Supporting information

for

Synthesis and cytostatic activity of 7-arylsulfanyl-7-deazapurine bases and ribonucleosides

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Contents:

1. Table of cytostatic activities
2. Experimental section
3. Copies of NMR spectra

Table S1. Cytostatic actitities of all compounds

	IC_{50} (μM)											
	A549	CCRF-CEM	CEM-DNR	HCT116	HCT116p53--	K562	K562-TAX	HepG2	HL60	HeLa S3	BJ	MRC-5
1b	> 50	> 50	> 50	> 50	> 50	> 50	> 50	>25	>25	>25	132.45 ± 27.23	141.14 ± 13.73
1c	48.80 ± 1.85	> 50	> 50	48.29 ± 2.01	48.58 ± 2.19	> 50	> 50	>25	>25	>25	141.72 ± 12.95	138.32 ± 15.75
1d	46.15 ± 3.02	42.40 ± 1.16	> 50	45.05 ± 4.18	44.98 ± 7.40	47.83 ± 3.37	> 50	>25	>25	>25	141.23 ± 8.31	121.28 ± 19.39
1e	> 50	> 50	> 50	> 50	> 50	> 50	> 50	>25	>25	>25	> 150	> 150
1f	> 50	> 50	> 50	> 50	> 50	> 50	> 50	>25	>25	>25	> 150	> 150
1g	49.09 ± 1.39	44.87 ± 1.50	48.14 ± 3.33	40.98 ± 3.79	43.67 ± 5.07	27.23 ± 5.22	49.36 ± 1.56	>25	>25	>25	140.22 ± 11.07	135.79 ± 18.48
1h	> 50	46.91 ± 4.22	> 50	45.03 ± 3.89	47.78 ± 3.69	49.34 ± 1.04	48.28 ± 2.30	>25	>25	>25	> 150	> 150
2b	16.19 ± 1.34	10.55 ± 2.90	17.67 ± 0.53	13.03 ± 3.03	5.06 ± 0.18	5.14 ± 0.18	21.66 ± 1.34	>25	21.1 ± 1.7	>25	23.38 ± 3.57	54.48 ± 9.06
2c	11.43 ± 2.61	7.73 ± 1.36	20.83 ± 1.72	6.75 ± 2.72	19.53 ± 1.23	4.26 ± 0.47	18.90 ± 0.85	>25	7.63 ± 1.1	8.49 ± 2.62	22.06 ± 2.77	32.87 ± 1.79
2d	64.23 ± 2.65	60.85 ± 7.39	56.77 ± 1.44	38.12 ± 32.56	29.10 ± 3.19	13.99 ± 0.59	64.63 ± 2.76	>25	>25	>25	> 150	135.50 ± 17.33
2e	19.80 ± 2.79	14.63 ± 0.55	35.25 ± 2.36	11.01 ± 3.70	27.54 ± 5.08	3.83 ± 0.25	22.14 ± 1.14	>25	>25	>25	144.56 ± 8.46	> 150
2f	28.58 ± 6.38	14.72 ± 0.73	26.15 ± 1.06	18.98 ± 2.77	45.30 ± 2.58	4.95 ± 0.39	21.00 ± 0.44	>25	13.5 ± 0.4	17.6 ± 2.9	132.24 ± 17.89	148.21 ± 2.77
2g	22.82 ± 1.91	16.68 ± 0.98	20.34 ± 1.34	22.79 ± 2.03	86.56 ± 2.40	17.88 ± 0.69	17.92 ± 0.74	>25	13.9 ± 0.6	17.9 ± 1.0	> 150	135.71 ± 22.15
2h	21.47 ± 2.33	18.23 ± 0.99	64.59 ± 2.11	17.15 ± 2.28	65.91 ± 6.13	50.33 ± 6.13	43.95 ± 2.24	>25	>25	23.9 ± 0.6	122.60 ± 26.55	148.13 ± 2.90
7b	62.50 ± 7.32	30.07 ± 2.55	53.04 ± 4.06	53.35 ± 6.10	50.76 ± 3.27	62.54 ± 1.85	65.89 ± 1.40	>25	>25	>25	98.17 ± 7.44	104.633.37
7c	> 50	> 50	> 50	> 50	> 50	> 50	> 50	>25	>25	>25	> 150	> 150
7d	76.41 ± 16.18	98.28 ± 1.89	78.57 ± 11.23	94.98 ± 5.51	> 100	78.79 ± 7.17	> 100	>25	>25	>25	112.55 ± 22.39	113.16 ± 24.20
7e	> 50	> 50	> 50	> 50	> 50	> 50	> 50	>25	>25	>25	> 150	> 150
7f	> 50	> 50	> 50	> 50	> 50	> 50	> 50	>25	>25	>25	> 150	> 150
7g	22.91 ± 3.14	33.96 ± 1.42	> 50	20.80 ± 3.59	22.41 ± 8.45	23.09 ± 6.24	29.62 ± 2.41	>25	>25	>25	67.88 ± 8.49	67.70 ± 14.43
7h	70.02 ± 24.37	68.85 ± 3.31	72.74 ± 2.25	85.0 ± 15.05	99.00 ± 1.55	84.96 ± 1.97	98.06 ± 3.02	>25	>25	>25	116.67 ± 25.82	133.33 ± 25.82
8b	76.96 ± 13.91	48.10 ± 2.91	75.21 ± 5.11	55.23 ± 7.98	47.19 ± 2.64	57.21 ± 3.97	70.45 ± 1.64	>25	>25	>25	109.58 ± 20.62	125.7210.94
8c	74.36 ± 11.67	58.93 ± 2.64	79.59 ± 4.71	81.65 ± 6.33	74.36 ± 3.15	80.81 ± 4.59	72.88 ± 4.64	>25	>25	>25	121.00 ± 6.63	145.70 ± 3.45
8d	83.49 ± 15.90	85.60 ± 5.24	88.42 ± 3.02	97.48 ± 4.37	> 100	96.64 ± 5.21	90.84 ± 4.68	>25	>25	>25	149.86 ± 0.34	> 150
8e	89.58 ± 12.73	96.21 ± 3.61	> 100	> 100	> 100	> 100	88.93 ± 5.14	>25	>25	>25	146.53 ± 8.49	> 150
8f	87.61 ± 14.76	96.69 ± 4.78	> 100	92.56 ± 7.75	> 100	> 100	81.85 ± 2.55	>25	>25	>25	> 150	> 150
8g	43.76 ± 4.93	64.66 ± 1.66	> 100	36.72 ± 7.81	23.18 ± 1.04	23.43 ± 1.13	55.77 ± 2.97	>25	>25	>25	93.59 ± 7.76	138.24 ± 10.63
8h	85.47 ± 6.43	89.29 ± 7.83	83.60 ± 2.00	98.80 ± 2.95	> 100	99.82 ± 0.43	86.69 ± 7.54	>25	>25	>25	141.79 ± 9.86	> 150

1. Experimental Section

General

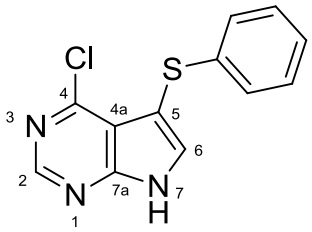
6-Chloro-7-deazapurine was purchased from commercial supplier and used without any further purification. Dry DMF and THF were used as received from supplier. All compounds were fully characterized by NMR and spectra were recorded on a Bruker Avance II 600 (^{1}H at 600.1 MHz, ^{13}C at 150.9 MHz) or on a Bruker Avance II 500 (499.8 or 500.0 MHz for ^{1}H and 125.7 MHz for ^{13}C) spectrometer or on a Bruker Avance II 400 (^{1}H at 400 MHz, ^{13}C at 100.6 MHz) ^{1}H and ^{13}C resonances were assigned using H,C-HSQC and H,C-HMBC spectra. The samples were measured in $CDCl_3$ or DMSO and chemical shifts (in ppm, δ-scale) were referenced to solvent signal ($\delta(^{1}H)$ = 7.26 ppm, $\delta(^{1}H)$ = 77.0 ppm) or in or DMSO ($\delta(^{1}H)$ = 2.50 ppm, $\delta(^{1}H)$ = 39.43 ppm) Coupling constants (*J*) are given in Hz. High performance flash chromatography (HPFC) were performed with Biotage SP1 apparatus on KP-Sil columns. Reverse phase - high performance flash chromatography (RP-HPFC) purifications were performed with Biotage SP1 apparatus on KP-C18-HS columns. Optical rotations were measured at 25 °C, $[\alpha]_D$ values are given in 10^{-1}degcm^{2}g^{-1}.IR spectra (wavenumbers in cm^{-1}) were recorded on Bruker Alpha FT-IR spectrometer using ATR technique. High resolution mass spectra were measured on a LTQ Orbitrap XL (Thermo Fisher Scientific) spectrometer using EI ionization technique. Melting points were determined on a Buchi Melting Point B-545 and are uncorrected. Elemental analyses were measured on PE 2400 Series II CHNS/O (Perkin Elmer, USA, 1999).

Preparation of starting compounds:

Sulfenytion of 7-deazapurines. General Procedure:

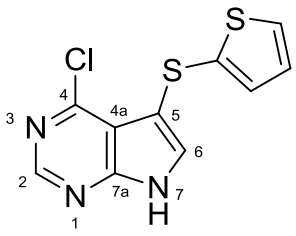
A mixture of 6-chloro-7-deazapurine (15.36 g, 100 mmol), disulphides (100 mmol), CuI (1.9 g, 10 mmol) and dtbpy (5.37 g, 20 mmol) in DMF (300 mL) was stirred at 110°C under oxygen for 18 hours until complete consumption of staring material as monitored by TLC. The solution was then cooled to room temperature, diluted with EtOAc (200 mL), washed with 1M solution of sodium salt of EDTA (100 mL). Aqueous solution was then extracted three times with EtOAc and combitated organic layers were dried over Na_2SO_4, filtered, and evaporated under vacuum. The crude product was purified by column chromatography on silica gel.

4-Chloro-5-(phenylsulfanyl)-7*H*-pyrrolo[2,3-*d*]pyrimidine
(6-chloro-7-(phenylsulfanyl)-7-deazapurine) (1a)



Diphenyldisulfide (21.83 g, 100 mmol) was used as starting compounds to give product **1a** (22.25 g, 85%) as yellowish solids. Chromatography was started with pure hexane (to remove excess of disulphide) and followed by hexane/EtOAc 5:1 to 1:1. Crystallization from ethanol gave white crystals. ^{1}H NMR was compared with published data[1].

4-Chloro-5-[(thiophen-2-yl)sulfanyl]-7*H*-pyrrolo[2,3-*d*]pyrimidine
(6-chloro-7-[(thiophen-2-yl)sulfanyl]-7-deazapurine) (2a)



2-Thienyl disulfide (23.04 g, 100 mmol) was used as starting compounds to give product **2a** (25.5 g, 95%) as white solids. Chromatography was started with pure hexane (to remove excess of disulphide) and followed by hexane/EtOAc 5:1 to 1:1

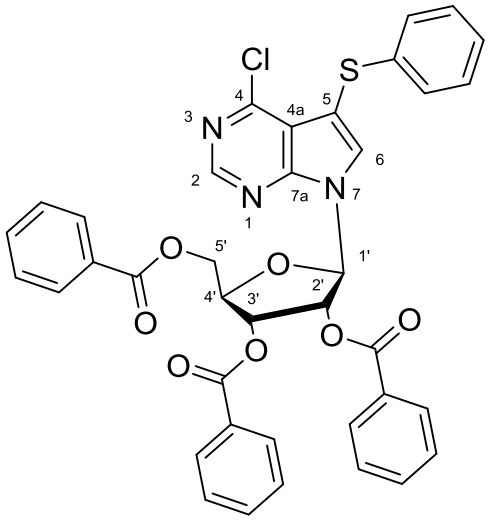
M.p. 176 °C. ^{1}H NMR (500 MHz, DMSO-d_6): 6.98 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-thienyl); 7.21 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-thienyl); 7.51 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-thienyl); 8.06 (s, 1H, H-6); 8.59 (s, 1H, H-2); 13.03 (bs, 1H, NH). ^{13}C NMR (125.7 MHz, DMSO-d_6): 104.01 (C-5); 115.45 (C-4a); 128.03 (CH-4-thienyl); 129.25 (CH-5-thienyl); 130.61 (CH-3-thienyl); 134.76 (CH-6); 136.71 (C-2-thienyl); 150.92 (C-4); 151.40 (CH-2); 152.76 (C-7a). IR (KBr): 3066, 2944, 2809, 2770, 1601, 1556, 1446, 1401, 1401, 1332, 1239, 1216, 1003, 973, 848, 716, 623. HRMS (ESI) calculated for $C_{10}H_7N_3ClS_2$: 267.9767; found: 267.9764.

Glycosylation of 7-sulfanyl-7-deazapurines. General Procedure:

7-Sulfanyl-7-deazapurine **1a-2a** (40 mmol) was suspended in acetonitrile (200 ml) and BSA (10.4 ml, 40 mmol) was added. Reaction mixture was stirred for 15 min at rt (during this time clear solution was formed). Then TMSOTf (14.46 ml, 80 mmol) and protected ribofuranose (20.2 g, 40 mmol) were added. Mixture was heated to 80 °C for 6 h. After cooling to rt, the mixture was extracted with EtOAc and water, organic layer was washed with $NaHCO_3$ and again with water, dried over $MgSO_4$ and evaporated under reduced pressure. Crude product was purified using column chromatography with chloroform.

4-Chloro-5-(phenylsulfanyl)-9-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-pyrrolo[2,3-d]pyrimidine

(6-chloro-7-(phenylsulfanyl)-9-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-7-deazapurine) (3a)



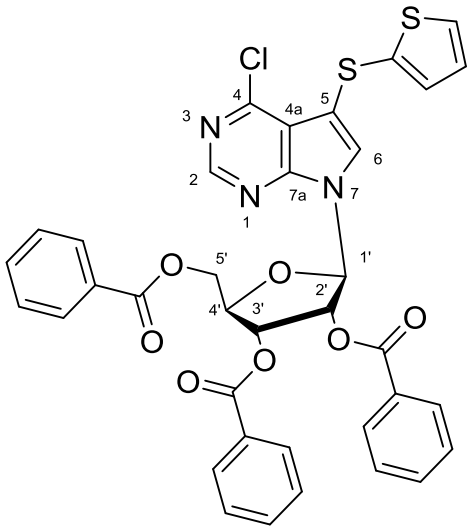
Reaction of **1a** (10.4 g, 40 mmol) according to the general procedure afforded compound **3a** (13.84 g, 49%) as yellowish foam.

M.p. 89 °C. ^{1}H NMR (600.1 MHz, $CDCl_3$): 4.71 (dd, 1H, J_{gem} = 12.3, $J_{5'b,4'}$ = 3.8, H-5'b); 4.82 (ddd, 1H, $J_{4',3'}$ = 4.7, $J_{4',5'}$ = 3.8, 3.1, H-4'); 4.89 (dd, 1H, J_{gem} = 12.3, $J_{5'a,4'}$ = 3.1, H-5'a); 6.14 (dd, 1H, $J_{3',2'}$ = 5.8, $J_{3',4'}$ = 4.7, H-3'); 6.23 (dd, 1H, $J_{2',3'}$ = 5.8, $J_{2',1'}$ = 5.4, H-2'); 6.66 (d, 1H, $J_{1',2'}$ = 5.4, H-1'); 7.12 (m, 2H, H-*o*-Ph); 7.13 (m, 1H, H-*p*-Ph); 7.21 (m, 2H, H-*m*-Ph); 7.37, 7.41, 7.42 (3 × m, 3 × 2H, H-*m*-Bz); 7.55, 7.59 (2 × m, 3H, H-*p*-Bz); 7.64 (s, 1H, H-6); 7.93, 8.01, 8.08 (3 × m, 3 × 2H, H-*o*-Bz); 8.58 (s, 1H, H-2). ^{13}C NMR (150.9 MHz, $CDCl_3$): 63.47 (CH_2-5'); 71.43 (CH-3'); 74.09 (CH-2'); 80.64 (CH-4'); 87.17 (CH-1'); 105.38 (C-5); 117.87 (C-4a); 125.92 (CH-*p*-Ph); 127.26 (CH-*o*-Ph); 128.38 (C-*i*-Bz); 128.53, 128.57 (CH-*m*-Bz); 128.67 (C-*i*-Bz); 128.68 (CH-*m*-Bz); 128.99 (CH-*m*-Ph); 129.21 (C-*i*-Bz); 129.66, 129.83, 129.84 (CH-*o*-Bz); 132.79 (CH-6); 133.52, 133.77, 133.81 (CH-*p*-Bz); 137.52 (C-*i*-Ph); 151.81 (CH-2); 152.48 (C-7a); 153.22 (C-4); 165.04, 165.35, 166.12 (CO-Bz). IR (KBr): 3123, 3058, 3028, 3004, 2947, 1727, 1601, 1574, 1541, 1452, 1263, 1123, 1090, 707. HRMS (ESI) calculated for $C_{38}H_{28}O_7N_3ClNaS$: 728.1229; found: 728.1233.

4-Chloro-5-(2-thienylsulfanyl)-9-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-pyrrolo[2,3-d]pyrimidine

(6-chloro-7-[(thiophen-2-yl)sulfanyl]-9-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-7-deazapurine) (4a)

To form clear solution double amount of BSA (20.8 ml, 80 mmol) was added.
Reaction of **2a** (10.71 g, 40 mmol) according to the general procedure afforded compound **4a** (8.5 g, 30%) as white foam.

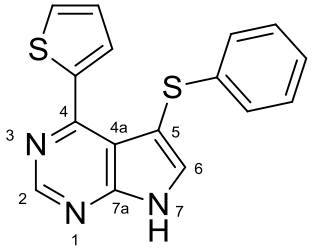


M.p. 72 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 4.67 (dd, 1H, J_{gem} = 12.2 Hz, $J_{5'a,4'}$ = 3.9 Hz, H-5′a); 4.79 (m, 1H, H-4′); 4.86 (dd, 1H, J_{gem} = 12.2 Hz, $J_{5'b,4'}$ = 3.1 Hz, H-5′b); 6.12 (dd, 1H, $J_{3',2'}$ = 5.8 Hz, $J_{3',4'}$ = 4.5 Hz, H-3'); 6.20 (t, 1H, $J_{2',1'} = J_{2',3'}$ = 5.7 Hz, H-2'); 6.61 (d, 1H, $J_{1',2'}$ = 5.6 Hz, H-1'); 6.89 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.15 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.25 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.36 and 7.40 (2×m, 2×2H, CH-*m*-Bz); 7.44 (s, 1H, H-6); 7.48 (m, 2H, H-*m*-Bz); 7.51 – 7.64 (m, 3H, H-*p*-Bz); 7.92, 7.99 and 8.10 (3×m, 3×2H, H-*o*-Bz); 8.57 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 63.62 (CH_2-5′); 71.41 (CH-3′); 74.01 (CH-2′); 80.59 (CH-4′); 87.04 (CH-1′); 109.91 (C-5); 117.06 (C-4a); 127.56 (CH-4-Sthienyl); 128.40 (C-*i*-Bz); 128.49 and 128.53 (CH-*m*-Bz); 128.68 (C-*i*-Bz); 128.70 (CH-*m*-Bz); 128.28 (C-*i*-Bz); 129.53 (CH-5-Sthienyl); 129.73, 129.80 and 129.82 (CH-*o*-Bz); 130.00 (CH-6); 132.70 (CH-3-Sthienyl); 133.52, 133.72 and 133.75 (CH-*p*-Bz); 133.86 (C-2-Sthienyl); 151.63 (CH-2); 151.97 (C-7a); 152.90 (C-4); 165.02, 165.32 and 166.09 (CO-Bz). IR (KBr): 3102, 3087, 3066, 3031, 3007, 2950, 1730, 1601, 1583, 1538, 1452, 1314, 1263, 1219, 1120, 1096, 1069, 707. HRMS (ESI) calculated for $C_{36}H_{26}O_7N_3ClNaS_2$: 734.0796; found: 734.0793.

General procedure for the Stille coupling

Compound **1a-4a** (1 equiv), tributylstannane (1.2 equiv) and $PdCl_2(PPh_3)_2$ (5 mol%) under argon atmosphere were dissolved in anhydrous DMF and heated to 100 °C for 8 h. Then, solvent was evaporated under reduced pressure and crude product was purified using HPFC.

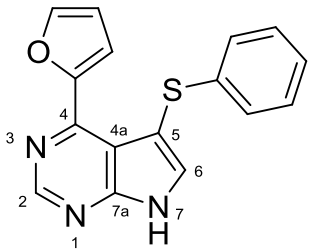
4-(Thiophen-2-yl)-5-(phenylsulfanyl)-7*H*-pyrrolo[2,3-*d*]pyrimidine (6-(thiophen-2-yl)-7-(phenylsulfanyl)-7-deazapurine) (1b)



Deazapurine **1a** (523 mg, 2 mmol), 2-(tributylstannyl)thiophene (0.762 mL, 2.4 mmol) and 15 mL DMF were used according to the general procedure. Crude product was purified using HPFC (hexane/EtOAc, 0–50% EtOAc) and product **1b** was obtained as yellowish solid (496 mg, 80%). Crystallization in ethanol/H_2O gave yellowish needles.

M.p. 240 °C. ^{1}H NMR (600.1 MHz, DMSO-d_6): 6.92 (m, 2H, H-*o*-Ph); 7.03 (m, 1H, H-*p*-Ph); 7.07 (dd, 1H, $J_{4,5}$ = 5.1, $J_{4,3}$ = 3.8, H-4-thienyl); 7.15 (m, 2H, H-*m*-Ph); 7.69 (dd, 1H, $J_{5,4}$ = 5.1, $J_{5,3}$ = 1.1, H-5-thienyl); 8.10 (s, 1H, H-6); 8.39 (dd, 1H, $J_{3,4}$ = 3.8, $J_{3,5}$ = 1.1, H-3-thienyl); 8.78 (s, 1H, H-2); 12.93 (bs, 1H, NH). ^{13}C NMR (150.9 MHz, DMSO-d_6): 98.90 (C-5); 113.95 (C-4a); 128.11 (CH-*p*-Ph); 125.64 (CH-*o*-Ph); 128.11 (CH-4-thienyl); 129.18 (CH-*m*-Ph); 130.66 (CH-5-thienyl); 131.87 (CH-3-thienyl); 136.84 (CH-6); 139.19 (C-*i*-Ph); 141.57 (C-2-thienyl); 151.15 (CH-2); 152.10 (C-4); 154.33 (C-7a). IR (KBr): 3105, 2986, 2869, 2827, 1595, 1541, 1479, 1431, 1308, 1260, 806, 740, 707. HRMS (ESI) calculated for $C_{16}H_{12}N_3S_2$: 310.0468; found: 310.0467. Anal. calculated for $C_{16}H_{11}N_3S_2 \cdot 0.15H_2O$: C, 61.57; H, 3.65; N, 13.46; S, 20.54. Found: C, 61.85; H, 3.55; N, 13.39; S, 20.26.

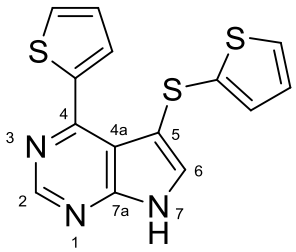
4-(Furan-2-yl)-5-(phenylsulfanyl)-7*H*-pyrrolo[2,3-*d*]pyrimidine
(6-(furan-2-yl)-7-(phenylsulfanyl)-7-deazapurine) (1c)



Deazapurine **1a** (523 mg, 2 mmol), 2-(tributylstannyl)furane (0.755 mL, 2.4 mmol) and 15 mL DMF were used according to the general procedure. Crude product was purified using HPFC (hexane/EtOAc, 0–50% EtOAc) and product **1c** was obtained as yellowish solid (510 mg, 87%). Crystallization in ethanol/H_2O gave yellowish needles.

M.p. 234 °C. ^{1}H NMR (500.0 MHz, DMSO-d_6): 7.58 (dd, 1H, $J_{4,3}$ = 3.4, $J_{4,5}$ = 1.7, H-4-furyl); 6.99 (m, 2H, H-*o*-Ph); 7.03 (m, 1H, H-*p*-Ph); 7.16 (m, 2H, H-*m*-Ph); 7.40 (dd, 1H, $J_{3,4}$ = 3.4, $J_{3,5}$ = 0.8, H-3-furyl); 7.70 (dd, 1H, $J_{5,4}$ = 1.7, $J_{5,3}$ = 0.8, H-5-furyl); 8.05 (s, 1H, H-6); 8.81 (s, 1H, H-2); 12.87 (bs, 1H, NH). ^{13}C NMR (150.9 MHz, DMSO-d_6): 99.51 (C-5); 112.27 (CH-4-furyl); 113.48 (C-4a); 114.77 (CH-3-furyl); 125.14 (CH-*p*-Ph); 125.65 (CH-*o*-Ph); 129.01 (CH-*m*-Ph); 136.57 (CH-6); 139.69 (C-*i*-Ph); 145.53 (CH-5-furyl); 147.73 (C-4); 150.92 (C-2-furyl); 151.32 (CH-2); 154.33 (C-7a). IR (KBr): 3108, 2989, 2869, 2821, 1580, 1541, 1479, 1443, 1314, 827, 731. HRMS (ESI) calculated for $C_{16}H_{12}ON_3S$: 294.0696; found: 294.0696. Anal. calculated for $C_{16}H_{11}N_3OS \cdot 0.25H_2O$: C, 64.52; H, 3.89; N, 14.11; S, 10.76. Found: C, 64.65; H, 3.73; N, 13.99; S, 10.47.

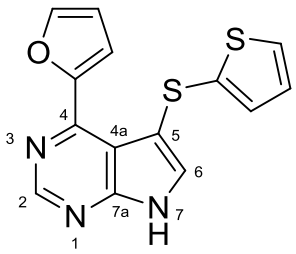
4-(Thiophen-2-yl)-5-[(thiophen-2-yl)sulfanyl]-7*H*-pyrrolo[2,3-*d*]pyrimidine
(6-(thiophen-2-yl)-7-[(thiophen-2-yl)sulfanyl]-7-deazapurine) (2b)



Deazapurine **2a** (535 mg, 2 mmol), 2-(tributylstannyl)thiophene (0.762 mL, 2.4 mmol) and 15 mL DMF were used according to the general procedure. Crude product was purified using HPFC (EtOAc/MeOH, 0–5% MeOH) and product **2b** was obtained as yellowish solid (360 mg, 57%).

M.p. 224 °C. ^{1}H NMR (500 MHz, DMSO-d_6): 6.78 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 6.84 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.29 (dd, 1H, $J_{4,5}$ = 5.1 Hz, $J_{4,3}$ = 3.7 Hz, H-4-thienyl); 7.39 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.84 (dd, 1H, $J_{5,4}$ = 5.1 Hz, $J_{5,3}$ = 1.1 Hz, H-5-thienyl); 8.07 (s, 1H, H-6); 8.44 (dd, 1H, $J_{3,4}$ = 3.7 Hz, $J_{3,5}$ = 1.1 Hz, H-3-thienyl); 8.76 (s, 1H, H-2); 12.83 (bs, 1H, NH). ^{13}C NMR (150.9 MHz, DMSO-d_6): 103.50 (C-5); 113.19 (C-4a); 127.76 (CH-4-Sthienyl); 128.21 (CH-4-thienyl); 129.08 (CH-5-Sthienyl); 129.85 (CH-3-Sthienyl); 160.73 (CH-5-thienyl); 132.46 (CH-3-thienyl); 135.19 (CH-6); 137.15 (C-2-Sthienyl); 141.41 (C-2-thienyl); 151.16 (CH-2); 152.01 (C-4); 153.80 (C-7a). IR (KBr): 2977, 2860, 2812, 1598, 1547, 1443, 1320, 809, 701. HRMS (ESI) calculated for $C_{14}H_{10}N_3S_3$: 316.0031; found: 316.0033.

4-(Furan-2-yl)-5-[(thiophen-2-yl)sulfanyl]-7*H*-pyrrolo[2,3-*d*]pyrimidine (6-(furan-2-yl)-7-[(thiophen-2-yl)sulfanyl]-7-deazapurine) (2c)

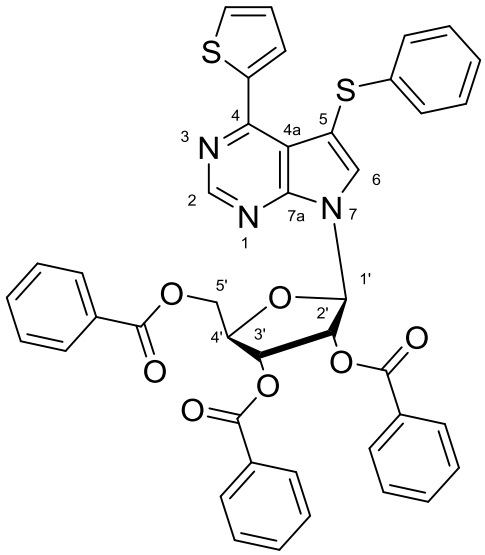


Deazapurine **2a** (535 mg, 2 mmol), 2-(tributylstannyl)furane (0.755 mL, 2.4 mmol) and 15 mL DMF were used according to the general procedure. Crude product was purified using HPFC (EtOAc/MeOH, 0–5% MeOH) and product **2c** was obtained as yellowish solid (434 mg, 72%).

M.p. 201°C. ^{1}H NMR (500 MHz, DMSO-d_6): 6.76 (dd, 1H, $J_{4,3}$ = 3.5 Hz, $J_{4,5}$ = 1.7 Hz, H-4-furyl); 6.92 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 6.99 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.45 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.48 (dd, 1H, $J_{3,4}$ = 3.5 Hz, $J_{3,5}$ = 0.8 Hz, H-3-furyl); 7.84 (s, 1H, H-6); 8.02 (dd, 1H, $J_{5,4}$ = 1.7 Hz, $J_{5,3}$ = 0.8 Hz, H-5-furyl); 8.77 (s, 1H, H-2); 12.70 (vbs, 1H, NH). ^{13}C NMR (125.7 MHz, DMSO-

d_6): 104.76 (C-5); 112.39 (C-4a); 112.56 (CH-4-furyl); 114. 86 (CH-3-furyl); 127.86 (CH-4-Sthienyl); 129.24 (CH-5-Sthienyl); 130.57 (CH-3-Sthienyl); 133.65 (CH-6); 136.87 (C-2-Sthienyl); 145.80 (CH-5-furyl); 147.52 (C-4); 151.18 (C-2-furyl); 151.26 (CH-2); 153.75 (C-7a). IR (KBr): 3105, 2989, 2860, 2830, 1601, 1586, 1532, 1317, 824, 749. HRMS (ESI) calculated for $C_{14}H_{10}ON_3S_2$: 300.0260; found: 300.0261.

4-(Thiophen-2-yl)-5-(phenylsulfanyl)-7-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine
(6-(thiophen-2-yl)-7-(phenylsulfanyl)-9-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-7-deazapurine) (3b)



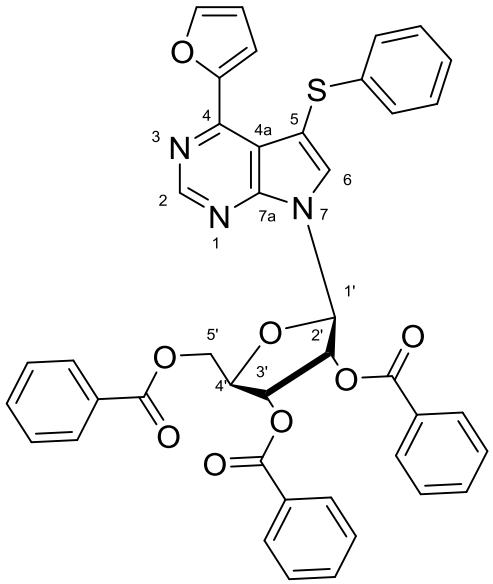
Nucleoside **3a** (706 mg, 1 mmol), 2-(tributylstannyl)thiophene (0.381 mL, 1.2 mmol) and 10 mL DMF were used according to the general procedure. Crude product was purified using HPFC (hexane/EtOAc, 0–20% EtOAc) and product **3b** was obtained as yellowish solid (540 mg, 72%).

M.p. 76 °C. ^{1}H NMR (500.0 MHz, $CDCl_3$): 4.73 (dd, 1H, J_{gem} = 12.2, $J_{5'b,4'}$ = 3.7, H-5'b); 4.83 (ddd, 1H, $J_{4',3'}$ = 4.6, $J_{4',5'}$ = 3.7, 3.1, H-4'); 4.90 (dd, 1H, J_{gem} = 12.2, $J_{5'a,4'}$ = 3.1, H-5'a); 6.16 (dd, 1H, $J_{3',2'}$ = 5.8, $J_{3',4'}$ = 4.6, H-3'); 6.26 (dd, 1H, $J_{2',3'}$ = 5.8, $J_{2',1'}$ = 5.6, H-2'); 6.80 (d, 1H, $J_{1',2'}$ = 5.6, H-1'); 6.90 (m, 2H, H-*o*-Ph); 7.01 (dd, 1H, $J_{4,5}$ = 5.1, $J_{4,3}$ = 3.8, H-4-thienyl); 7.02 (m, 1H, H-*p*-Ph); 7.07 (m, 2H, H-*m*-Ph); 7.36, 7.39, 7.41 (3 × m, 3 × 2H, H-*m*-Bz); 7.42 (dd, 1H, $J_{5,4}$ = 5.1, $J_{5,3}$ = 1.1, H-5-thienyl); 7.54, 7.55, 7.59 (3 × m, 3 × 1H, H-*p*-Bz); 7.71 (s, 1H,

H-6); 7.95, 8.02, 8.10 (3 × m, 3 × 2H, H-*o*-Bz); 8.20 (dd, 1H, $J_{3,4}$ = 3.8, $J_{3,5}$ = 1.1, H-3-thienyl); 8.84 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 63.63 (CH_2-5'); 71.50 (CH-3'); 74.04 (CH-2'); 80.48 (CH-4'); 86.54 (CH-1'); 104.35 (C-5); 115.63 (C-4a); 125.55 (CH-*p*-Ph); 126.70 (CH-*o*-Ph); 127.68 (CH-4-thienyl); 128.49, 128.53, 128.65 (C-*i*-Bz, CH-*m*-Bz); 128.78 (CH-*m*-Ph); 129.23 (C-*i*-Bz); 129.66, 129.83, 129.85 (CH-*o*-Bz); 129.93 (CH-5-thienyl); 132.57 (CH-3-thienyl); 133.24 (CH-6); 133.44, 133.72 (CH-*p*-Bz); 137.57 (C-*i*-Ph); 140.15 (C-2-thienyl); 151.64 (CH-2); 153.48 (C-7a); 154.15 (C-4); 165.07, 165.37, 166.15 (CO-Bz). IR (KBr): 3055, 3040, 3004, 2950, 2923, 1730, 1541, 1452, 1440, 1317, 1263, 1126, 1093, 1069, 1024, 704. HRMS (ESI) calculated for $C_{42}H_{32}O_7N_3S_2$: 754.1676; found: 754.1682.

4-(Furan-2-yl)-5-(phenylsulfanyl)-7-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine

(6-(furan-2-yl)-7-(phenylsulfanyl)-9-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-7-deazapurine) (3c)

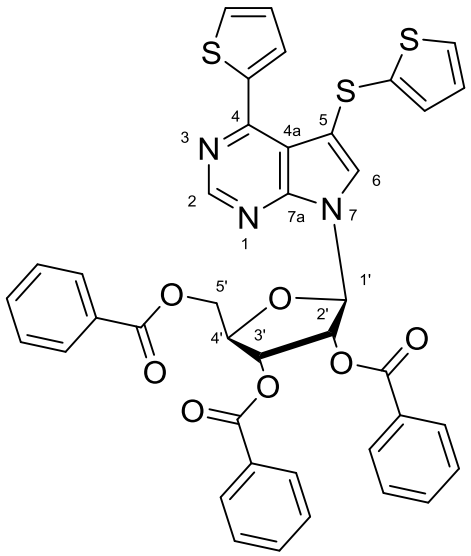


Nucleoside **3a** (706 mg, 1 mmol), 2-(tributylstannyl)furane (0.378 mL, 1.2 mmol) and 10 mL DMF were used according to the general procedure. Crude product was purified using HPFC (hexane/EtOAc, 0–20% EtOAc) and product **3c** was obtained as yellowish solid (677 mg, 92%).

M.p. 67 °C. ^{1}H NMR (500.0 MHz, $CDCl_3$): 4.72 (dd, 1H, J_{gem} = 12.2 Hz, $J_{5'a,4'}$ = 3.8 Hz, H-5′a); 4.82 (bdt, 1H, $J_{4',3'}$ = 4.6 Hz, $J_{4',5'a}$ = $J_{4',5'b}$ = 3.4 Hz, H-4′); 4.88 (dd, 1H, J_{gem} = 12.2 Hz,

$J_{5'b,4'}$ = 3.1 Hz, H-5′b); 6.15 (bdd, 1H, $J_{3',2'}$ = 5.9 Hz, $J_{3',4'}$ = 4.6 Hz, H-3'); 6.25 (t, 1H, $J_{2',1'}$ = $J_{2',3'}$ = 5.7 Hz, H-2'); 6.45 (dd, 1H, $J_{4,3}$ = 3.5 Hz, $J_{4,5}$ = 1.7 Hz, H-4-furyl); 6.77 (d, 1H, $J_{1',2'}$ = 5.5 Hz, H-1'); 7.01 (m, 2H, H-*o*-SPh); 7.04 (m, 1H, H-*p*-SPh); 7.12 (m, 2H, H-*m*-SPh); 7.34 – 7.44 (m, 6H, CH-*m*-Bz); 7.45 (dd, 1H, $J_{5,4}$ = 1.7 Hz, $J_{5,3}$ = 0.8 Hz, H-5-furyl); 7.57 (dd, 1H, $J_{3,4}$ = 3.5 Hz, $J_{3,5}$ = 0.8 Hz, H-3-furyl); 7.50 – 7.61 (m, 3H, H-*p*-Bz); 7.67 (s, 1H, H-6); 7.94, 8.01 and 8.09 (3×m, 3×2H, H-*o*-Bz); 8.87 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 63.62 (CH_2-5′); 71.48 (CH-3′); 74.05 (CH-2′); 80.44 (CH-4′); 86.60 (CH-1′); 104.34 (C-5); 112.04 (CH-4-furyl); 144.95 (C-4a); 116.15 (CH-3-furyl); 125.43 (CH-*p*-SPh); 126.46 (CH-*o*-SPh); 128.48 (C-*i*-Bz); 128.49, 128.54 and 128.64 (CH-*m*-Bz); 128.72 (C-*i*-Bz); 128.81 (CH-*m*-SPh); 129.23 (C-*i*-Bz); 129.66, 129.83 and 129.85 (CH-*o*-Bz); 133.39 (CH-6); 133.42, 133.71 and 133.72 (CH-*p*-Bz); 138.27 (C-*i*-SPh); 145.11 (CH-5-furyl); 149.26 (C-4); 150.26 (C-2-furyl); 151.84 (CH-2); 153.59 (C-7a); 165.06, 165.37 and 166.16 (CO-Bz).IR (KBr): 3117, 3063, 3031, 2959, 2920, 2857, 1730, 1538, 1452, 1317, 1263, 1120, 1093, 707. HRMS (ESI) calculated for $C_{42}H_{32}O_8N_3S$: 738.1905; found: 738.1908.

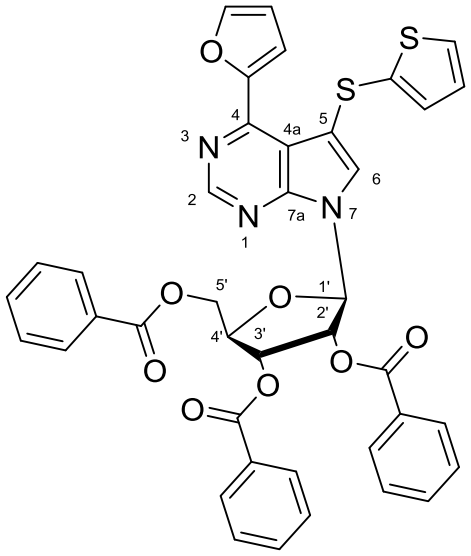
4-(Thiophen-2-yl)-5-[(thiophen-2-yl)sulfanyl]-7-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-pyrrolo[2,3-d]pyrimidine
(6-(thiophen-2-yl)-7-[(thiophen-2-yl)sulfanyl]-9-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-7-deazapurine) (4b)



Nucleoside **4a** (712 mg, 1 mmol), 2-(tributylstannyl)thiophene (0.381 mL, 1.2 mmol) and 10 mL DMF were used according to the general procedure. Crude product was purified using HPFC with pure DCM and product **4b** was obtained as yellowish solid (595 mg, 78%).

M.p. 77 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 4.70 (dd, 1H, J_{gem} = 12.2 Hz, $J_{5'a,4'}$ = 3.9 Hz, H-5′a); 4.81 (m, 1H, H-4′); 4.87 (dd, 1H, J_{gem} = 12.2 Hz, $J_{5'b,4'}$ = 3.1 Hz, H-5′b); 6.12 (dd, 1H, $J_{3',2'}$ = 5.8 Hz, $J_{3',4'}$ = 4.3 Hz, H-3'); 6.18 (t, 1H, $J_{2',1'}$ = $J_{2',3'}$ = 5.8 Hz, H-2'); 6.67 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 6.73 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 6.76 (d, 1H, $J_{1',2'}$ = 5.8 Hz, H-1'); 7.13 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.24 (dd, 1H, $J_{4,5}$ = 5.1 Hz, $J_{4,3}$ = 3.7 Hz, H-4-thienyl); 7.37, 7.41 and 7.49 (3×m, 3×2H, H-*m*-Bz); 7.52 – 7.65 (m, 4H, H-*p*-Bz, H-5-thienyl); 7.58 (s, 1H, H-6); 7.93, 8.00 and 8.14 (3×m, 3×2H, H-*o*-Bz); 8.26 (dd, 1H, $J_{3,4}$ = 3.8 Hz, $J_{3,5}$ = 1.2 Hz, H-3-thienyl); 8.86 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 63.78 (CH_2-5′); 71.53 (CH-3′); 74.07 (CH-2′); 80.63 (CH-4′); 86.42 (CH-1′); 109.61 (C-5); 114.89 (C-4a); 127.33 (CH-4-Sthienyl); 127.94 (CH-4-thienyl); 128.42 (C-*i*-Bz); 128.51 and 128.55 (CH-*m*-Bz); 128.71 (C-*i*-Bz); 128.76 (CH-*m*-Bz); 129.32 (C-*i*-Bz); 129.42 (CH-5-Sthienyl); 129.77, 129.84 and 129.87 (CH-*o*-Bz); 130.77 (CH-5-thienyl); 131.07 (CH-6); 132.19 (CH-3-Sthienyl); 133.45 (CH-3-thienyl); 133.54, 133.74 and 133.76 (CH-*p*-Bz); 133.5 (C-2-thienyl); 134.09 (C-2-Sthienyl); 150.91 (CH-2); 153.03 (C-4,7a); 165.08, 165.38 and 166.14 (CO-Bz). IR (KBr): 3108, 3060, 3037, 3007, 2953, 2926, 2851, 1727, 1550, 1455, 1317, 1269, 1126, 1090, 1066, 1030, 713. HRMS (ESI) calculated for $C_{40}H_{30}O_7N_3S_3$: 760.1240; found: 760.1243.

4-(Furan-2-yl)-5-[(thiophen-2-yl)sulfanyl]-7-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine
(6-(furan-2-yl)-7-[(thiophen-2-yl)sulfanyl]-9-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-7-deazapurine) (4c)



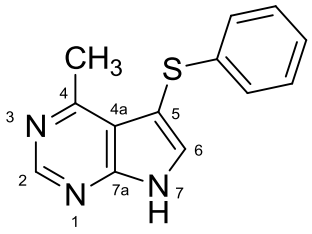
Nucleoside **4a** (712 mg, 1 mmol), 2-(tributylstannyl)furane (0.378 mL mg, 1.2 mmol) and 10 mL DMF were used according to the general procedure Crude product was purified using HPFC with pure DCM and product **4c** obtained as yellowish solid (303 mg, 41%).

M.p. 64 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 4.67 (dd, 1H, J_{gem} = 12.0 Hz, $J_{5'a,4'}$ = 3.9 Hz, H-5′a); 4.78 (m, 1H, H-4′); 4.81 (dd, 1H, J_{gem} = 12.0 Hz, $J_{5'b,4'}$ = 3.2 Hz, H-5′b); 6.12 (dd, 1H, $J_{3',2'}$ = 5.8 Hz, $J_{3',4'}$ = 4.4 Hz, H-3'); 6.19 (t, 1H, $J_{2',1'}$ = $J_{2',3'}$ = 5.7 Hz, H-2'); 6.64 (dd, 1H, $J_{4,3}$ = 3.5 Hz, $J_{4,5}$ = 1.8 Hz, H-4-furyl); 6.70 (d, 1H, $J_{1',2'}$ = 5.7 Hz, H-1'); 6.81 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 6.93 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.18 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.34 (s, 1H, H-6); 7.36, 7.40 and 7.48 (3×m, 3×2H, H-*m*-Bz); 7.51 - 7.62 (m, 3H, H-*p*-Bz); 7.60 (dd, 1H, $J_{3,4}$ = 3.5 Hz, $J_{3,5}$ = 0.9 Hz, H-3-furyl); 7.74 (dd, 1H, $J_{5,4}$ = 1.8 Hz, $J_{5,3}$ = 0.9 Hz, H-5-furyl); 7.93, 7.99 and 8.12 (3×m, 3×2H, H-*o*-Bz); 8.83 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 63.88 (CH_2-5′); 71.53 (CH-3′); 73.99 (CH-2′); 80.39 (CH-4′); 86.44 (CH-1′); 110.33 (C-5); 112.30 (CH-4-furyl); 113.79 (C-4a); 115.70 (CH-3-furyl); 127.47 (CH-4-Sthienyl); 128.45 and 128.51 (CH-*m*-Bz); 128.57 (C-*i*-Bz); 128.67 (CH-*m*-Bz); 128.80 (C-*i*-Bz); 129.16 (CH-6); 129.40 (C-*i*-Bz); 129.49 (CH-5-Sthienyl); 129.79, 129.82 and 129.86 (CH-*o*-Bz); 131.61 (CH-3-Sthienyl); 133.42 and 133.65 (CH-*p*-Bz); 134.30 (C-2-Sthienyl); 145.11 (CH-5-furyl); 148.86 (C-4); 150.90 (C-2-furyl); 151.70 (CH-2); 153.18 (C-7a); 165.05, 165.35 and 166.13 (CO-Bz). IR (KBr): 3102, 3055, 3034, 3004, 2956, 2926, 2866, 2851, 1733, 1562, 1535, 1452, 1269, 1123, 1099, 713. HRMS (ESI) calculated for $C_{40}H_{30}O_8N_3S_2$: 744.1469; found: 744.1471.

General procedure for methylation

Me_3Al (3 equiv, 2 M in toluene) was added to solution of compound **1a-4a** (1 equiv), and $Pd(PPh_3)_4$ (5 mol%) in THF. The reaction mixture was stirred at 70 °C for 12 h. Then the solution was dropped in water (decomposition of Me_3Al) and extracted three times with EtOAc and combitated organic layers were dried over Na_2SO_4, filtered, and evaporated under vacuum. The crude product was purified by HPFC.

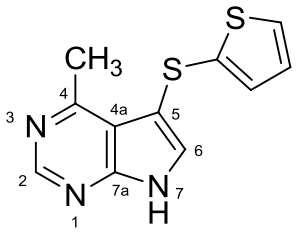
4-Methyl-5-(phenylsulfanyl)-7*H*-pyrrolo[2,3-*d*]pyrimidine
(6-methyl-7-(phenylsulfanyl)-7-deazapurine) (1d)



Deazapurine **1a** (523 mg, 2 mmol), Me_3Al (3 mL, 6 mmol, 2 M in toluene) and 40 mL THF were used according to the general procedure. Crude product was purified using HPFC (hexane/EtOAc, 0–50% EtOAc) and product **1d** was obtained as yellowish solid (355 mg, 73%). Crystallization in ethanol/H_2O gave yellowish needles.

M.p. 230 °C. ^{1}H NMR (500.0 MHz, DMSO-d_6): 2.60 (s, 3H, CH_3); 7.03 (m, 2H, H-*o*-Ph); 7.12 (m, 1H, H-*p*-Ph); 7.25 (m, 2H, H-*m*-Ph); 7.95 (s, 1H, H-6); 8.66 (s, 1H, H-2); 12.64 (bs, 1H, NH). ^{13}C NMR (125.7 MHz, DMSO-d_6): 20.89 (CH_3); 98.18 (C-5); 117.17 (C-4a); 125.46 (CH-*o*-Ph); 125.49 (CH-*p*-Ph); 129.44 (CH-*m*-Ph); 134.49 (CH-6); 139.52 (C-*i*-Ph); 151.82 (CH-2); 152.26 (C-7a); 159.36 (C-4). IR (KBr): 3123, 2986, 2842, 1577, 1473, 1434, 1332, 1263, 1227, 737. HRMS (ESI) calculated for $C_{13}H_{12}N_3S$: 242.0746; found: 242.0747. Anal. calculated for $C_{13}H_{11}N_3S \cdot 0.25H_2O$: C, 63.52; H, 4.72; N, 17.09; S, 13.04. Found: C, 63.48; H, 4.49; N, 16.93; S, 13.28.

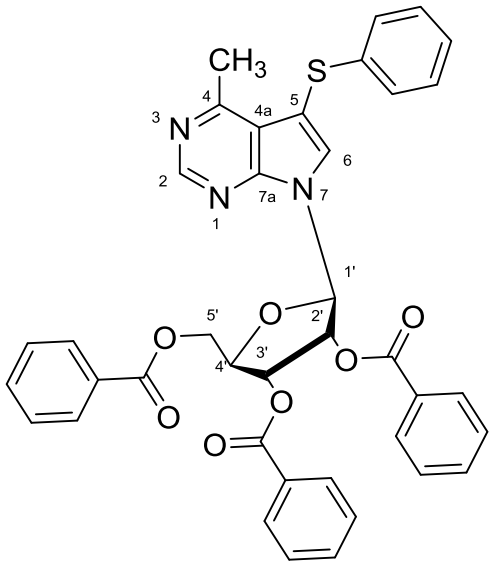
4-Methyl-5-[(thiophen-2-yl)sulfanyl]-7*H*-pyrrolo[2,3-*d*]pyrimidine
(6-methyl-7-[(thiophen-2-yl)sulfanyl]-7-deazapurine) (2d)



Deazapurine **2a** (535 mg, 2 mmol), Me_3Al (3 mL, 6 mmol, 2 M in toluene) and 40 mL THF were used according to the general procedure. Crude product was purified using HPFC (EtOAc/MeOH, 0–5% MeOH) and product **2d** was obtained as yellowish solid (325 mg, 66%).

M.p. 198 °C. ^{1}H NMR (500 MHz, DMSO-d_6): 2.81 (s, 3H, CH_3-4); 6.98 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.12 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.47 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.94 (s, 1H, H-6); 8.64 (s, 1H, H-2); 12.55 (vbs, 1H, NH). ^{13}C NMR (125.7 MHz, DMSO-d_6): 21.54 (CH_3-4); 103.13 (C-5); 116.39 (C-4a); 128.08 (CH-4-Sthienyl); 128.37 (CH-5-Sthienyl); 128.77 (CH-3-Sthienyl); 133.34 (CH-6); 138.22 (C-2-Sthienyl); 151.72 (C-7a); 151.74 (CH-2); 159.23 (C-4). IR (KBr): 3075, 2962, 2803, 2773, 2753, 2702, 2576, 1598, 2574, 1434, 1410, 1338, 1132, 1006, 696, 626. HRMS (ESI) calculated for $C_{11}H_{10}N_3S_2$: 248.0311; found: 248.0311.

4-Methyl-5-(phenylsulfanyl)-7-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine
(6-methyl-7-(phenylsulfanyl)-9-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-7-deazapurine) (3d)

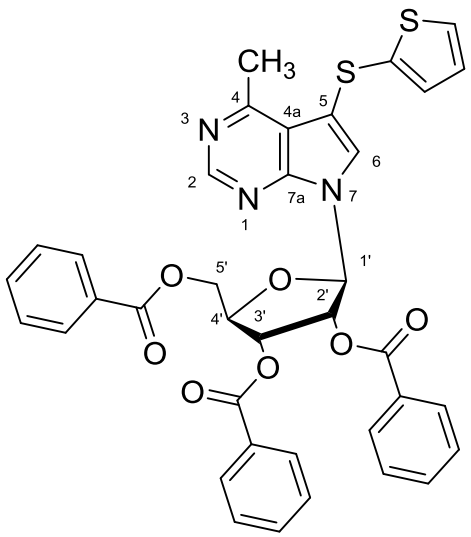


Nucleoside **3a** (706 mg, 1 mmol), Me_3Al (1.5 mL, 3 equiv, 2 M in toluene) and 20 mL THF were used according to the general procedure. Crude product was purified using HPFC (hexane/EtOAc, 0–20% EtOAc) and product **3d** was obtained as white foam (380 mg, 55%).

M.p. 61 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 2.69 (s, 3H, CH_3); 4.71 (dd, 1H, J_{gem} = 12.2 Hz, $J_{5'a,4'}$ = 3.9 Hz, H-5′a); 4.81 (bdt, 1H, $J_{4',3'}$ = 4.7 Hz, $J_{4',5'a}$ = $J_{4',5'b}$ = 3.6 Hz, H-4′); 4.89 (dd, 1H, J_{gem} = 12.2 Hz, $J_{5'b,4'}$ = 3.2 Hz, H-5′b); 6.16 (dd, 1H, $J_{3',2'}$ = 5.9 Hz, $J_{3',4'}$ = 4.7 Hz, H-3'); 6.24 (t, 1H, $J_{2',1'}$ = $J_{2',3'}$ = 5.7 Hz, H-2'); 6.71 (d, 1H, $J_{1',2'}$ = 5.5 Hz, H-1'); 7.05 (m, 2H, H-*o*-SPh); 7.11 (m, 1H, H-*p*-SPh); 7.20 (m, 2H, H-*m*-SPh); 7.37, 7.40 and 7.41 (3×m, 3×2H, H-*m*-Bz); 7.53, 7.55 and 7.58 (3×m, 3×1H, H-*p*-Bz); 7.61 (s, 1H, H-6); 7.94, 8.01 and 8.07 (3×m, 3×2H, H-*o*-Bz); 8.73 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 21.24 (CH_3); 63.59 (CH_2-5′); 71.50 (CH-3′); 74.10 (CH-2′); 80.44 (CH-4′); 86.71 (CH-1′); 104.19 (C-5); 118.59 (C-4a); 125.56 (CH-*p*-SPh); 126.14 (CH-*o*-SPh); 128.50 (CH-*m*-Bz); 128.52 (C-*i*-Bz); 128.54 and 128.64 (CH-*m*-Bz); 128.877 (C-*i*-Bz); 129.09 (CH-*m*-SPh); 129.31 (C-*i*-Bz); 129.69, 129.84 and 129.86 (CH-*o*-Bz); 132.14 (CH-6); 133.43, 133.71 and 133.73 (CH-*p*-Bz); 138.34 (C-*i*-SPh); 151.82 (C-7a); 152.28 (CH-2); 161.52 (C-4); 165.09, 165.38 and 166.17 (CO-Bz). IR (KBr): 3058, 3028, 3007, 2956, 2926, 2869, 2854, 1730, 1571, 1449, 1263, 1120, 1096, 1069, 1027, 707. HRMS (ESI) calculated for $C_{39}H_{32}O_7N_3S$: 686.1956; found: 686.1958.

4-Methyl-5-[(thiophen-2-yl)sulfanyl]-7-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine

(6-methyl-7-[(thiophen-2-yl)sulfanyl]-9-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-7-deazapurine) (4d)



Nucleoside **4a** (712 mg, 1 mmol), Me_3Al (1.5 mL, 3 equiv, 2 M in toluene) and 20 mL THF were used according to the general procedure. Crude product was purified using HPFC (DCM/MeOH, 0–5% MeOH) and product **4d** was obtained as white foam (469 mg, 67%).

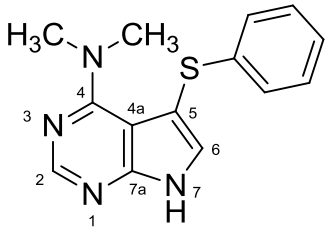
M.p. 59 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 2.92 (s, 3H, CH_3); 4.69 (dd, 1H, J_{gem} = 12.2 Hz, $J_{5'a,4'}$ = 3.9 Hz, H-5′a); 4.79 (m, 1H, H-4′); 4.87 (dd, 1H, J_{gem} = 12.2 Hz, $J_{5'b,4'}$ = 3.2 Hz, H-5′b); 6.15 (dd, 1H, $J_{3',2'}$ = 5.9 Hz, $J_{3',4'}$ = 4.5 Hz, H-3'); 6.21 (t, 1H, $J_{2',1'} = J_{2',3'}$ = 5.7 Hz, H-2'); 6.68 (d, 1H, $J_{1',2'}$ = 5.5 Hz, H-1'); 6.88 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 6.99 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.20 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.36, 7.40 and 7.47 (3×m, 3×2H, H-*m*-Bz); 7.53 (s, 1H, H-6); 7.51 – 7.62 (m, 3H, H-*p*-Bz); 7.93, 7.99 and 8.12 (3×m, 3×2H, H-*o*-Bz); 8.72 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 21.94 (CH_3); 63.71 (CH_2-5′); 71.51 (CH-3′); 74.05 (CH-2′); 80.43 (CH-4′); 86.60 (CH-1′); 108.20 (C-5); 118.00 (C-4a); 127.51 (CH-4-Sthienyl); 128.19 (CH-5-Sthienyl); 128.46 and 128.52 (CH-*m*-Bz); 128.56 (C-*i*-Bz); 128.67 (CH-*m*-Bz); 128.80 and 129.41 (C-*i*-Bz); 129.76, 129.83 and 129.85 (CH-*o*-Bz); 130.06 (CH-6); 130.34 (CH-3-Sthienyl); 133.44 and 133.67 (CH-*p*-Bz); 136.26 (C-2-Sthienyl); 151.38 (C-7a); 152.18 (CH-2); 161.21 (C-4); 165.07, 165.36 and 166.15 (CO-Bz). IR (KBr): 3111, 3066, 3028, 3007,

2932, 2851, 1730, 1568, 1452, 1317, 1269, 1177, 1123, 1093, 1069, 1024, 713. HRMS (ESI) calculated for $C_{37}H_{30}O_7N_3S_2$: 692.1520; found: 692.1521.

General procedure for dimethylamination

Dimethylamine (3 equiv, 2 M in THF) was added to solution of compound **1a-4a** (1 equiv), in propan-2-ol (25 mL) and the reaction mixture was stirred at 70 °C for 24 h. Volatiles were removed under reduced pressure and crude product was purified by HPFC.

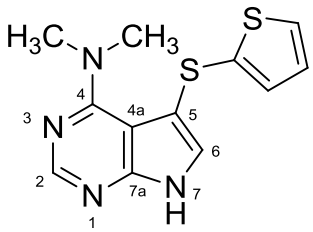
4-(*N*,*N*-Dimethylamino)-5-(phenylsulfanyl)-7*H*-pyrrolo[2,3-*d*]pyrimidine (6-(*N*,*N*-Dimethylamino)-7-(phenylsulfanyl)-7-deazapurine) (1e)



Deazapurine **1a** (523 mg, 2 mmol) was used according to the general procedure. Crude product was purified using HPFC (hexane/EtOAc, 0–50% EtOAc) and product **1e** was obtained as yellowish solid (454 mg, 84%). Crystallization in ethanol/H_2O gave white needles.

M.p. 201 °C. ^{1}H NMR (600.1 MHz, DMSO-d_6): 3.11 (s, 6H, $(CH_3)_2N$); 7.00 (m, 2H, H-*o*-Ph); 7.09 (m, 1H, H-*p*-Ph); 7.23 (m, 2H, H-*m*-Ph); 7.66 (s, 1H, H-6); 8.21 (s, 1H, H-2); 12.36 (bs, 1H, NH). ^{13}C NMR (150.9 MHz, DMSO-d_6): 41.23 ($(CH_3)_2N$); 98.32 (C-5); 104.52 (C-4a); 125.15 (CH-*o*-Ph); 125.21 (CH-*p*-Ph); 129.22 (CH-*m*-Ph); 131.94 (CH-6); 140.13 (C-*i*-Ph); 150.85 (CH-2); 153.58 (C-7a); 159.41 (C-4). IR (KBr): 3090, 2968, 2863, 2818, 1589, 1559, 1488, 1416, 1398, 1063, 922, 860 743. HRMS (ESI) calculated for $C_{14}H_{15}N_4S$: 271.1012; found: 271.1012. Anal. calculated for: $C_{14}H_{14}N_4S$: C, 62.20; H, 5.22; N, 20.72; S, 11.86; found: C, 61.97; H, 5.18; N, 20.64; S, 11.73.

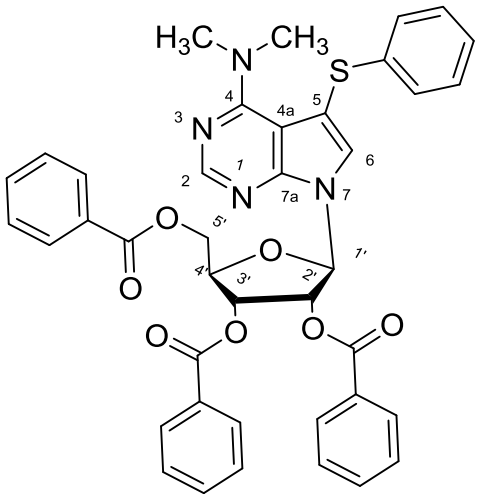
4-(*N*,*N*-Dimethylamino)-5-[(thiophen-2-yl)sulfanyl]-7*H*-pyrrolo[2,3-*d*]pyrimidine (6-(*N*,*N*-Dimethylamino)-7-[(thiophen-2-yl)sulfanyl]-7-deazapurine) (2e)



Deazapurine **2a** (535 mg, 2 mmol) was used according to the general procedure. Crude product was purified using HPFC (EtOAc/MeOH, 0–5% MeOH) and product **2e** was obtained as brownish solid (346 mg, 63%).

M.p. 185 °C. ^{1}H NMR (401 MHz, DMSO-d_6): 3.22 (s, 6H, $(CH_3)_2N$); 6.95 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.07 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.44 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.62 (s, 1H, H-6); 8.19 (s, 1H, H-2); 12.23 (bs, 1H, NH). ^{13}C NMR (100.8 MHz, DMSO-d_6): 41.45 ($(CH_3)_2N$); 103.09 (C-5); 103.91 (C-4a); 127.95 (CH-4-Sthienyl); 128.47 (CH-5-Sthienyl); 129.14 (CH-3-Sthienyl); 130.17 (CH-6); 138.47 (C-2-Sthienyl); 150.82 (CH-2); 153.12 (C-7a); 159.48 (C-4). IR (KBr): 3081, 2941, 2860, 2806, 1589, 1559, 1416, 1401, 1060, 928, 848, 692. HRMS (ESI) calculated for $C_{12}H_{13}N_4S_2$: 277.0576; found: 277.0576.

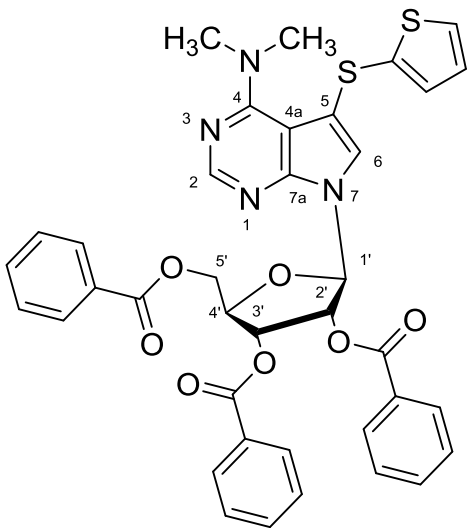
4-(*N*,*N*-Dimethylamino)-5-(phenylsulfanyl)-7-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine (6-(*N*,*N*-Dimethylamino)-7-(phenylsulfanyl)-9-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-7-deazapurine) (3e)



Nucleoside **3a** (706 mg, 1 mmol) was used according to the general procedure. Crude product was purified using HPFC (hexane/EtOAc, 0–20% EtOAc) and product **3e** was obtained as white foam (624 mg, 88%).

M.p. 67 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 3.17 (s, 6H, $(CH_3)_2N$); 4.70 (dd, 1H, J_{gem} = 12.1 Hz, $J_{5'a,4'}$ = 3.8 Hz, H-5′a); 4.78 (bdt, 1H, $J_{4',3'}$ = 4.6 Hz, $J_{4',5'a}$ = $J_{4',5'b}$ = 3.5 Hz, H-4′); 4.85 (dd, 1H, J_{gem} = 12.1 Hz, $J_{5'b,4'}$ = 3.2 Hz, H-5′b); 6.11 (dd, 1H, $J_{3',2'}$ = 5.9 Hz, $J_{3',4'}$ = 4.6 Hz, H-3'); 6.18 (t, 1H, $J_{2',1'}$ = $J_{2',3'}$ = 5.8 Hz, H-2'); 6.75 (d, 1H, $J_{1',2'}$ = 5.6 Hz, H-1'); 7.01 (m, 2H, H-*o*-SPh); 7.08 (m, 1H, H-*p*-SPh); 7.17 (m, 2H, H-*m*-SPh); 7.33 – 7.42 (m, 6H, CH-*m*-Bz); 7.45 (s, 1H, H-6); 7.48 – 7.60 (m, 3H, H-*p*-Bz); 7.95, 7.98 and 8.08 (3×m, 3×2H, H-*o*-Bz); 8.33 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 41.17 ($(CH_3)_2N$); 63.54 (CH_2-5′); 71.32 (CH-3′); 73.79 (CH-2′); 80.03 (CH-4′); 85.94 (CH-1′); 102.88 (C-5); 105.52 (C-4a); 125.21 (CH-*p*-SPh); 125.70 (CH-*o*-SPh); 128.44, 128.48 and 128.58 (CH-*m*-Bz); 128.64 and 128.81 (C-*i*-Bz); 128.88 (CH-*m*-SPh); 129.35 (CH-6); 129.67, 129.83 and 129.88 (CH-*o*-Bz); 133.30 and 133.61 (CH-*p*-Bz); 138.79 (C-*i*-SPh); 151.13 (CH-2); 152.95 (C-7a); 159.55 (C-4); 164.83, 165.14 and 165.94 (CO-Bz). IR (KBr): 3123, 3058, 3031, 3010, 2950, 2926, 2881, 2806, 1727, 1565, 1544, 1455, 1419, 1401, 1317, 1263, 1126, 1096, 1072, 1027, 707.. HRMS (ESI) calculated for $C_{40}H_{35}O_7N_4S$: 715.2221; found: 715.2223.

4-(*N*,*N*-Dimethylamino)-5-[(thiophen-2-yl)sulfanyl]-7-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-pyrrolo[2,3-d]pyrimidine
(6-(*N*,*N*-Dimethylamino)-7-[(thiophen-2-yl)sulfanyl]-9-(2,3,5-tri-O-benzoyl-β-D-ribofuranosyl)-7-deazapurine) (4e)



Nucleoside **4a** (712 mg, 1 mmol was used according to the general procedure. Crude product was purified using HPFC (DCM/MeOH, 0–5% MeOH) and product **4e** was obtained as white foam (634 mg, 88%).

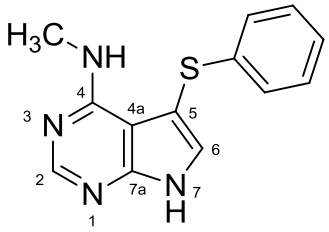
M.p. 81 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 3.29 (s, 6H, $(CH_3)_2N$); 4.67 (dd, 1H, J_{gem} = 12.1 Hz, $J_{5'a,4'}$ = 3.9 Hz, H-5′a); 4.75 (m, 1H, H-4′); 4.82 (dd, 1H, J_{gem} = 12.1 Hz, $J_{5'b,4'}$ = 3.2 Hz, H-5′b); 6.09 (dd, 1H, $J_{3',2'}$ = 5.8 Hz, $J_{3',4'}$ = 4.4 Hz, H-3'); 6.15 (t, 1H, $J_{2',1'} = J_{2',3'}$ = 5.8 Hz, H-2'); 6.70 (d, 1H, $J_{1',2'}$ = 5.7 Hz, H-1'); 6.83 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 6.93 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.16 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.32 (s, 1H, H-6); 7.36, 7.38 and 7.47 (3×m, 3×2H, H-*m*-Bz); 7.53, 7.56 and 7.59 (3×m, 3×1H, H-*p*-Bz); 7.95, 7.96 and 8.14 (3×m, 3×2H, H-*o*-Bz); 8.33 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 41.64 $(CH_3)_2N$); 63.94 (CH_2-5′); 71.55 (CH-3′); 73.98 (CH-2′); 80.23 (CH-4′); 86.05 (CH-1′); 105.34 (C-5); 107.91 (C-4a); 126.92 (CH-6); 127.38 (CH-4-Sthienyl); 128.13 (CH-5-Sthienyl); 128.42, 128.46 and 128.65 (CH-*m*-Bz); 128.70, 128.84 and 129.48 (C-*i*-Bz); 129.79, 129.82 and 129.89 (CH-*o*-Bz); 130.28 (CH-3-Sthienyl); 133.35 and 133.58 (CH-*p*-Bz); 136.59 (C-2-Sthienyl); 151.37 (CH-2); 152.82 (C-7a); 160.04 (C-4); 165.07, 165.37 and 166.18 (CO-Bz). IR (KBr): 3066, 2926, 2887, 2854, 1724, 1562, 1544,

1449, 1407, 1317, 1266, 1123, 1096, 1069, 1024, 710. HRMS (ESI) calculated for $C_{38}H_{33}O_7N_4S_2$: 721.1785; found: 721.1787.

General procedure for methylamination

Compound **1a-4a** (1 equiv), aq. methylamine (40% [w/w], 5 mL) in dioxane (5 mL) was stirred at autoclave at 120 °C for 18 h. Solvent was then evaporated under reduced pressure and crude products were purified using RP-HPFC (0→100% of MeOH in H_2O)

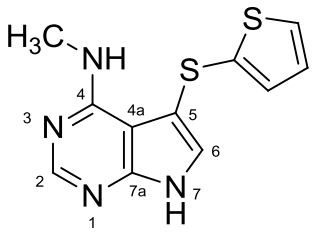
4-(*N*-Methylamino)-5-(phenylsulfanyl)-7*H*-pyrrolo[2,3-*d*]pyrimidine (6-(*N*-Methylamino)-7-(phenylsulfanyl)-7-deazapurine) (1f)



Reaction of deazapurine **1a** (523 mg, 2 mmol) according to the general procedure afforded compound **1f** as brownish solid (423 mg, 83 %). Crystallization in ethanol/H_2O gave yellowish needles.

M.p. 230 °C. ^{1}H NMR (500 MHz, DMSO-d_6): 2.90 (d, 3H, $J_{CH3,NH}$ = 4.8 Hz, C**H**$_3$NH); 6.48 (q, 1H, $J_{NH,CH3}$ = 4.8 Hz, CH_3N**H**); 7.09 (m, 2H, H-*o*-Ph); 7.13 (m, 1H, H-*p*-Ph); 7.26 (m, 2H, H-*m*-Ph); 7.55 (s, 1H, H-6); 8.19 (s, 1H, H-2); 12.19 (vbs, 1H, NH). ^{13}C NMR (125.7 MHz, DMSO-d_6): 27.62 (CH_3NH); 97.87 (C-5); 103.25 (C-4a); 125.71 (CH-*p*-Ph); 126.02 (CH-*o*-Ph); 129.33 (CH-*m*-Ph); 129.59 (CH-6); 139.07 (C-*i*-Ph); 150.99 (C-7a); 152.67 (CH-2); 157.08 (C-4). IR (KBr): 3374, 3099, 3058, 2962, 2902, 2860, 2812, 1607, 1586, 1491, 1485, 1383, 881, 737. HRMS (ESI) calculated for $C_{13}H_{13}N_4S$: 257.0855 ; found: 257.0855. Anal. calculated for $C_{13}H_{12}N_4S$: C, 60.91; H, 4.72; N, 21.86; S, 12.51 ; found: C, 60.66; H, 4.71; N, 21.75; S, 12.17.

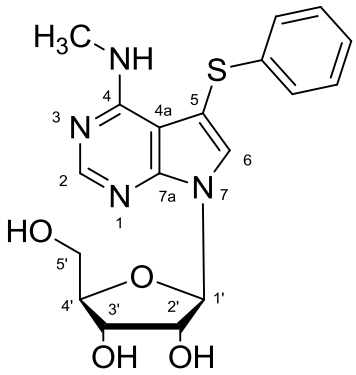
4-(*N*-Methylamino)-5-[(thiophen-2-yl)sulfanyl]-7*H*-pyrrolo[2,3-*d*]pyrimidine (6-(*N*-Methylamino)-7-[(thiophen-2-yl)sulfanyl]-7-deazapurine) (2f)



Reaction of deazapurine **2a** (535 mg, 2 mmol) according to the general procedure afforded compound **2f** as brownish solid (303 mg, 58 %).

M.p. 212 °C. ^{1}H NMR (401 MHz, DMSO-d$_6$): 3.01 (d, 3H, $J_{CH3,NH}$ = 4.8 Hz, C**H$_3$**NH); 6.71 (q, 1H, $J_{NH,CH3}$ = 4.8 Hz, CH$_3$N**H**); 6.97 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.24 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.49 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.54 (s, 1H, H-6); 8.17 (s, 1H, H-2); 12.10 (bs, 1H, NH). ^{13}C NMR (100.8 MHz, DMSO-d$_6$): 27.74 (CH$_3$NH); 102.26 (C-5); 102.53 (C-4a); 127.97 (CH-4-Sthienyl); 128.50 (CH-6); 129.19 (CH-5-Sthienyl); 129.92 (CH-3-Sthienyl); 137.86 (C-2-Sthienyl); 150.46 (C-7a); 152.43 (CH-2); 156.76 (C-4). IR (KBr): 3392, 3102, 3060, 2995, 2965, 2905, 2863, 2788, 1607, 1595, 1488, 1413, 1383, 1350, 1314, 881, 626. HRMS (ESI) calculated for $C_{11}H_{11}N_4S_2$: 263.0420 ; found: 263.0420.

4-(*N*-Methylamino)-5-(phenylsulfanyl)-7-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine (6-(*N*-Methylamino)-7-(phenylsulfanyl)-9-β-D-ribofuranosyl)-7-deazapurine) (3f)

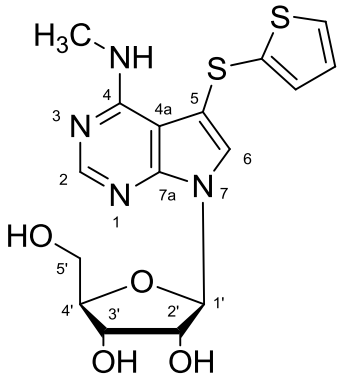


Reaction of nucleoside **3a** (706 mg, 1 mmol) according to the general procedure afforded compound **3f** as white solid (352 mg, 90 %). Crystallization in MeOH/H_2O gave white foam.

M.p. 157 °C. $[\alpha]_D$ −57.9 (0.21). ^{1}H NMR (600.1 MHz, DMSO-d_6): 2.91 (d, 3H, $J_{CH3,NH}$ = 4.8 Hz, C**H$_3$**NH); 3.55 (ddd, 1H, J_{gem} = 12.0 Hz, $J_{5'a,OH}$ = 6.2 Hz, $J_{5'a,4'}$ = 3.7 Hz, H-5′a); 3.65 (ddd, 1H, J_{gem} = 12.0 Hz, $J_{5'b,OH}$ = 5.0 Hz, $J_{5'b,4'}$ = 3.7 Hz, H-5′b); 3.92 (q, 1H, $J_{4',5'a}$ = $J_{4',5'b}$ = $J_{4',3'}$ = 3.5 Hz, H-4′); 4.10 (td, 1H, $J_{3',2'}$ = $J_{3',OH}$ = 5.0 Hz, $J_{3',4'}$ = 3.2 Hz, H-3'); 4.43 (td, 1H, $J_{2',1'}$ = $J_{2',OH}$ = 6.2 Hz, $J_{2',3'}$ = 5.1 Hz, H-2'); 5.14 (d, 1H, $J_{OH,3'}$ = 4.9 Hz, OH-3′); 5.21 (dd, 1H, $J_{OH,5'a}$ = 6.2 Hz, $J_{OH,5'b}$ = 5.0 Hz, OH-5′); 5.37 (d, 1H, $J_{OH,2'}$ = 6.3 Hz, OH-2′); 6.08 (d, 1H, $J_{1',2'}$ = 6.0 Hz, H-1'); 6.59 (q, 1H, $J_{NH,CH3}$ = 4.8 Hz, CH_3N**H**); 7.13 (m, 2H, H-*o*-SPh); 7.16 (m, 1H, H-*p*-SPh); 7.29 (m, 2H, H-*m*-SPh); 7.87 (s, 1H, H-6); 8.23 (s, 1H, H-2). ^{13}C NMR (150.9 MHz, DMSO-d_6): 27.74 (CH_3NH); 61.65 (CH_2-5′); 70.63 (CH-3′); 74.25 (CH-2′); 85.46 (CH-4′); 87.63 (CH-1′); 99.16 (C-5); 103.82 (C-4a); 125.95 (CH-*p*-SPh); 126.24 (CH-*o*-SPh); 129.44 (CH-*m*-SPh); 129.96 (CH-6); 138.39 (C-*i*-SPh); 150.33 (C-7a); 152.62 (CH-2); 157.10 (C-4). IR (KBr): 3398, 3180, 3126, 2941, 2917, 2905, 2896, 2866, 1613, 1562, 1488, 1389, 1099, 1060, 740, 629. HRMS (ESI) calculated for $C_{18}H_{21}O_4N_4S$: 389.1278; found: 389.1281. Anal. calculated for $C_{18}H_{20}N_4O_4S \cdot 1.25H_2O$: C, 52.61; H, 5.52; N, 13.63; S, 7.8; found: C, 52.79; H, 5.35; N, 13.46; S, 7.98.

4-(*N*-Methylamino)-5-[(thiophen-2-yl)sulfanyl]-7-β-D-ribofuranosyl)-pyrrolo[2,3-d]pyrimidine

(6-(*N*-Methylamino)-7-[(thiophen-2-yl)sulfanyl]-9-β-D-ribofuranosyl)-7-deazapurine) (4f)



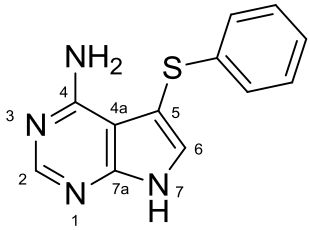
Reaction of nucleoside **4a** (712 mg, 1 mmol) according to the general procedure afforded compound **4f** as white solid (297 mg, 75 %).

M.p. 187 °C. $[\alpha]_D$ −51.7 (0.23). ^{1}H NMR (401.0 MHz, DMSO-d_6): 3.03 (d, 3H, $J_{CH3,NH}$ = 4.8 Hz, C**H**$_3$NH); 3.54 (dd, 1H, J_{gem} = 12.0 Hz, $J_{5'a,4'}$ = 3.7 Hz, H-5′a); 3.65 (dd, 1H, J_{gem} = 12.0 Hz, $J_{5'b,4'}$ = 3.7 Hz, H-5′b); 3.91 (q, 1H, $J_{4',5'a}$ = $J_{4',5'b}$ = $J_{4',3'}$ = 3.6 Hz, H-4′); 4.09 (dd, 1H, $J_{3',2'}$ = 5.1 Hz, $J_{3',4'}$ = 3.4 Hz, H-3'); 4.39 (dd, 1H, $J_{2',1'}$ = 6.0 Hz, $J_{2',3'}$ = 5.1 Hz, H-2'); 5.02 – 5.50 (m, 3H, OH-2′,3′,5′); 6.03 (d, 1H, $J_{1',2'}$ = 6.0 Hz, H-1'); 6.80 (q, 1H, $J_{NH,CH3}$ = 4.8 Hz, CH_3N**H**); 6.99 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.27 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.53 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.87 (s, 1H, H-6); 8.20 (s, 1H, H-2). ^{13}C NMR (100.8 MHz, DMSO-d_6): 27.80 (CH_3NH); 61.68 (CH_2-5′); 70.61 (CH-3′); 74.20 (CH-2′); 85.47 (CH-4′); 87.66 (CH-1′); 103.17 and 103.33 (C-4a,5); 128.04 (CH-4-Sthienyl); 128.87 (CH-6); 129.62 (CH-5-Sthienyl); 130.57 (CH-3-Sthienyl); 136.89 (C-2-Sthienyl); 149.88 (C-7a); 152.59 (CH-2); 156.95 (C-4). IR (KBr): 3503, 3404, 3279, 3126, 3099, 2929, 2869, 1613, 1568, 1491, 1416, 1398, 1338, 1308, 1126, 1084, 1048, 701. HRMS (ESI) calculated for $C_{16}H_{19}O_4N_4S_2$: 395.0842; found: 395.0842.

General procedure for amination

Compound **1a-4a** (1 equiv), aq ammonia (25% [w/w], 5 mL) in dioxane (5 mL) was stirred at autoclave at 120 °C for 18h. After cooling to rt precipitate was formed and filtered off.

4-Amino-5-(phenylsulfanyl)-7*H*-pyrrolo[2,3-*d*]pyrimidine
(6-Amino-7-(phenylsulfanyl)-7-deazapurine) (1g)

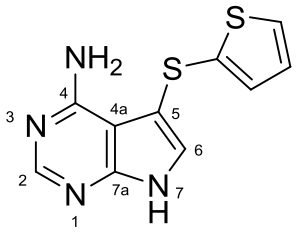


Reaction of deazapurine **1a** (523 mg, 2 mmol) according to the general procedure afforded compound **1g** (411 mg, 85%) as white powder.

^{1}H NMR was compared with published data[1].

4-Amino-5-[(thiophen-2-yl)sulfanyl]-7*H*-pyrrolo[2,3-*d*]pyrimidine

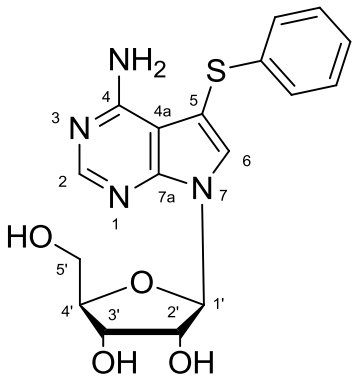
(6-Amino-7-[(thiophen-2-yl)sulfanyl]-7-deazapurine) (2g)



Reaction of deazapurine **2a** (535 mg, 2 mmol) according to the general procedure afforded compound **2g** (425 mg, 85%) as white powder.

M.p. 281 °C. ^{1}H NMR (500 MHz, DMSO-d_6): 6.70 (bs, 2H, NH_2); 6.97 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.20 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.49 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.57 (s, 1H, H-6); 8.08 (s, 1H, H-2); 12.01 (vbs, 1H, NH). ^{13}C NMR (125.7 MHz, DMSO-d_6): 102.18 and 102.22 (C-5,4a); 128.00 (CH-4-Sthienyl); 128.84 (CH-6); 128.88 (CH-5-Sthienyl); 129.35 (CH-3-Sthienyl); 137.99 (C-2-Sthienyl); 151.42 (C-7a); 152.76 (CH-2); 157.43 (C-4). IR (KBr): 3099, 3069, 2980, 2806, 2672, 1643, 1583, 1320, 719, 686. HRMS (ESI) calculated for $C_{10}H_9N_4S_2$: 249.0263; found: 249.0264.

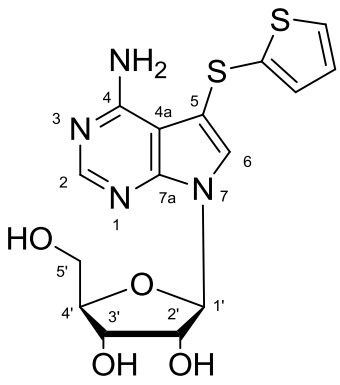
4-Amino-5-(phenylsulfanyl)-7-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine
(6-amino-7-(phenylsulfanyl)-9-β-D-ribofuranosyl)-7-deazapurine) (3g)



Reaction of nucleoside **3a** (706 mg, 1 mmol) according to the general procedure afforded compound **3g** (321 mg, 86%) as white powder.

M.p. 214 °C. $[\alpha]_D$ −62.8 (0.21). ^{1}H NMR (600.1 MHz, DMSO-d_6): 3.55 (ddd, 1H, J_{gem} = 12.0 Hz, $J_{5'a,OH}$ = 6.2 Hz, $J_{5'a,4'}$ = 3.7 Hz, H-5′a); 3.65 (ddd, 1H, J_{gem} = 12.0 Hz, $J_{5'b,OH}$ = 5.0 Hz, $J_{5'b,4'}$ = 3.7 Hz, H-5′b); 3.93 (q, 1H, $J_{4',5'a}$ = $J_{4',5'b}$ = $J_{4',3'}$ = 3.6 Hz, H-4′); 4.11 (td, 1H, $J_{3',2'}$ = $J_{3',OH}$ = 4.9 Hz, $J_{3',4'}$ = 3.3 Hz, H-3'); 4.34 (td, 1H, $J_{2',1'}$ = $J_{2',OH}$ = 6.2 Hz, $J_{2',3'}$ = 5.1 Hz, H-2'); 5.15 (d, 1H, $J_{OH,3'}$ = 4.8 Hz, OH-3′); 5.22 (dd, 1H, $J_{OH,5'a}$ = 6.3 Hz, $J_{OH,5'b}$ = 5.0 Hz, OH-5′); 5.39 (d, 1H, $J_{OH,2'}$ = 6.3 Hz, OH-2′); 6.09 (d, 1H, $J_{1',2'}$ = 6.1 Hz, H-1'); 7.12 (m, 2H, H-*o*-SPh); 7.16 (m, 1H, H-*p*-SPh); 7.29 (m, 2H, H-*m*-SPh); 7.90 (s, 1H, H-6); 8.14 (s, 1H, H-2). ^{13}C NMR (150.9 MHz, DMSO-d_6): 61.67 (CH_2-5′); 70.67 (CH-3′); 74.23 (CH-2′); 85.49 (CH-4′); 87.58 (CH-1′); 99.34 (C-5); 103.39 (C-4a); 125.94 (CH-*p*-SPh); 126.01 (CH-*o*-SPh); 129.50 (CH-*m*-SPh); 130.28 (CH-6); 138.29 (C-*i*-SPh); 151.21 (C-7a); 152.77 (CH-2); 157.65 (C-4). IR (KBr): 3407, 3282, 3147, 3087, 1646, 1586, 1556, 1473, 1440, 1329, 1317, 1144, 1015, 749. HRMS (ESI) calculated for $C_{17}H_{19}O_4N_4S$: 375.1122; found: 375.1123. Anal. calculated for: $C_{17}H_{18}N_4O_4S \cdot 0.60H_2O$: C, 53; H, 5.02; N, 14.54; S, 8.32; found: C, 52.77; H, 4.72; N, 14.29; S, 8.54.

4-Amino-5-[(thiophen-2-yl)sulfanyl]-7-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine (6-amino-7-[(thiophen-2-yl)sulfanyl]-9-β-D-ribofuranosyl)-7-deazapurine) (4g)

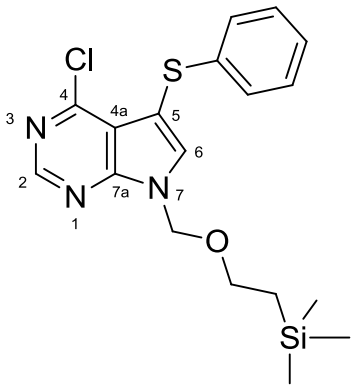


Reaction of nucleoside **4a** (712 mg, 1 mmol) according to the general procedure. Precipitate was not formed. Solvent was then evaporated under reduced pressure and crude products were purified using RP-HPFC (0→100% of MeOH in H_2O) and product **4g** was obtained as white powder (267 mg, 70 %).

M.p. 178 °C. $[\alpha]_D$ −49.5 (0.23). ^{1}H NMR (401.0 MHz, DMSO-d_6): 3.55 (ddd, 1H, J_{gem} = 12.0 Hz, $J_{5'a,OH}$ = 6.2 Hz, $J_{5'a,4'}$ = 3.7 Hz, H-5′a); 3.65 (ddd, 1H, J_{gem} = 12.0 Hz, $J_{5'b,OH}$ = 4.9 Hz, $J_{5'b,4'}$ = 3.7 Hz, H-5′b); 3.91 (bq, 1H, $J_{4',5'a}$ = $J_{4',5'b}$ = $J_{4',3'}$ = 3.5 Hz, H-4′); 4.09 (td, 1H, $J_{3',2'}$ = $J_{3',OH}$ = 4.9 Hz, $J_{3',4'}$ = 3.3 Hz, H-3'); 4.40 (td, 1H, $J_{2',1'}$ = $J_{2',OH}$ = 6.2 Hz, $J_{2',3'}$ = 5.1 Hz, H-2'); 5.13 (d, 1H, $J_{OH,3'}$ = 4.8 Hz, OH-3′); 5.23 (dd, 1H, $J_{OH,5'a}$ = 6.2 Hz, $J_{OH,5'b}$ = 4.9 Hz, OH-5′); 5.35 (d, 1H, $J_{OH,2'}$ = 6.4 Hz, OH-2′); 6.03 (d, 1H, $J_{1',2'}$ = 6.1 Hz, H-1'); 6.88 (vbs, 2H, NH_2); 6.99 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.24 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.53 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.90 (s, 1H, H-6); 8.11 (s, 1H, H-2). ^{13}C NMR (100.8 MHz, DMSO-d_6): 61.68 (CH_2-5′); 70.63 (CH-3′); 74.17 (CH-2′); 85.47 (CH-4′); 87.60 (CH-1′); 102.77 (C-4a); 103.45 (C-5); 128.09 (CH-4-Sthienyl); 129.23 (CH-6); 129.37 (CH-5-Sthienyl); 130.10 (CH-3-Sthienyl); 136.95 (C-2-Sthienyl); 150.74 (C-7a); 152.70 (CH-2); 157.58 (C-4). IR (KBr): 3285, 3102, 1625, 1589, 1556, 1479, 1437, 1344, 1311, 1129, 1045, 701. HRMS (ESI) calculated for $C_{15}H_{17}O_4N_4S_2$: 381.0686; found: 381.0687.

Methoxylation

4-Chloro-5-(phenylsulfanyl)-7-{([2-(trimethylsilyl)ethoxy]methyl}-7H-pyrrolo[2,3-d]pyrimidine
(6-chloro-7-(phenylsulfanyl)-9-{([2-(trimethylsilyl)ethoxy]methyl}-7-deazapurine) (5a)

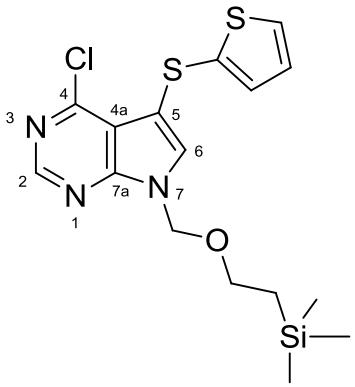


A mixture of **1a** (1.044 g, 4 mmol) and DMF (10 mL) was cooled to – 5 °C in an ice/brine bath. Sodium hydride (NaH, 60 wt%, 178 mg, 4.4 mmol, 1.1 equiv) was added in portions as a solid under stirring. The solution darkened over 15 minutes. 2-(Trimethylsilyl)ethoxymethyl chloride (SEM-Cl, 0.78 mL, 4.4 mmol, 1.1 equiv) was added

slowly via syringe so that the temperature did not exceed 5 °C. The reaction was stirred for 30 minutes to completion (monitored by TLC). Water (25 mL) was slowly added to quench the reaction. The mixture was then diluted with water (100 mL) and ether (200 mL). The layers were separated and the aqueous layer was extracted with ether (200 mL). The combined organic layers were washed with water (2 x 100 mL) and brine (100 mL), dried over sodium sulfate (Na_2SO_4), and concentrated under the reduced pressure. Crude product was purified using by HPFC (hexane/EtOAc, 0–20% EtOAc) and product **5a** (1.25 g, 80%) was obtained as a pale yellow oil which solidified upon standing at room temperature.

M.p. 107 °C. ^{1}H NMR (500 MHz, $CDCl_3$): -0.04 (s, 9H, CH_3Si); 0.93 (m, 2H, $OCH_2C\mathbf{H_2}Si$); 3.57 (m, 2H, $OC\mathbf{H_2}CH_2Si$); 5.67 (s, 2H, NCH_2O); 7.13 – 7.18 (m, 3H, H-*o*,*p*-SPh); 7.23 (m, 2H, H-*m*-SPh); 7.60 (s, 1H, H-6); 8.68 (bs, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): -1.46 (CH_3Si); 17.74 ($OCH_2\mathbf{C}H_2Si$); 67.15 ($O\mathbf{C}H_2CH_2Si$); 73.38 (NCH_2O); 104.42 (C-5); 116.9 (C-4a); 125.95 (CH-*p*-SPh); 127.25 (CH-*o*-SPh); 129.02 (CH-*m*-SPh); 134.96 (CH-6); 137.86 (C-*i*-SPh); 151.84 (CH-2); 152.82 (C-7a); 153.01 (C-4). IR (KBr): 3060, 3004, 2953, 2923, 1586, 1541, 1452, 1368, 1335, 1248, 1227, 1090, 979, 863, 830, 734, 629. HRMS (ESI) calculated for $C_{18}H_{23}ON_3ClSSi$: 392.1014; found: 392.1015.

4-Chloro-5-[(thiophen-2-yl)sulfanyl]-7-{([2-(trimethylsilyl)ethoxy]methyl}-7*H*-pyrrolo[2,3-*d*]pyrimidine
(6-chloro-7-[(thiophen-2-yl)sulfanyl]-9-{([2-(trimethylsilyl)ethoxy]methyl}-7-deazapurine) (6a)

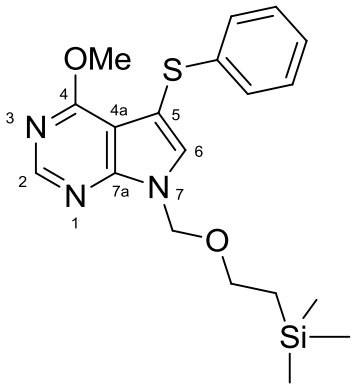


Compound **6a** was prepared as described for **5a** from 6-chloro-7-[(thiophen-2-yl)sulfanyl]-7-deazapurine (**2a**) (1.107 g, 4 mmol) to give protected deazapurine **6a** (1.09 g, 69%) as a pale yellow oil which solidified upon standing at room temperature.

M.p. 57°C. ^{1}H NMR (500 MHz, $CDCl_3$): -0.06 (s, 9H, CH_3Si); 0.89 (m, 2H, $OCH_2C\mathbf{H_2}Si$); 3.51 (m, 2H, $OC\mathbf{H_2}CH_2Si$); 5.60 (s, 2H, NCH_2O); 6.97 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.26 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.33 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.41 (s, 1H, H-6); 8.65 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): -1.48 (CH_3Si); 17.68 ($OCH_2\mathbf{C}H_2Si$); 67.03 ($O\mathbf{C}H_2CH_2Si$); 73.26 (NCH_2O); 108.87 (C-5); 116.09 (C-4a); 127.64 (CH-4-Sthienyl); 129.44 (CH-5-Sthienyl); 132.25 (CH-6); 132.49 (CH-3-Sthienyl); 134.53 (C-2-Sthienyl); 151.65 (CH-2); 152.30 (C-7a); 152.66 (C-4). IR (KBr): 3090, 3060, 2950, 2896, 1571, 1538, 1452, 1440, 1422, 1401, 1356, 1332, 1251, 1216, 1180, 1096, 979, 866, 836, 713, 689, 632. HRMS (ESI) calculated for $C_{16}H_{21}ON_3ClS_2Si$: 398.0578; found: 398.0579.

4-Methoxy-5-(phenylsulfanyl)-7-{([2-(trimethylsilyl)ethoxy]methyl}-7*H*-pyrrolo[2,3-*d*]pyrimidine

(6-methoxy-7-(phenylsulfanyl)-9-{([2-(trimethylsilyl)ethoxy]methyl}-7-deazapurine) (5h)

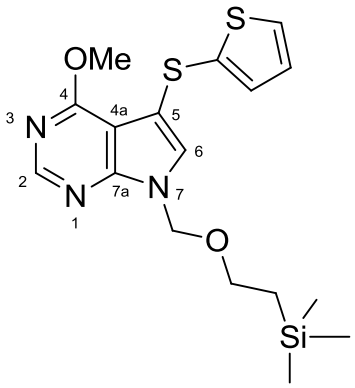


Protected deazapurine **5a** (950 mg, 2.5 mmol, 1 equiv) was disolved in acetone (10 mL) and 1 M solution of MeONa in MeOH (5 mL, 2 equiv) was added. Reaction mixture was stirred at rt overnight. Solvents were evaporated under reduced pressure and the mixture was then diluted with water (25 mL) and EtOAc (25 mL). The layers were separated and the aqueous layer was extracted two times with EtOAc (25 mL). The combined organic layers were dried over sodium sulfate (Na_2SO_4), and concentrated under the reduced pressure to give product **5h** (1.01 g, 99%) was as a yellow oil which solidified upon standing at room temperature.

M.p. 286 °C. ^{1}H NMR (500 MHz, $CDCl_3$): -0.05 (s, 9H, CH_3Si); 0.91 (m, 2H, $OCH_2C\mathbf{H_2}Si$); 3.56 (m, 2H, $OC\mathbf{H_2}CH_2Si$); 3.97 (s, 3H, CH_3O-4); 5.61 (s, 2H, NCH_2O); 7.13 (m, 1H, H-*p*-SPh); 7.19 – 7.24 (m, 4H, H-*o*,*m*-SPh); 7.33 (s, 1H, H-6); 8.48 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): -1.47 (CH_3Si); 17.74 ($OCH_2\mathbf{C}H_2Si$); 53.75 (CH_3O-4); 66.69 ($O\mathbf{C}H_2CH_2Si$);

73.17 (NCH_2O); 104.00 (C-5); 106.33 (C-4a); 125.72 (CH-*p*-SPh); 127.74 and 128.74 (CH-*m*,*o*-SPh); 130.67 (CH-6); 138.19 (C-*i*-SPh); 152.11 (CH-2); 153.28 (C-7a); 163.65 (C-4). IR (KBr): 3087, 3052, 2992, 2947, 2935, 2896, 1589, 1562, 1476, 1407, 1338, 1323, 1248, 1222, 1093, 863, 842, 743. HRMS (ESI) calculated for $C_{19}H_{25}O_2N_3NaSSi$: 410.1329; found: 410.1331.

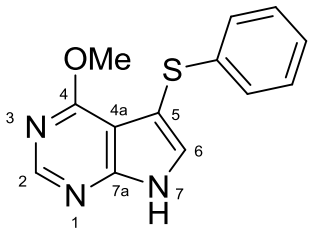
4-Methoxy-5-[(thiophen-2-yl)sulfanyl]-7-{([2-(trimethylsilyl)ethoxy]methyl}-7*H*-pyrrolo[2,3-*d*]pyrimidine
(6-methoxy-7-[(thiophen-2-yl)sulfanyl]-9-{([2-(trimethylsilyl)ethoxy]methyl}-7-deazapurine) (6h)



Compound **6h** was prepared as described for **5h** from 6-methoxy-7-[(thiophen-2-yl)sulfanyl]-9-{([2-(trimethylsilyl)ethoxy]methyl}-7-deazapurine (**6a**) (995 mg, 2.5 mmol) to give 6-methoxy deazapurine **6h** (930 mg, 95%) as a pale yellow oil which solidified upon standing at room temperature.

M.p. 101 °C. ^{1}H NMR (500 MHz, $CDCl_3$): -0.07 (s, 9H, CH_3Si); 0.88 (m, 2H, $OCH_2C\mathbf{H_2}Si$); 3.50 (m, 2H, $OC\mathbf{H_2}CH_2Si$); 4.15 (s, 3H, CH_3O); 5.54 (s, 2H, NCH_2O); 6.96 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.16 (s, 1H, H-6); 7.24 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.31 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 8.46 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): -1.48 (CH_3Si); 17.70 ($OCH_2\mathbf{C}H_2Si$); 53.70 (CH_3O); 66.60 ($O\mathbf{C}H_2CH_2Si$); 73.06 (NCH_2O); 105.45 (C-4a); 108.19 (C-5); 127.39 (CH-4-Sthienyl); 128.13 (CH-6); 129.26 (CH-5-Sthienyl); 132.73 (CH-3-Sthienyl); 135.02 (C-2-Sthienyl); 151.98 (CH-2); 152.87 (C-7a); 163.53 (C-4). IR (KBr):3087, 3004, 2953, 2890, 1589, 1562, 1479, 1410, 1341, 1326, 1248, 1224, 1174, 1096, 1078, 866, 833. HRMS (ESI) calculated for $C_{17}H_{24}O_2N_3S_2Si$: 394.1074; found: 394.1074.

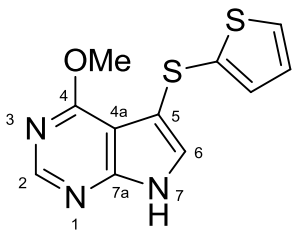
4-Methoxy-5-(phenylsulfanyl)-7*H*-pyrrolo[2,3-*d*]pyrimidine
(6-methoxy-7-(phenylsulfanyl)-7-deazapurine) (1h)



Protected deazapurine **5h** (774 mg, 2.0 mmol, 1 equiv) was disolved in trifluoroacetic acid (2 mL) and the reaction mixture was stirred at rt overnight. The mixture was then diluted with $NaHCO_3$ (check pH=7!) and EtOAc (25 mL). The layers were separated and the aqueous layer was extracted two times with EtOAc (25 mL). The combined organic layers were dried over sodium sulfate (Na_2SO_4), and concentrated under the reduced pressure to give white solid. The solid was then diluted with aq. ammonia (25% [w/w], 15 mL) and stirred at rt overnight to form white precipitate. The precipitate was filtered to give product **1h** (460 mg, 90%) as a white powder.

M.p. 200 °C. ^{1}H NMR (600.1 MHz, DMSO-d_6): 3.86 (s, 3H, CH_3O); 7.07 (m, 2H, H-*o*-SPh); 7.10 (m, 1H, H-*p*-SPh); 7.23 (m, 2H, H-*m*-SPh); 7.71 (s, 1H, H-6); 8.41 (s, 1H, H-2); 12.54 (bs, 1H, NH). ^{13}C NMR (150.9 MHz, DMSO-d_6): 53.57 (CH_3O); 99.54 (C-5); 105.71 (C-4a); 125.30 (CH-*p*-SPh); 126.20 (CH-*o*-SPh); 129.04 (CH-*m*-SPh); 131.31 (CH-6); 139.40 (C-*i*-SPh); 151.51 (CH-2); 153.58 (C-7a); 162.90 (C-4). IR (KBr): 3096, 2974, 2941, 2896, 2851, 2821, 1598, 1583, 1485, 1434, 1398, 1335, 1326, 1093, 878, 737. HRMS (ESI) calculated for $C_{13}H_{12}ON_3S$: 258.0696; found: 258.0696.

4-Methoxy-5-[(thiophen-2-yl)sulfanyl]-7*H*-pyrrolo[2,3-*d*]pyrimidine
(6-methoxy-7-[(thiophen-2-yl)sulfanyl]-7-deazapurine) (2h)



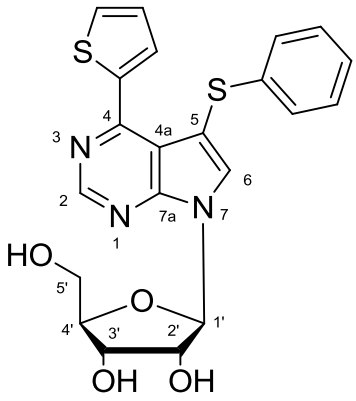
Compound **2h** was prepared as described for **1h** from 6-chloro-7-[(thiophen-2-yl)sulfanyl]-9-{([2-(trimethylsilyl)ethoxy]methyl}-7-deazapurine (**6h**) (787 mg, 2.0 mmol) to give deazapurine **2h** (462 mg, 88%) as a white powder.

M.p. 167 °C. ^{1}H NMR (401 MHz, DMSO-d_6): 4.03 (s, 3H, CH_3O); 6.98 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.20 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.50 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.60 (s, 1H, H-6); 8.39 (s, 1H, H-2); 12.41 (bs, 1H, NH). ^{13}C NMR (100.8 MHz, DMSO-d_6): 53.56 (CH_3O); 104.10 (C-5); 104.91 (C-4a); 127.84 (CH-4-Sthienyl); 129.25 (CH-6); 129.46 (CH-5-Sthienyl); 131.46 (CH-3-Sthienyl); 136.58 (C-2-Sthienyl); 151.52 (CH-2); 153.07 (C-7a); 162.81 (C-4). IR (KBr):3096, 2992, 2947, 2899, 2857, 2824, 1595, 1583, 1476, 1395, 1338, 1317, 1102, 713, 626. HRMS (ESI) calculated for C11 H10 O N3 S2: 264.0260; found: 264.0261.

General procedure for deprotection of 7-substituted nucleosides

A protected nucleoside **3b-3e** or **4b-4e** (1 equiv) was disolved in methanol and 1 M solution of MeONa in MeOH (1.5 equiv) was added. Reaction mixture was stirred at rt overnight. Solvent was evaporated under reduced pressure and crude products were purified using RP-HPFC (0→100% of MeOH in H_2O).

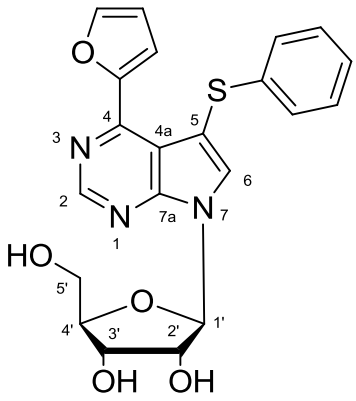
4-(Thiophen-2-yl)-5-(phenylsulfanyl)-7-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine (6-(thiophen-2-yl)-7-(phenylsulfanyl)-9-β-D-ribofuranosyl)-7-deazapurine) (7b)



Deprotection of **3b** (376 mg, 0.5 mmol) according to the general procedure afforded compound **7b** (164 mg, 75%) as yellow solid. Crystallization in MeOH/H_2O gave yellow foam.

M.p. 77 °C $[\alpha]_D$ −49.3 (0.19). ^{1}H NMR (401.0 MHz, DMSO-d_6): 3.59 (dd, 1H, J_{gem} = 12.0 Hz, $J_{5'a,4'}$ = 3.7 Hz, H-5′a); 3.69 (dd, 1H, J_{gem} = 12.0 Hz, $J_{5'b,4'}$ = 3.7 Hz, H-5′b); 3.97 (q, 1H, $J_{4',5'a} = J_{4',5'b} = J_{4',3'}$ = 3.7 Hz, H-4′); 4.15 (dd, 1H, $J_{3',2'}$ = 5.0 Hz, $J_{3',4'}$ = 3.6 Hz, H-3'); 4.46 (bt, 1H, $J_{2',1'} = J_{2',3'}$ = 5.4 Hz, H-2'); 5.06 – 5.64 (m, 3H, OH-2′,3′,5′); 6.33 (d, 1H, $J_{1',2'}$ = 5.8 Hz, H-1'); 6.93 (m, 2H, H-*o*-SPh); 7.04 (m, 1H, H-*p*-SPh); 7.08 (dd, 1H, $J_{4,5}$ = 5.1 Hz, $J_{4,3}$ = 3.8 Hz, H-4-thienyl); 7.16 (m, 2H, H-*m*-SPh); 7.71 (dd, 1H, $J_{5,4}$ = 5.1 Hz, $J_{5,3}$ = 1.1 Hz, H-5-thienyl); 8.35 (dd, 1H, $J_{3,4}$ = 3.8 Hz, $J_{3,5}$ = 1.1 Hz, H-3-thienyl); 8.41 (s, 1H, H-6); 8.84 (s, 1H, H-2). ^{13}C NMR (100.8 MHz, DMSO-d_6): 61.37 (CH_2-5′); 70.51 (CH-3′); 74.60 (CH-2′); 85.56 (CH-4′); 87.26 (CH-1′); 100.37 (C-5); 114.66 (C-4a); 125.67 (CH-*p*-SPh); 125.83 (CH-*o*-SPh); 128.20 (CH-4-thienyl); 129.27 (CH-*m*-SPh); 131.08 (CH-5-thienyl); 132.28 (CH-3-thienyl); 136.12 (CH-6); 138.44 (C-*i*-SPh); 140.89 (C-2-thienyl); 151.22 (CH-2); 152.62 (C-4); 153.50 (C-7a). IR (KBr): 3117, 3052, 2923, 2869, 1559, 1482, 1437, 1404, 1195, 1105, 1081, 1048, 1021, 737.. HRMS (ESI) calculated for $C_{21}H_{20}O_4N_3S_2$: 442.0890; found: 442.0890.

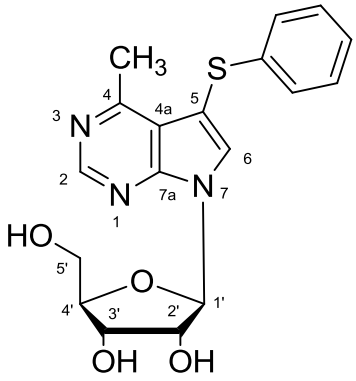
4-(Furan-2-yl)-5-(phenylsulfanyl)-7-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine (6-(furan-2-yl)-7-(phenylsulfanyl)-9-β-D-ribofuranosyl)-7-deazapurine) (7c)



Deprotection of **3c** (552 mg, 0.75 mmol) according to the general procedure afforded compound **7c** (248 mg, 78%) as yellow solid. Crystallization in MeOH/H_2O gave yellow solid.

M.p. 120 °C $[\alpha]_D$ −39.3 (0.19). ^{1}H NMR (500 MHz, DMSO-d_6): 3.59 (ddd, 1H, J_{gem} = 12.0 Hz, $J_{5'a,OH}$ = 5.5 Hz, $J_{5'a,4'}$ = 3.7 Hz, H-5′a); 3.68 (ddd, 1H, J_{gem} = 12.0 Hz, $J_{5'b,OH}$ = 5.3 Hz, $J_{5'b,4'}$ = 3.9 Hz, H-5′b); 3.97 (q, 1H, $J_{4',5'a}$ = $J_{4',5'b}$ = $J_{4',3'}$ = 3.7 Hz, H-4′); 4.15 (td, 1H, $J_{3',2'}$ = $J_{3',OH}$ = 5.0 Hz, $J_{3',4'}$ = 3.6 Hz, H-3'); 4.46 (td, 1H, $J_{2',1'}$ = $J_{2',OH}$ = 6.0 Hz, $J_{2',3'}$ = 5.0 Hz, H-2'); 5.13 (t, 1H, $J_{OH,5'a}$ = $J_{OH,5'b}$ = 5.4 Hz, OH-5′); 5.22 (d, 1H, $J_{OH,3'}$ = 5.0 Hz, OH-3′); 5.50 (d, 1H, $J_{OH,2'}$ = 6.1 Hz, OH-2′); 6.32 (d, 1H, $J_{1',2'}$ = 5.8 Hz, H-1'); 6.59 (dd, 1H, $J_{4,3}$ = 3.5 Hz, $J_{4,5}$ = 1.7 Hz, H-4-furyl); 7.01 (m, 2H, H-*o*-SPh); 7.05 (m, 1H, H-*p*-SPh); 7.18 (m, 2H, H-*m*-SPh); 7.42 (dd, 1H, $J_{3,4}$ = 3.5 Hz, $J_{3,5}$ = 0.9 Hz, H-3-furyl); 7.71 (dd, 1H, $J_{5,4}$ = 1.7 Hz, $J_{5,3}$ = 0.9 Hz, H-5-furyl); 8.38 (s, 1H, H-6); 8.87 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, DMSO-d_6): 61.39 (CH_2-5′); 70.54 (CH-3′); 74.54 (CH-2′); 85.55 (CH-4′); 87.09 (CH-1′); 100.90 (C-5); 112.43 (CH-4-furyl); 114.11 (C-4a); 115.27 (CH-3-furyl); 125.36 (CH-*p*-SPh); 125.76 (CH-*o*-SPh); 129.11 (CH-*m*-SPh); 135.98 (CH-6); 139.03 (C-*i*-SPh); 145.91 (CH-5-furyl); 148.09 (C-4); 150.63 (C-2-furyl); 151.41 (CH-2); 153.57 (C-7a). IR (KBr): 3294, 3135, 3117, 2947, 2920, 2902, 1580, 1556, 1482, 1440, 1326, 1204, 1108, 988, 734. HRMS (ESI) calculated for $C_{21}H_{20}O_5N_3S$: 426.1118; found: 426.1118. Anal. calculated for $C_{21}H_{19}N_3O_5S \cdot 0.75H_2O$: C, 57.46; H, 4.71; N, 9.57; S, 7.3; found: C, 57.77; H, 4.56; N, 9.21; S, 7.17.

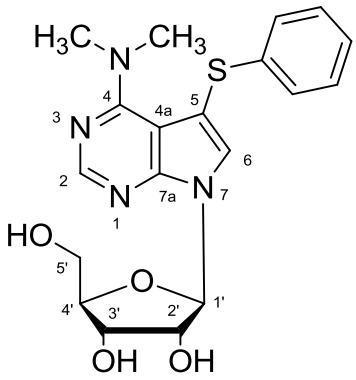
4-Methyl-5-(phenylsulfanyl)-7-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine (6-methyl-7-(phenylsulfanyl)-9-β-D-ribofuranosyl)-7-deazapurine) (7d)



Deprotection of **3d** (274 mg, 0.4 mmol) according to the general procedure afforded compound **7d** (130 mg, 87%) as white solid. Crystallization in MeOH/H_2O gave white solid.

M.p. 182 °C $[\alpha]_D$ −54.5 (0.21). ^{1}H NMR (600.1 MHz, DMSO-d_6): 2.60 (s, 3H, CH_3-4); 3.58 (ddd, 1H, J_{gem} = 11.9 Hz, $J_{5'a,OH}$ = 5.5 Hz, $J_{5'a,4'}$ = 3.7 Hz, H-5′a); 3.67 (ddd, 1H, J_{gem} = 11.9 Hz, $J_{5'b,OH}$ = 5.2 Hz, $J_{5'b,4'}$ = 3.9 Hz, H-5′b); 3.95 (q, 1H, $J_{4',5'a}$ = $J_{4',5'b}$ = $J_{4',3'}$ = 3.7 Hz, H-4′); 4.14 (td, 1H, $J_{3',2'}$ = $J_{3',OH}$ = 4.9 Hz, $J_{3',4'}$ = 3.4 Hz, H-3'); 4.45 (td, 1H, $J_{2',1'}$ = $J_{2',OH}$ = 6.0 Hz, $J_{2',3'}$ = 5.0 Hz, H-2'); 5.11 (t, 1H, $J_{OH,5'a}$ = $J_{OH,5'b}$ = 5.4 Hz, OH-5′); 5.20 (d, 1H, $J_{OH,3'}$ = 4.9 Hz, OH-3′); 5.45 (d, 1H, $J_{OH,2'}$ = 6.1 Hz, OH-2′); 6.25 (d, 1H, $J_{1',2'}$ = 5.9 Hz, H-1'); 7.07 (m, 2H, H-*o*-SPh); 7.15 (m, 1H, H-*p*-SPh); 7.28 (m, 2H, H-*m*-SPh); 8.27 (s, 1H, H-6); 8.73 (s, 1H, H-2). ^{13}C NMR (150.9 MHz, DMSO-d_6): 20.81 (CH_3-4); 61.46 (CH_2-5′); 70.56 (CH-3′); 74.44 (CH-2′); 85.53 (CH-4′); 87.10 (CH-1′); 100.54 (C-5); 117.80 (C-4a); 125.58 (CH-*o*-SPh); 125.69 (CH-*p*-SPh); 129.15 (CH-*m*-SPh); 134.14 (CH-6); 138.79 (C-*i*-SPh); 151.62 (C-7a); 151.79 (CH-2); 159.62 (C-4). IR (KBr): 3455, 3422, 3225, 3072, 2953, 2914, 2881, 2839, 1580, 1568, 1470, 1428, 1338, 1213, 1054, 1042, 740, 629. HRMS (ESI) calculated for $C_{18}H_{20}O_4N_3S$: 374.1169; found: 374.1170. Anal. calculated for $C_{18}H_{19}N_3O_4S \cdot 0.3\ H_2O$: C, 57.07; H, 5.21; N, 11.09; S, 8.46; found: C, 57.12; H, 5.16; N, 10.92; S, 8.29.

4-(*N*,*N*-Dimethylamino)-5-(phenylsulfanyl)-7-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine
(6-(*N*,*N*-Dimethylamino)-7-(phenylsulfanyl)-9-β-D-ribofuranosyl)-7-deazapurine) (7e)

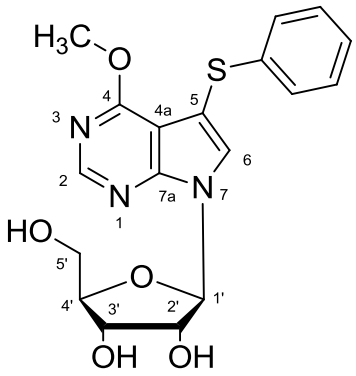


Deprotection of **3e** (535 mg, 0.75 mmol) according to the general procedure afforded compound **7e** (302 mg, 87%) as white solid. Crystallization in MeOH/H_2O gave white solid.

M.p. 112 °C $[\alpha]_D$ −44.4 (0.18). ^{1}H NMR (600.1 MHz, DMSO-d_6): 3.12 (s, 6H, $(CH_3)_2N$); 3.55 (ddd, 1H, J_{gem} = 12.0 Hz, $J_{5'a,OH}$ = 5.7 Hz, $J_{5'a,4'}$ = 3.7 Hz, H-5′a); 3.65 (ddd, 1H, J_{gem} = 12.0 Hz, $J_{5'b,OH}$ = 4.9 Hz, $J_{5'b,4'}$ = 3.7 Hz, H-5′b); 3.92 (q, 1H, $J_{4',5'a}$ = $J_{4',5'b}$ = $J_{4',3'}$ = 3.6 Hz, H-4′);

4.10 (m, 1H, H-3'); 4.41 (m, 1H, H-2'); 5.13 – 5.17 (m, 2H, OH-3′,5′); 5.39 (m, 1H, OH-2′); 6.18 (d, 1H, $J_{1',2'}$ = 5.9 Hz, H-1'); 7.03 (m, 2H, H-*o*-SPh); 7.12 (m, 1H, H-*p*-SPh); 7.26 (m, 2H, H-*m*-SPh); 8.01 (s, 1H, H-6); 8.25 (s, 1H, H-2). ^{13}C NMR (150.9 MHz, DMSO-d_6): 41.26 ($(CH_3)_2$N); 61.51 (CH_2-5′); 70.53 (CH-3′); 74.30 (CH-2′); 85.32 (CH-4′); 87.33 (CH-1′); 99.61 (C-5); 104.95 (C-4a); 125.31 (CH-*o*-SPh); 125.45 (CH-*p*-SPh); 129.32 (CH-*m*-SPh); 131.82 (CH-6); 139.39 (C-*i*-SPh); 150.78 (CH-2); 152.83 (C-7a); 159.39 (C-4).
IR (KBr): 3515, 3407, 3192, 3114, 3052, 2941, 2902, 2863, 1571, 1547, 1491, 1416, 1296, 1096, 1057, 1030,740. HRMS (ESI) calculated for $C_{19}H_{23}O_4N_4S$: 403.1435; found: 403.1436. Anal. calculated for $C_{19}H_{22}N_4O_4S \cdot 1.15H_2O$: C, 53.93; H, 5.79; N, 13.24; S, 7.58; found: C, 54.14; H, 5.72; N, 13.03; S, 7.48.

4-Methoxy-5-(phenylsulfanyl)-7-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine (6-methoxy-7-(phenylsulfanyl)-9-β-D-ribofuranosyl)-7-deazapurine) (7h)

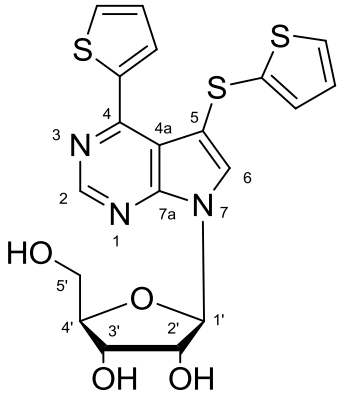


Deprotection and methoxylation of **3a** (706 mg, 1 mmol) according to the general procedure (4 equiv of NaOMe were used) afforded compound **7h** (290 mg, 75%) as white solid. Crystallization in MeOH/H_2O gave white solid.

M.p. 162 °C $[\alpha]_D$ −55.1 (0.18). ^{1}H NMR (600.1 MHz, DMSO-d_6): 3.56 (ddd, 1H, J_{gem} = 12.0 Hz, $J_{5'a,OH}$ = 5.7 Hz, $J_{5'a,4'}$ = 3.8 Hz, H-5′a); 3.65 (ddd, 1H, J_{gem} = 12.0 Hz, $J_{5'b,OH}$ = 5.2 Hz, $J_{5'b,4'}$ = 3.9 Hz, H-5′b); 3.87 (s, 3H, CH_3O-4); 3.93 (q, 1H, $J_{4',5'a} = J_{4',5'b} = J_{4',3'}$ = 3.7 Hz, H-4′); 4.11 (td, 1H, $J_{3',2'} = J_{3',OH}$ = 4.9 Hz, $J_{3',4'}$ = 3.4 Hz, H-3'); 4.42 (m, 1H, H-2'); 5.10 (t, 1H, $J_{OH,5'a} = J_{OH,5'b}$ = 5.4 Hz, OH-5′); 5.18 (d, 1H, $J_{OH,3'}$ = 4.8 Hz, OH-3′); 5.42 (d, 1H, $J_{OH,2'}$ = 6.0 Hz, OH-2′); 6.18 (d, 1H, $J_{1',2'}$ = 6.0 Hz, H-1'); 7.11 (m, 2H, H-*o*-SPh); 7.13 (m, 1H, H-*p*-SPh); 7.26 (m, 2H, H-*m*-SPh); 8.04 (s, 1H, H-6); 8.48 (s, 1H, H-2). ^{13}C NMR (150.9 MHz, DMSO-d_6): 53.83 (CH_3O-4); 61.52 (CH_2-5′); 70.59 (CH-3′); 74.44 (CH-2′); 85.53 (CH-4′);

87.40 (CH-1′); 101.02 (C-5); 106.28 (C-4a); 125.61 (CH-*p*-SPh); 126.51 (CH-*o*-SPh); 129.14 (CH-*m*-SPh); 131.09 (CH-6); 138.61 (C-*i*-SPh); 151.73 (CH-2); 152.96 (C-7a); 162.97 (C-4). IR (KBr): 3225, 3150, 3058, 3016, 3001, 2941, 2908, 2869, 2848, 1589, 1556, 1479, 1449, 1419, 1344, 1299, 1072, 1051, 737. HRMS (ESI) calculated for $C_{18}H_{20}O_5N_3S$: 390.1118; found: 390.1119.

4-(Thiophen-2-yl)-5-[(thiophen-2-yl)sulfanyl]-7-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine
(6-(thiophen-2-yl)-7-[(thiophen-2-yl)sulfanyl]-9-β-D-ribofuranosyl)-7-deazapurine) (8b)

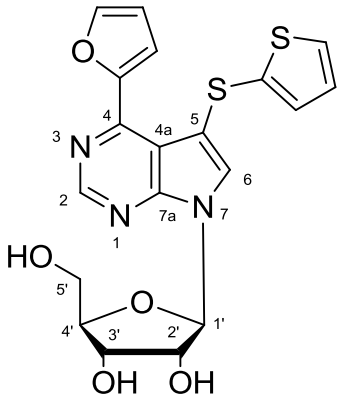


Deprotection of **4b** (462 mg, 0.65 mmol) according to the general procedure afforded compound **8b** (171 mg, 59%) as yellowish solid.

M.p. 165 °C $[\alpha]_D$ −31.2 (0.19). ^{1}H NMR (401.0 MHz, DMSO-d_6): 3.60 (dd, 1H, J_{gem} = 12.0 Hz, $J_{5'a,4'}$ = 3.8 Hz, H-5′a); 3.70 (dd, 1H, J_{gem} = 12.0 Hz, $J_{5'b,4'}$ = 3.8 Hz, H-5′b); 3.97 (q, 1H, $J_{4',5'a}$ = $J_{4',5'b}$ = $J_{4',3'}$ = 3.7 Hz, H-4′); 4.15 (dd, 1H, $J_{3',2'}$ = 5.0 Hz, $J_{3',4'}$ = 3.6 Hz, H-3'); 4.43 (bt, 1H, $J_{2',1'}$ = $J_{2',3'}$ = 5.4 Hz, H-2'); 4.96 – 5.66 (m, 3H, OH-2′,3′,5′); 6.28 (d, 1H, $J_{1',2'}$ = 5.7 Hz, H-1'); 6.79 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 6.85 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.30 (dd, 1H, $J_{4,5}$ = 5.1 Hz, $J_{4,3}$ = 3.7 Hz, H-4-thienyl); 7.41 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.86 (dd, 1H, $J_{5,4}$ = 5.1 Hz, $J_{5,3}$ = 1.1 Hz, H-5-thienyl); 8.38 (s, 1H, H-6); 8.40 (dd, 1H, $J_{3,4}$ = 3.7 Hz, $J_{3,5}$ = 1.1 Hz, H-3-thienyl); 8.82 (s, 1H, H-2). ^{13}C NMR (100.8 MHz, DMSO-d_6): 61.41 (CH_2-5′); 70.51 (CH-3′); 74.56 (CH-2′); 85.57 (CH-4′); 87.29 (CH-1′); 104.92 (C-5); 113.99 (C-4a); 127.83 (CH-4-Sthienyl); 128.31 (CH-4-thienyl); 129.54 (CH-5-Sthienyl); 130.56 (CH-3-Sthienyl); 131.15 (CH-5-thienyl); 132.91 (CH-3-thienyl); 134.53 (CH-6); 136.03 (C-2-Sthienyl); 140.81 (C-2-thienyl); 151.21

(CH-2); 152.52 (C-4); 152.94 (C-7a). IR (KBr): 3291, 3111, 2932, 2869, 1556, 1443, 1401, 1192, 1099, 1075, 1045, 803, 716, 629. HRMS (ESI) calculated for $C_{19}H_{18}O_4N_3S_3$: 448.0454; found: 448.0453.

4-(Furan-2-yl)-5-[(thiophen-2-yl)sulfanyl]-7-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine
(6-(furan-2-yl)-7-[(thiophen-2-yl)sulfanyl]-9-β-D-ribofuranosyl)-7-deazapurine) (8c)

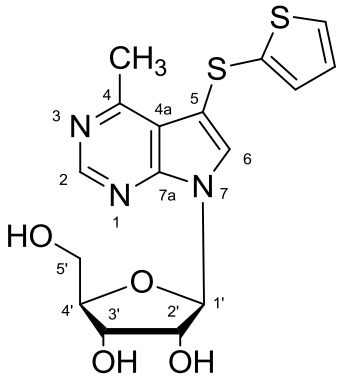


Deprotection of **4c** (260 mg, 0.35 mmol) according to the general procedure afforded compound **8c** (85 mg, 57%) as yellow solid.

M.p. 172 °C $[\alpha]_D$ −41.7 (0.21). ^{1}H NMR (401.0 MHz, DMSO-d_6): 3.56 (dd, 1H, J_{gem} = 11.9 Hz, $J_{5'a,4'}$ = 3.8 Hz, H-5′a); 3.66 (dd, 1H, J_{gem} = 11.9 Hz, $J_{5'b,4'}$ = 3.8 Hz, H-5′b); 3.95 (q, 1H, $J_{4',5'a}$ = $J_{4',5'b}$ = $J_{4',3'}$ = 3.7 Hz, H-4′); 4.11 (dd, 1H, $J_{3',2'}$ = 5.0 Hz, $J_{3',4'}$ = 3.6 Hz, H-3'); 4.39 (bt, 1H, $J_{2',1'}$ = $J_{2',3'}$ = 5.4 Hz, H-2'); 4.96 – 5.67 (m, 3H, OH-2′,3′,5′); 6.25 (d, 1H, $J_{1',2'}$ = 5.8 Hz, H-1'); 6.78 (dd, 1H, $J_{4,3}$ = 3.5 Hz, $J_{4,5}$ = 1.8 Hz, H-4-furyl); 6.93 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.02 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.48 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.50 (dd, 1H, $J_{3,4}$ = 3.5 Hz, $J_{3,5}$ = 0.9 Hz, H-3-furyl); 8.05 (dd, 1H, $J_{5,4}$ = 1.8 Hz, $J_{5,3}$ = 0.9 Hz, H-5-furyl); 8.17 (s, 1H, H-6); 8.83 (s, 1H, H-2). ^{13}C NMR (100.8 MHz, DMSO-d_6): 61.44 (CH_2-5′); 70.55 (CH-3′); 74.51 (CH-2′); 85.52 (CH-4′); 87.16 (CH-1′); 106.19 (C-5); 112.74 (CH-4-furyl); 113.10 (C-4a); 115.42 (CH-3-furyl); 127.95 (CH-4-Sthienyl); 129.71 (CH-5-Sthienyl); 131.25 (CH-3-Sthienyl); 133.07 (CH-6); 135.74 (C-2-Sthienyl); 146.18 (CH-5-furyl); 147.87 (C-4); 150.87 (C-2-furyl); 151.33 (CH-2); 152.96 (C-7a). IR (KBr): 3252, 3162, 3138, 2944, 2914, 2872, 1586,

1562, 1532, 1461, 1338, 1198, 1096, 1054, 1024, 979, 812, 752, 704. HRMS (ESI) calculated for $C_{19}H_{17}O_5N_3NaS_2$: 454.0502; found: 454.0502.

4-Methyl-5-[(thiophen-2-yl)sulfanyl]-7-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine (6-methyl-7-[(thiophen-2-yl)sulfanyl]-9-β-D-ribofuranosyl)-7-deazapurine) (8d)

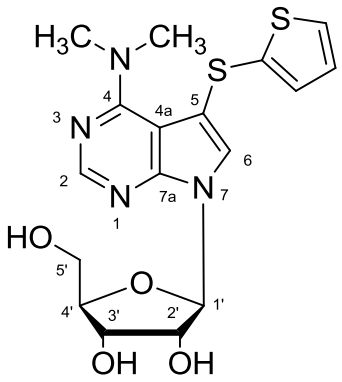


Deprotection of **4d** (415 mg, 0.6 mmol) according to the general procedure afforded compound **8d** (146 mg, 64%) as white solid.

M.p. 140 °C $[\alpha]_D$ −53.0 (0.22). ^{1}H NMR (401.0 MHz, DMSO-d_6): 2.83 (s, 3H, CH_3); 3.57 (dd, 1H, J_{gem} = 11.9 Hz, $J_{5'a,4'}$ = 3.8 Hz, H-5′a); 3.68 (dd, 1H, J_{gem} = 11.9 Hz, $J_{5'b,4'}$ = 3.9 Hz, H-5′b); 3.94 (q, 1H, $J_{4',5'a} = J_{4',5'b} = J_{4',3'}$ = 3.7 Hz, H-4′); 4.12 (dd, 1H, $J_{3',2'}$ = 5.1 Hz, $J_{3',4'}$ = 3.5 Hz, H-3'); 4.41 (dd, 1H, $J_{2',1'}$ = 5.9 Hz, $J_{2',3'}$ = 5.1 Hz, H-2'); 5.00 – 5.55 (m, 3H, OH-2′,3′,5′); 6.20 (d, 1H, $J_{1',2'}$ = 5.9 Hz, H-1'); 7.00 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.16 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.51 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 8.27 (s, 1H, H-6); 8.71 (s, 1H, H-2). ^{13}C NMR (100.8 MHz, DMSO-d_6): 21.60 (CH_3); 61.50 (CH_2-5′); 70.55 (CH-3′); 74.45 (CH-2′); 85.54 (CH-4′); 87.18 (CH-1′); 104.53 (C-5); 117.14 (C-4a); 128.20 (CH-4-Sthienyl); 128.85 (CH-5-Sthienyl); 129.50 (CH-3-Sthienyl); 133.11 (CH-6); 137.12 (C-2-Sthienyl); 151.10 (C-7a); 151.76 (CH-2); 159.90 (C-4). IR (KBr): 3425, 3282, 3108, 2950, 2932, 2881, 2842, 1583, 1562, 1416, 1407, 1338, 1219, 1207, 1117, 1096, 1057, 1039, 976, 848, 710, 695, 626. HRMS (ESI) calculated for $C_{16}H_{17}O_4N_3NaS_2$: 402.0553; found: 402.0553.

4-(*N*,*N*-Dimethylamino)-5-[(thiophen-2-yl)sulfanyl]-7-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine

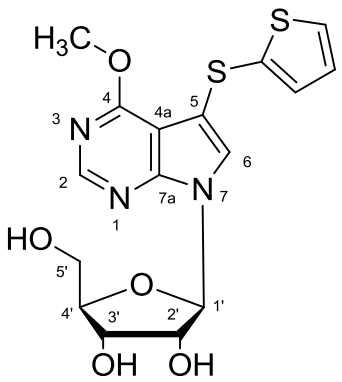
(6-(*N*,*N*-Dimethylamino)-7-[(thiophen-2-yl)sulfanyl]-9-β-D-ribofuranosyl)-7-deazapurine) (8e)



Deprotection of **4e** (540 mg, 0.75 mmol) according to the general procedure afforded compound **8e** (197 mg, 65%) as white solid.

M.p. 199 °C $[\alpha]_D$ −41.8 (0.19). ^{1}H NMR (401.0 MHz, DMSO-d$_6$): 3.24 (s, 6H, $(CH_3)_2N$); 3.55 (dd, 1H, J_{gem} = 11.9 Hz, $J_{5'a,4'}$ = 3.7 Hz, H-5′a); 3.65 (dd, 1H, J_{gem} = 11.9 Hz, $J_{5'b,4'}$ = 3.7 Hz, H-5′b); 3.91 (q, 1H, $J_{4',5'a} = J_{4',5'b} = J_{4',3'}$ = 3.7 Hz, H-4′); 4.09 (dd, 1H, $J_{3',2'}$ = 5.1 Hz, $J_{3',4'}$ = 3.6 Hz, H-3'); 4.36 (dd, 1H, $J_{2',1'}$ = 5.8 Hz, $J_{2',3'}$ = 5.1 Hz, H-2'); 5.00 – 5.51 (m, 3H, OH-2′,3′,5′); 6.11 (d, 1H, $J_{1',2'}$ = 5.8 Hz, H-1'); 6.97 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.10 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.48 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 8.23 (s, 1H, H-6); 8.30 (s, 1H, H-2). ^{13}C NMR (100.8 MHz, DMSO-d$_6$): 41.54 ($(CH_3)_2N$); 61.57 (CH_2-5′); 70.54 (CH-3′); 74.32 (CH-2′); 85.33 (CH-4′); 87.44 (CH-1′); 104.37 and 104.47 (C-4a,5); 128.05 (CH-4-Sthienyl); 128.93 (CH-5-Sthienyl); 129.82 (CH-3-Sthienyl); 130.12 (CH-6); 137.39 (C-2-Sthienyl); 150.76 (CH-2); 152.35 (C-7a); 159.53 (C-4). IR (KBr):3494, 3282, 3222, 3117, 2941, 2887, 1577, 1538, 1497, 1437, 1419, 1404, 1299, 1219, 1135, 1030, 698. HRMS (ESI) calculated for $C_{17}H_{21}O_4N_4S_2$: 409.0999; found: 409.1002.

4-Methoxy-5-[(thiophen-2-yl)sulfanyl]-7-β-D-ribofuranosyl)-pyrrolo[2,3-*d*]pyrimidine (6-methoxy-7-[(thiophen-2-yl)sulfanyl]-9-β-D-ribofuranosyl)-7-deazapurine) (8h)



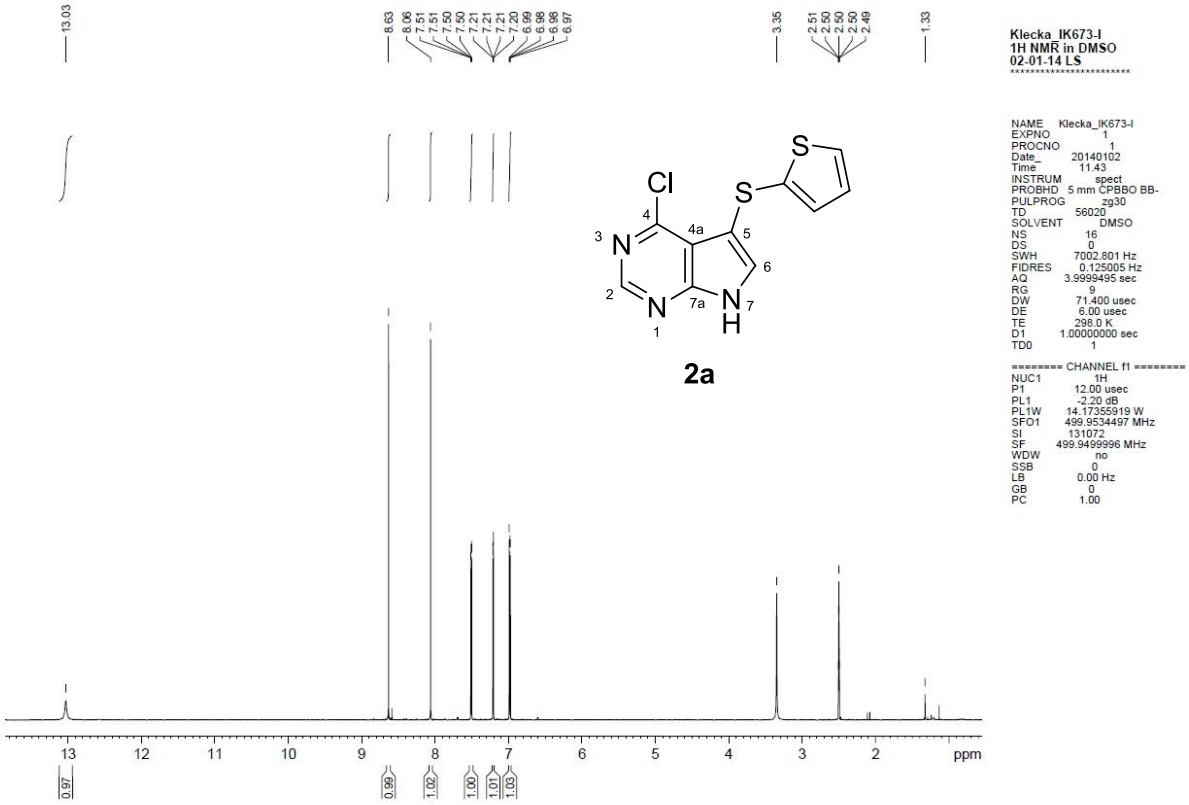
Deprotection and methoxylation of **4a** (712 mg, 1 mmol) according to the general procedure (4 equiv of NaOMe were used) afforded compound **8h** (305 mg, 77%) as white solid.

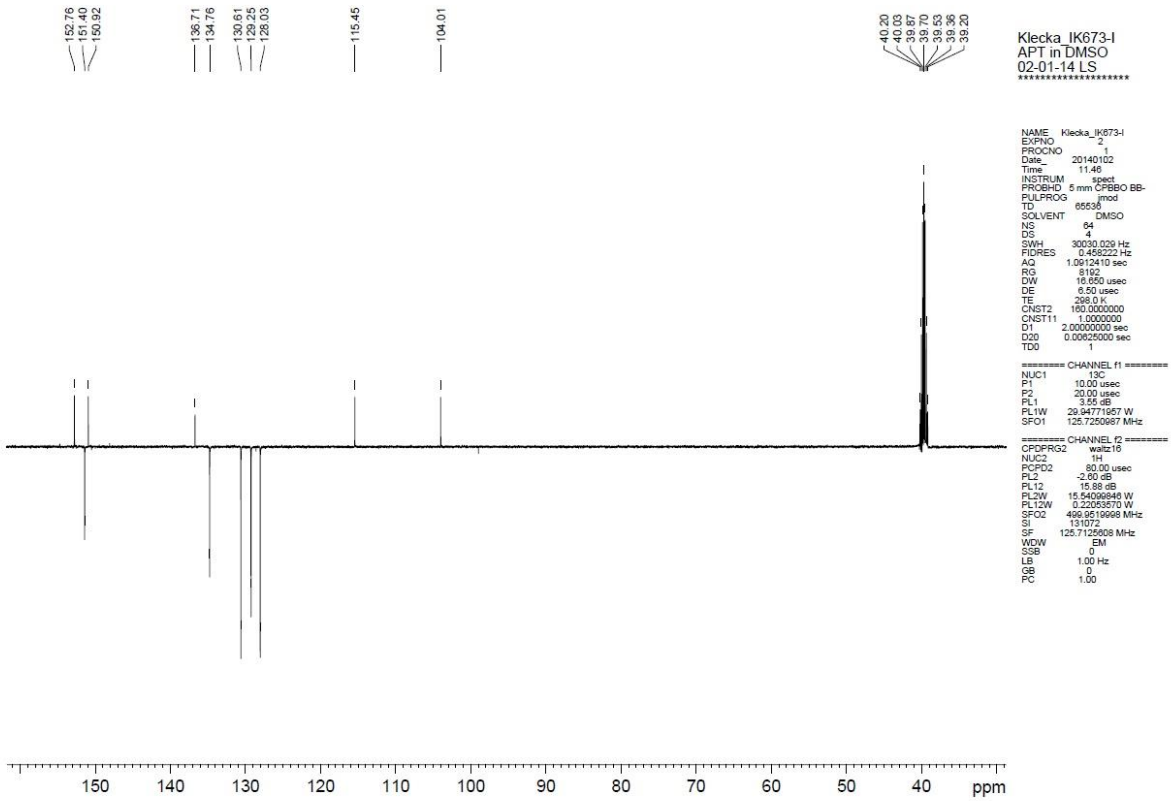
M.p. 194 °C $[\alpha]_D$ −51.1 (0.17). ^{1}H NMR (401.0 MHz, DMSO-d_6): 3.55 (dd, 1H, J_{gem} = 11.9 Hz, $J_{5'a,4'}$ = 3.8 Hz, H-5′a); 3.64 (dd, 1H, J_{gem} = 11.9 Hz, $J_{5'b,4'}$ = 3.8 Hz, H-5′b); 3.91 (q, 1H, $J_{4',5'a}$ = $J_{4',5'b}$ = $J_{4',3'}$ = 3.6 Hz, H-4′); 4.06 (s, 3H, CH_3O); 4.09 (dd, 1H, $J_{3',2'}$ = 5.1 Hz, $J_{3',4'}$ = 3.4 Hz, H-3'); 4.37 (dd, 1H, $J_{2',1'}$ = 6.1 Hz, $J_{2',3'}$ = 5.1 Hz, H-2'); 4.98 – 5.52 (m, 3H, OH-2′,3′,5′); 6.12 (d, 1H, $J_{1',2'}$ = 6.1 Hz, H-1'); 7.00 (dd, 1H, $J_{4,5}$ = 5.3 Hz, $J_{4,3}$ = 3.6 Hz, H-4-Sthienyl); 7.24 (dd, 1H, $J_{3,4}$ = 3.6 Hz, $J_{3,5}$ = 1.3 Hz, H-3-Sthienyl); 7.55 (dd, 1H, $J_{5,4}$ = 5.3 Hz, $J_{5,3}$ = 1.3 Hz, H-5-Sthienyl); 7.93 (s, 1H, H-6); 8.45 (s, 1H, H-2). ^{13}C NMR (100.8 MHz, DMSO-d_6): 53.83 (CH_3O); 61.55 (CH_2-5′); 70.60 (CH-3′); 74.36 (CH-2′); 85.54 (CH-4′); 87.39 (CH-1′); 105.39 and 105.53 (C-4a,5); 127.93 (CH-4-Sthienyl); 129.13 (CH-6); 129.96 (CH-5-Sthienyl); 132.18 (CH-3-Sthienyl); 135.41 (C-2-Sthienyl); 151.73 (CH-2); 152.47 (C-7a); 162.89 (C-4). IR (KBr): 3512, 3285, 3025, 2992, 2935, 2920, 2869, 1589, 1556, 1482, 1443, 1407, 1338, 1326, 1302, 1138, 1081, 1036, 704. HRMS (ESI) calculated for $C_{16}H_{18}O_5N_3S_2$: 396.0682; found: 396.0682.

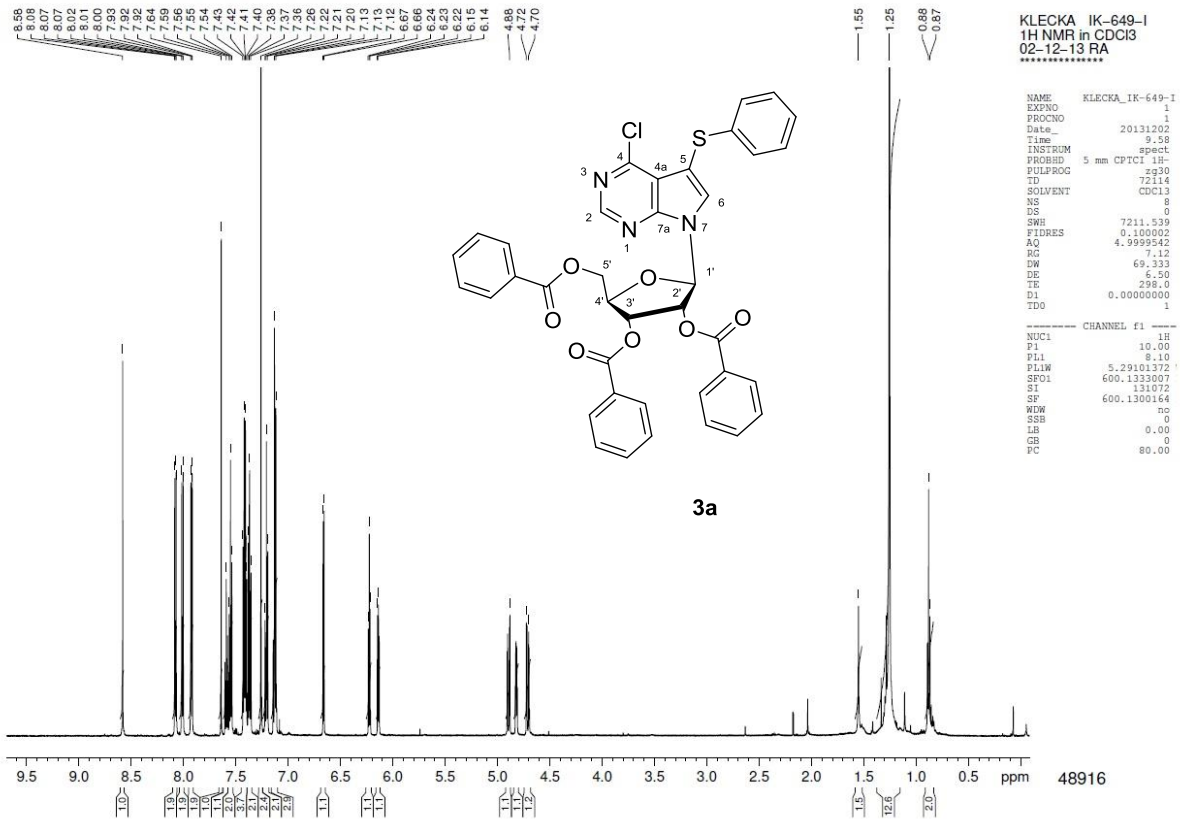
References:

1. Klečka, M.; Pohl, R.; Klepetářová, B; Hocek, M. *Org. Biomol. Chem.* **2013**, *11,* 5189-5193.

2. Copies of NMR spectra



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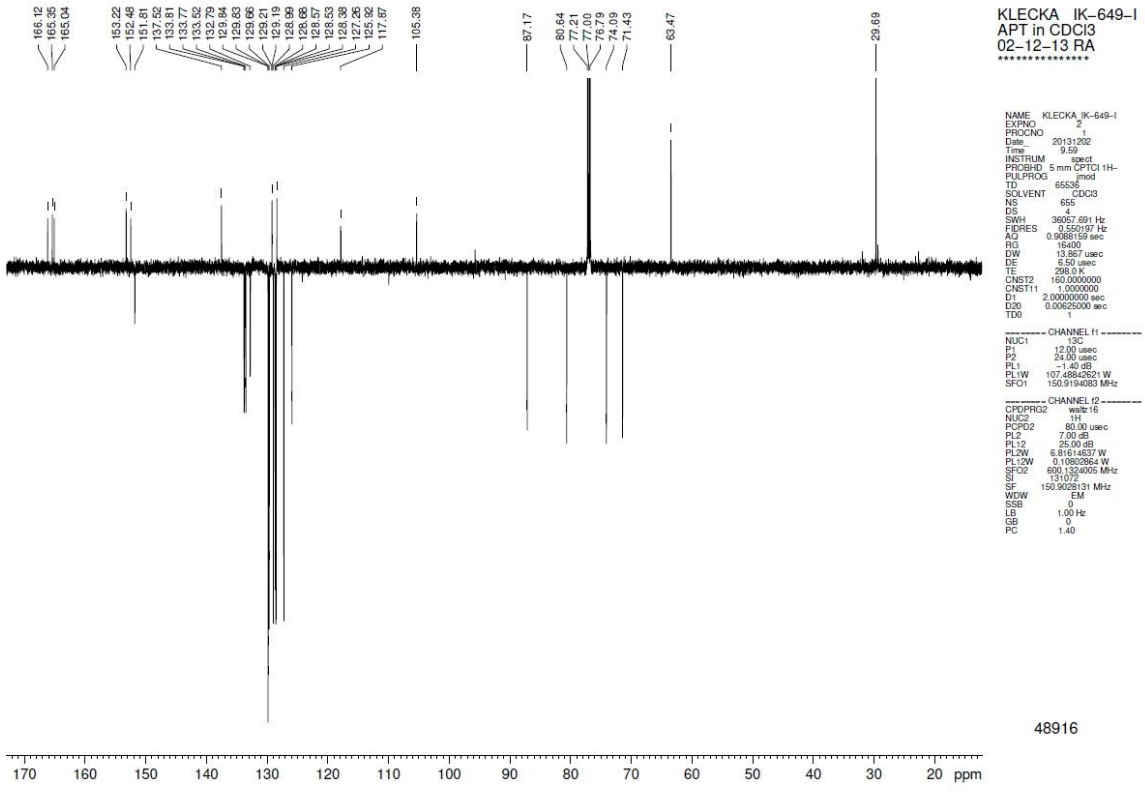
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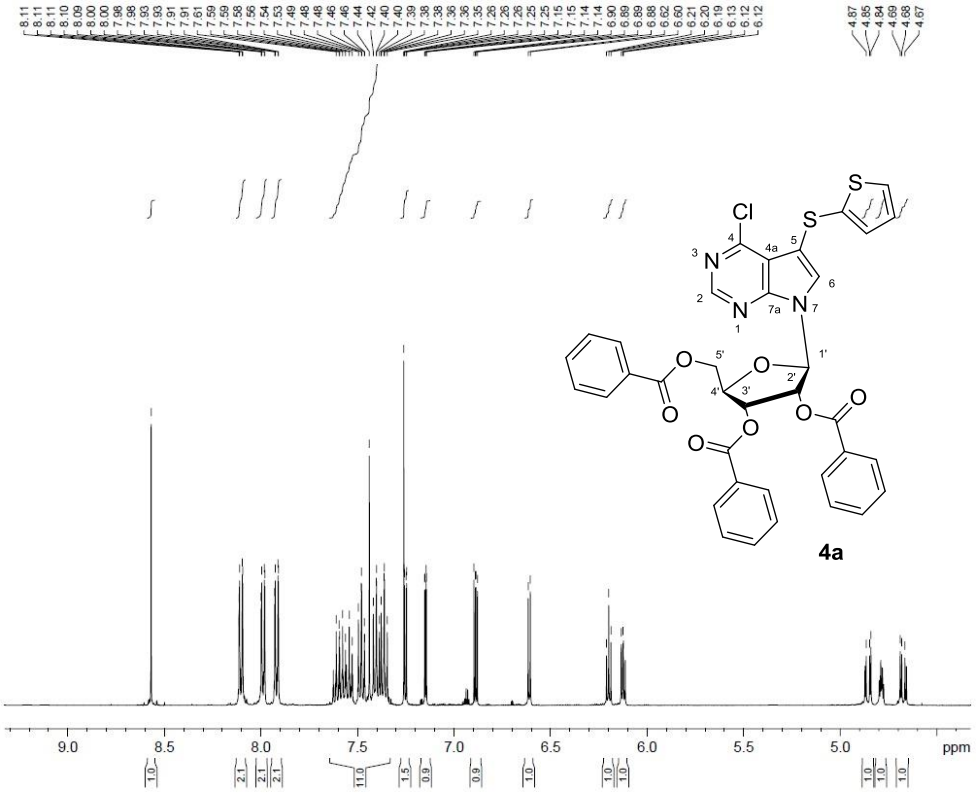

Klecka_IK689-I
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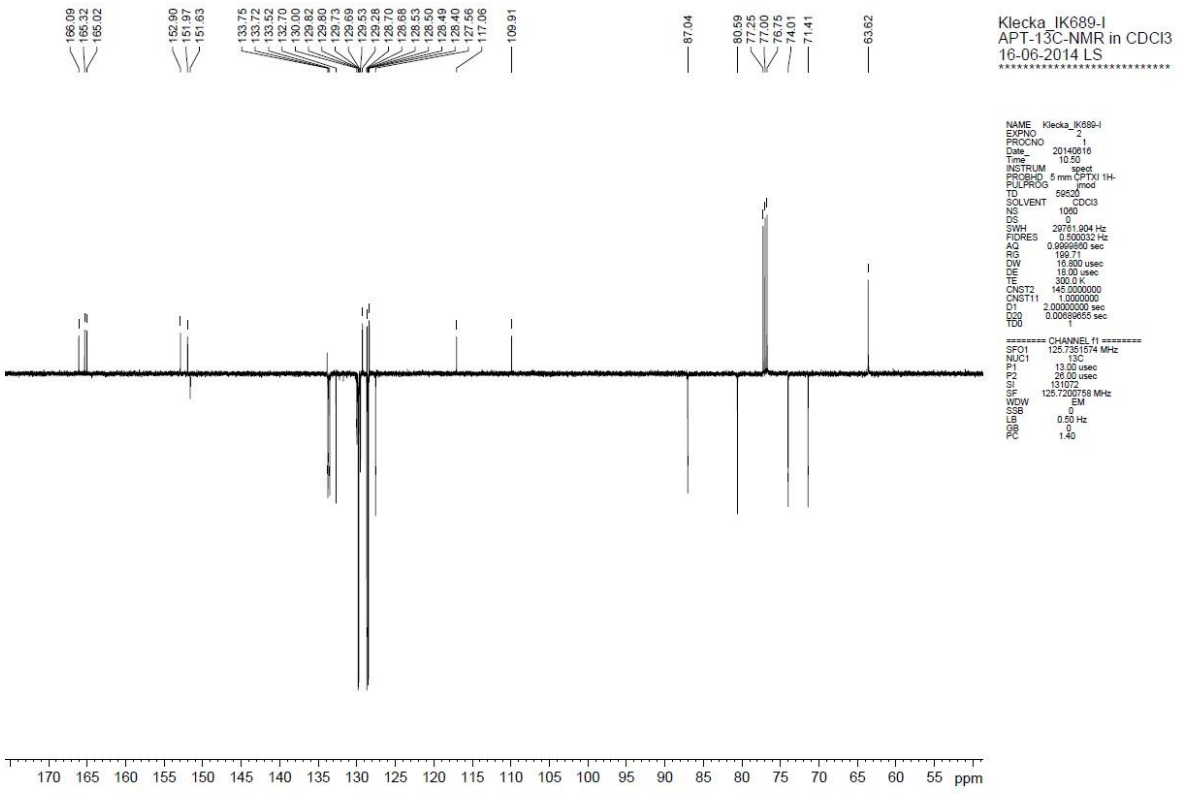
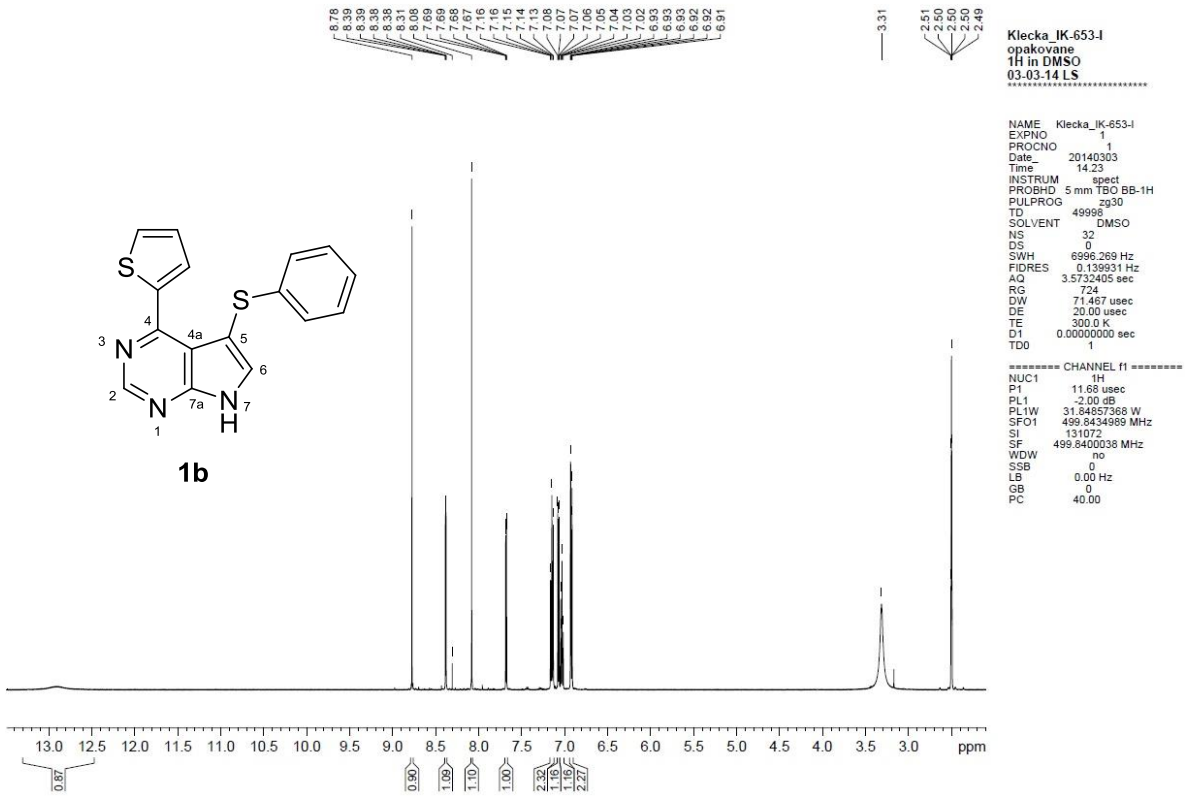
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.00
```

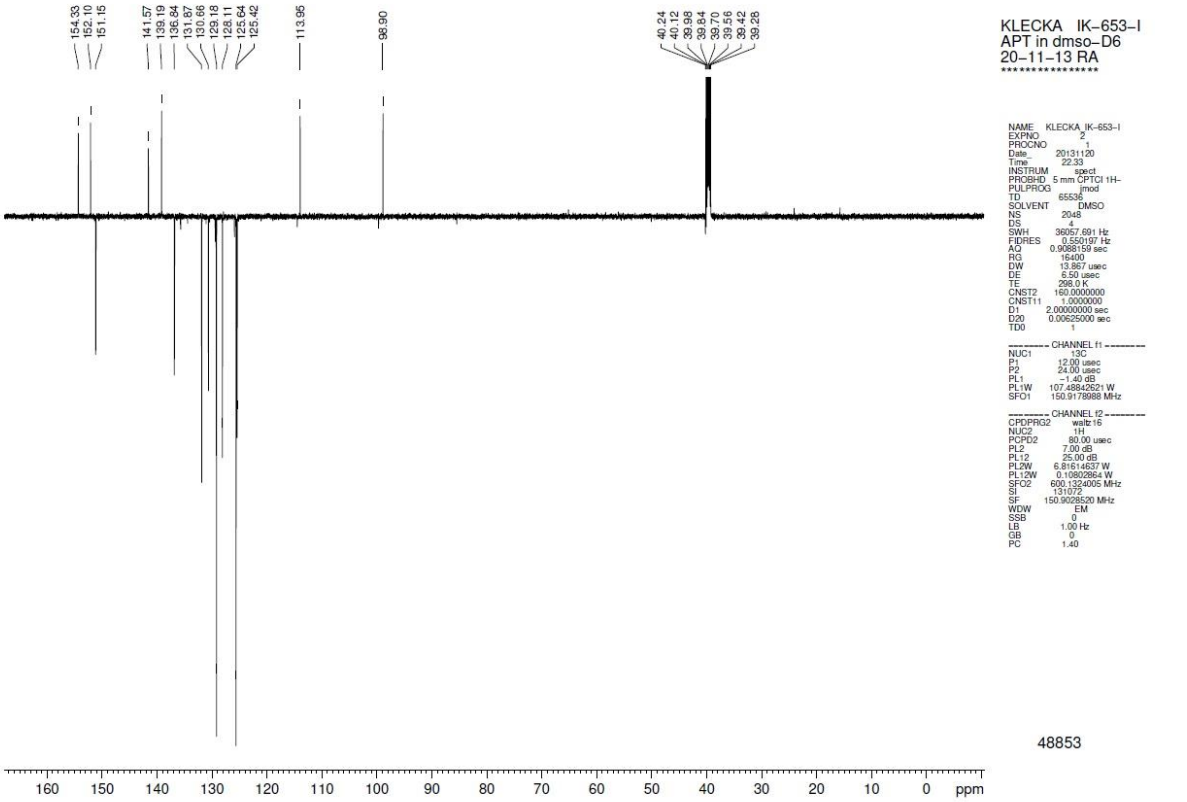


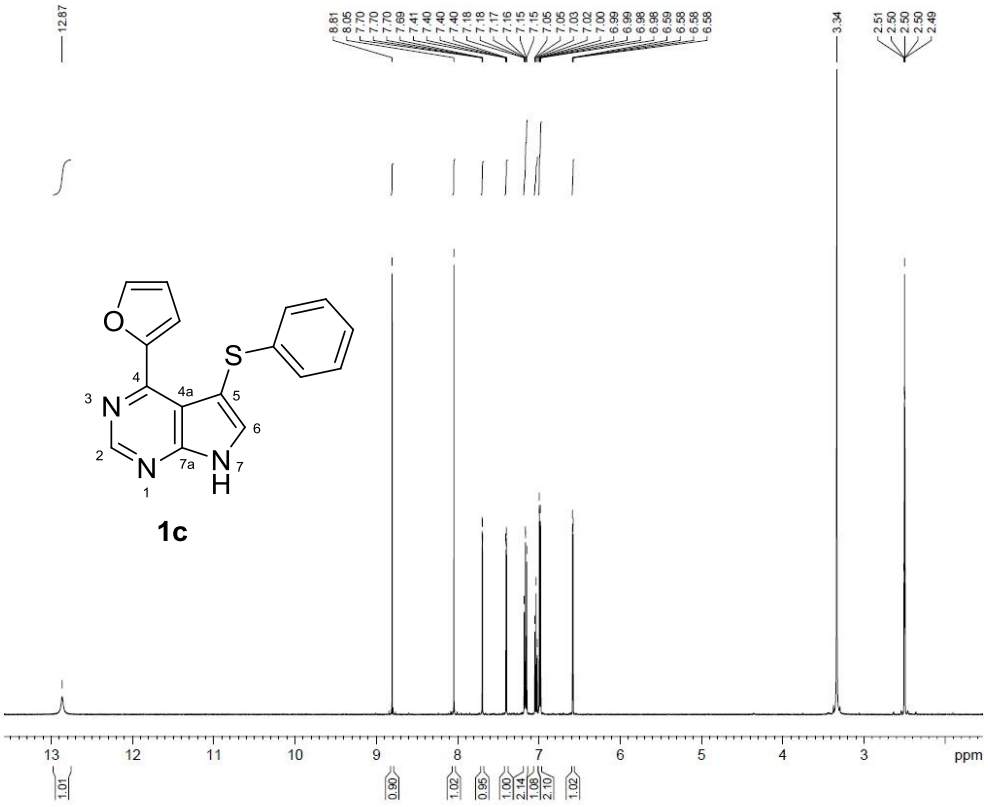
Klecka_IK689-I
APT-13C-NMR in CDCl3
16-06-2014 LS

```
NAME   Klecka_IK689-I
EXPNO              2
PROCNO             1
Date_       20140616
Time            10.50
INSTRUM         spect
PROBHD  5 mm CPTXI 1H-
PULPROG         jmod
TD             65620
SOLVENT        CDCl3
NS              1060
DS                 0
SWH        29761.904 Hz
FIDRES      0.500032 Hz
AQ          0.9999960 sec
RG             199.71
DW             16.800 usec
DE             18.00 usec
TE             300.0 K
CNST2          145.0000000
CNST11           1.0000000
D1          2.00000000 sec
D20         0.00689655 sec
TD0                1

======== CHANNEL f1 ========
SFO1       125.7351574 MHz
NUC1             13C
P1             13.00 usec
P2             26.00 usec
SI             131072
SF         125.7200768 MHz
WDW              EM
SSB               0
LB             0.50 Hz
GB                0
PC             1.40
```


8.78
8.39
8.39
8.38
8.38
8.31
8.08
7.69
7.68
7.67
7.16
7.16
7.15
7.14
7.13
7.08
7.07
7.07
7.06
7.05
7.04
7.03
7.02
6.93
6.93
6.92
6.92
6.91
3.31
2.51
2.50
2.50
2.50
2.49
Klecka_IK-653-I
opakovane
1H in DMSO
03-03-14 LS
NAME Klecka_IK-653-I
EXPNO 1
PROCNO 1
Date_ 20140303
Time 14.23
INSTRUM spect
PROBHD 5 mm TBO BB-1H
PULPROG zg30
TD 49998
SOLVENT DMSO
NS 32
DS 0
SWH 6996.269 Hz
FIDRES 0.139931 Hz
AQ 3.5732405 sec
RG 724
DW 71.467 usec
DE 20.00 usec
TE 300.0 K
D1 0.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 11.68 usec
PL1 -2.00 dB
PL1W 31.84857368 W
SFO1 499.8434989 MHz
SI 131072
SF 499.8400038 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 40.00
1b
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 ppm
0.67
0.90
1.09
1.10
1.00
2.32
1.16
1.16
2.27

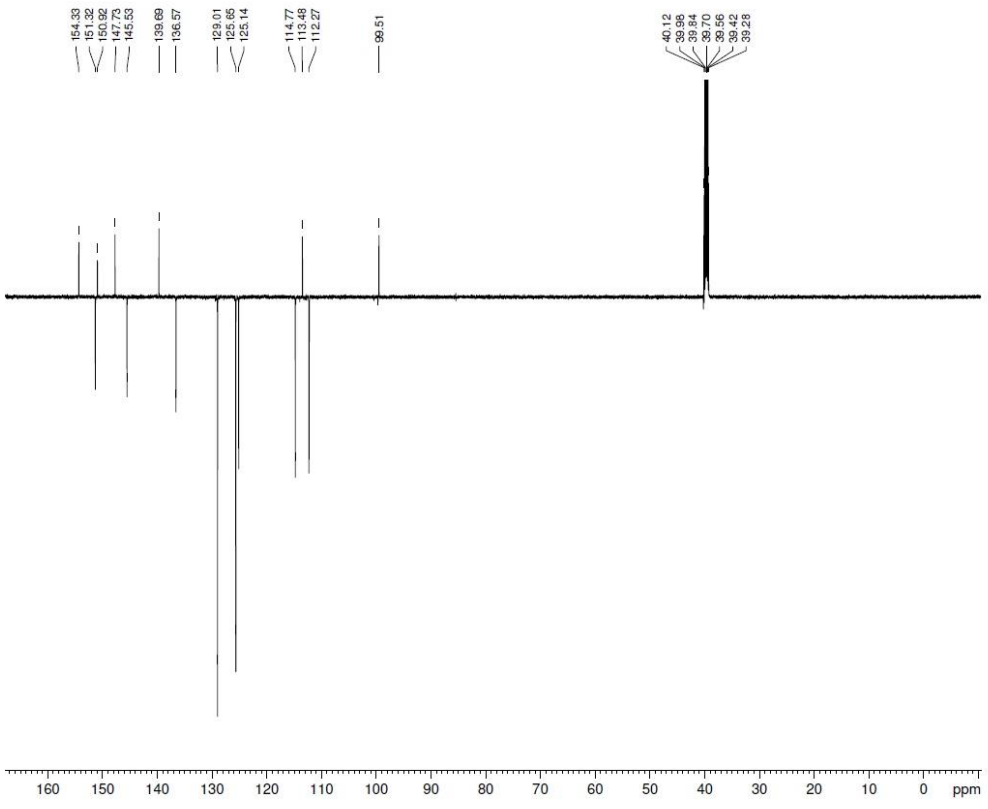

154.33
152.10
151.15
141.57
139.19
136.84
131.87
130.66
129.18
128.11
125.64
125.42
113.95
99.90
40.24
40.12
39.98
39.84
39.70
39.56
39.42
39.28
KLECKA IK-653-I
APT in dmso-D6
20-11-13 RA
NAME KLECKA_IK-653-I
EXPNO 2
PROCNO 1
Date_ 20131120
Time 22.33
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 16400
DW 13.867 usec
DE 6.50 usec
TE 298.0 K
CNST2 160.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00625000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 12.00 usec
P2 24.00 usec
PL1 -1.40 dB
PL1W 107.48842621 W
SFO1 150.9178988 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 7.00 dB
PL12 25.00 dB
PL2W 6.81614637 W
PL12W 0.10802864 W
SFO2 600.1324005 MHz
SI 131072
SF 150.9028520 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
48853
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



Klecka_IK-659-I
1H in DMSO
22-01-14 LS

NAME Klecka_IK-659-I
EXPNO 1
PROCNO 1
Date_ 20140122
Time 11.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 49998
SOLVENT DMSO
NS 32
DS 0
SWH 6996.269 Hz
FIDRES 0.139931 Hz
AQ 3.5732405 sec
RG 724
DW 71.467 usec
DE 20.00 usec
TE 300.0 K
D1 0.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.68 usec
PL1 -2.00 dB
PL1W 31.84857368 W
SFO1 499.8434989 MHz
SI 131072
SF 499.8400038 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 40.00



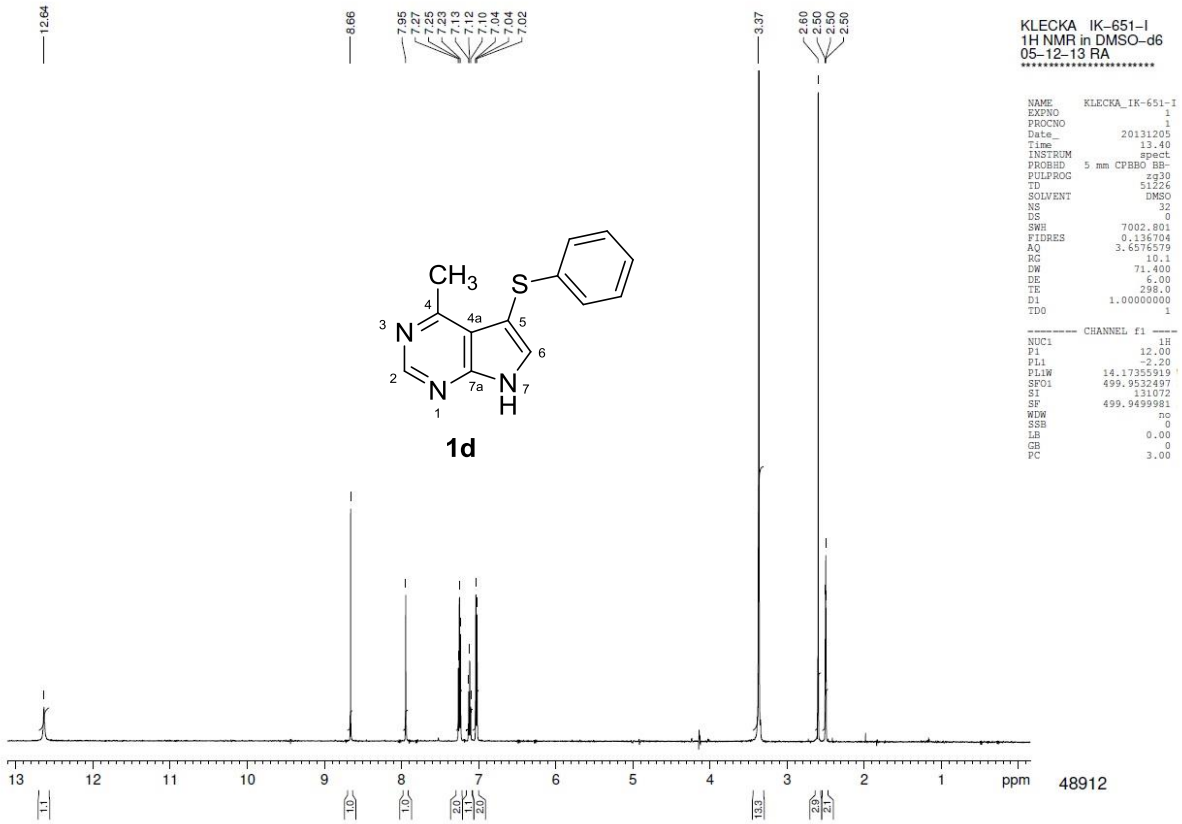
KLECKA IK-659-I
APT in dmso-D6
20-11-13 RA

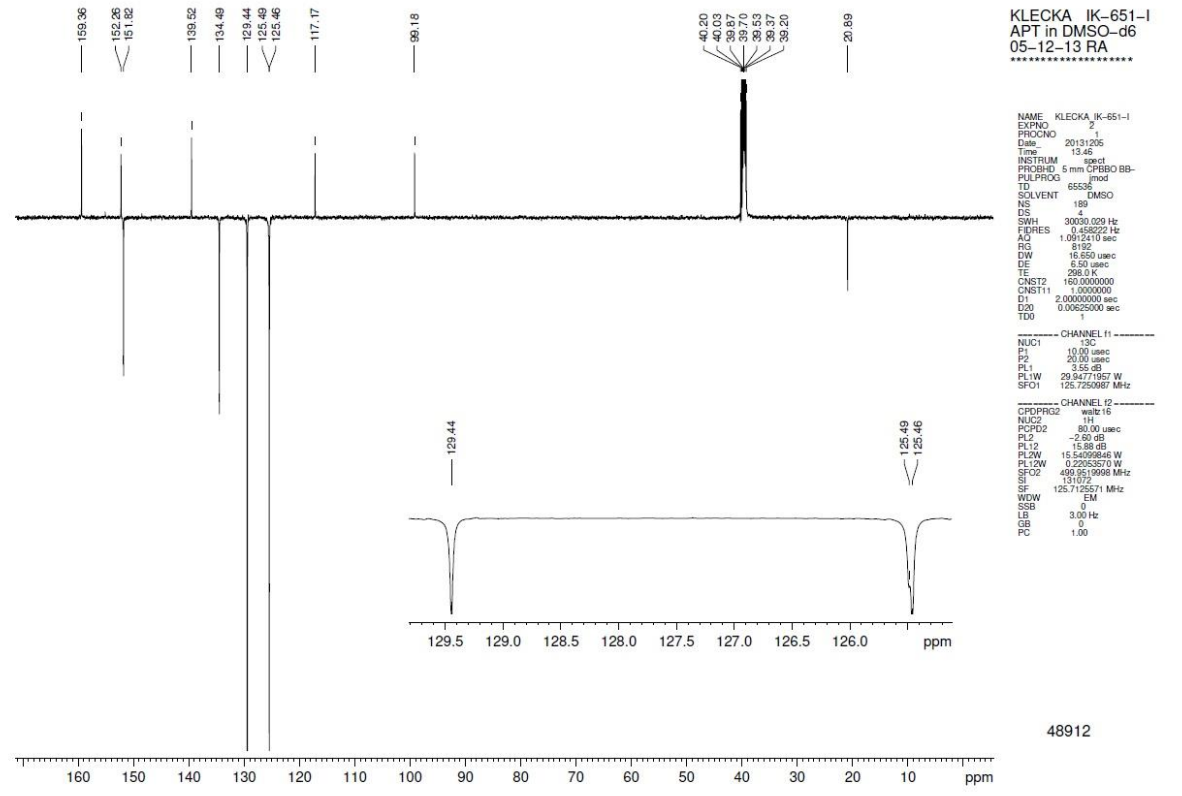
NAME KLECKA_IK-659-I
EXPNO 2
PROCNO 1
Date_ 20131121
Time 4.18
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 16400
DW 13.867 usec
DE 6.50 usec
TE 298.0 K
CNST2 160.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00625000 sec
TD0 1

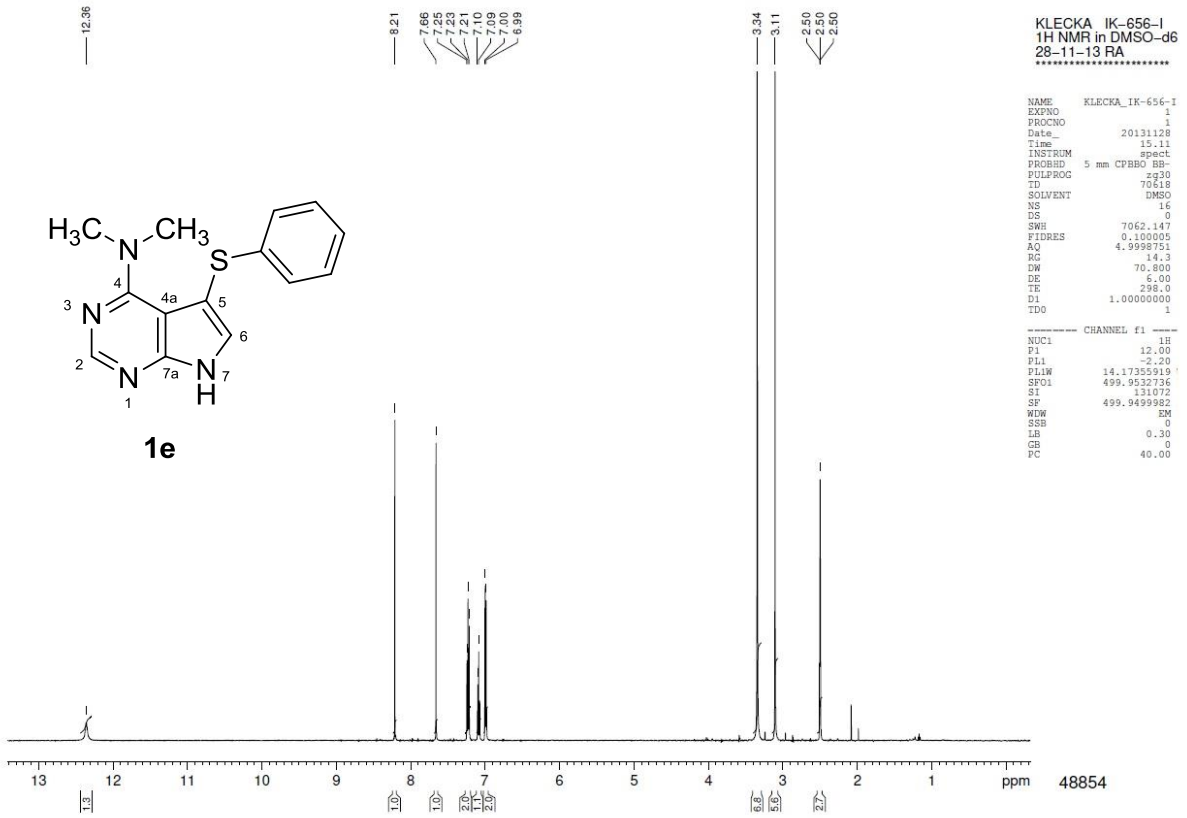
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
P2 24.00 usec
PL1 -1.40 dB
PL1W 107.48842621 W
SFO1 150.9179988 MHz

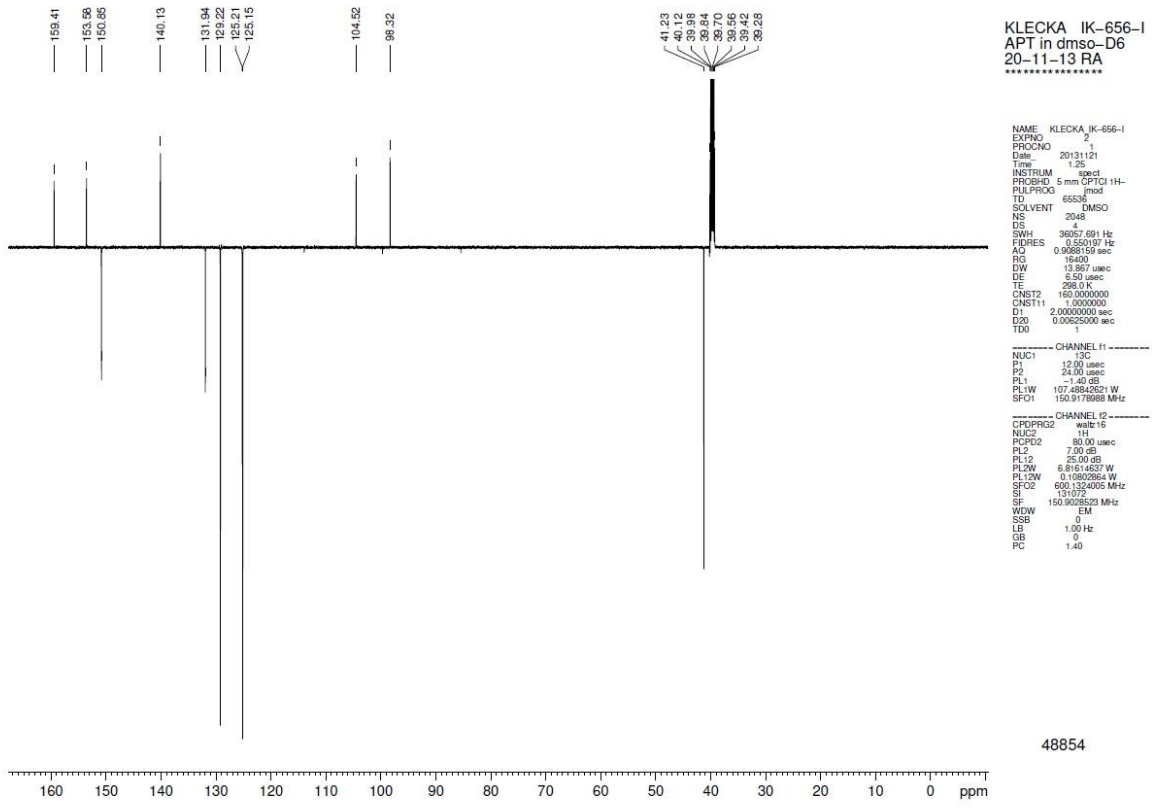
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 7.00 dB
PL12 25.00 dB
PL2W 6.81614637 W
PL12W 0.10802864 W
SFO2 600.1324005 MHz
SI 131072
SF 150.9025525 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

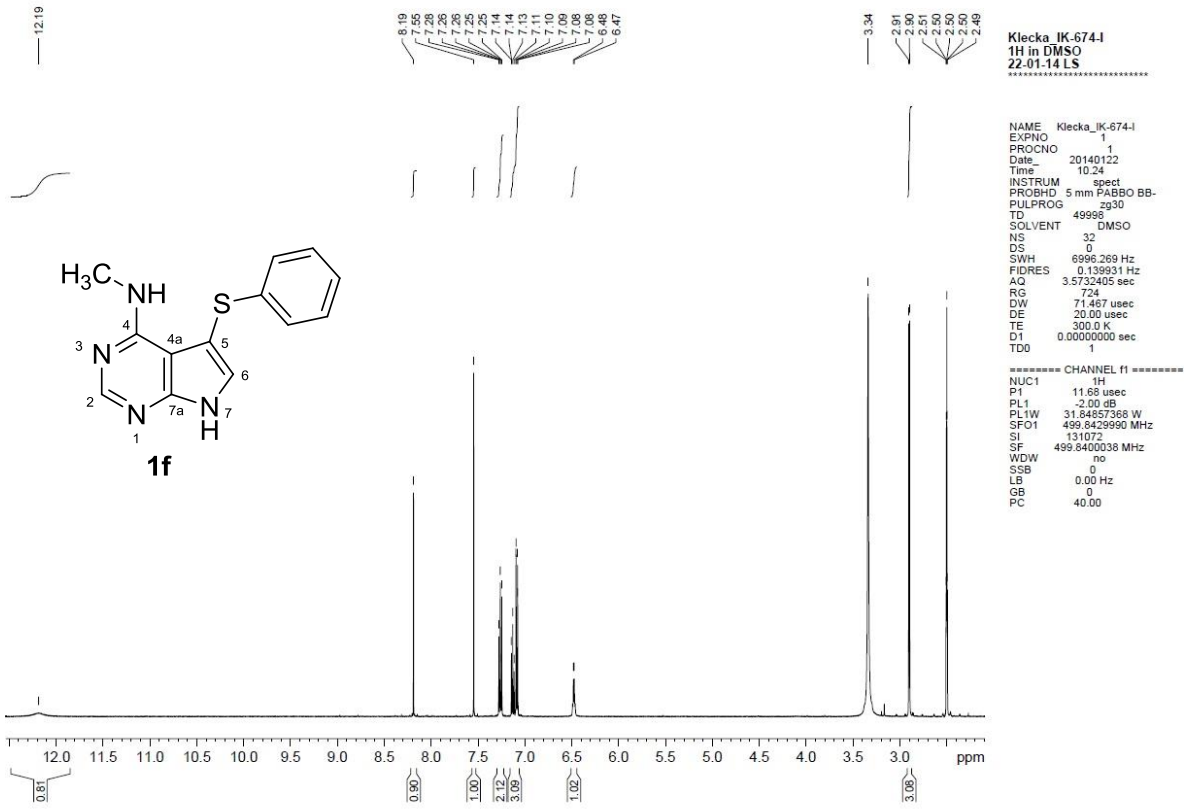
48855

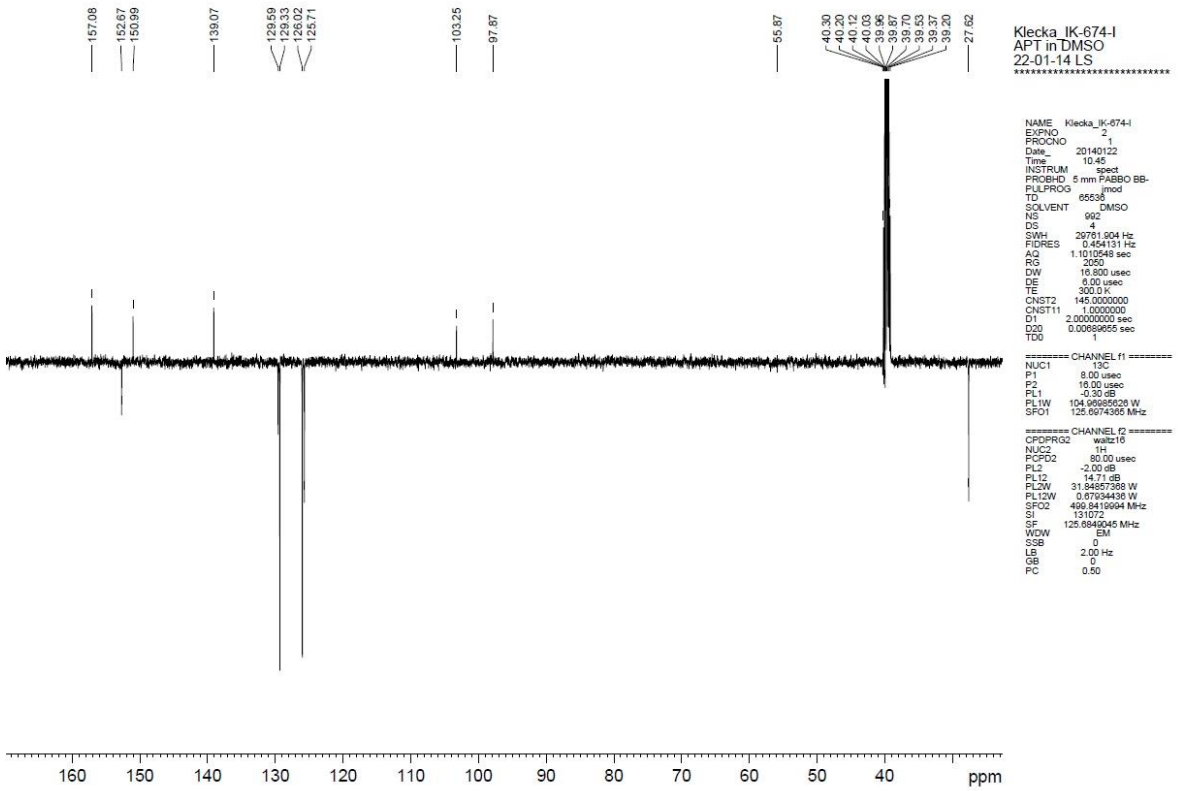

12.64
8.66
7.95
7.27
7.25
7.23
7.13
7.12
7.10
7.04
7.02
3.37
2.60
2.50
2.50
2.50
KLECKA IK-651-I
1H NMR in DMSO-d6
05-12-13 RA
NAME KLECKA_IK-651-I
EXPNO 1
PROCNO 1
Date_ 20131205
Time 13.40
INSTRUM spect
PROBHD 5 mm CPBBO BB-
PULPROG zg30
TD 51226
SOLVENT DMSO
NS 32
DS 0
SWH 7002.801
FIDRES 0.136704
AQ 3.6576579
RG 10.1
DW 71.400
DE 6.00
TE 298.0
D1 1.00000000
TD0 1
CHANNEL f1
NUC1 1H
P1 12.00
PL1 -2.20
PL1W 14.17355919
SFO1 499.9532497
SI 131072
SF 499.9499981
WDW no
SSB 0
LB 0.00
GB 0
PC 3.00
CH3
S
N
N
N
H
1d
ppm
48912

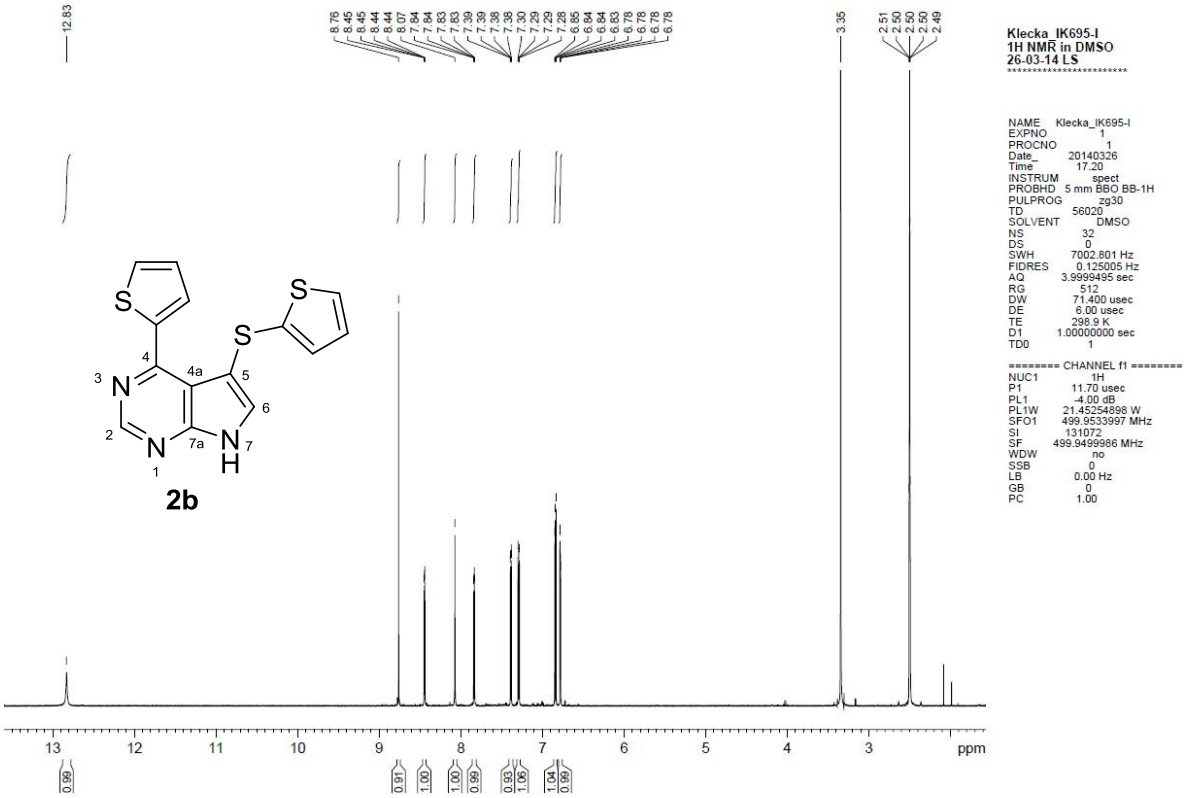

159.36
152.26
151.82
139.52
134.49
129.44
125.49
125.46
117.17
99.18
40.20
40.03
39.87
39.70
39.53
39.37
39.20
29.89
KLECKA IK-651-I
APT in DMSO-d6
05-12-13 RA
NAME KLECKA_IK-651-I
EXPNO 2
PROCNO 1
Date_ 20131205
Time 13.46
INSTRUM spect
PROBHD 5 mm CPBBO BB-
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 189
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 8192
DW 16.650 usec
DE 6.50 usec
TE 298.0 K
CNST2 160.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00625000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 10.00 usec
P2 20.00 usec
PL1 3.55 dB
PL1W 29.94771957 W
SFO1 125.7250987 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.60 dB
PL12 15.88 dB
PL2W 15.54099846 W
PL12W 0.22053570 W
SFO2 499.9519998 MHz
SI 131072
SF 125.7125571 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
129.44
125.49
125.46
ppm
48912

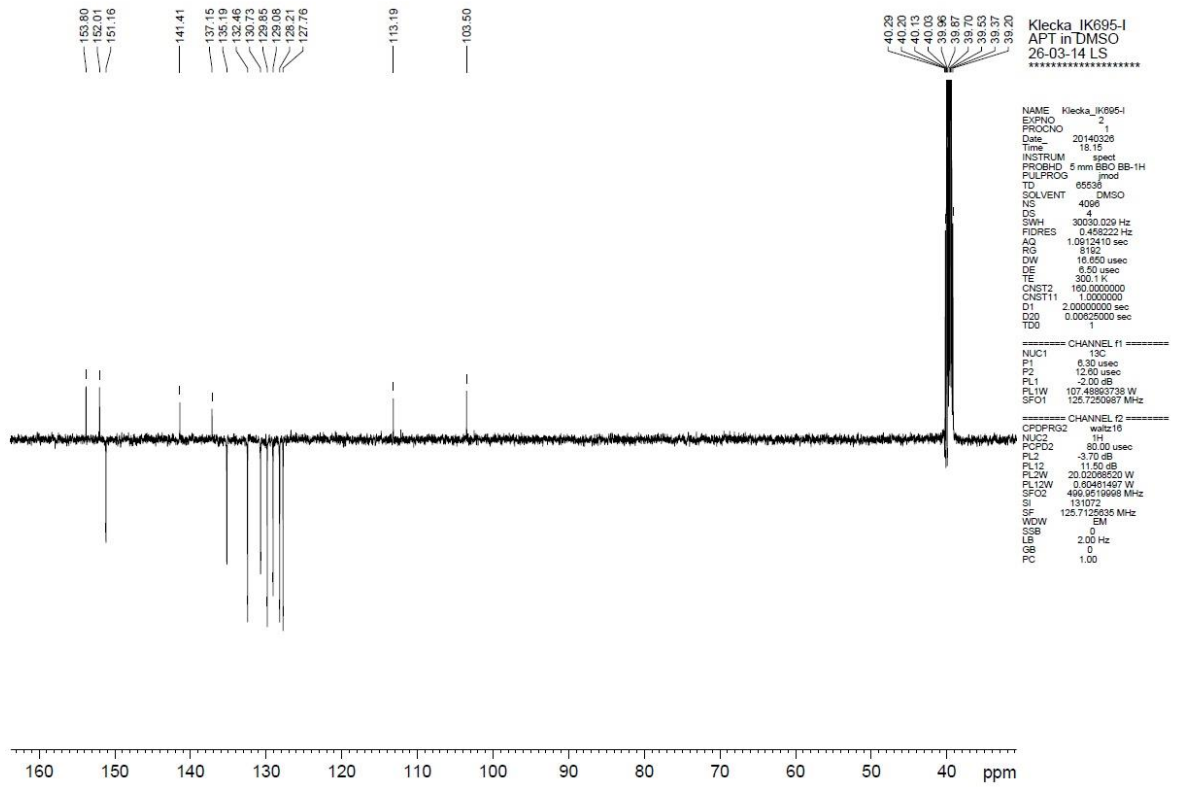

12.36
8.21
7.66
7.25
7.23
7.21
7.10
7.09
7.00
6.99
3.34
3.11
2.50
2.50
2.50
KLECKA IK-656-I
1H NMR in DMSO-d6
28-11-13 RA
NAME KLECKA_IK-656-I
EXPNO 1
PROCNO 1
Date_ 20131128
Time 15.11
INSTRUM spect
PROBHD 5 mm CPBBO BB-
PULPROG zg30
TD 70618
SOLVENT DMSO
NS 16
DS 0
SWH 7062.147
FIDRES 0.100005
AQ 4.9998751
RG 14.3
DW 70.800
DE 6.00
TE 298.0
D1 1.00000000
TD0 1
CHANNEL f1
NUC1 1H
P1 12.00
PL1 -2.20
PL1W 14.17355919
SFO1 499.9532736
SI 131072
SF 499.9499982
WDW EM
SSB 0
LB 0.30
GB 0
PC 40.00
H3C
N
CH3
S
4
4a
5
3
N
6
2
N
7a
N
7
H
1
1e
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.3
1.0
1.0
2.0
1.1
2.0
6.8
5.6
2.7
48854

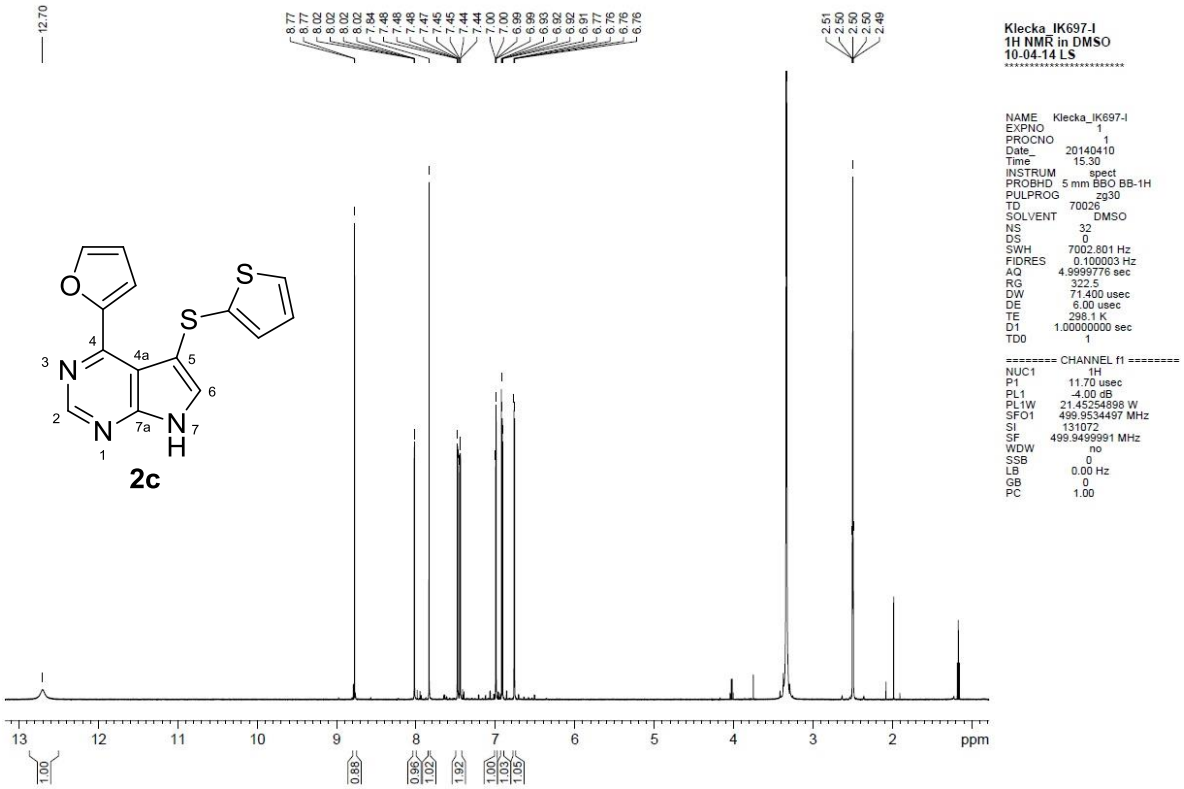

159.41
153.58
150.85
140.13
131.94
129.22
125.21
125.15
104.52
98.32
41.23
40.12
39.98
39.84
39.70
39.56
39.42
39.28
KLECKA IK-656-I
APT in dmso-D6
20-11-13 RA
NAME KLECKA_IK-656-I
EXPNO 2
PROCNO 1
Date_ 20131121
Time 1.25
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 16400
DW 13.867 usec
DE 6.50 usec
TE 298.0 K
CNST2 160.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00625000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 12.00 usec
P2 24.00 usec
PL1 -1.40 dB
PL1W 107.48842621 W
SFO1 150.9178988 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 7.00 dB
PL12 25.00 dB
PL2W 6.81614637 W
PL12W 0.10802864 W
SFO2 600.1324005 MHz
SI 131072
SF 150.9028523 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
48854

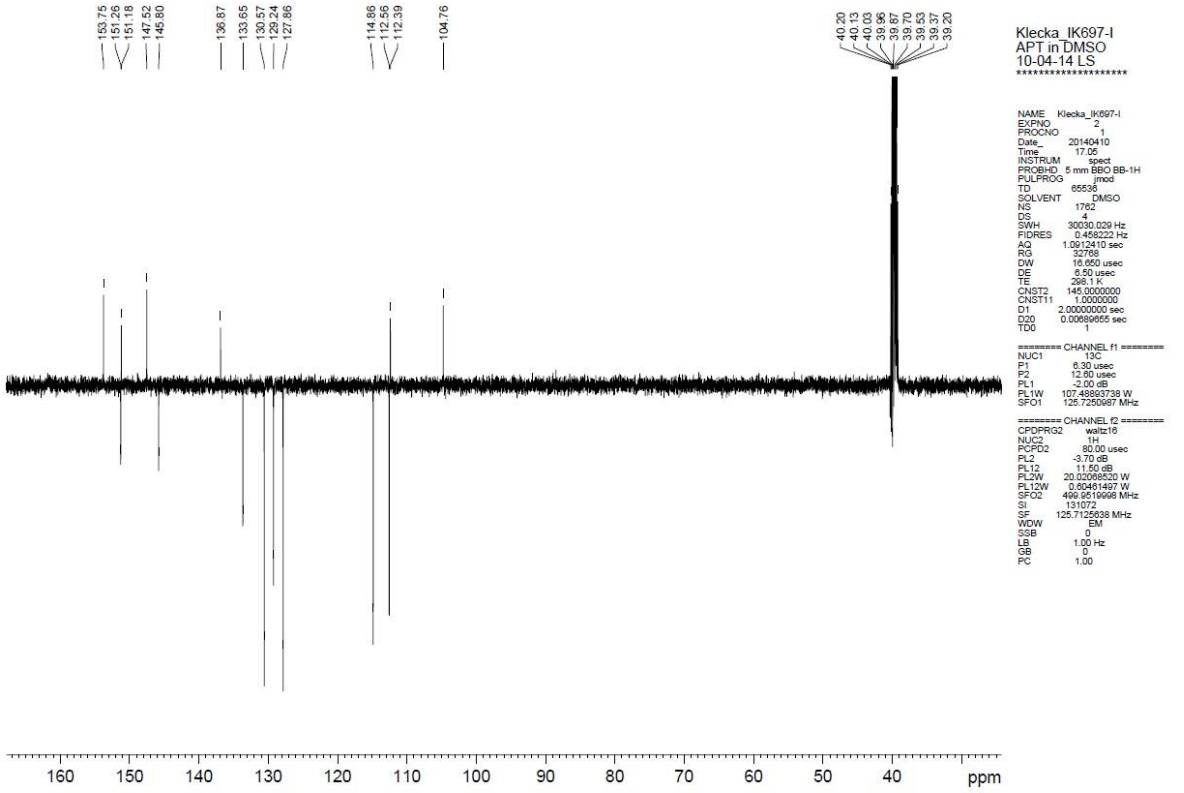

12.19
8.19
7.55
7.28
7.26
7.25
7.25
7.14
7.13
7.11
7.10
7.09
7.08
7.08
6.48
6.47
3.34
2.91
2.90
2.51
2.50
2.50
2.50
2.49
Klecka_IK-674-I
1H in DMSO
22-01-14 LS
NAME Klecka_IK-674-I
EXPNO 1
PROCNO 1
Date_ 20140122
Time 10.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 49998
SOLVENT DMSO
NS 32
DS 0
SWH 6996.269 Hz
FIDRES 0.139931 Hz
AQ 3.5732405 sec
RG 724
DW 71.467 usec
DE 20.00 usec
TE 300.0 K
D1 0.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.68 usec
PL1 -2.00 dB
PL1W 31.84857368 W
SFO1 499.8429990 MHz
SI 131072
SF 499.8400038 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 40.00
H3C
NH
S
N
N
N
H
1
2
3
4
4a
5
6
7
7a
1f
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
ppm
0.81
0.90
1.00
2.12
3.09
1.02
3.06


157.08
152.67
150.99
139.07
129.59
129.33
126.02
125.71
103.25
97.87
55.87
40.30
40.20
40.12
40.03
39.96
39.87
39.70
39.53
39.37
39.20
27.62
Klecka_IK-674-I
APT in DMSO
22-01-14 LS
NAME Klecka_IK-674-I
EXPNO 2
PROCNO 1
Date_ 20140122
Time 10.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 992
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
P2 16.00 usec
PL1 -0.30 dB
PL1W 104.96985626 W
SFO1 125.6974365 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.71 dB
PL2W 31.84857368 W
PL12W 0.67934436 W
SFO2 499.8419994 MHz
SI 131072
SF 125.6849045 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 0.50
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm


12.83
8.76
8.45
8.45
8.44
8.44
8.07
7.64
7.64
7.63
7.63
7.39
7.38
7.38
7.30
7.29
7.29
7.28
6.85
6.84
6.84
6.83
6.78
6.78
6.78
6.78
3.35
2.51
2.50
2.50
2.50
2.49
S
S
S
4
4a
5
3
N
6
2
N
7a
N
7
H
1
2b
13
12
11
10
9
8
7
6
5
4
3
ppm
0.99
0.91
1.00
1.00
0.99
0.93
1.06
1.04
0.99
Klecka_IK695-I
1H NMR in DMSO
26-03-14 LS
NAME Klecka_IK695-I
EXPNO 1
PROCNO 1
Date_ 20140326
Time 17.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 56020
SOLVENT DMSO
NS 32
DS 0
SWH 7002.801 Hz
FIDRES 0.125005 Hz
AQ 3.9999495 sec
RG 512
DW 71.400 usec
DE 6.00 usec
TE 298.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 -4.00 dB
PL1W 21.45254898 W
SFO1 499.9533997 MHz
SI 131072
SF 499.9499986 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00


153.80
152.01
151.16
141.41
137.15
135.19
132.46
130.73
129.85
129.08
128.21
127.76
113.19
103.50
40.29
40.20
40.13
40.03
39.96
39.87
39.70
39.53
39.37
39.20
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm
Klecka_IK695-I
APT in DMSO
26-03-14 LS
NAME Klecka_IK695-I
EXPNO 2
PROCNO 1
Date_ 20140326
Time 18.15
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 4096
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 8192
DW 16.650 usec
DE 6.50 usec
TE 300.1 K
CNST2 160.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00625000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.30 usec
P2 12.60 usec
PL1 -2.00 dB
PL1W 107.49883738 W
SFO1 125.7250987 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.70 dB
PL12 11.50 dB
PL2W 20.02066520 W
PL12W 0.80461497 W
SFO2 499.9519998 MHz
SI 131072
SF 125.7125935 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00


12.70
8.77
8.77
8.02
8.02
8.02
7.94
7.48
7.48
7.48
7.47
7.45
7.45
7.44
7.44
7.00
7.00
6.99
6.99
6.93
6.92
6.92
6.91
6.77
6.76
6.76
6.76
2.51
2.50
2.50
2.50
2.49
Klecka_IK697-I
1H NMR in DMSO
10-04-14 LS
NAME Klecka_IK697-I
EXPNO 1
PROCNO 1
Date_ 20140410
Time 15.30
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 70026
SOLVENT DMSO
NS 32
DS 0
SWH 7002.801 Hz
FIDRES 0.100003 Hz
AQ 4.9999776 sec
RG 322.5
DW 71.400 usec
DE 6.00 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 -4.00 dB
PL1W 21.45254898 W
SFO1 499.9534497 MHz
SI 131072
SF 499.9499991 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
O
S
S
N
N
N
H
4
4a
5
6
7
7a
3
2
1
2c
13
12
11
10
9
8
7
6
5
4
3
2
ppm
1.00
0.88
0.96
1.02
1.92
1.00
1.03
1.05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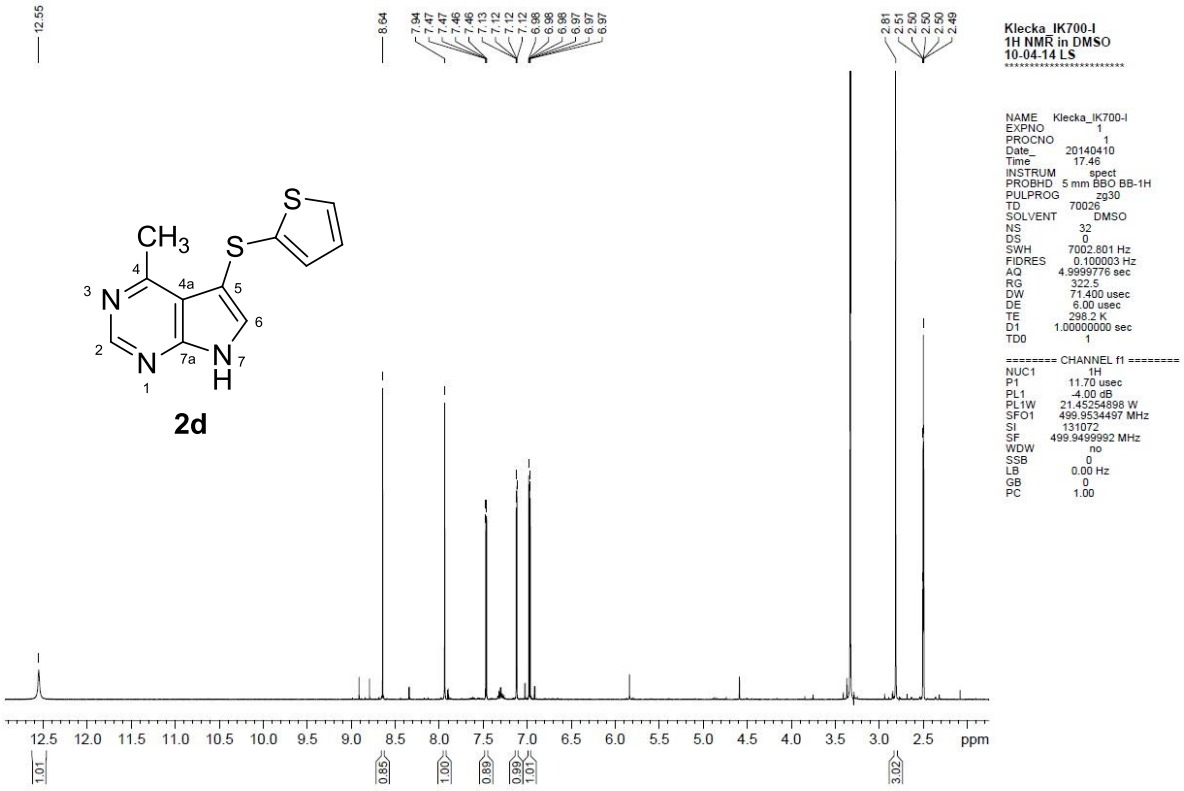
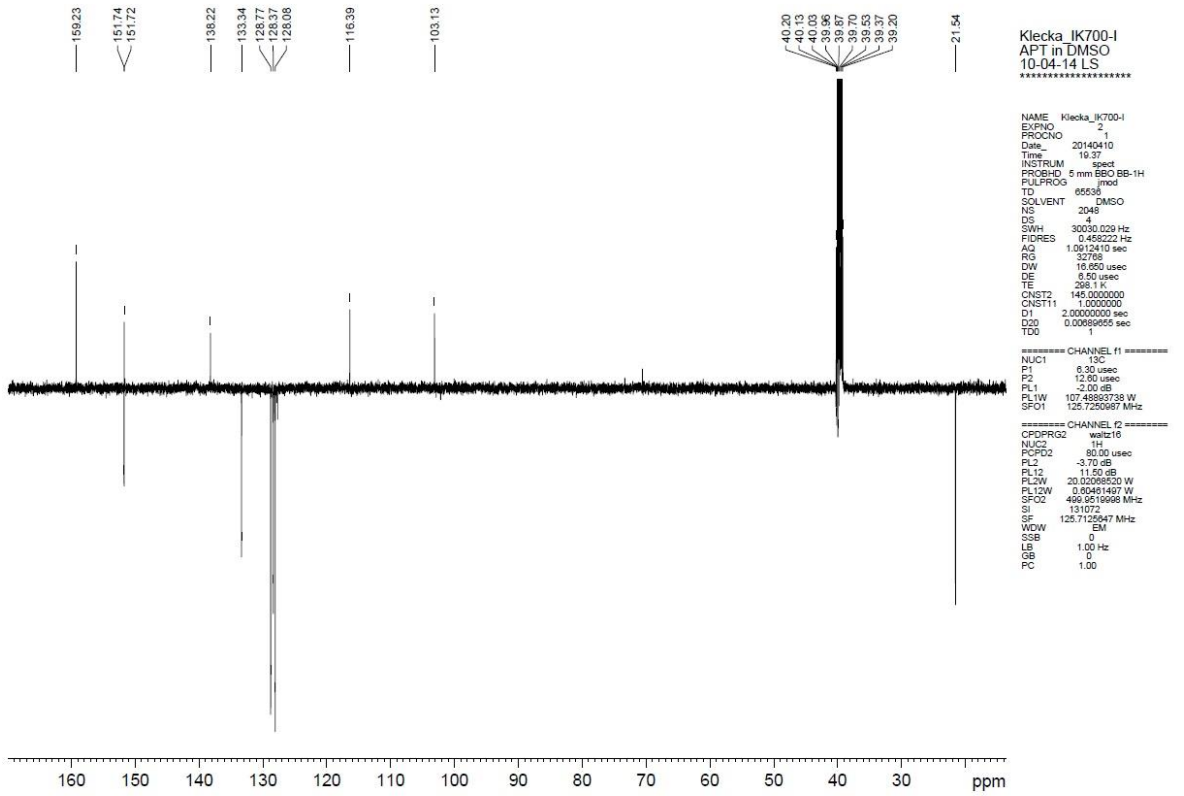
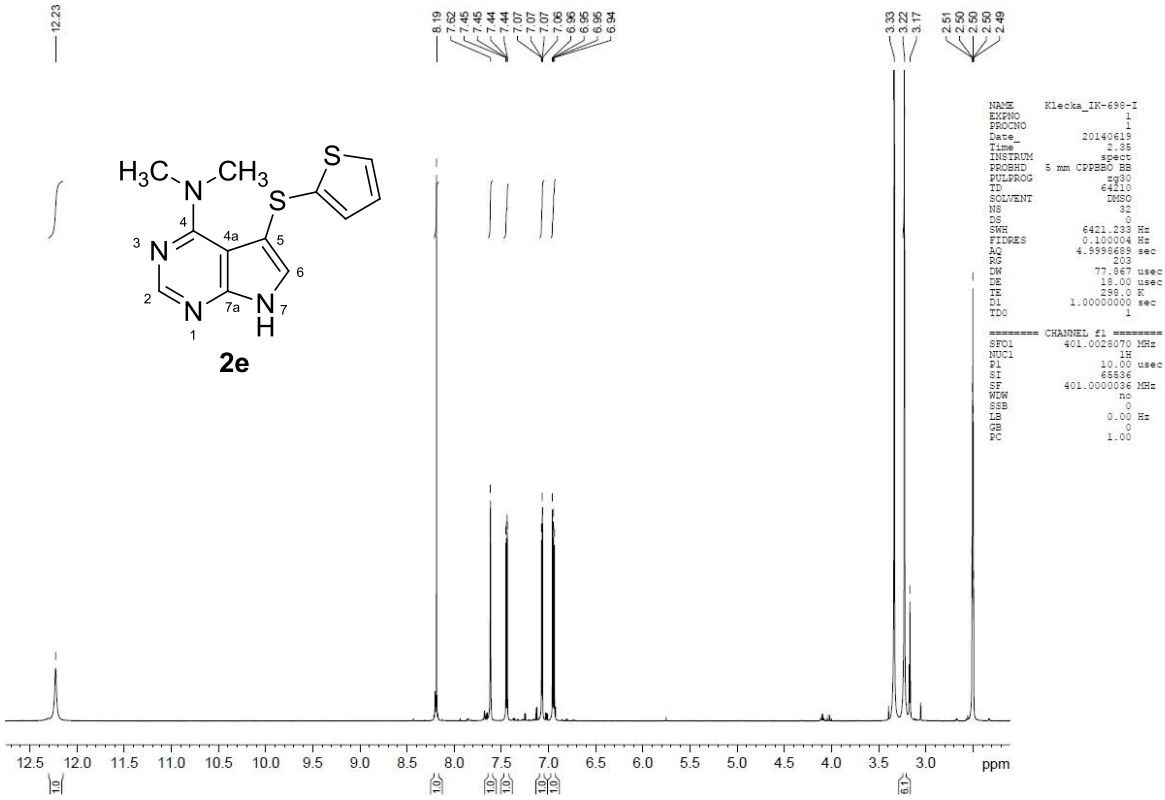
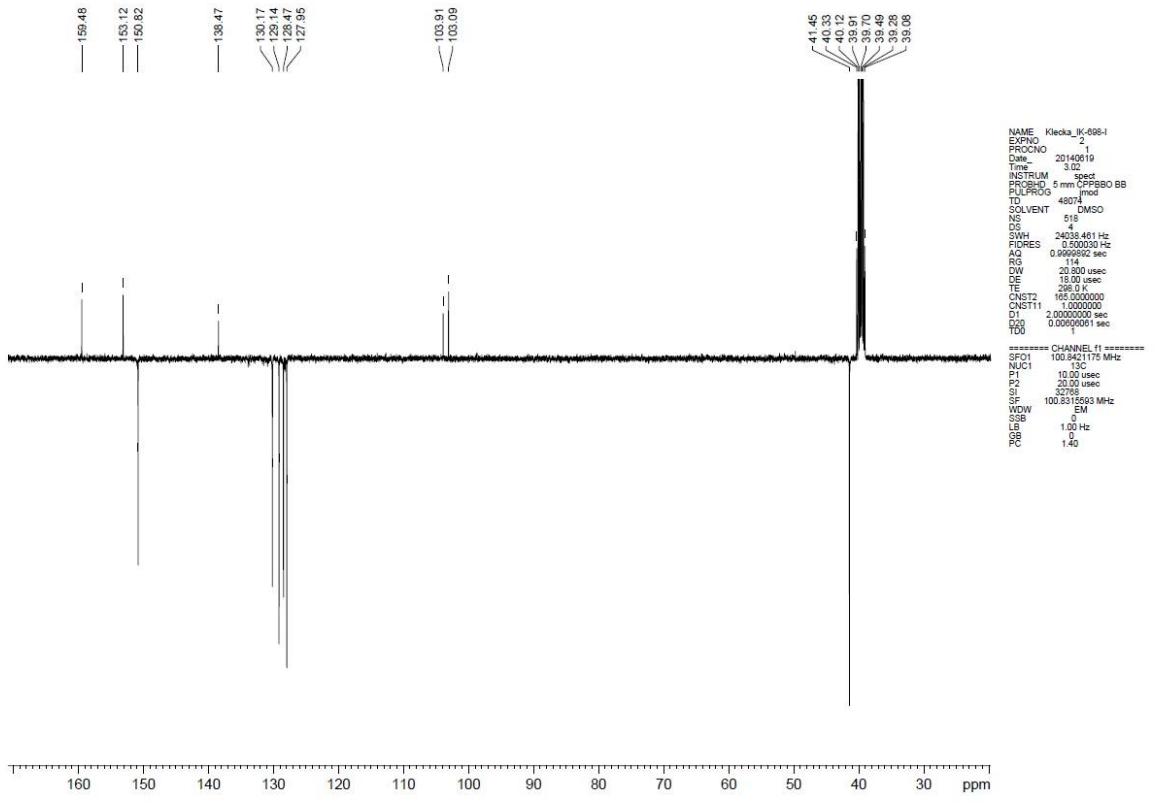
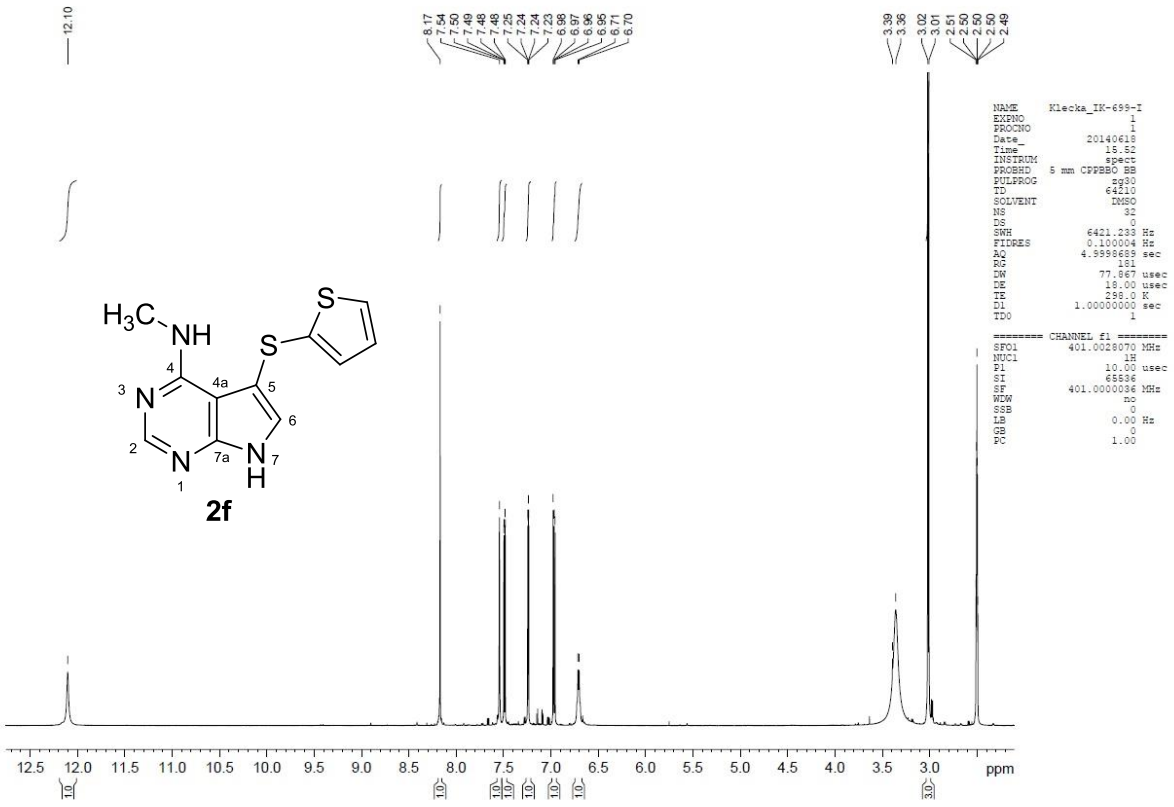
151.26
151.18
147.52
145.80
136.87
133.65
130.57
129.24
127.86
114.86
112.55
112.39
104.76
40.20
40.13
40.03
39.96
39.87
39.70
39.53
39.37
39.20
Klecka_IK697-I
APT in DMSO
10-04-14 LS
NAME Klecka_IK697-I
EXPNO 2
PROCNO 1
Date_ 20140410
Time 17.05
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 1762
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.1 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.30 usec
P2 12.60 usec
PL1 -2.00 dB
PL1W 107.48888738 W
SFO1 125.7250987 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.70 dB
PL12 11.50 dB
PL2W 20.02066520 W
PL12W 0.80461497 W
SFO2 499.9519998 MHz
SI 131072
SF 125.7125838 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm

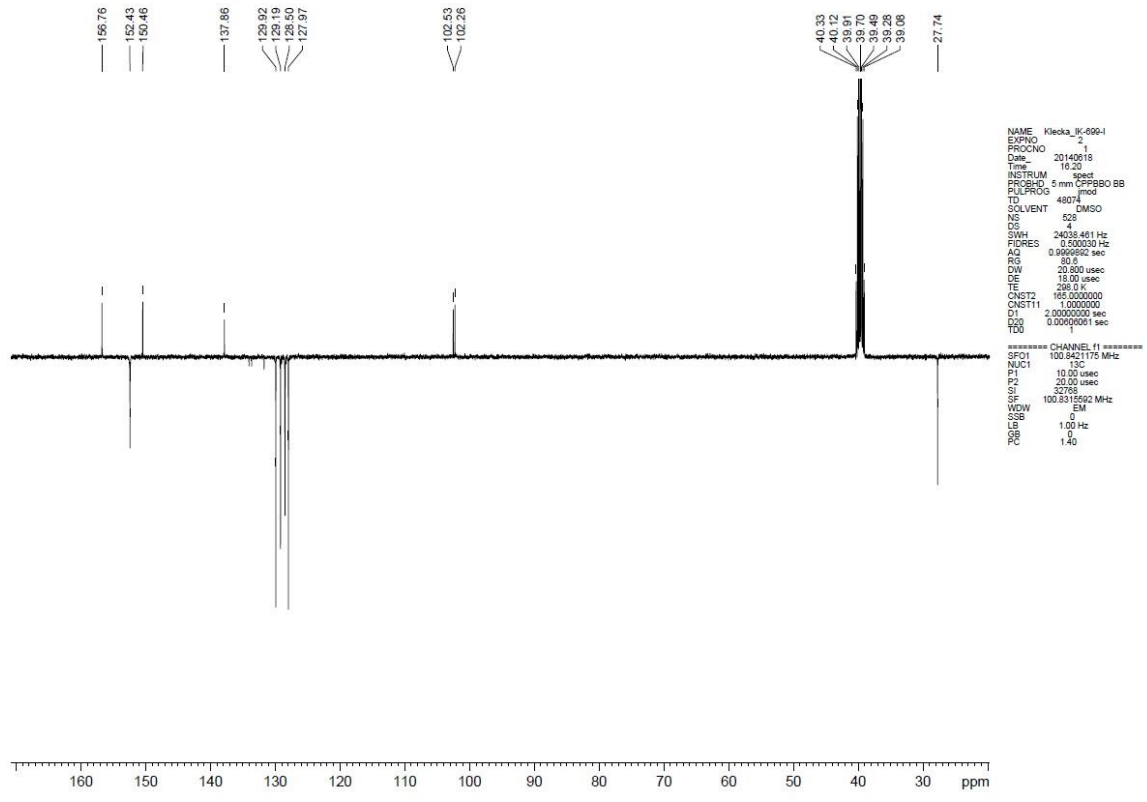

12.55
8.64
7.94
7.47
7.47
7.46
7.46
7.13
7.12
7.12
6.98
6.98
6.98
6.97
6.97
6.97
2.81
2.51
2.50
2.50
2.50
2.49
Klecka_IK700-I
1H NMR in DMSO
10-04-14 LS
NAME Klecka_IK700-I
EXPNO 1
PROCNO 1
Date_ 20140410
Time 17.46
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 70026
SOLVENT DMSO
NS 32
DS 0
SWH 7002.801 Hz
FIDRES 0.100003 Hz
AQ 4.9999776 sec
RG 322.5
DW 71.400 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 -4.00 dB
PL1W 21.45254898 W
SFO1 499.9534497 MHz
SI 131072
SF 499.9499992 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
CH3
S
S
4
4a
5
3
N
6
2
N
7a
N
7
H
1
2d
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 ppm
1.01
0.85
1.00
0.89
0.99
1.01
3.02

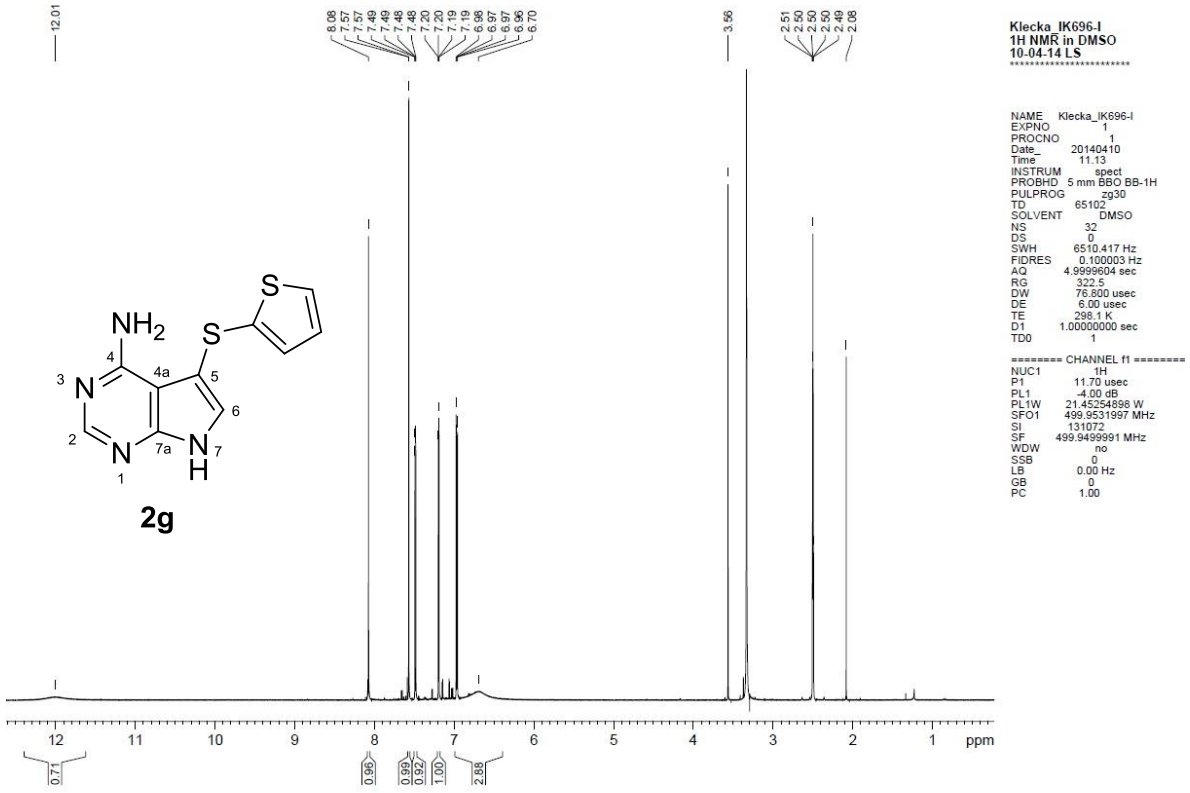

159.23
151.74
151.72
138.22
133.34
128.77
128.37
128.08
116.39
103.13
40.20
40.13
40.03
39.96
39.87
39.70
39.53
39.37
39.20
21.54
Klecka_IK700-I
APT in DMSO
10-04-14 LS
NAME Klecka_IK700-I
EXPNO 2
PROCNO 1
Date_ 20140410
Time 19.37
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.1 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.30 usec
P2 12.60 usec
PL1 -2.00 dB
PL1W 107.48893738 W
SFO1 125.7250987 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.70 dB
PL12 11.50 dB
PL2W 20.02068520 W
PL12W 0.60461497 W
SFO2 499.9519998 MHz
SI 131072
SF 125.7125647 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm


12.23
8.19
7.62
7.45
7.45
7.44
7.44
7.07
7.07
7.07
7.06
6.96
6.95
6.95
6.94
3.33
3.22
3.17
2.51
2.50
2.50
2.50
2.49
S
H3C
N
CH3
S
4
4a
5
3
N
6
2
N
7a
N
7
H
1
2e
ppm


159.48
153.12
150.82
138.47
130.17
129.14
126.47
127.95
103.91
103.09
41.45
40.33
40.12
39.91
39.70
39.49
39.28
39.08
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm


12.10
8.17
7.54
7.50
7.49
7.48
7.48
7.25
7.24
7.24
7.23
6.98
6.97
6.96
6.95
6.71
6.70
3.39
3.36
3.02
3.01
2.51
2.50
2.50
2.50
2.49
H3C
NH
S
S
N
N
N
H
1
2
3
4
4a
5
6
7
7a
2f
ppm

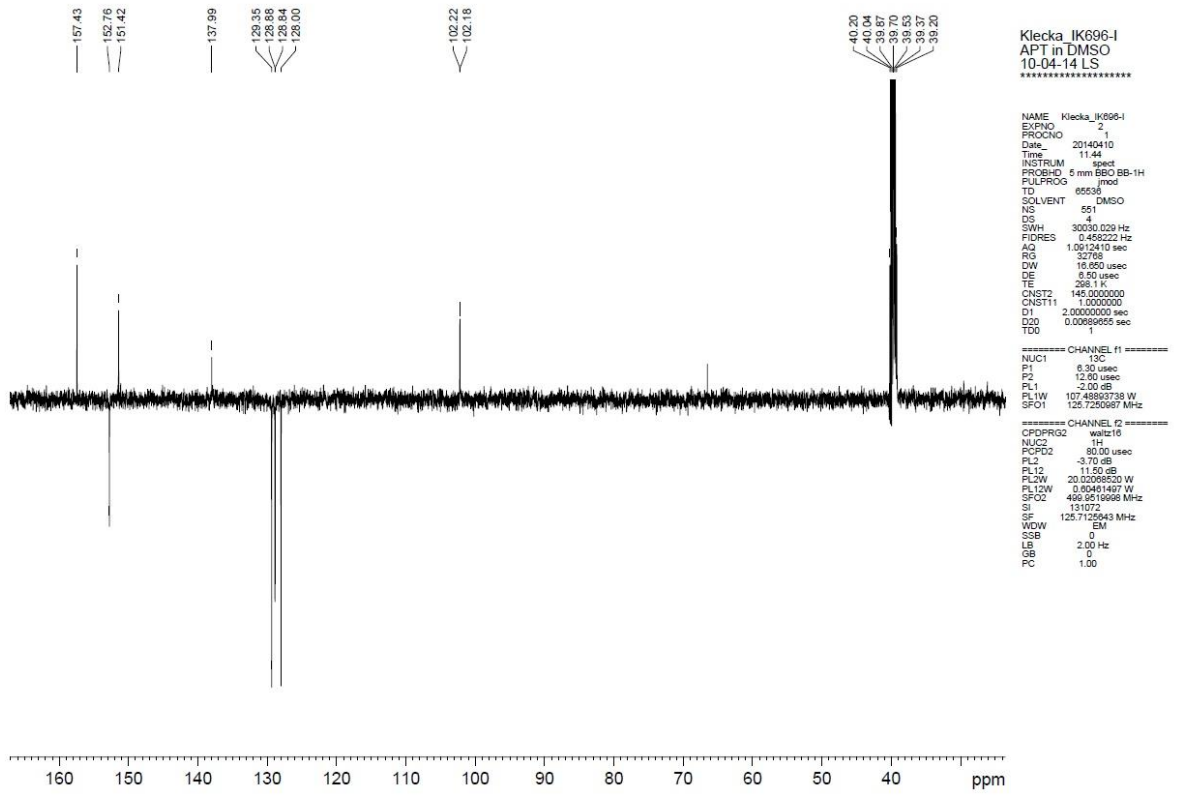

156.76
152.43
150.46
137.86
129.92
129.19
128.50
127.97
102.53
102.26
40.33
40.12
39.91
39.70
39.49
39.28
39.06
27.74
ppm



Klecka_IK696-I
1H NMR in DMSO
10-04-14 LS

```
NAME        Klecka_IK696-I
EXPNO               1
PROCNO              1
Date_           20140410
Time            11.13
INSTRUM         spect
PROBHD   5 mm BBO BB-1H
PULPROG         zg30
TD              65102
SOLVENT         DMSO
NS                32
DS                 0
SWH         6510.417 Hz
FIDRES      0.100003 Hz
AQ          4.9999604 sec
RG              322.5
DW              76.800 usec
DE               6.00 usec
TE              298.1 K
D1          1.00000000 sec
TD0                1

======== CHANNEL f1 ========
NUC1               1H
P1              11.70 usec
PL1             -4.00 dB
PL1W        21.45254898 W
SFO1        499.9531997 MHz
SI              131072
SF          499.9499991 MHz
WDW               no
SSB                0
LB              0.00 Hz
GB                 0
PC              1.00
```

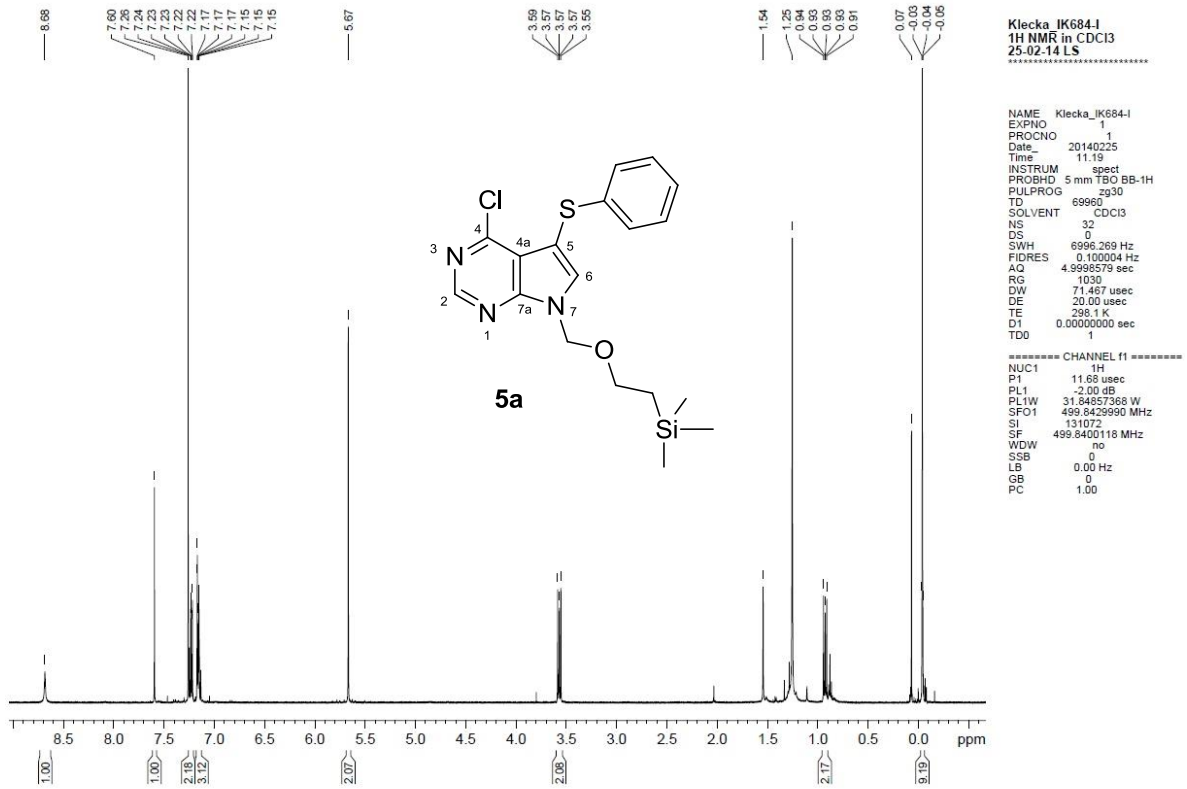


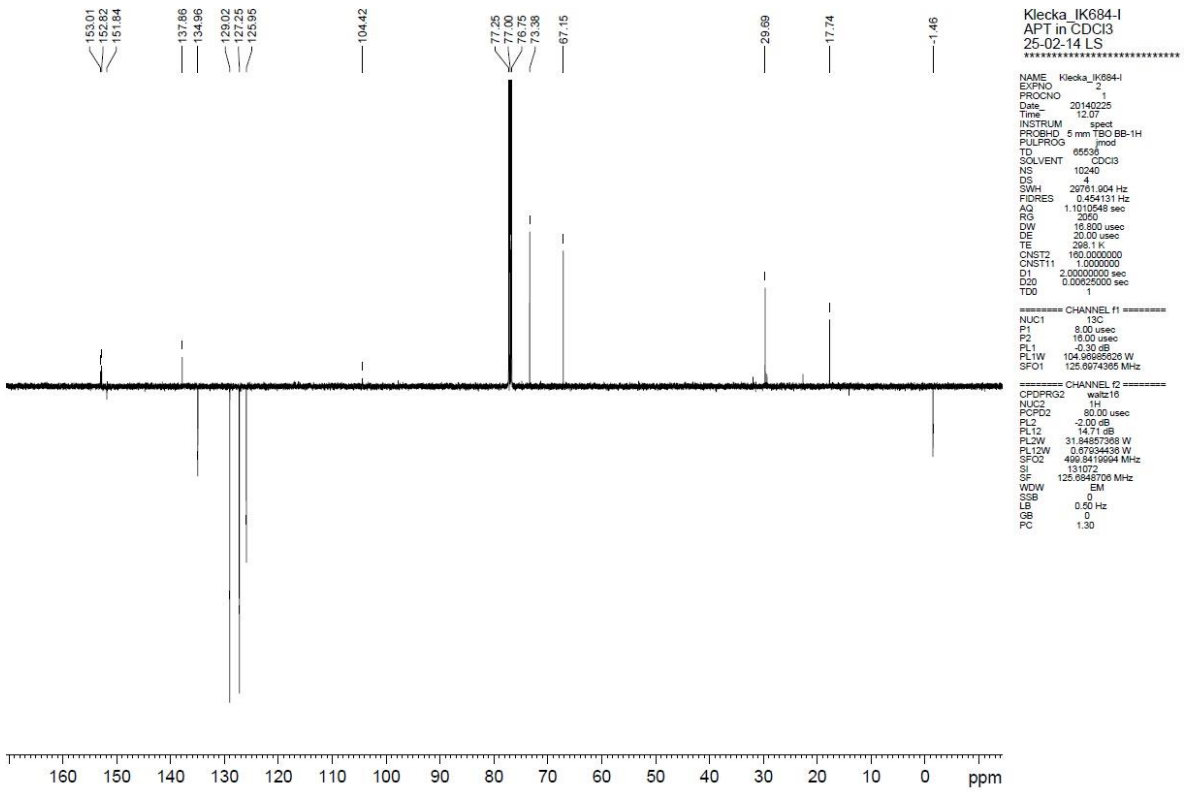
Klecka_IK696-I
APT in DMSO
10-04-14 LS

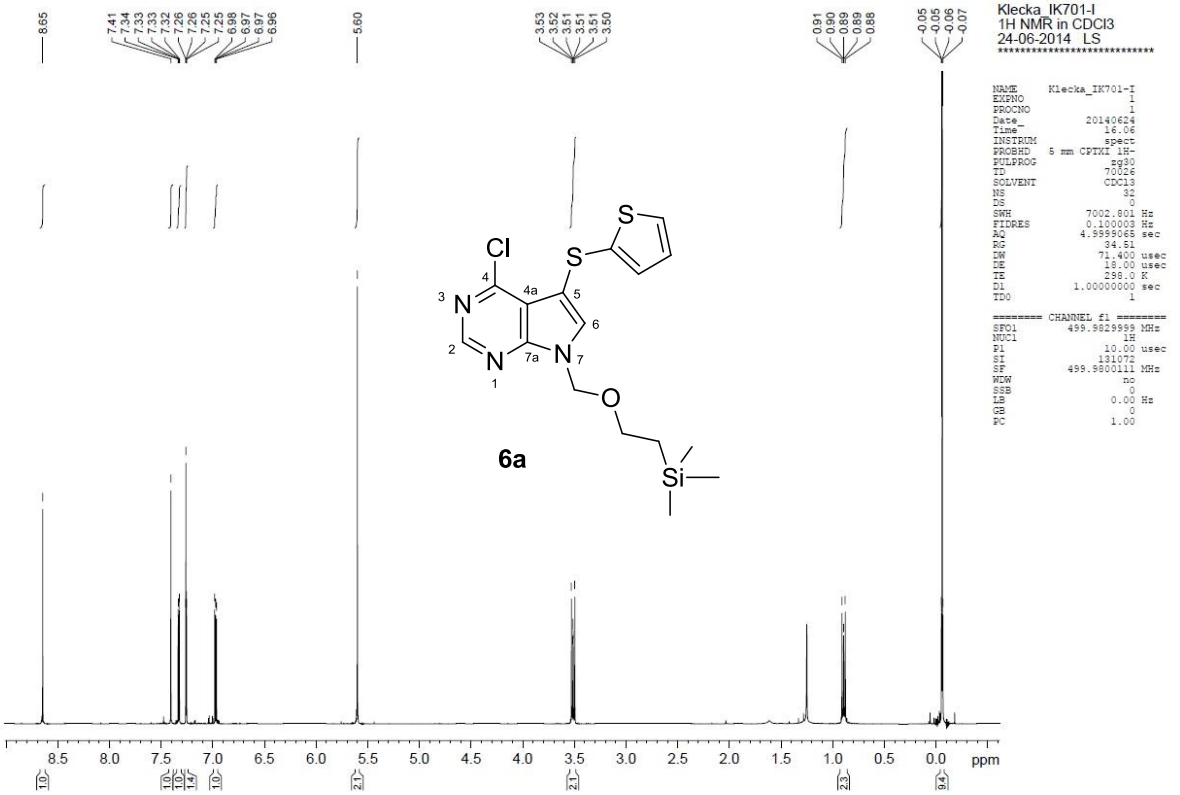
```
NAME        Klecka_IK696-I
EXPNO               2
PROCNO              1
Date_           20140410
Time            11.44
INSTRUM         spect
PROBHD   5 mm BBO BB-1H
PULPROG         jmod
TD              65536
SOLVENT         DMSO
NS                551
DS                 4
SWH         30030.029 Hz
FIDRES      0.458222 Hz
AQ          1.0912410 sec
RG              32768
DW              16.650 usec
DE               6.50 usec
TE              298.1 K
CNST2       145.0000000
CNST11        1.0000000
D1          2.00000000 sec
D20         0.00689655 sec
TD0                1

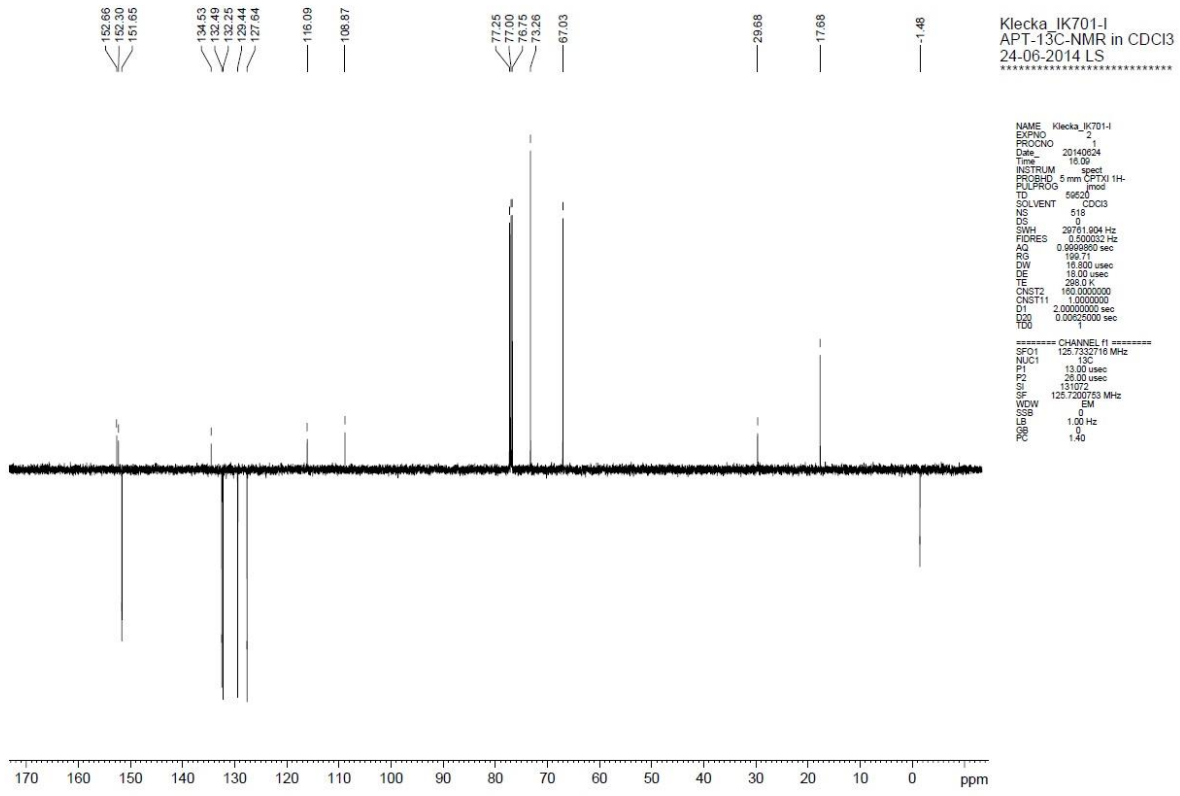
======== CHANNEL f1 ========
NUC1               13C
P1               6.30 usec
P2              12.60 usec
PL1             -2.00 dB
PL1W        107.48893738 W
SFO1        125.7250987 MHz

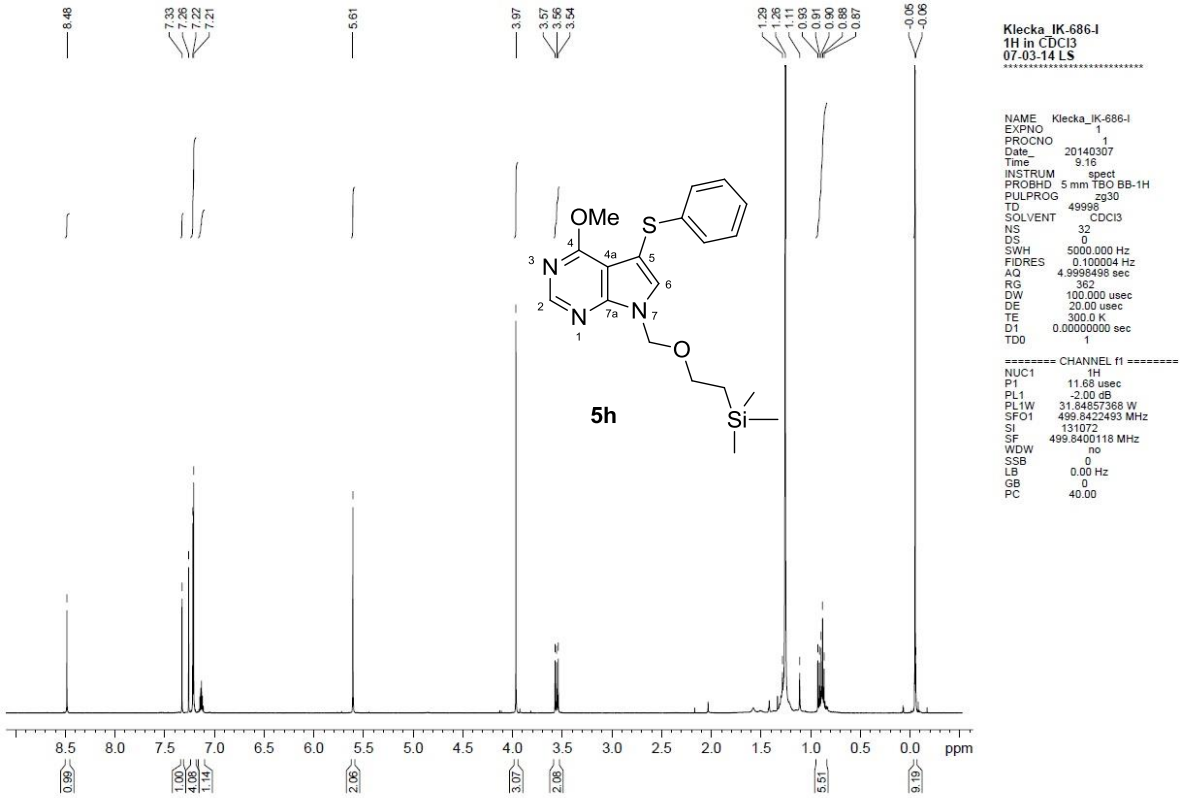
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2           80.00 usec
PL2             -3.70 dB
PL12            11.50 dB
PL2W        20.02066520 W
PL12W       0.60461497 W
SFO2        499.9519998 MHz
SI              131072
SF          125.7125643 MHz
WDW               EM
SSB                0
LB              2.00 Hz
GB                 0
PC              1.00
```


8.68
7.60
7.26
7.24
7.23
7.22
7.17
7.17
7.15
7.15
5.67
3.59
3.57
3.57
3.55
1.54
1.25
0.94
0.93
0.93
0.91
0.07
-0.03
-0.04
-0.05
Cl
S
N
N
N
O
Si
5a
Klecka_IK684-I
1H NMR in CDCl3
25-02-14 LS
NAME Klecka_IK684-I
EXPNO 1
PROCNO 1
Date_ 20140225
Time 11.19
INSTRUM spect
PROBHD 5 mm TBO BB-1H
PULPROG zg30
TD 69960
SOLVENT CDCl3
NS 32
DS 0
SWH 6996.269 Hz
FIDRES 0.100004 Hz
AQ 4.9998579 sec
RG 1030
DW 71.467 usec
DE 20.00 usec
TE 298.1 K
D1 0.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.68 usec
PL1 -2.00 dB
PL1W 31.84857368 W
SFO1 499.8429990 MHz
SI 131072
SF 499.8400118 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.00
2.18
3.12
2.07
2.08
2.17
9.19


153.01
152.82
151.84
137.86
134.96
129.02
127.25
125.95
104.42
77.25
77.00
76.75
73.38
67.15
29.69
17.74
1.46
Klecka_IK684-I
APT in CDCl3
25-02-14 LS
NAME Klecka_IK684-I
EXPNO 2
PROCNO 1
Date_ 20140225
Time 12.07
INSTRUM spect
PROBHD 5 mm TBO BB-1H
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 10240
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 20.00 usec
TE 298.1 K
CNST2 160.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00625000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
P2 16.00 usec
PL1 -0.30 dB
PL1W 104.96865626 W
SFO1 125.6974365 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.71 dB
PL2W 31.84857368 W
PL12W 0.67934436 W
SFO2 499.8419994 MHz
SI 131072
SF 125.6848706 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.30
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.65
7.41
7.34
7.33
7.33
7.32
7.26
7.26
7.25
7.25
6.98
6.97
6.97
6.96
5.60
3.53
3.52
3.51
3.51
3.51
3.50
0.91
0.90
0.89
0.89
0.88
-0.05
-0.05
-0.06
-0.07
Cl
S
S
N
N
N
O
Si
6a
Klecka_IK701-I
1H NMR in CDCl3
24-06-2014 LS
NAME Klecka_IK701-I
EXPNO 1
PROCNO 1
Date_ 20140624
Time 16.06
INSTRUM spect
PROBHD 5 mm CPTXI 1H-
PULPROG zg30
TD 70026
SOLVENT CDCl3
NS 32
DS 0
SWH 7002.801 Hz
FIDRES 0.100003 Hz
AQ 4.9999065 sec
RG 34.51
DW 71.400 usec
DE 18.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 499.9829999 MHz
NUC1 1H
P1 10.00 usec
SI 131072
SF 499.9800111 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.0
1.0
1.4
1.0
2.1
2.1
2.3
9.4

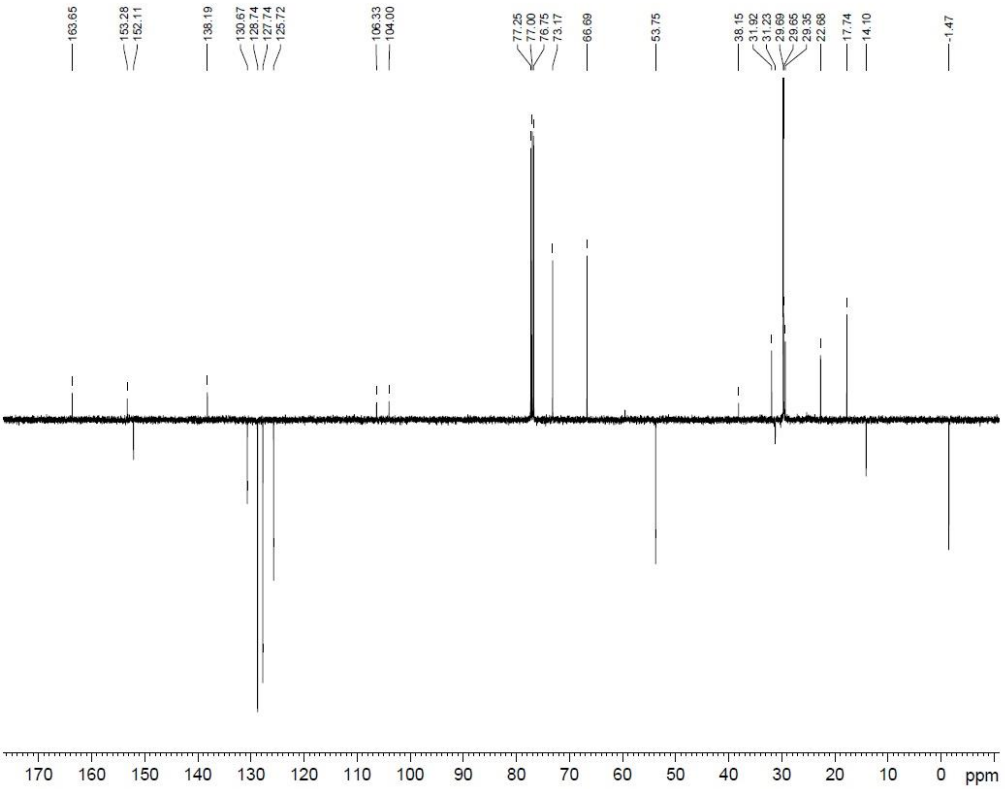

152.66
152.30
151.65
134.53
132.49
132.25
129.44
127.64
116.09
108.87
77.25
77.00
76.75
73.26
67.03
29.68
17.68
-1.48
Klecka_IK701-I
APT-13C-NMR in CDCl3
24-06-2014 LS
NAME Klecka_IK701-I
EXPNO 2
PROCNO 1
Date_ 20140624
Time 16.09
INSTRUM spect
PROBHD 5 mm CPTXI 1H-
PULPROG jmod
TD 59520
SOLVENT CDCl3
NS 518
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999860 sec
RG 199.71
DW 16.800 usec
DE 18.00 usec
TE 298.0 K
CNST2 160.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00625000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7332716 MHz
NUC1 13C
P1 13.00 usec
P2 26.00 usec
SI 131072
SF 125.7200753 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm




```
Klecka_IK-686-I
1H in CDCl3
07-03-14 LS
******************************

NAME    Klecka_IK-686-I
EXPNO          1
PROCNO         1
Date_      20140307
Time           9.16
INSTRUM        spect
PROBHD  5 mm TBO BB-1H
PULPROG        zg30
TD             49998
SOLVENT        CDCl3
NS             32
DS             0
SWH            5000.000 Hz
FIDRES         0.100004 Hz
AQ             4.9998498 sec
RG             362
DW             100.000 usec
DE             20.00 usec
TE             300.0 K
D1             0.00000000 sec
TD0            1

======== CHANNEL f1 ========
NUC1           1H
P1             11.68 usec
PL1            -2.00 dB
PL1W           31.84857368 W
SFO1           499.8422493 MHz
SI             131072
SF             499.8400118 MHz
WDW            no
SSB            0
LB             0.00 Hz
GB             0
PC             40.00
```

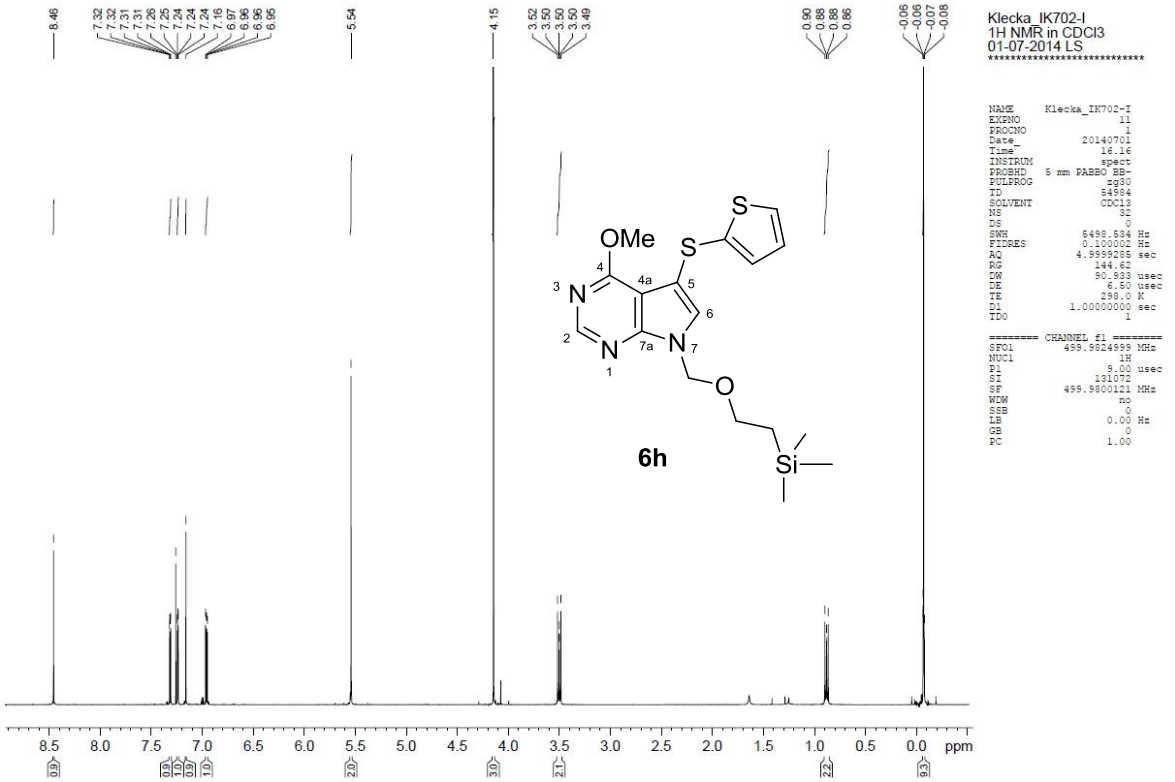



```
Klecka_IK-686-I
APT in CDCl3
07-03-14 LS
******************************

NAME    Klecka_IK-686-I
EXPNO          2
PROCNO         1
Date_      20140307
Time           10.23
INSTRUM        spect
PROBHD  5 mm TBO BB-1H
PULPROG        jmod
TD             65536
SOLVENT        CDCl3
NS             1276
DS             4
SWH            29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010548 sec
RG             2050
DW             16.800 usec
DE             6.00 usec
TE             300.0 K
CNST2          145.0000000
CNST11         1.0000000
D1             2.00000000 sec
D20            0.00689655 sec
TD0            1

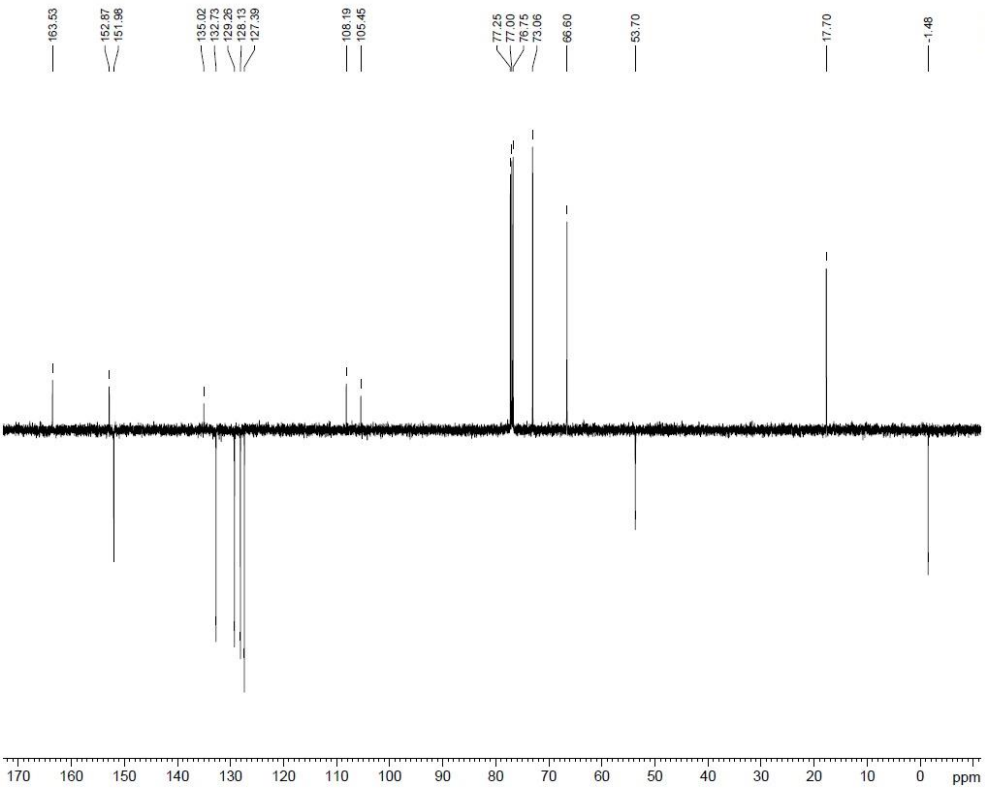
======== CHANNEL f1 ========
NUC1           13C
P1             8.00 usec
P2             16.00 usec
PL1            -0.30 dB
PL1W           104.96965626 W
SFO1           125.6974365 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2           1H
PCPD2          80.00 usec
PL2            -2.00 dB
PL12           14.71 dB
PL2W           31.84857368 W
PL12W          0.67934436 W
SFO2           499.8419994 MHz
SI             131072
SF             125.6848899 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             0.50
```



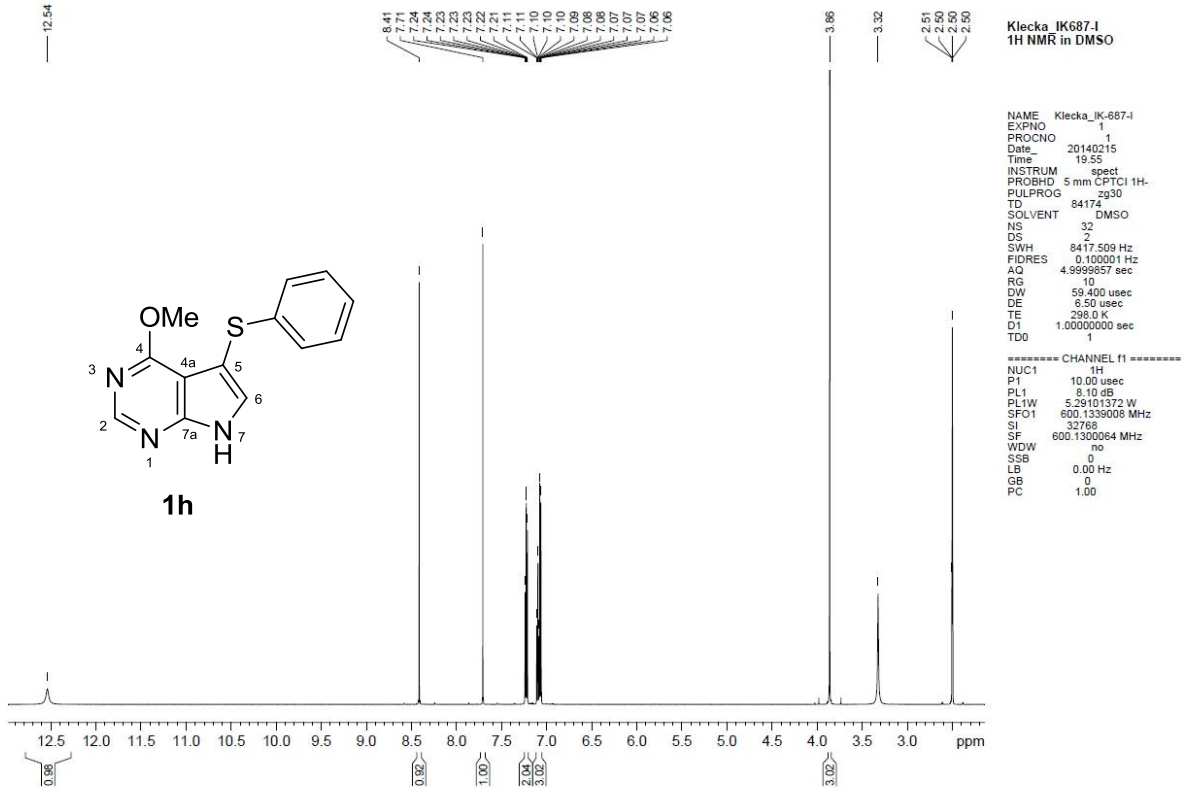
Klecka_IK702-I
1H NMR in CDCl3
01-07-2014 LS

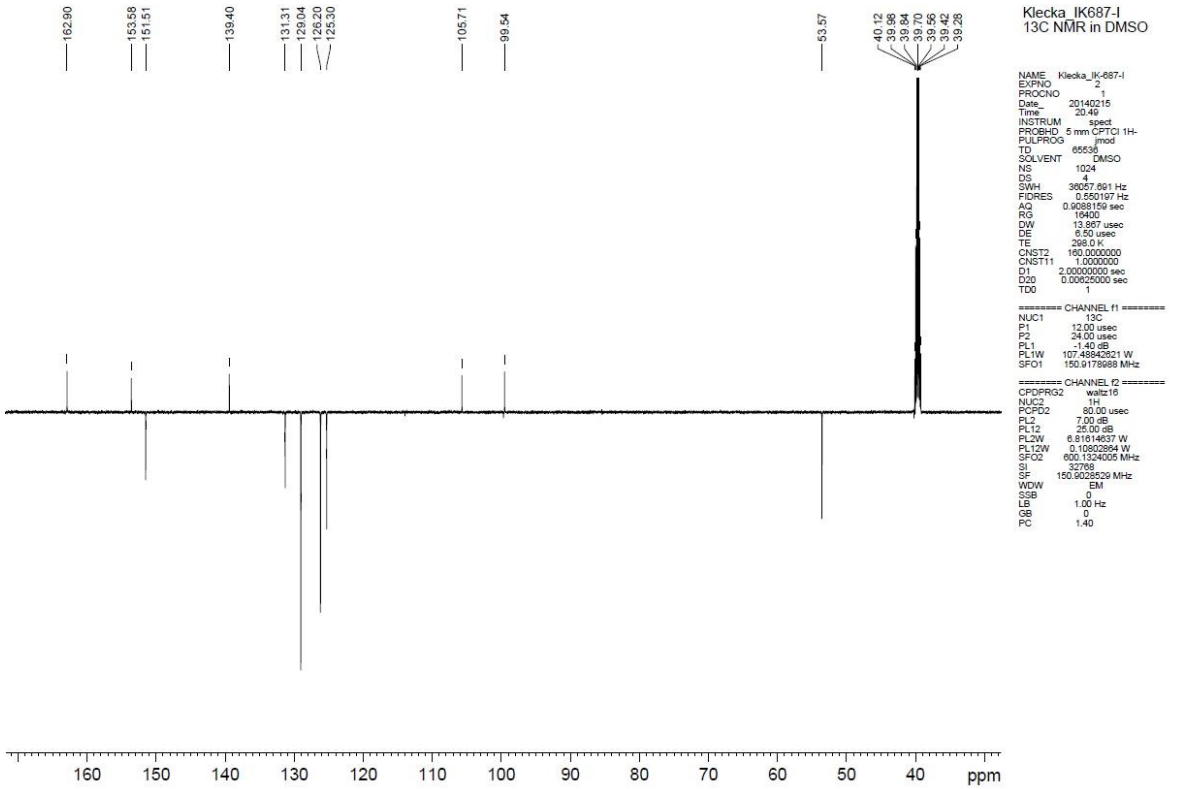
NAME	Klecka_IK702-I
EXPNO	11
PROCNO	1
Date_	20140701
Time	16.16
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	54984
SOLVENT	CDCl3
NS	32
DS	0
SWH	5498.534 Hz
FIDRES	0.100002 Hz
AQ	4.9999285 sec
RG	144.62
DW	90.933 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	499.9824999 MHz
NUC1	1H
P1	9.00 usec
SI	131072
SF	499.9800121 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

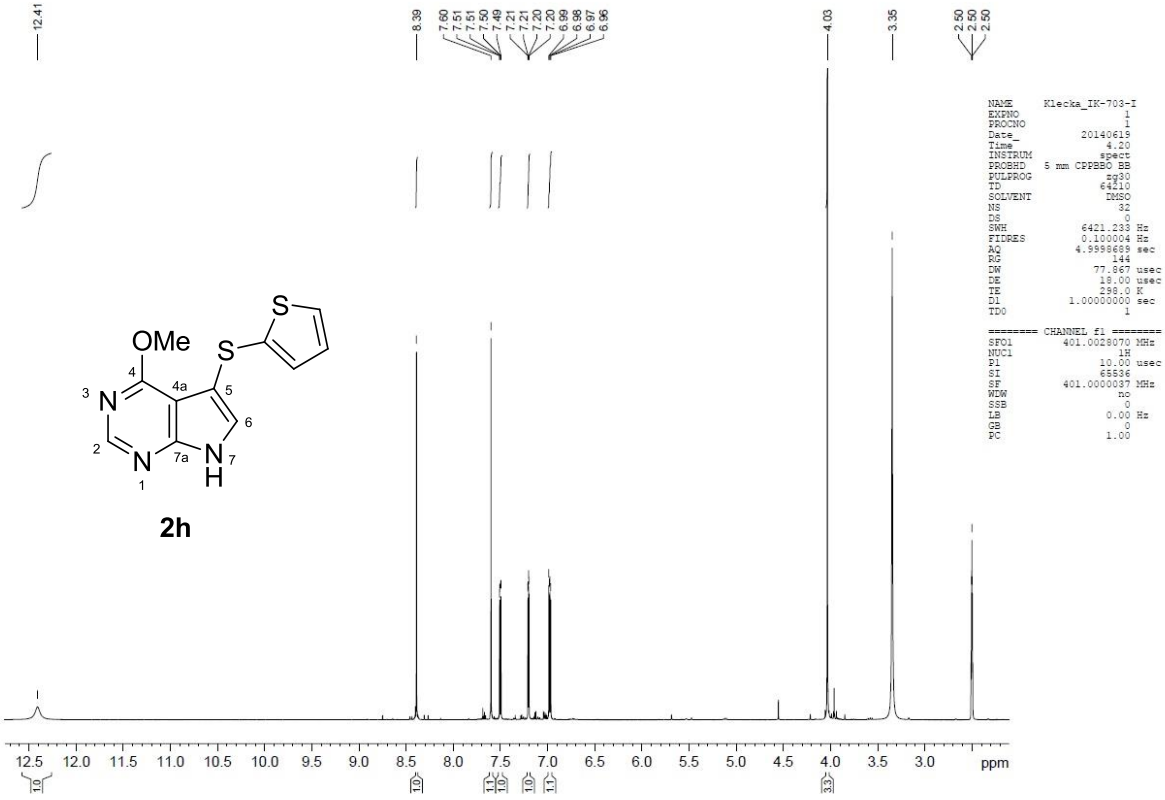


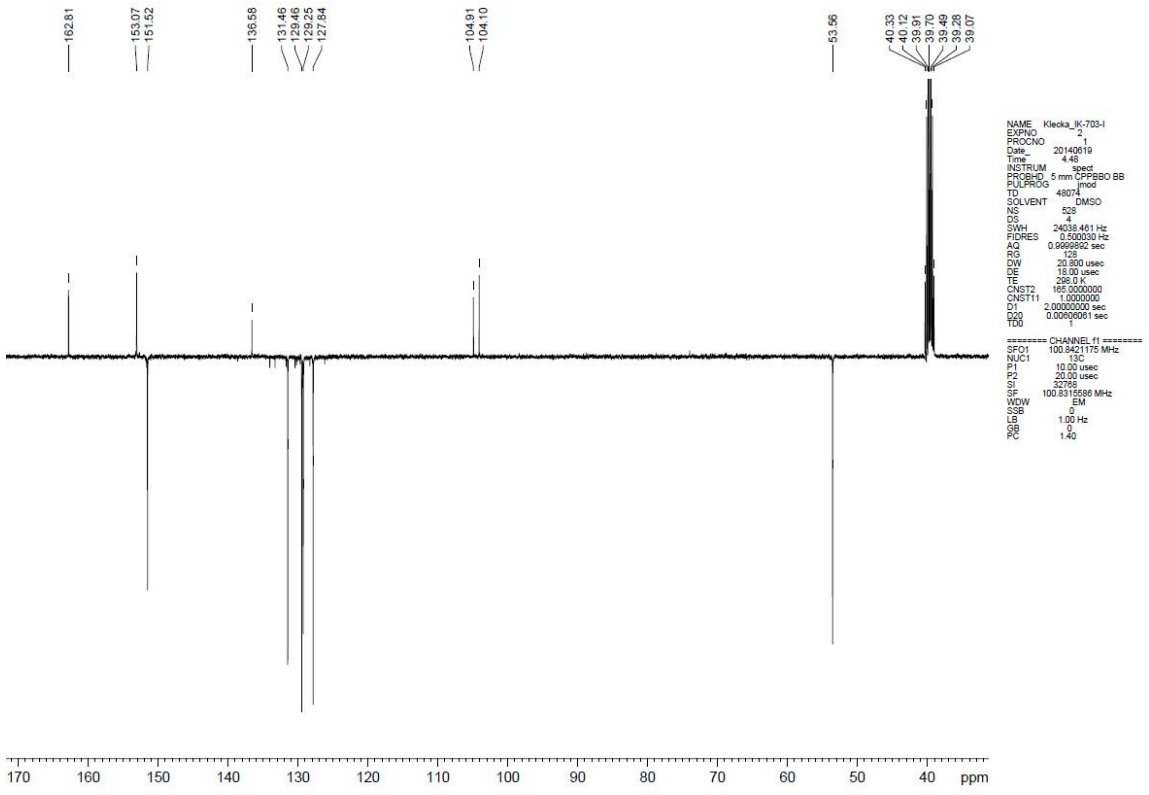
Klecka_IK702-I
APT-13C-NMR in CDCl3
01-07-2014 LS

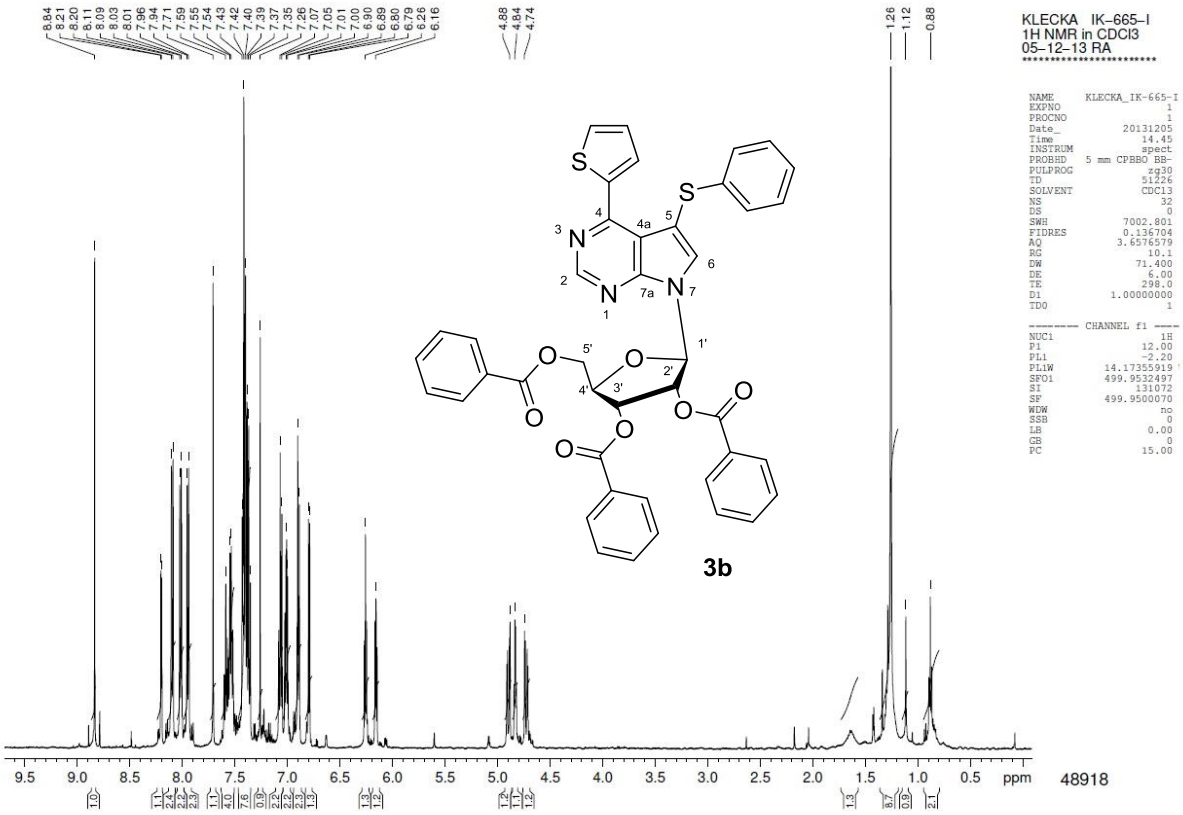
NAME	Klecka_IK702-I
EXPNO	22
PROCNO	1
Date_	20140701
Time	16.43
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	jmod
TD	59520
SOLVENT	CDCl3
NS	630
DS	2
SWH	29761.904 Hz
FIDRES	0.500032 Hz
AQ	0.9999990 sec
RG	199.71
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
CNST2	160.0000000
CNST11	1.0000000
D1	2.00000000 sec
D20	0.00625000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	125.7332716 MHz
NUC1	13C
P1	7.60 usec
P2	15.20 usec
SI	131072
SF	125.7200729 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

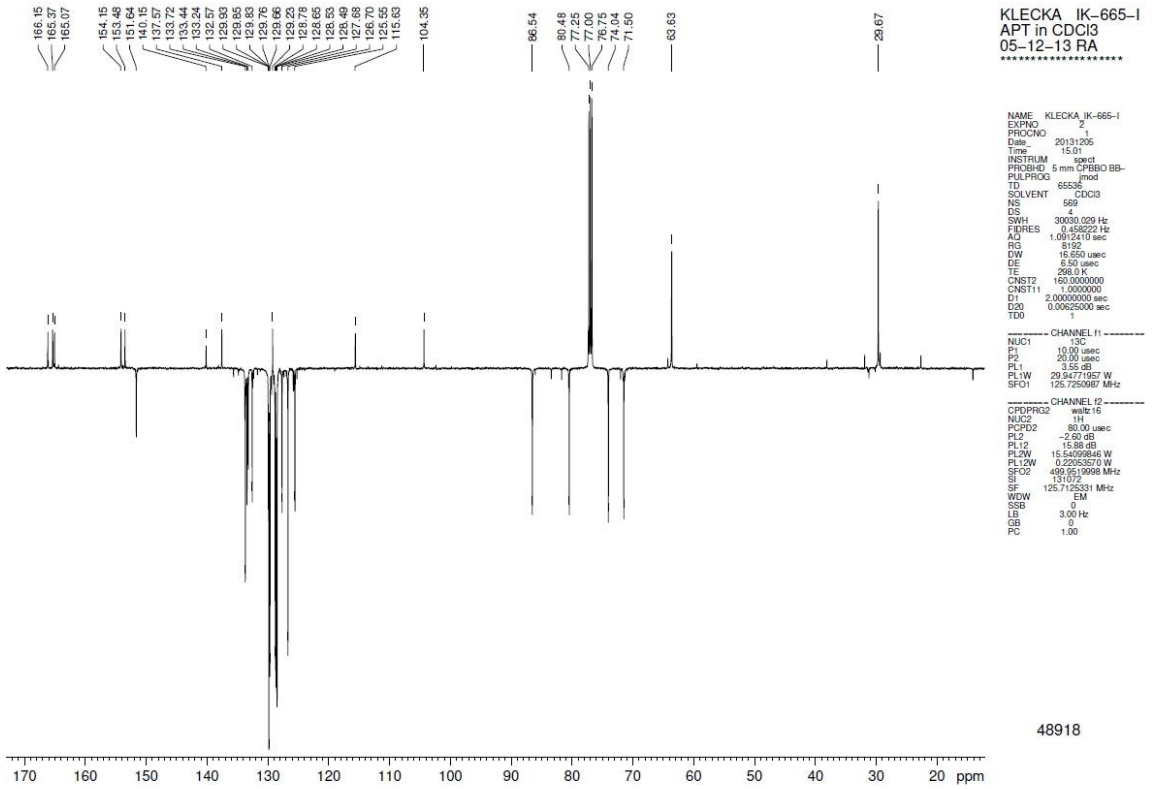

12.54
8.41
7.71
7.24
7.24
7.23
7.23
7.23
7.22
7.21
7.11
7.11
7.10
7.10
7.09
7.08
7.08
7.07
7.07
7.07
7.06
7.05
3.86
3.32
2.51
2.50
2.50
2.50
Klecka_IK687-I
1H NMR in DMSO
NAME Klecka_IK-687-I
EXPNO 1
PROCNO 1
Date_ 20140215
Time 19.55
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 84174
SOLVENT DMSO
NS 32
DS 2
SWH 8417.509 Hz
FIDRES 0.100001 Hz
AQ 4.9999857 sec
RG 10
DW 59.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 8.10 dB
PL1W 5.29101372 W
SFO1 600.1339008 MHz
SI 32768
SF 600.1300064 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
OMe
S
4
4a
5
3
N
6
2
N
7a
N
7
H
1
1h
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 ppm
0.98
0.92
1.00
2.04
3.02
3.02

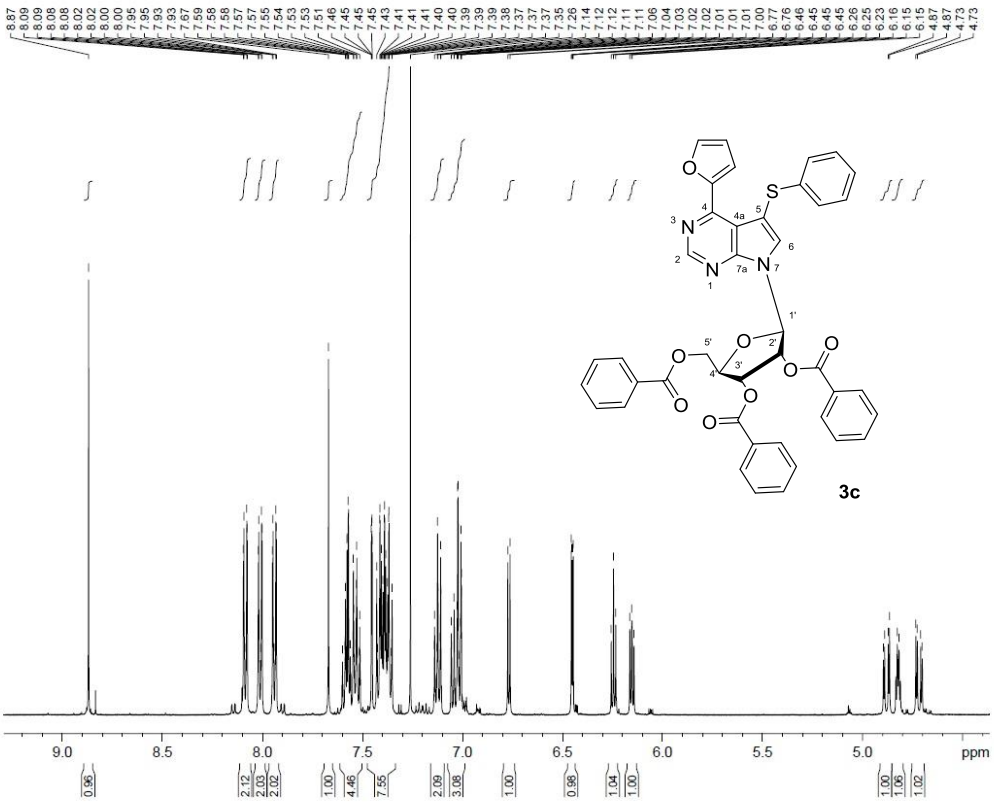

162.90
153.58
151.51
139.40
131.31
129.04
126.20
125.30
105.71
99.54
53.57
40.12
39.98
39.84
39.70
39.56
39.42
39.28
Klecka_IK687-I
13C NMR in DMSO
NAME Klecka_IK-687-I
EXPNO 2
PROCNO 1
Date_ 20140215
Time 20.49
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 38067.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 19400
DW 13.867 usec
DE 6.50 usec
TE 298.0 K
CNST2 160.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00625000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
P2 24.00 usec
PL1 -1.40 dB
PL1W 107.46842621 W
SFO1 150.9178988 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 7.00 dB
PL12 25.00 dB
PL2W 6.81614637 W
PL12W 0.10802864 W
SFO2 600.1324005 MHz
SI 32768
SF 150.9028529 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 ppm


12.41
8.39
7.60
7.51
7.51
7.50
7.49
7.21
7.21
7.20
7.20
6.99
6.98
6.97
6.96
4.03
3.35
2.50
2.50
2.50
OMe
S
S
N
N
H
2h
ppm


162.81
153.07
151.52
136.58
131.46
129.46
129.25
127.84
104.91
104.10
53.56
40.33
40.12
39.91
39.70
39.49
39.28
39.07
ppm


KLECKA IK–665–I
1H NMR in CDCl3
05–12–13 RA
3b
48918
ppm

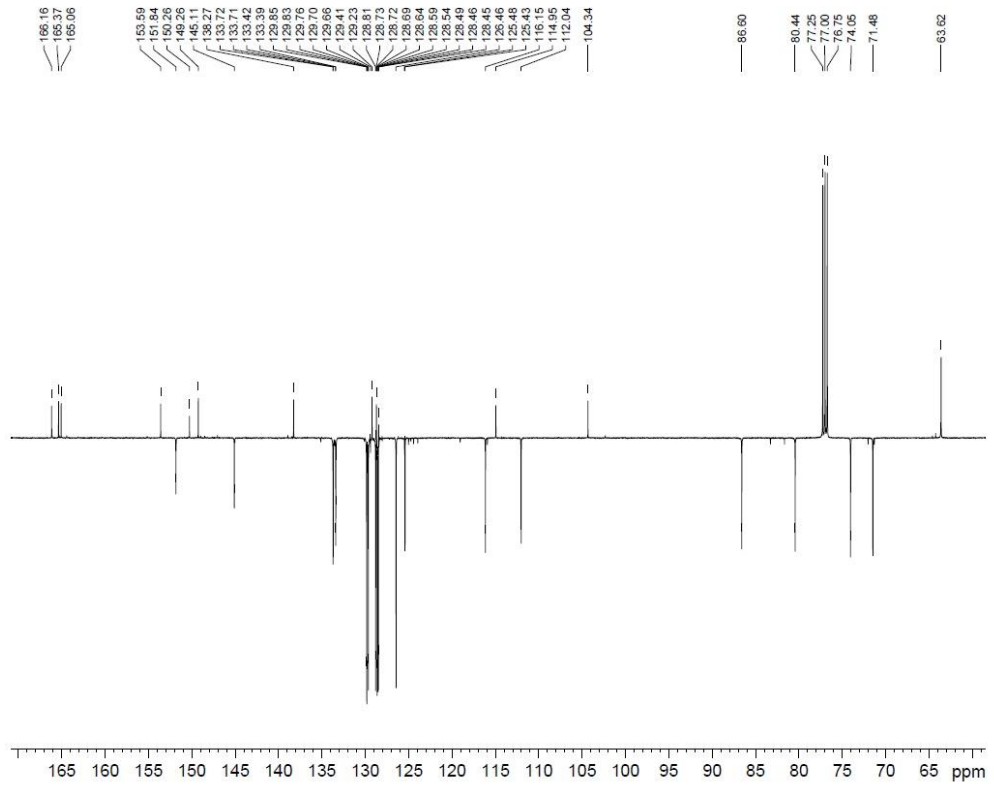

KLECKA IK–665–I
APT in CDCl3
05–12–13 RA
48918
ppm



KLECKA IK-664-I
1H NMR in CDCl3
13-12-13 RA

NAME KLECKA_IK-664-Inew
EXPNO 1
PROCNO 1
Date_ 20131213
Time 8.19
INSTRUM spect
PROBHD 5 mm CPBBO BB-
PULPROG zg30
TD 51226
SOLVENT CDCl3
NS 32
DS 0
SWH 5122.951 Hz
FIDRES 0.100007 Hz
AQ 4.9998050 sec
RG 10.1
DW 97.600 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.20 dB
PL1W 14.17355919 W
SFO1 499.9524608 MHz
SI 131072
SF 499.9500078 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 3.00

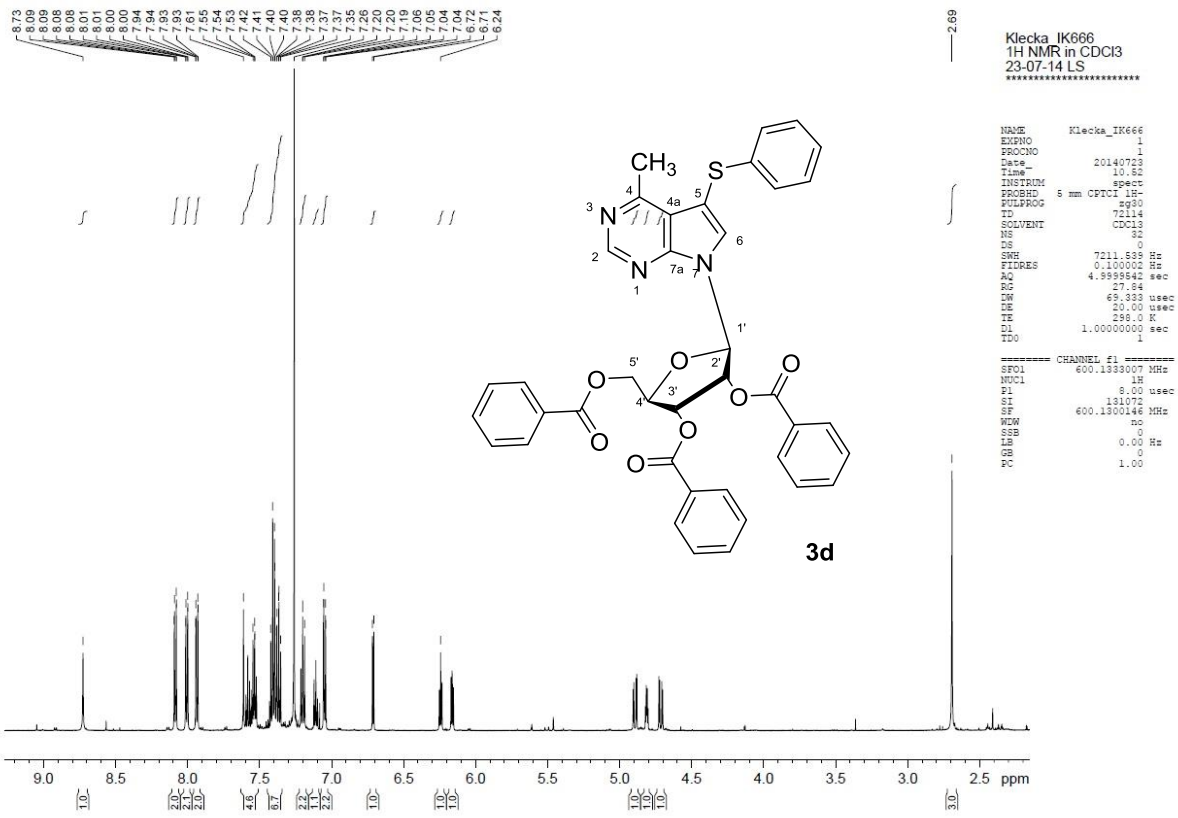


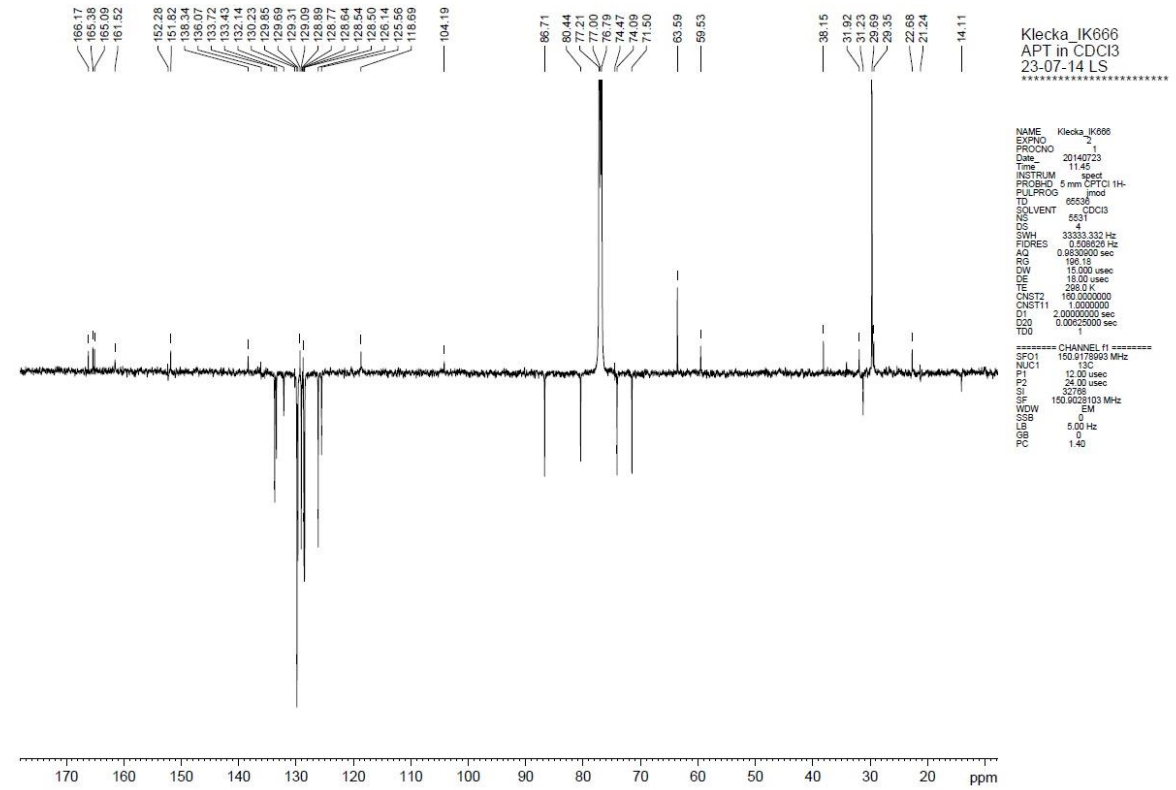
KLECKA IK-664-I
APT in CDCl3
13-12-13 RA

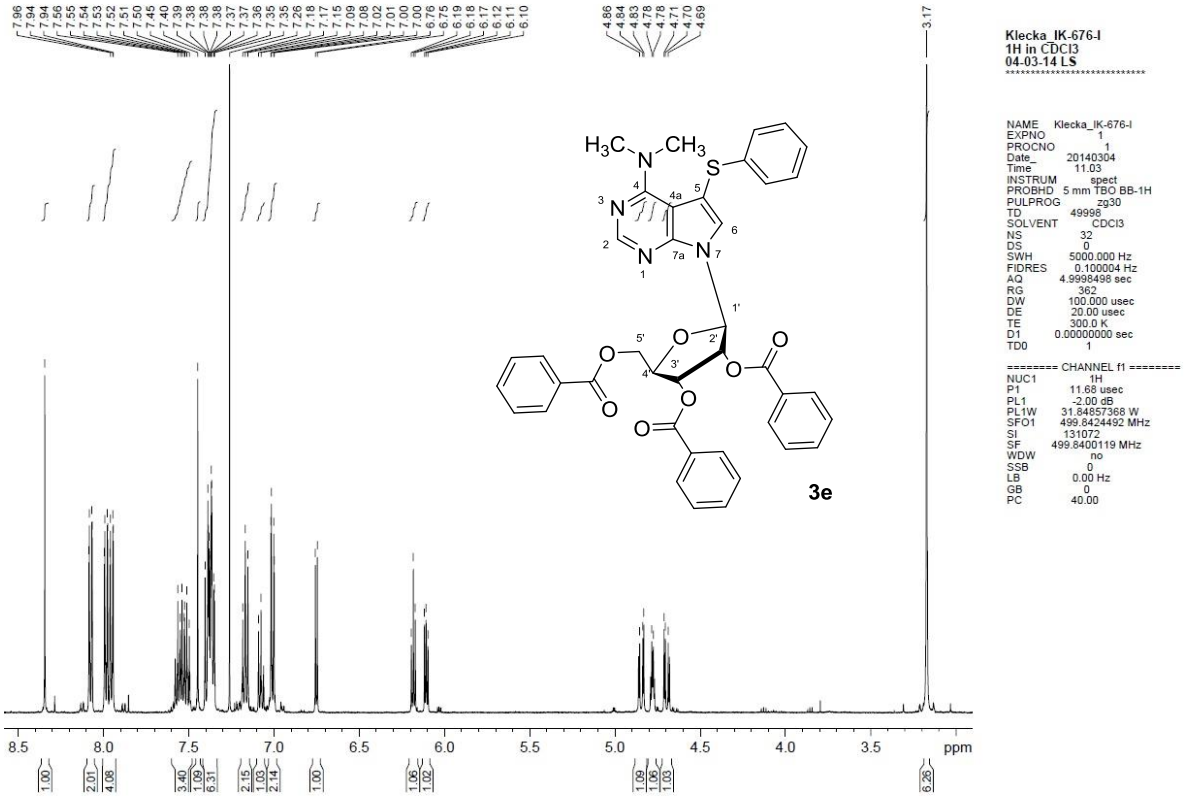
NAME KLECKA_IK-664-Inew
EXPNO 2
PROCNO 1
Date_ 20131213
Time 8.28
INSTRUM spect
PROBHD 5 mm CPBBO BB-
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 2001
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 8192
DW 16.650 usec
DE 6.50 usec
TE 298.0 K
CNST2 160.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00625000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
P2 20.00 usec
PL1 3.55 dB
PL1W 29.94771957 W
SFO1 125.7250967 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.60 dB
PL12 15.88 dB
PL2W 15.54009848 W
PL12W 0.22053570 W
SFO2 499.9519998 MHz
SI 131072
SF 125.7125318 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00


Klecka_IK666
1H NMR in CDCl3
23-07-14 LS
3d
ppm

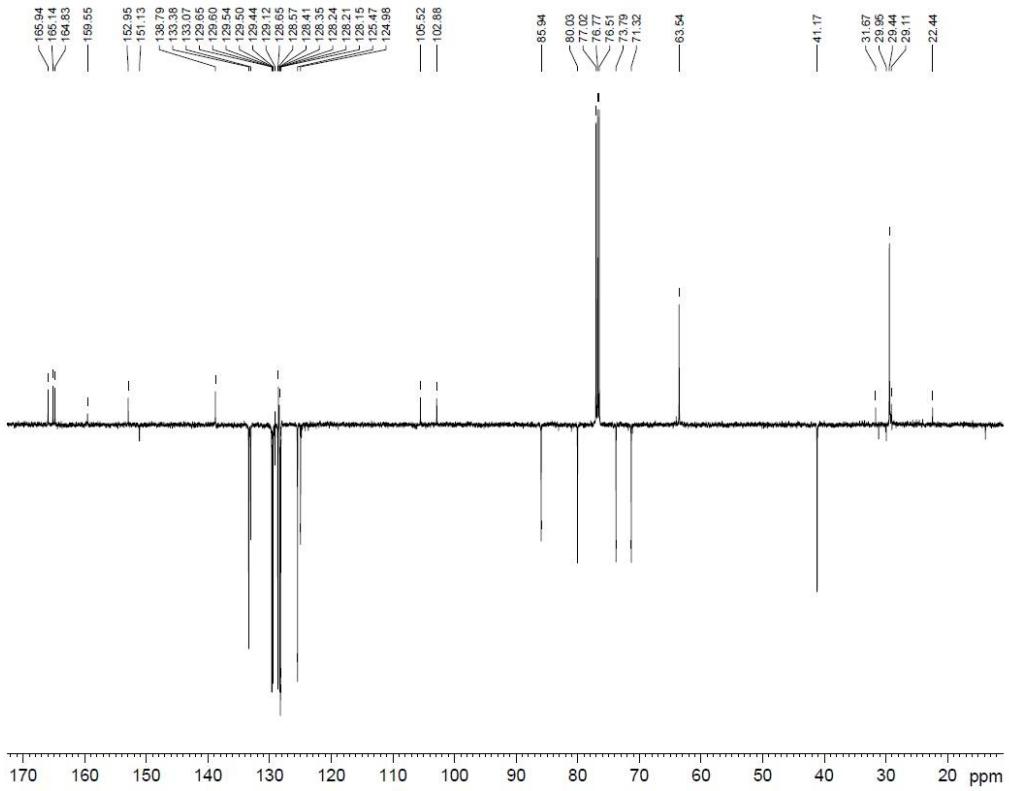

Klecka_IK666
APT in CDCl3
23-07-14 LS
ppm



Klecka_IK-676-I
1H in CDCl3
04-03-14 LS
****

NAME Klecka_IK-676-I
EXPNO 1
PROCNO 1
Date_ 20140304
Time 11.03
INSTRUM spect
PROBHD 5 mm TBO BB-1H
PULPROG zg30
TD 49998
SOLVENT CDCl3
NS 32
DS 0
SWH 5000.000 Hz
FIDRES 0.100004 Hz
AQ 4.9998498 sec
RG 362
DW 100.000 usec
DE 20.00 usec
TE 300.0 K
D1 0.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.68 usec
PL1 -2.00 dB
PL1W 31.84857368 W
SFO1 499.8424492 MHz
SI 131072
SF 499.8400119 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 40.00

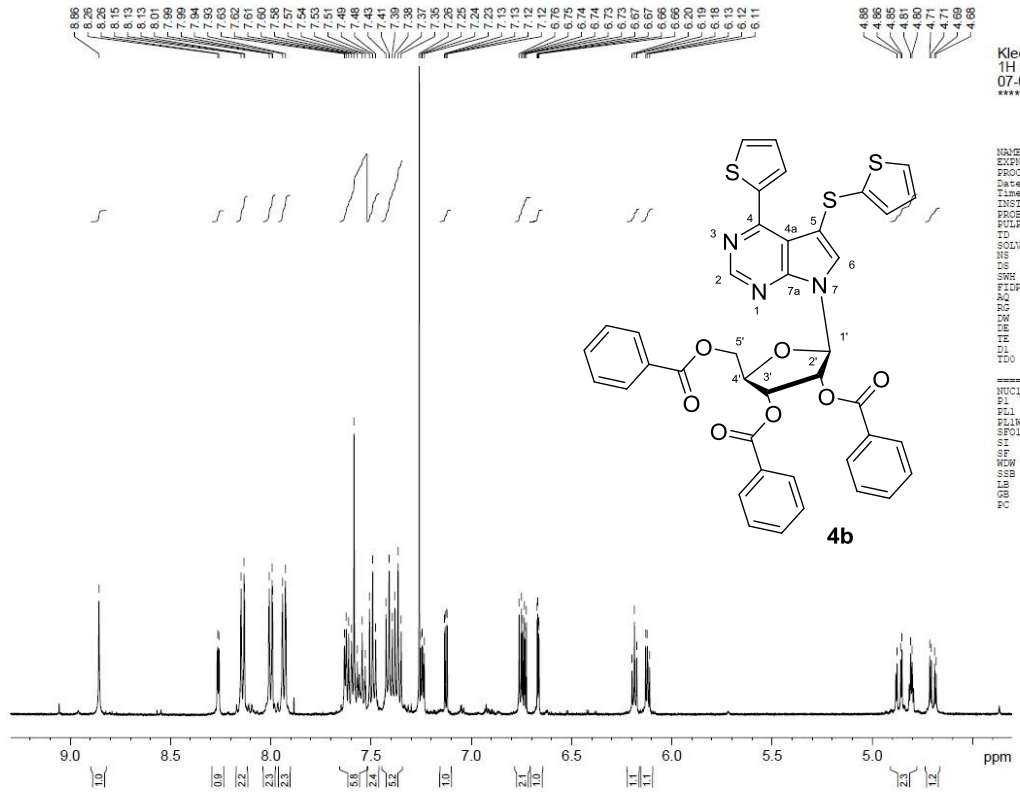


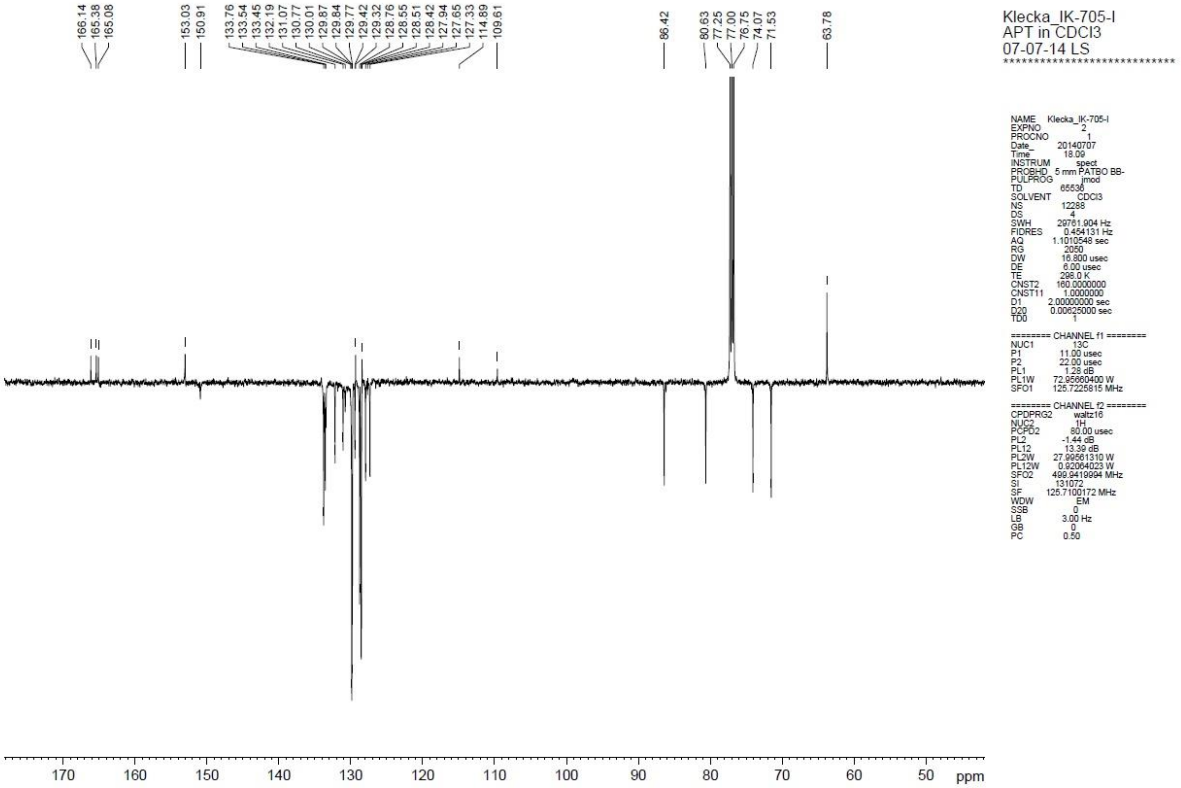
Klecka_IK-676-I
APT in CDCl3
04-03-14 LS
****

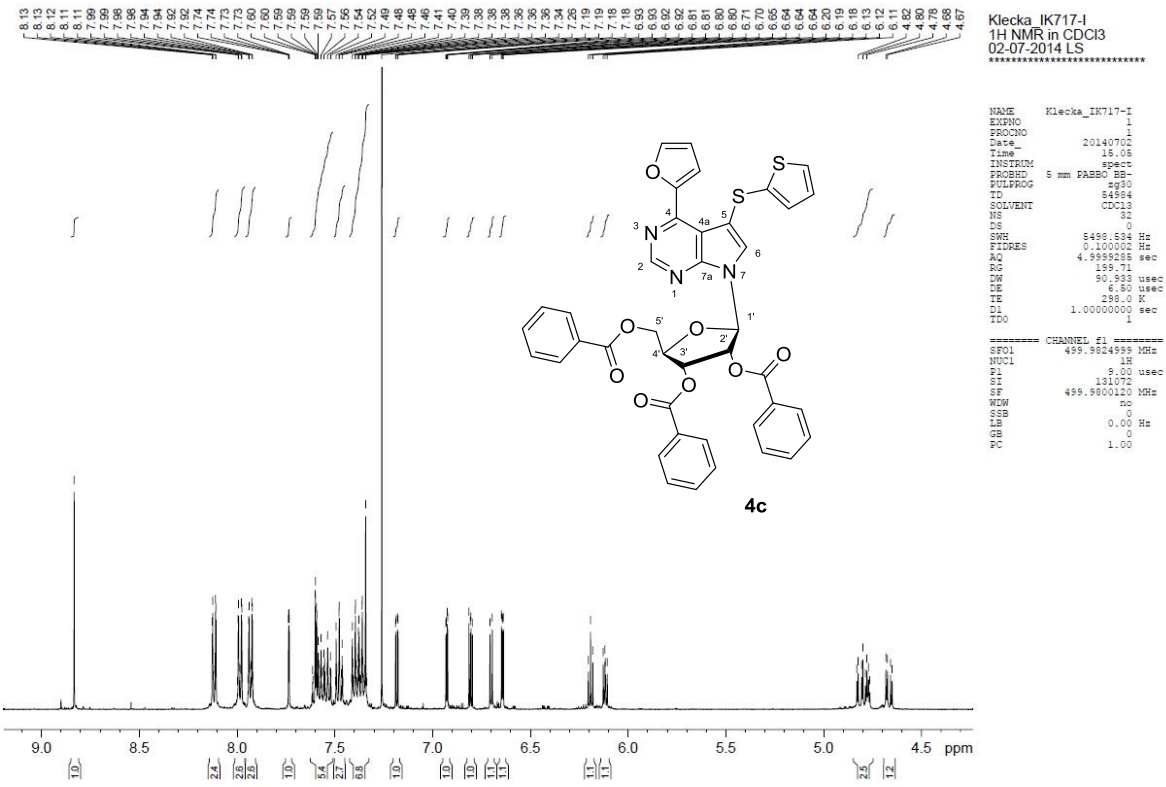
NAME Klecka_IK-676-I
EXPNO 2
PROCNO 1
Date_ 20140304
Time 11.23
INSTRUM spect
PROBHD 5 mm TBO BB-1H
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 2302
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00689655 sec
TD0 1

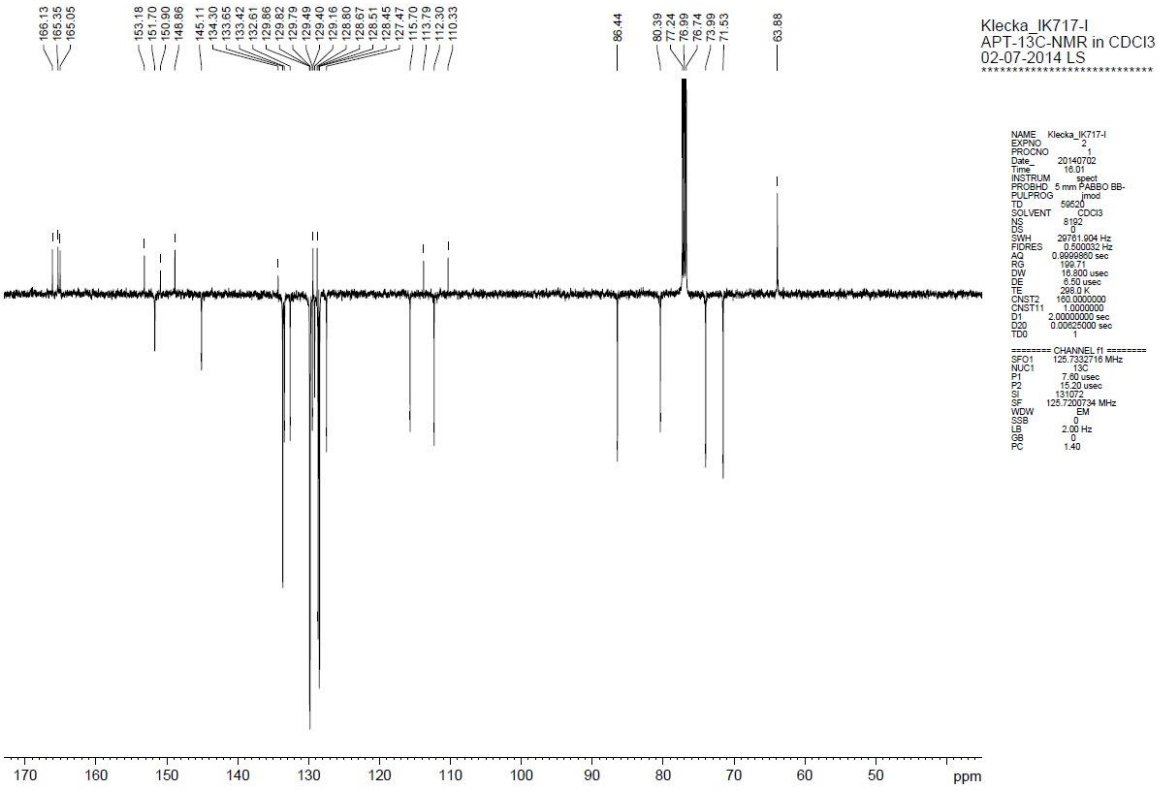
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
P2 16.00 usec
PL1 -0.30 dB
PL1W 104.96985626 W
SFO1 125.6974365 MHz

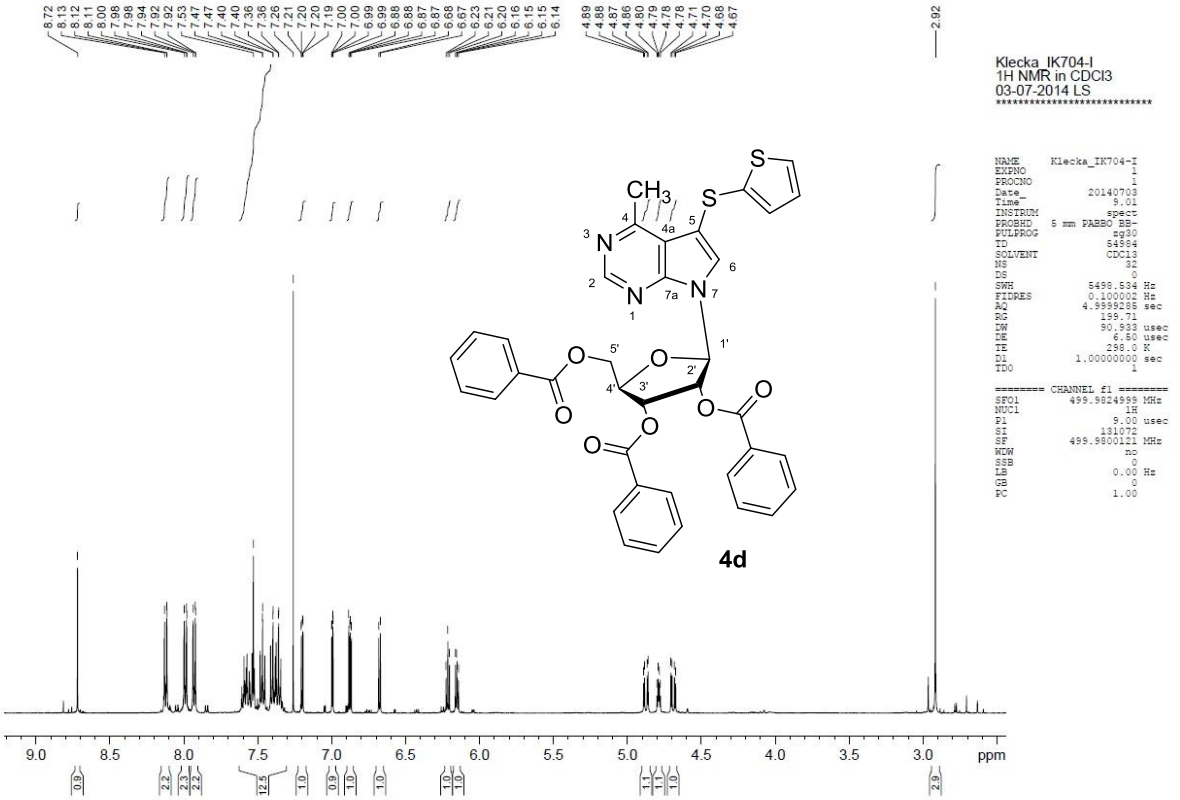
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.71 dB
PL2W 31.84857368 W
PL12W 0.67934436 W
SFO2 499.8419994 MHz
SI 131072
SF 125.6849011 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 0.50

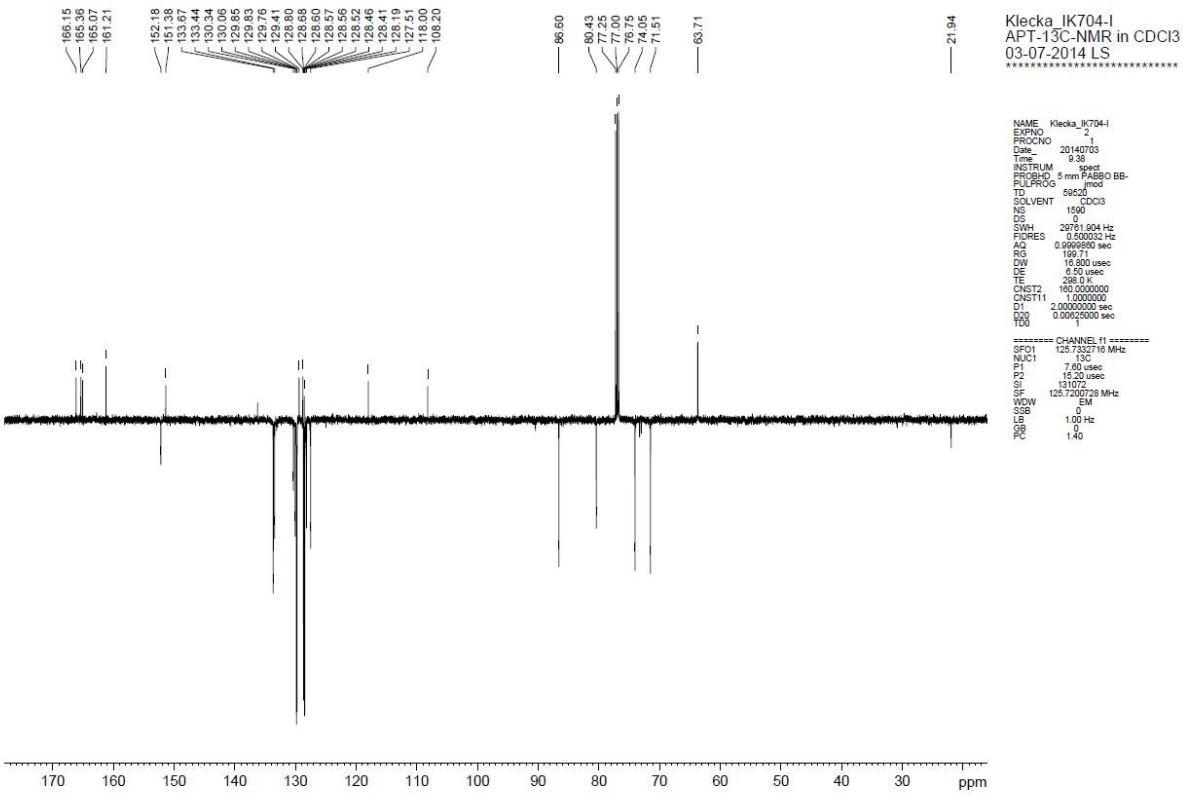

Klecka_IK-705-I
1H in CDCl3
07-07-14 LS
4b
ppm

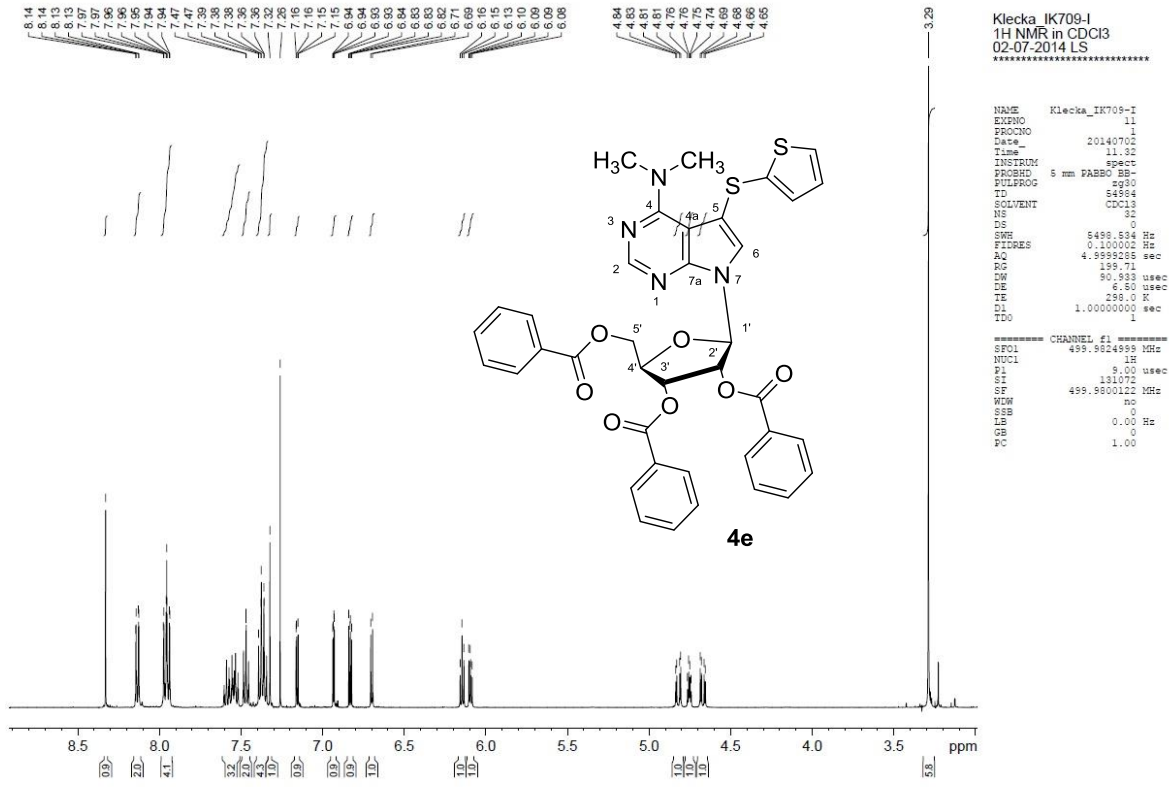

Klecka_IK-705-I
APT in CDCl3
07-07-14 LS
ppm

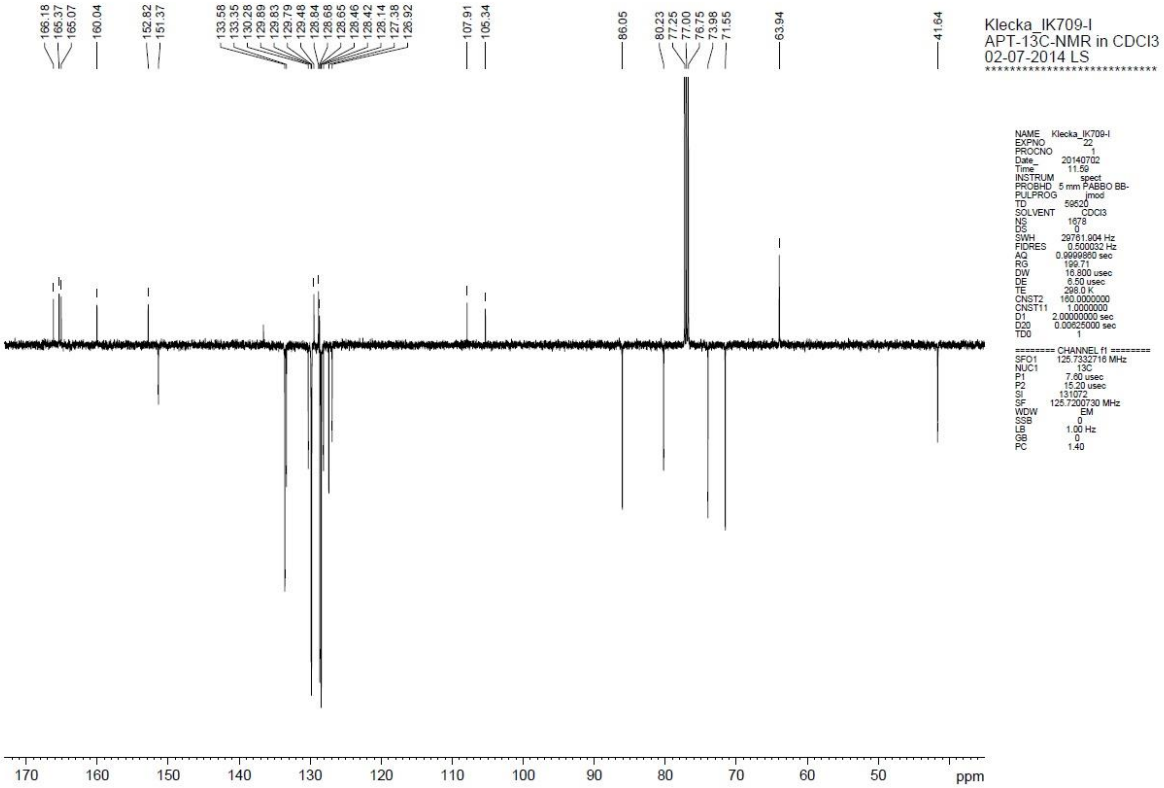

Klecka_IK717-I
1H NMR in CDCl3
02-07-2014 LS
4c
ppm

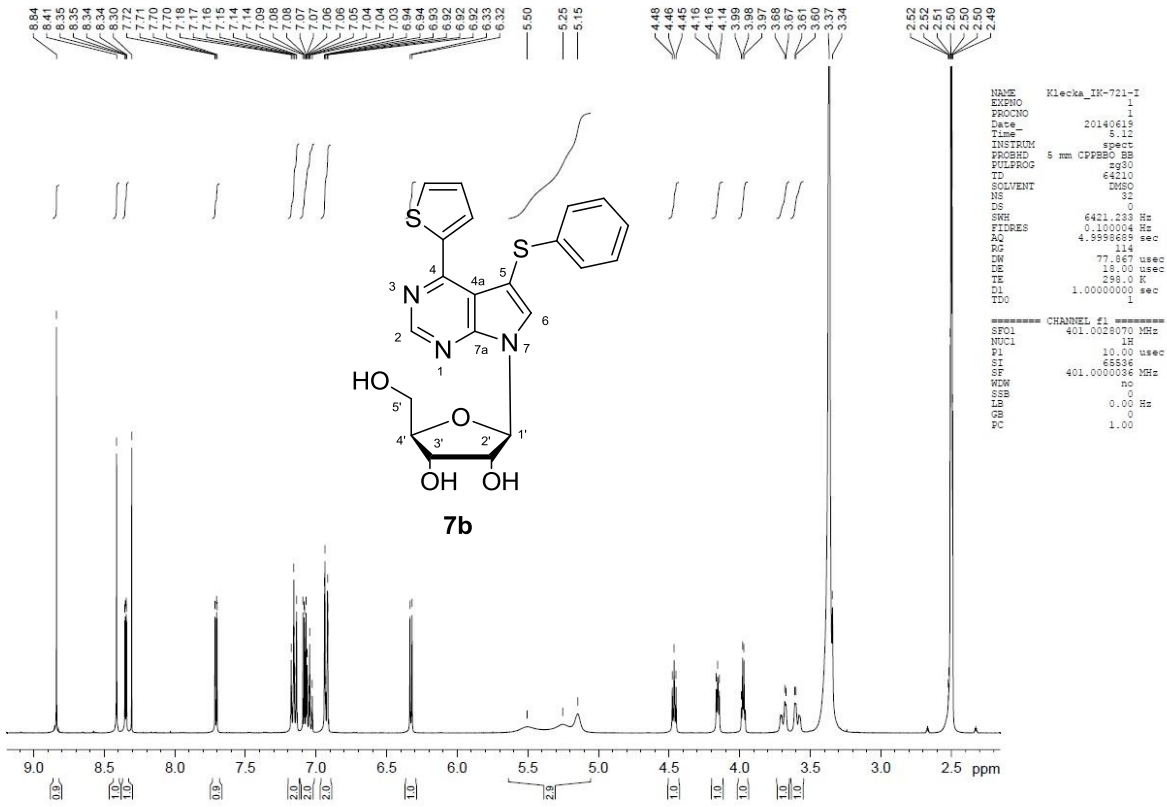

Klecka_IK717-I
APT-13C-NMR in CDCl3
02-07-2014 LS
ppm

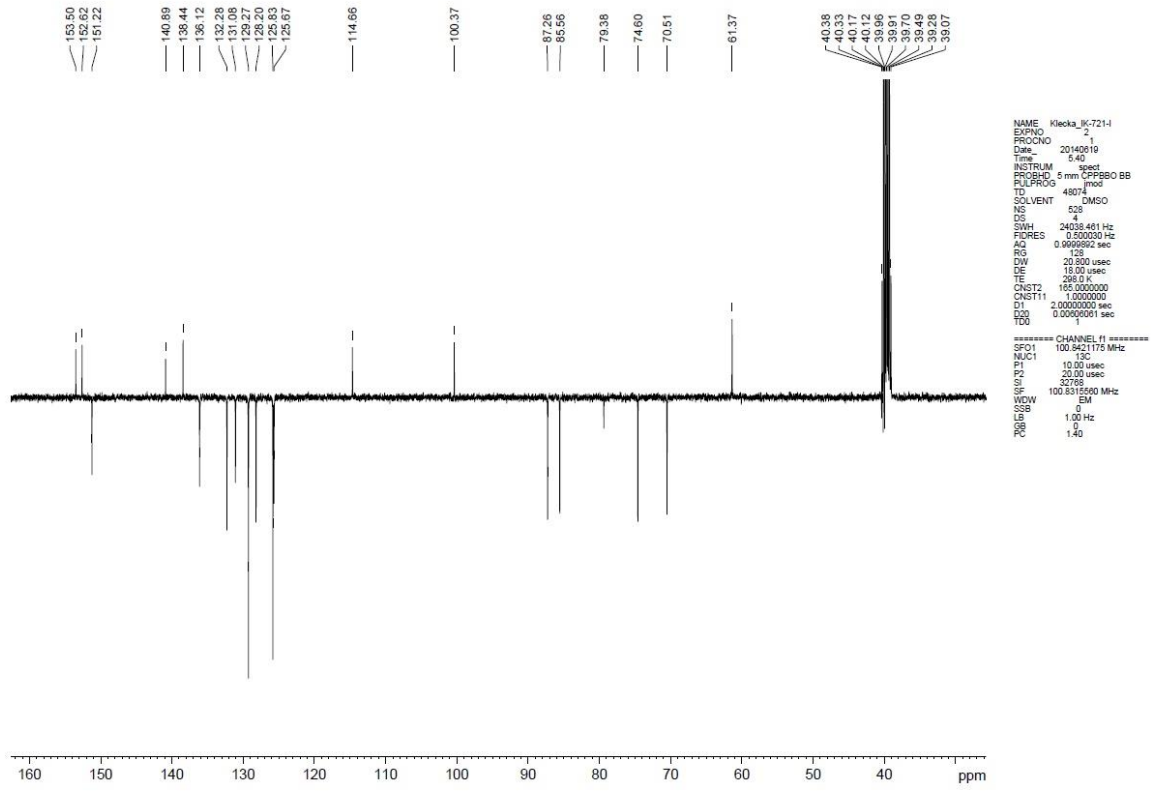

8.72 8.13 8.12 8.11 8.00 7.98 7.98 7.94 7.92 7.92 7.53 7.47 7.47 7.40 7.40 7.36 7.36 7.21 7.20 7.20 7.19 7.00 7.00 6.99 6.99 6.88 6.88 6.87 6.87 6.68 6.67 6.23 6.21 6.20 6.16 6.15 6.15 6.14
4.89 4.88 4.87 4.86 4.80 4.79 4.78 4.78 4.71 4.70 4.68 4.67
2.92
Klecka_IK704-I
1H NMR in CDCl3
03-07-2014 LS
NAME Klecka_IK704-I
EXPNO 1
PROCNO 1
Date_ 20140703
Time_ 9.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 54984
SOLVENT CDCl3
NS 32
DS 0
SWH 5498.534 Hz
FIDRES 0.100002 Hz
AQ 4.9999285 sec
RG 199.71
DW 90.933 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 499.9824999 MHz
NUC1 1H
P1 9.00 usec
SI 131072
SF 499.9800121 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
CH3
S
N
N
O
O
O
O
O
4d
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 ppm
0.9 2.2 2.3 2.2 12.5 1.0 0.9 1.0 1.0 1.0 1.0 1.1 1.1 1.0 2.9

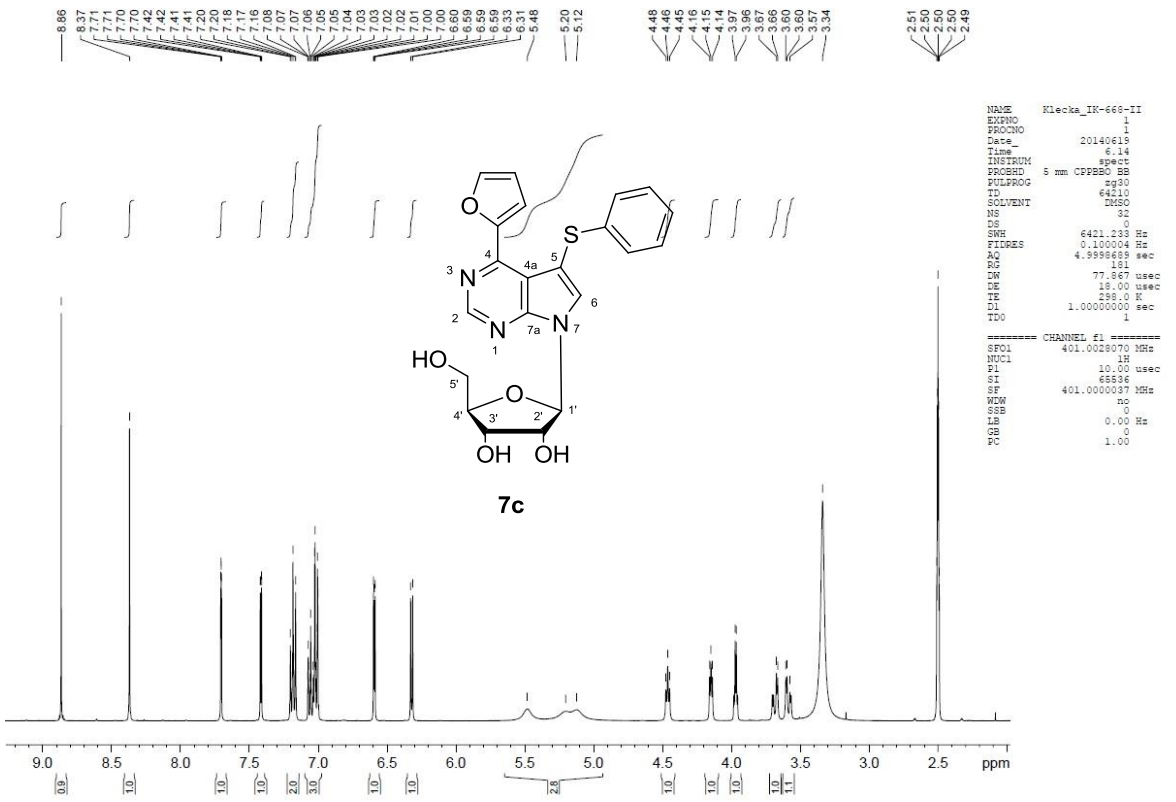

166.15 165.36 165.07 161.21
152.18 151.38 133.67 133.44 130.34 130.06 129.85 129.83 129.76 129.41 128.80 128.68 128.60 128.57 128.56 128.52 128.46 128.41 128.19 127.51 118.00 108.20
86.60
80.43 77.25 77.00 76.75 74.05 71.51
63.71
21.94
Klecka_IK704-I
APT-13C-NMR in CDCl3
03-07-2014 LS
NAME Klecka_IK704-I
EXPNO 2
PROCNO 1
Date_ 20140703
Time_ 9.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 59520
SOLVENT CDCl3
NS 1590
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999990 sec
RG 199.71
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
CNST2 160.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00625000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7332716 MHz
NUC1 13C
P1 7.60 usec
P2 15.20 usec
SI 131072
SF 125.7200728 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm

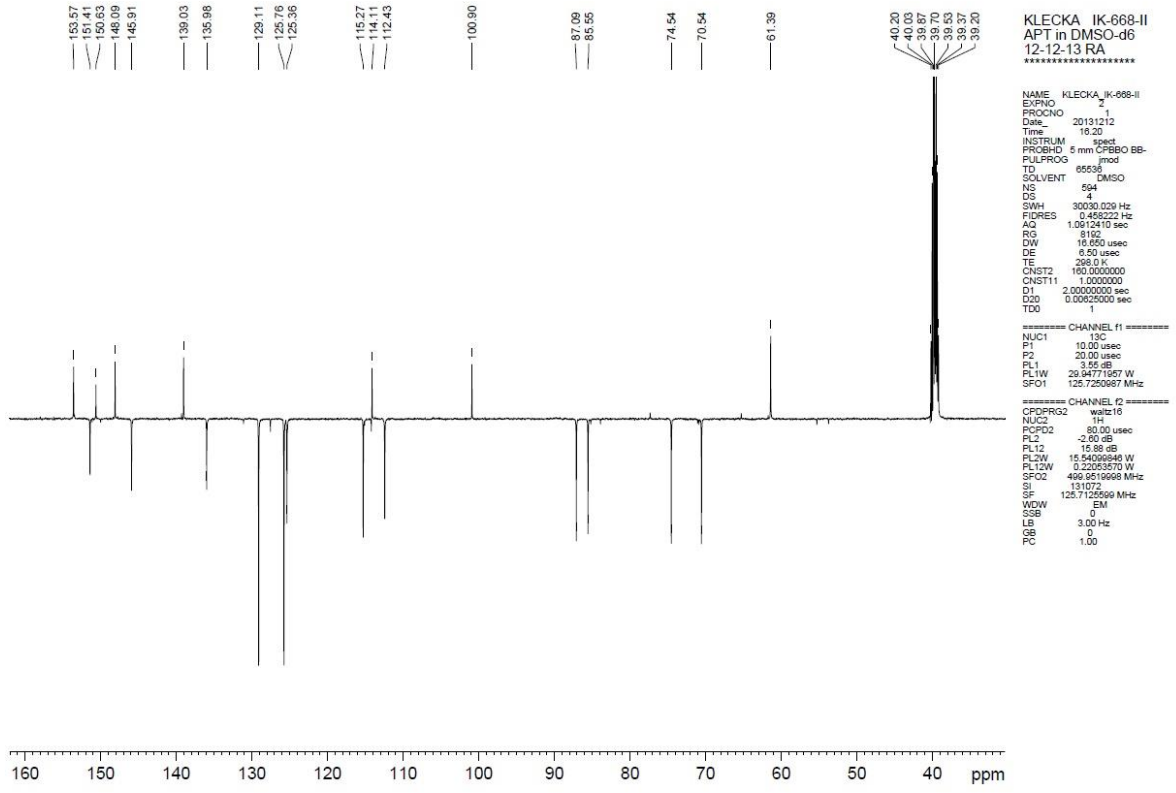

Klecka_IK709-I
1H NMR in CDCl3
02-07-2014 LS
4e
ppm

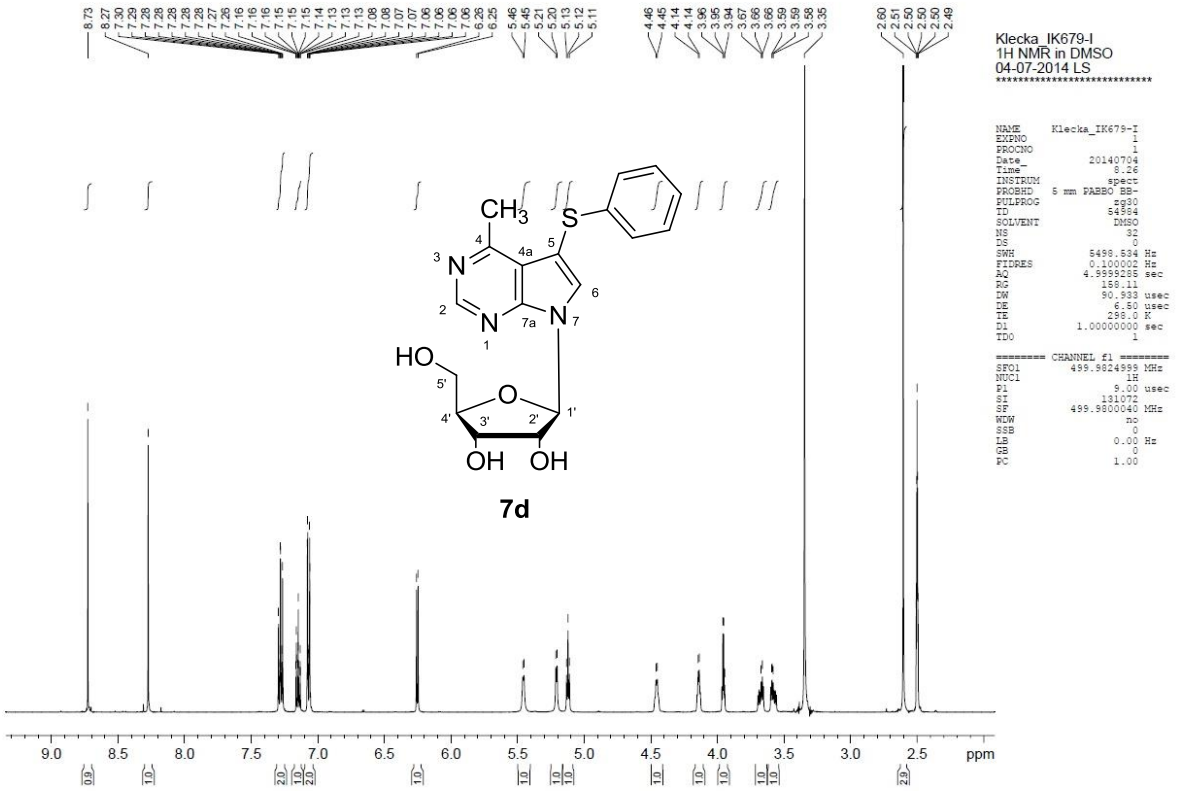

Klecka_IK709-I
APT-13C-NMR in CDCl3
02-07-2014 LS
ppm

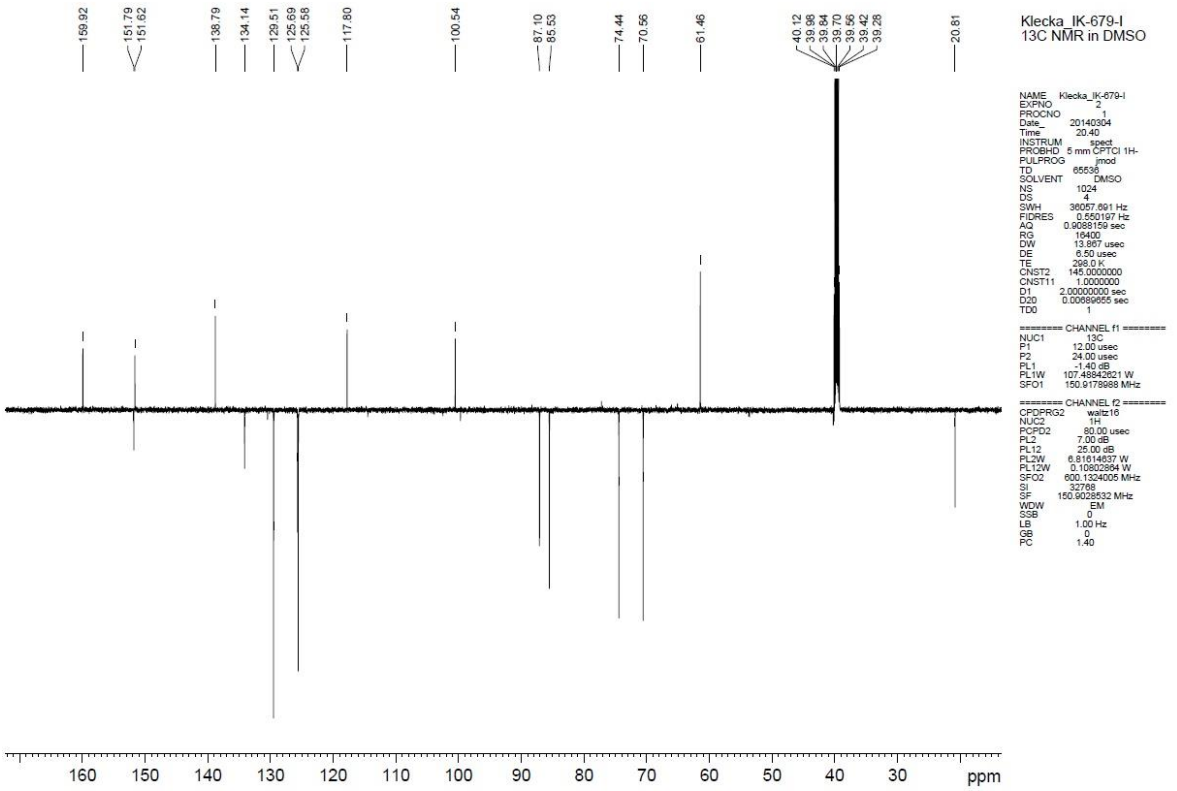

8.84 8.41 8.35 8.35 8.34 8.34 8.30 7.72 7.71 7.70 7.70 7.18 7.17 7.16 7.15 7.14 7.14 7.09 7.08 7.08 7.07 7.07 7.06 7.06 7.05 7.04 7.04 7.03 6.94 6.94 6.93 6.92 6.92 6.92 6.33 6.32 5.50 5.25 5.15 4.48 4.46 4.45 4.45 4.16 4.14 3.99 3.98 3.97 3.68 3.67 3.61 3.60 3.37 3.34 2.52 2.52 2.51 2.50 2.50 2.50 2.49
NAME Klecka_IK-721-I
EXPNO 1
PROCNO 1
Date_ 20140619
Time_ 5.12
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 64210
SOLVENT DMSO
NS 32
DS 0
SWH 6421.233 Hz
FIDRES 0.100004 Hz
AQ 4.9998689 sec
RG 114
DW 77.867 usec
DE 18.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 401.0028070 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 401.0000036 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7b
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 ppm
0.9 1.0 1.0 0.9 2.0 2.0 2.0 1.0 2.9 1.0 1.0 1.0 1.0 1.0

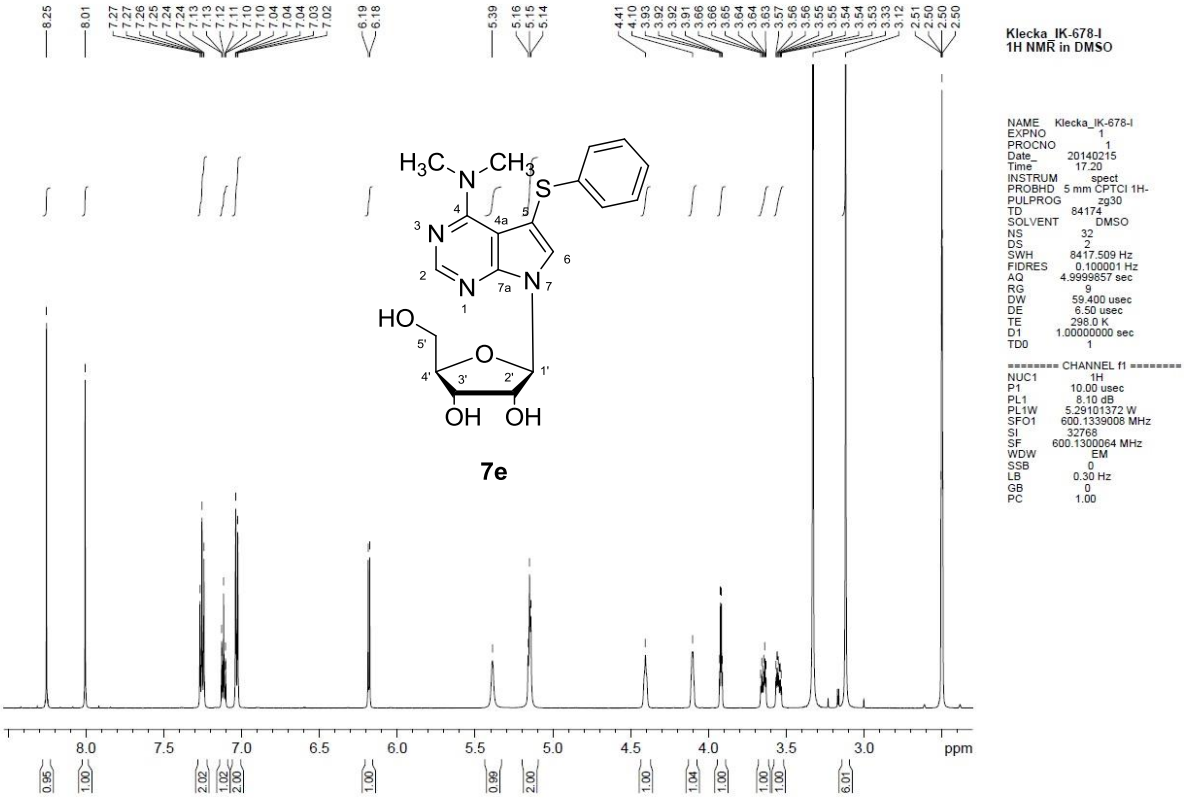

153.50 152.62 151.22 140.89 138.44 136.12 132.28 131.08 129.27 128.20 125.83 125.67 114.66 100.37 87.26 85.56 79.38 74.60 70.51 61.37 40.38 40.33 40.17 40.12 39.96 39.91 39.70 39.49 39.28 39.07
NAME Klecka_IK-721-I
EXPNO 2
PROCNO 1
Date_ 20140619
Time_ 5.40
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG jmod
TD 48074
SOLVENT DMSO
NS 528
DS 4
SWH 24038.461 Hz
FIDRES 0.500030 Hz
AQ 0.9999892 sec
RG 128
DW 20.800 usec
DE 18.00 usec
TE 298.0 K
CNST2 165.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00606061 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.8421175 MHz
NUC1 13C
P1 10.00 usec
P2 20.00 usec
SI 32768
SF 100.8315560 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 ppm


8.86
9.37
7.71
7.70
7.42
7.41
7.20
7.18
7.17
7.16
7.08
7.07
7.06
7.05
7.04
7.03
7.02
7.01
7.00
6.60
6.59
6.59
6.33
6.31
5.48
5.20
5.12
4.48
4.46
4.45
4.16
4.15
4.14
3.97
3.96
3.67
3.66
3.60
3.60
3.57
3.34
2.51
2.50
2.50
2.50
2.49
NAME Klecka_IK-668-II
EXPNO 1
PROCNO 1
Date_ 20140619
Time 6.14
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 64210
SOLVENT DMSO
NS 32
DS 0
SWH 6421.233 Hz
FIDRES 0.100004 Hz
AQ 4.9998689 sec
RG 181
DW 77.867 usec
DE 18.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 401.0028070 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 401.0000037 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
O
S
N
N
N
HO
O
OH
OH
7c
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm


153.57
151.41
150.63
148.09
145.91
139.03
135.98
129.11
125.76
125.36
115.27
114.11
112.43
100.90
87.09
85.55
74.54
70.54
61.39
40.20
40.03
39.87
39.70
39.53
39.37
39.20
KLECKA IK-668-II
APT in DMSO-d6
12-12-13 RA
****
NAME KLECKA_IK-668-II
EXPNO 2
PROCNO 1
Date_ 20131212
Time 16.20
INSTRUM spect
PROBHD 5 mm CPBBO BB-
PULPROG apt.mod
TD 65536
SOLVENT DMSO
NS 594
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 8192
DW 16.650 usec
DE 6.50 usec
TE 298.0 K
CNST2 160.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00625000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
P2 20.00 usec
PL1 3.55 dB
PL1W 29.94771957 W
SFO1 125.7250987 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.60 dB
PL12 15.88 dB
PL2W 15.54099846 W
PL12W 0.22053570 W
SFO2 499.9519998 MHz
SI 131072
SF 125.7125699 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm


Klecka_IK679-I
1H NMR in DMSO
04-07-2014 LS
7d


Klecka_IK-679-I
13C NMR in DMSO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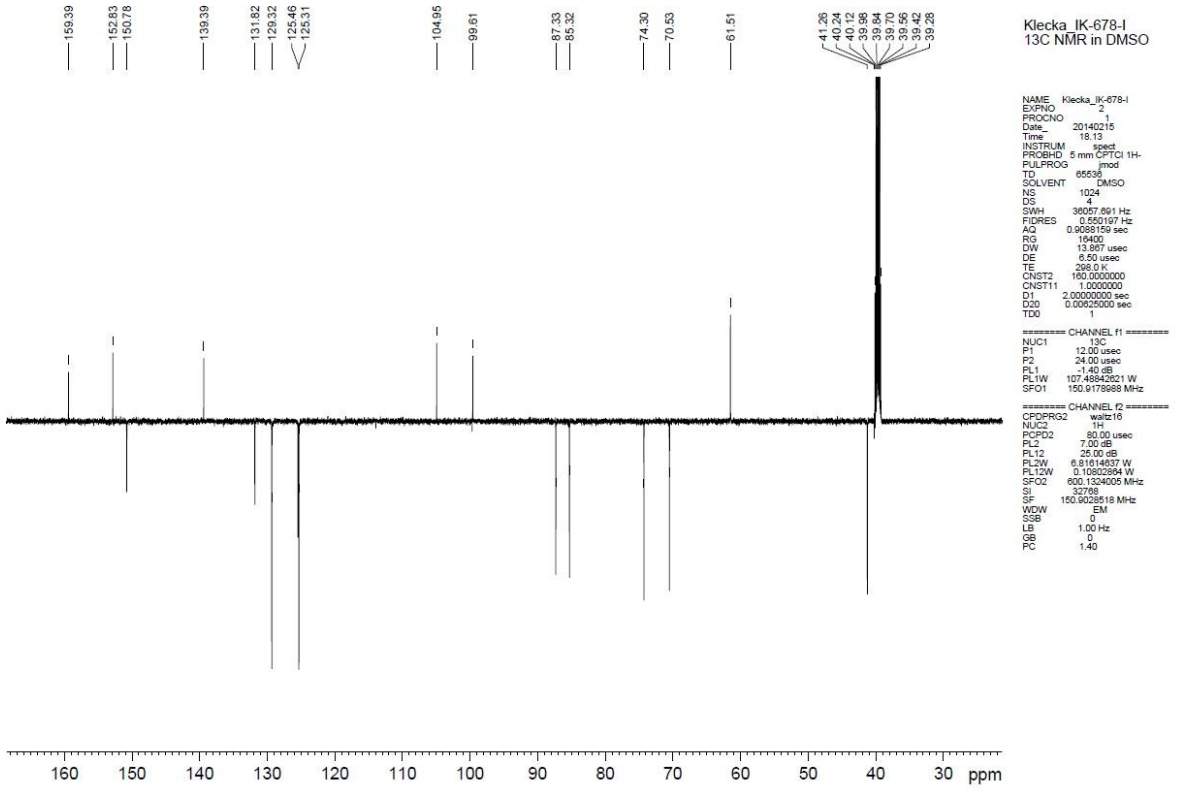
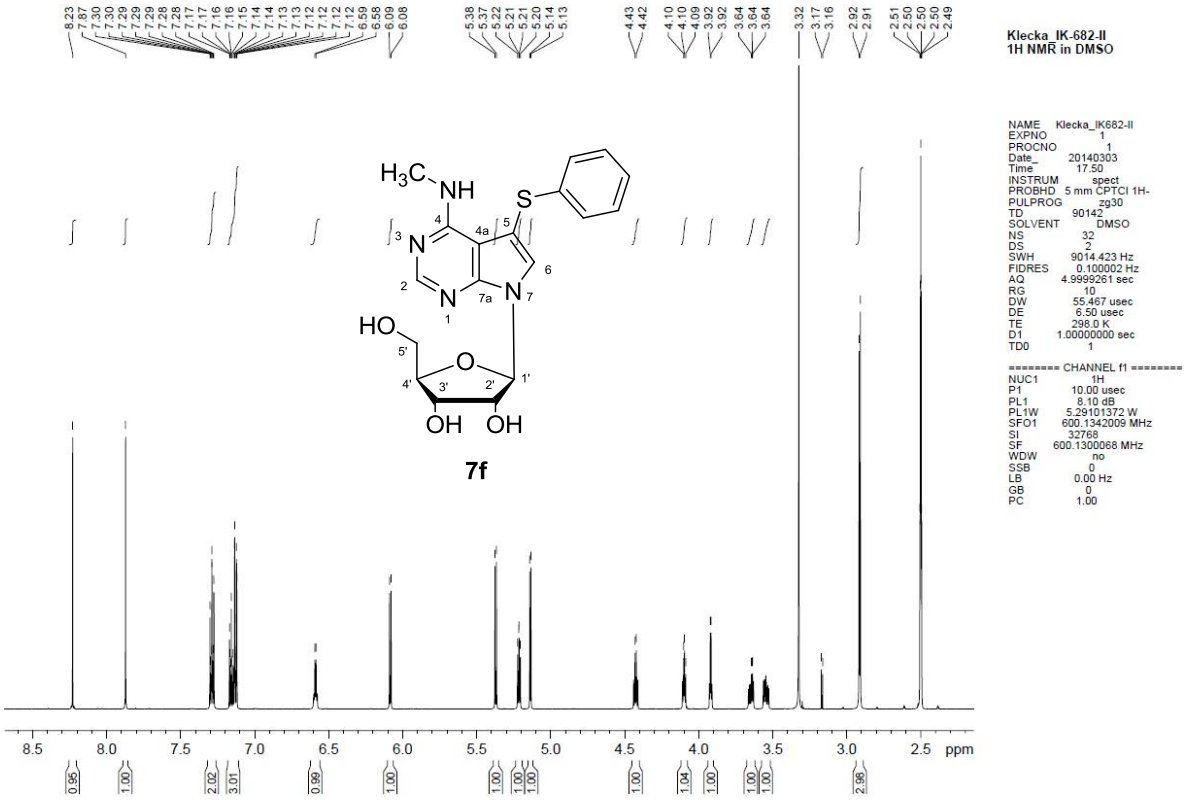
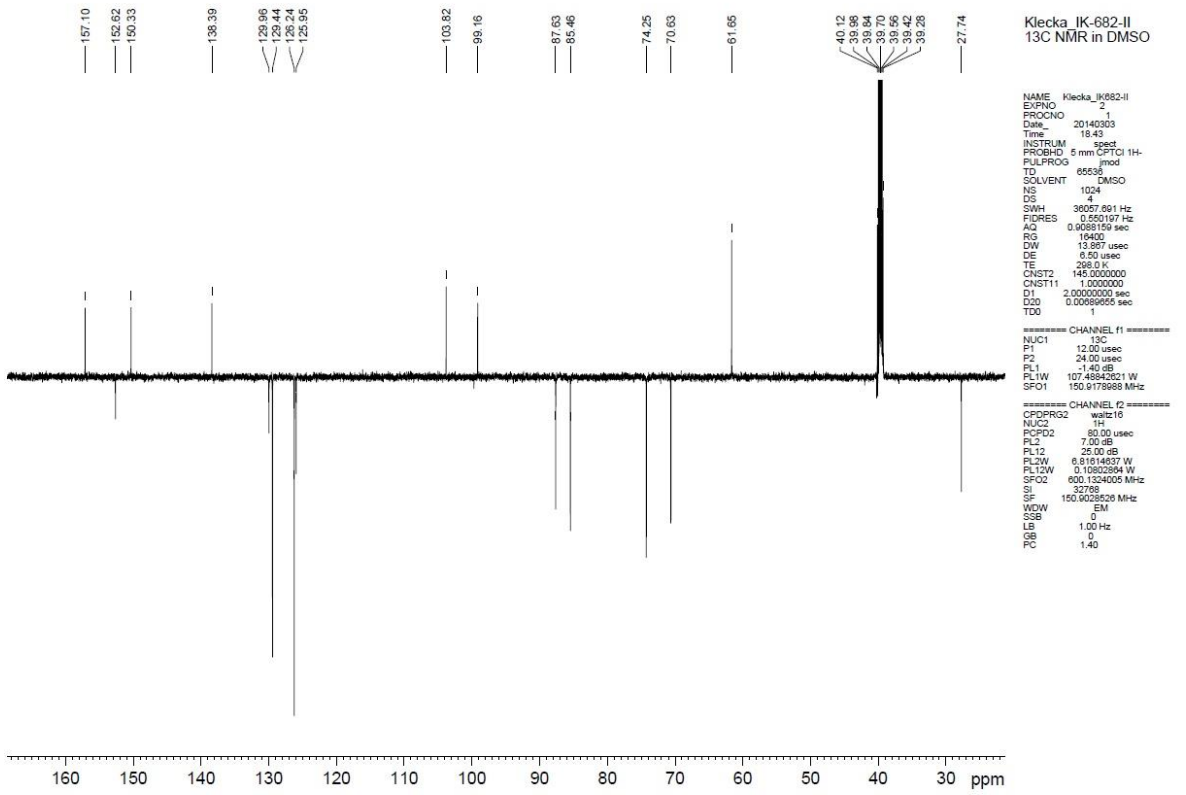
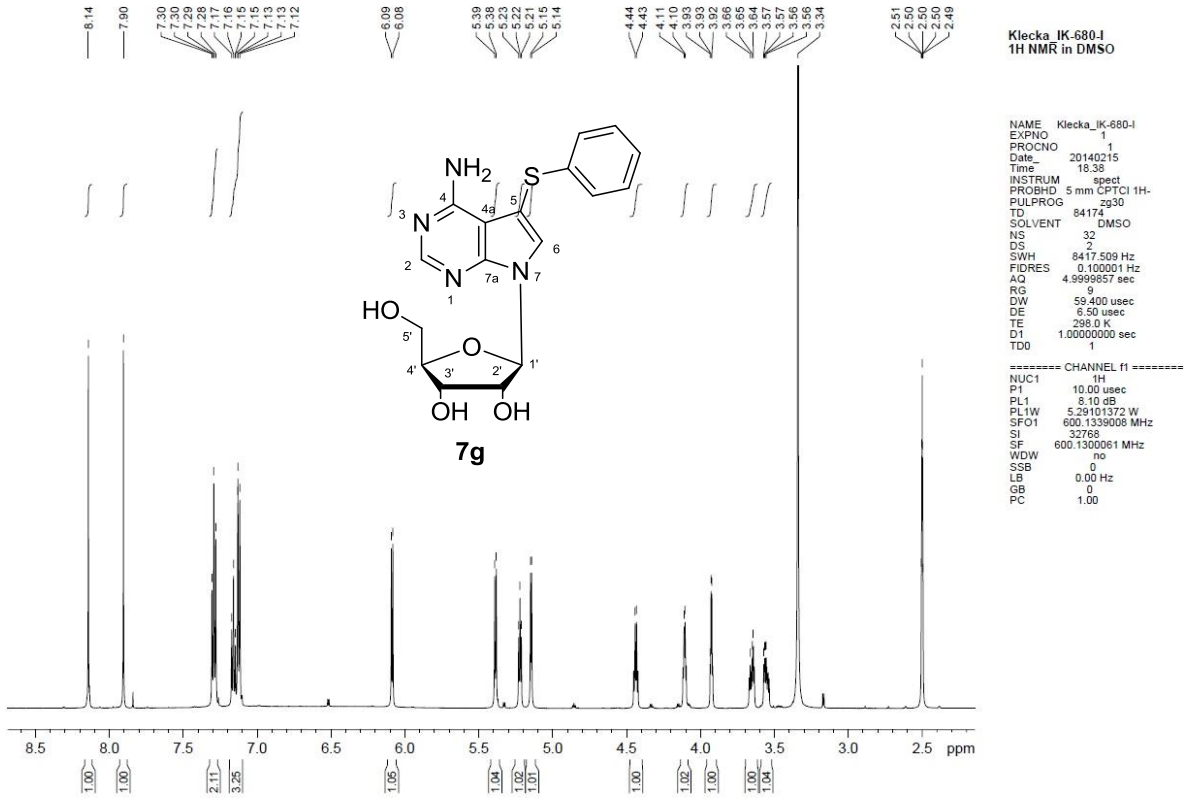
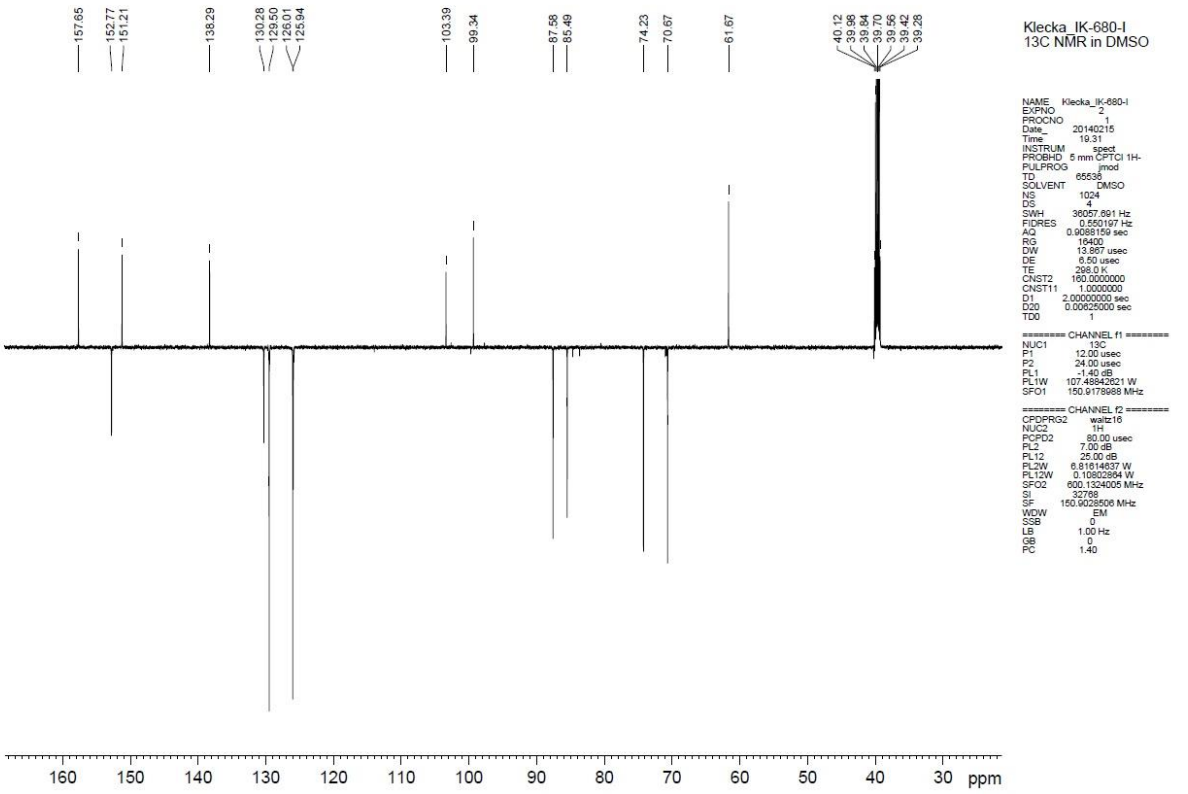
8.01
7.27
7.26
7.25
7.24
7.13
7.12
7.11
7.10
7.04
7.03
7.02
6.19
6.18
5.39
5.16
5.15
5.14
4.41
4.10
3.93
3.92
3.91
3.66
3.65
3.64
3.57
3.56
3.55
3.54
3.53
3.33
3.12
2.51
2.50
2.50
2.50
Klecka_IK-678-I
1H NMR in DMSO
NAME Klecka_IK-678-I
EXPNO 1
PROCNO 1
Date_ 20140215
Time 17.20
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 84174
SOLVENT DMSO
NS 32
DS 2
SWH 8417.509 Hz
FIDRES 0.100001 Hz
AQ 4.9999857 sec
RG 9
DW 59.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 8.10 dB
PL1W 5.29101372 W
SFO1 600.1339008 MHz
SI 32768
SF 600.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7e
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
ppm
0.95
1.00
2.02
1.02
2.00
1.00
0.99
2.00
1.00
1.04
1.00
1.00
1.00
6.01

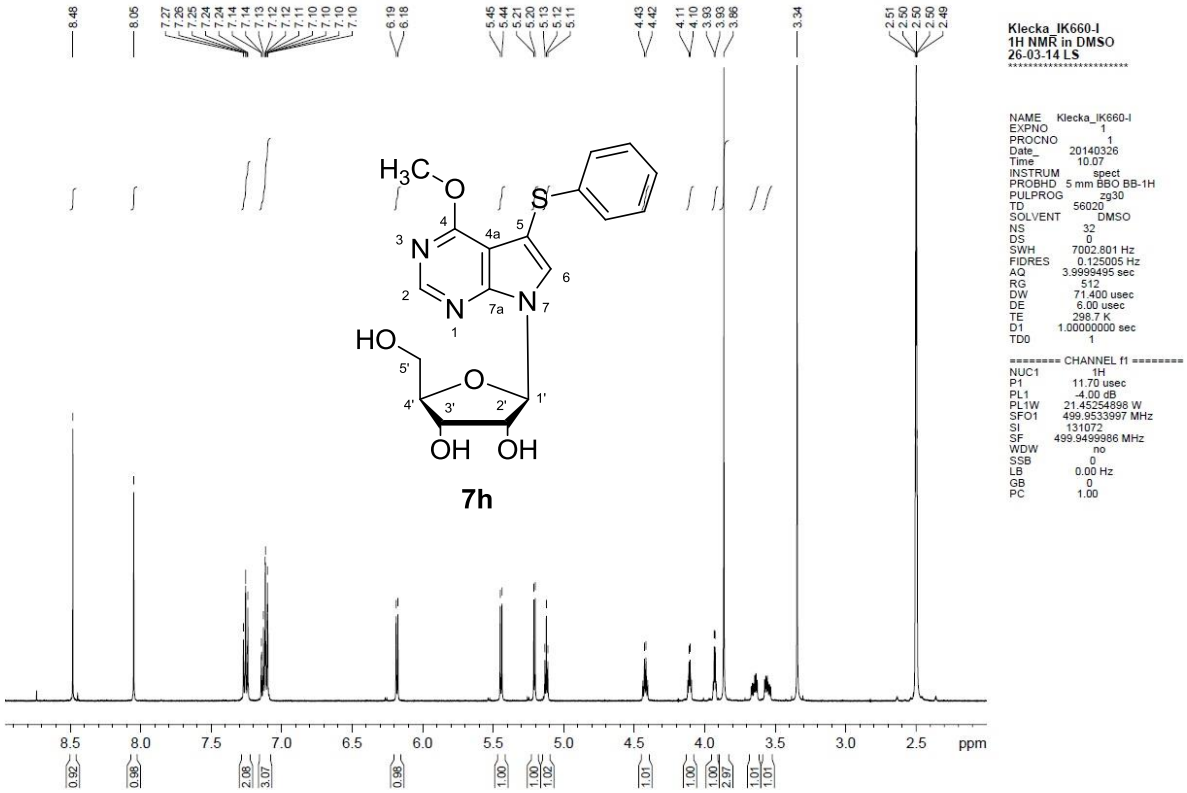

159.39
152.83
150.78
139.39
131.82
129.32
125.46
125.31
104.95
99.61
87.33
85.32
74.30
70.53
61.51
41.26
40.24
40.03
39.98
39.84
39.70
39.56
39.42
39.28
Klecka_IK-678-I
13C NMR in DMSO
NAME Klecka_IK-678-I
EXPNO 2
PROCNO 1
Date_ 20140215
Time 18.13
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 16400
DW 13.867 usec
DE 6.50 usec
TE 298.0 K
CNST2 160.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00625000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
P2 24.00 usec
PL1 -1.40 dB
PL1W 107.48842621 W
SFO1 150.9178988 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 7.00 dB
PL12 25.00 dB
PL2W 6.81614637 W
PL12W 0.10802894 W
SFO2 600.1324005 MHz
SI 32768
SF 150.9028518 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

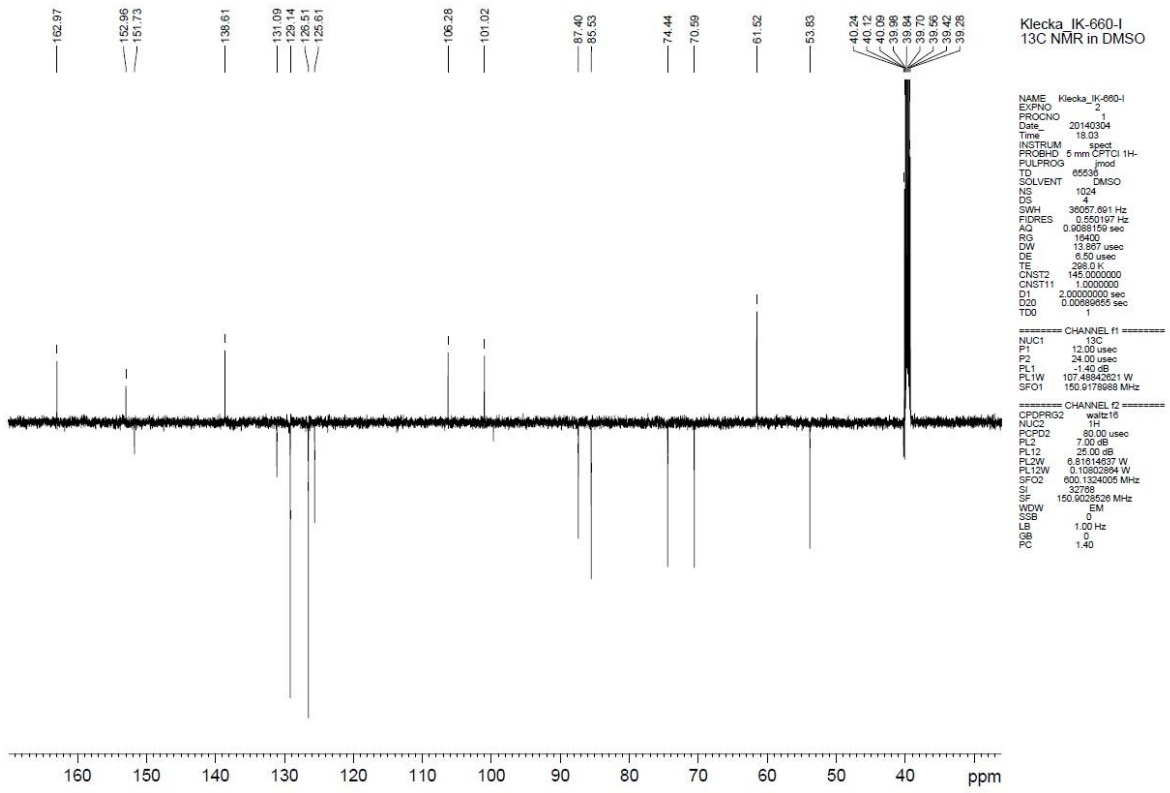

Klecka_IK-682-II
1H NMR in DMSO
7f

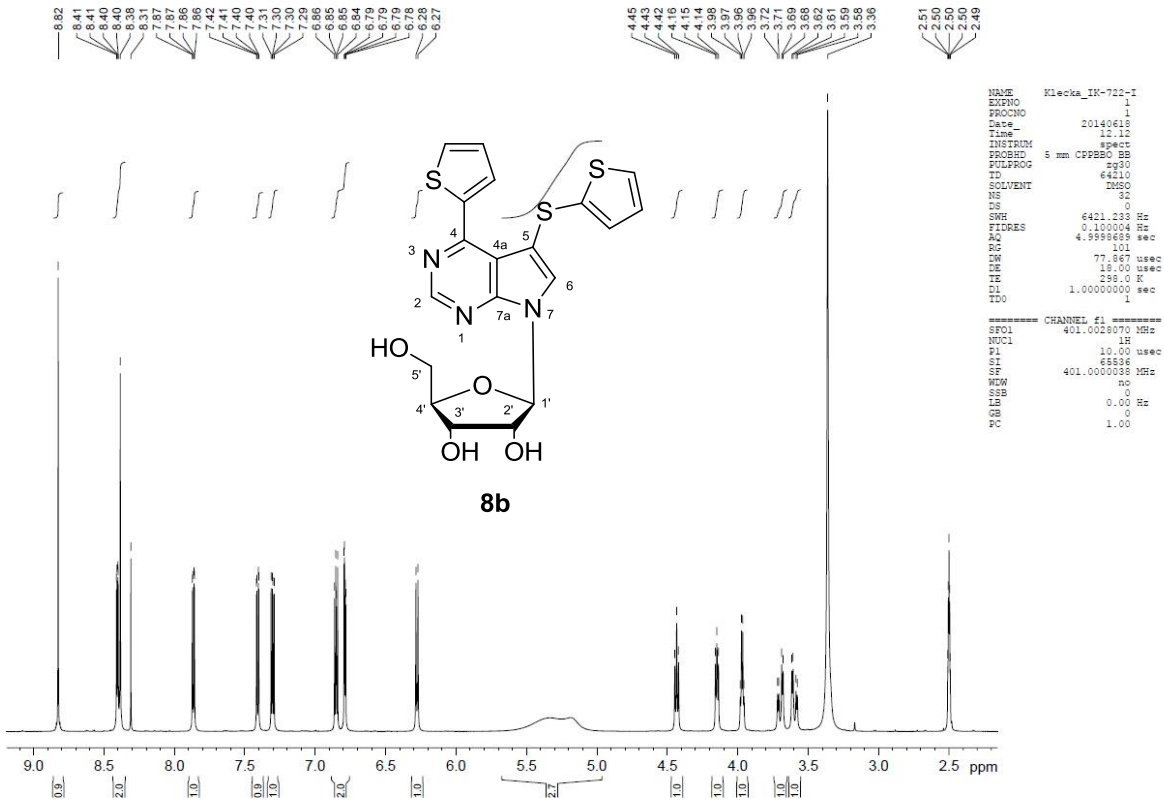

Klecka_IK-682-II
13C NMR in DMSO

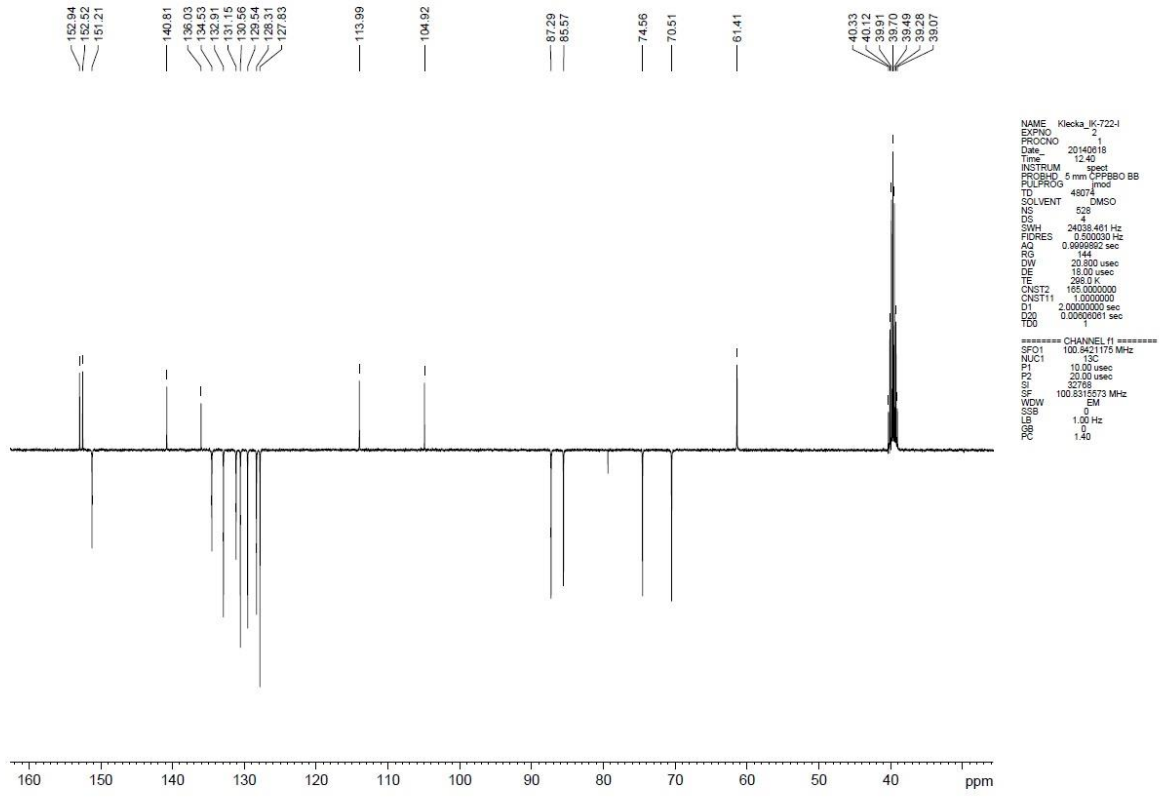

8.14
7.90
7.30
7.30
7.29
7.28
7.17
7.16
7.15
7.15
7.13
7.13
7.12
6.09
6.08
5.39
5.38
5.23
5.22
5.21
5.15
5.14
4.44
4.43
4.11
4.10
3.93
3.93
3.92
3.66
3.65
3.64
3.57
3.57
3.56
3.56
3.34
2.51
2.50
2.50
2.50
2.49
NH2
S
N
N
N
HO
OH
OH
O
7g
Klecka_IK-680-I
1H NMR in DMSO
NAME Klecka_IK-680-I
EXPNO 1
PROCNO 1
Date_ 20140215
Time 18.38
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 84174
SOLVENT DMSO
NS 32
DS 2
SWH 8417.509 Hz
FIDRES 0.100001 Hz
AQ 4.9999857 sec
RG 9
DW 59.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 8.10 dB
PL1W 5.29101372 W
SFO1 600.1339008 MHz
SI 32768
SF 600.1300061 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
1.00
1.00
2.11
3.25
1.05
1.04
1.02
1.01
1.00
1.02
1.00
1.00
1.04

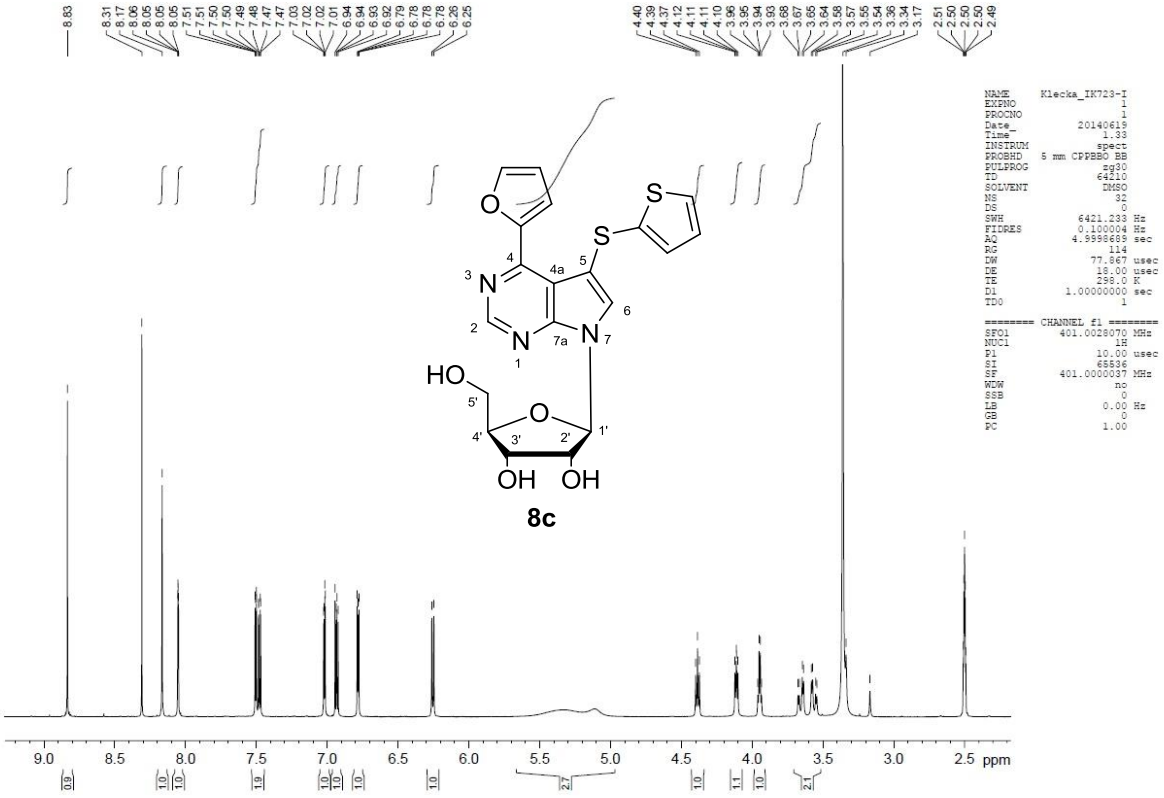

157.65
152.77
151.21
138.29
130.28
129.50
126.01
125.94
103.39
99.34
87.58
85.49
74.23
70.67
61.67
40.12
39.96
39.94
39.70
39.56
39.42
39.28
Klecka_IK-680-I
13C NMR in DMSO
NAME Klecka_IK-680-I
EXPNO 2
PROCNO 1
Date_ 20140215
Time 19.31
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 16400
DW 13.867 usec
DE 6.50 usec
TE 298.0 K
CNST2 160.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00625000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
P2 24.00 usec
PL1 -1.40 dB
PL1W 107.48842621 W
SFO1 150.9178988 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 7.00 dB
PL12 25.00 dB
PL2W 6.81614637 W
PL12W 0.10802864 W
SFO2 600.1324005 MHz
SI 32768
SF 150.9028506 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm


Klecka_IK660-I
1H NMR in DMSO
26-03-14 LS
7h
ppm


Klecka_IK-660-I
13C NMR in DMSO
ppm


8.82 8.41 8.41 8.40 8.40 8.38 8.31 7.87 7.87 7.86 7.86 7.42 7.41 7.40 7.40 7.31 7.30 7.30 7.29 6.86 6.85 6.85 6.84 6.79 6.79 6.78 6.28 6.27
4.45 4.43 4.42 4.16 4.15 4.14 3.98 3.97 3.96 3.72 3.71 3.69 3.68 3.62 3.61 3.59 3.58 3.36
2.51 2.50 2.50 2.50 2.50 2.49
NAME Klecka_IK-722-I
EXPNO 1
PROCNO 1
Date_ 20140618
Time 12.12
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 64210
SOLVENT DMSO
NS 32
DS 0
SWH 6421.233 Hz
FIDRES 0.100004 Hz
AQ 4.9998689 sec
RG 101
DW 77.867 usec
DE 18.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 401.0028070 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 401.0000038 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
HO
5'
O
4'
3'
2'
1'
OH
OH
S
S
S
4
4a
5
3
N
6
2
N
7a
N
7
1
8b
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 ppm
0.9 2.0 1.0 0.9 1.0 2.0 1.0 2.7 1.0 1.0 1.0 1.0 1.0


152.94 152.52 151.21 140.81 136.03 134.53 132.91 131.15 130.56 129.54 128.31 127.83 113.99 104.92 87.29 85.57 74.56 70.51 61.41 40.33 40.12 39.91 39.70 39.49 39.28 39.07
NAME Klecka_IK-722-I
EXPNO 2
PROCNO 1
Date_ 20140618
Time 12.40
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG jmod
TD 48074
SOLVENT DMSO
NS 528
DS 4
SWH 24038.461 Hz
FIDRES 0.500030 Hz
AQ 0.9999992 sec
RG 144
DW 20.800 usec
DE 18.00 usec
TE 298.0 K
CNST2 165.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00606061 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.8421175 MHz
NUC1 13C
P1 10.00 usec
P2 20.00 usec
SI 32768
SF 100.8315573 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 ppm


8.83
8.31
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8.06
8.05
8.05
8.05
7.51
7.51
7.51
7.50
7.50
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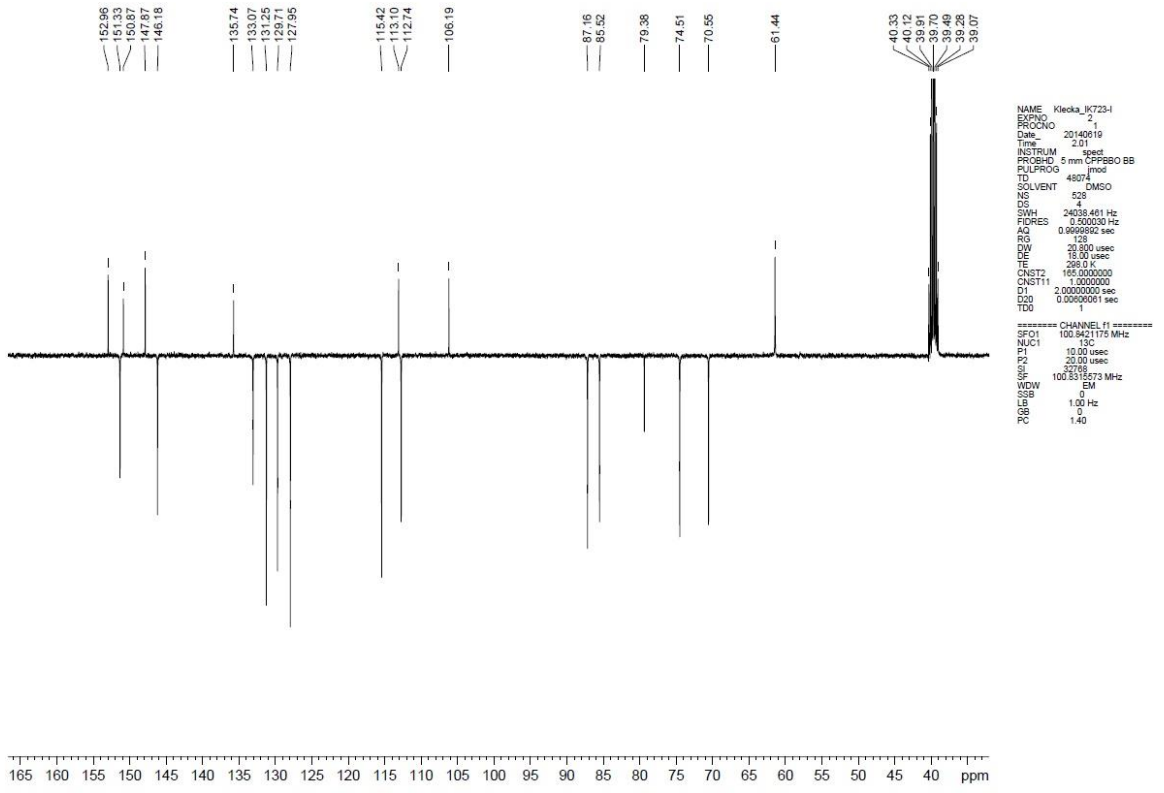
7.48
7.47
7.47
7.03
7.02
7.02
7.01
6.94
6.94
6.93
6.92
6.79
6.78
6.78
6.78
6.26
6.25
4.40
4.39
4.37
4.12
4.11
4.11
4.10
3.96
3.95
3.94
3.93
3.68
3.67
3.66
3.64
3.58
3.57
3.55
3.54
3.36
3.34
3.17
2.51
2.50
2.50
2.50
2.50
2.49
NAME Klecka_IK723-I
EXPNO 1
PROCNO 1
Date_ 20140619
Time_ 1.33
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 64210
SOLVENT DMSO
NS 32
DS 0
SWH 6421.233 Hz
FIDRES 0.100004 Hz
AQ 4.9998689 sec
RG 114
DW 77.867 usec
DE 18.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 401.0028070 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 401.0000037 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
O
S
S
4
4a
5
3
N
6
2
N
7a
N
7
1
HO
5'
O
4'
3'
2'
1'
OH
OH
8c
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
0.9
1.0
1.9
1.9
1.0
1.0
1.0
1.0
2.7
1.0
1.1
1.0
2.1


152.96
151.33
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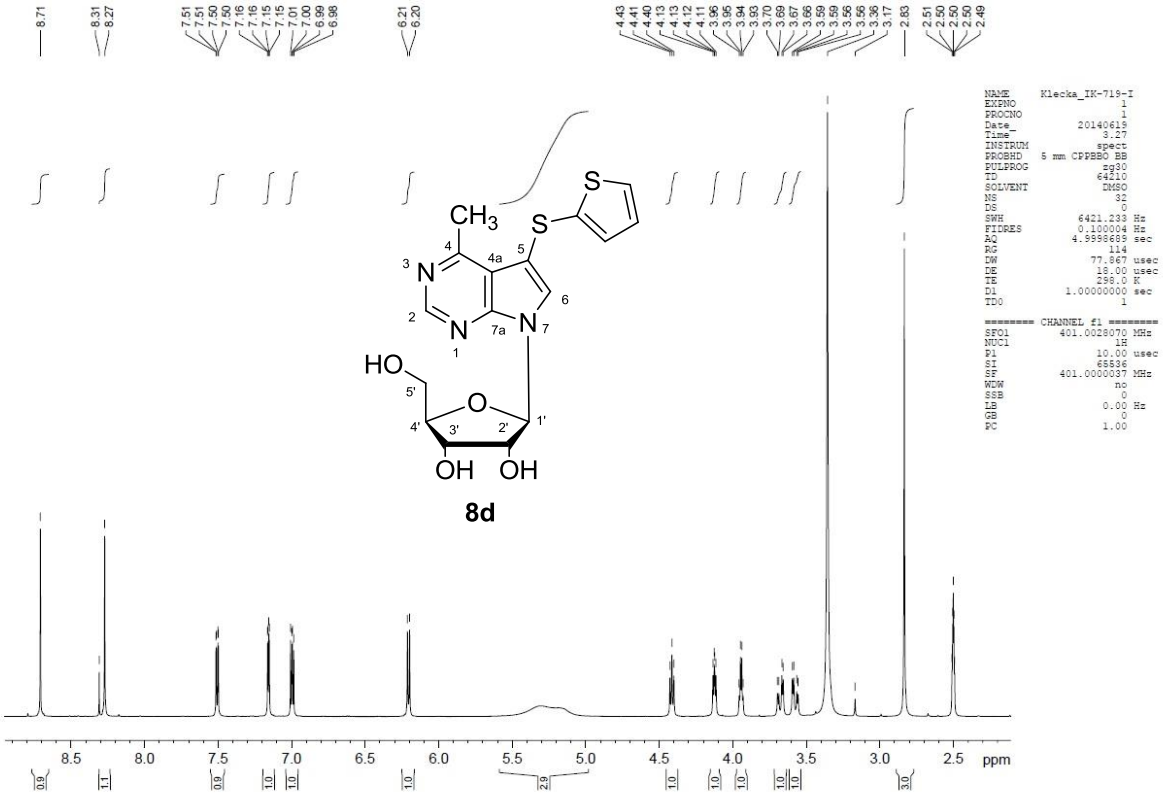
147.87
146.18
135.74
133.07
131.25
129.71
127.95
115.42
113.10
112.74
106.19
87.16
85.52
79.38
74.51
70.55
61.44
40.33
40.12
39.91
39.70
39.49
39.28
39.07
NAME Klecka_IK723-I
EXPNO 2
PROCNO 1
Date_ 20140619
Time_ 2.01
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG jmod
TD 48074
SOLVENT DMSO
NS 528
DS 4
SWH 24038.461 Hz
FIDRES 0.500030 Hz
AQ 0.9999892 sec
RG 128
DW 20.800 usec
DE 18.00 usec
TE 298.0 K
CNST2 165.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00606061 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.8421175 MHz
NUC1 13C
P1 10.00 usec
P2 20.00 usec
SI 32768
SF 100.8315573 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
ppm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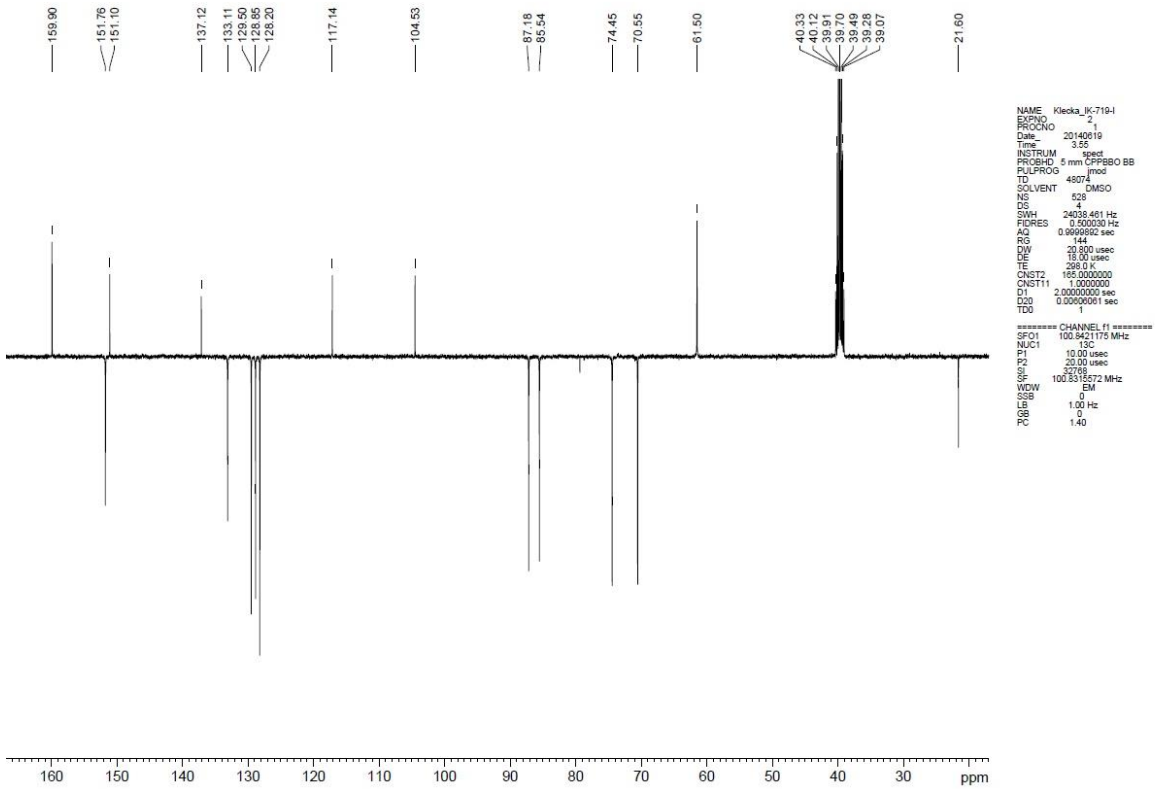
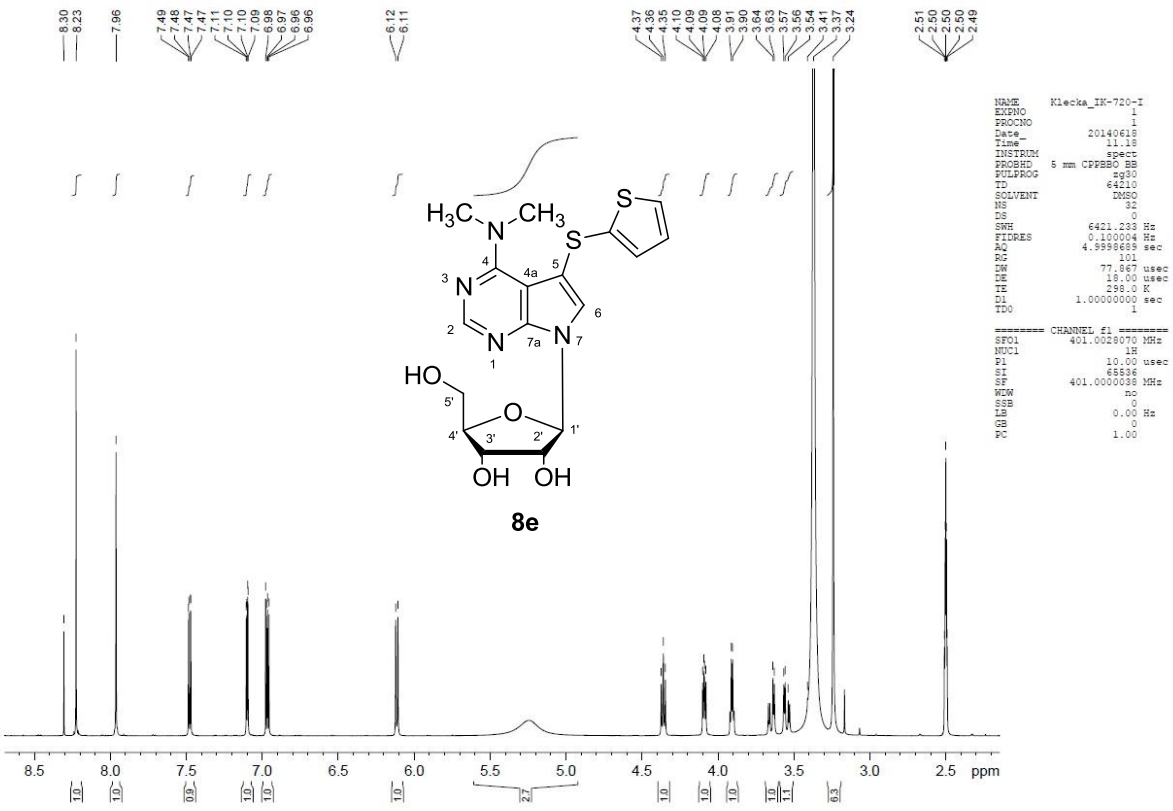
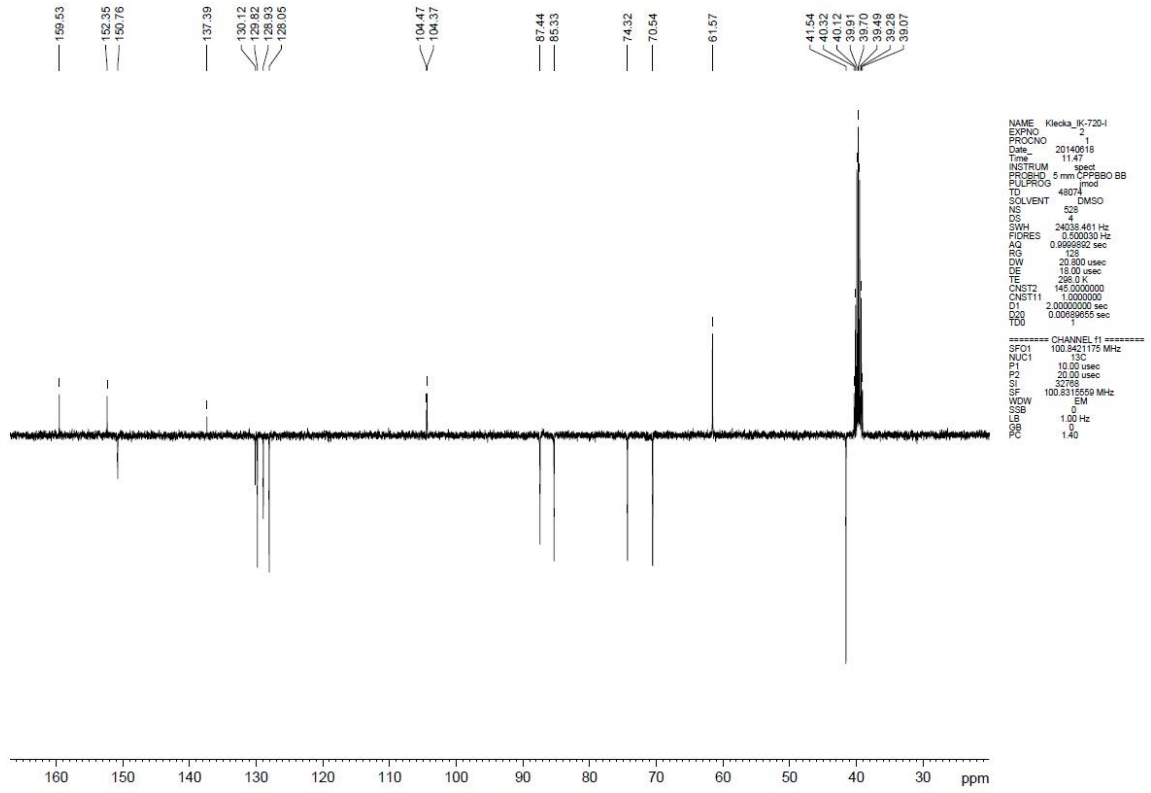
8.31
8.27
7.51
7.51
7.50
7.50
7.16
7.16
7.15
7.15
7.01
7.00
6.99
6.98
6.21
6.20
4.43
4.41
4.40
4.13
4.13
4.12
4.11
3.96
3.95
3.94
3.93
3.70
3.69
3.67
3.66
3.59
3.56
3.56
3.36
3.17
2.83
2.51
2.50
2.50
2.50
2.49
CH3
S
S
HO
OH
OH
8d
ppm


159.90
151.76
151.10
137.12
133.11
129.50
128.85
128.20
117.14
104.53
87.18
85.54
74.45
70.55
61.50
40.33
40.12
39.91
39.70
39.49
39.28
39.07
21.60
ppm


8.30
8.23
7.96
7.49
7.48
7.47
7.47
7.11
7.10
7.10
7.09
6.98
6.97
6.96
6.96
6.12
6.11
4.37
4.36
4.35
4.10
4.09
4.09
4.08
3.91
3.90
3.64
3.63
3.57
3.56
3.54
3.41
3.37
3.24
2.51
2.50
2.50
2.50
2.50
2.49
NAME Klecka_IK-720-I
EXPNO 1
PROCNO 1
Date_ 20140618
Time_ 11.18
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 64210
SOLVENT DMSO
NS 32
DS 0
SWH 6421.233 Hz
FIDRES 0.100004 Hz
AQ 4.9998689 sec
RG 101
DW 77.867 usec
DE 18.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 401.0028070 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 401.0000038 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
HO
5'
O
4'
3'
2'
1'
OH
OH
8e
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
1.0
1.0
0.9
1.0
1.0
1.0
2.7
1.0
1.0
1.0
1.0
1.1
6.3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152.35
150.76
137.39
130.12
129.82
128.93
128.05
104.47
104.37
87.44
85.33
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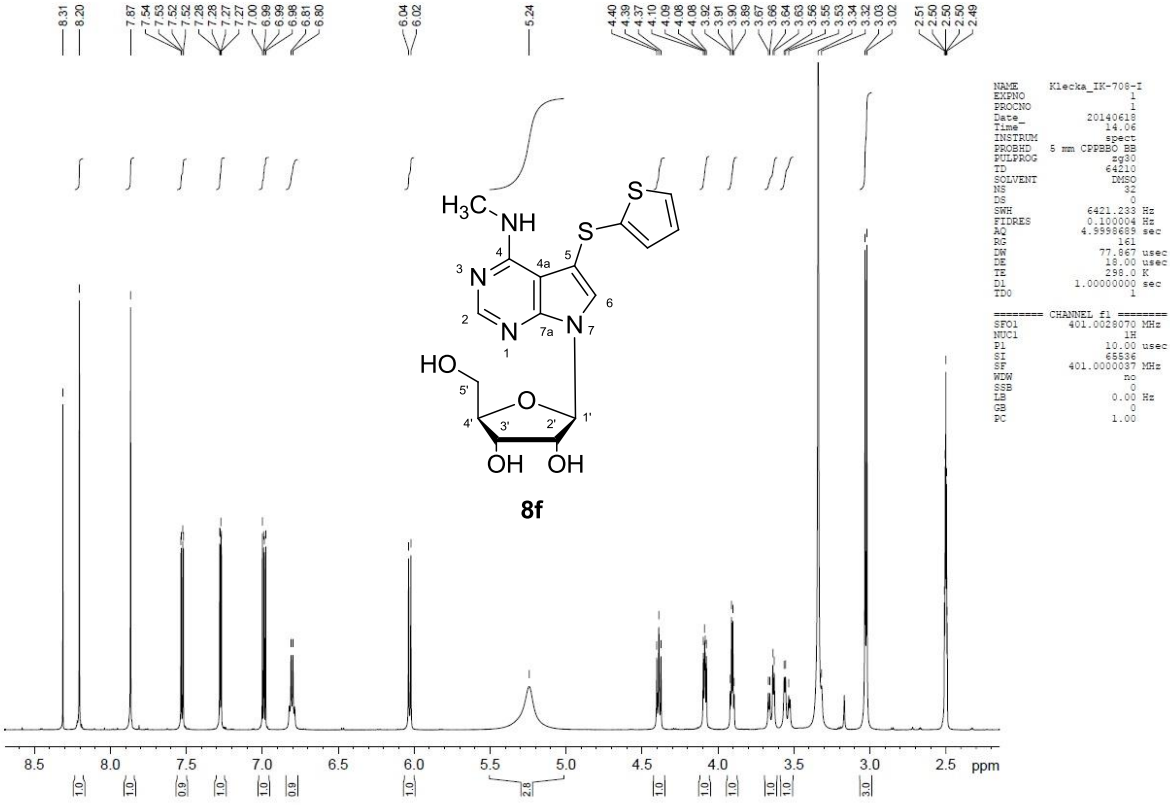
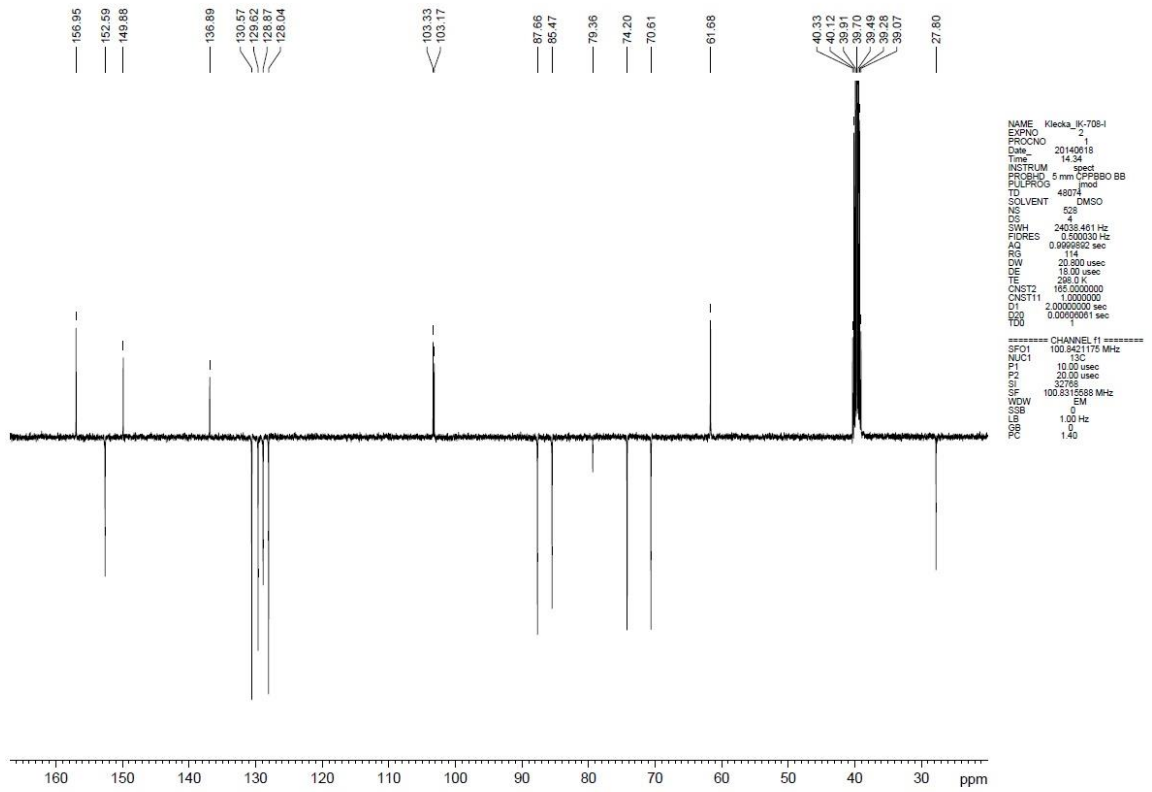
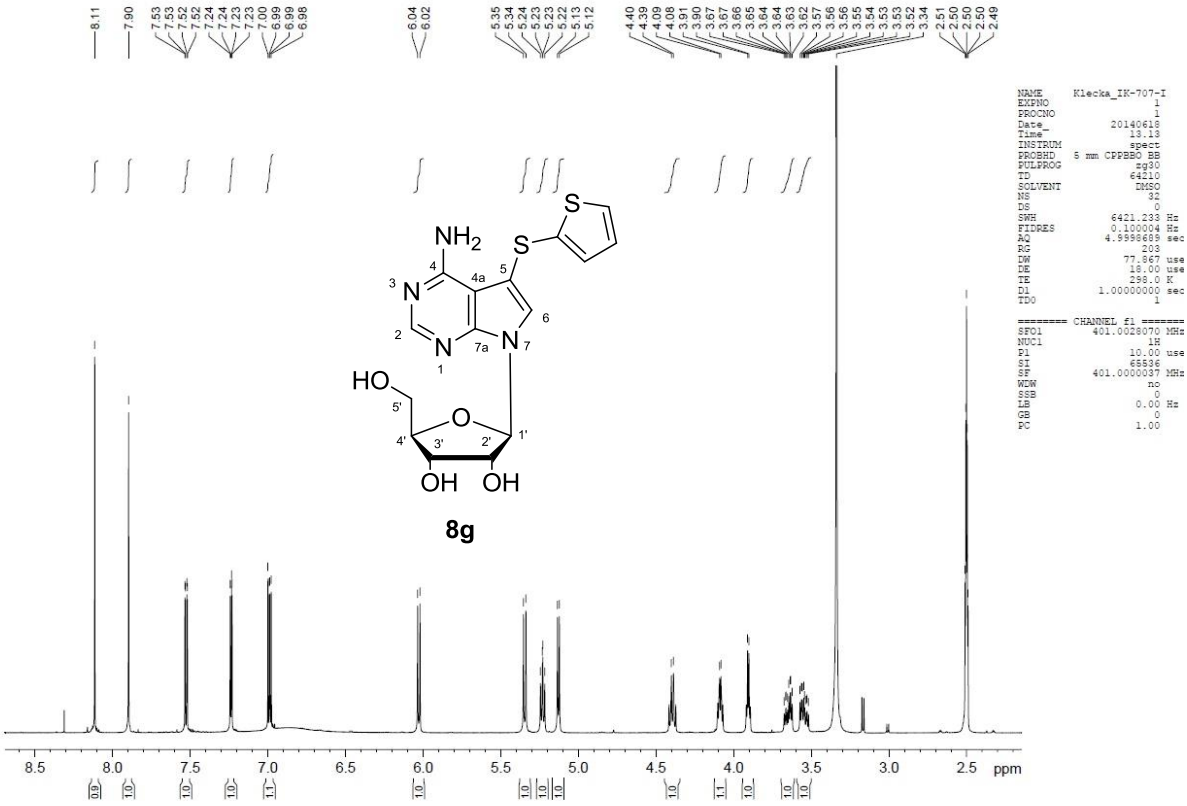
70.54
61.57
41.54
40.32
40.12
39.91
39.70
39.49
39.28
39.07
NAME Klecka_IK-720-I
EXPNO 2
PROCNO 1
Date_ 20140618
Time 11.47
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG jmod
TD 48074
SOLVENT DMSO
NS 528
DS 4
SWH 24038.461 Hz
FIDRES 0.500030 Hz
AQ 0.9999892 sec
RG 128
DW 20.800 usec
DE 18.00 usec
TE 298.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.8421175 MHz
NUC1 13C
P1 10.00 usec
P2 20.00 usec
SI 32768
SF 100.8315559 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm


8.31
8.20
7.87
7.54
7.53
7.52
7.52
7.28
7.28
7.27
7.27
7.00
6.99
6.99
6.98
6.81
6.80
6.04
6.02
5.24
4.40
4.39
4.37
4.10
4.09
4.08
4.08
3.92
3.91
3.90
3.89
3.67
3.66
3.64
3.63
3.56
3.55
3.53
3.34
3.32
3.03
3.02
2.51
2.50
2.50
2.50
2.49
NAME Klecka_IK-708-I
EXPNO 1
PROCNO 1
Date_ 20140618
Time_ 14.06
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 64210
SOLVENT DMSO
NS 32
DS 0
SWH 6421.233 Hz
FIDRES 0.100004 Hz
AQ 4.9998689 sec
RG 161
DW 77.867 usec
DE 18.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 401.0028070 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 401.0000037 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
HO
5'
O
4'
3'
2'
1'
OH
OH
N
NH
S
S
1
2
3
4
4a
5
6
7
7a
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
1.0
1.0
0.9
1.0
1.0
0.9
1.0
2.8
1.0
1.0
1.0
1.0
3.0

8f


156.95
152.59
149.88
136.89
130.57
129.62
128.87
128.04
103.33
103.17
87.66
85.47
79.36
74.20
70.61
61.68
40.33
40.12
39.91
39.70
39.49
39.28
39.07
27.80
NAME Klecka_IK-708-I
EXPNO 2
PROCNO 1
Date_ 20140618
Time_ 14.34
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG jmod
TD 48074
SOLVENT DMSO
NS 528
DS 4
SWH 24038.461 Hz
FIDRES 0.500030 Hz
AQ 0.9999692 sec
RG 114
DW 20.800 usec
DE 18.00 usec
TE 296.0 K
CNST2 165.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00000061 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.8421175 MHz
NUC1 13C
P1 10.00 usec
P2 20.00 usec
SI 32768
SF 100.8315588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm


8.11
7.90
7.53
7.52
7.24
7.23
7.00
6.99
6.98
6.04
6.02
5.35
5.34
5.24
5.23
5.22
5.13
5.12
4.40
4.39
4.09
4.08
3.91
3.90
3.67
3.66
3.65
3.64
3.63
3.62
3.57
3.56
3.55
3.54
3.53
3.52
3.34
2.51
2.50
2.49
NH2
S
N
HO
O
OH
OH
8g
8.5
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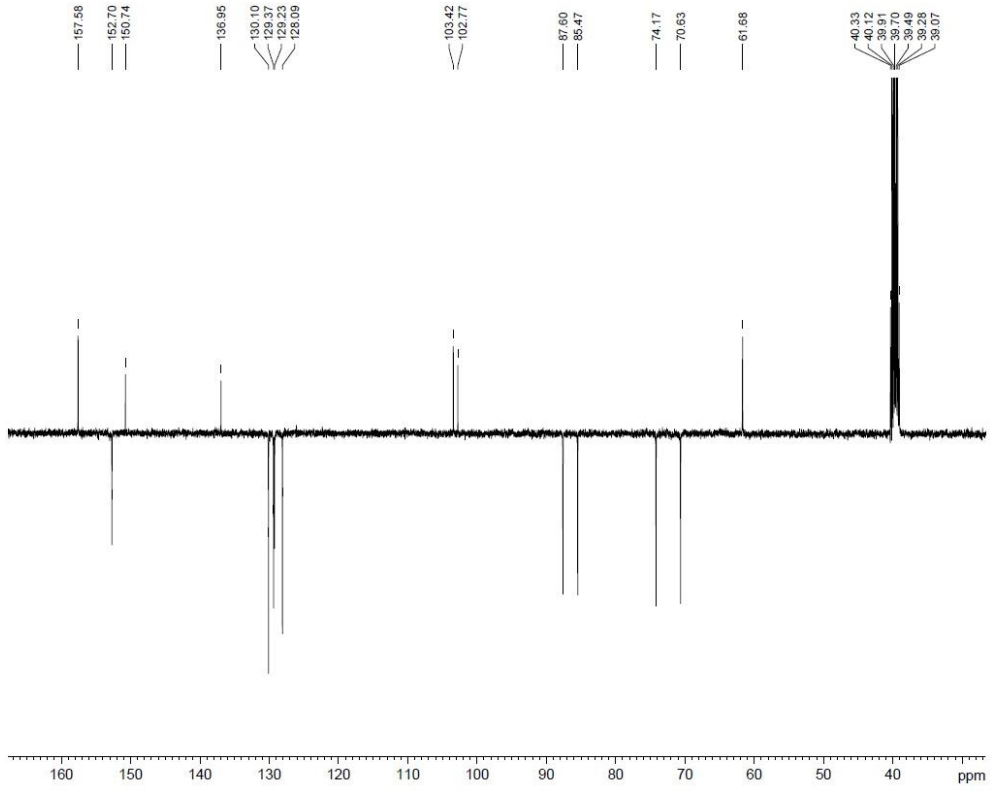
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm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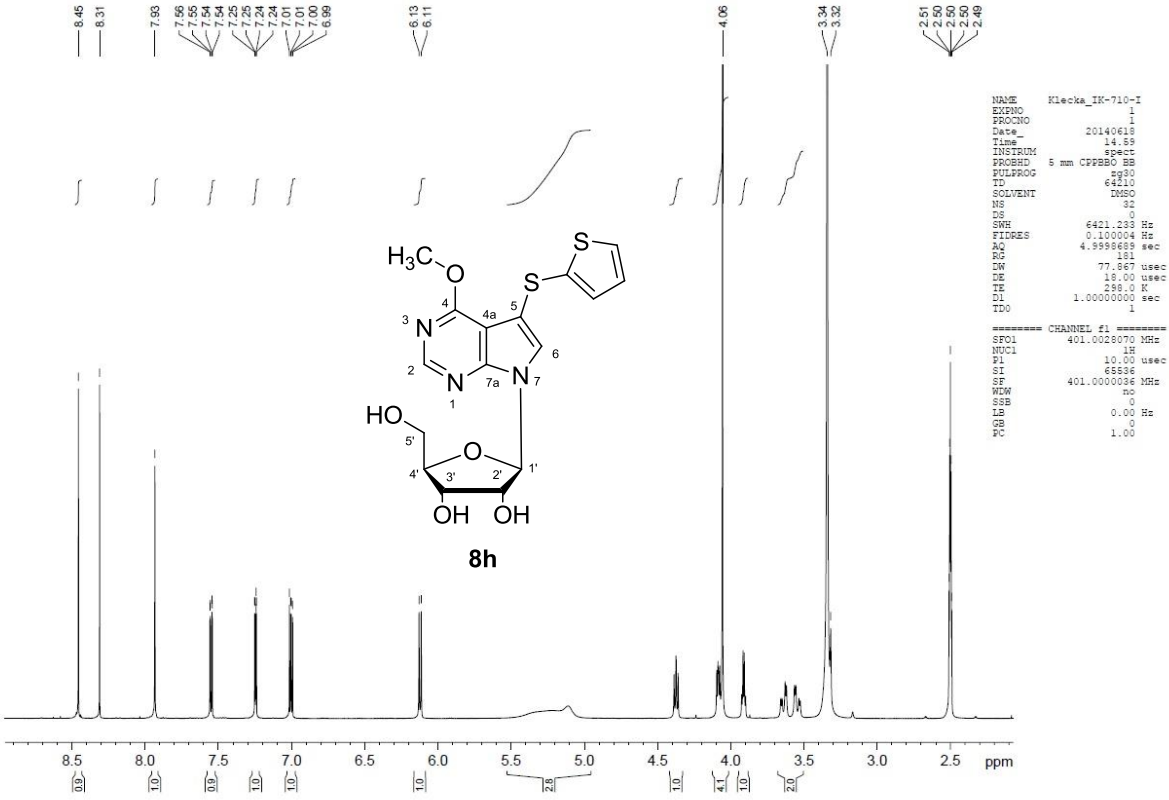
129.37
129.23
128.09
103.42
102.77
87.60
85.47
74.17
70.63
61.68
40.33
40.12
39.91
39.70
39.49
39.28
39.07
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm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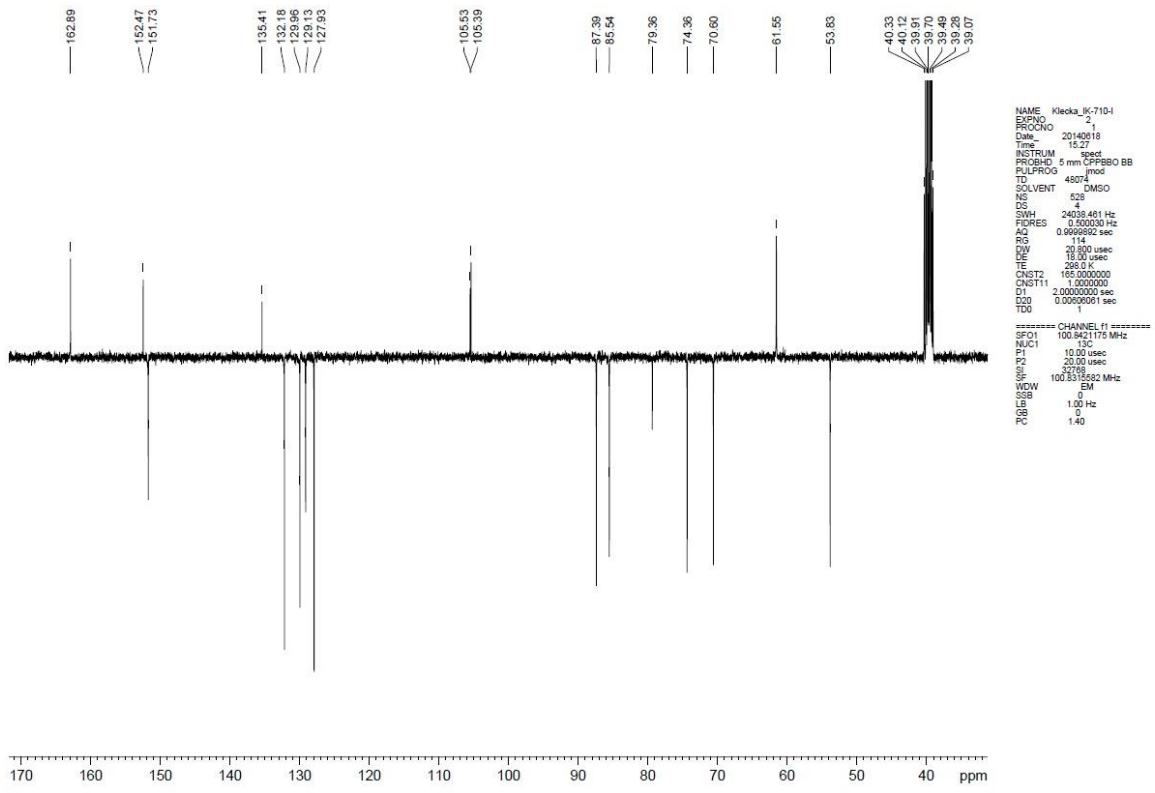
7.66
7.55
7.54
7.54
7.25
7.25
7.24
7.24
7.01
7.01
7.00
6.99
6.13
6.11
4.06
3.34
3.32
2.51
2.50
2.50
2.50
2.50
2.49
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm

H3C
O
S
S
4
4a
5
3
N
6
2
N
7a
N
7
1
HO
5'
O
4'
3'
2'
1'
OH
OH

8h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151.73
135.41
132.18
129.96
129.13
127.93
105.53
105.39
87.39
85.54
79.36
74.36
70.60
61.55
53.83
40.33
40.12
39.91
39.70
39.49
39.28
39.07
170
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm