Electronic Supplementary Material (ESI) for RSC Advances.

Metal-free multicomponent synthesis of novel macrocyclic tetrathiadienes with cyano and amino groups

Nail S. Akhmadiev, Ekaterina S. Mescheryakova, Vnira R. Akhmetova*, Ashat G. Ibragimov

Institute of Petrochemistry and Catalysis, Russian Academy of Sciences, 141 Prospekt Oktyabrya, 450075 Ufa, Russian Federation. E-mail: vnirara@mail.ru; Tel./fax: +7 3472 842750

Corresponding author:* vnirara@mail.ru (V.R. Akhmetova)

SUPPLEMENTARY INFORMATION

5,12-Diamino-7,14-*bis*(4-fluorophenyl)-1,4,8,11-tetrathiacyclotetradeca-5,12-diene-6,13-dicarbonitrile **4a**

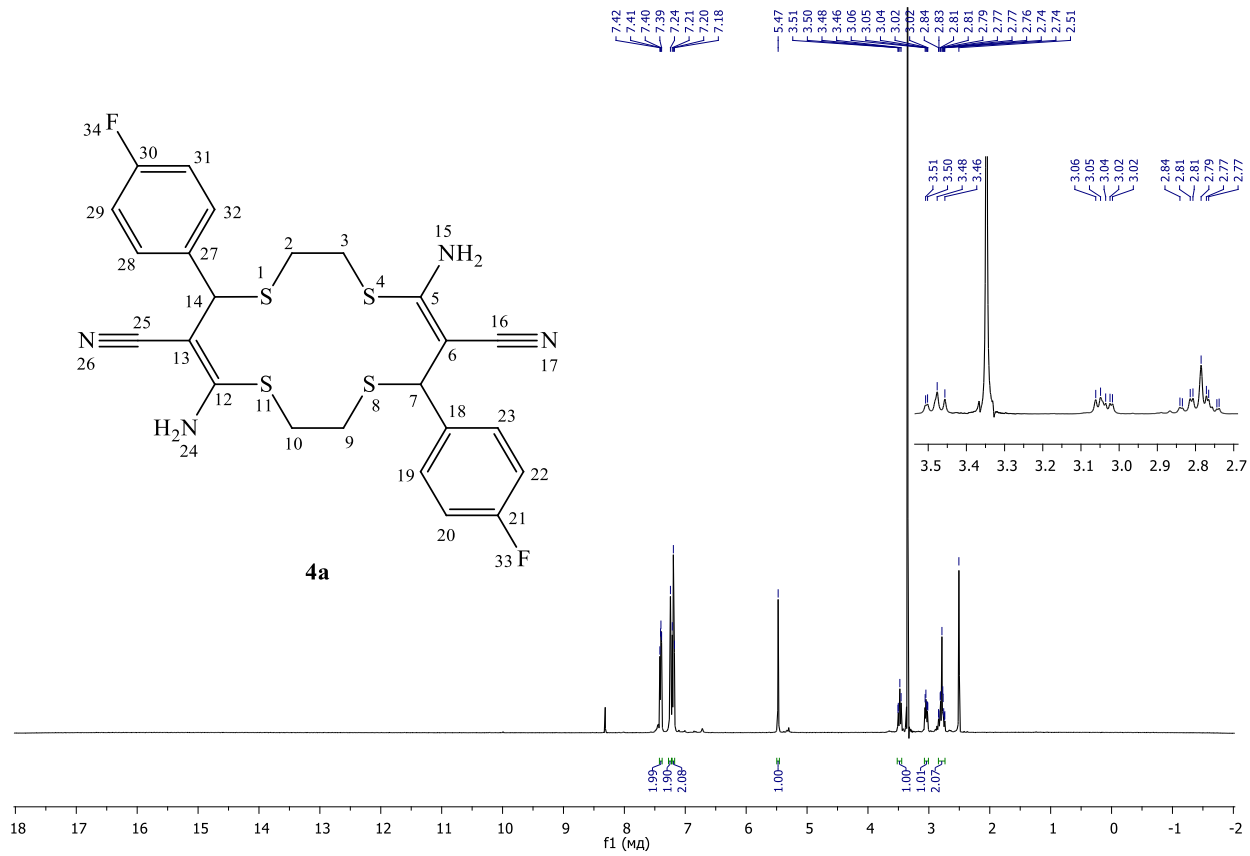

Fig. S1. ^{1}H NMR spectrum of compound **4a** in DMSO-D_6 (500 MHz)

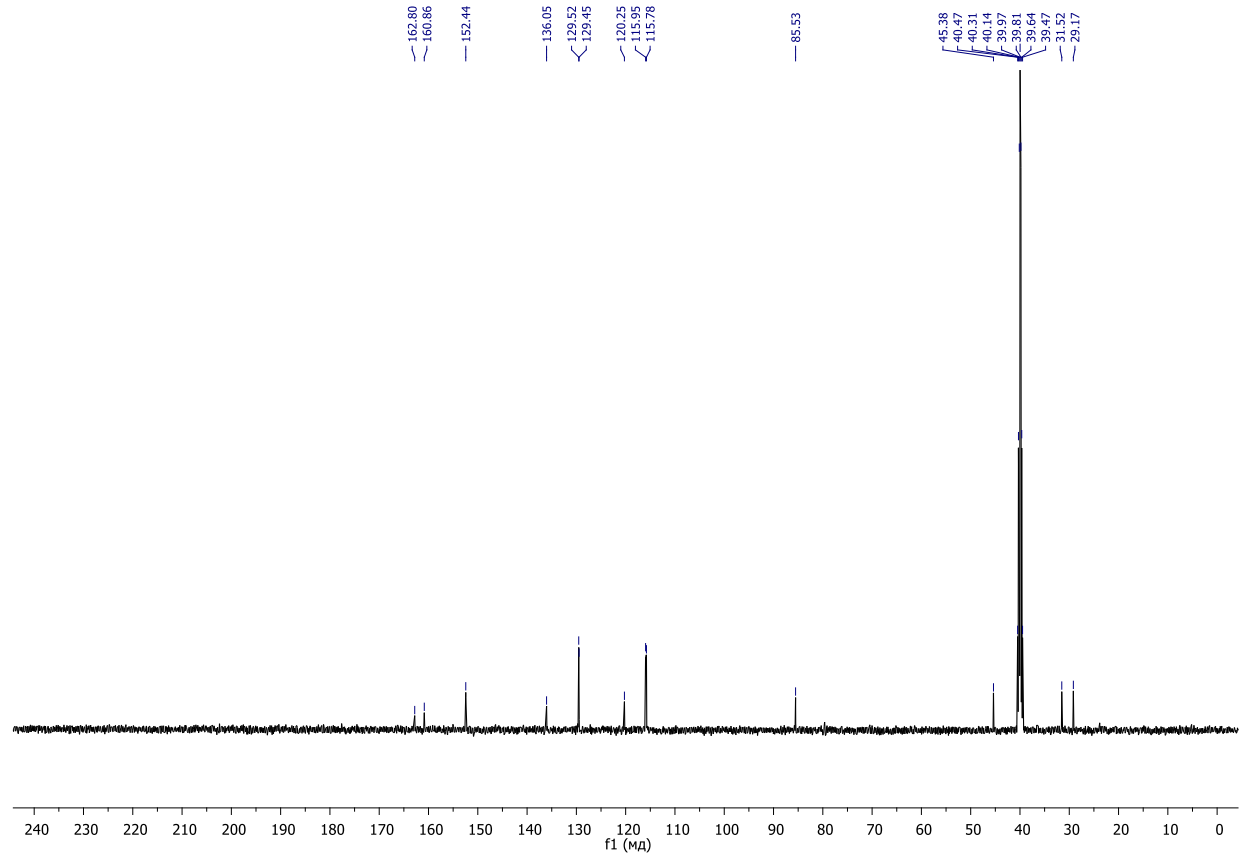

Fig. S2. ^{13}C NMR spectrum of compound **4a** in DMSO-D_6 (125 MHz)

5,12-Diamino-7,14-*bis*(4-chlorophenyl)-1,4,8,11-tetrathiacyclotetradeca-5,12-diene-6,13-dicarbonitrile **4b**

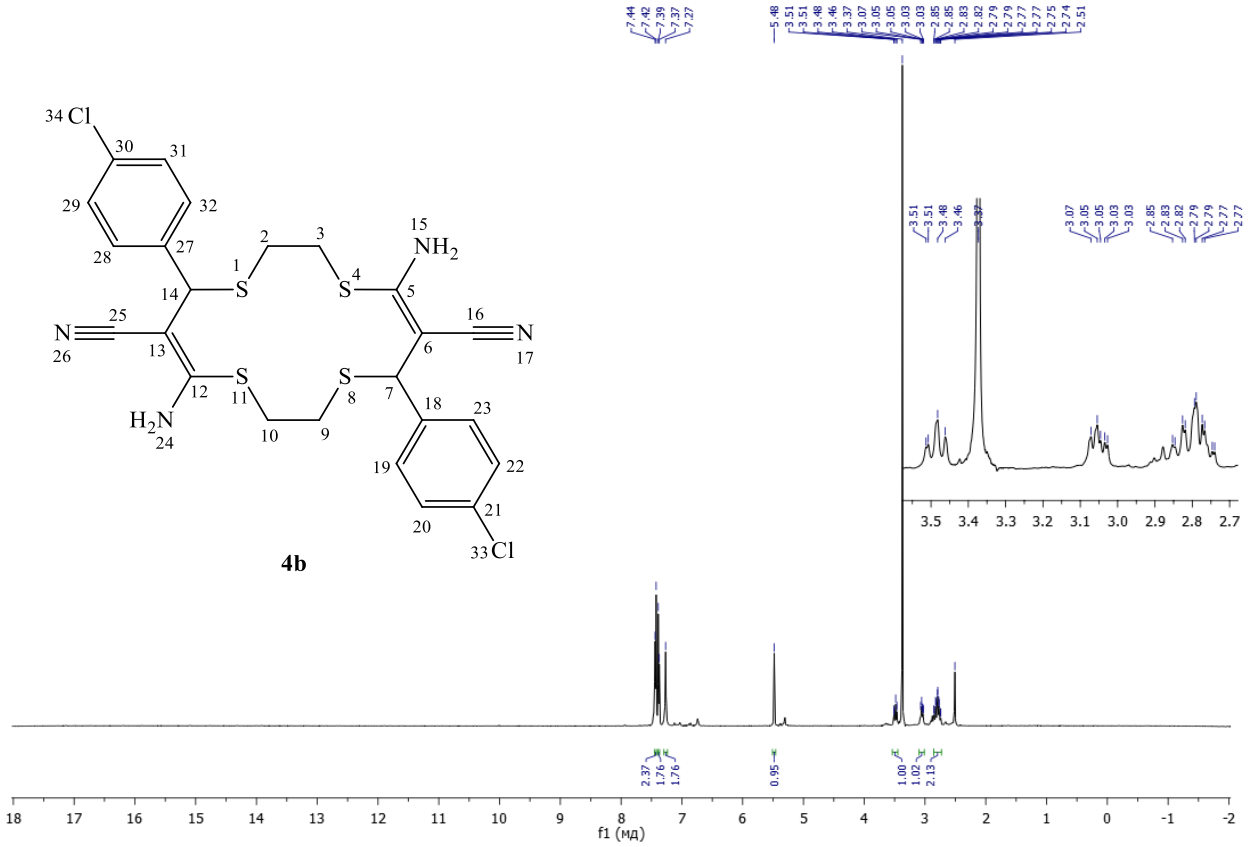

Fig. S3. ^{1}H NMR spectrum of compound **4b** in DMSO-D_6 (500 MHz)

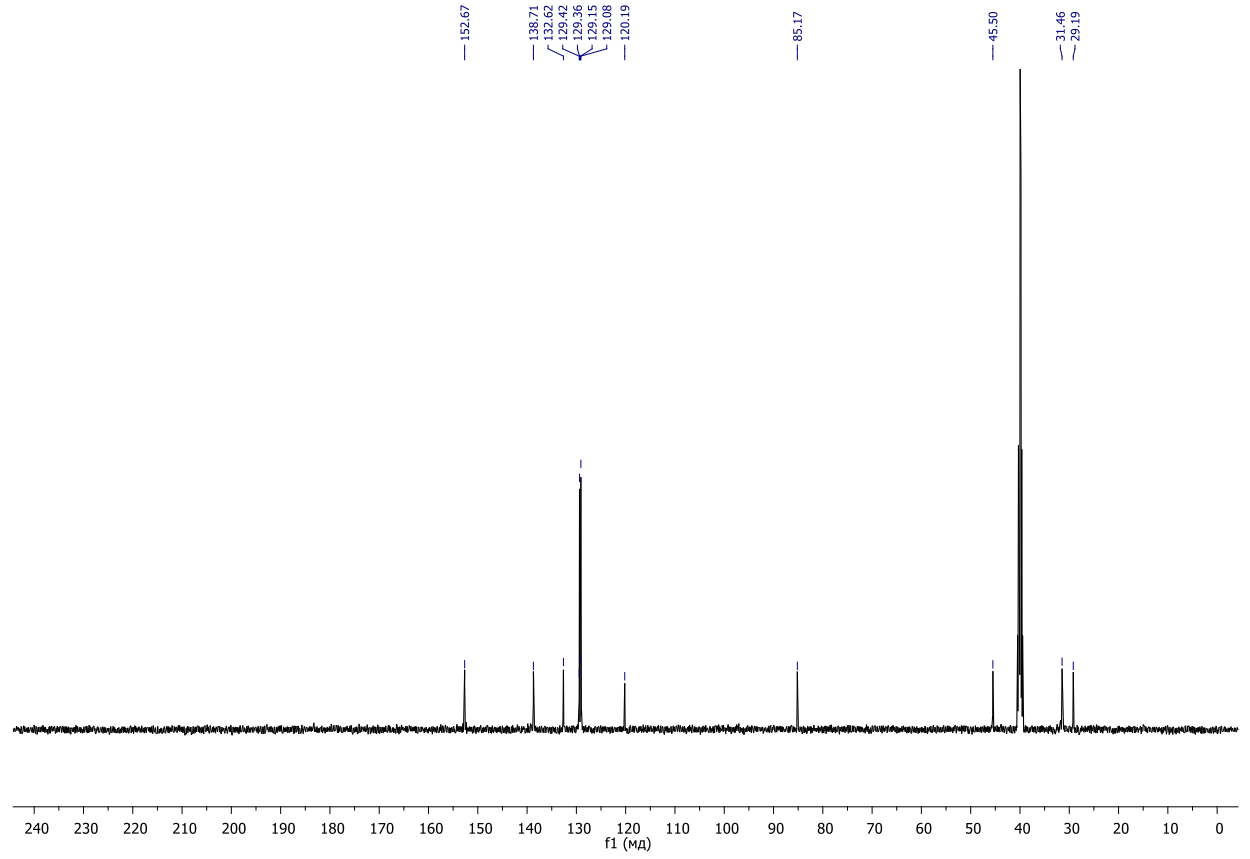

Fig. S4. ^{13}C NMR spectrum of compound **4b** in DMSO-D_6 (125 MHz)

5,12-Diamino-7,14-*bis*(3-fluorophenyl)-1,4,8,11-tetrathiacyclotetradeca-5,12-diene-6,13-dicarbonitrile **4c**

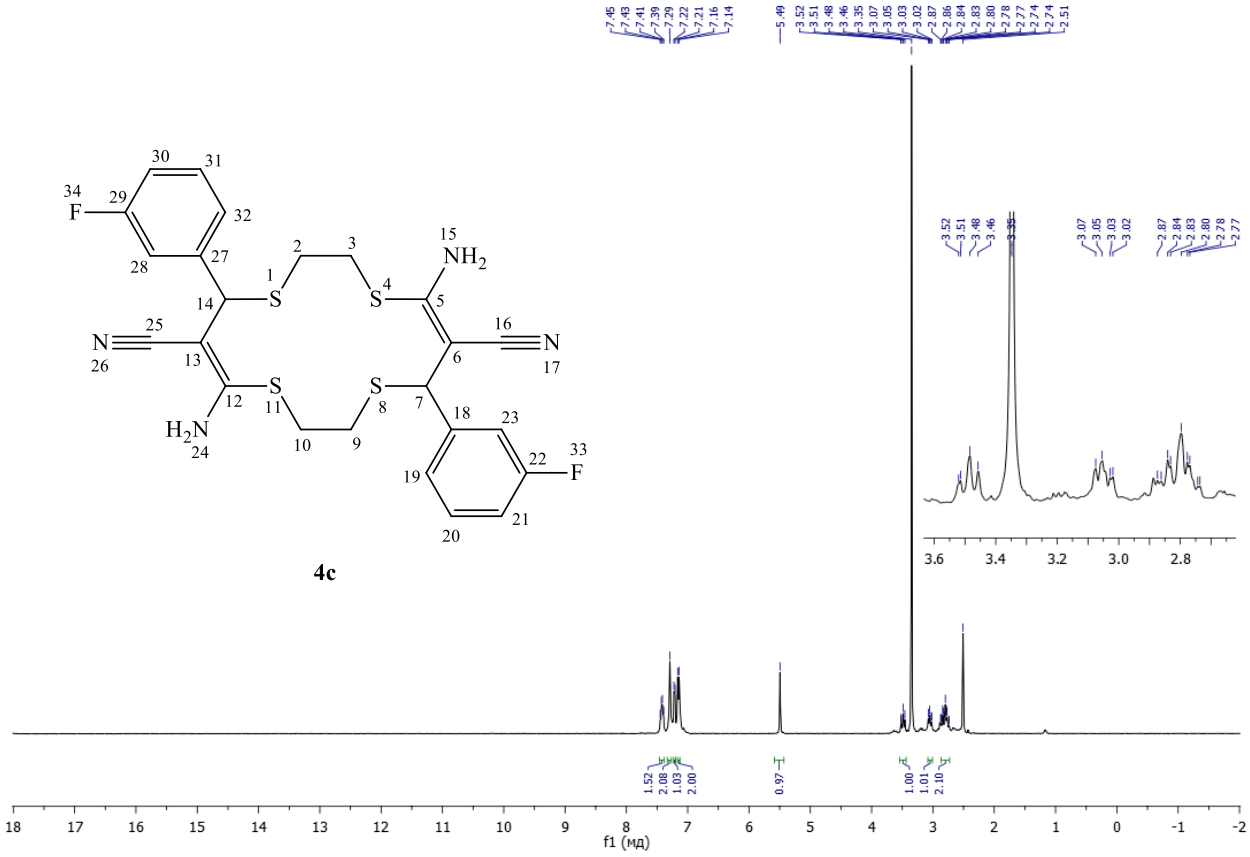

Fig. S5. ^{1}H NMR spectrum of compound **4c** in DMSO-D_6 (400 MHz)

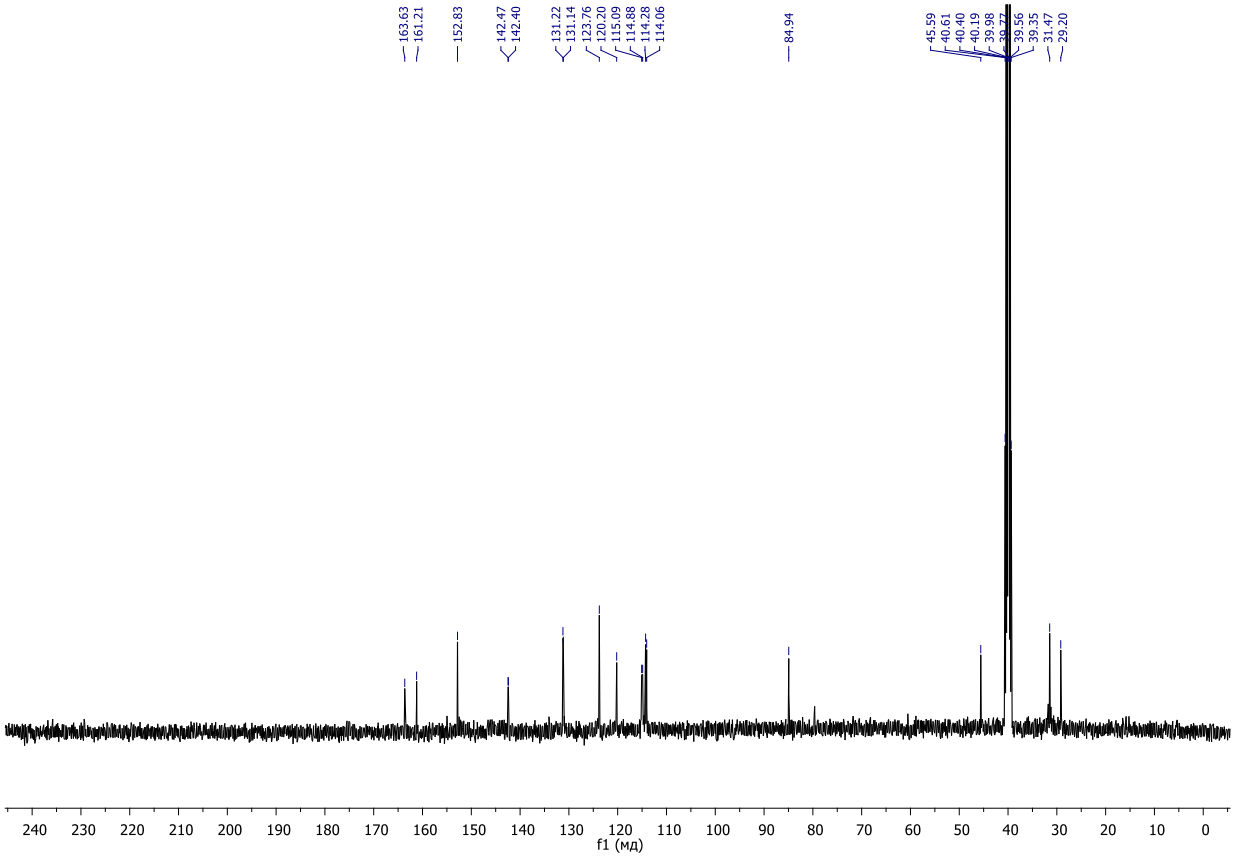

Fig. S6. ^{13}C NMR spectrum of compound **4c** in DMSO-D_6 (100 MHz)

5,12-Diamino-7,14-*bis*[4-(trifluoromethyl)phenyl]-1,4,8,11-tetrathiacyclotetradeca-5,12-diene-6,13-dicarbonitrile **4d**

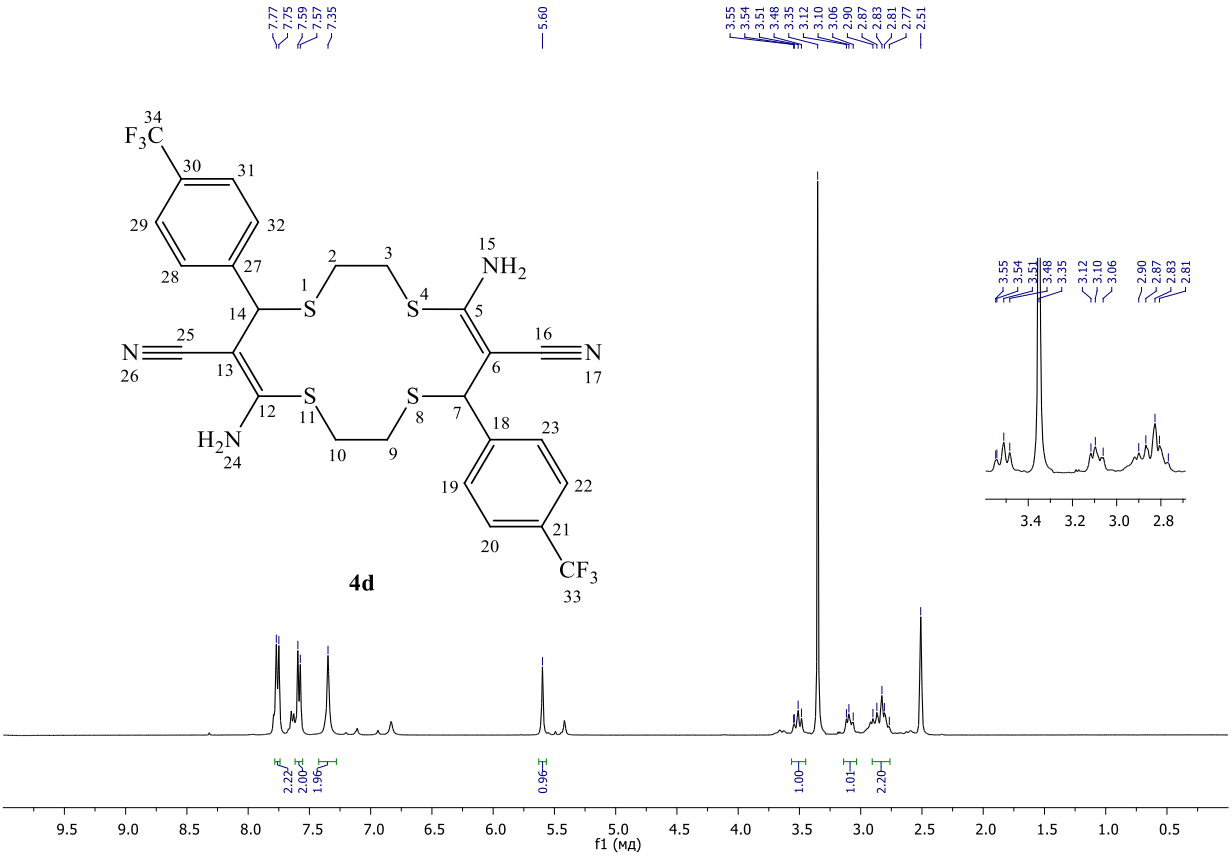

Fig. S7. ^{1}H NMR spectrum of compound **4d** in DMSO-D_6 (400 MHz)

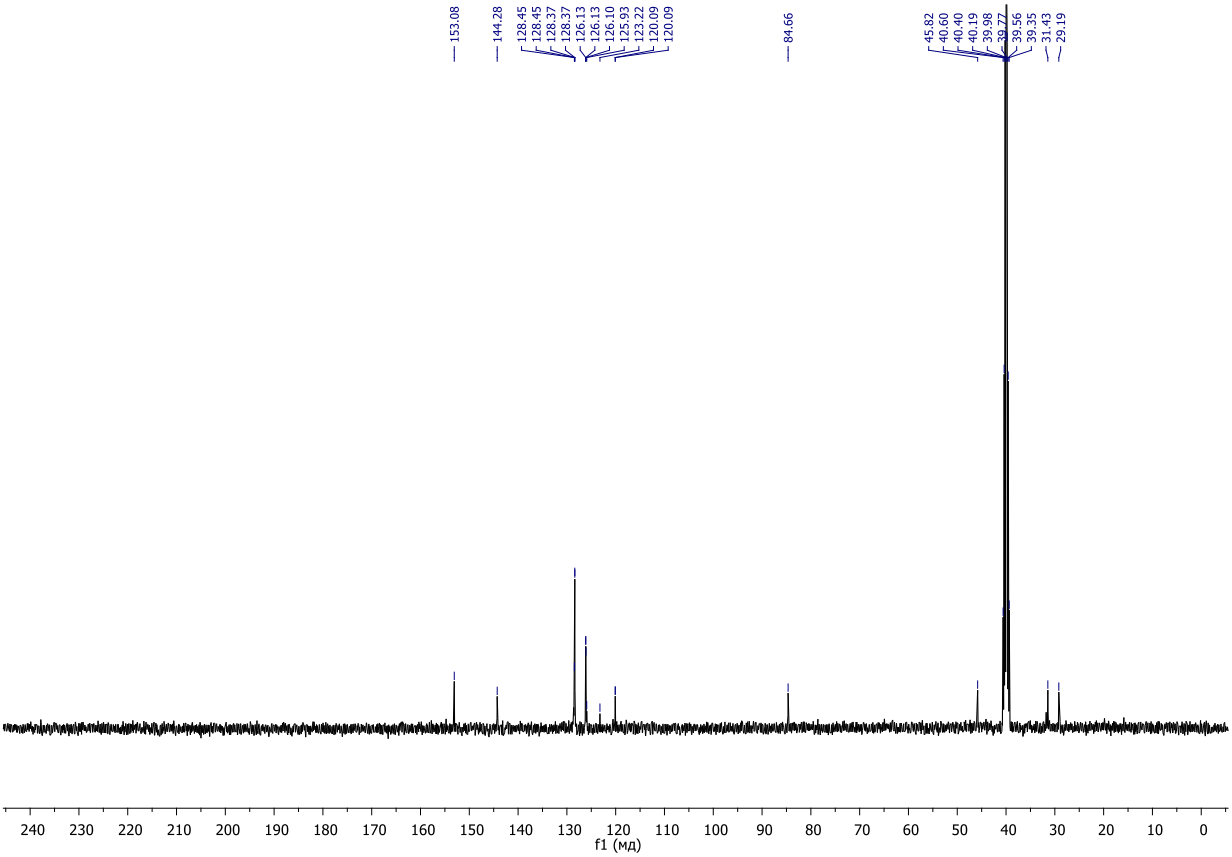

Fig. S8. ^{13}C NMR spectrum of compound **4d** in DMSO-D_6 (100 MHz)

5,12-Diamino-7,14-*bis*(4-methoxyphenyl)-1,4,8,11-tetrathiacyclotetradeca-5,12-diene-6,13-dicarbonitrile **4e**

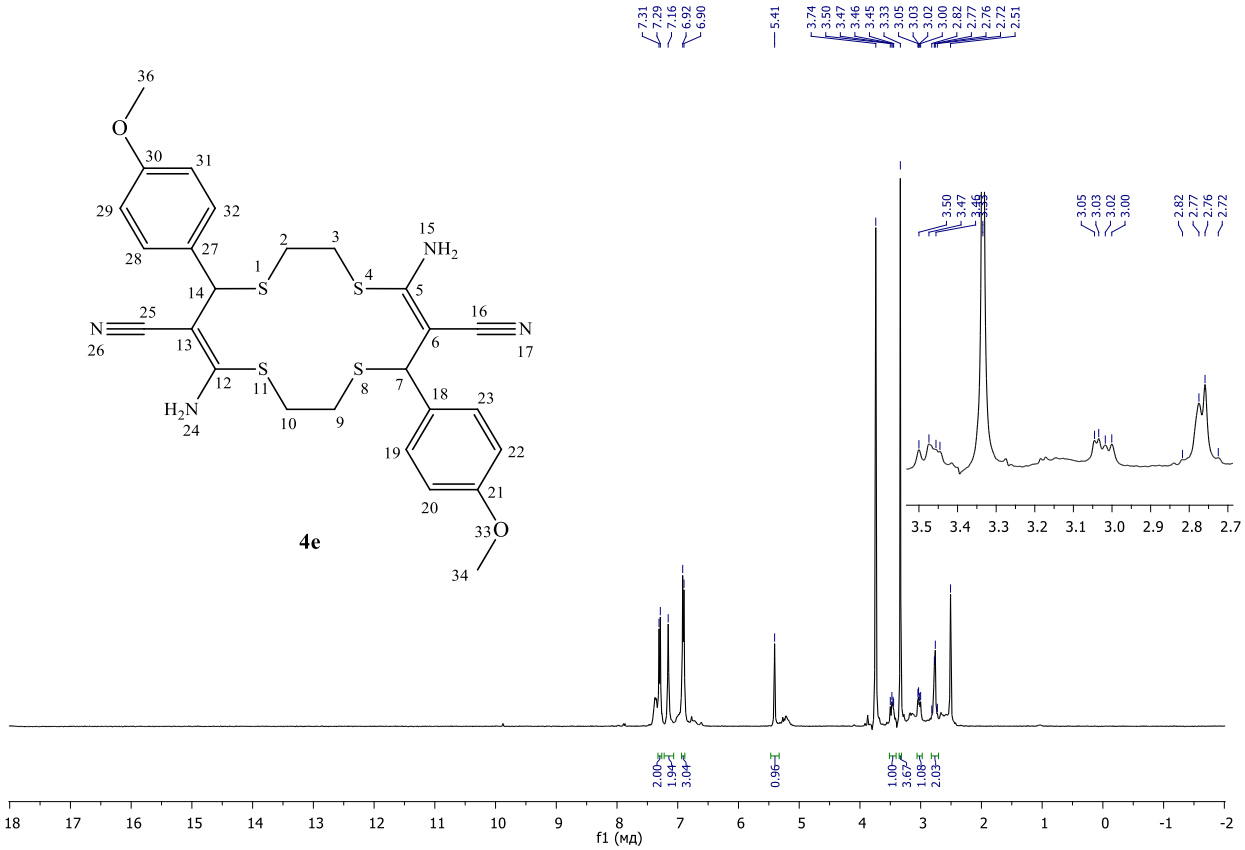

Fig. S9. ^{1}H NMR spectrum of compound **4e** in DMSO-D_6 (400 MHz)

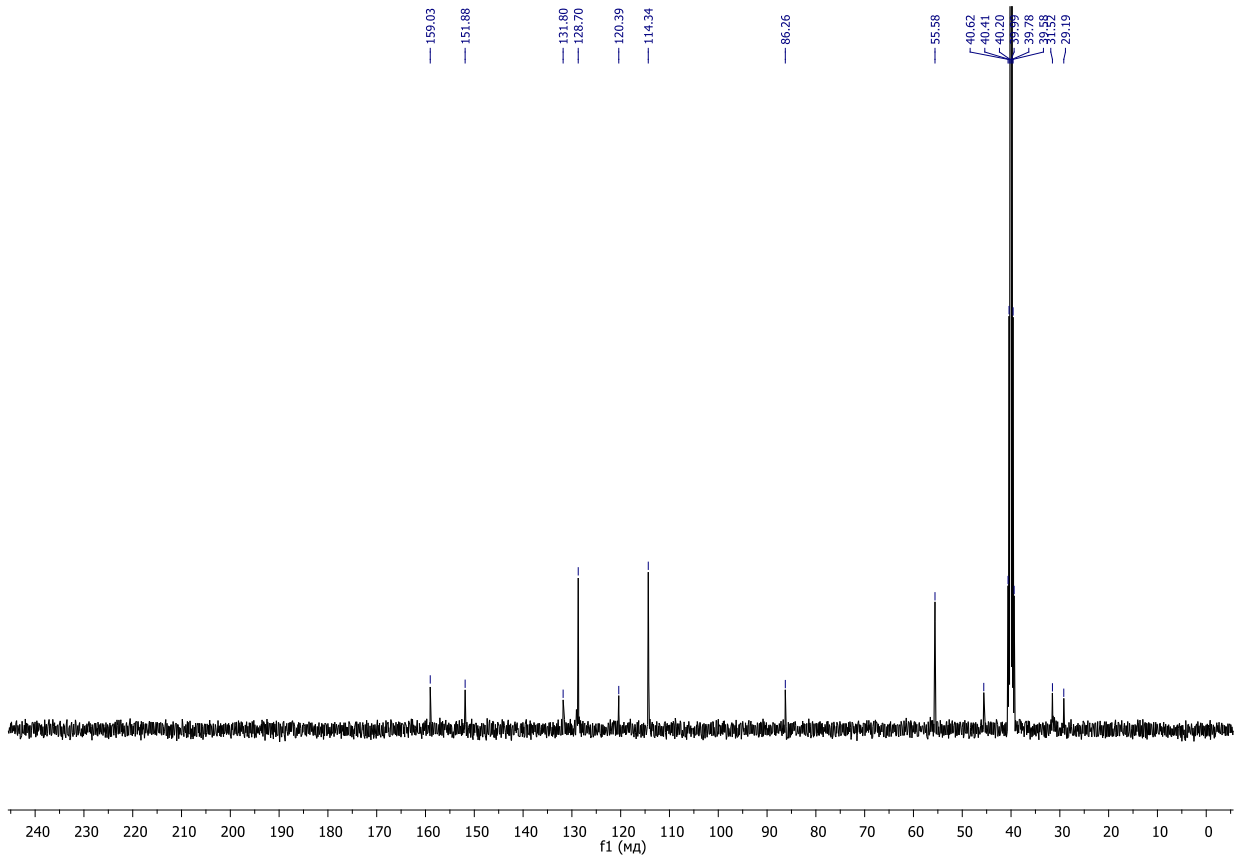

Fig. S10. ^{13}C NMR spectrum of compound **4e** in DMSO-D_6 (100 MHz)

5,12-Diamino-7,14-*bis*(3,4-dimethoxyphenyl)-1,4,8,11-tetrathiacyclotetradeca-5,12-diene-6,13-dicarbonitrile **4f**

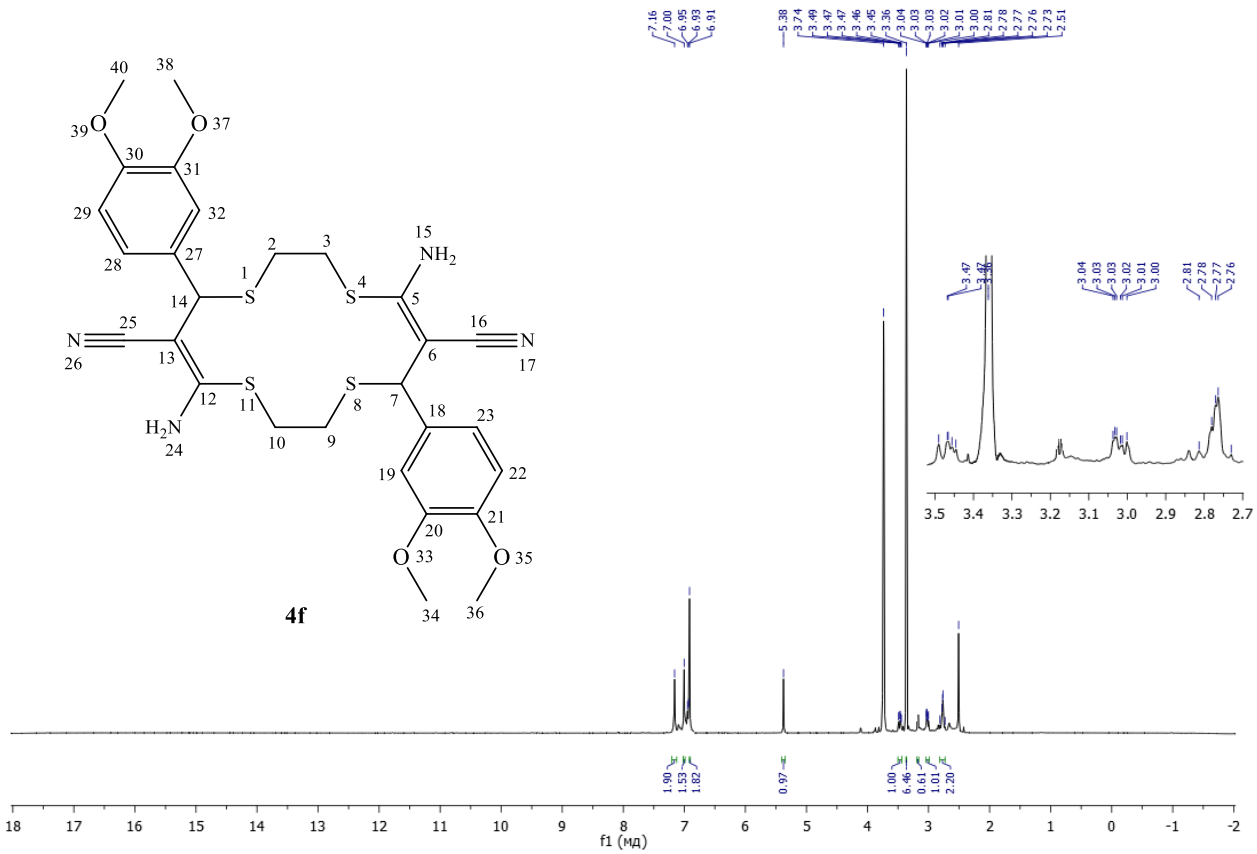

Fig. S11. ^{1}H NMR spectrum of compound **4f** in DMSO-D_6 (500 MHz)

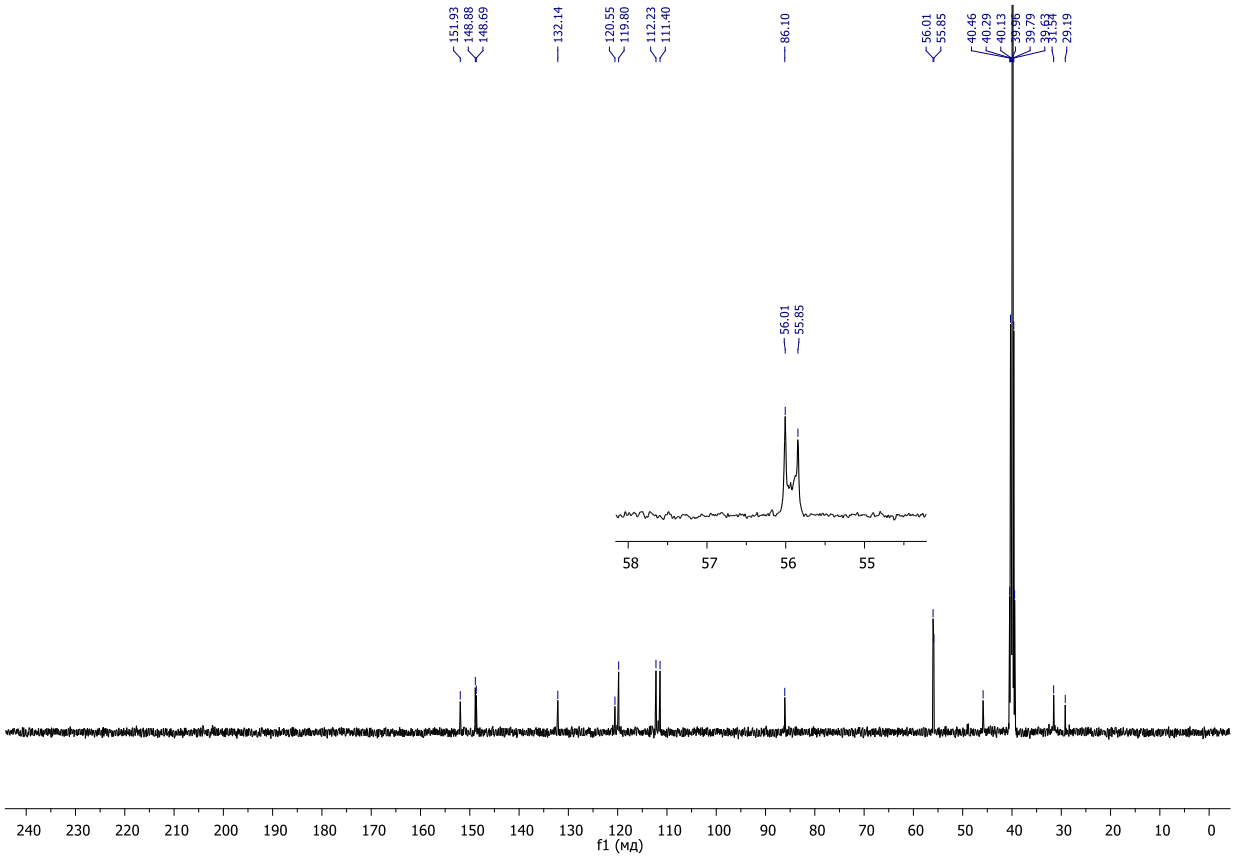

Fig. S12. ^{13}C NMR spectrum of compound **4f** in DMSO-D_6 (125 MHz)

5,12-Diamino-7,14-*bis*(4-methylphenyl)-1,4,8,11-tetrathiacyclotetradeca-5,12-diene-6,13-dicarbonitrile **4g**

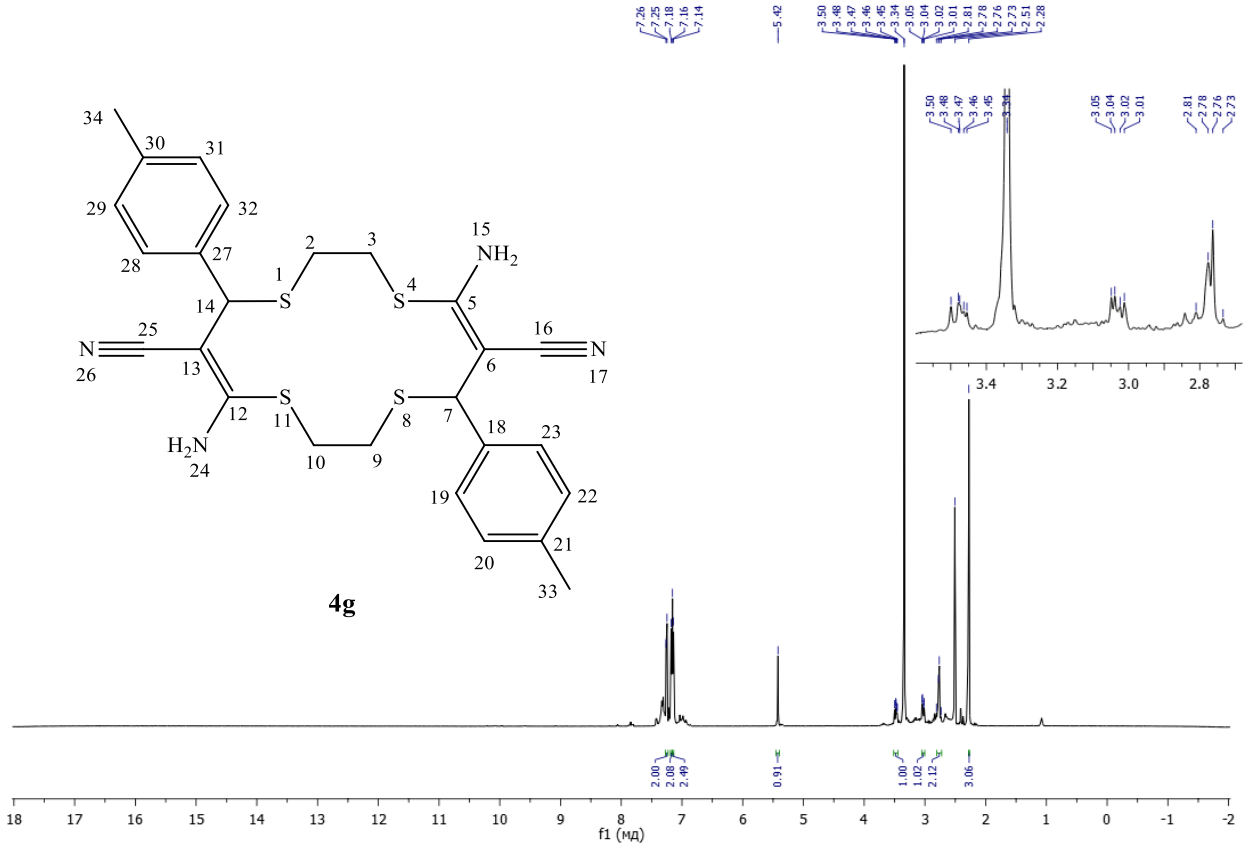

Fig. S13. ^{1}H NMR spectrum of compound **4g** in DMSO-D_6 (500 MHz)

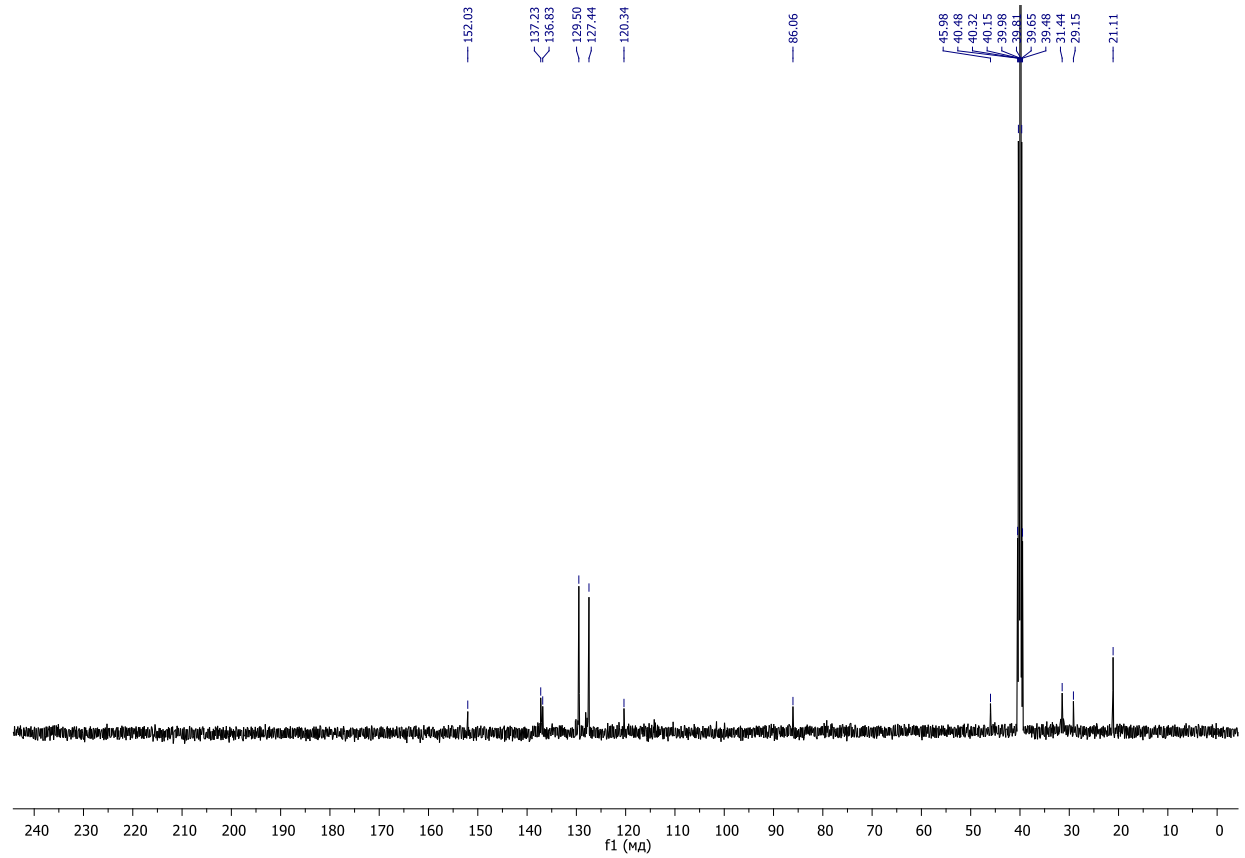

Fig. S14. ^{13}C NMR spectrum of compound **4g** in DMSO-D_6 (125 MHz)

5,12-Diamino-7,14-*bis*[4-(dimethylamino)phenyl]-1,4,8,11-tetrathiacyclotetradeca-5,12-diene-6,13-dicarbonitrile **4h**

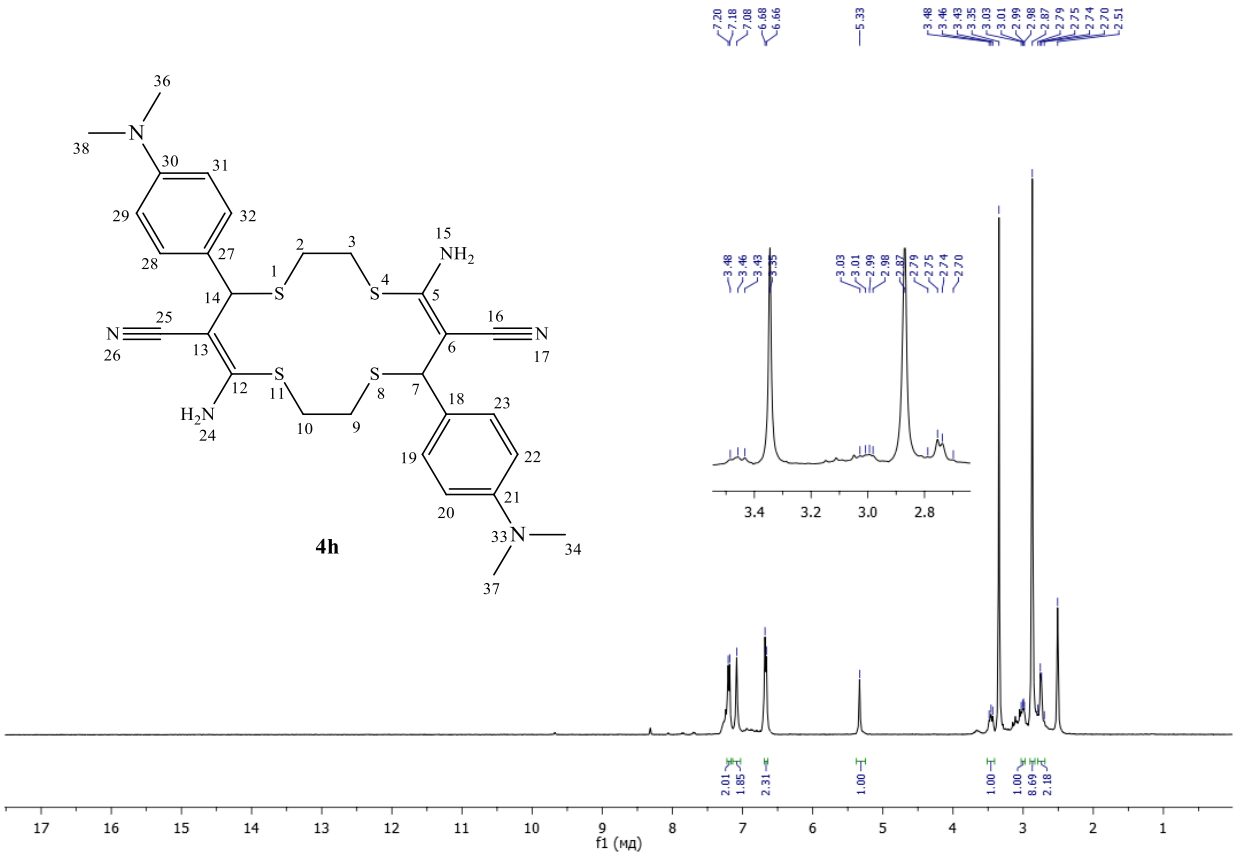

Fig. S15. ^{1}H NMR spectrum of compound **4h** in DMSO-D_6 (400 MHz)

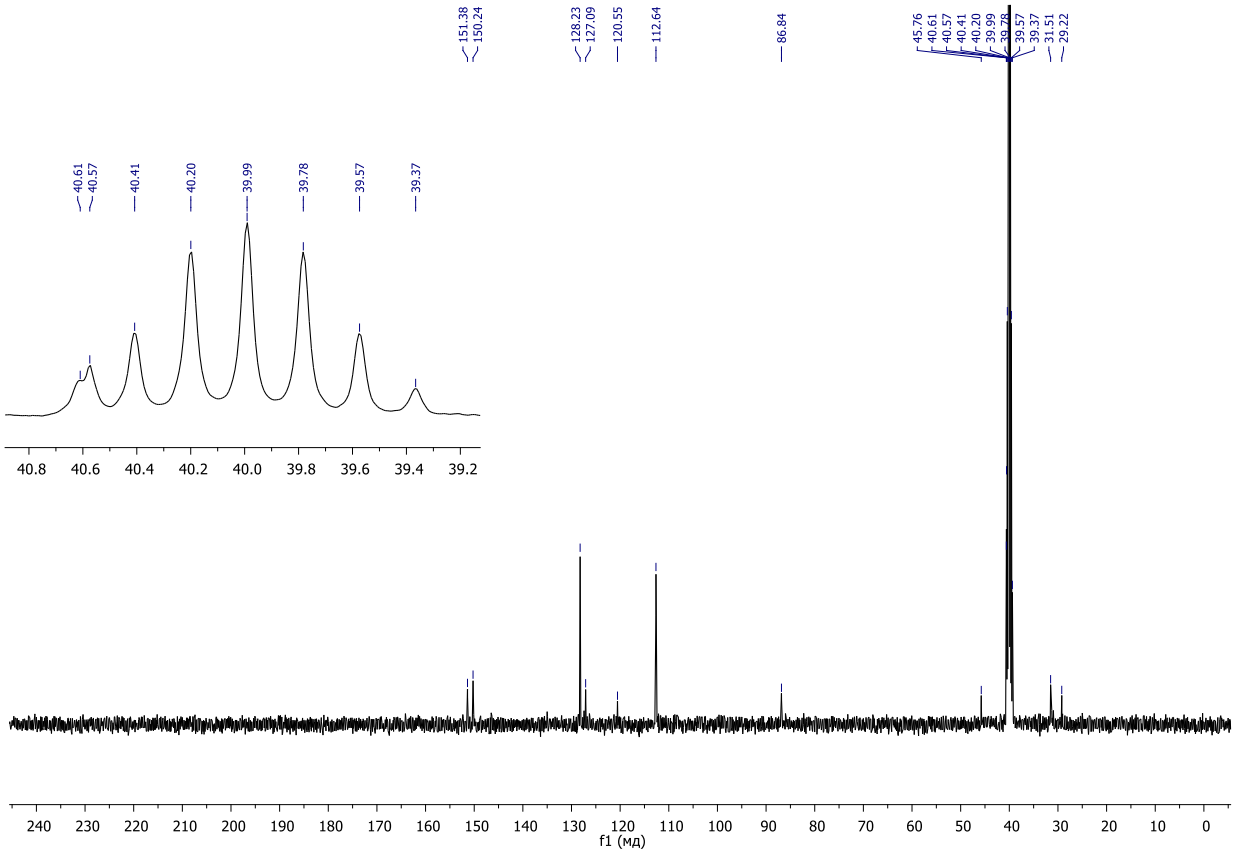

Fig. S16. ^{13}C NMR spectrum of compound **4h** in DMSO-D_6 (100 MHz)

5,12-Diamino-7,14-*bis*(1,3-benzodioxol-5-yl)-1,4,8,11-tetrathiacyclotetradeca-5,12-diene-6,13-dicarbonitrile **4i**

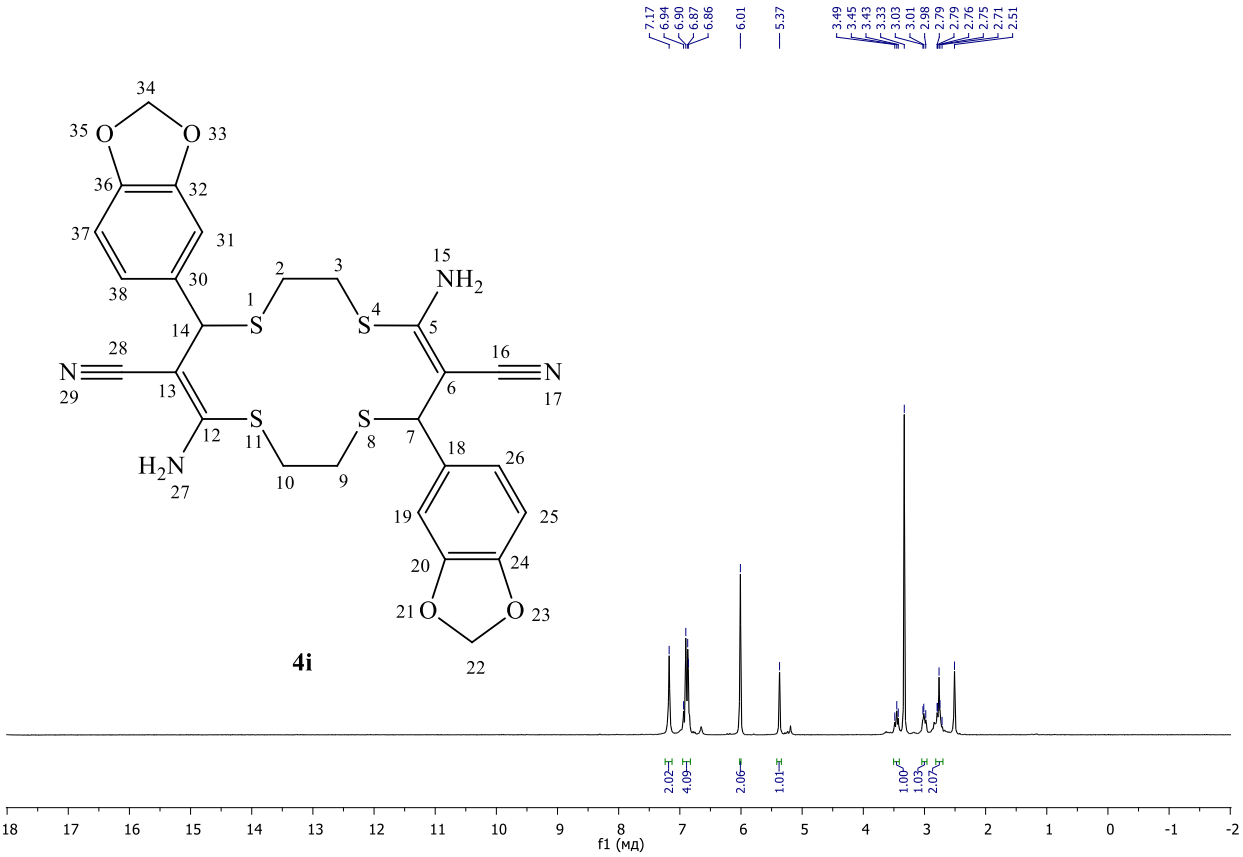

Fig. S17. ^{1}H NMR spectrum of compound **4i** in DMSO-D_6 (400 MHz)

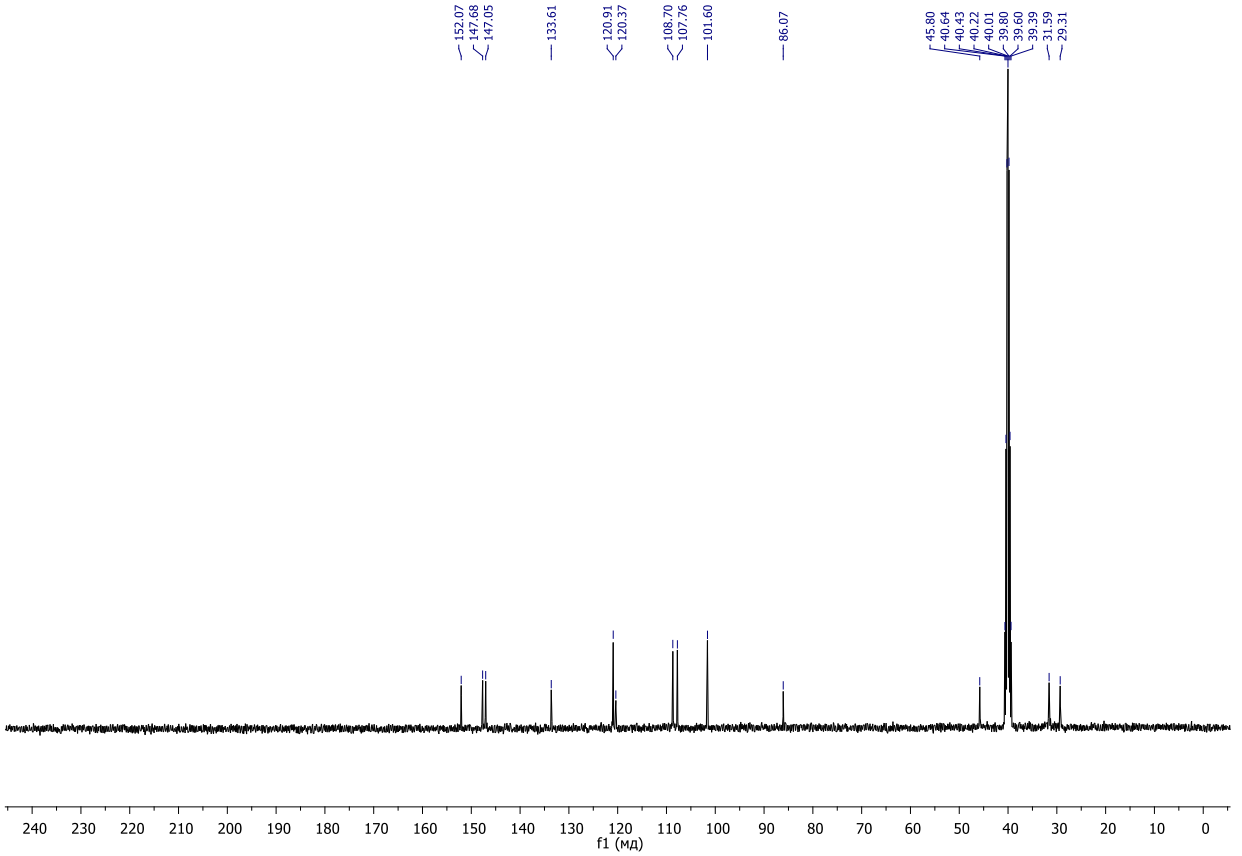

Fig. S18. ^{13}C NMR spectrum of compound **4i** in DMSO-D_6 (100 MHz)

Table S1. Crystal data and structure refinement of the compounds **4a**,**b**,**5**.

Parameters	**4a**	**4b**	**5**
Empirical formula	$C_{32}H_{46}F_2N_4O_4S_8$	$C_{36}H_{58}Cl_2N_4O_6S_{10}$	$C_{10}H_5FN_2$
Formula weight	845.21	1034.36	172.16
Temperature(K)	293(2)	293(2)	293(2)
Wavelength(Å)	0.71073	0.71073	0.71073
Crystal system	triclinic	orthorhombic	triclinic
Space group	P-1	Pbca	P-1
a (Å)	8.1311(4)	18.600(2)	6.9541(7)
b (Å)	11.9456(7)	9.7722(10)	7.3664(5)
c (Å)	12.1000(12)	28.091(6)	9.1492(10)
α (°)	103.820(7)	90	107.194(8)
β (°)	109.146(6)	90	99.036(9)
γ (°)	93.699(4)	90	102.848(8)
Volume ($Å^3$)	1064.98(14)	5105.9(13)	423.86(7)
Z	1	4	2
Density (g/cm^3)	1.318	1.346	1.349
μ, Absorption coefficient (mm^{-1})	0.466	0.580	0.099
F(000)	444.0	2176.0	176.0
Radiation	$MoK_\alpha(\lambda = 0.71073)$	$MoK_\alpha(\lambda = 0.71073)$	$MoK_\alpha(\lambda = 0.71073)$
2Theta range for the collection	4.372 to 58.398°	4.38 to 58.78°	4.8 to 62.168°
Index range	$10 \le h \le 9$, $-16 \le k \le 14$, $-14 \le l \le 16$	$-13 \le h \le 25$, $-10 \le k \le 12$, $-36 \le l \le 36$	$-8 \le h \le 9$, $-10 \le k \le 10$, $-13 \le l \le 10$
Reflection collected	8840	15833	3675
Independent reflections	4905 [*R*(int) = 0.0209]	5993 [*R*(int) = 0.1149]	2396[*R*(int) = 0.0132]
Goodness of fit on F^2	0.877	0.901	1.049
Final R indices [I>2sigma(I)]	$R_1 = 0.0477$, $wR_2 = 0.1315$	$R_1 = 0.0998$, $wR_2 = 0.2187$	$R_1 = 0.0460$, $wR_2 = 0.1190$
Largest diff. peak and hole ($eÅ^{-3}$)	0.34 and -0.37	0.44 and -0.25	0.21 and -0.18