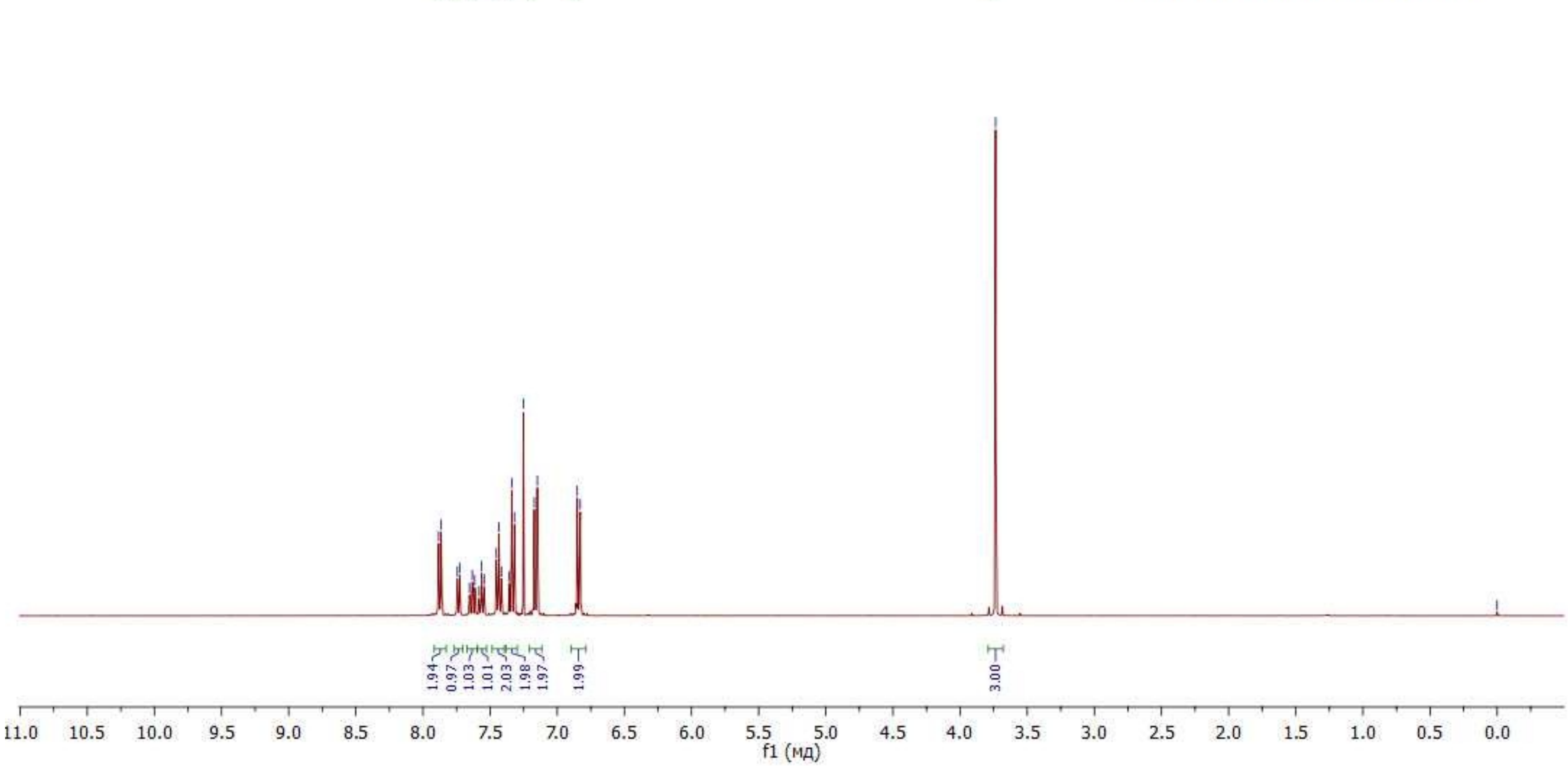
^{1}H NMR spectrum of **11ba** ($CDCl_3$)

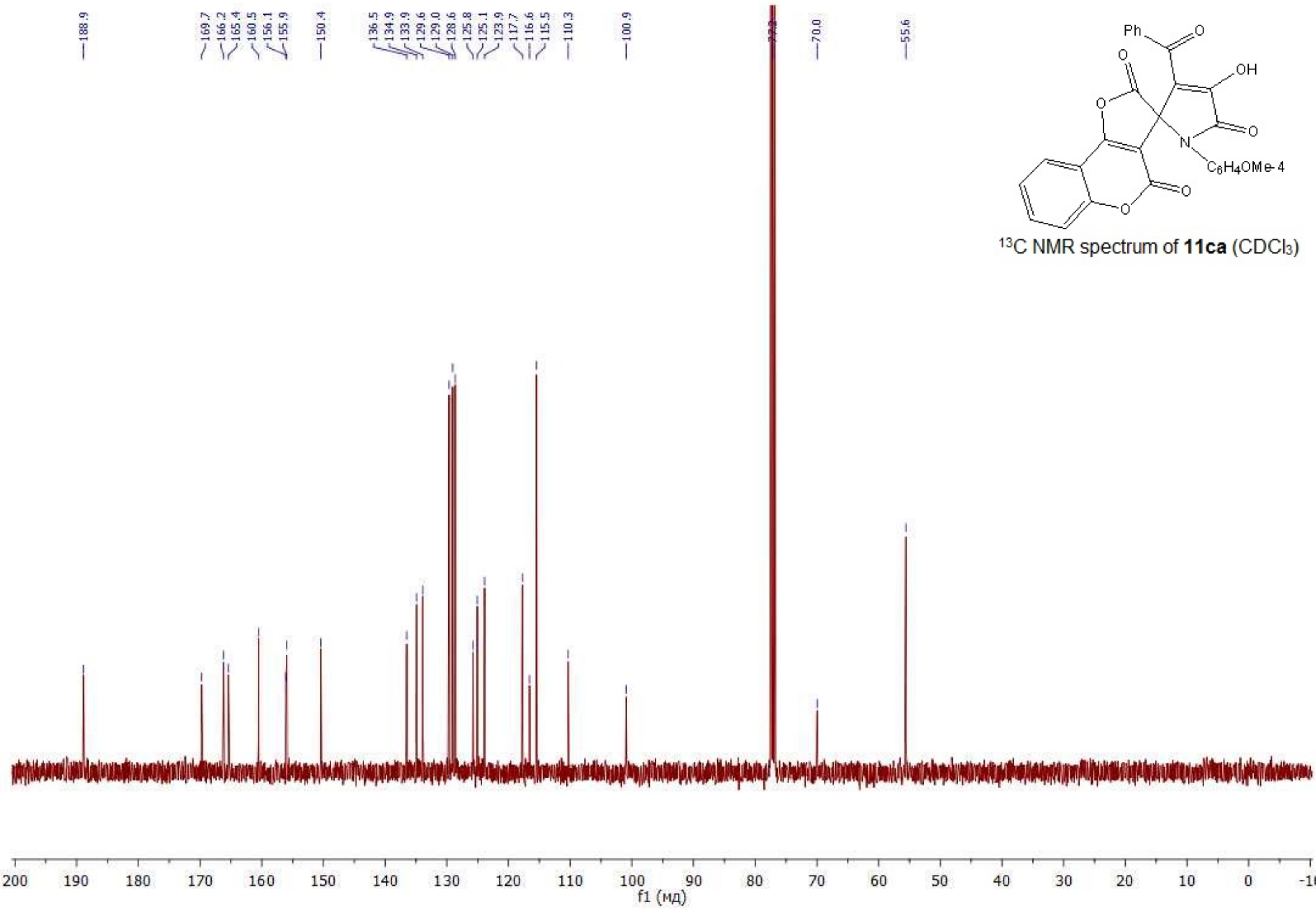


^{13}C NMR spectrum of **11ba** ($CDCl_3$)

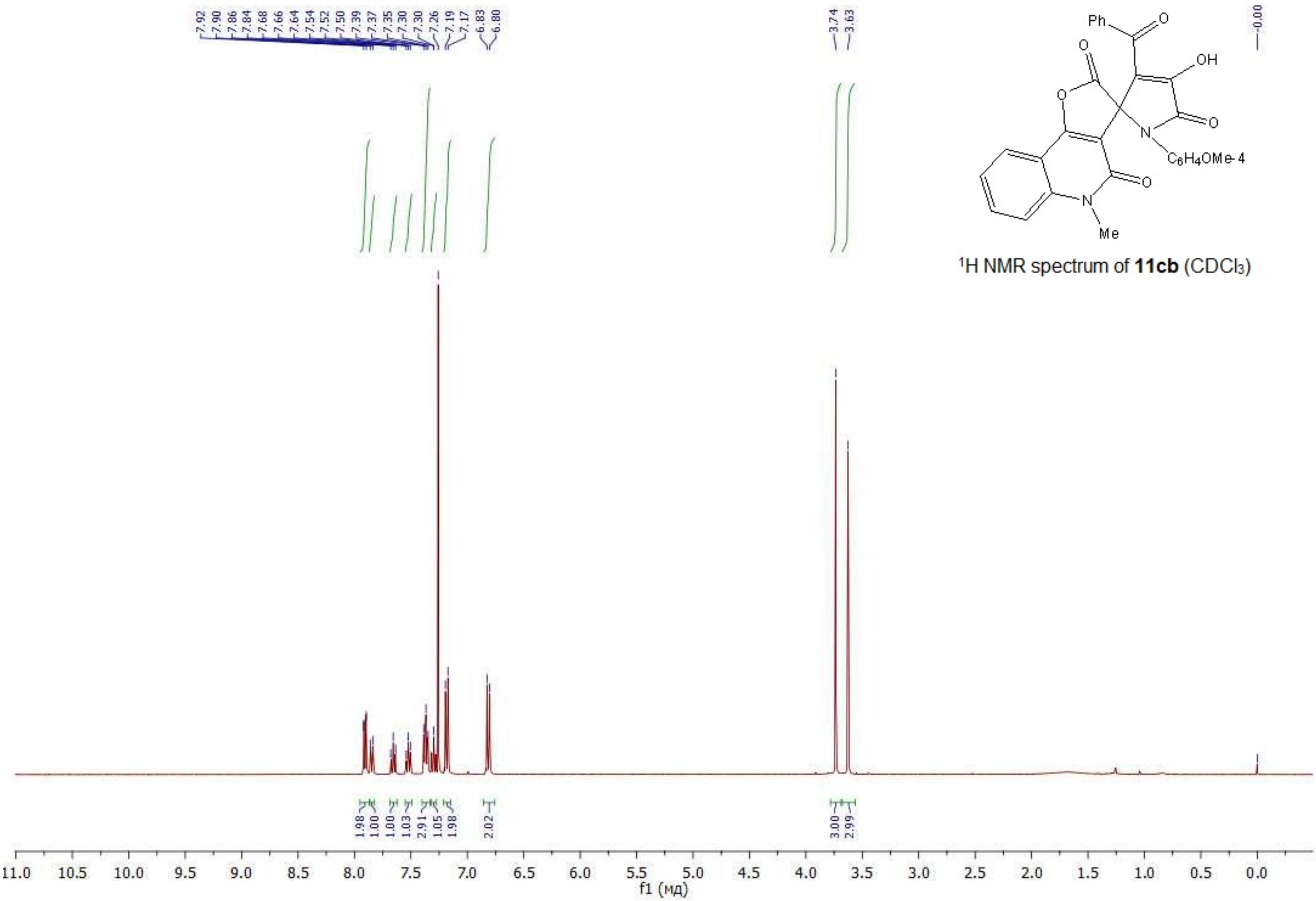


^{1}H NMR spectrum of **11ca** ($CDCl_3$)

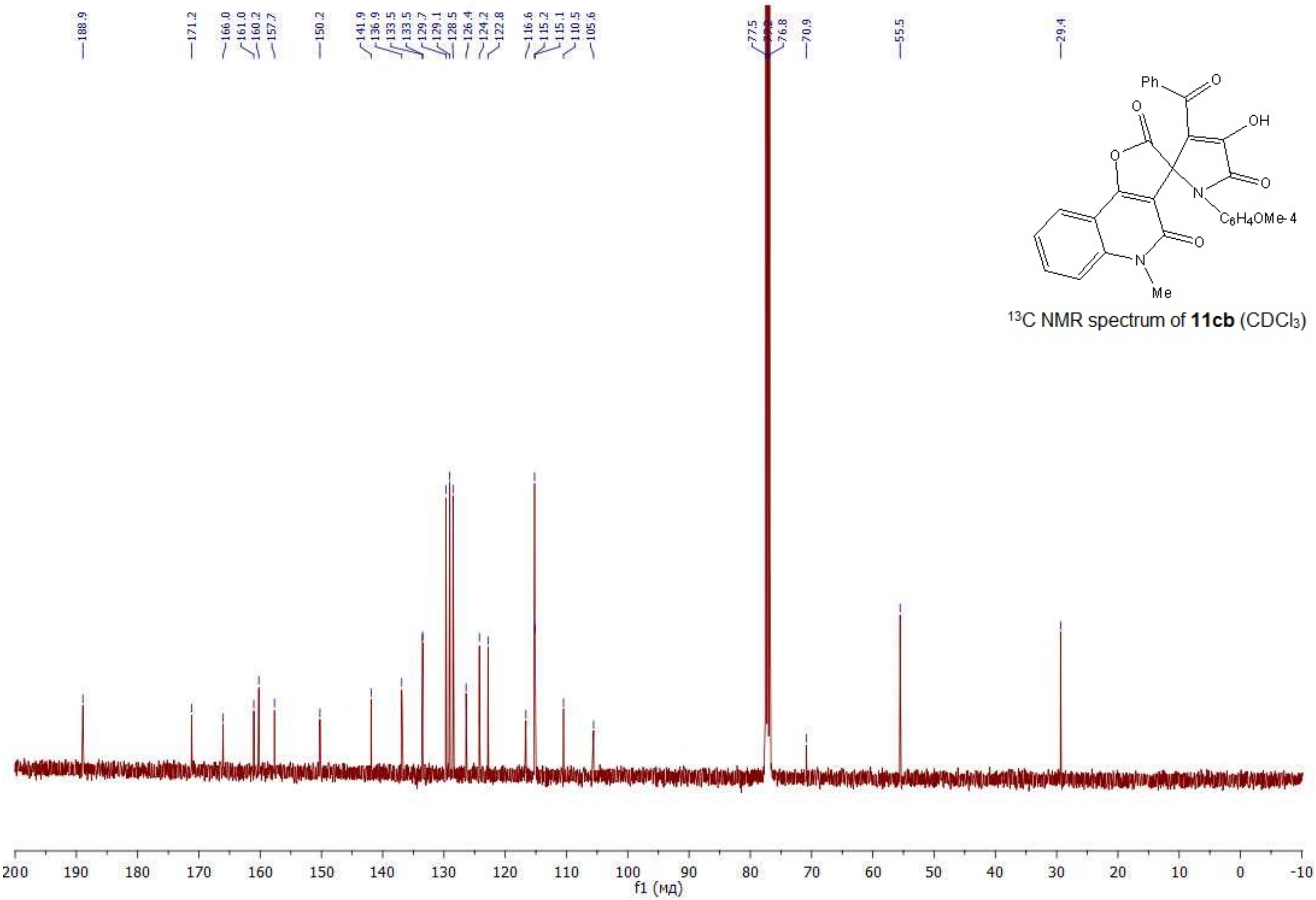




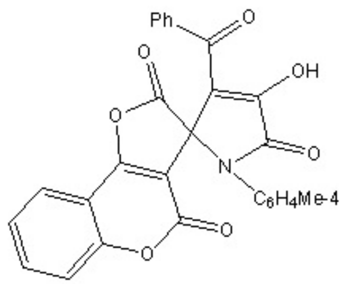
^{13}C NMR spectrum of **11ca** ($CDCl_3$)



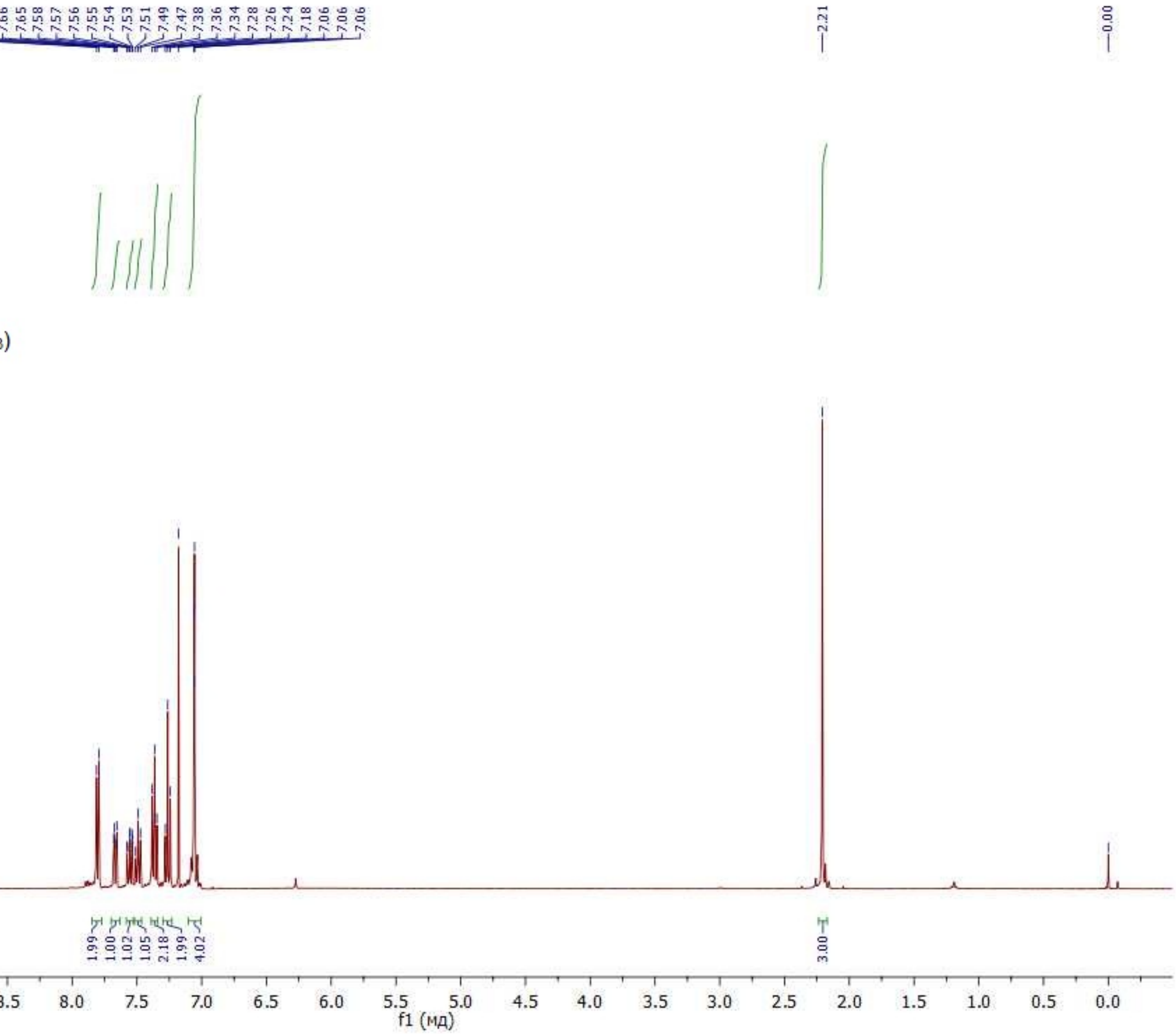
[1]H NMR spectrum of **11cb** ($CDCl_3$)

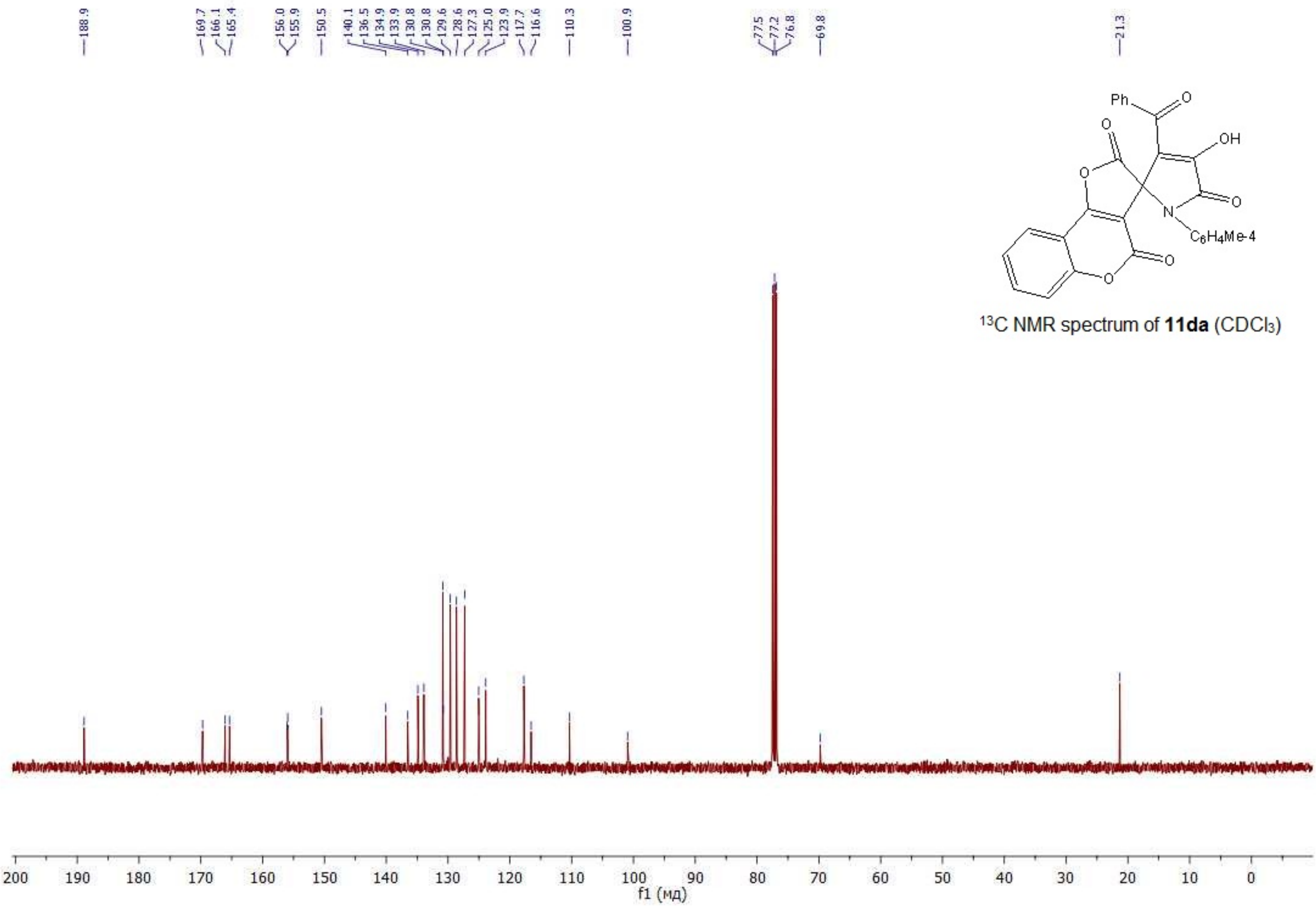


^{13}C NMR spectrum of **11cb** ($CDCl_3$)

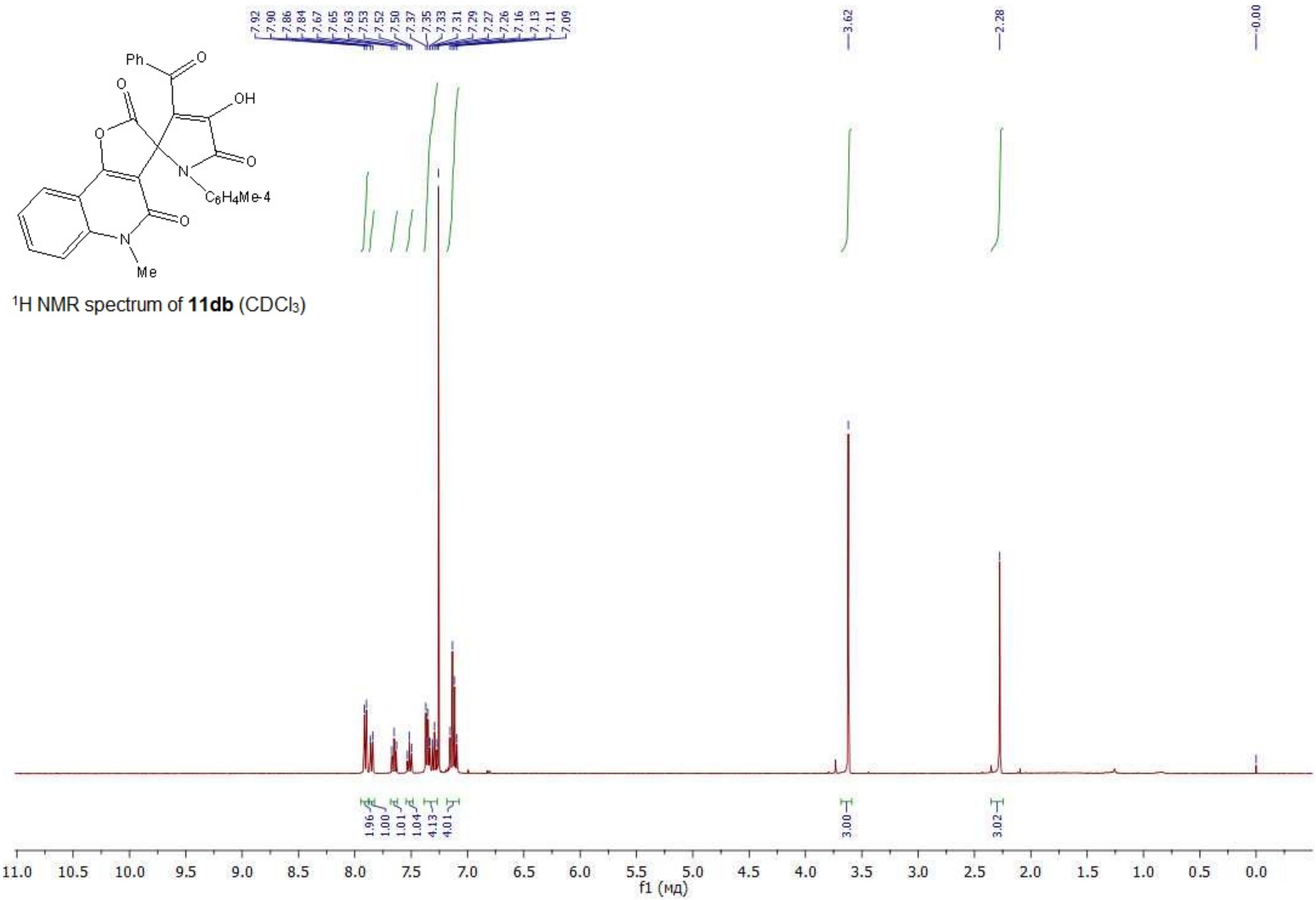


^{1}H NMR spectrum of **11da** ($CDCl_3$)

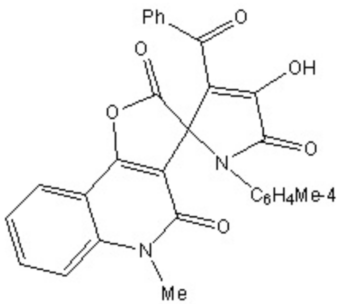




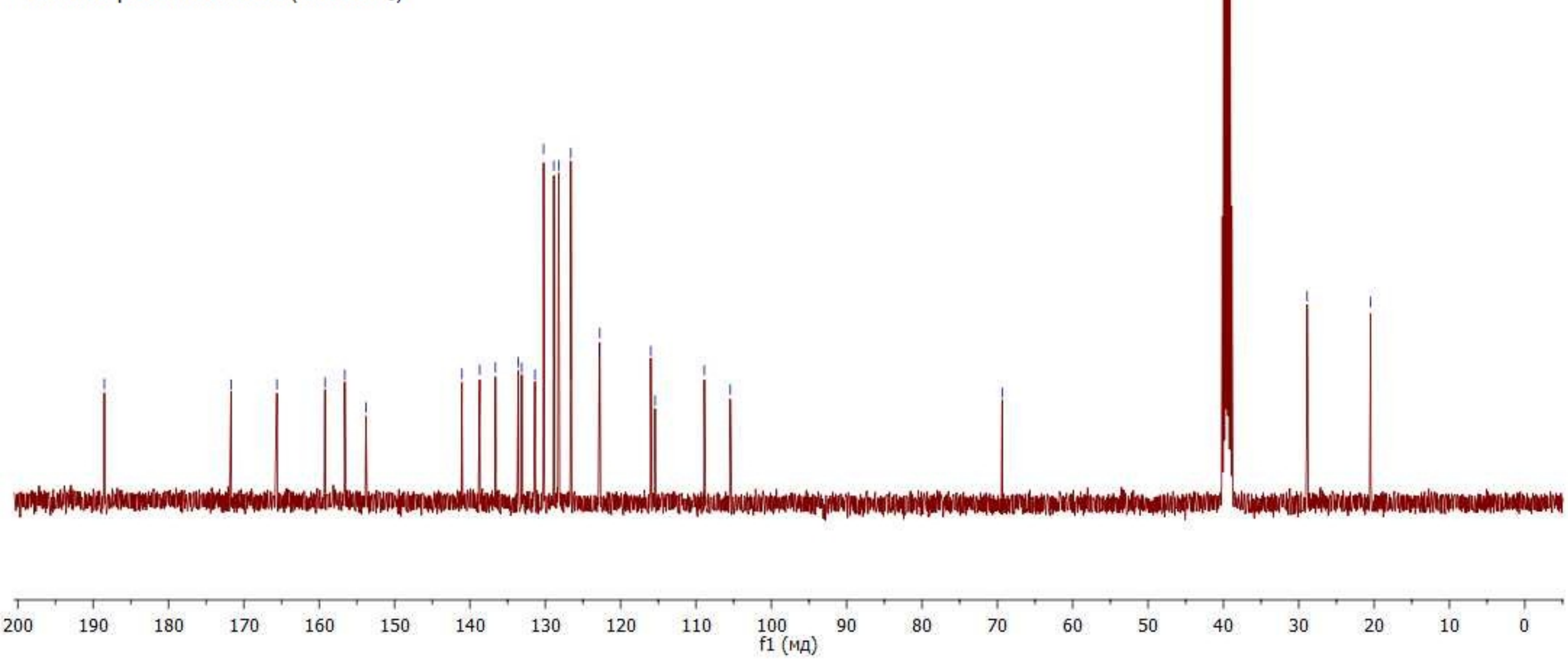
^{13}C NMR spectrum of **11da** ($CDCl_3$)

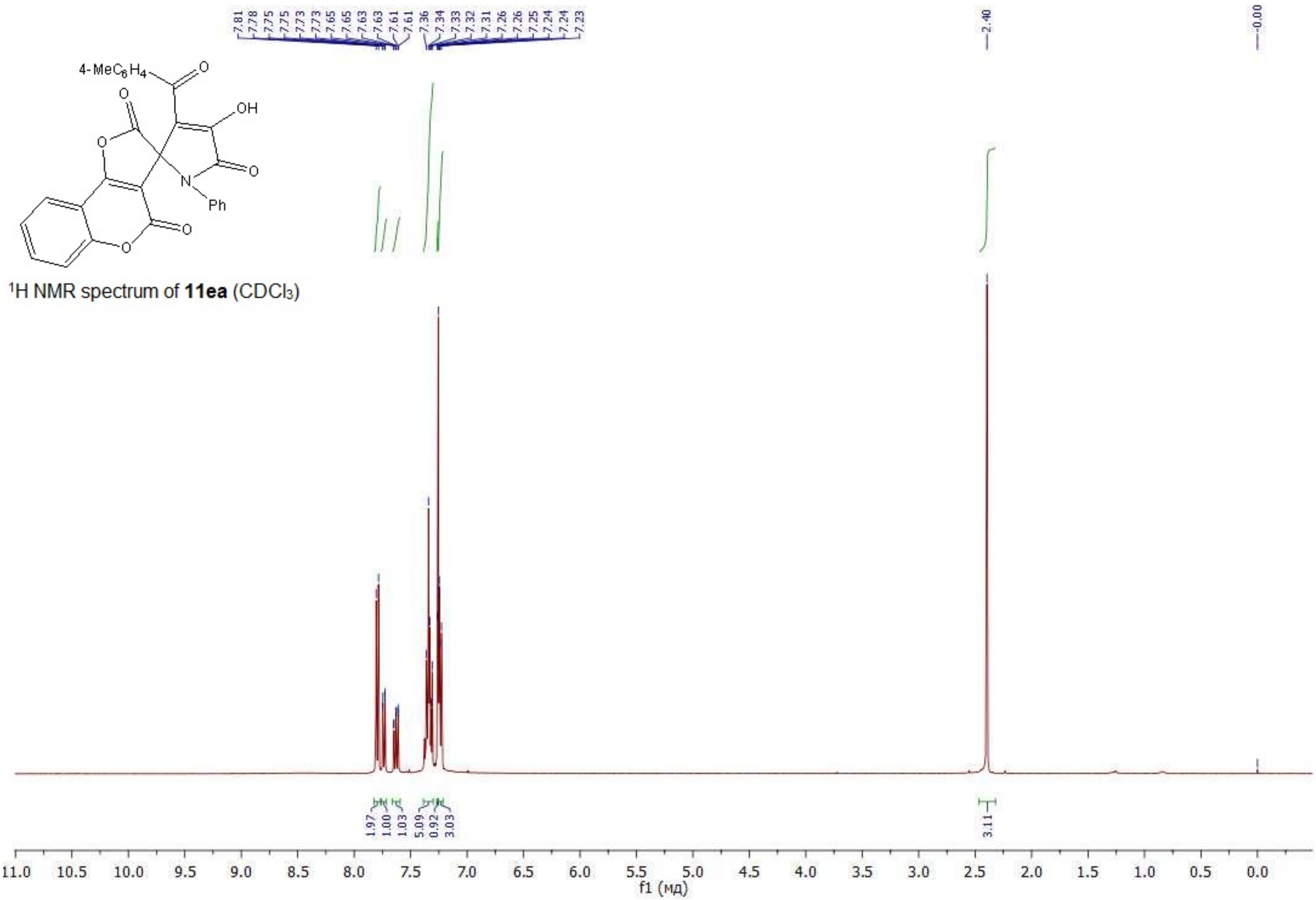


[1]H NMR spectrum of **11db** ($CDCl_3$)

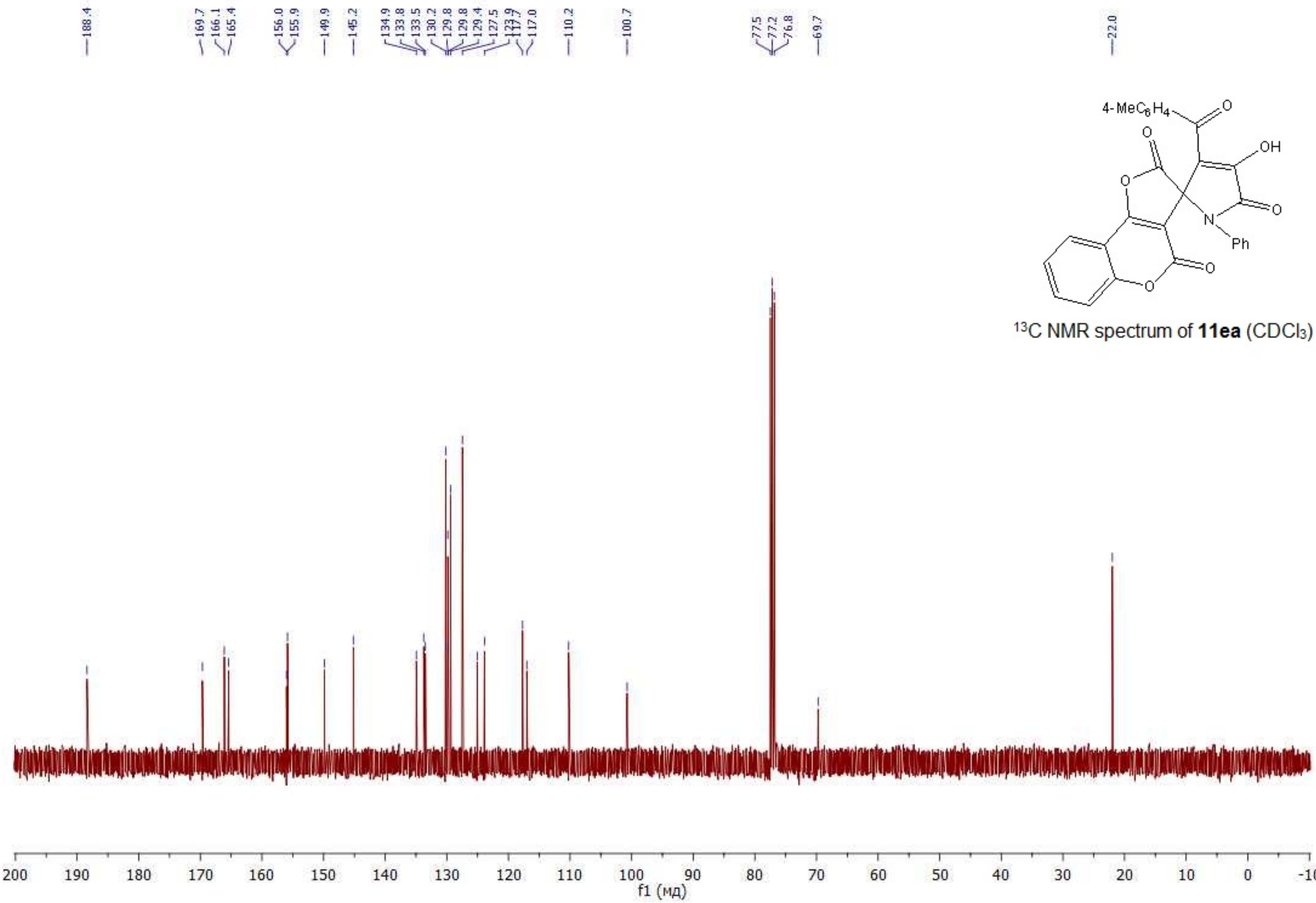


^{13}C NMR spectrum of **11db** (DMSO-d_6)

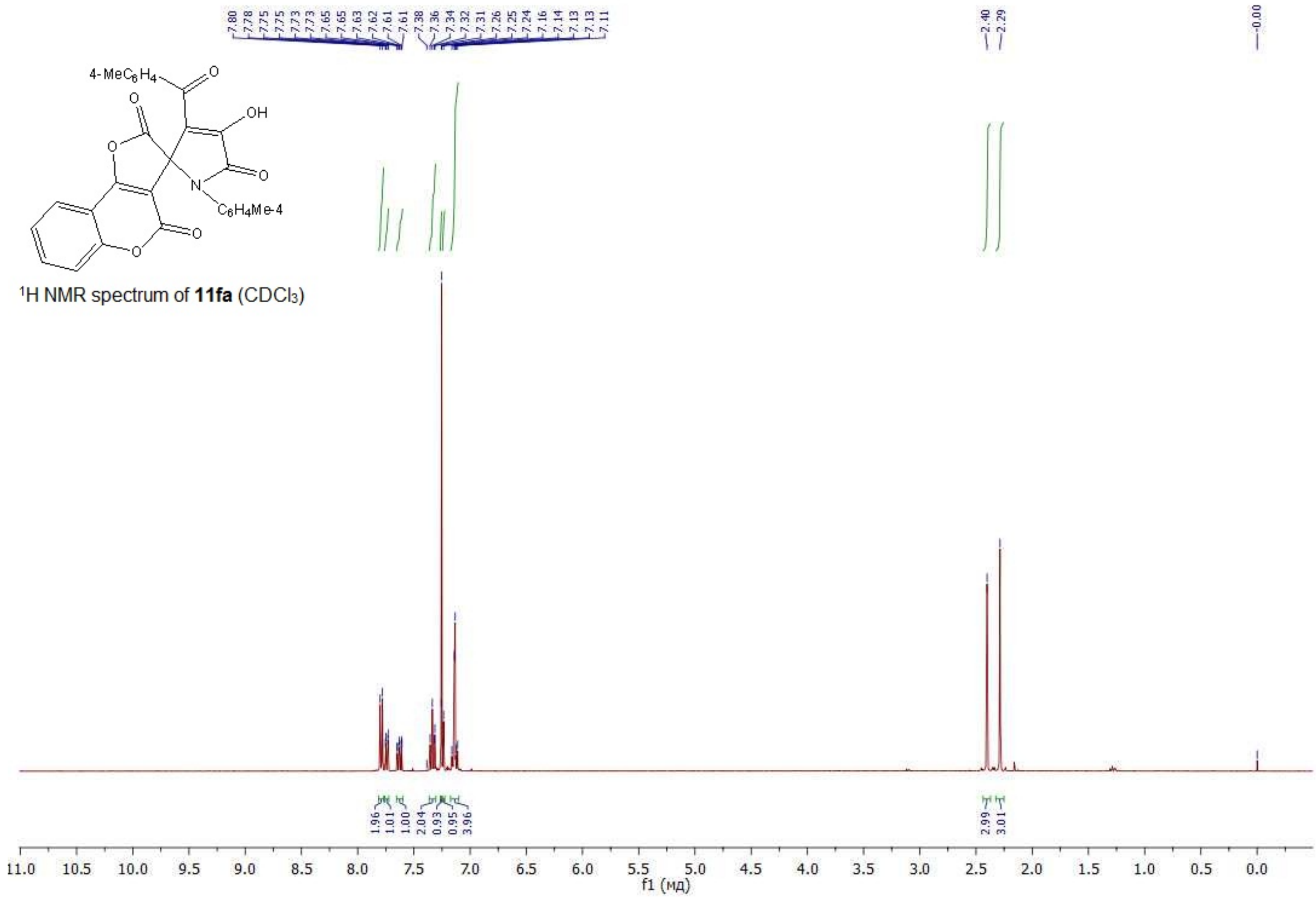




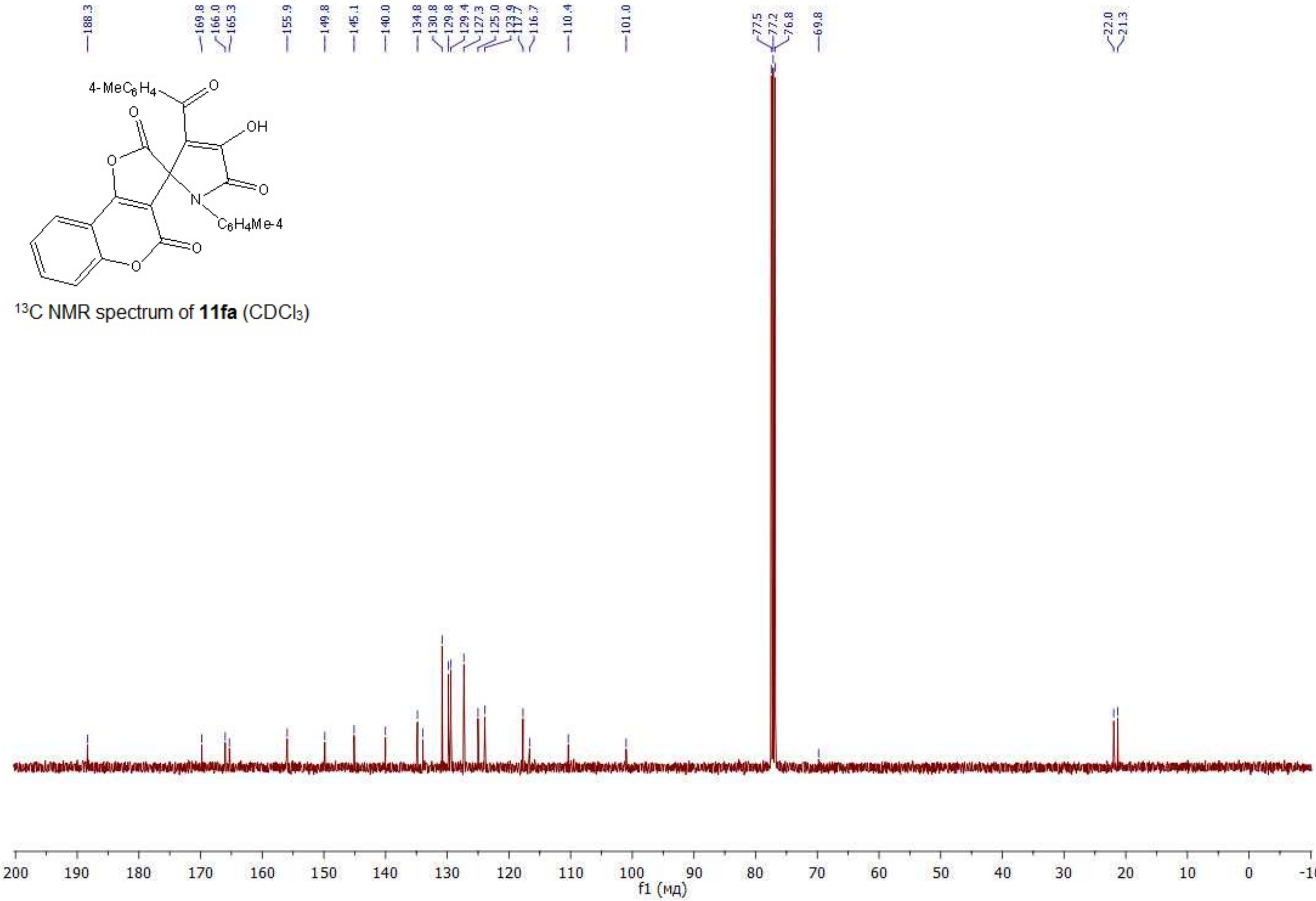
[1]H NMR spectrum of **11ea** ($CDCl_3$)



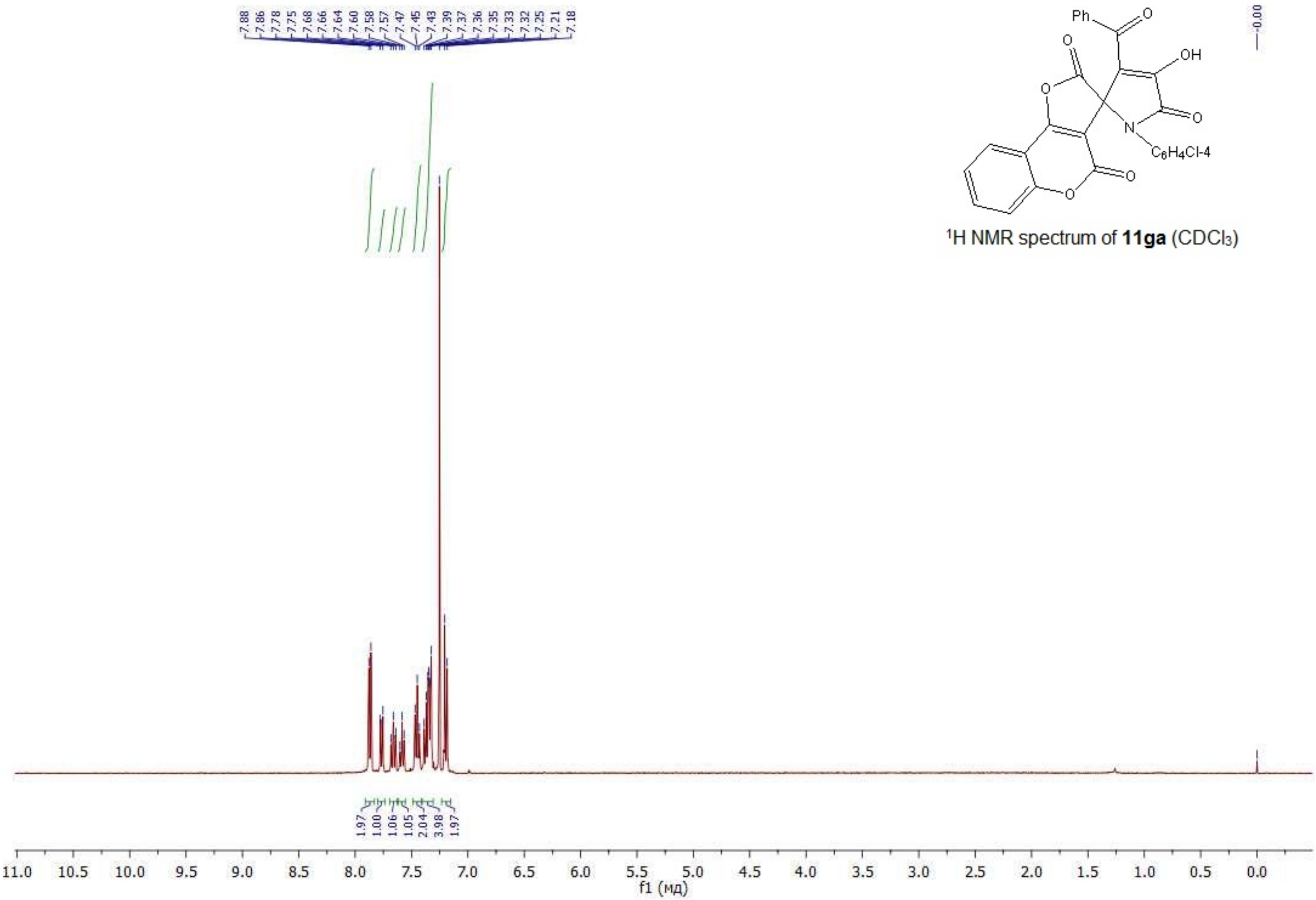
^{13}C NMR spectrum of **11ea** ($CDCl_3$)



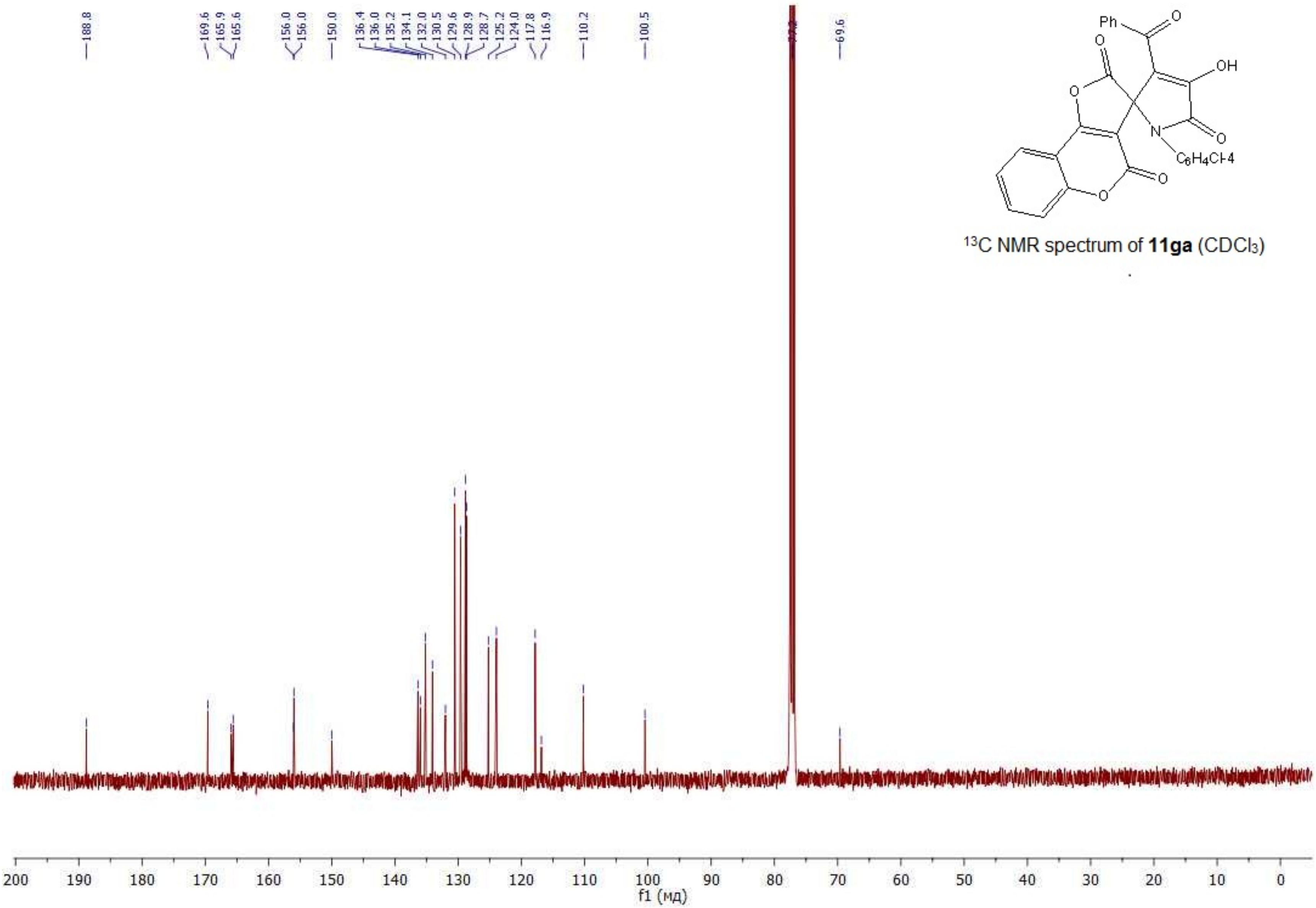
^{1}H NMR spectrum of **11fa** ($CDCl_3$)



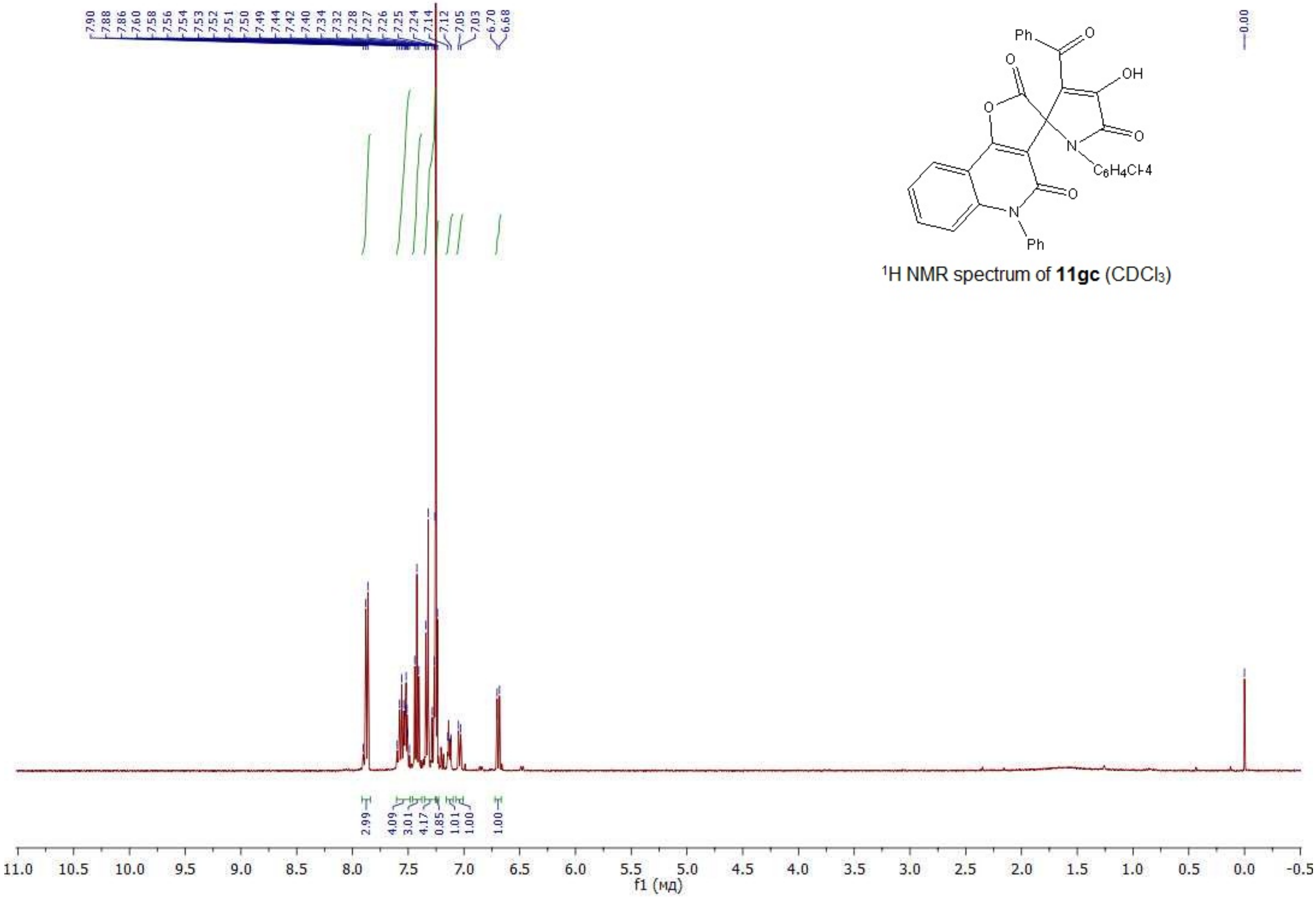
^{13}C NMR spectrum of **11fa** ($CDCl_3$)



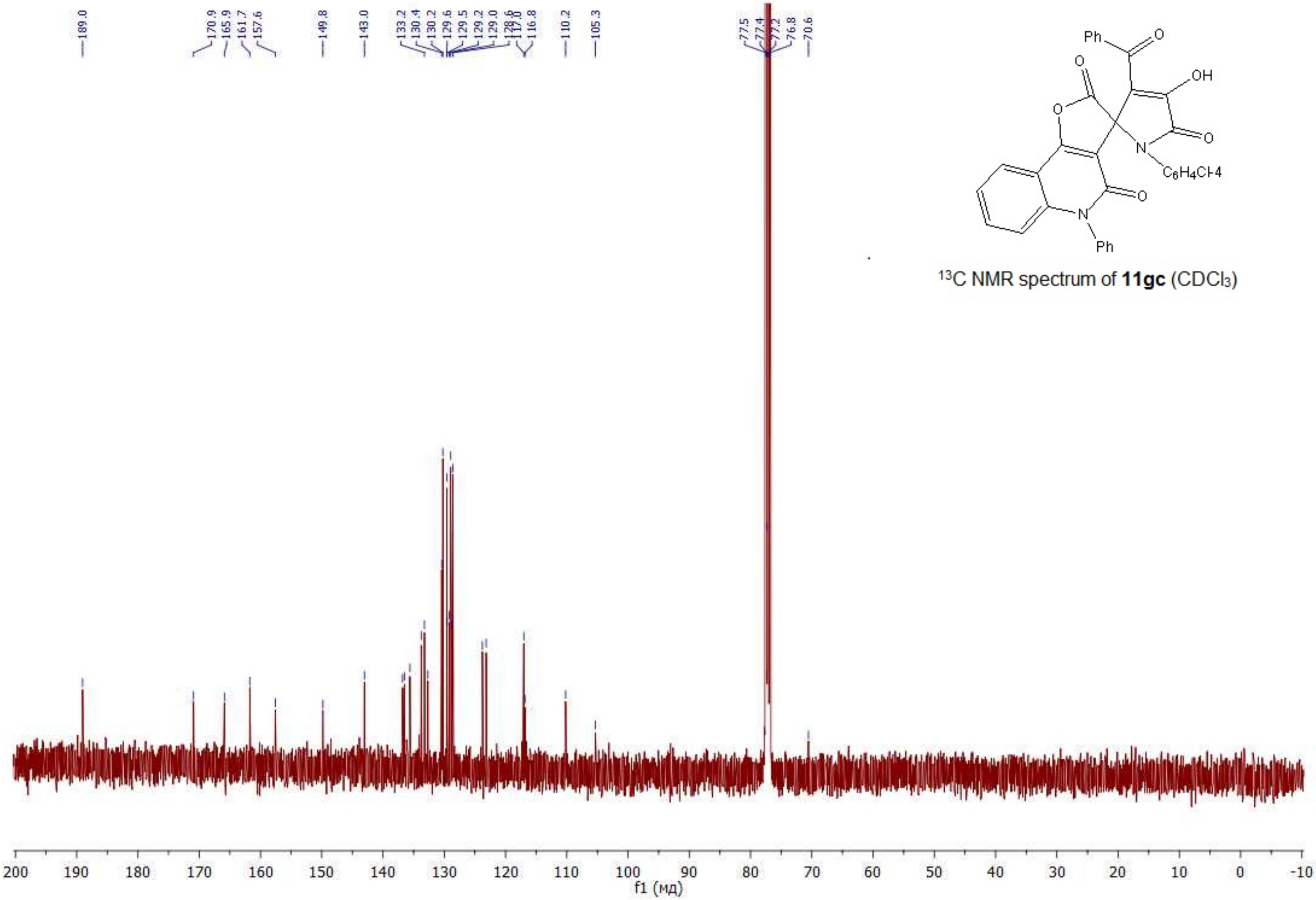
¹H NMR spectrum of **11ga** ($CDCl_3$)



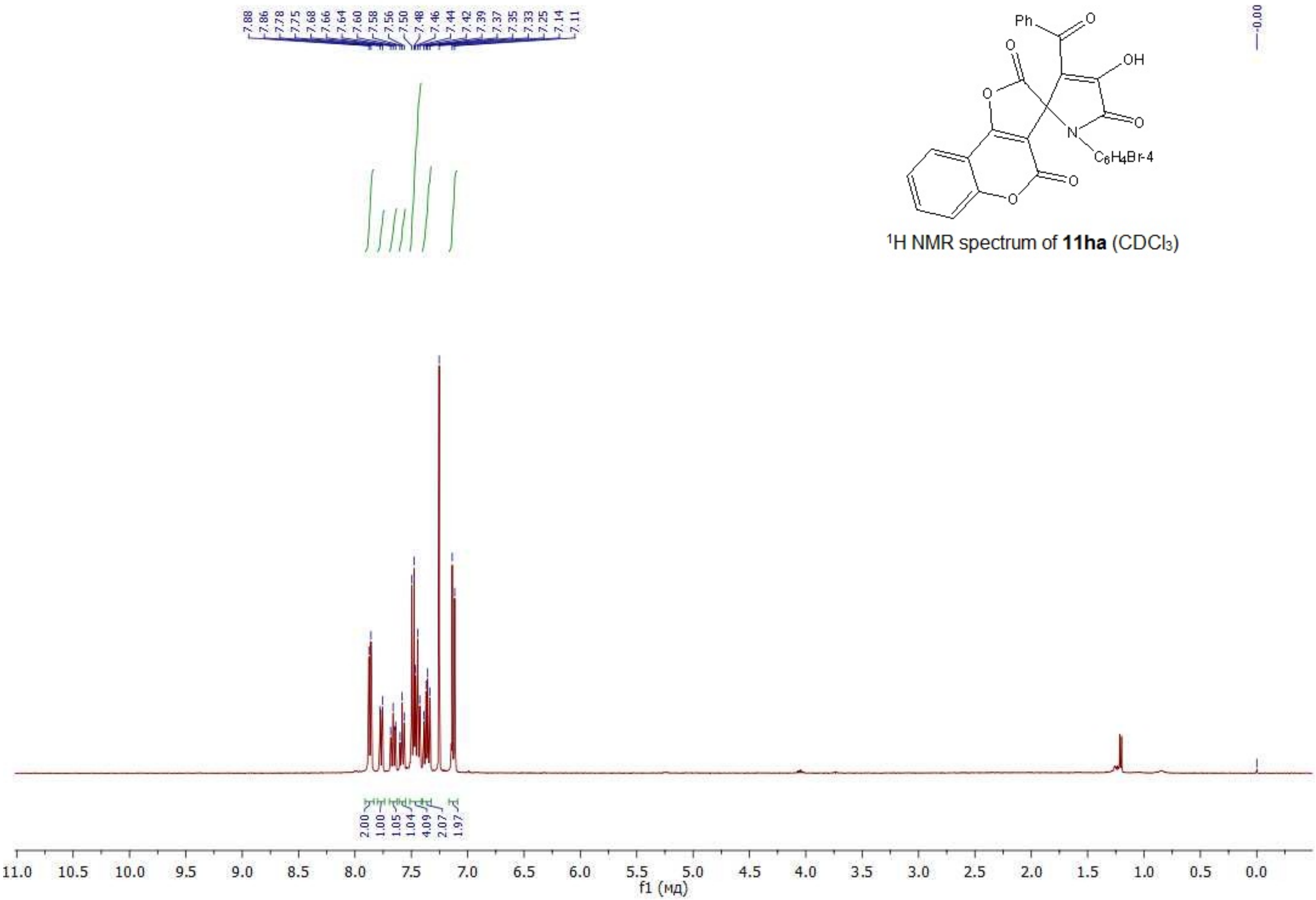
^{13}C NMR spectrum of **11ga** ($CDCl_3$)



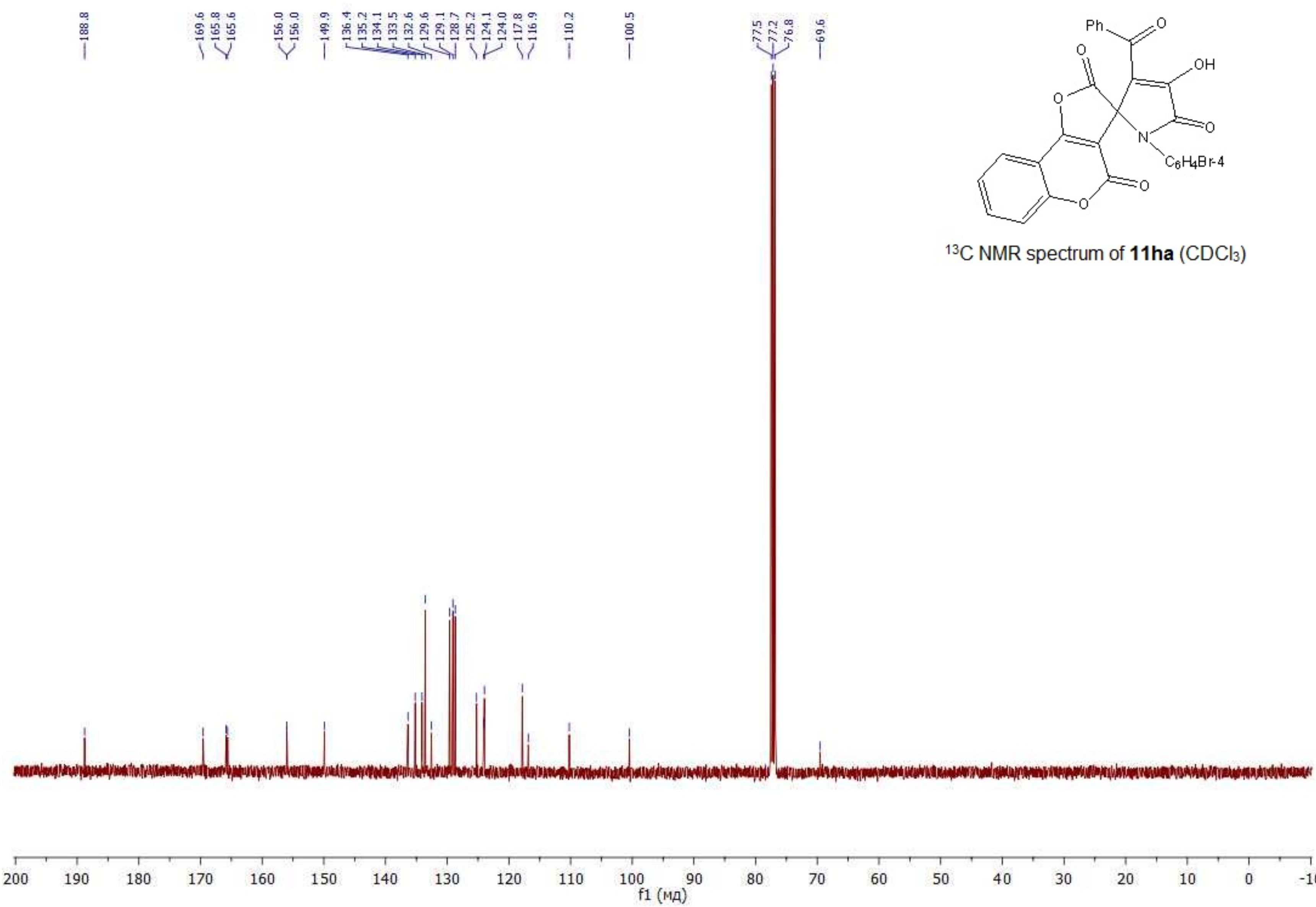
[1]H NMR spectrum of **11gc** ($CDCl_3$)



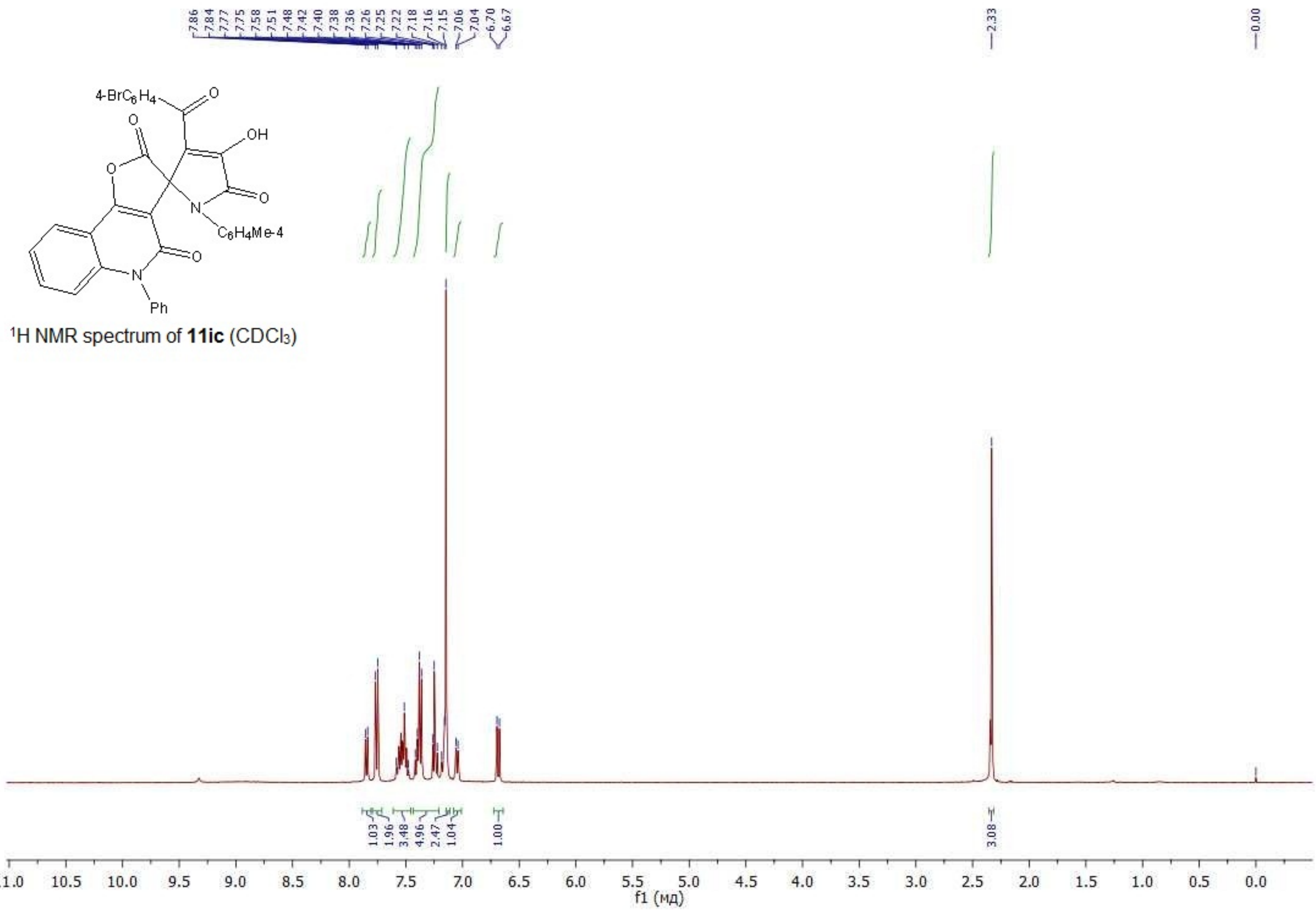
^{13}C NMR spectrum of **11gc** ($CDCl_3$)



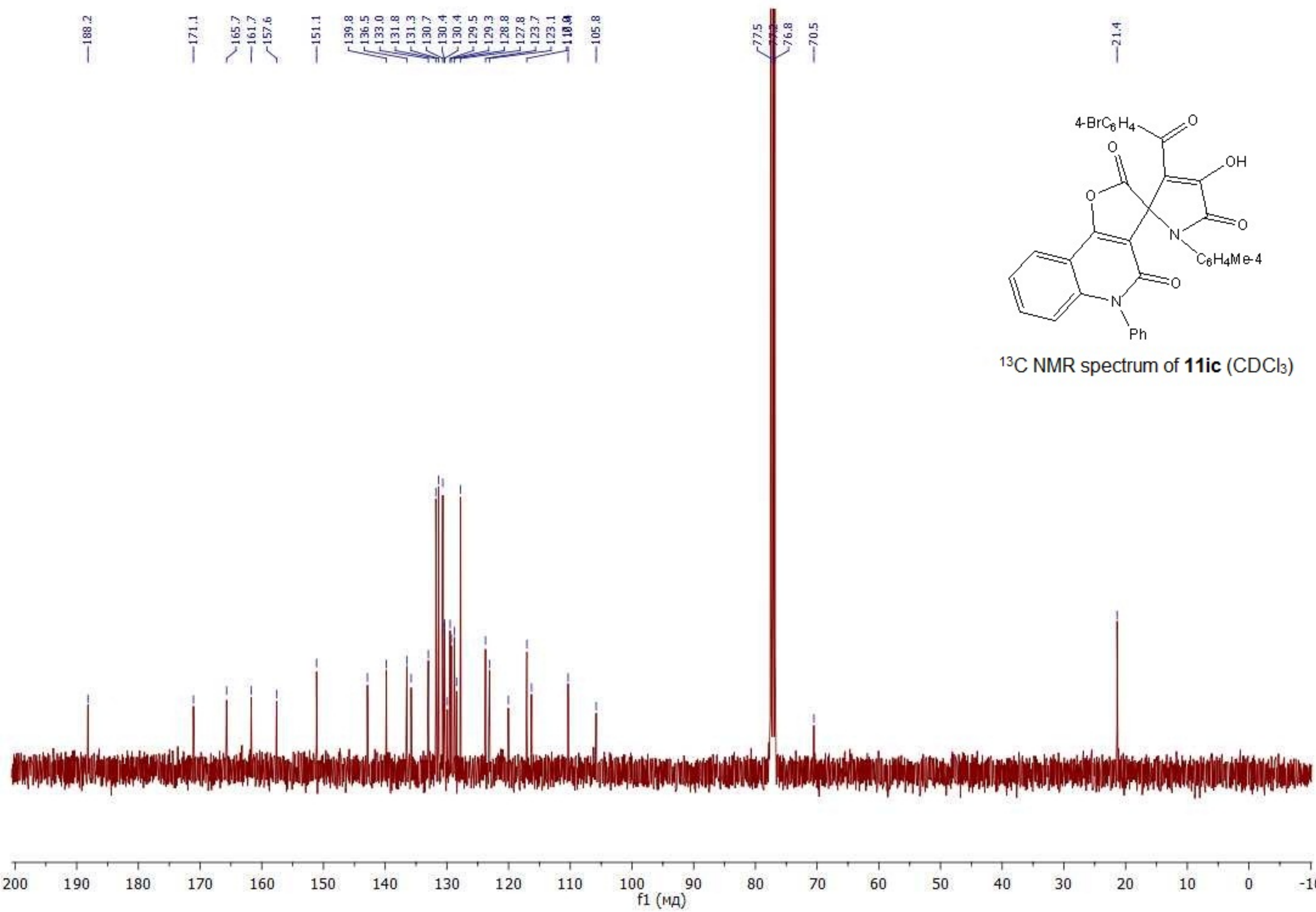
[1]H NMR spectrum of **11ha** ($CDCl_3$)



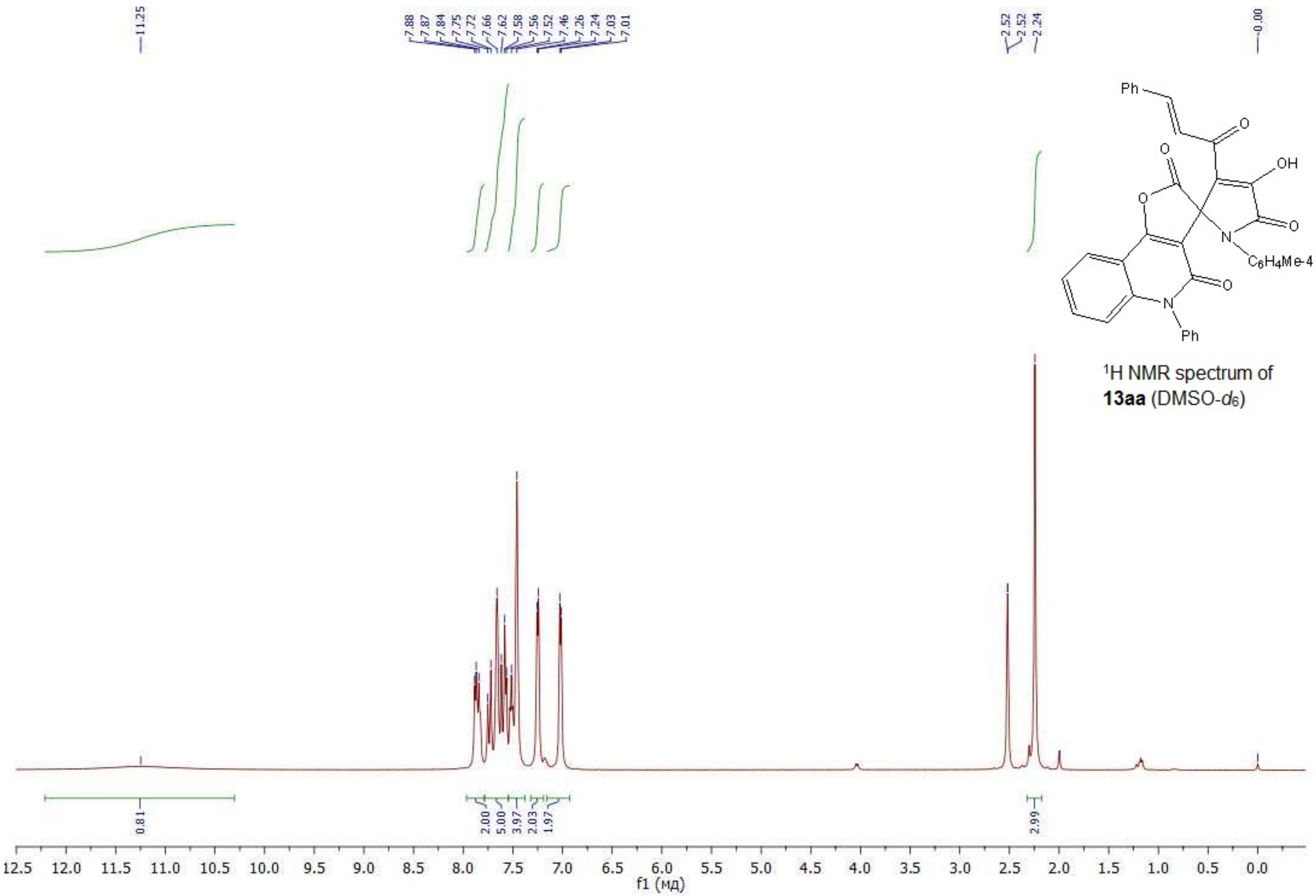
^{13}C NMR spectrum of **11ha** ($CDCl_3$)



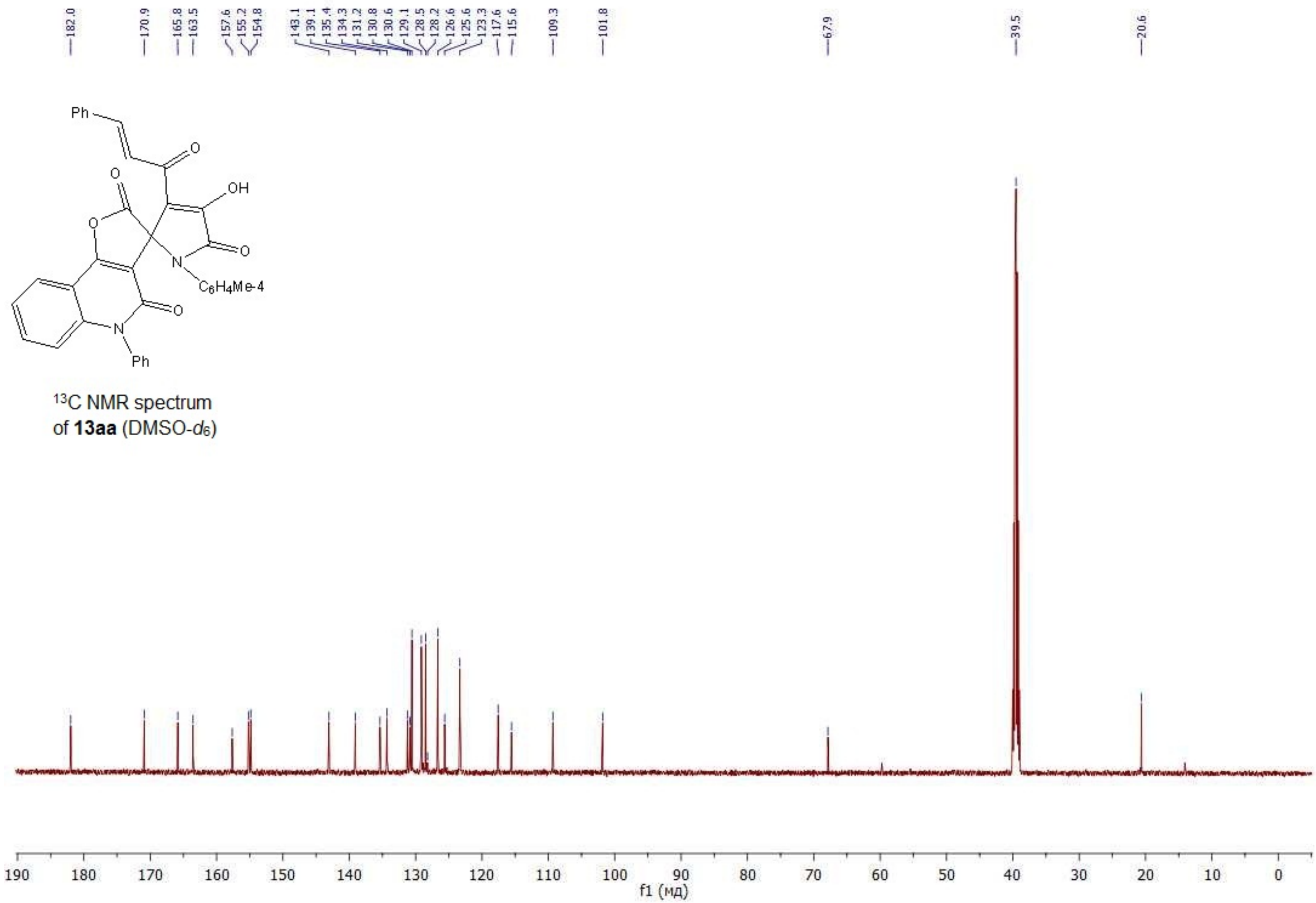
^{1}H NMR spectrum of **11ic** ($CDCl_3$)



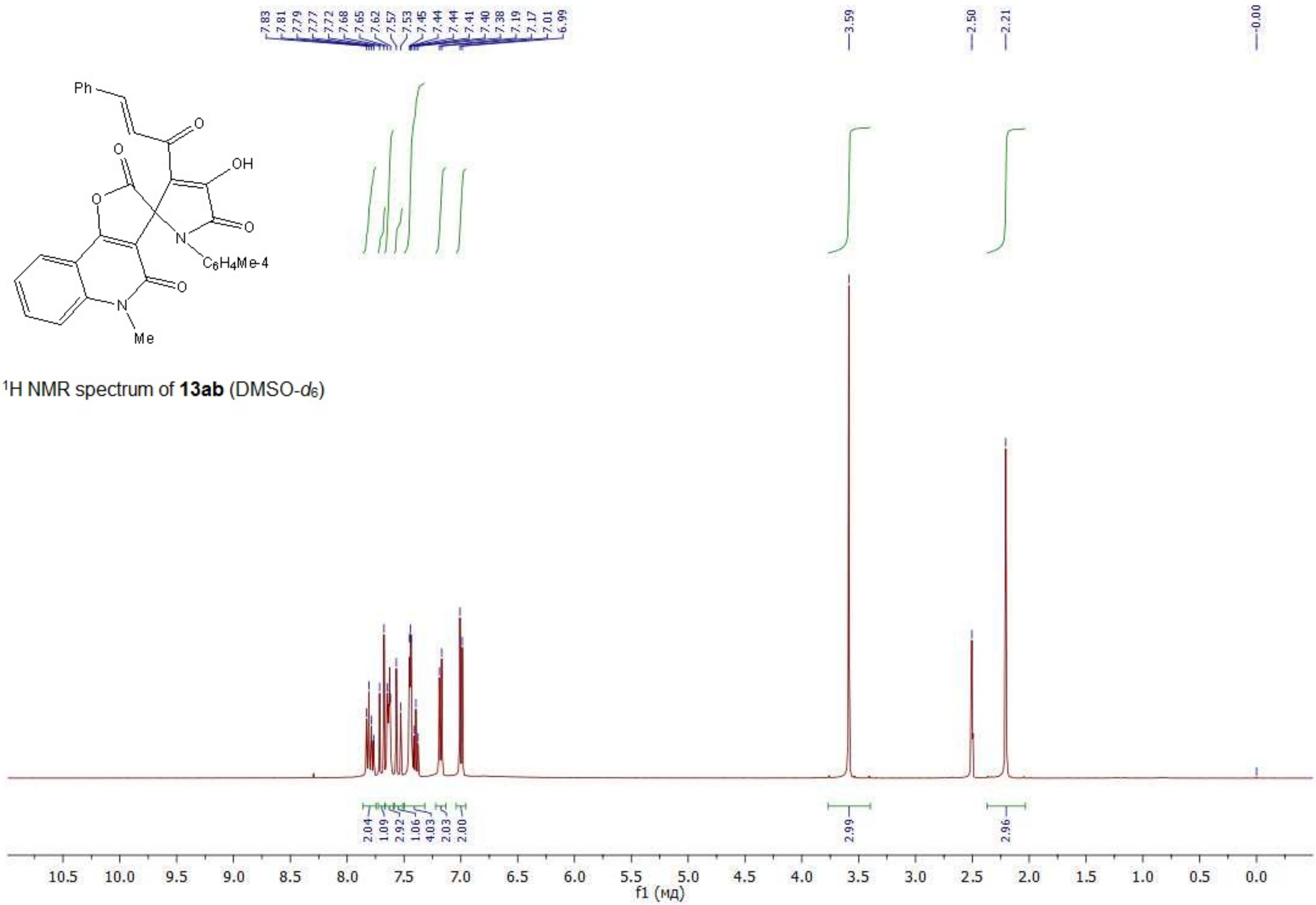
^{13}C NMR spectrum of **11ic** ($CDCl_3$)



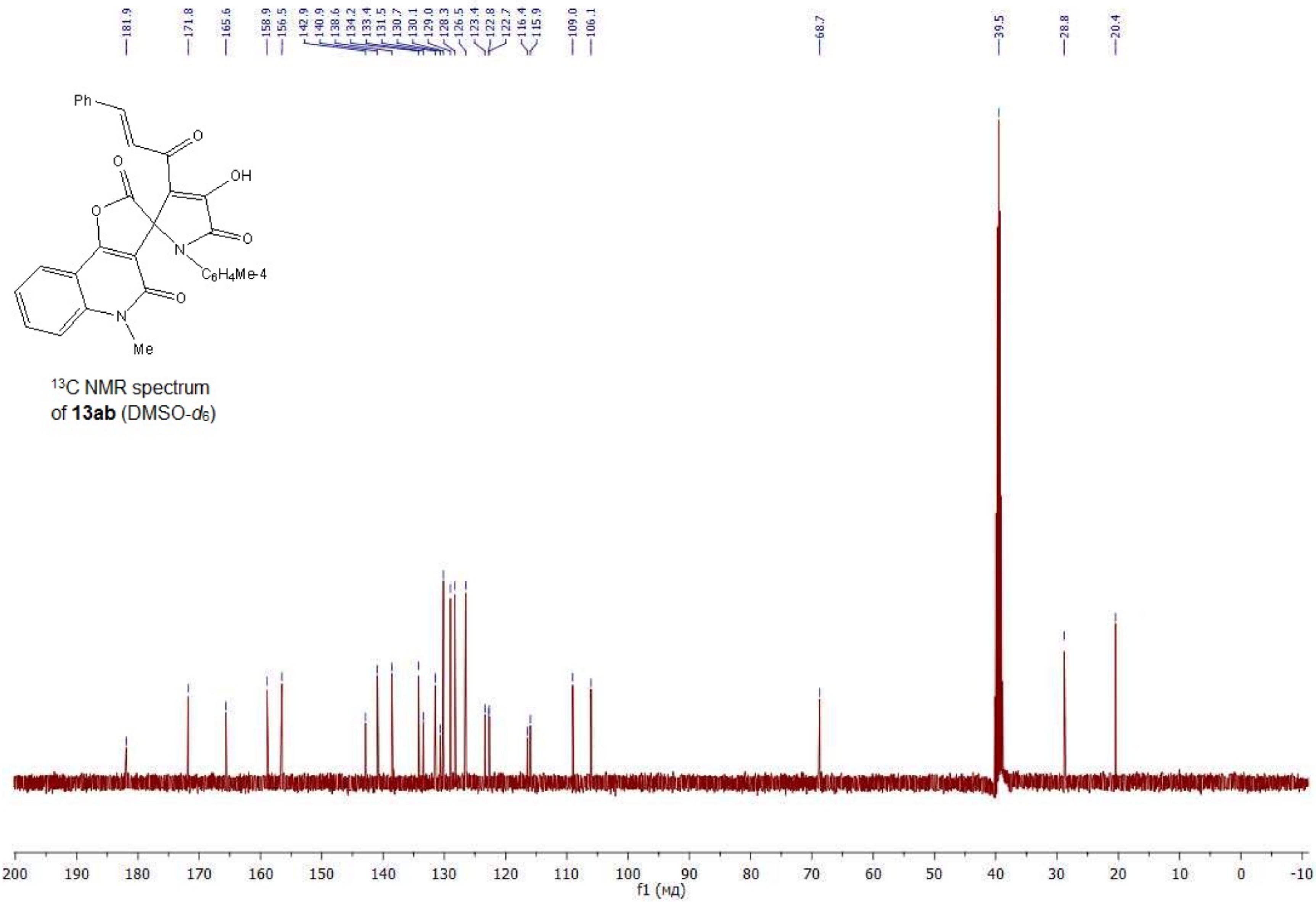
[1]H NMR spectrum of **13aa** (DMSO-d_6)



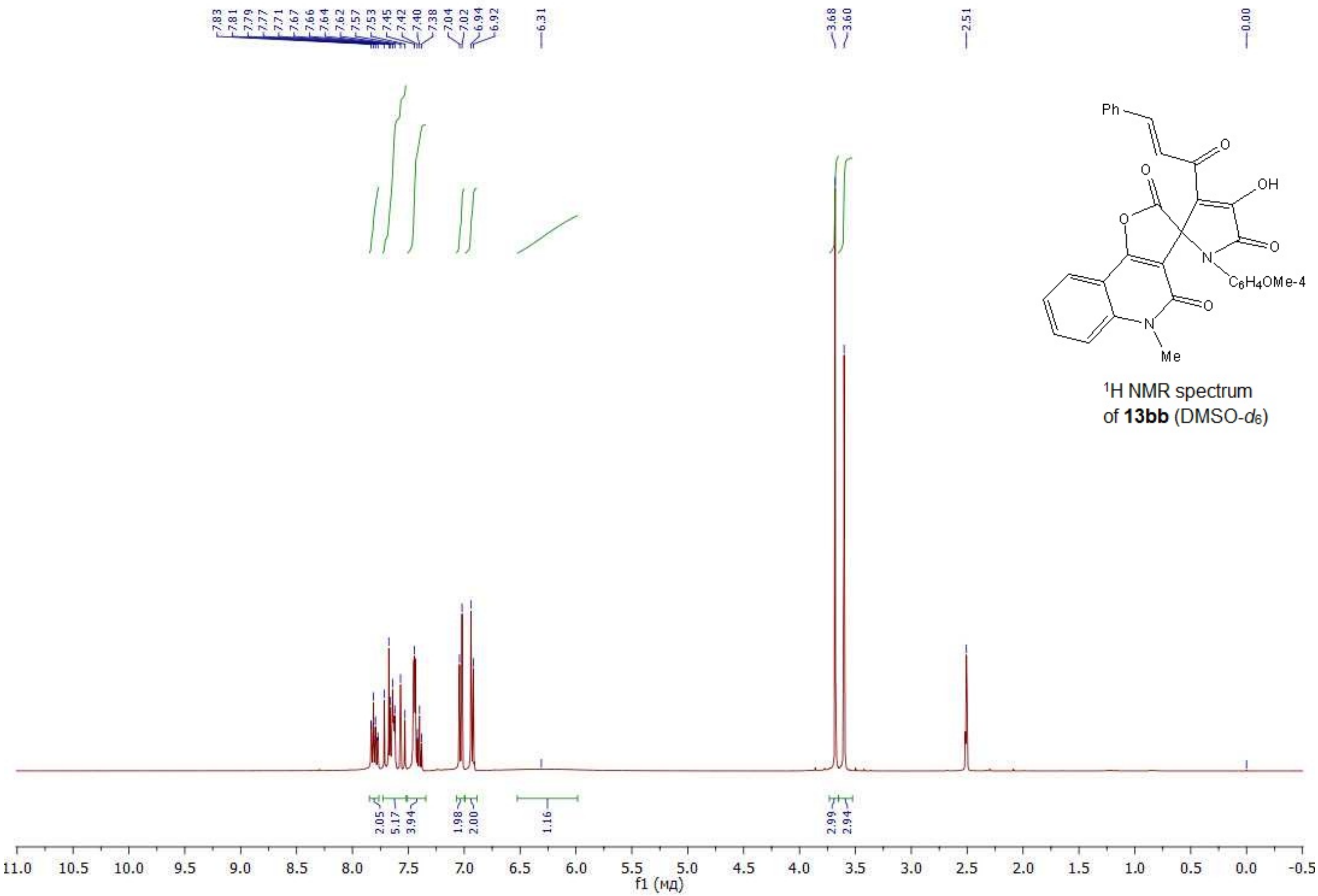
^{13}C NMR spectrum of **13aa** (DMSO-d_6)



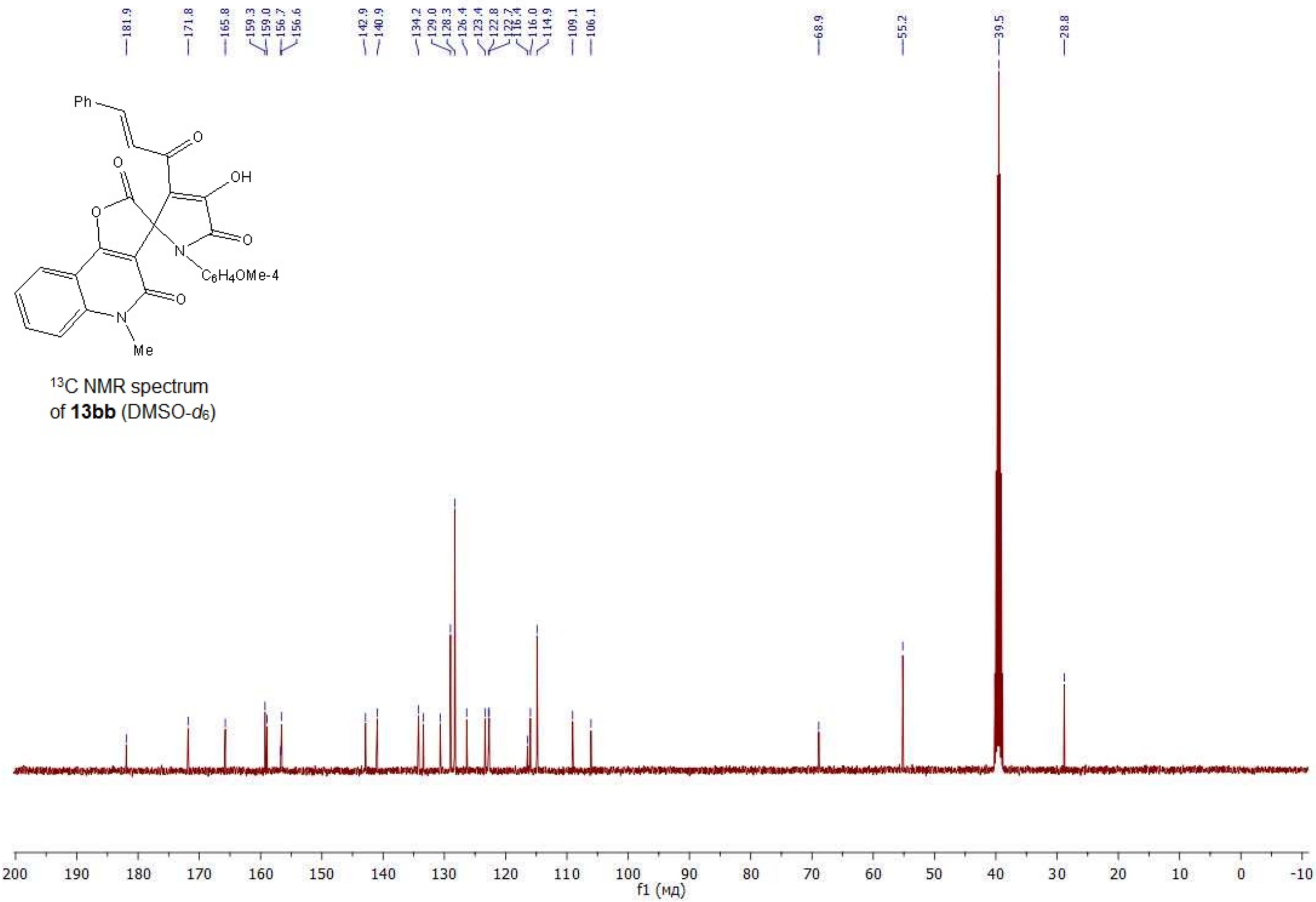
[1]H NMR spectrum of **13ab** (DMSO-d_6)



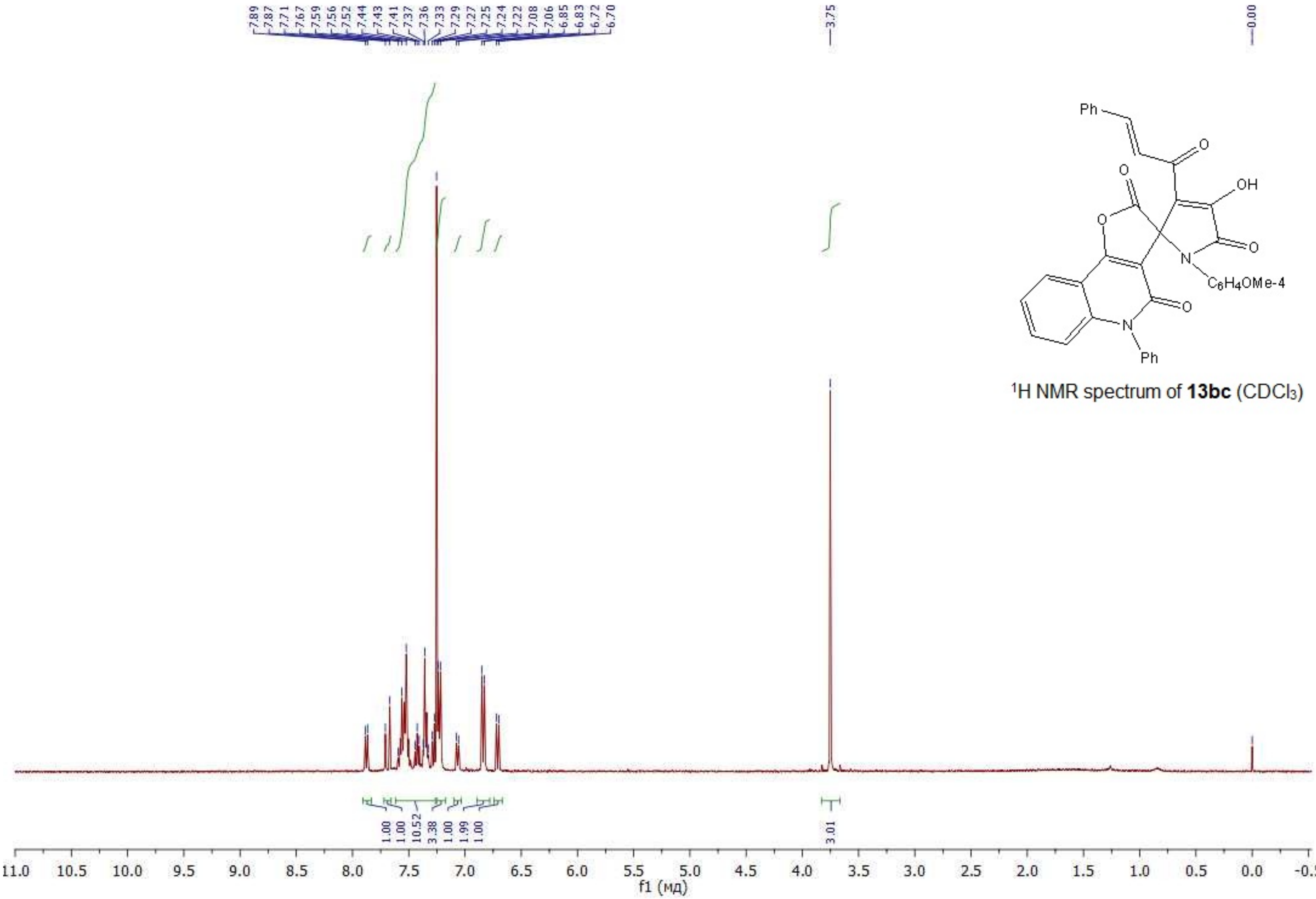
^{13}C NMR spectrum of **13ab** (DMSO-d_6)



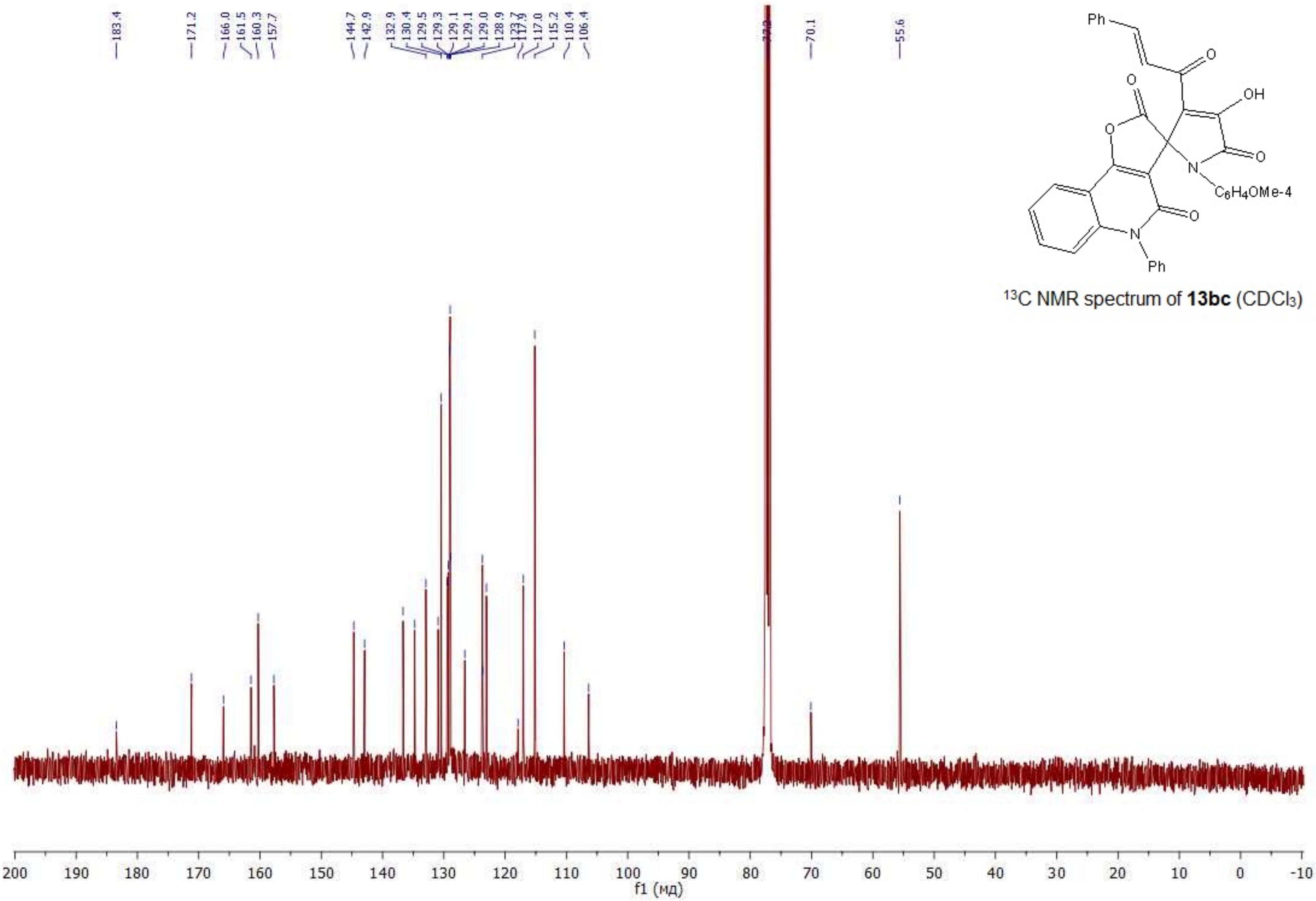
^{1}H NMR spectrum of **13bb** (DMSO-d_6)



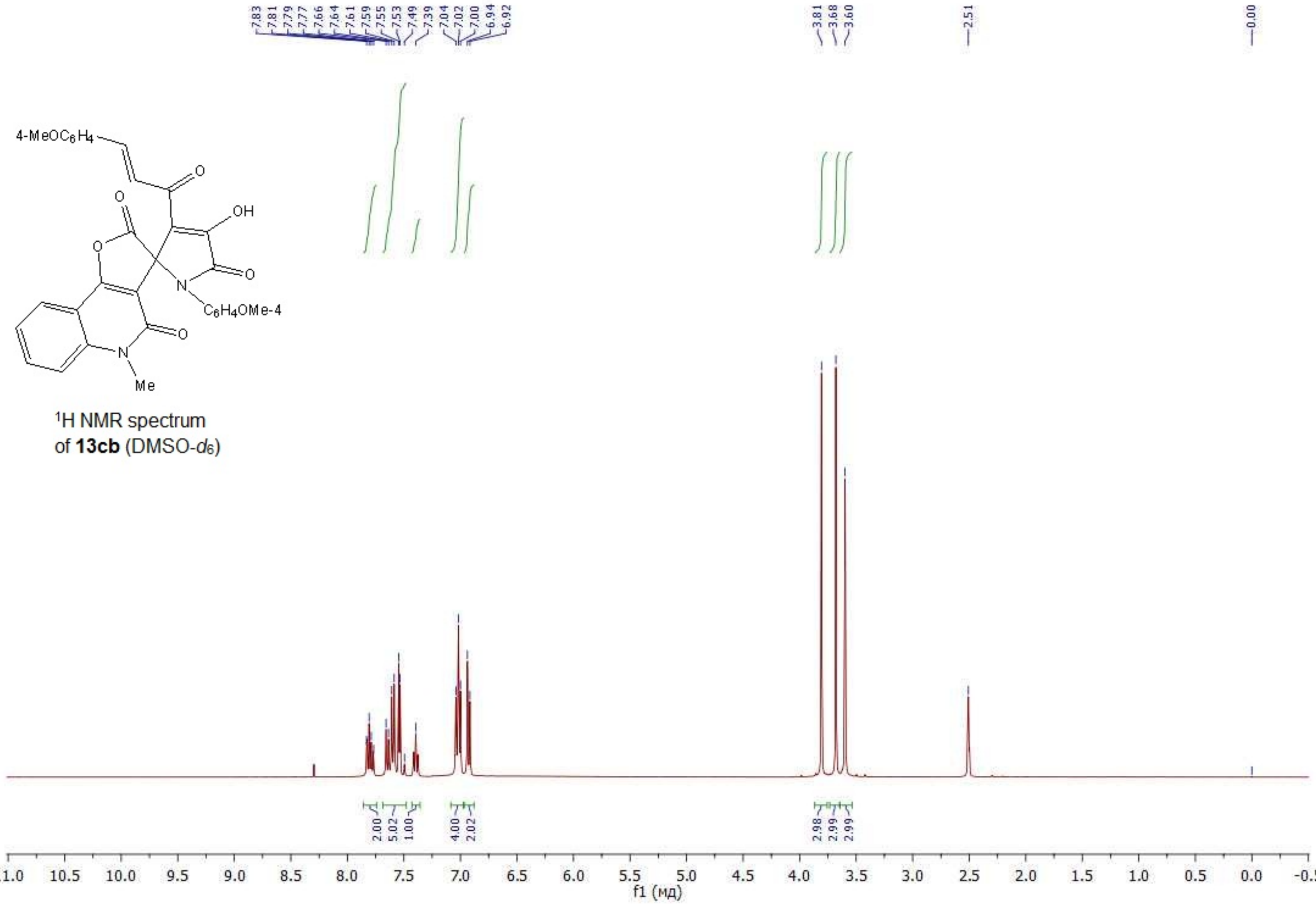
^{13}C NMR spectrum of **13bb** (DMSO-d_6)



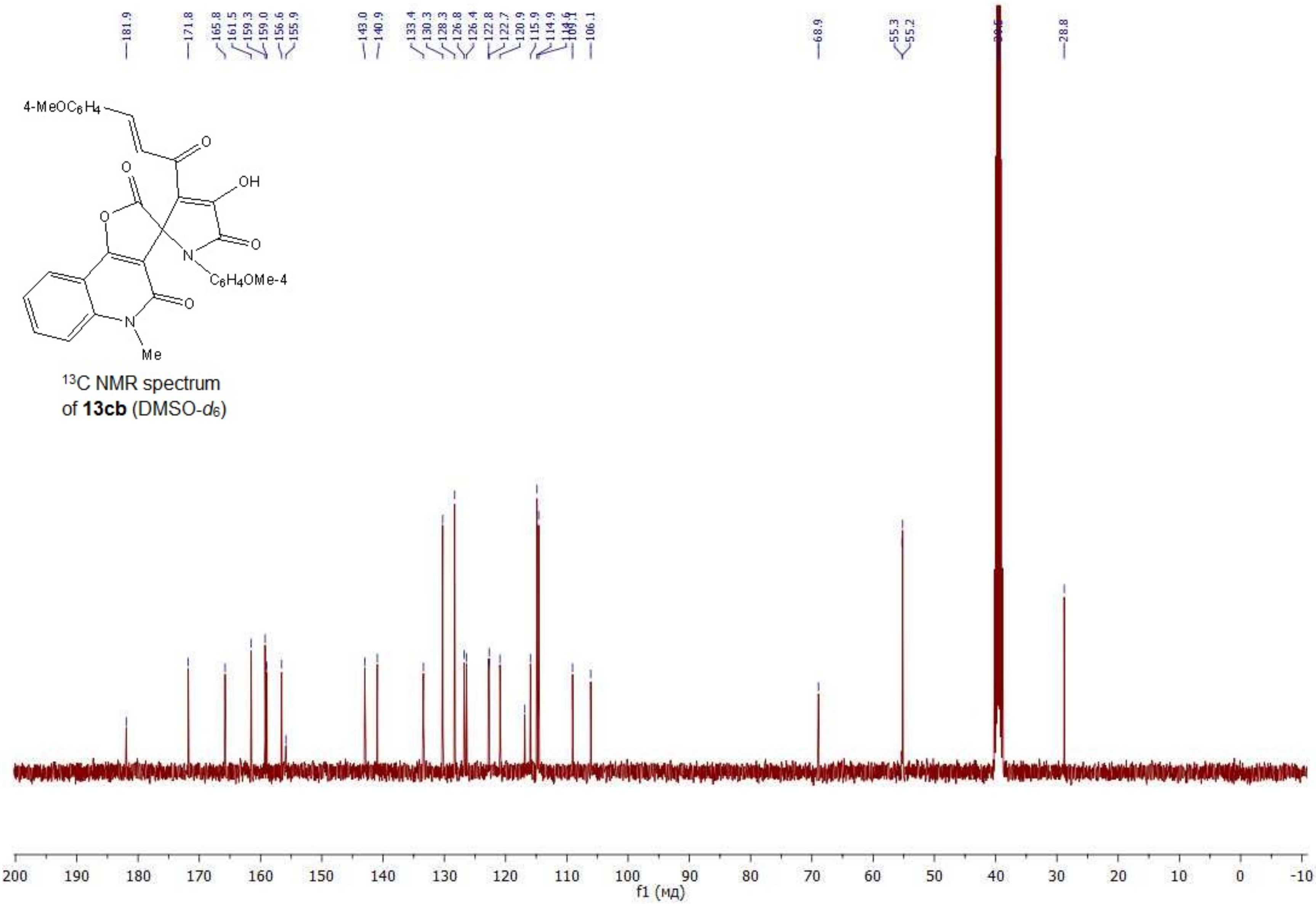
[1]H NMR spectrum of **13bc** ($CDCl_3$)



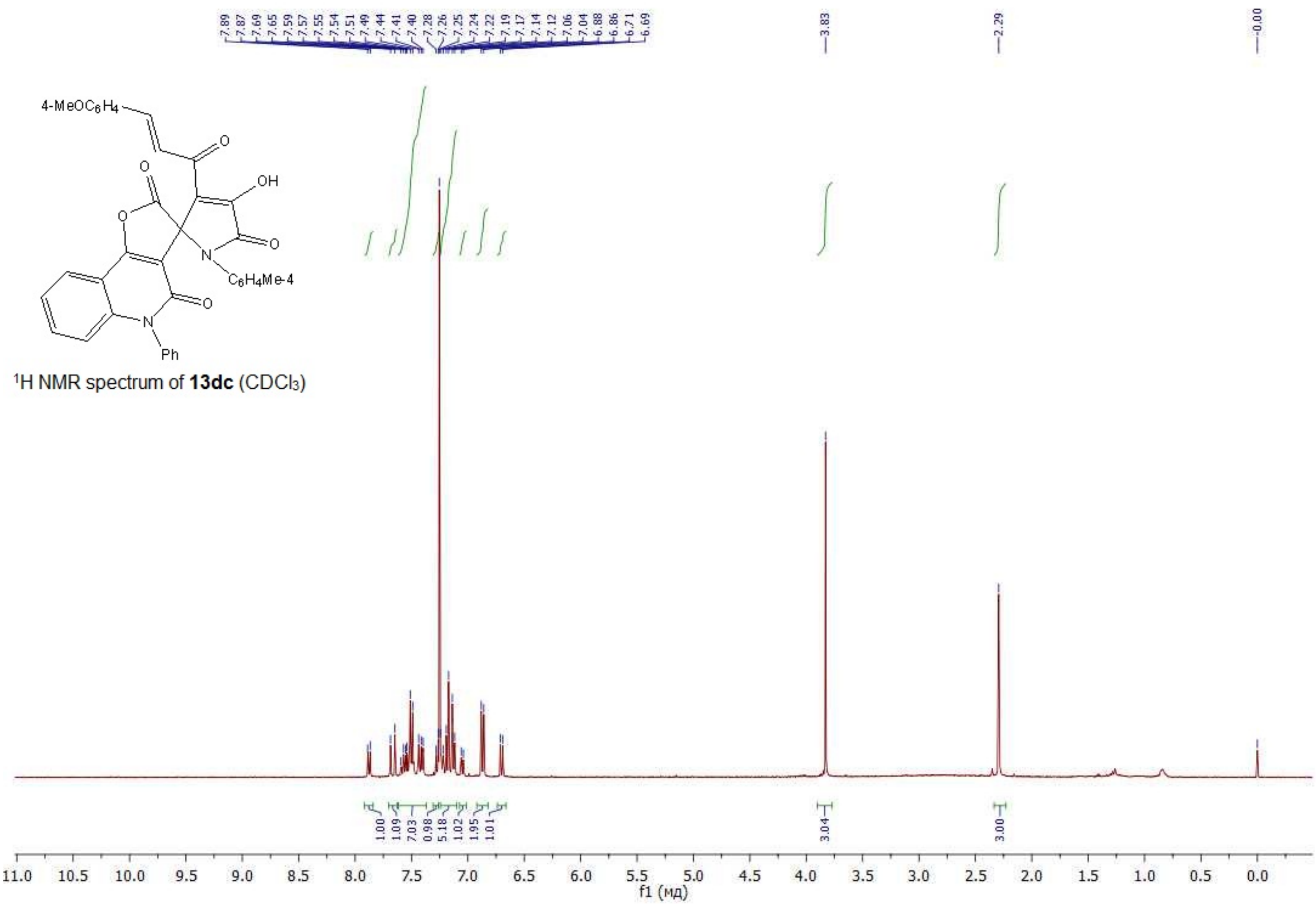
^{13}C NMR spectrum of **13bc** ($CDCl_3$)



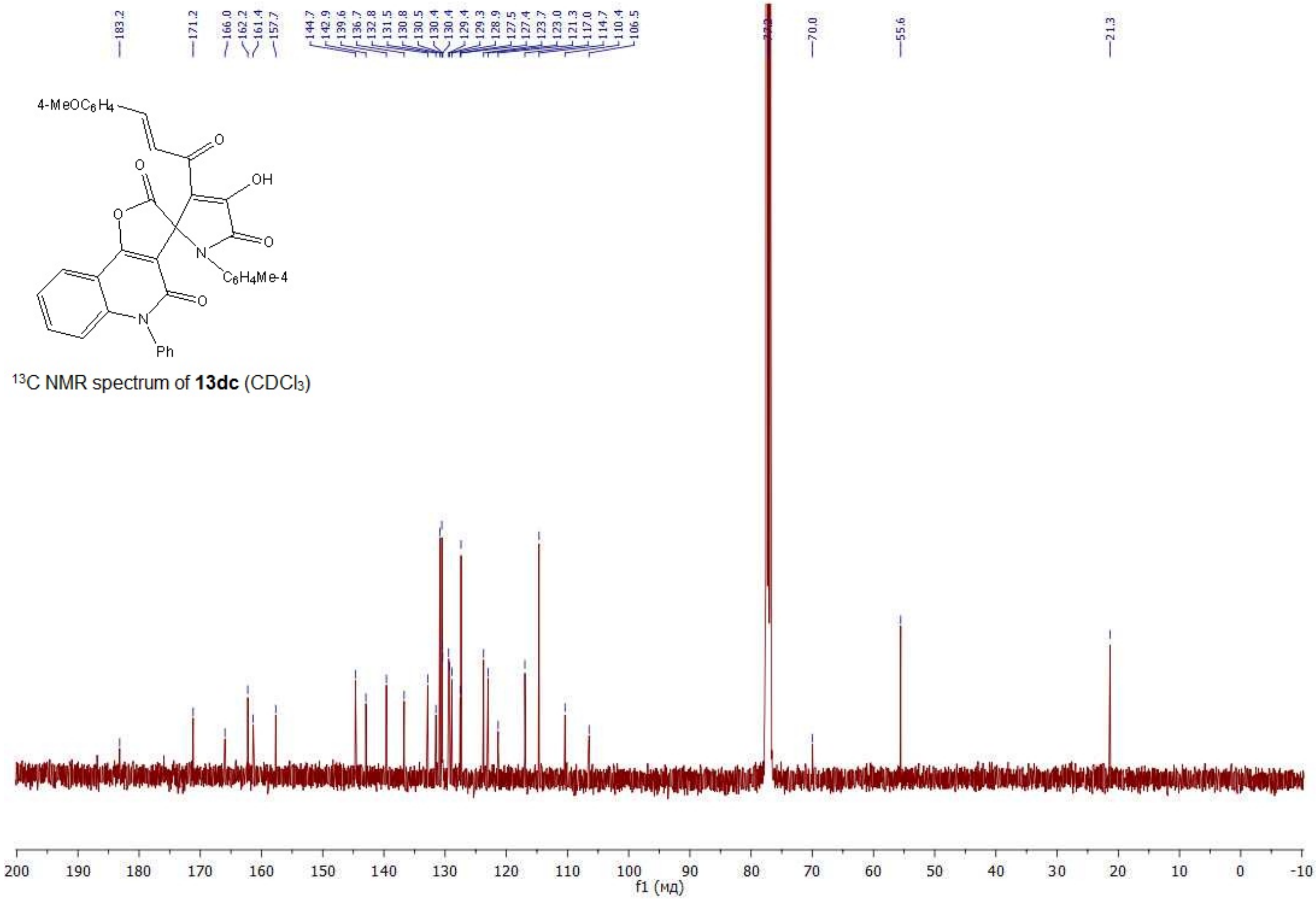
[1]H NMR spectrum of **13cb** (DMSO-d_6)



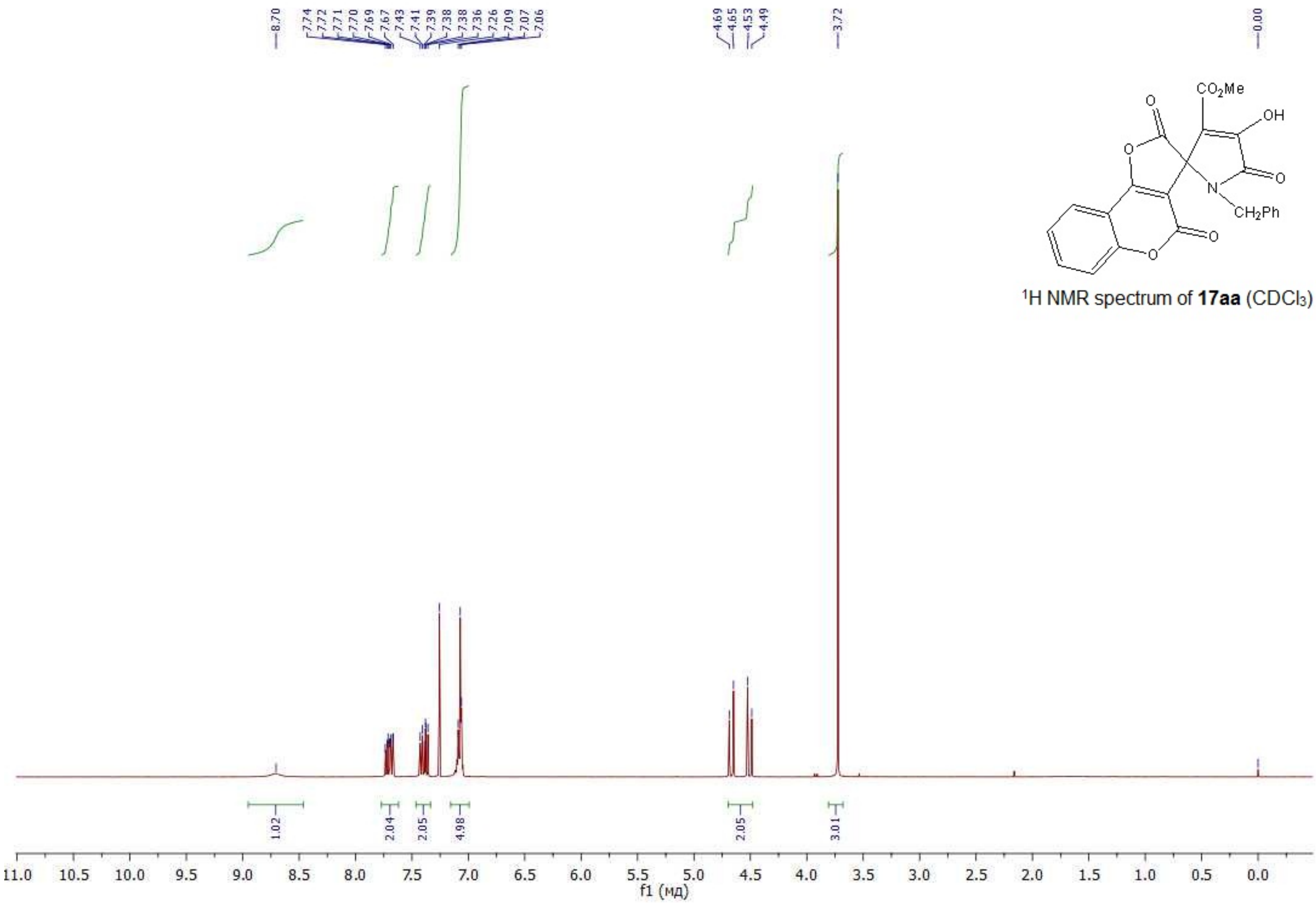
^{13}C NMR spectrum
of **13cb** (DMSO-d_6)



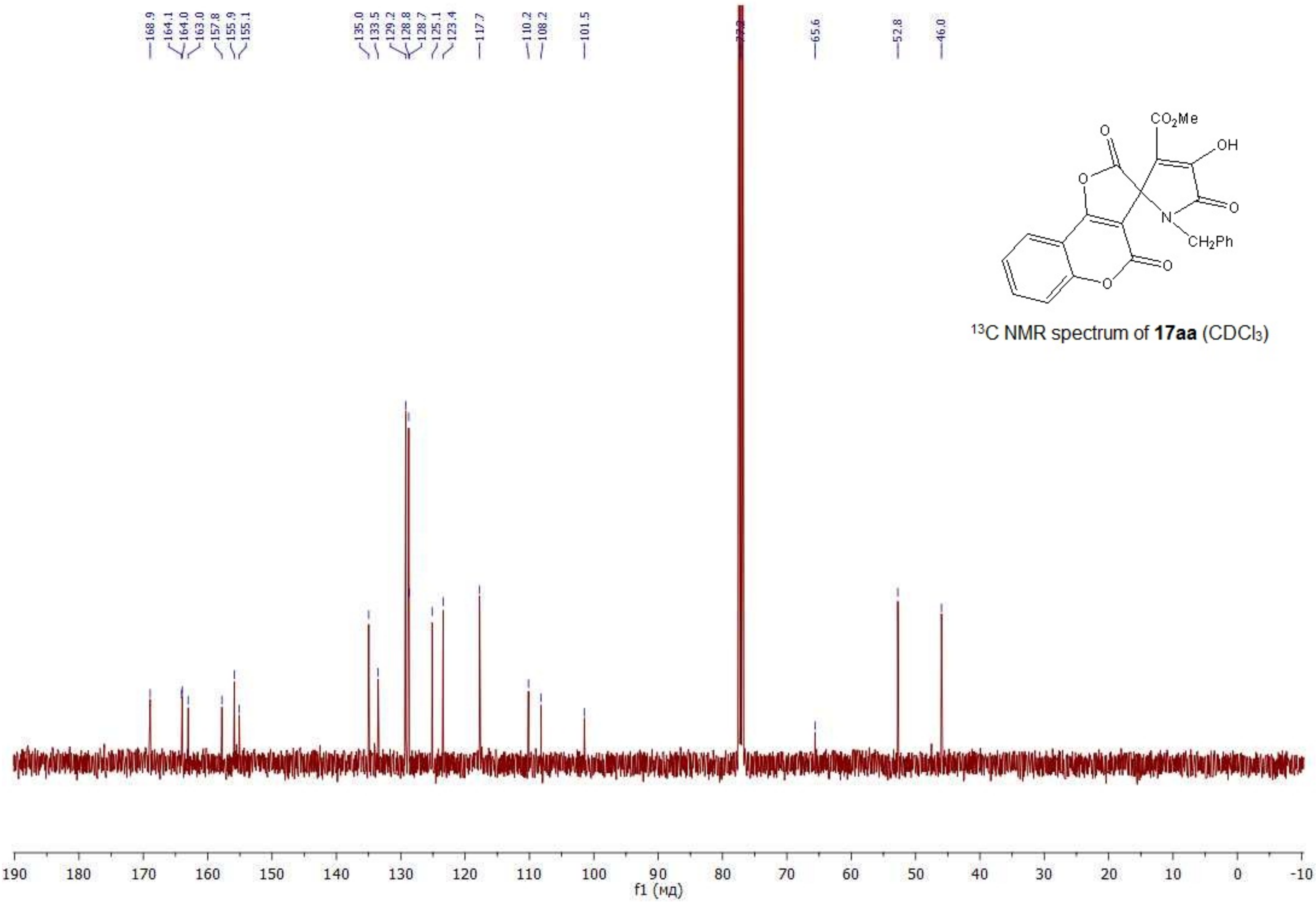
[1]H NMR spectrum of **13dc** (CDCl3)



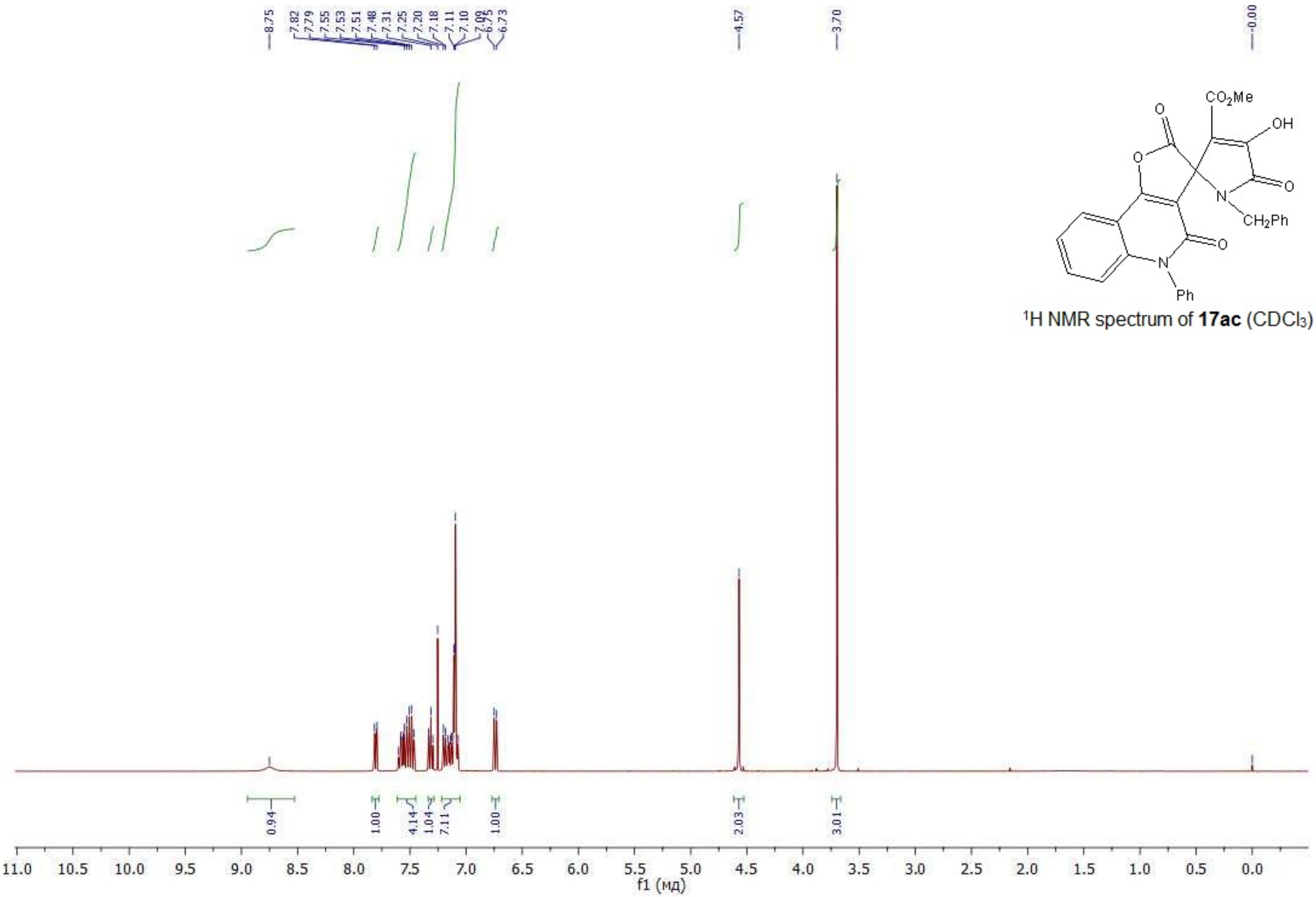
^{13}C NMR spectrum of **13dc** ($CDCl_3$)



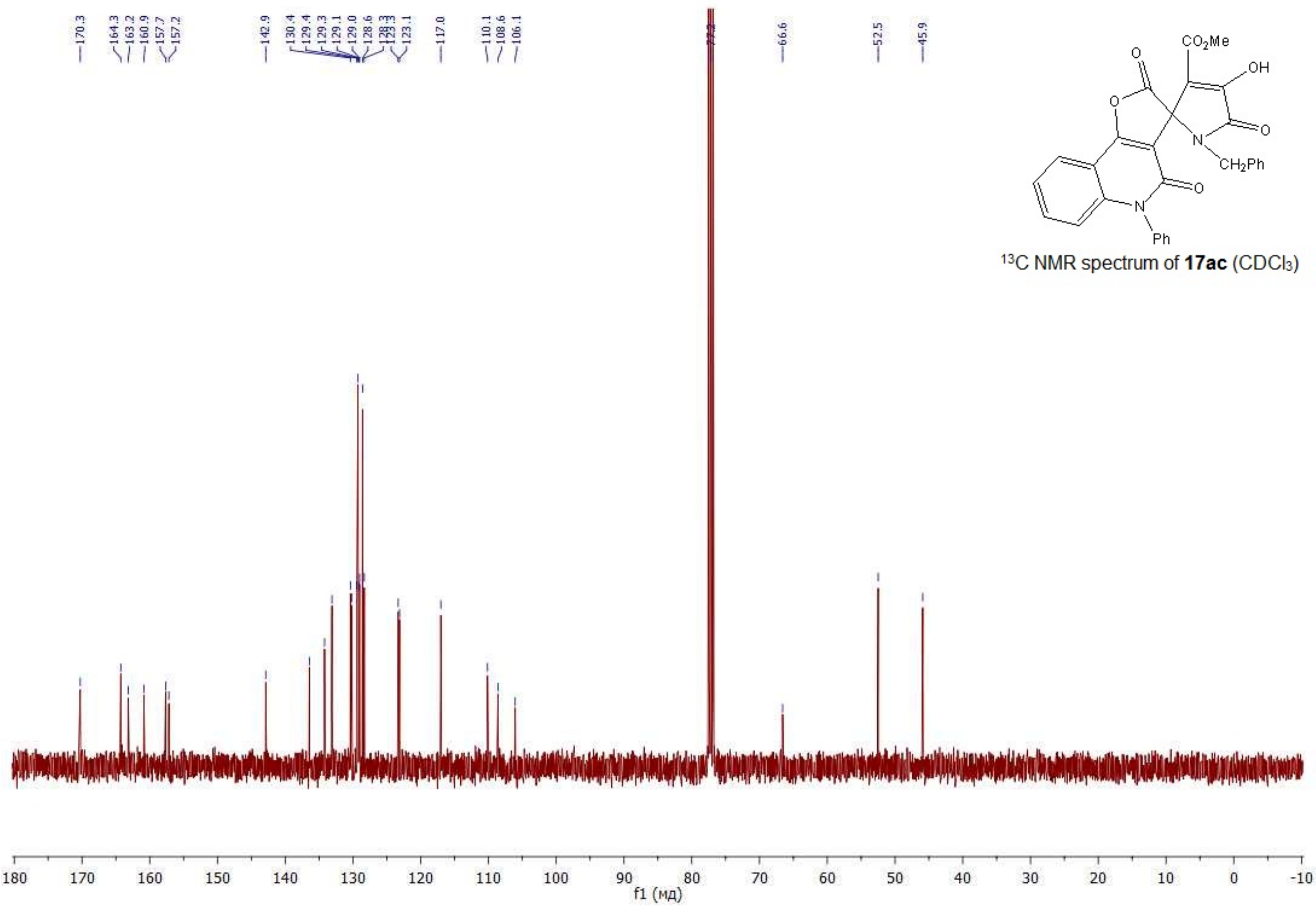
[1]H NMR spectrum of **17aa** ($CDCl_3$)



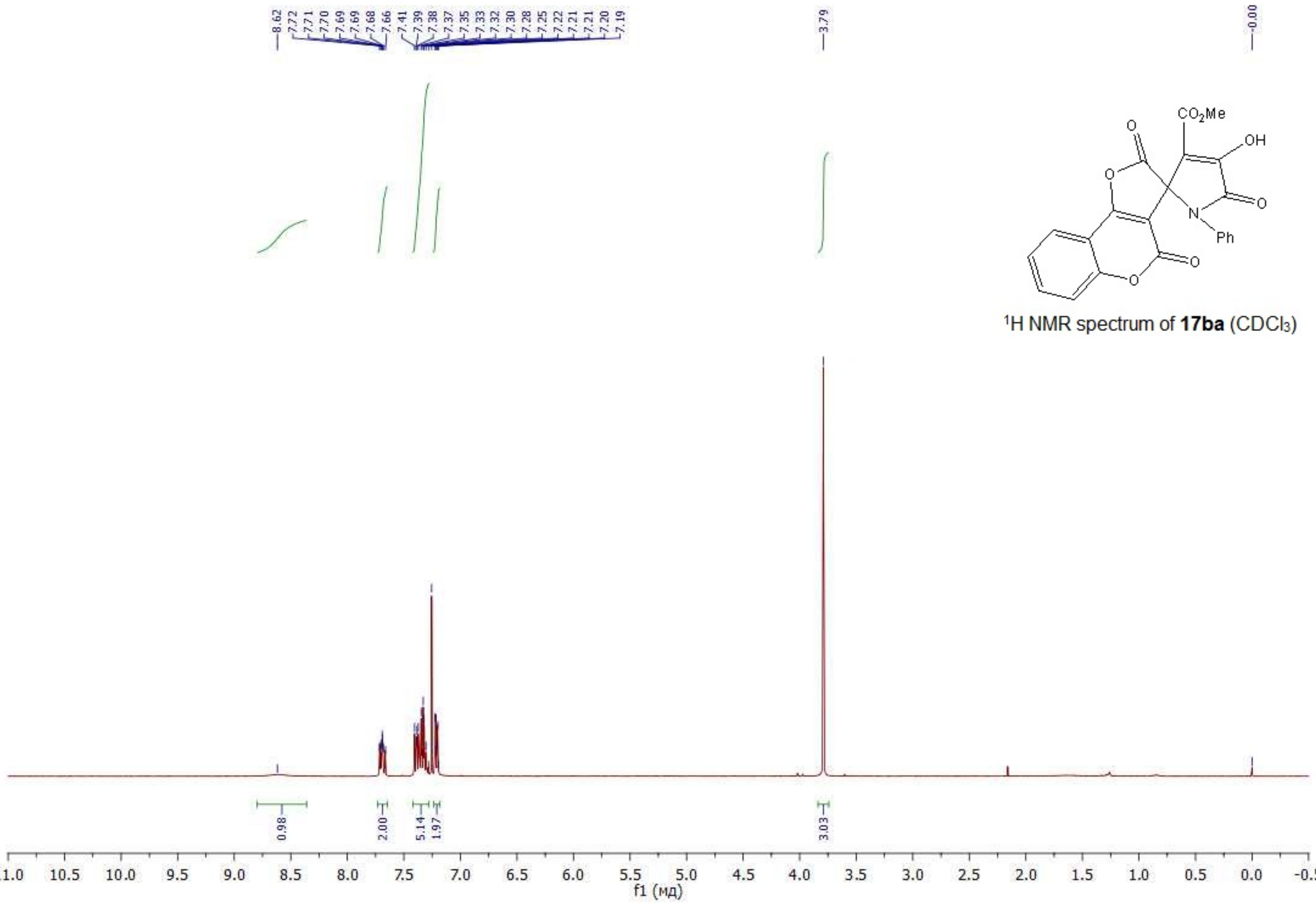
^{13}C NMR spectrum of **17aa** ($CDCl_3$)



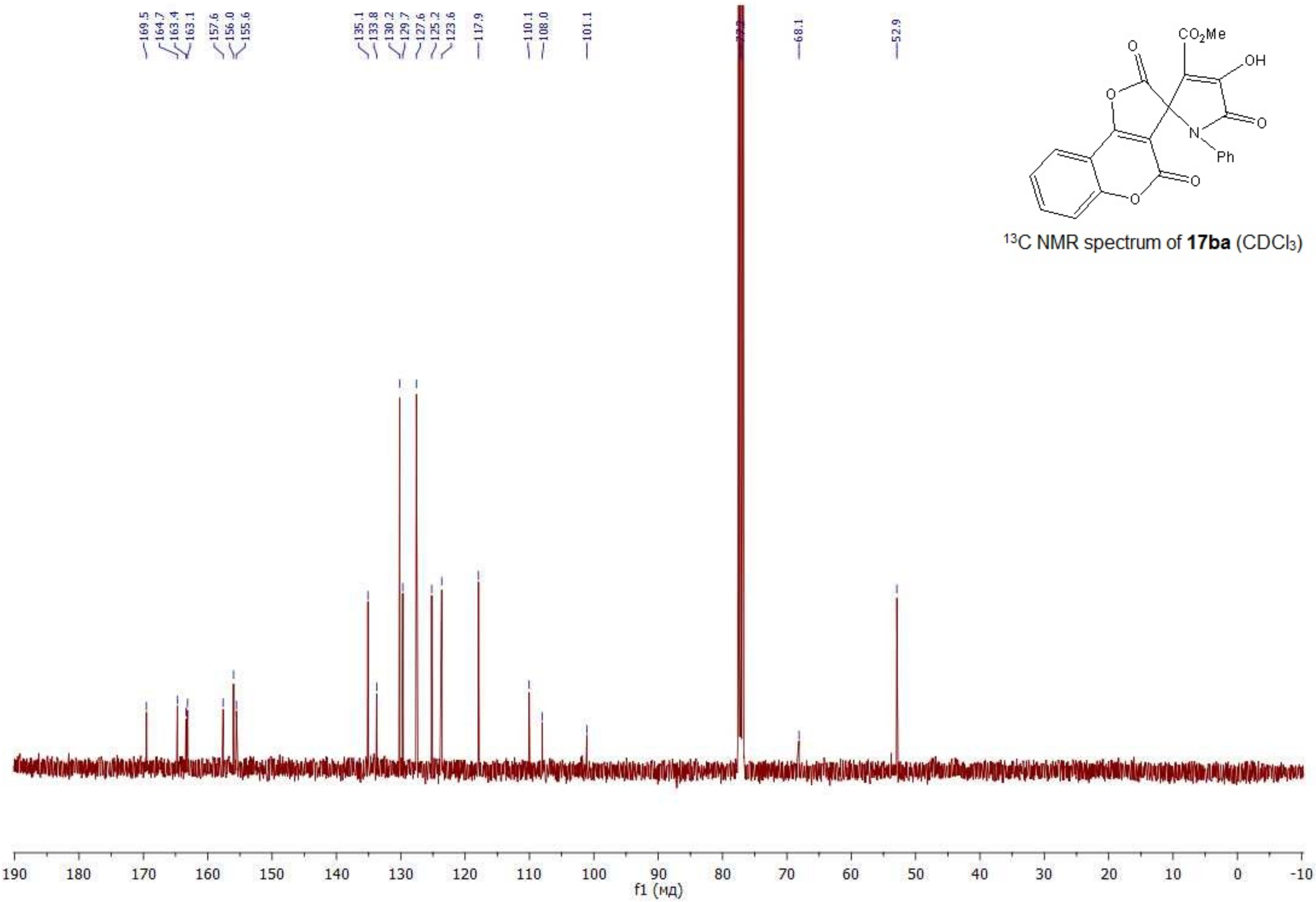
[1]H NMR spectrum of **17ac** ($CDCl_3$)



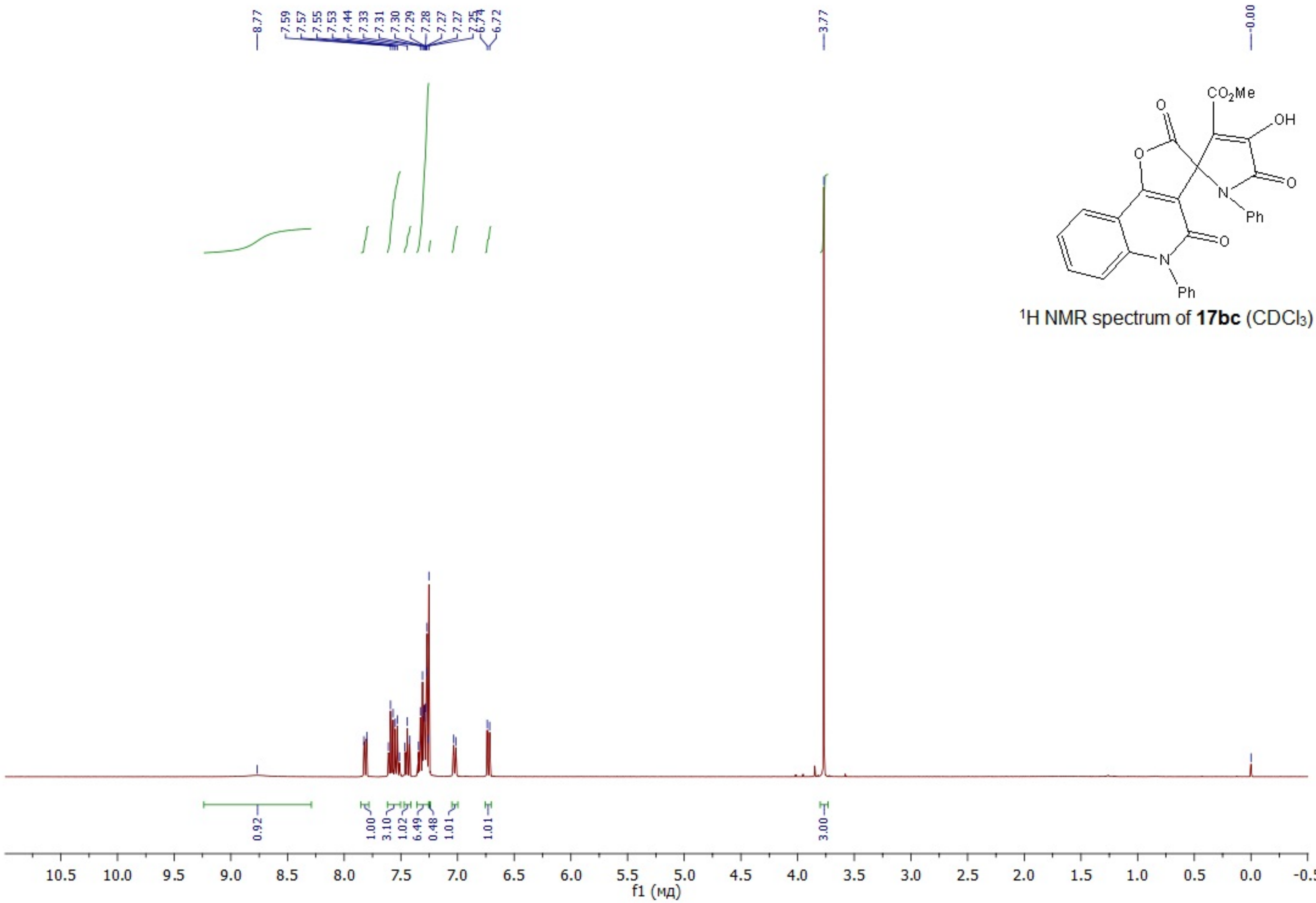
^{13}C NMR spectrum of **17ac** ($CDCl_3$)



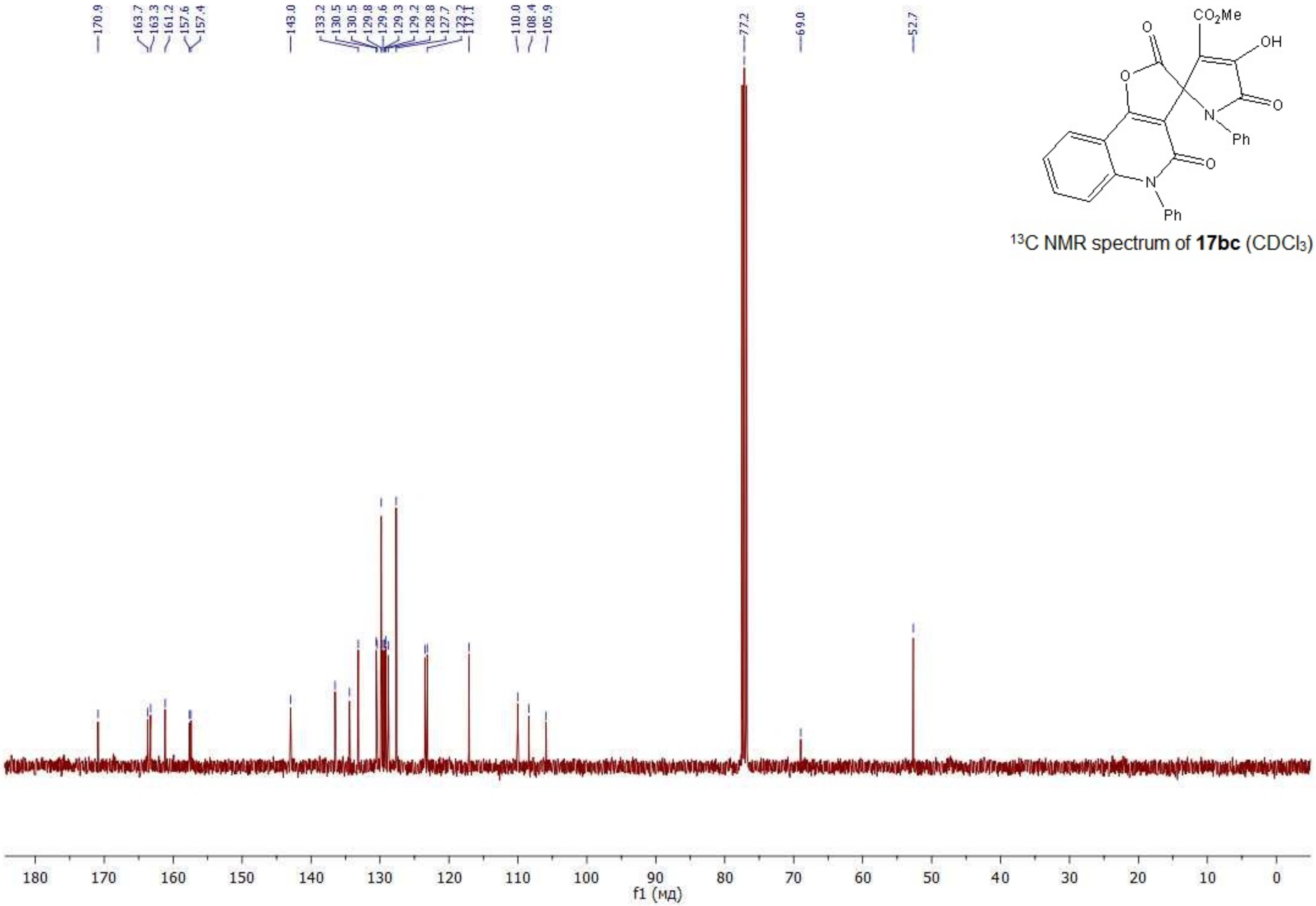
^{1}H NMR spectrum of **17ba** ($CDCl_3$)



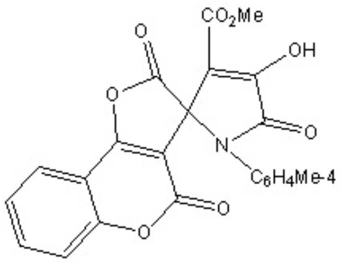
^{13}C NMR spectrum of **17ba** ($CDCl_3$)



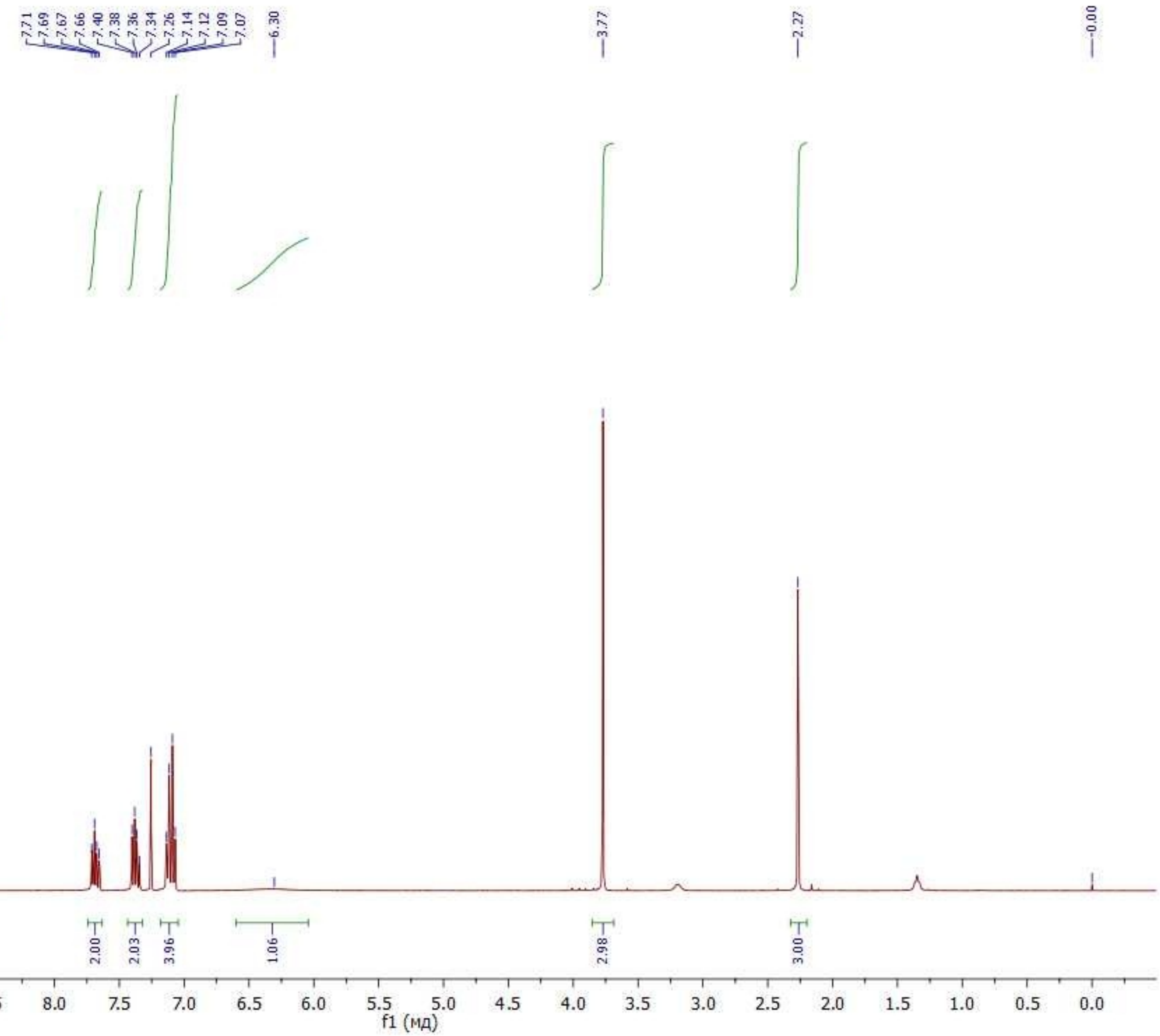
[1]H NMR spectrum of **17bc** ($CDCl_3$)

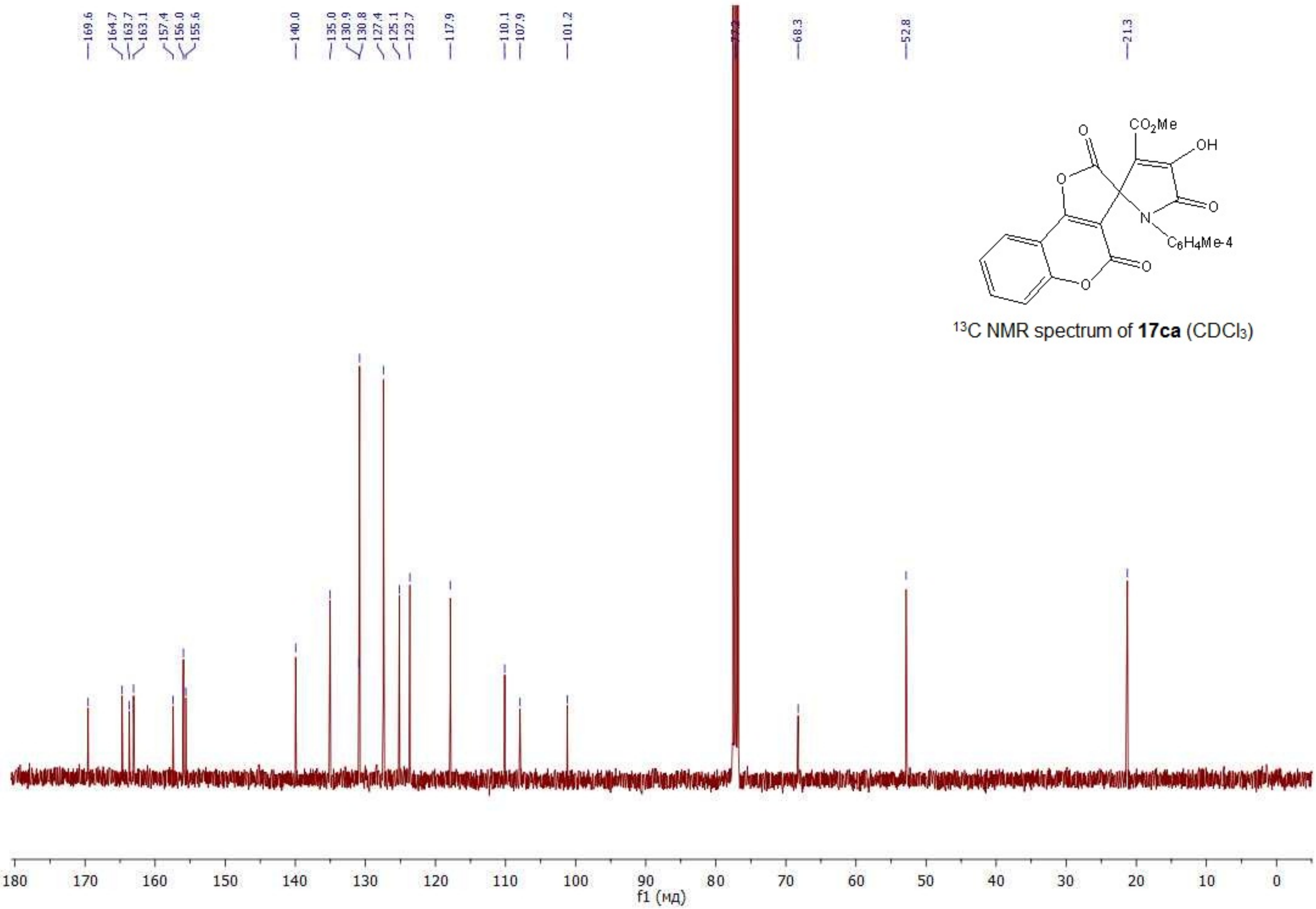


^{13}C NMR spectrum of **17bc** ($CDCl_3$)

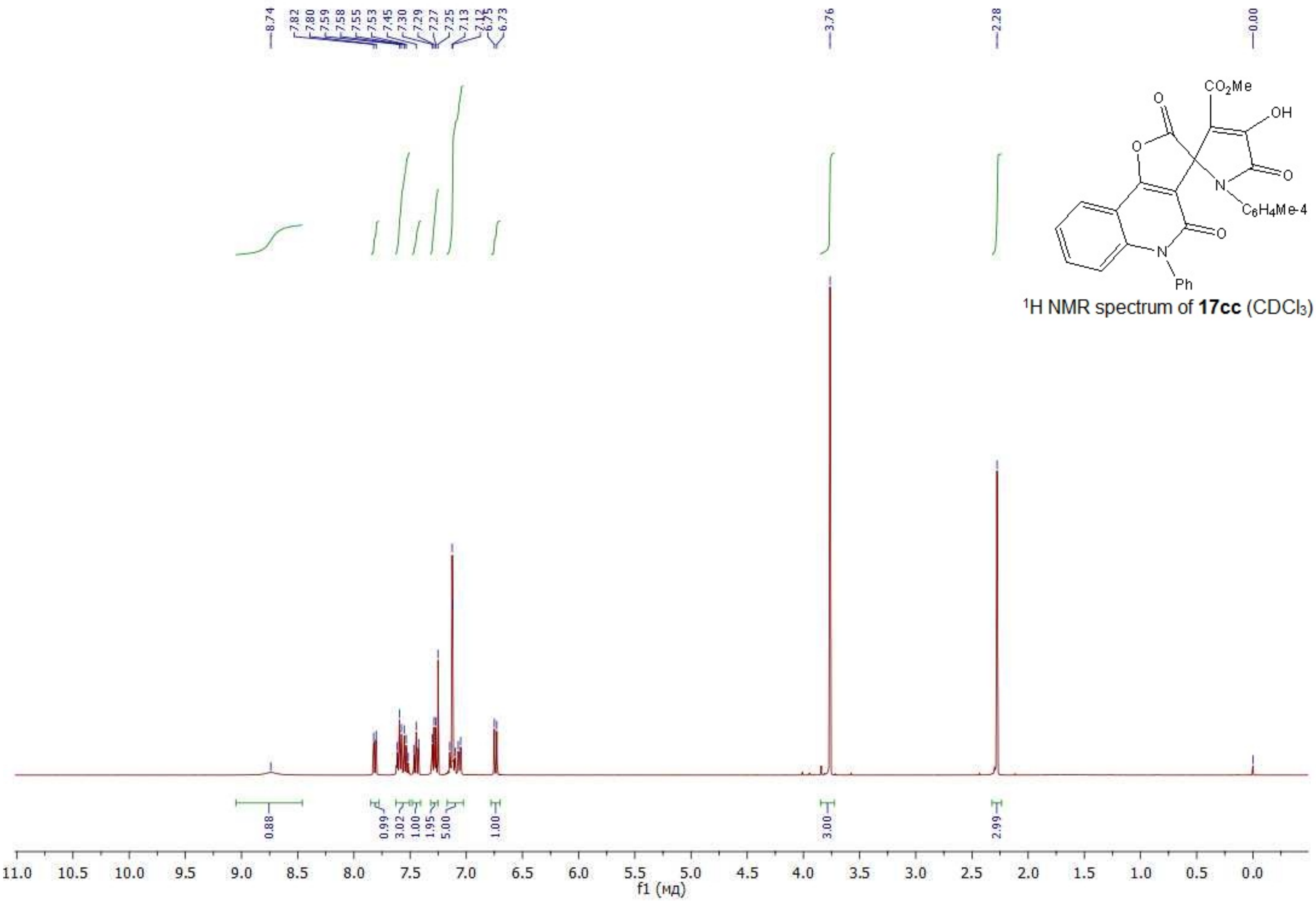


^{1}H NMR spectrum of **17ca** ($CDCl_3$)

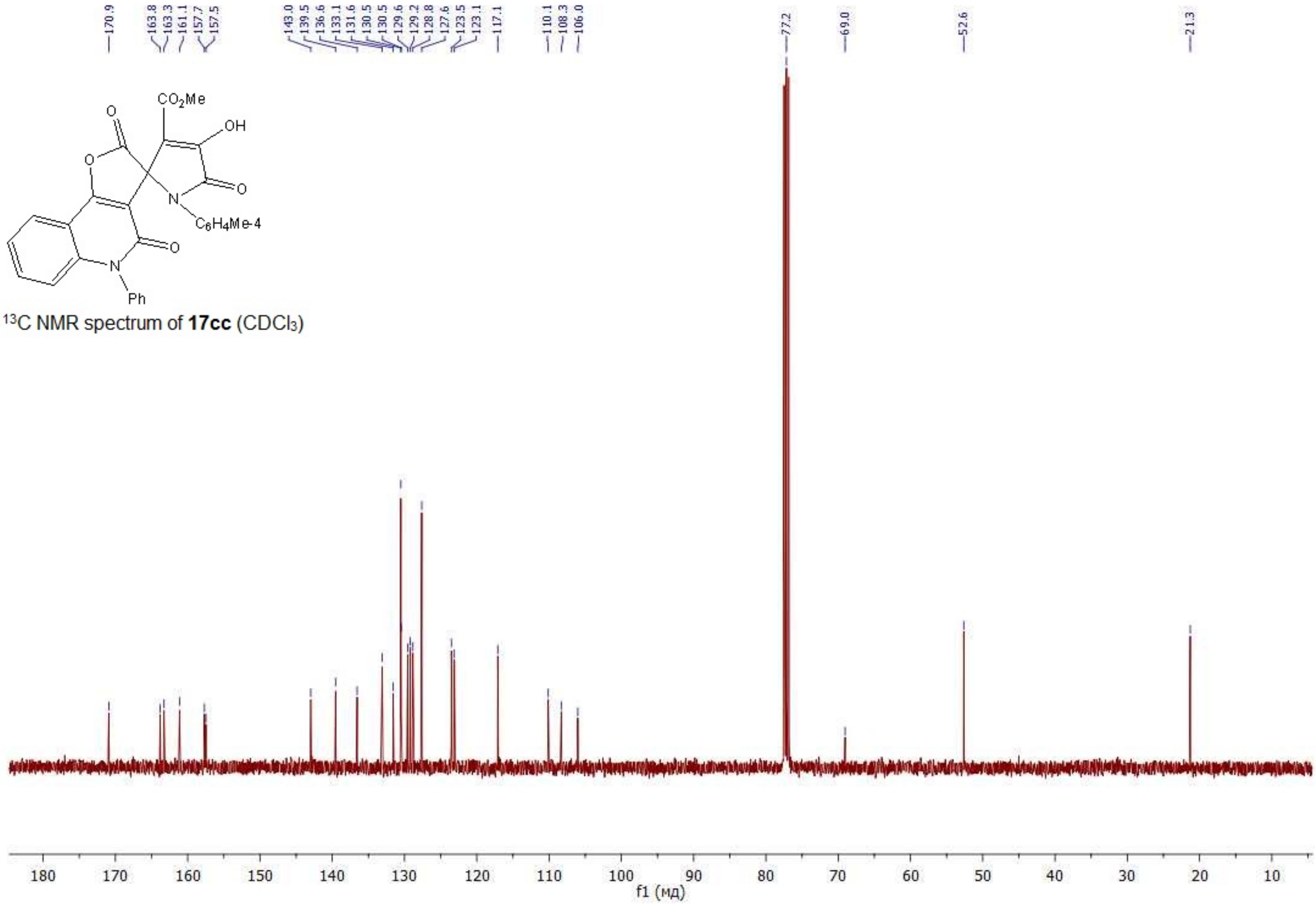




^{13}C NMR spectrum of **17ca** ($CDCl_3$)



[1]H NMR spectrum of **17cc** ($CDCl_3$)



^{13}C NMR spectrum of **17cc** ($CDCl_3$)

Preparation of X-ray quality crystals

The crystals for XRD experiments were prepared by crystallization as follows: Drum vial (volume 15 mL) was charged with crude solid material (100 mg) and mixture (1:1:1:1) of four solvents: ethyl acetate, acetone, acetonitrile, and toluene. The mixture was heated to boil until solid material was fully dissolved, then covered with glass lead and allowed to cool down to room temperature. After three days the formed crystalline crop was filtered on a sintered funnel, which was turned upside down. Upon drying the crystals were gradually dropping down to a Petri dish. Several well-formed transparent X-ray quality crystals were picked manually.

Crystal structure determination

The unit cell parameters and the X-ray diffraction intensities were measured on a Xcalibur Ruby diffractometer. The empirical absorption correction was introduced by multi-scan method using SCALE3 ABSPACK algorithm[1]. The structures were solved by direct method and refined by the full-matrix least-squares method in the anisotropic approximation for all non-hydrogen atoms using the SHELXS-97 and SHELXL-97[2] (**11ba**) or SHELXL-2014[3] and OLEX2[4] (**11db**) program packages. Hydrogen atoms bound to carbon were located from the Fourier synthesis of the electron density and refined using a riding model. The hydrogen atoms of hydroxyl groups were refined independently with isotropic displacement parameter.

*Crystal Data of **11ba**.* $C_{28}H_{17}NO_8$, M = 495.43, monoclinic, a = 8.2673(11) Å, b = 18.169(3) Å, c = 14.955(2) Å, β = 91.962(13) °, V = 2245.1(6) Å^3, T = 295(2) K, space group $P2_1/c$, Z = 4, μ(Mo Kα) = 0.109 mm^{-1}. The final refinement parameters: R_1 = 0.0480, wR_2 = 0.1208 (for observed 4053 reflections with $I > 2\sigma(I)$); R_1 = 0.0658, wR_2 = 0.1333 (for all independent 5271 reflections), S = 1.059. Largest diff. peak and hole 0.240 and –0.217 ēÅ^{-3}.

*Crystal Data of **11db**.* $C_{29}H_{20}N_2O_6{\cdot}C_2H_3N$, M = 533.52, triclinic, a = 10.0480(16) Å, b = 10.5278(12) Å, c = 13.3170(16) Å, α = 83.352(10) °, β = 74.653(13) °, γ = 89.492(11) °, V = 1349.0(3) Å^3, T = 295(2) K, space group P–1, Z = 2, μ(Mo Kα) = 0.093 mm^{-1}. The final refinement parameters: R_1 = 0.0515, wR_2 = 0.1284 (for observed 4550 reflections with $I > 2\sigma(I)$); R_1 = 0.0729, wR_2 = 0.1455 (for all independent 6306 reflections), S = 1.035. Largest diff. peak and hole 0.197 and –0.193 ēÅ^{-3}.

CCDC 1486439 (**11ba**) and 1486440 (**11db**) contain the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* http://www.ccdc.cam.ac.uk.

1. CrysAlisPro, Agilent Technologies, Version 1.171.37.33 (release 27-03-2014 CrysAlis171 .NET).
2. Sheldrick G.M. *ActaCryst*. **2008**, *A64*, 112.
3. Sheldrick G.M. *ActaCryst*. **2015**, *C71*, 3.
4. Dolomanov O.V., Bourhis L.J., Gildea R.J, Howard J.A.K., Puschmann H. *J. Appl. Cryst.* **2009**, *42*, 339.

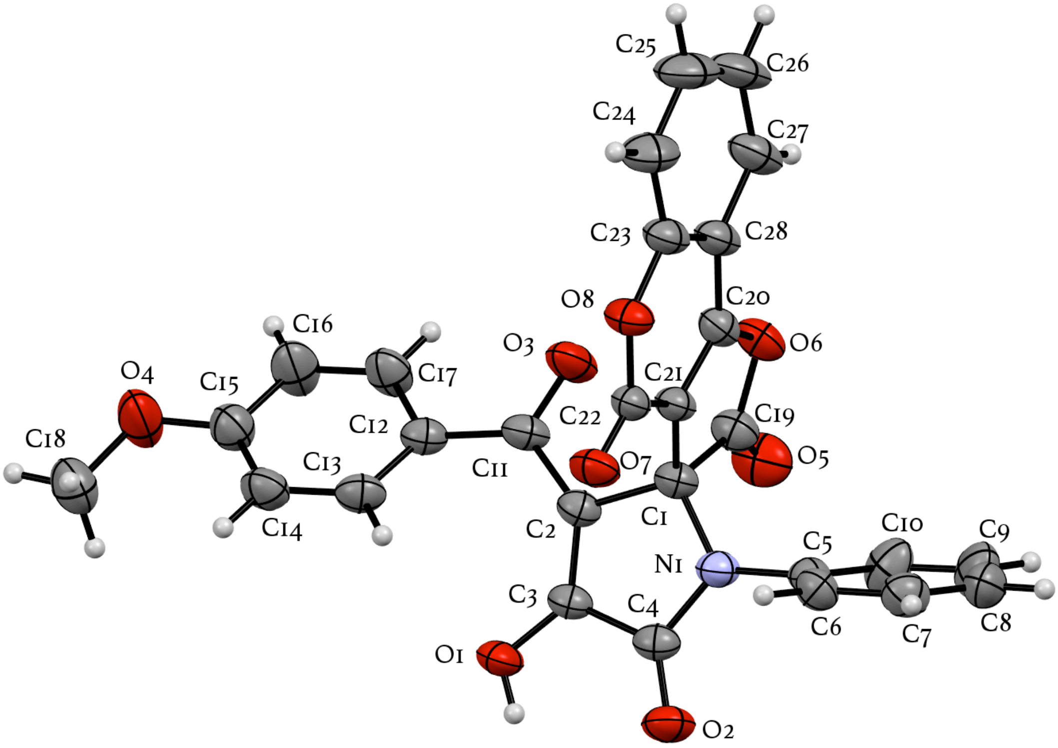


Figure 1. ORTEP drawing of compound **11ba**: showing 50% probability amplitude displacement ellipsoids.

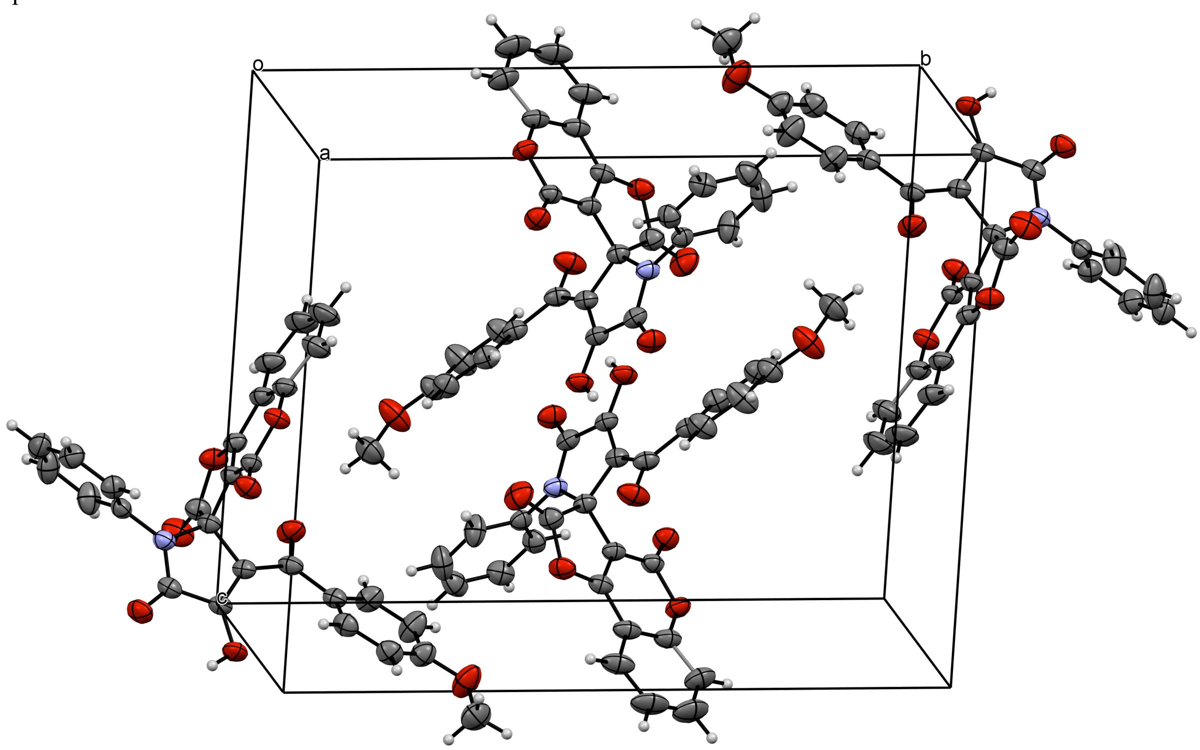


Figure 2. Packing diagram of the crystal structure of compound **11ba**.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) of compound 11ba.

	x	y	z	U_{iso}*/U_{eq}
H1	0.196 (3)	0.5217 (14)	0.5833 (18)	0.091 (8)*
O7	−0.10593 (14)	0.45365 (6)	0.30045 (7)	0.0452 (3)
O8	−0.05211 (13)	0.42250 (6)	0.16252 (7)	0.0422 (3)
O2	0.05126 (14)	0.61959 (6)	0.50532 (7)	0.0481 (3)
O1	0.24846 (15)	0.49477 (7)	0.54898 (7)	0.0454 (3)
O6	0.34408 (13)	0.55671 (6)	0.17134 (7)	0.0450 (3)
O3	0.45716 (15)	0.44537 (7)	0.28926 (7)	0.0541 (3)
N1	0.12561 (15)	0.59899 (7)	0.35966 (8)	0.0369 (3)
C20	0.21221 (17)	0.51171 (8)	0.16301 (10)	0.0361 (3)
C21	0.12847 (17)	0.50466 (8)	0.23821 (9)	0.0333 (3)
C22	−0.01469 (18)	0.46082 (8)	0.23954 (9)	0.0340 (3)
C11	0.39732 (18)	0.43620 (9)	0.36264 (10)	0.0378 (3)
C28	0.17419 (19)	0.47195 (9)	0.08319 (10)	0.0409 (4)
C3	0.22421 (17)	0.51860 (8)	0.46590 (9)	0.0342 (3)
C12	0.44289 (17)	0.37169 (8)	0.41705 (10)	0.0363 (3)
O5	0.46477 (15)	0.61610 (8)	0.28789 (9)	0.0622 (4)
C19	0.36013 (19)	0.57672 (9)	0.26213 (11)	0.0426 (4)
C4	0.12214 (17)	0.58522 (8)	0.44922 (10)	0.0366 (3)
C5	0.03917 (17)	0.65675 (8)	0.31350 (9)	0.0352 (3)
C13	0.34942 (18)	0.34214 (9)	0.48312 (10)	0.0424 (4)
H13	0.2535	0.3653	0.4976	0.051*
C23	0.0429 (2)	0.42445 (9)	0.08850 (10)	0.0417 (4)
C2	0.28003 (17)	0.49270 (8)	0.38895 (9)	0.0345 (3)
C1	0.21751 (17)	0.54313 (8)	0.31358 (9)	0.0344 (3)
C14	0.3962 (2)	0.27869 (9)	0.52793 (11)	0.0466 (4)
H14	0.3317	0.2593	0.5719	0.056*
O4	0.59913 (17)	0.18263 (8)	0.54856 (10)	0.0706 (4)
C16	0.6323 (2)	0.27271 (11)	0.44016 (13)	0.0597 (5)
H16	0.7280	0.2493	0.4256	0.072*
C17	0.58449 (19)	0.33476 (10)	0.39561 (12)	0.0490 (4)
H17	0.6472	0.3529	0.3501	0.059*
C7	−0.2082 (2)	0.70909 (9)	0.25652 (11)	0.0473 (4)
H7	−0.3202	0.7072	0.2488	0.057*
C6	−0.12738 (19)	0.65314 (9)	0.30158 (10)	0.0412 (4)
H6	−0.1843	0.6134	0.3237	0.049*
C27	0.2629 (2)	0.47271 (11)	0.00509 (11)	0.0533 (5)
H27	0.3494	0.5050	−0.0002	0.064*
C15	0.5388 (2)	0.24422 (10)	0.50730 (12)	0.0483 (4)
C24	0.0014 (2)	0.37674 (11)	0.01960 (12)	0.0583 (5)
H24	−0.0858	0.3448	0.0240	0.070*
C10	0.1236 (2)	0.71559 (9)	0.28022 (14)	0.0538 (5)
H10	0.2353	0.7182	0.2891	0.065*
C26	0.2214 (3)	0.42542 (14)	−0.06356 (11)	0.0661 (6)
H26	0.2803	0.4256	−0.1154	0.079*
C8	−0.1239 (2)	0.76766 (10)	0.22287 (12)	0.0537 (4)
H8	−0.1791	0.8053	0.1927	0.064*
C18	0.5252 (3)	0.15709 (12)	0.62683 (14)	0.0651 (5)
H18A	0.4157	0.1427	0.6123	0.098*
H18B	0.5254	0.1957	0.6706	0.098*
H18C	0.5843	0.1156	0.6505	0.098*
C9	0.0418 (2)	0.77058 (10)	0.23375 (14)	0.0613 (5)
H9	0.0988	0.8096	0.2098	0.074*
C25	0.0928 (3)	0.37773 (13)	−0.05596 (13)	0.0692 (6)
H25	0.0672	0.3456	−0.1027	0.083*

Geometric parameters (Å, º) of compound 11ba.

O7—C22	1.2094 (17)	C23—C24	1.381 (2)
O8—C22	1.3722 (17)	C2—C1	1.529 (2)
O8—C23	1.3793 (18)	C14—C15	1.379 (2)
O2—C4	1.2128 (17)	C14—H14	0.9300
O1—C3	1.3245 (18)	O4—C15	1.364 (2)
O1—H1	0.84 (3)	O4—C18	1.417 (2)
O6—C20	1.3650 (19)	C16—C17	1.361 (2)
O6—C19	1.4076 (19)	C16—C15	1.388 (2)
O3—C11	1.2304 (18)	C16—H16	0.9300
N1—C4	1.3638 (19)	C17—H17	0.9300
N1—C5	1.4334 (19)	C7—C8	1.377 (3)
N1—C1	1.4554 (19)	C7—C6	1.379 (2)
C20—C21	1.3467 (19)	C7—H7	0.9300
C20—C28	1.421 (2)	C6—H6	0.9300
C21—C22	1.427 (2)	C27—C26	1.373 (3)
C21—C1	1.498 (2)	C27—H27	0.9300
C11—C12	1.469 (2)	C24—C25	1.381 (3)
C11—C2	1.475 (2)	C24—H24	0.9300
C28—C23	1.391 (2)	C10—C9	1.380 (3)
C28—C27	1.401 (2)	C10—H10	0.9300
C3—C2	1.340 (2)	C26—C25	1.379 (3)
C3—C4	1.492 (2)	C26—H26	0.9300
C12—C13	1.383 (2)	C8—C9	1.375 (3)
C12—C17	1.396 (2)	C8—H8	0.9300
O5—C19	1.1772 (19)	C18—H18A	0.9600
C19—C1	1.555 (2)	C18—H18B	0.9600
C5—C10	1.379 (2)	C18—H18C	0.9600
C5—C6	1.384 (2)	C9—H9	0.9300
C13—C14	1.382 (2)	C25—H25	0.9300
C13—H13	0.9300		
C22—O8—C23	122.85 (12)	C21—C1—C19	99.92 (11)
C3—O1—H1	108.6 (18)	C2—C1—C19	110.93 (12)
C20—O6—C19	106.94 (11)	C15—C14—C13	119.84 (15)
C4—N1—C5	125.47 (12)	C15—C14—H14	120.1
C4—N1—C1	111.47 (12)	C13—C14—H14	120.1
C5—N1—C1	122.84 (12)	C15—O4—C18	118.75 (15)
C21—C20—O6	114.36 (13)	C17—C16—C15	120.21 (16)
C21—C20—C28	123.22 (14)	C17—C16—H16	119.9
O6—C20—C28	122.28 (13)	C15—C16—H16	119.9
C20—C21—C22	120.99 (13)	C16—C17—C12	121.16 (16)
C20—C21—C1	109.30 (13)	C16—C17—H17	119.4
C22—C21—C1	129.51 (13)	C12—C17—H17	119.4
O7—C22—O8	116.68 (13)	C8—C7—C6	120.37 (16)
O7—C22—C21	127.55 (14)	C8—C7—H7	119.8
O8—C22—C21	115.76 (12)	C6—C7—H7	119.8
O3—C11—C12	119.93 (13)	C7—C6—C5	119.40 (15)
O3—C11—C2	115.58 (14)	C7—C6—H6	120.3
C12—C11—C2	124.49 (13)	C5—C6—H6	120.3
C23—C28—C27	119.07 (15)	C26—C27—C28	119.50 (19)
C23—C28—C20	114.70 (13)	C26—C27—H27	120.2
C27—C28—C20	126.04 (16)	C28—C27—H27	120.2
O1—C3—C2	130.20 (14)	O4—C15—C14	124.98 (16)
O1—C3—C4	119.25 (13)	O4—C15—C16	115.43 (15)
C2—C3—C4	110.54 (13)	C14—C15—C16	119.59 (16)
C13—C12—C17	118.08 (14)	C23—C24—C25	118.28 (19)
C13—C12—C11	124.51 (13)	C23—C24—H24	120.9

C17—C12—C11	117.23 (14)	C25—C24—H24	120.9
O5—C19—O6	121.11 (15)	C5—C10—C9	119.84 (16)
O5—C19—C1	129.64 (15)	C5—C10—H10	120.1
O6—C19—C1	109.14 (12)	C9—C10—H10	120.1
O2—C4—N1	127.72 (14)	C27—C26—C25	120.35 (18)
O2—C4—C3	125.89 (14)	C27—C26—H26	119.8
N1—C4—C3	106.40 (12)	C25—C26—H26	119.8
C10—C5—C6	120.25 (15)	C9—C8—C7	120.08 (16)
C10—C5—N1	119.42 (14)	C9—C8—H8	120.0
C6—C5—N1	120.32 (13)	C7—C8—H8	120.0
C14—C13—C12	121.07 (14)	O4—C18—H18A	109.5
C14—C13—H13	119.5	O4—C18—H18B	109.5
C12—C13—H13	119.5	H18A—C18—H18B	109.5
O8—C23—C24	116.70 (16)	O4—C18—H18C	109.5
O8—C23—C28	121.91 (14)	H18A—C18—H18C	109.5
C24—C23—C28	121.40 (16)	H18B—C18—H18C	109.5
C3—C2—C11	136.27 (14)	C8—C9—C10	120.03 (17)
C3—C2—C1	107.76 (12)	C8—C9—H9	120.0
C11—C2—C1	115.42 (12)	C10—C9—H9	120.0
N1—C1—C21	115.51 (12)	C26—C25—C24	121.37 (18)
N1—C1—C2	103.75 (11)	C26—C25—H25	119.3
C21—C1—C2	114.84 (12)	C24—C25—H25	119.3
N1—C1—C19	112.18 (12)		
C19—O6—C20—C21	5.75 (18)	C4—N1—C1—C21	123.98 (14)
C19—O6—C20—C28	−170.13 (14)	C5—N1—C1—C21	−50.89 (19)
O6—C20—C21—C22	178.51 (13)	C4—N1—C1—C2	−2.60 (15)
C28—C20—C21—C22	−5.7 (2)	C5—N1—C1—C2	−177.47 (12)
O6—C20—C21—C1	−6.12 (18)	C4—N1—C1—C19	−122.40 (14)
C28—C20—C21—C1	169.72 (13)	C5—N1—C1—C19	62.73 (17)
C23—O8—C22—O7	−178.72 (13)	C20—C21—C1—N1	124.17 (14)
C23—O8—C22—C21	1.22 (19)	C22—C21—C1—N1	−61.0 (2)
C20—C21—C22—O7	−175.00 (15)	C20—C21—C1—C2	−115.09 (14)
C1—C21—C22—O7	10.7 (3)	C22—C21—C1—C2	59.76 (19)
C20—C21—C22—O8	5.1 (2)	C20—C21—C1—C19	3.63 (15)
C1—C21—C22—O8	−169.28 (13)	C22—C21—C1—C19	178.49 (15)
C21—C20—C28—C23	−0.2 (2)	C3—C2—C1—N1	1.07 (15)
O6—C20—C28—C23	175.32 (14)	C11—C2—C1—N1	−171.82 (12)
C21—C20—C28—C27	−175.11 (16)	C3—C2—C1—C21	−125.93 (13)
O6—C20—C28—C27	0.4 (2)	C11—C2—C1—C21	61.18 (17)
O3—C11—C12—C13	−157.20 (15)	C3—C2—C1—C19	121.71 (14)
C2—C11—C12—C13	22.0 (2)	C11—C2—C1—C19	−51.18 (17)
O3—C11—C12—C17	17.8 (2)	O5—C19—C1—N1	52.9 (2)
C2—C11—C12—C17	−163.00 (15)	O6—C19—C1—N1	−123.25 (13)
C20—O6—C19—O5	−179.54 (16)	O5—C19—C1—C21	175.83 (19)
C20—O6—C19—C1	−2.99 (16)	O6—C19—C1—C21	−0.34 (15)
C5—N1—C4—O2	−2.4 (2)	O5—C19—C1—C2	−62.6 (2)
C1—N1—C4—O2	−177.07 (14)	O6—C19—C1—C2	121.24 (13)
C5—N1—C4—C3	177.73 (13)	C12—C13—C14—C15	0.5 (2)
C1—N1—C4—C3	3.03 (16)	C15—C16—C17—C12	1.0 (3)
O1—C3—C4—O2	−3.1 (2)	C13—C12—C17—C16	−2.0 (3)
C2—C3—C4—O2	177.78 (15)	C11—C12—C17—C16	−177.31 (17)
O1—C3—C4—N1	176.81 (13)	C8—C7—C6—C5	0.7 (2)
C2—C3—C4—N1	−2.31 (17)	C10—C5—C6—C7	−0.3 (2)
C4—N1—C5—C10	109.74 (18)	N1—C5—C6—C7	−179.62 (14)
C1—N1—C5—C10	−76.12 (19)	C23—C28—C27—C26	−1.7 (2)
C4—N1—C5—C6	−70.96 (19)	C20—C28—C27—C26	173.04 (16)
C1—N1—C5—C6	103.18 (17)	C18—O4—C15—C14	−9.7 (3)

C17—C12—C13—C14	1.3 (2)	C18—O4—C15—C16	170.01 (18)
C11—C12—C13—C14	176.18 (15)	C13—C14—C15—O4	178.21 (16)
C22—O8—C23—C24	172.65 (14)	C13—C14—C15—C16	−1.5 (3)
C22—O8—C23—C28	−7.3 (2)	C17—C16—C15—O4	−178.98 (18)
C27—C28—C23—O8	−178.18 (14)	C17—C16—C15—C14	0.7 (3)
C20—C28—C23—O8	6.5 (2)	O8—C23—C24—C25	179.33 (16)
C27—C28—C23—C24	1.9 (2)	C28—C23—C24—C25	−0.8 (3)
C20—C28—C23—C24	−173.38 (15)	C6—C5—C10—C9	−0.9 (3)
O1—C3—C2—C11	−7.6 (3)	N1—C5—C10—C9	178.40 (16)
C4—C3—C2—C11	171.38 (16)	C28—C27—C26—C25	0.3 (3)
O1—C3—C2—C1	−178.31 (15)	C6—C7—C8—C9	0.2 (3)
C4—C3—C2—C1	0.68 (16)	C7—C8—C9—C10	−1.5 (3)
O3—C11—C2—C3	−155.49 (18)	C5—C10—C9—C8	1.8 (3)
C12—C11—C2—C3	25.3 (3)	C27—C26—C25—C24	0.9 (3)
O3—C11—C2—C1	14.7 (2)	C23—C24—C25—C26	−0.7 (3)
C12—C11—C2—C1	−164.56 (13)		

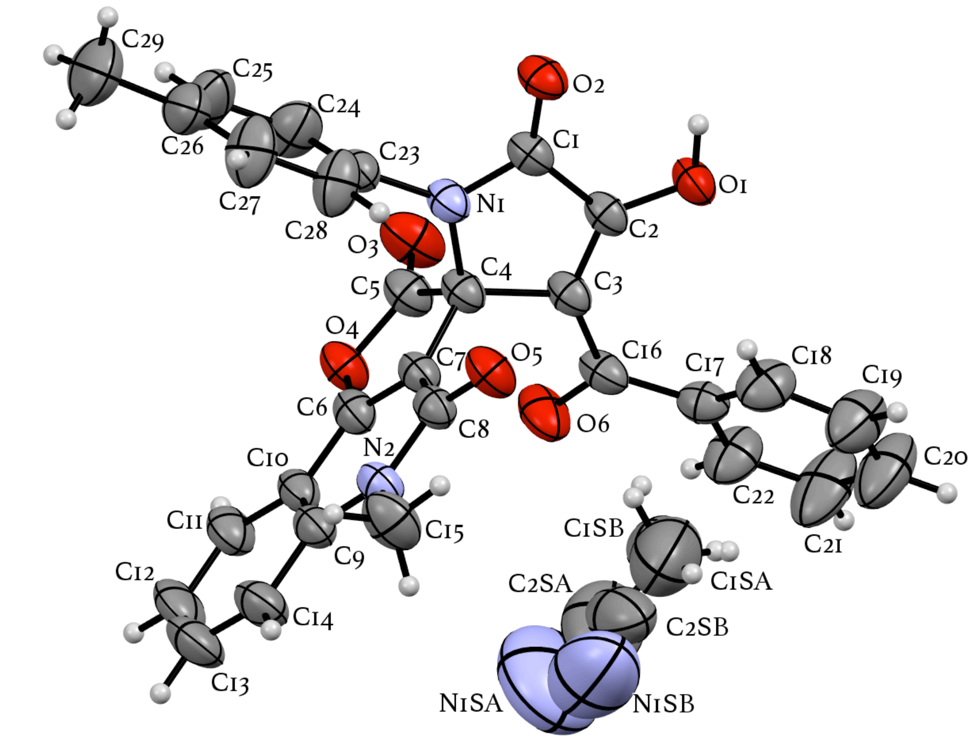


Figure 3. ORTEP drawing of compound **11db**: showing 50% probability amplitude displacement ellipsoids and a molecule of disordered crystallized solvent (acetonitrile).

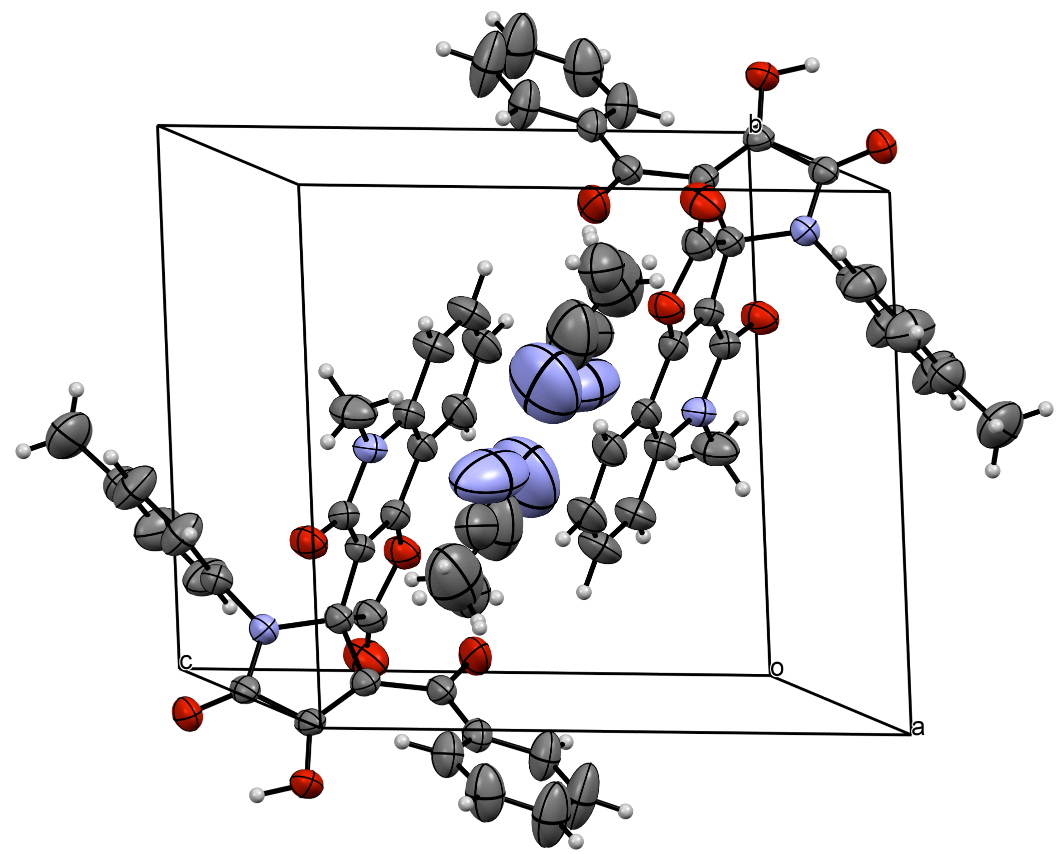


Figure 4. Packing diagram of the crystal structure of compound **11db**.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) of compound 11db.

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.50061 (12)	1.15678 (9)	0.09013 (9)	0.0486 (3)	
H1	0.533 (2)	1.182 (2)	0.013 (2)	0.097 (7)*	
O2	0.72395 (11)	1.05413 (10)	−0.05438 (8)	0.0482 (3)	
O3	0.81103 (15)	0.95631 (12)	0.27246 (11)	0.0694 (4)	
O4	0.73560 (12)	0.75301 (10)	0.32355 (9)	0.0518 (3)	
O5	0.45732 (12)	0.70715 (10)	0.10042 (9)	0.0535 (3)	
O6	0.49575 (15)	0.91692 (12)	0.38310 (9)	0.0690 (4)	
N1	0.72537 (13)	0.89679 (11)	0.08132 (10)	0.0426 (3)	
N2	0.47932 (14)	0.53482 (11)	0.21563 (11)	0.0464 (3)	
C1	0.67953 (15)	1.00324 (12)	0.03470 (11)	0.0373 (3)	
C2	0.56132 (15)	1.04937 (12)	0.11477 (11)	0.0377 (3)	
C3	0.53799 (16)	0.97166 (13)	0.20524 (11)	0.0399 (3)	
C4	0.64445 (16)	0.86681 (13)	0.19016 (11)	0.0408 (3)	
C5	0.73900 (18)	0.87173 (15)	0.26519 (13)	0.0498 (4)	
C6	0.64751 (16)	0.67488 (14)	0.29355 (12)	0.0429 (3)	
C7	0.59258 (15)	0.73116 (13)	0.21810 (11)	0.0407 (3)	
C8	0.50577 (16)	0.66173 (13)	0.17293 (12)	0.0416 (3)	
C9	0.53282 (16)	0.47705 (14)	0.29613 (12)	0.0466 (4)	
C10	0.62100 (17)	0.54568 (14)	0.33749 (12)	0.0461 (4)	
C11	0.67841 (19)	0.48668 (17)	0.41599 (13)	0.0577 (4)	
H11	0.7363	0.5328	0.4433	0.069*	
C12	0.6480 (2)	0.36024 (19)	0.45163 (16)	0.0724 (6)	
H12	0.6862	0.3197	0.5030	0.087*	
C13	0.5606 (2)	0.29268 (18)	0.41143 (17)	0.0758 (6)	
H13	0.5404	0.2070	0.4368	0.091*	
C14	0.5033 (2)	0.34836 (16)	0.33549 (16)	0.0647 (5)	
H14	0.4447	0.3008	0.3099	0.078*	
C15	0.3942 (2)	0.45865 (17)	0.17008 (18)	0.0702 (6)	
H15A	0.3154	0.4237	0.2240	0.105*	
H15B	0.4475	0.3903	0.1390	0.105*	
H15C	0.3638	0.5120	0.1173	0.105*	
C16	0.45246 (17)	0.97585 (13)	0.31357 (12)	0.0459 (4)	
C17	0.32096 (17)	1.04441 (14)	0.34005 (12)	0.0460 (4)	
C18	0.24201 (19)	1.07195 (19)	0.26998 (14)	0.0626 (5)	
H18	0.2743	1.0527	0.2014	0.075*	
C19	0.1153 (2)	1.1280 (3)	0.30147 (18)	0.0868 (7)	
H19	0.0606	1.1429	0.2551	0.104*	
C20	0.0701 (3)	1.1618 (3)	0.4012 (2)	0.1093 (10)	
H20	−0.0138	1.2023	0.4215	0.131*	
C21	0.1489 (3)	1.1359 (3)	0.47106 (19)	0.1069 (10)	
H21	0.1184	1.1596	0.5384	0.128*	
C22	0.2719 (2)	1.0754 (2)	0.44179 (14)	0.0681 (5)	
H22	0.3229	1.0550	0.4902	0.082*	
C23	0.83317 (16)	0.81754 (14)	0.02992 (12)	0.0437 (4)	
C24	0.96404 (19)	0.82697 (18)	0.04263 (18)	0.0683 (5)	
H24	0.9833	0.8840	0.0856	0.082*	
C25	1.06677 (19)	0.7511 (2)	−0.0089 (2)	0.0797 (7)	
H25	1.1547	0.7570	0.0007	0.096*	
C26	1.0423 (2)	0.66723 (18)	−0.07409 (17)	0.0658 (5)	
C27	0.9108 (2)	0.6582 (2)	−0.08420 (18)	0.0756 (6)	
H27	0.8915	0.6010	−0.1270	0.091*	
C28	0.8059 (2)	0.73184 (19)	−0.03245 (15)	0.0647 (5)	
H28	0.7171	0.7232	−0.0400	0.078*	

C29	1.1561 (3)	0.5895 (3)	−0.1345 (2)	0.1014 (9)	
H29A	1.1789	0.6221	−0.2075	0.152*	
H29B	1.1257	0.5018	−0.1259	0.152*	
H29C	1.2361	0.5952	−0.1086	0.152*	
N1SA	0.1293 (15)	0.5474 (13)	0.3909 (9)	0.256 (6)	0.5
C1SA	0.1077 (18)	0.7308 (14)	0.2621 (13)	0.165 (7)	0.5
H1SA	0.0398	0.7051	0.2291	0.248*	0.5
H1SB	0.1951	0.7451	0.2107	0.248*	0.5
H1SC	0.0798	0.8083	0.2925	0.248*	0.5
C2SA	0.120 (2)	0.6348 (15)	0.3403 (11)	0.159 (5)	0.5
N1SB	0.0491 (8)	0.5372 (7)	0.3141 (10)	0.204 (4)	0.5
C1SB	0.1412 (15)	0.7627 (9)	0.2911 (10)	0.101 (3)	0.5
H1SD	0.1791	0.7757	0.3482	0.151*	0.5
H1SE	0.0652	0.8188	0.2913	0.151*	0.5
H1SF	0.2110	0.7809	0.2260	0.151*	0.5
C2SB	0.0949 (12)	0.6357 (8)	0.3021 (9)	0.118 (4)	0.5

Geometric parameters (Å, º) of compound 11db.

O1—H1	1.00 (3)	C16—C17	1.479 (2)
O1—C2	1.3290 (17)	C17—C18	1.382 (2)
O2—C1	1.2126 (17)	C17—C22	1.390 (2)
O3—C5	1.181 (2)	C18—H18	0.9300
O4—C5	1.3889 (18)	C18—C19	1.381 (3)
O4—C6	1.3768 (19)	C19—H19	0.9300
O5—C8	1.2393 (18)	C19—C20	1.372 (4)
O6—C16	1.2276 (18)	C20—H20	0.9300
N1—C1	1.3544 (18)	C20—C21	1.376 (4)
N1—C4	1.4616 (19)	C21—H21	0.9300
N1—C23	1.4359 (19)	C21—C22	1.369 (3)
N2—C8	1.3890 (18)	C22—H22	0.9300
N2—C9	1.399 (2)	C23—C24	1.375 (2)
N2—C15	1.464 (2)	C23—C28	1.370 (2)
C1—C2	1.491 (2)	C24—H24	0.9300
C2—C3	1.3421 (19)	C24—C25	1.383 (3)
C3—C4	1.523 (2)	C25—H25	0.9300
C3—C16	1.476 (2)	C25—C26	1.373 (3)
C4—C5	1.555 (2)	C26—C27	1.368 (3)
C4—C7	1.4957 (19)	C26—C29	1.508 (3)
C6—C7	1.347 (2)	C27—H27	0.9300
C6—C10	1.415 (2)	C27—C28	1.383 (3)
C7—C8	1.431 (2)	C28—H28	0.9300
C9—C10	1.404 (2)	C29—H29A	0.9600
C9—C14	1.399 (2)	C29—H29B	0.9600
C10—C11	1.405 (2)	C29—H29C	0.9600
C11—H11	0.9300	N1SA—C2SA	1.094 (12)
C11—C12	1.369 (3)	C1SA—H1SA	0.9600
C12—H12	0.9300	C1SA—H1SB	0.9600
C12—C13	1.382 (3)	C1SA—H1SC	0.9600
C13—H13	0.9300	C1SA—C2SA	1.394 (13)
C13—C14	1.365 (3)	N1SB—C2SB	1.116 (9)
C14—H14	0.9300	C1SB—H1SD	0.9600
C15—H15A	0.9600	C1SB—H1SE	0.9600
C15—H15B	0.9600	C1SB—H1SF	0.9600
C15—H15C	0.9600	C1SB—C2SB	1.398 (10)
C2—O1—H1	109.0 (14)	O6—C16—C3	116.05 (15)
C6—O4—C5	107.13 (11)	O6—C16—C17	120.35 (14)

C1—N1—C4	111.27 (12)	C3—C16—C17	123.59 (13)
C1—N1—C23	125.09 (12)	C18—C17—C16	122.78 (15)
C23—N1—C4	123.52 (11)	C18—C17—C22	119.13 (17)
C8—N2—C9	123.56 (14)	C22—C17—C16	117.99 (15)
C8—N2—C15	116.93 (13)	C17—C18—H18	119.9
C9—N2—C15	119.48 (13)	C19—C18—C17	120.19 (18)
O2—C1—N1	128.06 (14)	C19—C18—H18	119.9
O2—C1—C2	125.13 (13)	C18—C19—H19	120.0
N1—C1—C2	106.81 (12)	C20—C19—C18	120.0 (2)
O1—C2—C1	119.02 (12)	C20—C19—H19	120.0
O1—C2—C3	130.60 (15)	C19—C20—H20	120.0
C3—C2—C1	110.33 (12)	C19—C20—C21	120.1 (2)
C2—C3—C4	107.76 (13)	C21—C20—H20	120.0
C2—C3—C16	135.59 (14)	C20—C21—H21	119.9
C16—C3—C4	115.85 (12)	C22—C21—C20	120.2 (2)
N1—C4—C3	103.82 (11)	C22—C21—H21	119.9
N1—C4—C5	110.02 (12)	C17—C22—H22	119.9
N1—C4—C7	114.09 (12)	C21—C22—C17	120.28 (19)
C3—C4—C5	111.11 (12)	C21—C22—H22	119.9
C7—C4—C3	117.57 (13)	C24—C23—N1	120.52 (15)
C7—C4—C5	100.32 (11)	C28—C23—N1	119.91 (14)
O3—C5—O4	121.49 (15)	C28—C23—C24	119.57 (16)
O3—C5—C4	129.07 (14)	C23—C24—H24	120.2
O4—C5—C4	109.34 (13)	C23—C24—C25	119.52 (19)
O4—C6—C10	121.97 (13)	C25—C24—H24	120.2
C7—C6—O4	114.32 (12)	C24—C25—H25	119.1
C7—C6—C10	123.70 (15)	C26—C25—C24	121.73 (19)
C6—C7—C4	108.88 (13)	C26—C25—H25	119.1
C6—C7—C8	121.78 (13)	C25—C26—C29	121.6 (2)
C8—C7—C4	129.25 (12)	C27—C26—C25	117.64 (18)
O5—C8—N2	120.68 (14)	C27—C26—C29	120.7 (2)
O5—C8—C7	124.49 (13)	C26—C27—H27	119.1
N2—C8—C7	114.82 (13)	C26—C27—C28	121.7 (2)
N2—C9—C10	120.83 (13)	C28—C27—H27	119.1
N2—C9—C14	120.97 (16)	C23—C28—C27	119.78 (18)
C14—C9—C10	118.18 (15)	C23—C28—H28	120.1
C9—C10—C6	115.26 (14)	C27—C28—H28	120.1
C9—C10—C11	120.93 (14)	C26—C29—H29A	109.5
C11—C10—C6	123.81 (16)	C26—C29—H29B	109.5
C10—C11—H11	120.5	C26—C29—H29C	109.5
C12—C11—C10	119.05 (19)	H29A—C29—H29B	109.5
C12—C11—H11	120.5	H29A—C29—H29C	109.5
C11—C12—H12	119.9	H29B—C29—H29C	109.5
C11—C12—C13	120.12 (17)	H1SA—C1SA—H1SB	109.5
C13—C12—H12	119.9	H1SA—C1SA—H1SC	109.5
C12—C13—H13	119.1	H1SB—C1SA—H1SC	109.5
C14—C13—C12	121.73 (17)	C2SA—C1SA—H1SA	109.5
C14—C13—H13	119.1	C2SA—C1SA—H1SB	109.5
C9—C14—H14	120.0	C2SA—C1SA—H1SC	109.5
C13—C14—C9	120.0 (2)	N1SA—C2SA—C1SA	169.3 (19)
C13—C14—H14	120.0	H1SD—C1SB—H1SE	109.5
N2—C15—H15A	109.5	H1SD—C1SB—H1SF	109.5
N2—C15—H15B	109.5	H1SE—C1SB—H1SF	109.5
N2—C15—H15C	109.5	C2SB—C1SB—H1SD	109.5
H15A—C15—H15B	109.5	C2SB—C1SB—H1SE	109.5
H15A—C15—H15C	109.5	C2SB—C1SB—H1SF	109.5
H15B—C15—H15C	109.5	N1SB—C2SB—C1SB	175.2 (13)

O1—C2—C3—C4	−176.63 (14)	C6—O4—C5—O3	−177.17 (17)
O1—C2—C3—C16	−7.8 (3)	C6—O4—C5—C4	−0.66 (17)
O2—C1—C2—O1	−2.4 (2)	C6—C7—C8—O5	−176.82 (15)
O2—C1—C2—C3	179.86 (14)	C6—C7—C8—N2	2.3 (2)
O4—C6—C7—C4	−0.86 (18)	C6—C10—C11—C12	−178.74 (17)
O4—C6—C7—C8	175.99 (13)	C7—C4—C5—O3	176.35 (19)
O4—C6—C10—C9	−177.75 (14)	C7—C4—C5—O4	0.18 (16)
O4—C6—C10—C11	1.4 (2)	C7—C6—C10—C9	0.6 (2)
O6—C16—C17—C18	−155.89 (17)	C7—C6—C10—C11	179.83 (15)
O6—C16—C17—C22	20.3 (2)	C8—N2—C9—C10	−1.4 (2)
N1—C1—C2—O1	177.24 (12)	C8—N2—C9—C14	−179.51 (15)
N1—C1—C2—C3	−0.49 (16)	C9—N2—C8—O5	178.78 (14)
N1—C4—C5—O3	55.8 (2)	C9—N2—C8—C7	−0.3 (2)
N1—C4—C5—O4	−120.34 (13)	C9—C10—C11—C12	0.4 (3)
N1—C4—C7—C6	117.93 (14)	C10—C6—C7—C4	−179.36 (14)
N1—C4—C7—C8	−58.6 (2)	C10—C6—C7—C8	−2.5 (2)
N1—C23—C24—C25	−179.00 (17)	C10—C9—C14—C13	−0.5 (3)
N1—C23—C28—C27	178.20 (17)	C10—C11—C12—C13	−0.8 (3)
N2—C9—C10—C6	1.3 (2)	C11—C12—C13—C14	0.5 (3)
N2—C9—C10—C11	−177.92 (14)	C12—C13—C14—C9	0.1 (3)
N2—C9—C14—C13	177.65 (17)	C14—C9—C10—C6	179.42 (15)
C1—N1—C4—C3	0.44 (15)	C14—C9—C10—C11	0.2 (2)
C1—N1—C4—C5	−118.54 (13)	C15—N2—C8—O5	0.8 (2)
C1—N1—C4—C7	129.64 (13)	C15—N2—C8—C7	−178.32 (15)
C1—N1—C23—C24	101.64 (19)	C15—N2—C9—C10	176.49 (16)
C1—N1—C23—C28	−78.3 (2)	C15—N2—C9—C14	−1.6 (2)
C1—C2—C3—C4	0.75 (16)	C16—C3—C4—N1	−172.08 (12)
C1—C2—C3—C16	169.60 (16)	C16—C3—C4—C5	−53.86 (17)
C2—C3—C4—N1	−0.73 (15)	C16—C3—C4—C7	60.87 (18)
C2—C3—C4—C5	117.49 (13)	C16—C17—C18—C19	175.34 (18)
C2—C3—C4—C7	−127.79 (14)	C16—C17—C22—C21	−178.3 (2)
C2—C3—C16—O6	−151.39 (18)	C17—C18—C19—C20	2.9 (4)
C2—C3—C16—C17	29.4 (3)	C18—C17—C22—C21	−1.9 (3)
C3—C4—C5—O3	−58.6 (2)	C18—C19—C20—C21	−2.2 (5)
C3—C4—C5—O4	125.25 (14)	C19—C20—C21—C22	−0.6 (5)
C3—C4—C7—C6	−120.16 (14)	C20—C21—C22—C17	2.6 (4)
C3—C4—C7—C8	63.3 (2)	C22—C17—C18—C19	−0.9 (3)
C3—C16—C17—C18	23.3 (2)	C23—N1—C1—O2	−4.3 (2)
C3—C16—C17—C22	−160.45 (16)	C23—N1—C1—C2	176.08 (13)
C4—N1—C1—O2	179.62 (14)	C23—N1—C4—C3	−175.73 (13)
C4—N1—C1—C2	−0.01 (16)	C23—N1—C4—C5	65.30 (17)
C4—N1—C23—C24	−82.7 (2)	C23—N1—C4—C7	−46.52 (19)
C4—N1—C23—C28	97.34 (19)	C23—C24—C25—C26	0.9 (3)
C4—C3—C16—O6	16.8 (2)	C24—C23—C28—C27	−1.7 (3)
C4—C3—C16—C17	−162.45 (13)	C24—C25—C26—C27	−1.9 (3)
C4—C7—C8—O5	−0.7 (3)	C24—C25—C26—C29	176.8 (2)
C4—C7—C8—N2	178.42 (14)	C25—C26—C27—C28	1.0 (3)
C5—O4—C6—C7	0.97 (18)	C26—C27—C28—C23	0.7 (3)
C5—O4—C6—C10	179.50 (14)	C28—C23—C24—C25	0.9 (3)
C5—C4—C7—C6	0.38 (16)	C29—C26—C27—C28	−177.7 (2)
C5—C4—C7—C8	−176.16 (15)		