Supplementary Material

Acid-catalyzed reaction of 1-(2,2-dimethoxyethyl)ureas with phenols as an effective approach to diarylethanes and dibenzoxanthenes

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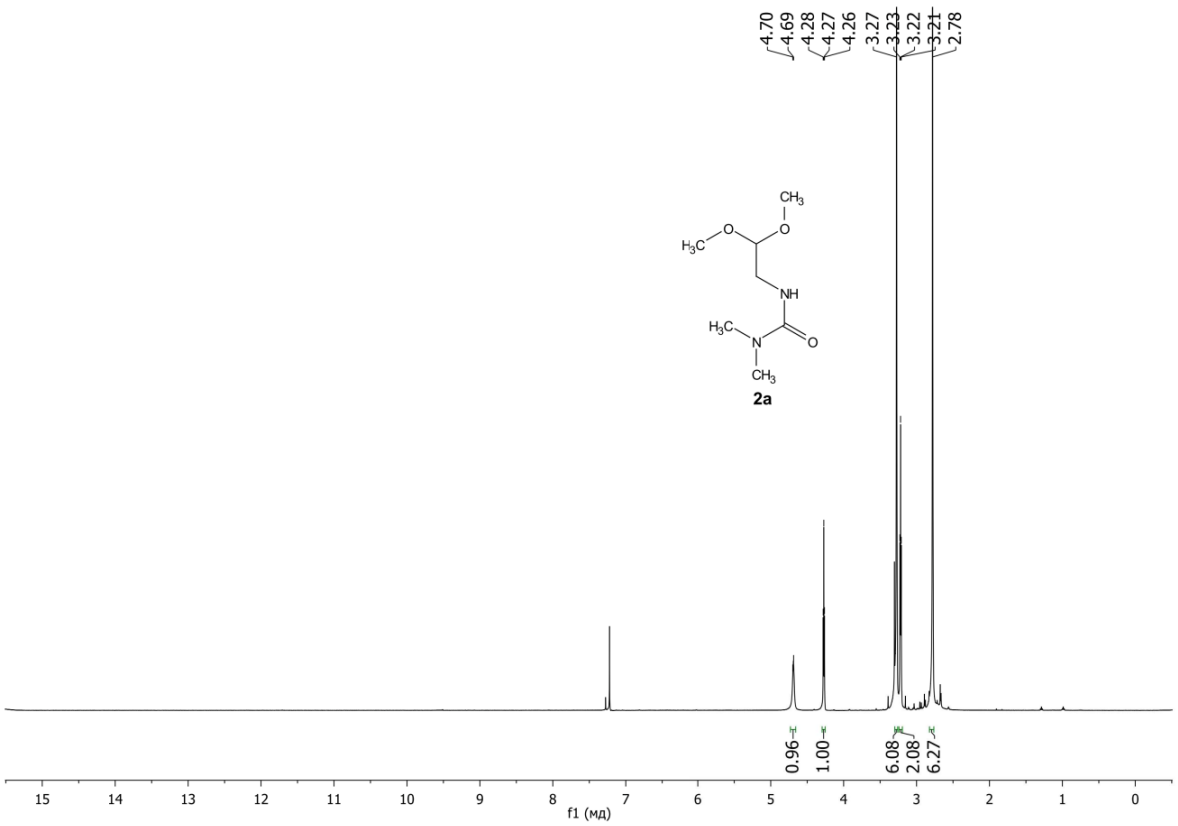

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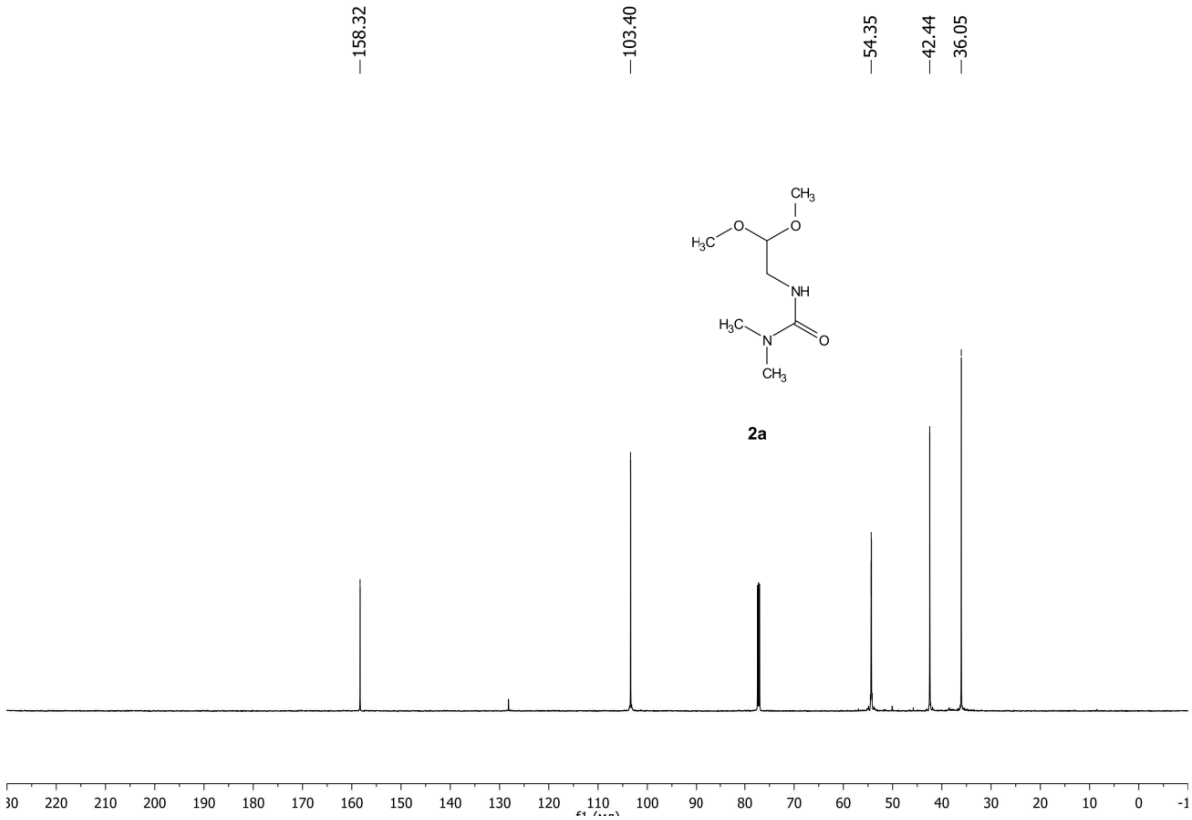

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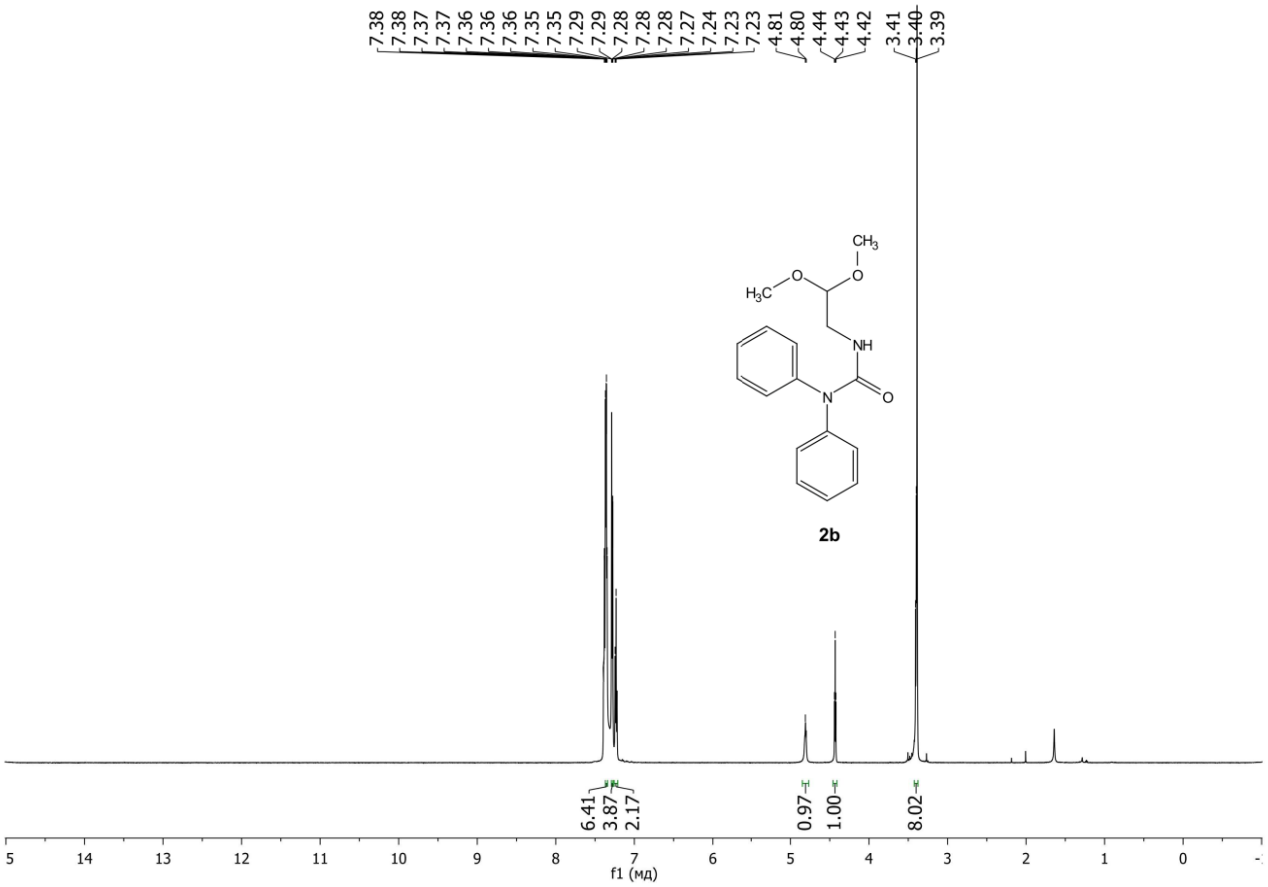

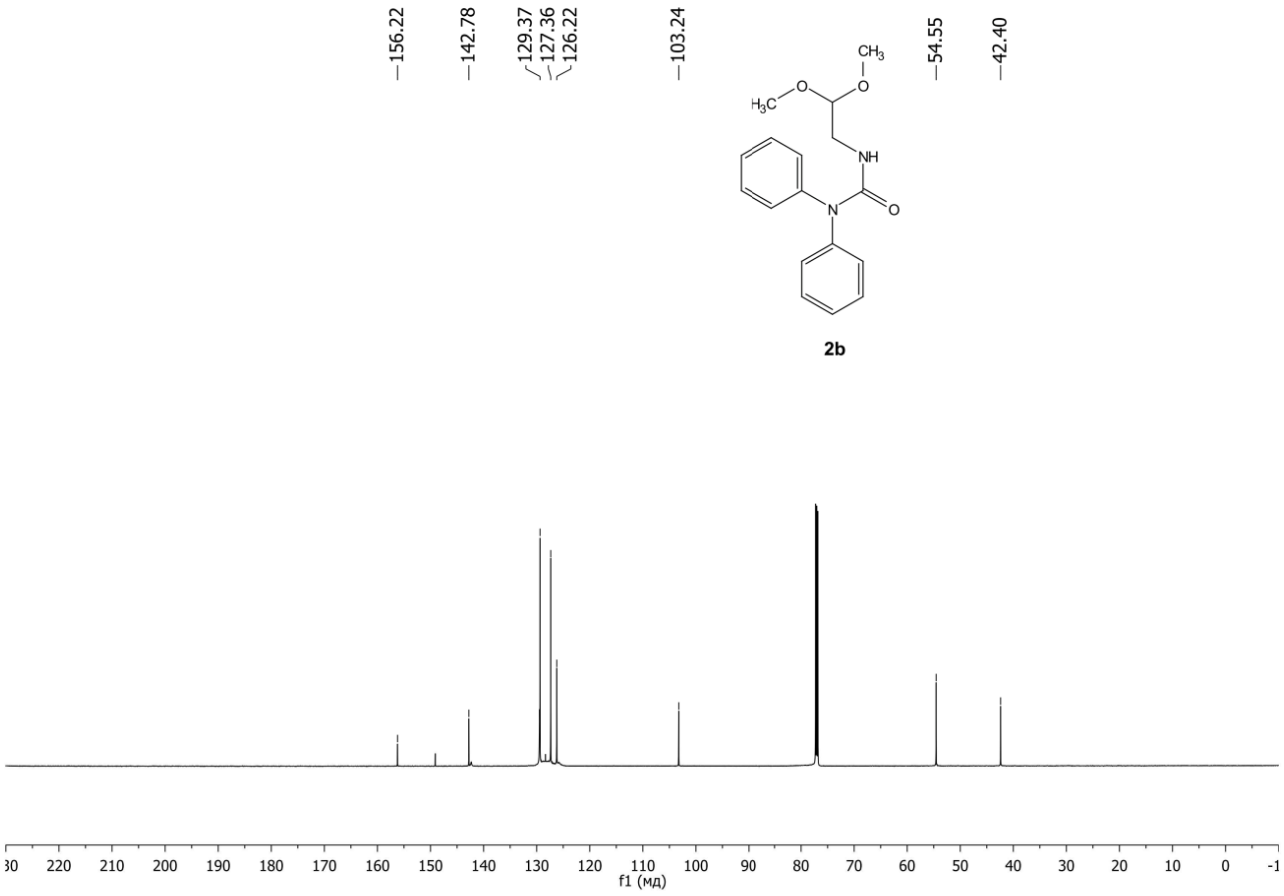

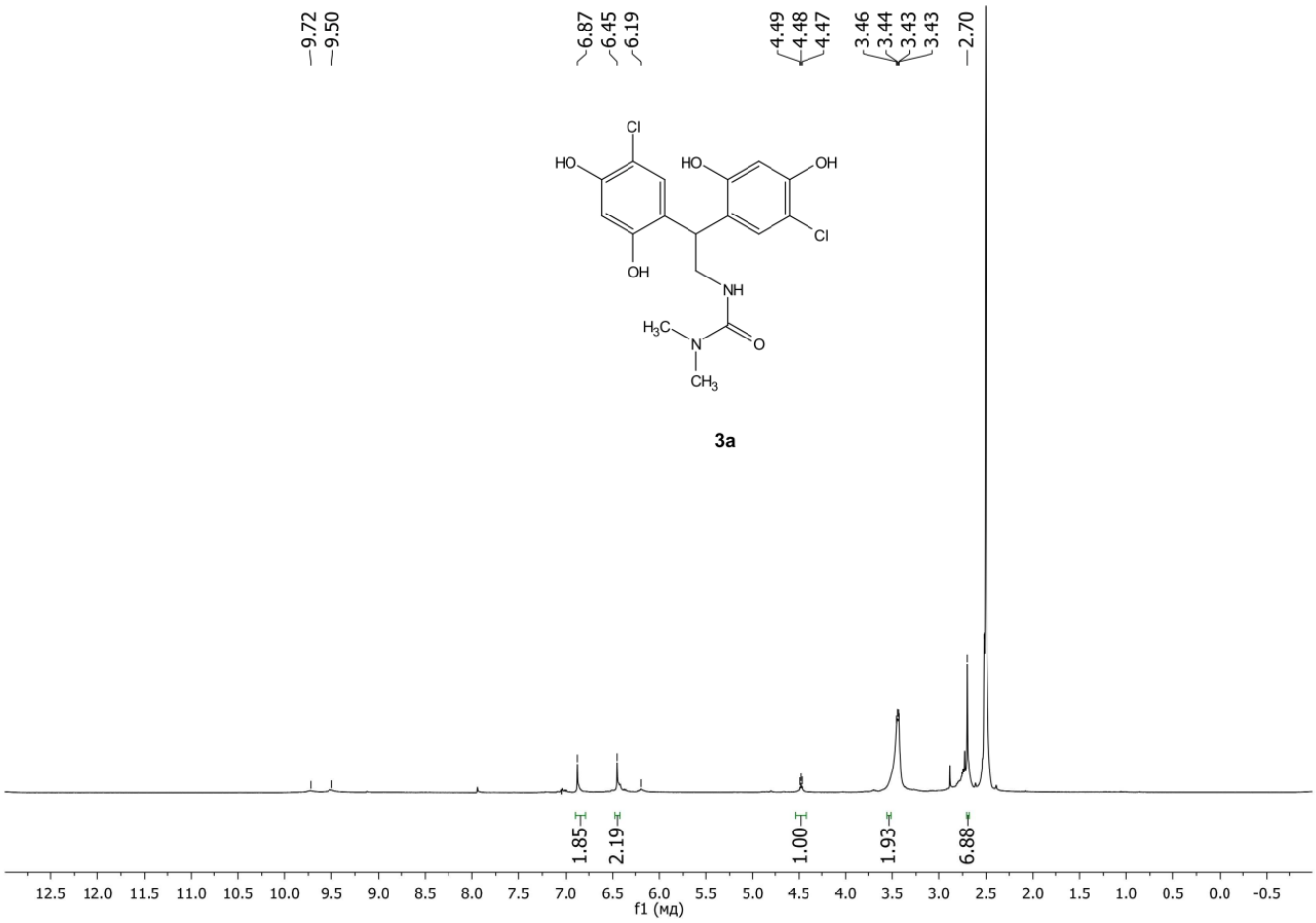

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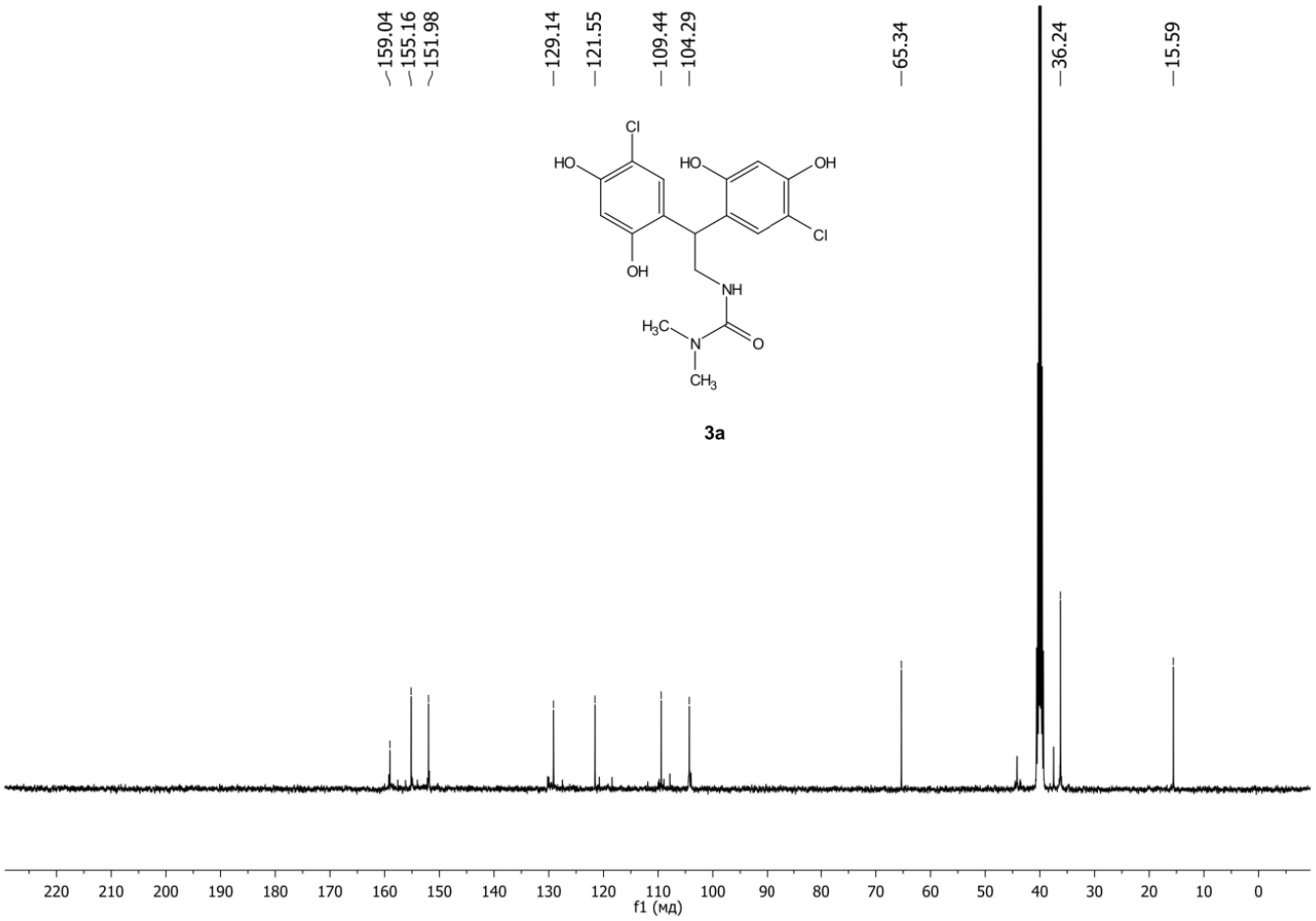

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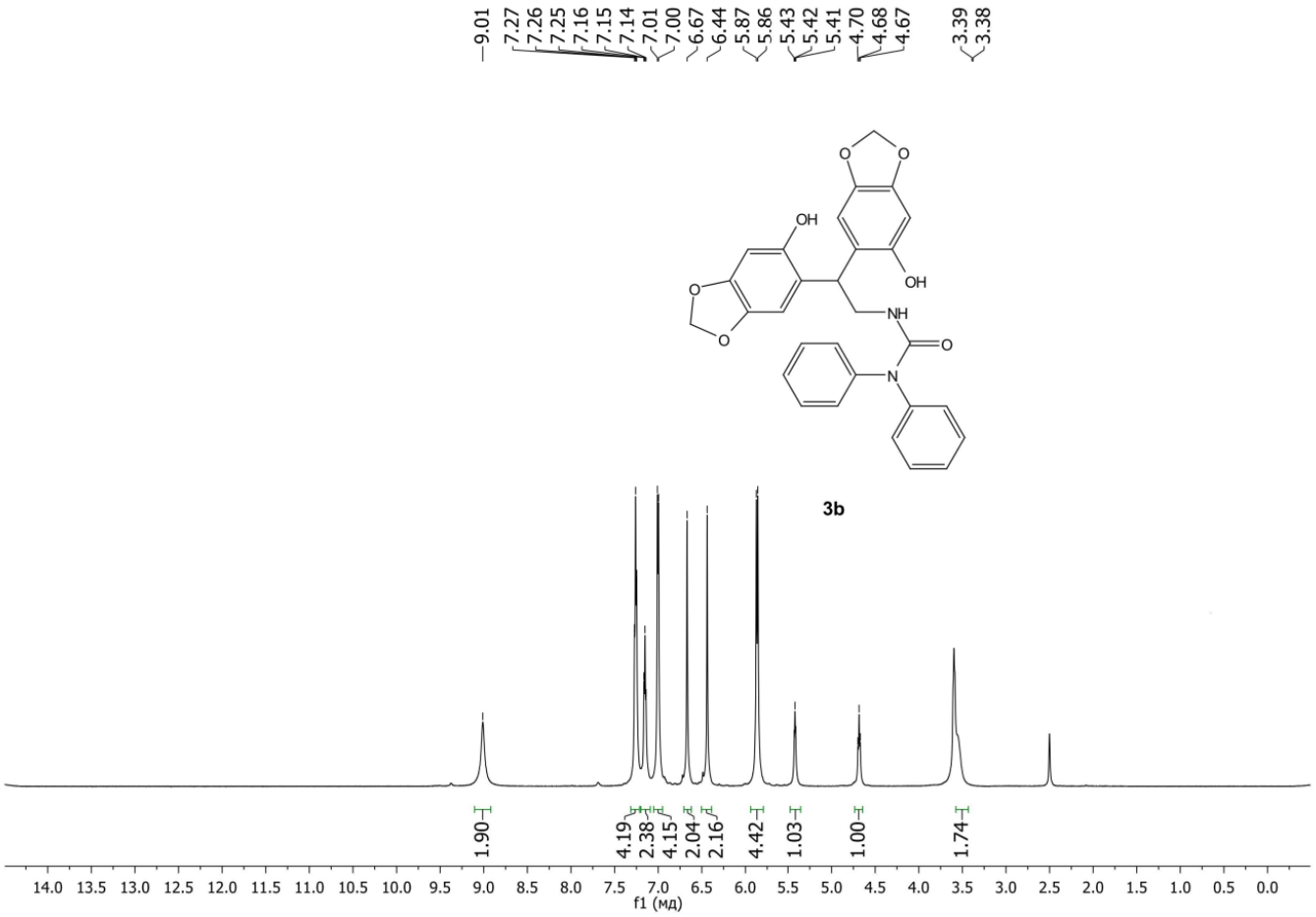

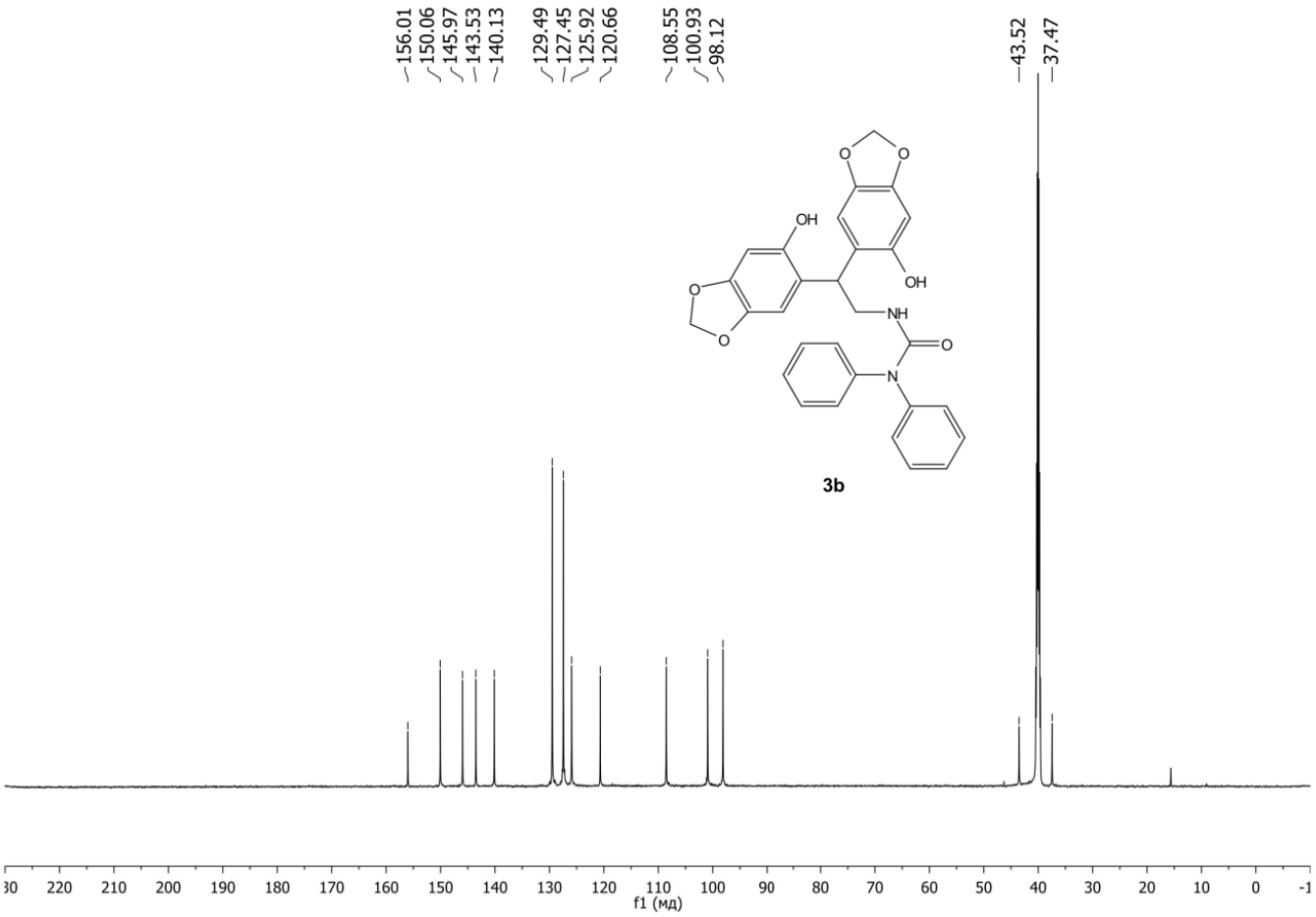

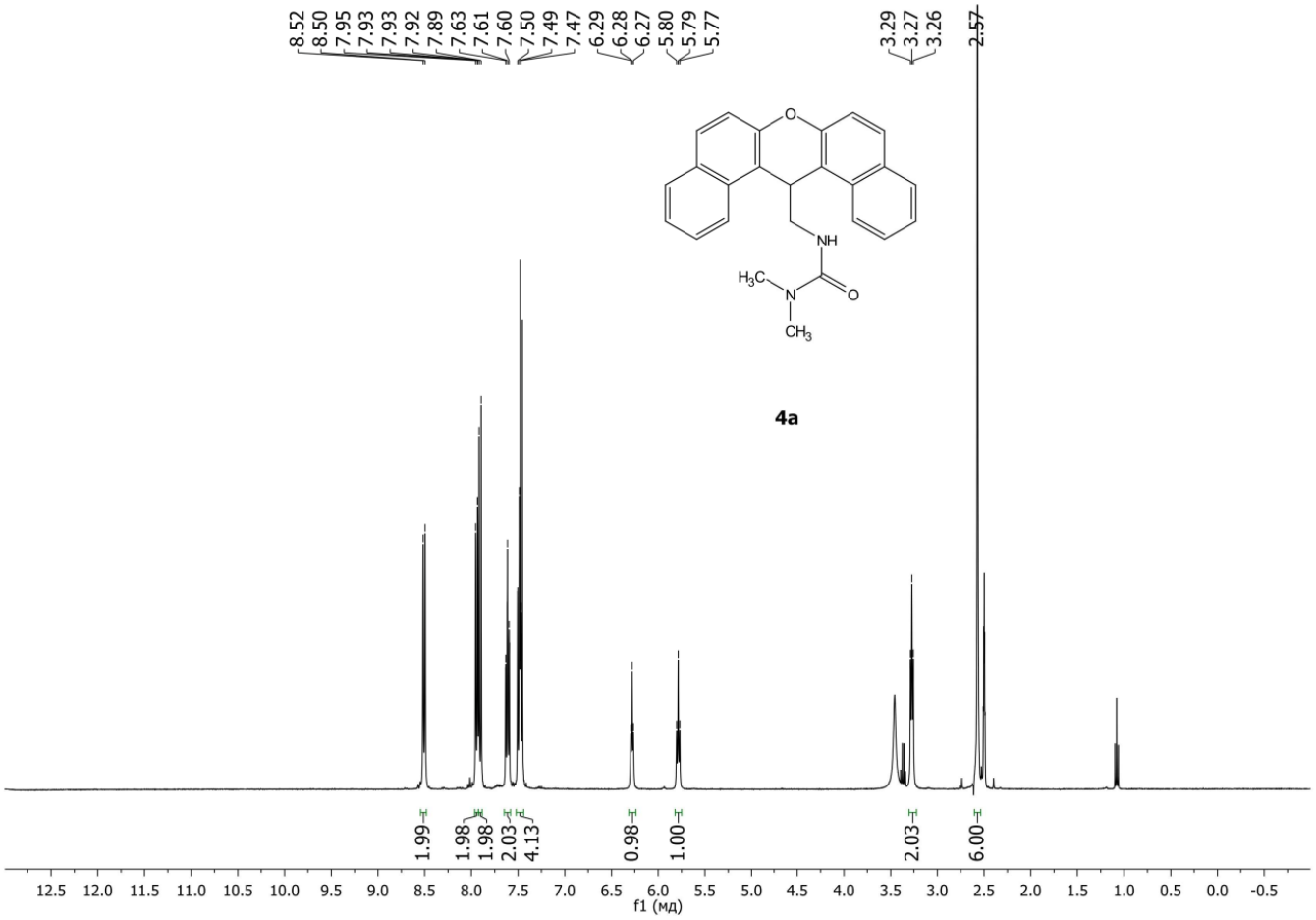

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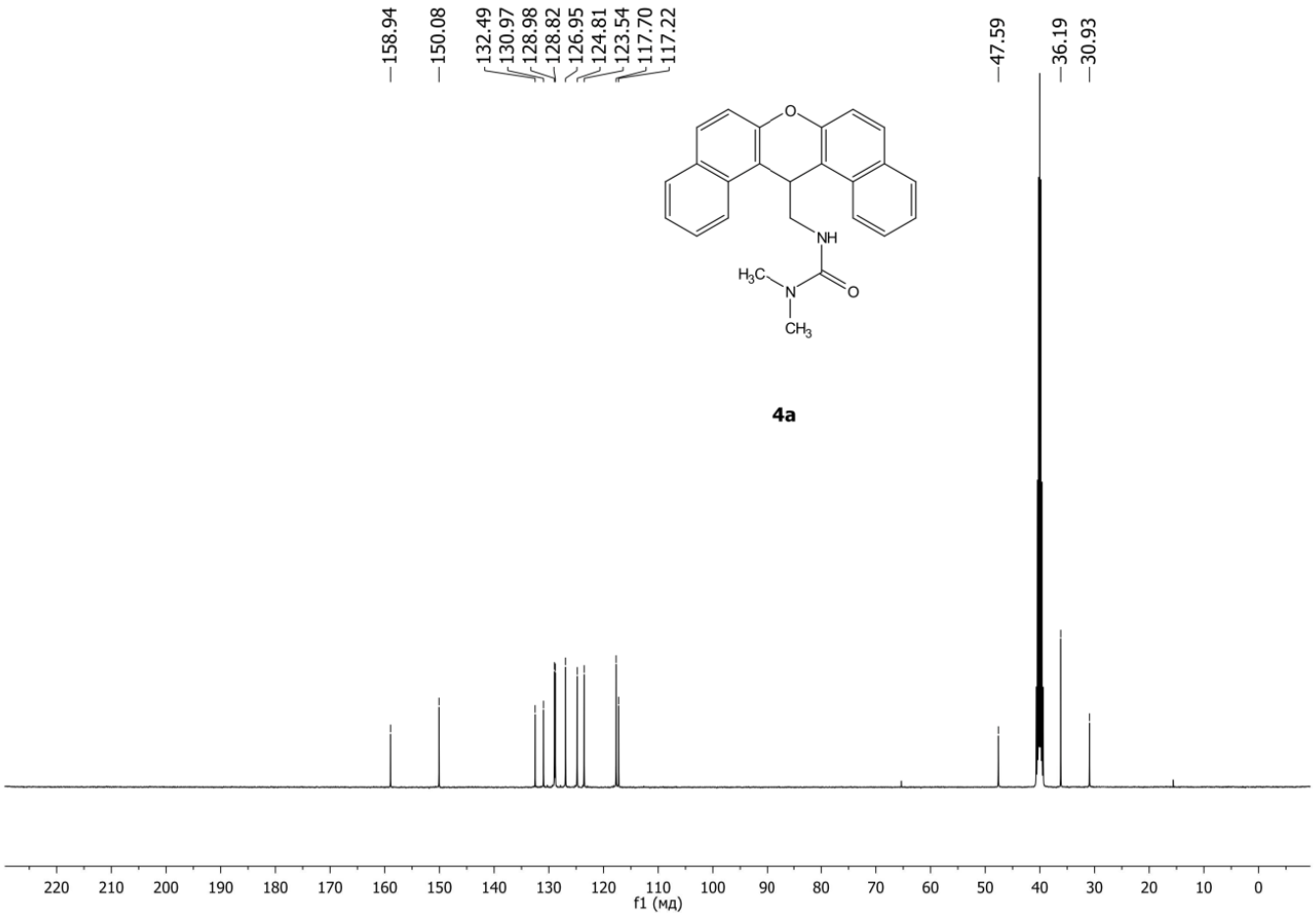

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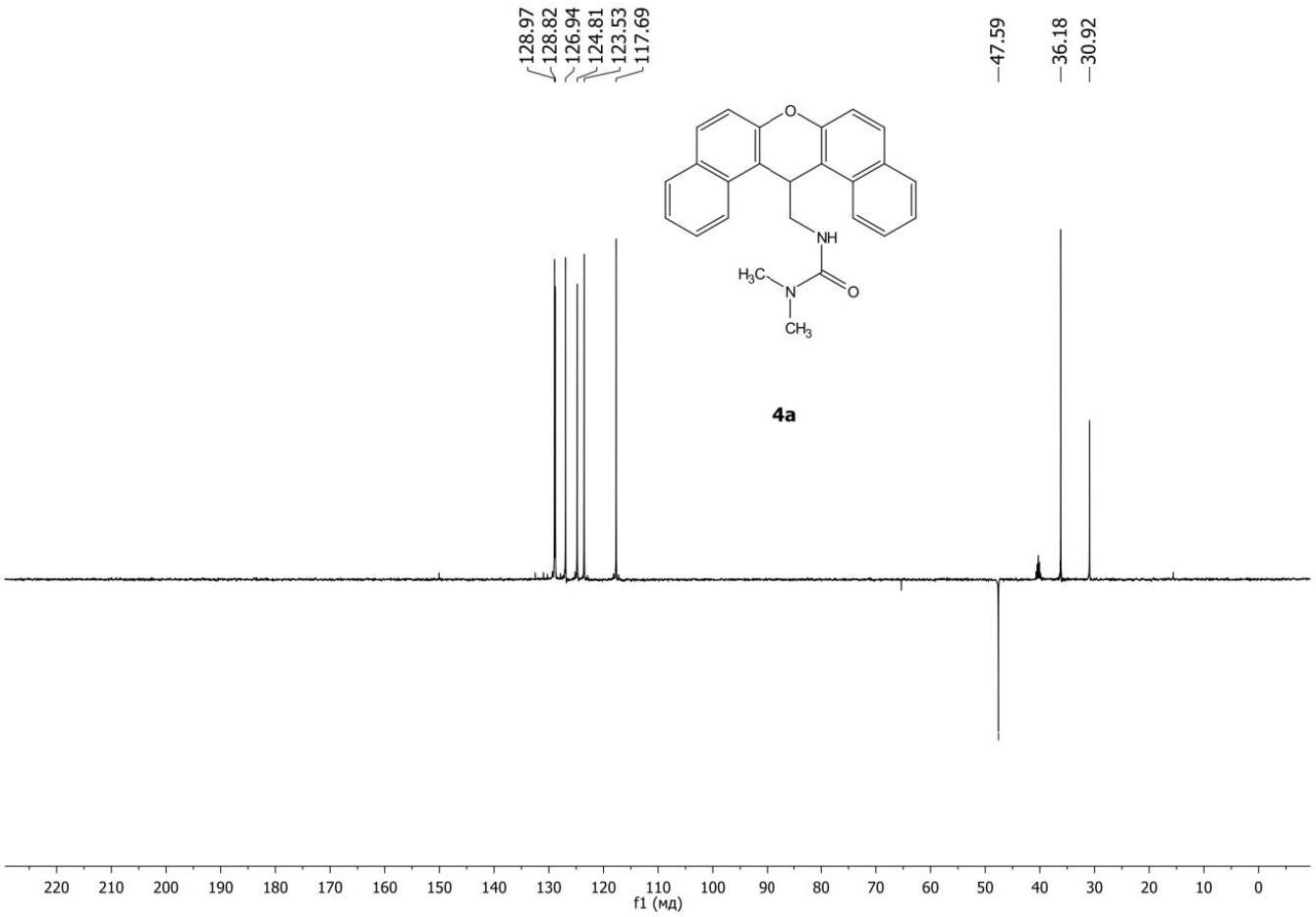

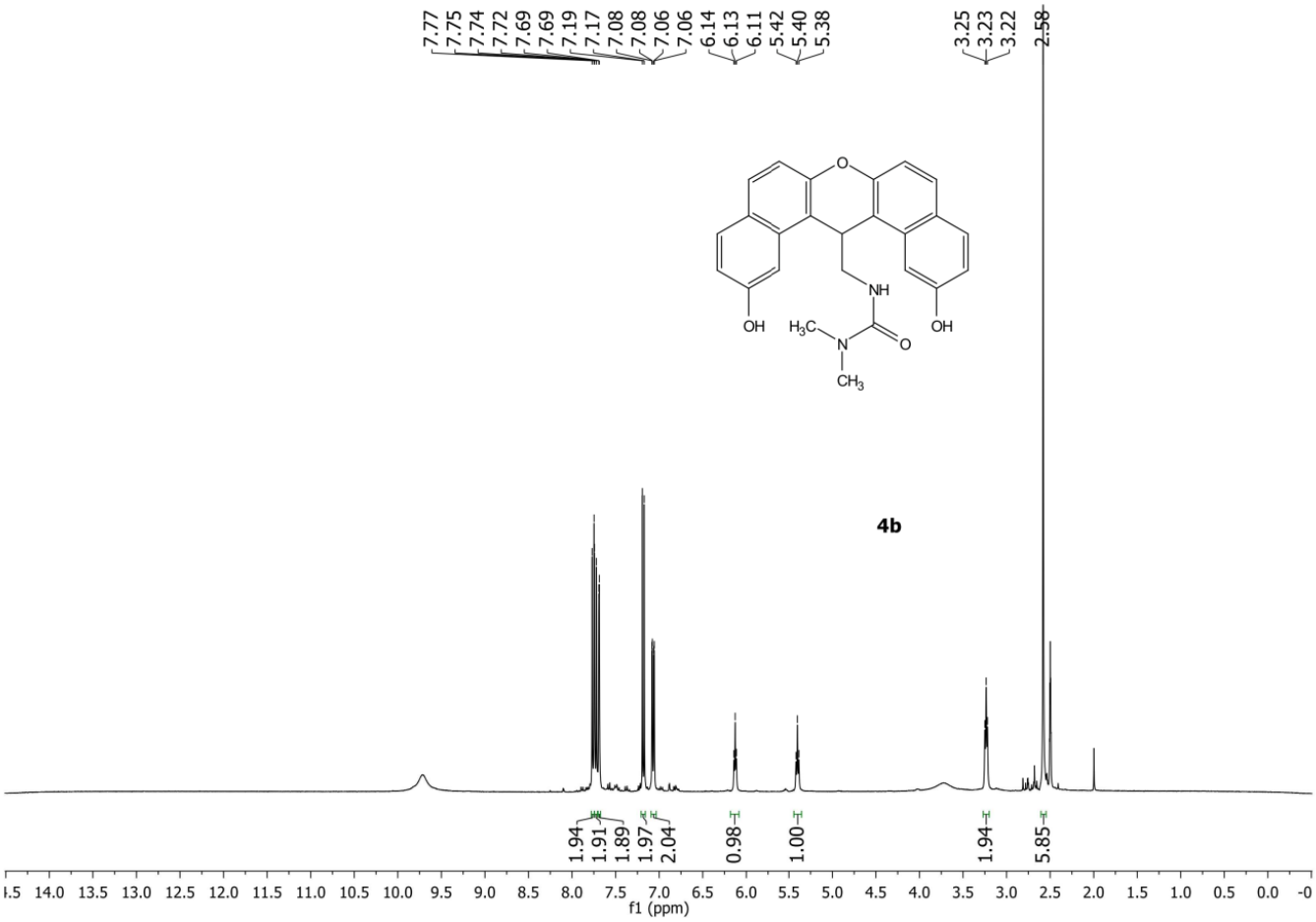

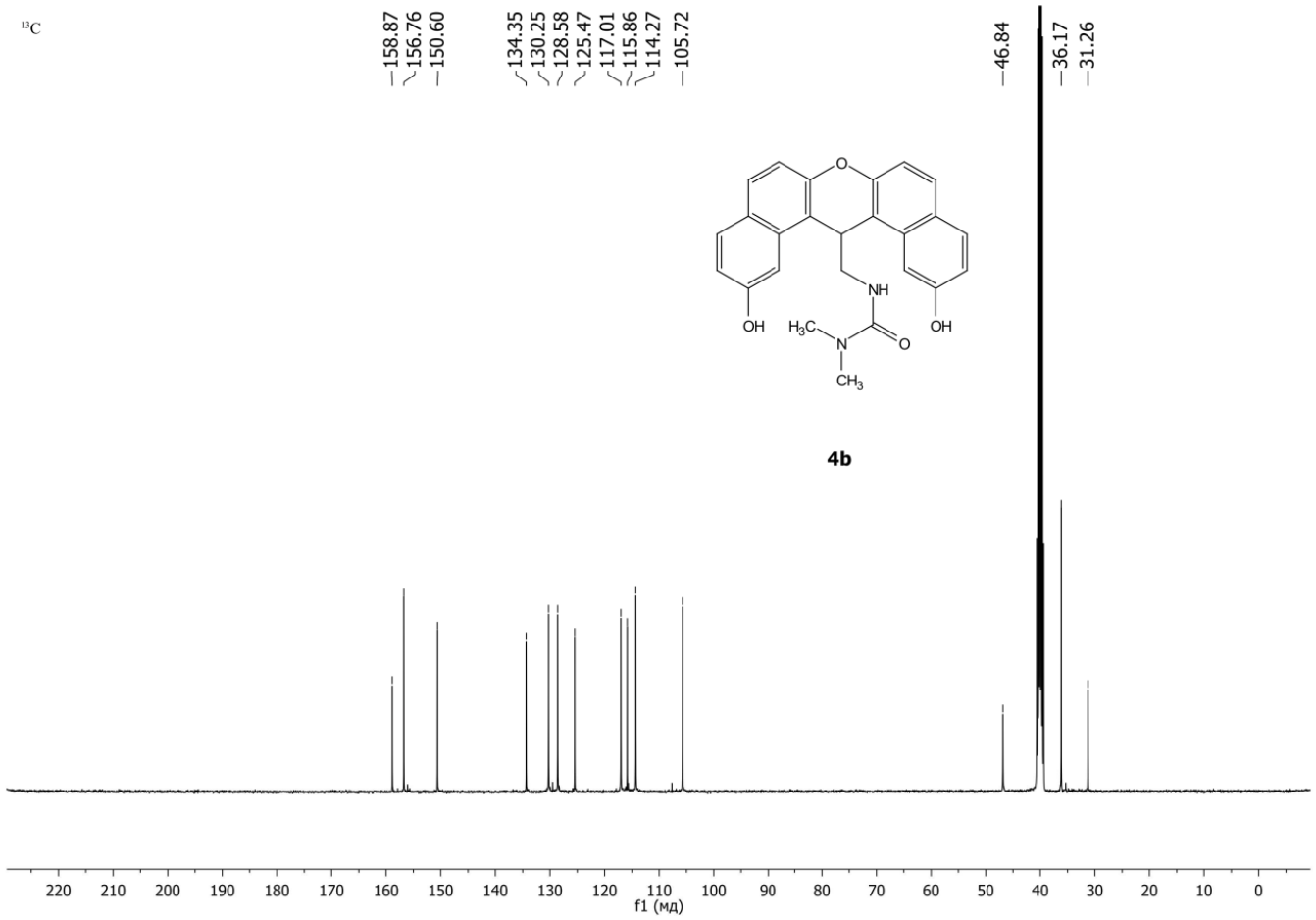

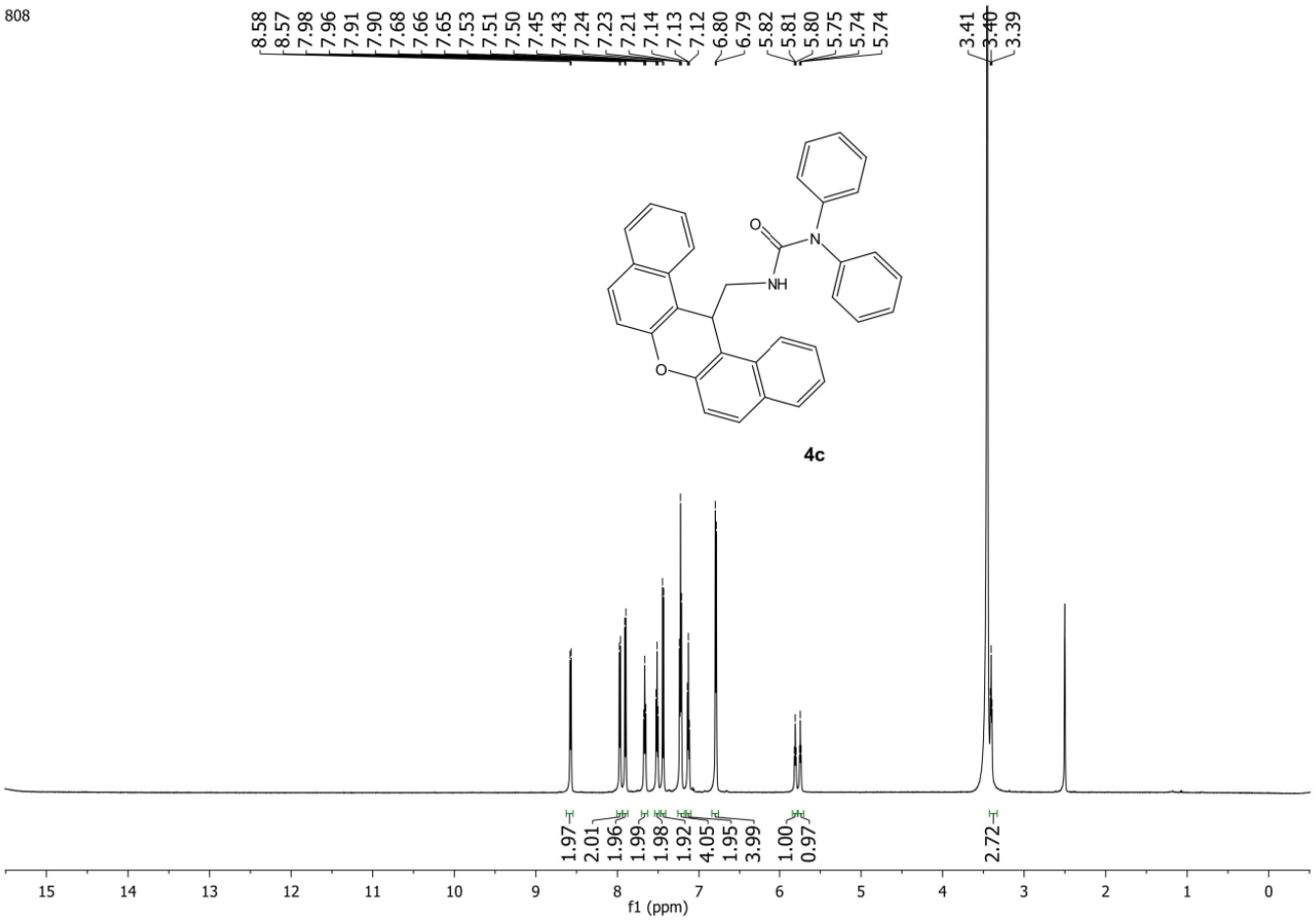

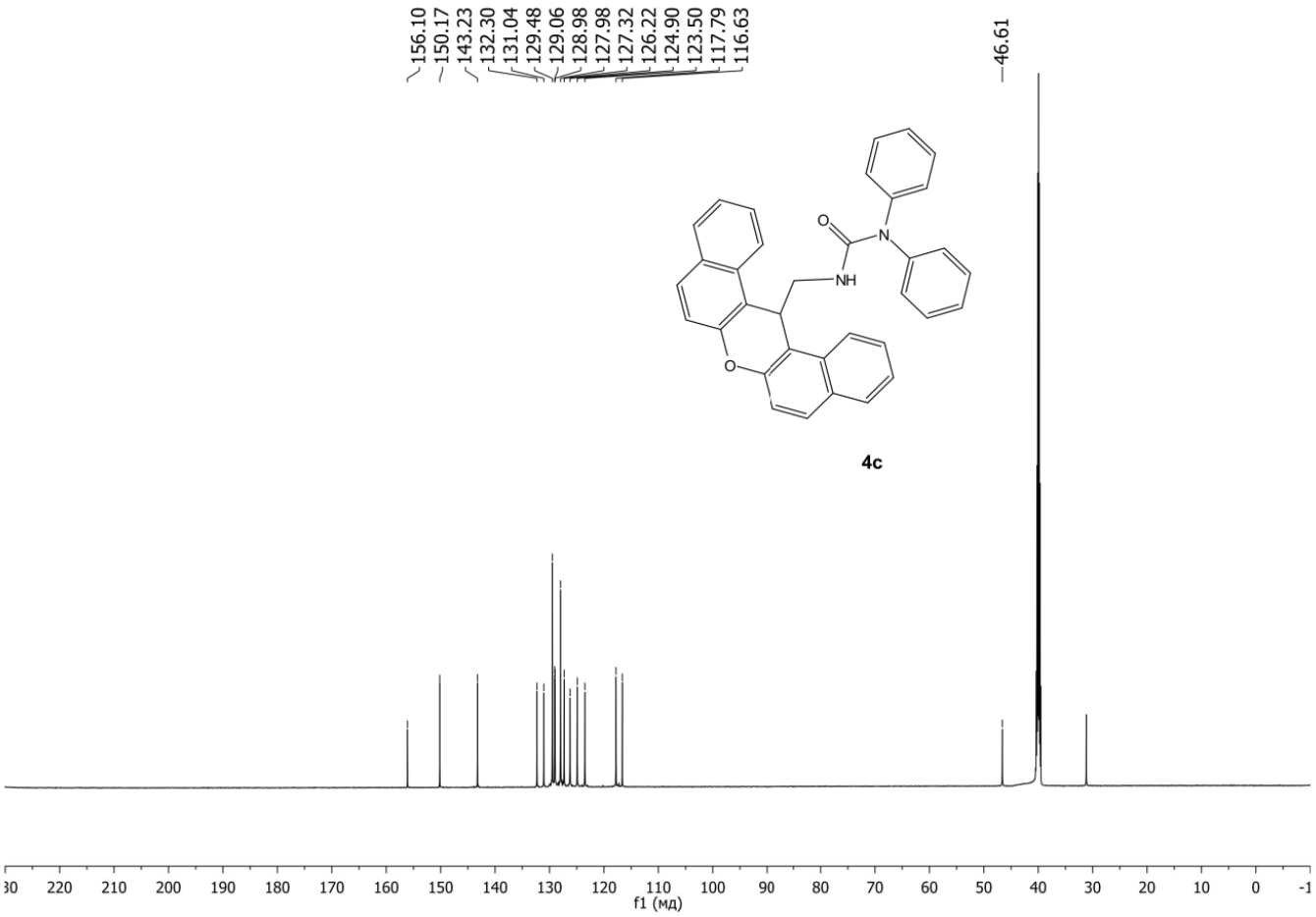

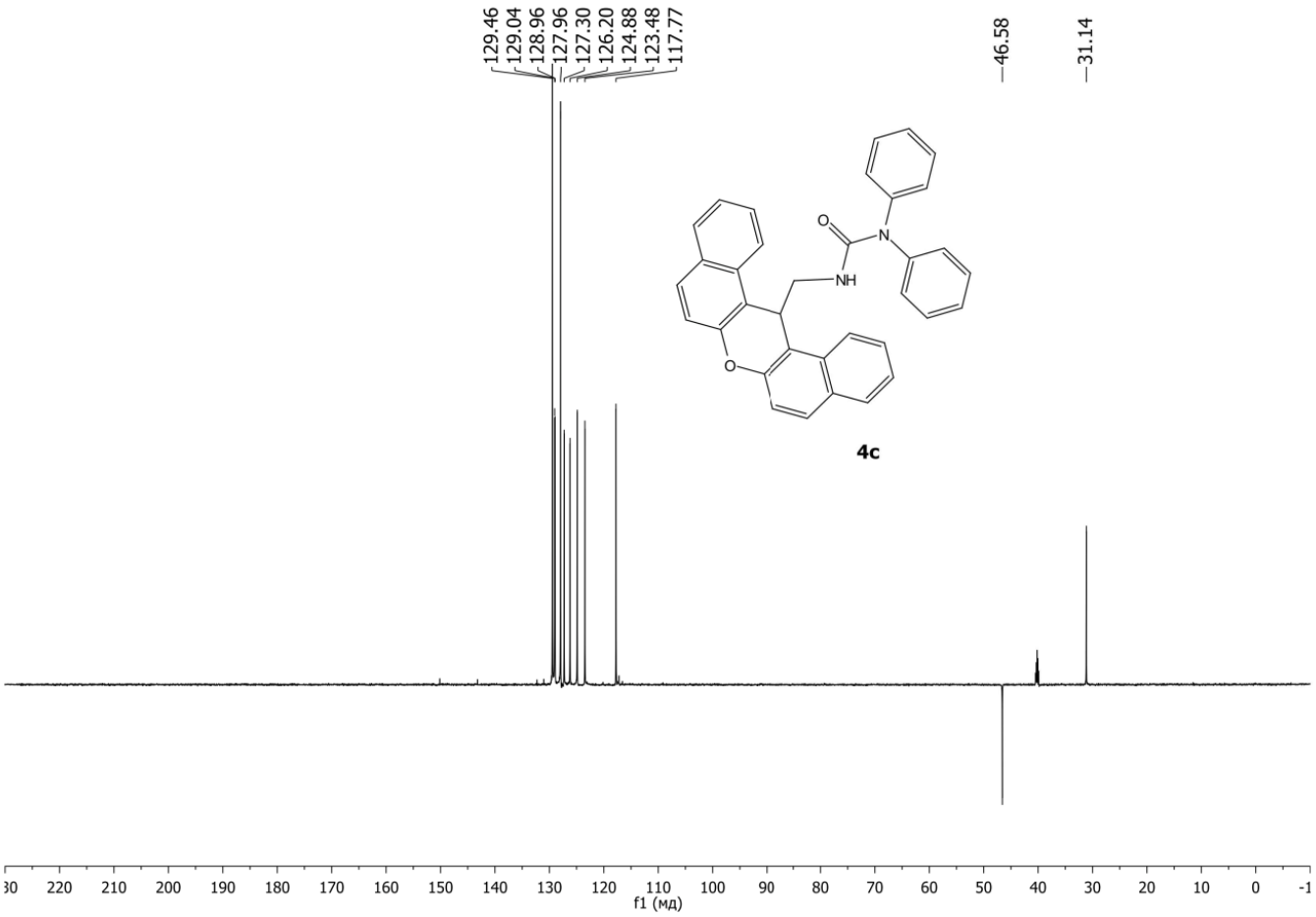

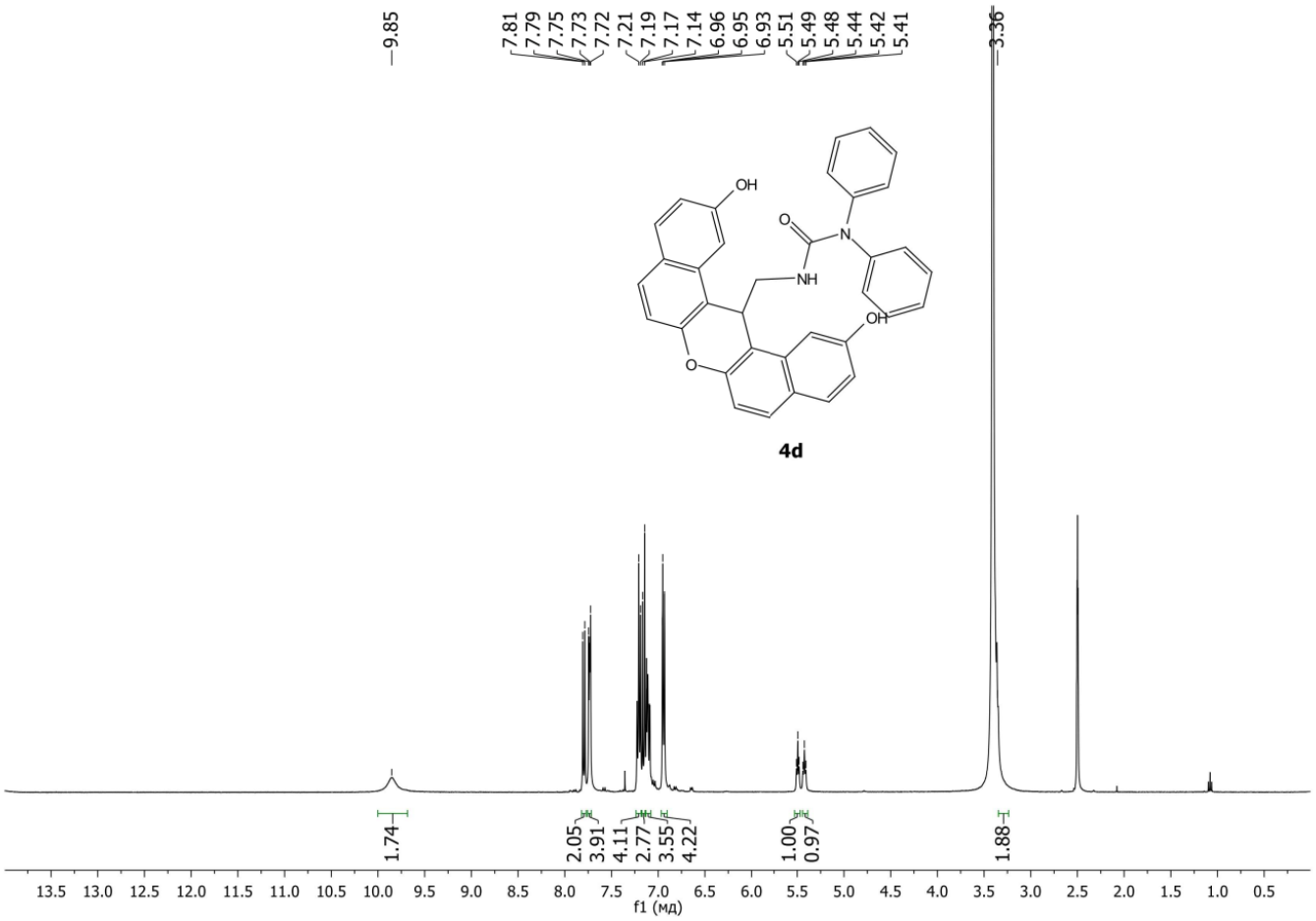

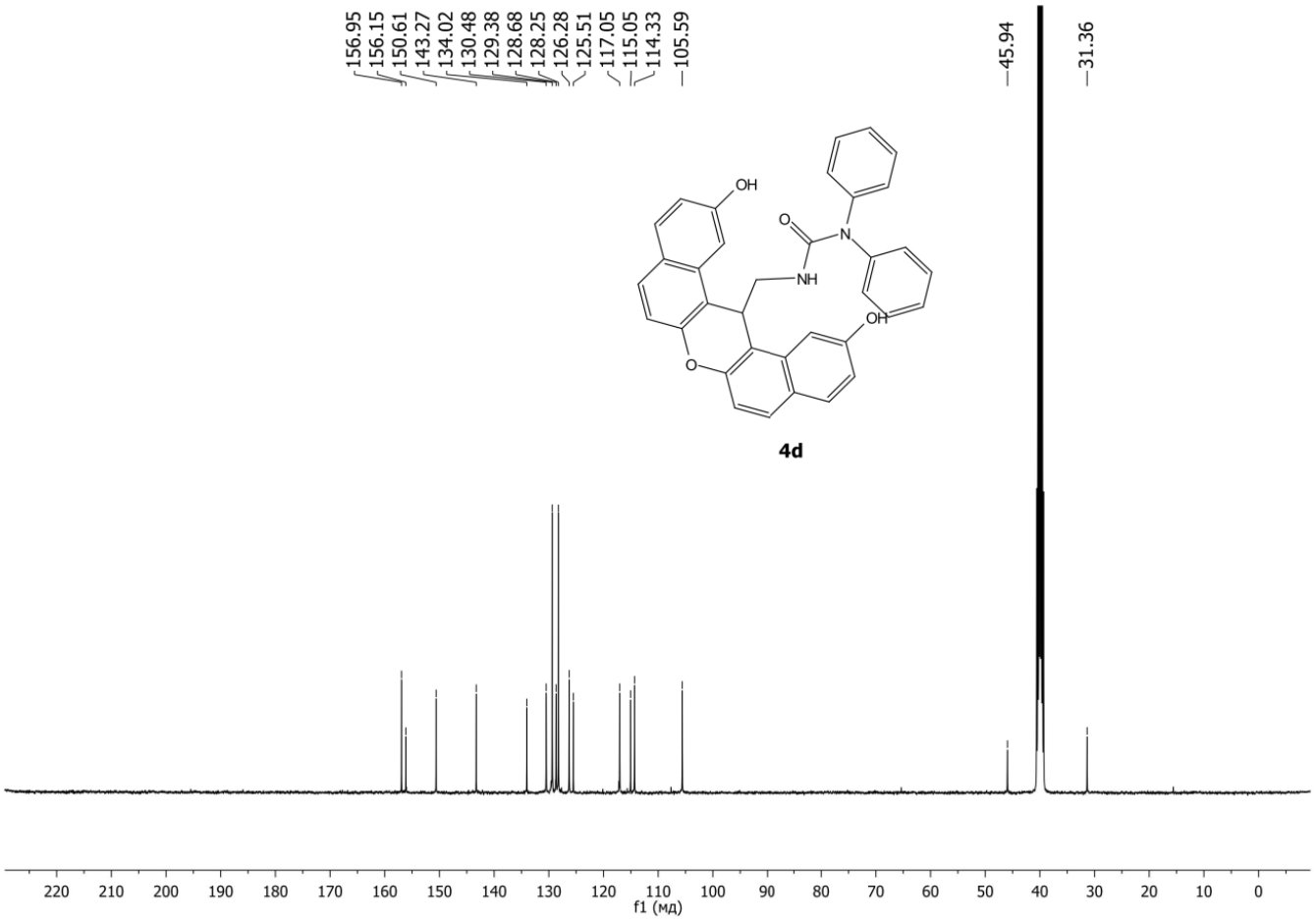

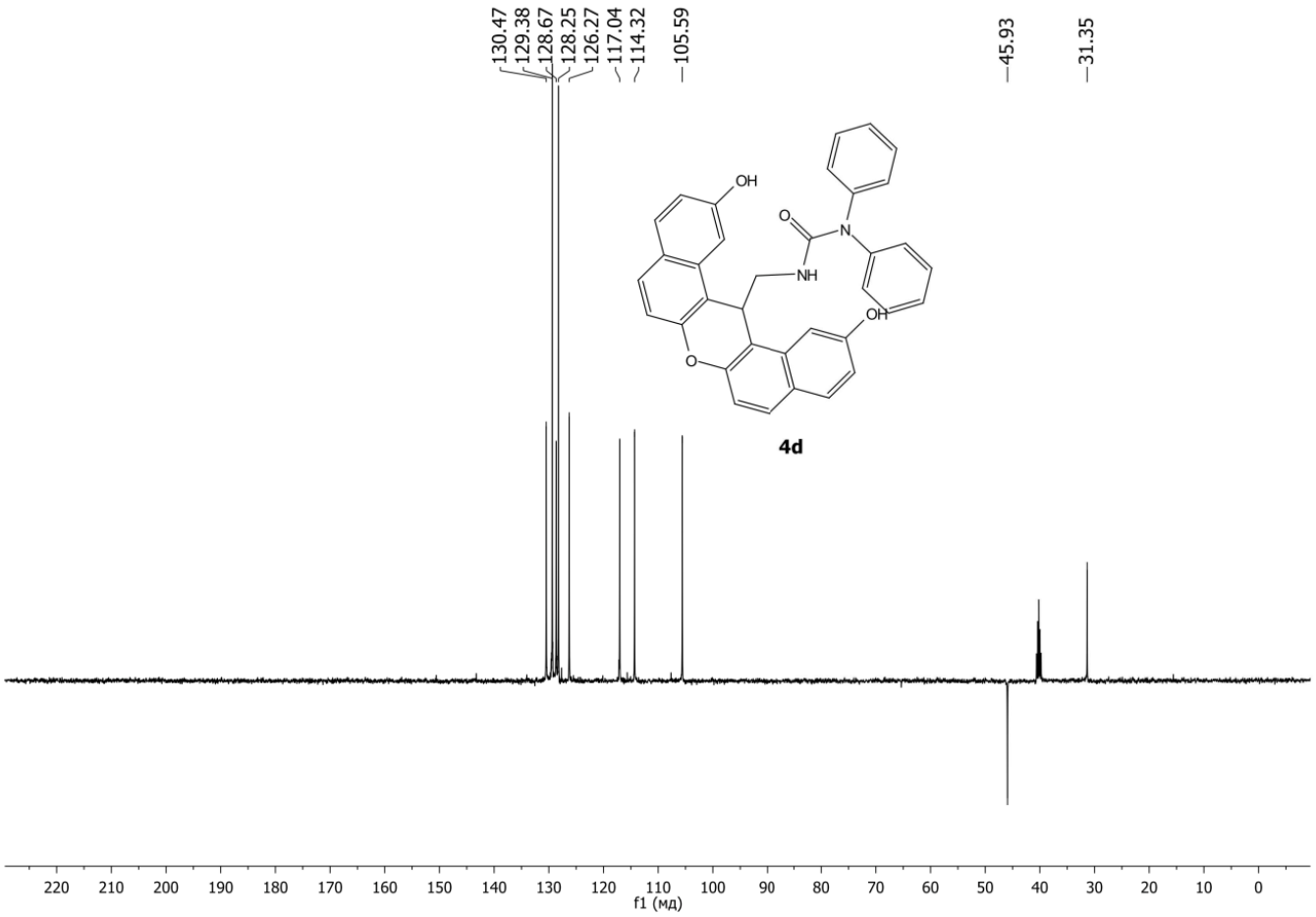

Experimental. The X-ray diffraction data for the crystal of **4c** were collected on a Smart Apex II automatic diffractometer using graphite monochromated radiation. The structures were solved by direct methods and refined by full-matrix least-squares using the SHELXL97[1] program. All the non-hydrogen atoms were refined with anisotropic atomic displacement parameters. All figures were made using the program OLEX2[2]. Crystallographic data for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Center (deposit number is 1950456).

Crystal data for 4c: $C_{35}H_{26}N_2O_2$, M = 506.58, colorless crystal, monoclinic, space group *P21/c*, Z = 4, a = 10.1977(9), b = 12.3675(13), c = 20.1934(18)Å, β=103.246(7)°, V = 2479.0(4) Å^3, ρ_{calc} = 1.357 g/cm^3, μ = 0.084 mm^{-1}, 19605 reflections collected (±h, ±k, ±l), 5950 independent (R_{int} =0.1283) and 2945 observed reflections [$I \geq 2\ \sigma(I)$], 353 refined parameters, R = 0.0780, wR^2 = 0.2132, residual electron density 0.327(-0.379)eÅ^{-3}.

The bond lengths, valence and torsion angles in the molecules of **4c** in crystal are in the ranges typical for every bond type. The conformation of molecule **4c** is folded and stabilized by CH(Ph)…π and π…π interactions.

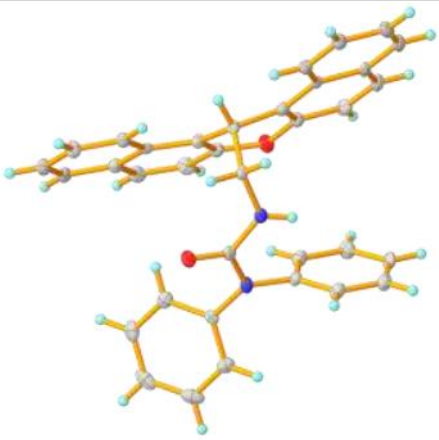

Figure 1. Molecular structure of compound **4c** in crystal. Ellipsoids are shown with 50% probability.

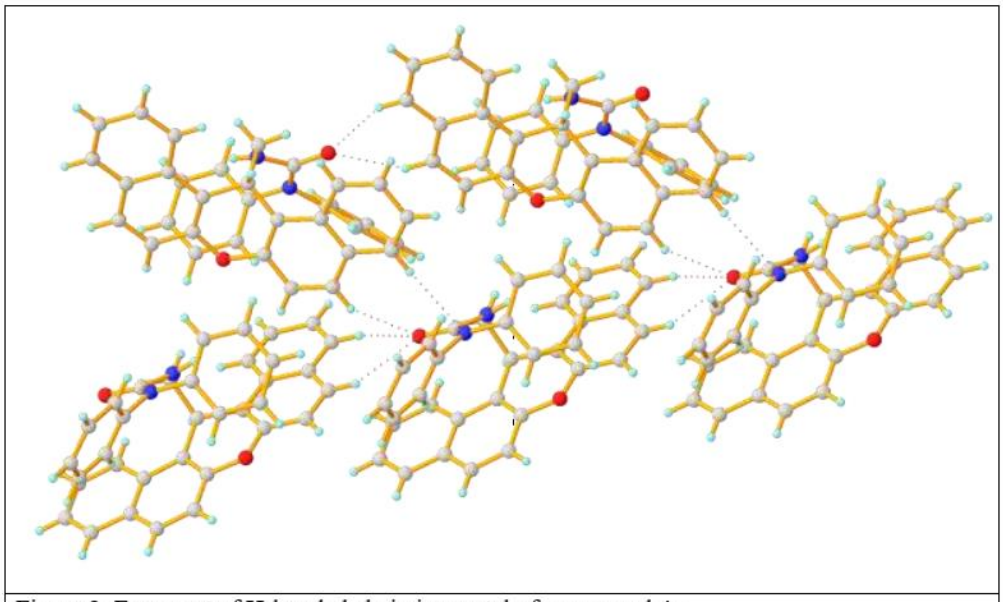

Figure 2. Fragment of H-bonded chain in crystal of compound **4c**.

Crystal packing of investigated compound is formed by the large number of non-covalent interactions. Infinite chains are formed by the hydrogen bonds with additional stabilization of Lp…π interactions. Folded layers are formed perpendicular to them due to stacking and CH…π interactions.

Table 1. H-bonds in crystal of compound **4c**.

H-bond	D – H, Å	H...A, Å	D...A, Å	D - H...A, °
C(7)-H(7)...O(15)	0.93	2.54	2.862(4)	101
C(17)-H(17B)...O(15)	0.97	2.37	2.775(4)	104
C(22)-H(22). ..O(15)	0.93	2.53	3.383(4)	153

Table 2. Lp…π interactions in crystals of **4c**.

Lp… π	X..Cg*	X-Perp	Gamma	Y-X..Cg
C(14)-O(15)... Cg(6)	3.800(3)	3.340	28.49	75.4(2)
C(14)-O(15)... Cg(7)	3.873(3)	3.378	29.29	111.4(2)

*Cg – centroid of the ring.

Table 3. C-H…π interactions in crystals of **4c**.

C-H…π	H..Cg, Å	H-Perp, Å	Gamma, °	X-H..Cg, °	X..Cg, Å	C-H…π
C(7)-H(7)...Cg(6)	2.96	2.79	19.37	153	3.811(4)	63
C(25)-H(25)...Cg(2)	2.84	-2.74	15.83	125	3.468(4)	51

Table 4. π…π interactions in crystals of **4c**.

π …π	Cg-Cg, Å	Alpha, °	Beta, °	Gamma, °	CgI_Perp, Å	CgJ_Perp, Å
Cg(2)...Cg(7)	5.391(2)	68.02(17)	42.6	61.2	2.5974(15)	3.9682(14)
Cg(3)...Cg(6)	5.589(2)	39.72(16)	59.8	48.3	3.7215(14)	2.8086(14)
Cg(4)...Cg(3)	4.849(2)	47.81(16)	10.0	38.0	3.8213(14)	-4.7751(14)
Cg(4)...Cg(5)	5.065(2)	1.44(17)	49.0	50.3	3.2354(15)	3.3254(15)
Cg(4)...Cg(7)	4.641(2)	10.56(17)	47.7	39.0	3.6043(14)	3.1224(14)
Cg(5)...Cg(4)	5.065(2)	1.44(17)	50.3	49.0	3.3254(15)	3.2355(15)
Cg(5)...Cg(5)	4.410(2)	0.03(17)	42.4	42.4	3.2580(15)	3.2580(15)
Cg(5)...Cg(7)	4.949(2)	10.99(17)	50.2	48.5	3.2778(15)	3.1707(14)
Cg(6)...Cg(3)	5.589(2)	39.72(16)	48.3	59.8	2.8085(14)	3.7215(14)
Cg(7)...Cg(4)	4.641(2)	10.56(17)	39.0	47.7	3.1224(14)	3.6044(14)
Cg(7)...Cg(5)	4.949(2)	10.99(17)	48.5	50.2	3.1707(14)	3.2778(15)

1. Sheldrick, G. M. SHELXTL v.6.12, Structure Determination Software Suite, Bruker AXS, Madison, WI, USA, 2000.
2. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. J. Appl. Crystallogr. 2009, 42, 339-341.

