Electronic Supporting Information

for

Direct C-H amination and C-H chloroamination of 7-deazapurines

Nazarii Sabat,[a,b] Martin Klečka,[a,b] Lenka Slavětínská,[b] Blanka Klepetářová[b] and Michal Hocek[a,b]*

[a] Department of Organic Chemistry, Faculty of Science, Charles University in Prague, Hlavova 8, CZ-12843 Prague 2, Czech Republic.

[b] Institute of Organic Chemistry and Biochemistry, Academy of Sciences of the Czech Republic, Gilead & IOCB Research Center, Flemingovo nam. 2, CZ-16610 Prague 6, Czech Republic

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1. Experimental section

General

6-Chloro-7-deazapurine, boronic acid, arylsulfochlorides were purchased from commercial suppliers and used without any further purification. Dry solvents 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. ^{1}H and ^{13}C resonances were assigned using H,C-HSQC and H,C-HMBC spectra. The samples were measured in $CDCl_3$ and chemical shifts (in ppm, δ-scale) were referenced to solvent signal (δ (^{1}H) = 7.26 ppm, δ (^{1}H) = 77.0 ppm) or in $(CD_3)_2CO$ (δ (^{1}H) = 2.05 ppm, δ (^{1}H) = 29.8 ppm) Coupling constants (*J*) are given in Hz. All compounds were isolated in pure form and crystallized. Any unassigned peaks in NMR spectra come either from minor traces of solvents used for crystallization (hexane and/or ethyl acetate in the spectra compounds **5a**, **6a**, **7a** and **7b**) or are inherent signals of deuterated solvents used for NMR (acetone and water in the spectra of compound **10a**). IR spectra were recorded on Nicolet Avatar 370 FT-IR using KBr method. Wavenumbers are given in cm^{-}1. 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 Kofler block and are uncorrected. X-ray diffraction experiment of single crystals was carried out on an Xcalibur X-ray diffractometer by monochromatized CuK_α radiation (λ=1.54180 Å) at 180 K.

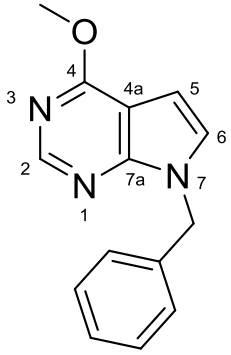
1.1. Preparation of starting compounds:

Compounds **1a**, **1d**, **1e** were synthetized according the previously published methods.[1]

Compounds **2-4** were prepared according the published protocol[2] from *N*-methyl-arylsulfonamides.[3]

7-Benzyl-4-methoxy-7H-pyrrolo[2,3-d]pyrimidine

(6-methoxy-9-benzyl-7-deazapurine) (1b)

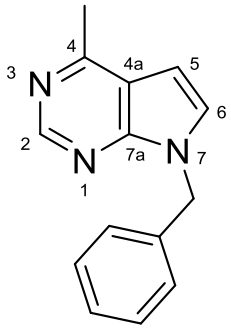


6-Chloro-9-benzyl-7-deazapurine (729 mg, 3 mmol) dissolved in methanol (9 ml) and then NaOMe 25 % (~4.4 M) solution in methanol was added dropwise and the mixture was stirred for 3 h at r.t., then quenched with H_2O (9 mL), extracted with EtOAc (20 ml), dried over anhydrous sodium sulphate, filtered and evaporated under reduced pressure. Product was isolated as a white solid (705 mg, 98 %).

M.p. 78-79 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 4.13 (s, 3H, CH_3O); 5.43 (s, 2H, CH_2-Ph); 6.55 (d, 1H, $J_{5,6}$ = 3.5 Hz, H-5); 7.00 (d, 1H, $J_{6,5}$ = 3.5 Hz, H-6); 7.19 (m, 2H, H-*o*-Bn); 7.25 – 7.34 (m, 3H, H-*m*,*p*-Bn); 8.51 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 48.19 (CH_2-Ph); 53.62 (CH_3O); 98.78 (CH-5); 105.37 (C-4a); 125.71 (CH-6); 127.45 (CH-*o*-Bn); 127.83 (CH-*p*-Bn); 128.79 (CH-*m*-Bn); 137.08 (C-*i*-Bn); 151.05 (CH-2); 151.94 (C-7a); 163.10 (C-4). IR(KBr): 3120, 3093, 1582, 1459, 1256, 1026, 698. HRMS (ESI) calculated for $C_{14}H_{14}ON_3$: 240.1131; found 240.1131.

7-Benzyl-4-methyl-7H-pyrrolo[2,3-d]pyrimidine

(6-methyl-9-benzyl-7-deazapurine) (1c)



6-Chloro-9-benzyl-7-deazapurine (972 mg, 4 mmol) $Pd(PPh_3)_4$ (0,2 mmol) were placed in an argon-purged vial and then THF (80 mL) was added. To this stirred reaction mixture, Me_3Al (2M solution in toluene, 4 mL, 8 mmol) was added dropwise at r.t.. The mixture was then stirred at 75 ° C for 8 h. After cooling to r.t., the reaction mixture was poured onto a mixture of H_2O (400 mL), NH_4Cl (4 g) and Na_2EDTA (1 g) and then extracted with chloroform (3 × 400 mL). The collected organic layers were dried with anhydrous Mg_2SO_4, filtered and evaporated under reduced pressure. The crude product was purified by column chromatography on silica gel, eluting with hexanes/EtOAc 5:1 to 1:2. Product was isolated as orange oil (760 mg, 85 %).

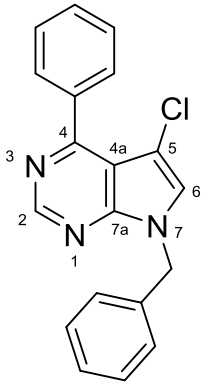
^{1}H NMR (500 MHz, $CDCl_3$): 2.75 (s, 3H, CH_3); 5.46 (s, 2H, CH_2-Ph); 6.57 (d, 1H, $J_{5,6}$ = 3.6 Hz, H-5); 7.13 (d, 1H, $J_{6,5}$ = 3.6 Hz, H-5); 7.21 (m, 2H, H-*o*-Bn); 7.27 – 7.35 (m, 3H, H-*m*,*p*-Bn); 8.80 (s, 1H, H-2). ^{13}C NMR (150.9 MHz, $CDCl_3$): 21.43 (CH_3); 47.92 (CH_2-Ph); 99.70 (CH-5); 117.80 (C-4a); 127.55 (CH-*o*-Bn); 127.63 (CH-6); 127.94 (CH-*p*-Bn); 128.85 (CH-*m*-Bn); 136.88 (C-*i*-Bn); 150.35 (C-7a); 151.40 (CH-2); 159.31 (C-4). IR(KBr): 3120, 3090, 1577, 1452, 1260, 1060, 630. HRMS (ESI) calculated for $C_{14}H_{14}N_3$: 224.1191; found 224.1191.

Chlorination of 6-phenyl-9-benzyl-7-deazapurine.

Method A: A mixture of 6-phenyl-7-deazapurine **1a** (285 mg, 1 mmol) and NCS (141 mg, 1.05 mmol) in DMF (1.5 mL) was stirred at r.t. for 90 h and then the mixture was evaporated under vacuum. The residue was purified by column chromatography on silica gel eluting with hexanes/EtOAc 5:1 to 2:1 to give product **9a** (272 mg, 85 %) as a colorless solid.

Method B: A mixture of 6-phenyl-7-deazapurine **1a** (285 mg, 1 mmol) and **4** (376, 1.5 mmol) in 1,4-dioxane (4 mL) was stirred at r.t. for 45 h and then evaporated under vacuum. The residue was purified by column chromatography on silica gel eluting with hexanes/EtOAc 5:1 to 2:1 to give product **9a** (250 mg, 78 %) as a colourless solid.

7-Benzyl-5-chloro-4-phenyl-7H-pyrrolo[2,3-d]pyrimidine
(6-phenyl-7-chloro-9-benzyl-7-deazapurine) (9a)



M.p. 117-118 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 5.49 (s, 2H, CH_2-Ph); 7.19 (s, 1H, H-6); 7.29 (m, 2H, H-*o*-Bn); 7.30 – 7.39 (m, 3H, H-*m*,*p*-Bn); 7.49 – 7.54 (m, 3H, H-*m*,*p*-Ph); 7.83 (m, 2H, H-*o*-Ph); 9.00 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 48.05 (CH_2-Ph); 104.27 (C-5); 113.14 (C-4a); 125.88 (C-6); 127.86 and 127.87 (CH-*o*-Bn, CH-*m*-Ph); 128.30 (CH-*p*-Bn); 129.00 (CH-*m*-Bn); 129.72 (CH-*p*-Ph); 130.30 (CH-*o*-Ph); 136.09 (C-*i*-Bn); 136.71 (C-*i*-Ph); 150.39 (C-7a); 151.92 (CH-2); 159.70 (C-4). IR(KBr): 3099, 3058, 1550, 1465, 1145, 976, 704. HRMS (ESI) calculated for $C_{19}H_{15}N_3Cl$: 320.0950; found 320.0949.

General procedures for C-H amination and C-H chloroamination of 7-deazapurines:

General procedure for C-H amination of 7-deazapurines

7-Deazapurine **1a-1e** (0.5 mmol), $Pd(OAc)_2$ (0.025 mmol), $Cu(acac)_2$ (0.05 mmol), bpy (0.05 mmol), Na_2CO_3 (3.5 mmol) and chlorosulfonamide (1-1,75 mmol) were placed in a vial which was purged with argon. Then 1,4-dioxane (2 mL) was added and the reaction mixture was stirred for 24 h at r.t., quenched with H_2O (2 mL), extracted with ethyl acetate (3 x 20 mL) and washed with brine (2 mL). The organic phases were combined and dried over sodium sulphate, filtered, and evaporated under vacuum. The crude product was purified by column chromatography on silica gel, eluting with hexanes/EtOAc (5:1 to 1:2) to afford the corresponding product.

General procedure for C-H chloroamination of 7-deazapurines

7-Deazapurine **1a-1e** (0.5 mmol), $Pd(OAc)_2$ (0.0125 mmol), CuCl (0.05 mmol), LiCl (1.0 mmol), Ag_2CO_3 (1.0 mmol) and chlorosulfonamide (1.5-1.75 mmol) were placed in a vial which was purged with argon. Then 1,4-dioxane (2 mL) was added and the reaction mixture was stirred for 24 h at r.t., quenched with H_2O (2 mL), extracted with ethyl acetate (3 x 20 mL) and washed with brine (2 mL). The organic phases were combined and dried over sodium sulphate, filtered, and evaporated under vacuum. The crude product was purified by column chromatography on silica gel, eluting with hexanes/EtOAc (5:1 to 1:2) to afford the corresponding product.

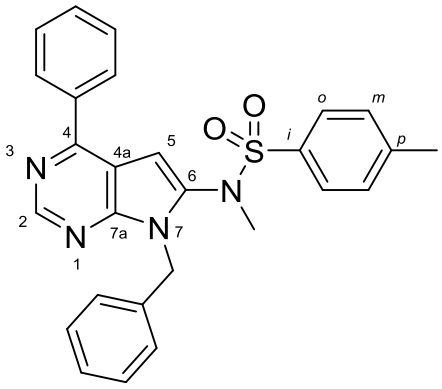
Table S1. Optimization for Palladium-Copper catalyzed C-H amination and C-H chloroamination of 6-phenyl-9-benzyl-7-deazapurine (**1a**) with *N*-chloro-*N*-methyl-2-nitrobenzenesulfonamide (**4**)

Entry	**4** (equiv.)	$Pd(OAc)_2$	Cu source (equiv.)	Additive (equiv.)	Base, (equiv.)	NMR conversion, (%)	
						7a	**8a**
1	1.5	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Na_2CO_3 (2)	10	-
2	2	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Na_2CO_3 (2)	15	-
3[a]	2	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Na_2CO_3 (2)	12	-
4	2	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Na_2CO_3 (5)	32	-
5	2	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Cs_2CO_3 (2)	17	-
6	2	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Cs_2CO_3 (5)	22	-
7	2	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	LiOtBu (5)	16	-
8	3.5	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Na_2CO_3 (5)	53	12
9	5	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Na_2CO_3 (5)	30	36
10	2	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Na_2CO_3 (10)	38	-
11	2	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Na_2CO_3 (7)	42	-
12	3	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Na_2CO_3 (7)	62	-
13	**3.5**	**5 %**	**$Cu(acac)_2$ (0.1)**	**bpy (0.1)**	**Na_2CO_3 (7)**	**65**	**-**
14	5	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Na_2CO_3 (7)	34	33
15[b]	3.5	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Na_2CO_3 (7)	40	-
16[c]	3.5	5 %	$Cu(acac)_2$ (0.1)	bpy (0.1)	Na_2CO_3 (7)	25	35
17	3.5	5 %	$Cu(acac)_2$ (0.1)	dtbpy (0.1)	Na_2CO_3 (7)	46	-
18	3.5	7.5 %	$Cu(acac)_2$ (0.15)	bpy (0.15)	Na_2CO_3 (7)	49	-
19	3.5	5 %	-	bpy (0.1)	Na_2CO_3 (7)	19	16
20	3.5	-	$Cu(acac)_2$ (0.1)	bpy (0.1)	Na_2CO_3 (7)	48	-
21	3.5	-	$Cu(acac)_2$ (0.2)	bpy (0.2)	Na_2CO_3 (7)	45	-
22	3.5	-	$Cu(acac)_2$ (0.4)	bpy (0.4)	Na_2CO_3 (7)	42	-
23	3.5	5	$Cu(acac)_2$ (0.2)	bpy (0.2)	Na_2CO_3 (7)	63	-
24	3.5	5	$Cu(acac)_2$ (0.4)	bpy (0.4)	Na_2CO_3 (7)	48	-
25	3.5	5 %	$Cu(acac)_2$ (0.1)	-	Na_2CO_3 (2)	31	19
26	3.5	5 %	$Cu(acac)_2$ (0.1)	-	Ag_2CO_3 (2)	28	23
27	3.5	5 %	CuCl (0.1)	-	Ag_2CO_3 (2)	12	33
28	3	2.5 %	CuCl (0.1)	-	Ag_2CO_3 (2)	14	38
29	3.5	2.5 %	CuCl (0.1)	-	Ag_2CO_3 (2)	10	39
30	3.5	2.5 %	CuCl (0.1)	LiCl (1)	Ag_2CO_3 (2)	6	47
31	**3.5**	**2.5 %**	**CuCl (0.1)**	**LiCl (2)**	**Ag_2CO_3 (2)**	**-**	**53**
32	3.5	-	CuCl (0.1)	LiCl (2)	Ag_2CO_3 (2)	13	45
33	3.5	-	CuCl (0.2)	LiCl (2)	Ag_2CO_3 (2)	17	40
34	3.5	2.5 %	CuCl (0.2)	LiCl (2)	Ag_2CO_3 (2)	8	50

[a] 70 °C. [b] in THF. [c] in toluene.

***N*-(7-Benzyl-4-phenyl-7H-pyrrolo[2,3-*d*]pyrimidin-6-yl)-*N*-methyl-4-methylbenzenesulfonamide (5a)**

6-phenyl-8-[*N*-(4-methylbenzenesulfonyl)-*N*-(methyl)amino]-9-benzyl-7-deazapurine (5a)

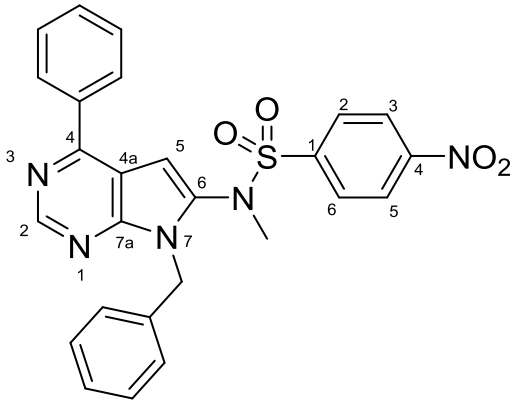


6-Phenyl-9-benzyl-7-deazapurine **1a** (285 mg, 1 mmol) and *N*-chloro-*N*-methyl-4-methylbenzenesulfonamide **2** (1098 mg, 5.0 mmol) were used as starting compounds to give product **5a** (334 mg, 68 %) as white needles after chromatography with hexanes/EtOAc 5:1 to 1:1 and crystallization from EtOAc/hexane.

M.p. 226-227 °C. ^{1}H NMR (500.0 MHz, $CDCl_3$): 2.51 (s 3H, CH_3-Ts); 2.78 (s, 3H, CH_3N); 5.73 (bs, 2H, CH_2Ph); 6.04 (s, 1H, H-5); 7.23-7.33 (m, 5H, H-*o*,*m*,*p*-Bn); 7.38 (m, 2H, H-*m*-Ts); 7.46-7.51 (m, 3H, H-*m*,*p*-Ph); 7.66 (m, 2H, H-*o*-Ts); 7.92 (m, 2H, H-*o*-Ph); 9.06 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 21.67 (CH_3-Ts); 39.77 (CH_3N); 45.25 (CH_2Ph); 96.99 (CH-5); 114.12 (C-4a); 127.65 (CH-*p*-Bn); 127.90 (CH-*o*-Bn); 128.57 (CH-*o*-Ts); 128.68 (CH-*m*-Bn); 128.76 (CH-*o*,*m*-Ph); 129.57 (CH-*m*-Ts); 130.20 (CH-*p*-Ph); 132.67 (C-*i*-Ts); 136.84 (C-*i*-Bn); 137.16 (C-*i*-Ph); 138.65 (C-6); 144.73 (C-*p*-Ts); 150.20 (C-7a); 152.45 (CH-2); 157.37 (C-4). IR(KBr): 2976, 2930, 2817, 1470, 1382, 1355, 1341, 1322, 1186, 1164, 1314, 852, 823, 691. HRMS (ESI) calculated for $C_{27}H_{24}N_4O_2S$: 469.1693; found 469.1693.

***N*-(7-benzyl-4-phenyl-7*H*-pyrrolo[2,3-*d*]pyrimidin-6-yl)-*N*-methyl-4-nitrobenzenesulfonamide (6a)**

9-benzyl-8-[*N*-(4-nitrophenylsulfonyl)-*N*-(methyl)amino]-6-phenyl-7-deazapurine (6a)

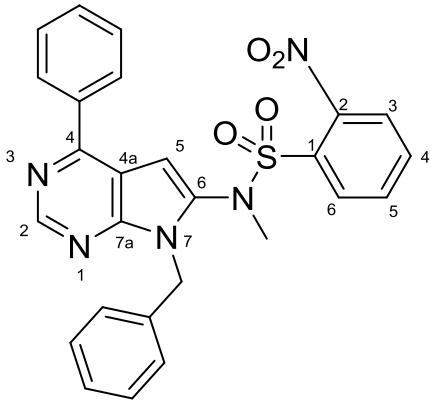


1a (285 mg, 1 mmol) and *N*-chloro-*N*-methyl-2-nitrobenzenesulfonamide **3** (877 mg, 3.0 mmol) were used as starting compounds to give product **6a** (235 mg, 47 %) as yellowish needles after chromatography with hexanes/EtOAc 5:1 to 1:1 and crystallization from EtOAc/hexane.

M.p. 231-232 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 2.97 (s, 3H, CH_3N); 5.74 (bs, 2H, CH_2-Ph); 6.04 (s, 1H, H-5); 7.28 (m, 2H, H-*o*-Bn); 7.28 – 7.35 (m, 3H, H-*p*,*m*-Bn); 7.46 – 7.52 (m, 3H, H-*m*,*p*-Ph); 7.90 (m, 2H, H-*o*-Ph); 7.97 (m, 2H, H-*o*-$C_6H_4NO_2$); 7.41 (m, 2H, H-*m*-$C_6H_4NO_2$); 9.08 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 39.91 (CH_3-N); 45.34 (CH_2-Ph); 97.21 (CH-5); 113.95 (C-4a); 124.21 (CH-*m*-$C_6H_4NO_2$); 127.86 (CH-*o*,*p*-Bn); 128.61(CH-*o*-Ph); 128.81 (CH-*m*-Bn); 128.92 (CH-*m*-Ph); 129.64 (CH-*o*-$C_6H_4NO_2$); 130.46 (CH-*p*-Ph); 136.85 (C-*i*-Bn); 137.24 (C-6); 137.48 (C-*i*-Ph); 141.52 (C-*i*-$C_6H_4NO_2$); 150.37 (C-7a); 150.67 (C-*p*-$C_6H_4NO_2$); 152.98 (CH-2); 158.02 (C-4).IR(KBr): 2825, 1537, 1374, 1366, 1362, 1340, 1317, 1305, 1177, 1158, 921. HRMS (ESI) calculated for $C_{26}H_{22}N_5O_4S$: 500.1386; found 500.1387.

***N*-(7-benzyl-4-phenyl-7*H*-pyrrolo[2,3-*d*]pyrimidin-6-yl)-*N*-methyl-2-nitrobenzenesulfonamide**

9-benzyl-8-[*N*-(2-nitrophenylsulfonyl)-*N*-(methyl)amino]-6-phenyl-7-deazapurine (7a)

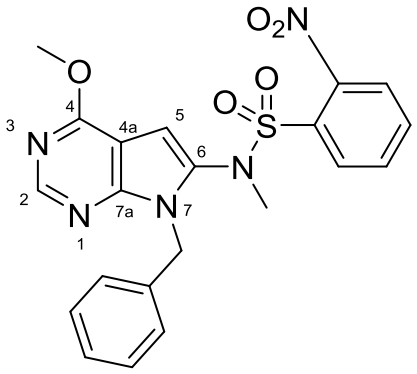


1a (285 mg, 1 mmol) and *N*-chloro-*N*-methyl-2-nitrobenzenesulfonamide **4** (877 mg, 3.5 mmol) were used as starting compounds to give product **7a** (310 mg, 62 %) as colourless crystals after chromatography with hexanes/EtOAc 5:1 to 1:1 and crystallization from EtOAc/hexane.

M.p. 102-103 °C. ^{1}H NMR (500.0 MHz, $CDCl_3$): 2.94 (s, 3H, CH_3N); 5.67 (bs, 2H, CH_2Ph); 6.45 (s, 1H, H-5); 7.22 (m, 2H, H-*o*-Bn); 7.27 (m, 1H, H-*p*-Bn); 7.30 (m, 2H, H-*m*-Bn); 7.50-7.53 (m, 3H, H-*m*,*p*-Ph); 7.61 (ddd, 1H, $J_{5,6}$ = 8.1, $J_{5,4}$ = 7.5, $J_{5,3}$ = 1.3, H-5-$C_6H_4NO_2$); 7.67 (ddd, 1H, $J_{3,4}$ = 8.0, $J_{3,5}$ = 1.3, $J_{3,6}$ = 0.5, H-3-$C_6H_4NO_2$); 7.76 (ddd, 1H, $J_{6,5}$ = 8.1, $J_{6,4}$ = 1.4, $J_{6,3}$ = 0.5, H-6-$C_6H_4NO_2$); 7.77 (ddd, 1H, $J_{4,3}$ = 8.0, $J_{4,5}$ = 7.5, $J_{4,6}$ = 1.4, H-4-$C_6H_4NO_2$); 7.97 (m, 2H, H-*o*-Ph); 9.10 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 40.53 (CH_3N); 45.29 (CH_2Ph); 96.40 (CH-5); 113.91 (C-4a); 124.12 (CH-3-$C_6H_4NO_2$); 127.65 (CH-*o*-Bn); 127.89 (CH-*p*-Bn); 128.85 (CH-*m*-Ph, CH-*o*-Bn); 128.94 (CH-*m*-Ph); 130.16 (C-1-$C_6H_4NO_2$); 130.64 (CH-*p*-Ph); 131.26 (CH-5-$C_6H_4NO_2$); 132.23 (CH-6-$C_6H_4NO_2$); 134.62 (CH-4-$C_6H_4NO_2$); 136.54 (C-*i*-Bn); 136.86 (C-*i*-Ph); 137.04 (C-6); 148.55 (C-2-$C_6H_4NO_2$); 150.56 (C-7a); 152.40 (CH-2); 157.48 (C-4). IR(KBr): 2821, 1545, 1376, 1368, 1360, 1343, 1318, 1309, 1180, 1163, 924. HRMS (ESI) calculated for $C_{26}H_{22}N_5O_4S$: 500.1387; found 500.1387.

***N*-(7-benzyl-4-methoxy-7*H*-pyrrolo[2,3-*d*]pyrimidin-6-yl)-*N*-methyl-2-nitrobenzenesulfonamide**

9-benzyl-6-methoxy-8-[*N*-(2-nitrophenylsulfonyl)-*N*-(methyl)amino]-7-deazapurine (7b)

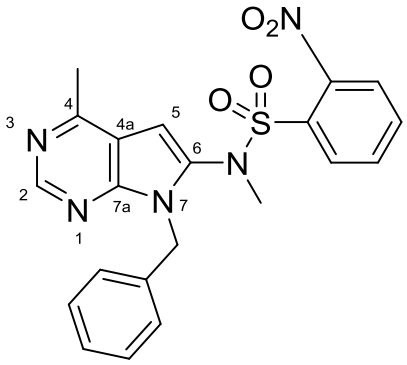


6-methoxy-9-Bn-7-deazapurine **1b** (240 mg, 1 mmol) and *N*-chloro-*N*-methyl-2-nitrobenzenesulfonamide **4** (501 mg, 2 mmol) were used as starting compounds to give product **7b** (274 mg, 60 %) as white needles after chromatography with hexanes/EtOAc 5:1 to 1:1 and crystallization from EtOAc/hexane.

M.p. 219-220 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 2.88 (s, 3H, CH_3N); 4.09 (s, 3H, CH_3O); 5.59 (bs, 2H, CH_2-Ph); 6.13 (s, 1H, H-5); 7.17 (m, 2H, H-*o*-Bn); 7.21 - 7.30 (m, 3H, H-*m*,*p*-Bn); 7.61 (ddd, 1H, $J_{5,6}$ = 8.0 Hz, $J_{5,4}$ = 7.4 Hz, $J_{5,3}$ = 1.3 Hz, H-5-$C_6H_4NO_2$); 7.64 (bdd, 1H, $J_{3,4}$ = 8.0 Hz, $J_{3,5}$ = 1.3 Hz, H-3-$C_6H_4NO_2$); 7.71 (bdd, 1H, $J_{6,5}$ = 8.0 Hz, $J_{6,4}$ = 1.4 Hz, H-6-$C_6H_4NO_2$); 7.75 (ddd, 1H, (ddd, 1H, $J_{4,3}$ = 8.0 Hz, $J_{4,5}$ = 7.4 Hz, $J_{4,6}$ = 1.4 Hz, H-4-$C_6H_4NO_2$); 8.57 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 40.61 (CH_3-N); 45.32 (CH_2-Ph); 53.77 (CH_3-O); 97.07 (CH-5); 103.94 (C-4a); 123.96 (CH-3-$C_6H_4NO_2$); 127.55 (CH-*o*-Bn); 127.68 (CH-*p*-Bn); 128.74 (CH-*m*-Bn); 130.29 (C-1-$C_6H_4NO_2$); 131.26 (CH-5-$C_6H_4NO_2$); 132.22 (CH-6-$C_6H_4NO_2$); 133.50 (C-6); 134.39 (CH-4-$C_6H_4NO_2$); 136.99 (C-*i*-Bn); 148.57 (C-2-$C_6H_4NO_2$); 150.54 (C-7a); 152.42 (CH-2); 163.04 (C-4). IR(KBr): 3090, 1580, 1549, 1377, 1352, 1262, 1160, 1030, 824, 516. HRMS (ESI) calculated for $C_{21}H_{20}N_5O_5S$: 454.1180; found 454.1179.

N-(7-benzyl-4-methyl-7H-pyrrolo[2,3-d]pyrimidin-6-yl)-N-methyl-2-nitrobenzenesulfonamide

9-benzyl-6-methyl-8-[*N*-(2-nitrophenylsulfonyl)-*N*-(methyl)amino]-7-deazapurine (7c)

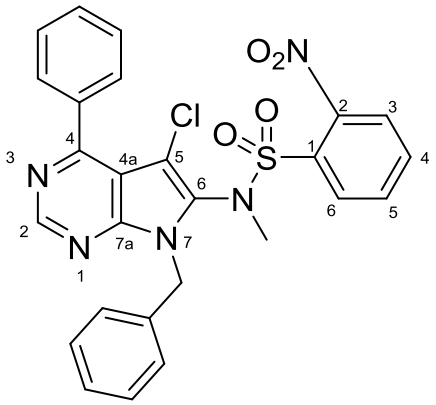


6-methyl-9-Bn-7-deazapurine **1c** (223 mg, 1 mmol) and *N*-chloro-*N*-methyl-2-nitrobenzenesulfonamide **4** (501 mg, 2 mmol) were used as starting compounds to give product **7c** (180 mg, 41 %) as yellowish crystals after chromatography with hexanes/EtOAc 5:1 to 1:1 and crystallization from EtOAc/hexane.

M.p. 186-187 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 2.65 (s, 3H, CH_3-4); 2.92 (s, 3H, CH_3N); 5.59 (bs, 2H, CH_2-Ph); 6.18 (s, 1H, H-5); 7.17 (m, 2H, H-*o*-Bn); 7.21 – 7.30 (m, 3H, H-*p*,*m*-Bn); 7.61 (ddd, 1H, $J_{5,6}$ = 8.0 Hz, $J_{5,4}$ = 7.4 Hz, $J_{5,3}$ = 1.3 Hz, H-5-$C_6H_4NO_2$); 7.66 (dd, 1H, $J_{3,4}$ = 8.0 Hz, $J_{3,5}$ = 1.3 Hz, H-3-$C_6H_4NO_2$); 7.72 (dd, 1H, $J_{6,5}$ = 8.0 Hz, $J_{6,4}$ = 1.4 Hz, H-6-$C_6H_4NO_2$); 7.76 (ddd, 1H, $J_{4,3}$ = 8.0 Hz, $J_{4,5}$ = 7.4 Hz, $J_{4,6}$ = 1.4 Hz, H-4-$C_6H_4NO_2$); 8.87 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 21.48 (CH_3-4); 40.57 (CH_3-N); 45.06 (CH_2-Ph); 97.91 (CH-5); 116.18 (C-4a); 124.07 (CH-3-$C_6H_4NO_2$); 127.58 (CH-*o*-Bn); 127.77 (CH-*p*-Bn); 128.79 (CH-*m*-Bn); 130.38 (C-1-$C_6H_4NO_2$); 131.22 (CH-5-$C_6H_4NO_2$); 132.24 (CH-6-$C_6H_4NO_2$); 134.51 (CH-4-$C_6H_4NO_2$); 135.44 (C-6); 136.80 (C-*i*-Bn); 148.56 (C-2-$C_6H_4NO_2$); 149.15 (C-7a); 152.80 (CH-2); 159.85 (C-4).IR(KBr): 3063, 1891, 1550, 1377, 1359, 1237, 1201, 1069, 1165, 893, 600. HRMS (ESI) calculated for $C_{21}H_{20}N_5O_4S$: 438.1232; found 438.1230.

***N*-(7-benzyl-5-chloro-4-phenyl-7*H*-pyrrolo[2,3-*d*]pyrimidin-6-yl)-*N*-methyl-2-nitrobenzenesulfonamide**

9-benzyl-7-chloro-8-[*N*-(2-nitrophenylsulfonyl)-*N*-(methyl)amino]-6-phenyl-7-deazapurine (8a)



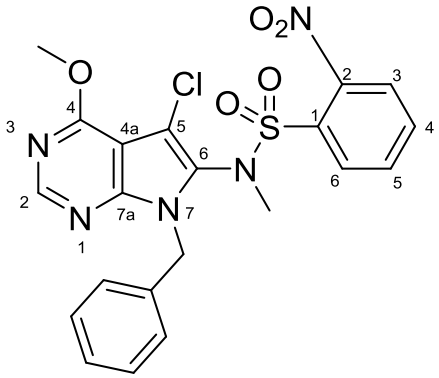
Method A, C-H chloroamination: **1a** (285 mg, 1 mmol) and **4** (877 mg, 3.5 mmol) were used as starting compounds to give product **8a** (273 mg, 51 %) as white crystals after chromatography with hexanes/EtOAc 5:1 to 1:1 and crystallization from EtOAc/hexane.

Method B, C-H amination: **9a** (285 mg, 1 mmol) and **4** (752 mg, 3.0 mmol) were used as starting compounds to give product **8a** (225 mg, 41 %) as white crystals after chromatography with hexanes/EtOAc 5:1 to 1:1 and crystallization from EtOAc/hexane.

M.p. 215-216 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 2.91 (s, 3H, CH_3N); 5.42 (d, 1H, J_{gem} = 15.3 Hz, CH_2a-Ph); 6.16 (d, 1H, J_{gem} = 15.3 Hz, CH_2b-Ph); 7.26 – 7.31 (m, 3H, H-*o*,*p*-Bn); 7.33 (m, 2H, H-*m*-Bn); 7.42 – 7.50 (m, 3H, H-*m*,*p*-Ph); 7.58 – 7.63 (m, 2H, H-3,5-$C_6H_4NO_2$); 7.71 (m, 2H, H-*o*-Ph); 7.73 (ddd, 1H, $J_{4,3}$ = $J_{4,5}$ = 7.7 Hz, $J_{4,6}$ = 1.4 Hz, H-4-$C_6H_4NO_2$); 7.84 (m, 1H, H-6-$C_6H_4NO_2$); 9.09 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 38.28 (CH_3-N); 45.68 (CH_2-Ph); 103.08 (C-5); 112.02 (C-4a); 124.00 (CH-3-$C_6H_4NO_2$); 127.84 (CH-*m*-Ph); 128.09 (CH-*p*-Bn); 128.11 (CH-*o*-Bn); 128.95 (CH-*m*-Bn); 129.86 (CH-*p*-Ph); 130.20 (CH-*o*-Ph); 131.45 (CH-5-$C_6H_4NO_2$); 131.62 (C-1-$C_6H_4NO_2$); 131.65 (CH-6-$C_6H_4NO_2$); 131.89 (C-6); 134.49 (CH-4-$C_6H_4NO_2$); 136.48 (C-*i*-Bn); 136.62 (C-*i*-Ph); 148.56 (C-2-$C_6H_4NO_2$); 148.78 (C-7a); 153.18 (CH-2); 160.20 (C-4). IR(KBr): 3050, 1583, 1545, 1374, 1345, 1165, 826, 558. HRMS (ESI) calculated for $C_{26}H_{21}N_5O_4SCl$: 534.0998; found: 534.0997.

***N*-(7-benzyl-5-chloro-4-methoxy-7*H*-pyrrolo[2,3-*d*]pyrimidin-6-yl)-*N*-methyl-2-nitrobenzenesulfonamide**

9-benzyl-7-chloro-6-methoxy-8-[*N*-(2-nitrophenylsulfonyl)-*N*-(methyl)amino]-7-deazapurine (8b)



1b (240 mg, 1 mmol) and **4** (752 mg, 3.5 mmol) were used as starting compounds to give product **8b** (205 mg, 42 %) as white crystals after chromatography with hexanes/EtOAc 5:1 to 1:1 and crystallization from EtOAc/hexane.

M.p. 177-179 °C. ^{1}H NMR (500 MHz, $CDCl_3$): 2.87 (s, 3H, CH_3N); 4.11 (s, 3H, CH_3O); 5.36 (d, 1H, J_{gem} = 15.4 Hz, CH_2a-Ph); 5.99 (d, 1H, J_{gem} = 15.4 Hz, CH_2b-Ph); 7.21 (m, 2H, H-*o*-Bn); 7.24 – 7.33 (m, 3H, H-*p*,*m*-Bn); 7.61 (m, 1H, H-3-$C_6H_4NO_2$); 7.62 (m, 1H, H-5-$C_6H_4NO_2$); 7.74 (bt, 1H, $J_{4,3}$ = $J_{4,5}$ = 7.8 Hz, H-4-$C_6H_4NO_2$); 7.81 (bd, 1H, $J_{6,5}$ = 7.9 Hz, H-6-$C_6H_4NO_2$); 8.57 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, $CDCl_3$): 38.30 (CH_3-N); 45.74 (CH_2-Ph); 54.07 (CH_3O); 102.24 (C-4a); 102.49 (C-5); 123.91 (CH-3-$C_6H_4NO_2$); 127.89 (CH-*o*-Bn); 127.94 (CH-*p*-Bn); 128.71 (C-6); 128.85 (CH-*m*-Bn); 131.51 (C-1-$C_6H_4NO_2$); 131.69 (CH-5-$C_6H_4NO_2$); 131.76 (CH-6-$C_6H_4NO_2$); 134.38 (CH-4-$C_6H_4NO_2$); 136.70 (C-*i*-Bn); 148.40 (C-2-$C_6H_4NO_2$); 148.66 (C-7a); 153.06 (CH-2); 163.08 (C-4). IR(KBr): 3068, 1580, 1374, 1352, 1262, 1160, 1030, 853, 517. HRMS (ESI) calculated for $C_{21}H_{19}N_5O_5SCl$: 488.0790; found 534.0789.

Deprotection of *N*-(7-benzyl-4-phenyl-7*H*-pyrrolo[2,3-*d*]pyrimidin-6-yl)-*N*-methyl-2-nitrobenzenesulfonamide (7a)

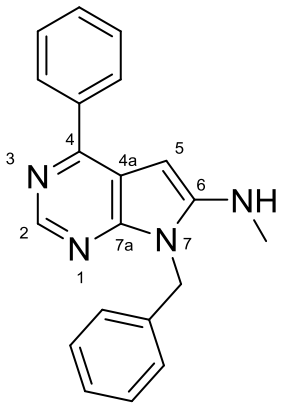
Compound **7a** (250 mg, 0.5 mmol) and Cs_2CO_3 (163 mg, 0.5 mmol) were dissolved in dry MeCN (4 mL) under Ar. Then, thiophenol (55 mg (0.051 ml), 0.5 mmol) was added dropwise to the stirred reaction mixture at r.t. and the stirring was continued for 1 h. Then the mixture was filtered and evaporated under vacuum. The residue was purified by column chromatography on silica gel eluting with hexanes/EtOAc 5:1 to 1:1 to get product **10a** 118 mg (75 %) as a yellow solid.

One pot C-H amination/deprotection

6-Phenyl-9-benzyl-7-deazapurine **1a** (285 mg, 1 mmol), $Pd(OAc)_2$ (0.05 mmol), $Cu(acac)_2$ (0.05 0.1 mmol), bpy (0.1 mmol), Na_2CO_3 (7 mmol) and *N*-chloro-*N*-methyl-2-nitrobenzenesulfonamide **4** (877 mg, 3.5 mmol) were placed in an argon-purged vial and then 1,4-dioxane (4 mL) was added. The reaction mixture was then stirred for 24 h at r.t., quenched with H_2O (4 mL), extracted with ethyl acetate (3 x 40 mL) and washed with brine (4 mL). The organic phases were combined and dried over sodium sulphate, filtered, and evaporated under vacuum. The crude intermediate was combined with Cs_2CO_3 (326 mg, 1 mmol) in an argon-purged vial and dissolved in dry MeCN (8 mL). Thiophenol (110 mg, 0.102 ml, 1 mmol) was added dropwise through septum to the stirred reaction mixture at r.t. and the stirring was continued for 1 h. The mixture was then quenched with H_2O (4 mL), extracted with ethyl acetate (3 x 40 mL) and washed with brine (4 mL). The organic phases were combined and dried over sodium sulphate, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel eluting with hexanes/EtOAc 5:1 to 1:1 to get product **10a** 110 mg (35 % in two steps) as a yellow solid.

7-Benzyl-*N*-methyl-4-phenyl-7*H*-pyrrolo[2,3-*d*]pyrimidin-6-amine

(9-benzyl-8-methylamino-6-phenyl-7-deazapurine) (10a)



M.p. 154-155 °C. ^{1}H NMR (500.0 MHz, acetone-d_6): 2.94 (d, 3H, J = 5.0, CH_3N); 5.48 (s, 2H, CH_2Ph); 5.68 (bq, 1H, J = 5.0, MeNH); 5.82 (d, 1H, J = 0.6, H-5); 7.20 (m, 2H, H-*o*-Bn); 7.24 (m, 1H, H-*p*-Bn); 7.30 (m, 2H, H-*m*-Bn); 7.44 (m, 1H, H-*p*-Ph); 7.53 (m, 2H, H-*m*-Ph); 8.25 (m, 2H, H-*o*-Ph); 8.57 (s, 1H, H-2). ^{13}C NMR (125.7 MHz, acetone-d_6): 30.97 (CH_3N); 43.94 (CH_2Ph); 74.20 (CH-5); 117.98 (C-4a); 127.65 (CH-*o*-Bn); 128.13 (CH-*p*-Bn); 129.00 (CH-*o*-Ph); 129.22 (CH-*m*-Ph); 129.37 (CH-*m*-Bn); 129.54 (CH-*p*-Ph); 138.14 (C-*i*-Bn); 140.35 (C-*i*-Ph); 148.33 (CH-2); 149.71 (C-4); 150.64 (C-6); 152.97 (C-7a). IR(KBr): 3416, 3220, 3061, 3031, 2820, 1604, 1583, 1570, 1495, 1452, 1344, 1181. HRMS (ESI) calculated for $C_{20}H_{19}N$: 315.1604; found 315.1604.

2. Single crystal X-ray structure analysis.

Crystallographic data for compounds **5a, 7a, 7b, 8a** were obtained from Xcalibur X-ray diffractometer by monochromatized CuK_α radiation (λ=1.54180 Å) at 180 K (**7a, 8a**), 200 K **5a** and **7b** 290 K. The structures were solved by direct methods (SIR92)[1] (**3c**, **3d**, **4c**) and by charge flipping (SUPERFLIP)[2] compound **5a**. They were all refined by full-matrix least-squares based on F with (CRYSTALS)[3]. The hydrogen atoms were found on difference Fourier map, recalculated into idealized positions and refined with riding constraints. All other atoms were refined anisotropically.

Crystal data for 5a (colourless, 0.09 x 0.18 x 0.37 mm)**:**

$C_{27}H_{24}N_4O_2S_1$, monoclinic, space group *C2/c*, a = 20.7725(4) Å, b = 10.3703(3) Å, c = 22.3779(5) Å, β = 104.039(2)°, V = 4676.61(18) Å^3, Z = 8, M = 468.58, 24824 reflections measured, 4828 independent reflections. Final R = 0.043, wR = 0.045, GoF = 1.109 for 3729 reflections with $I > 2\sigma(I)$ and 307 parameters. CCDC 1014819.

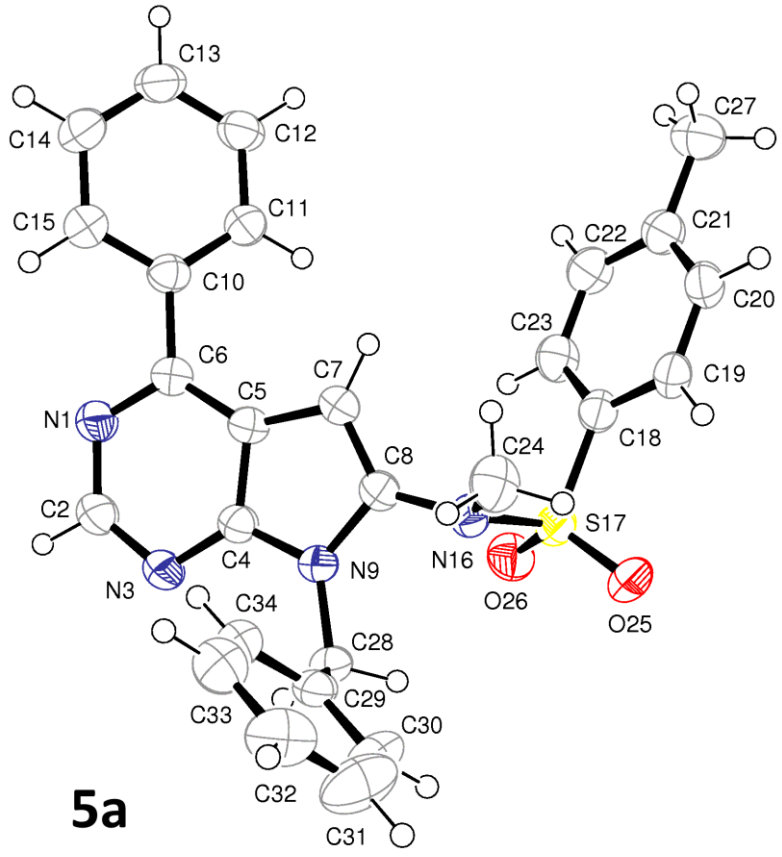


Figure 1. An ORTEP view of compounds **5a** shown with 50 % probability displacement ellipsoids.

Crystal data for 7a (orange, 0.45 x 0.68 x 0.72 mm)**:**

$C_{26}H_{21}N_5O_4S_1$, triclinic, space group *P*-1, *a* = 12.8359(2) Å, *b* = 14.6425(2) Å, *c* = 16.3039(3) Å, α = 81.5862(13)°, *β* = 70.0238(15)°, γ = 67.3666(15)°, *V* = 2657.75(8) Å^3, *Z* = 4, *M* = 499.54, 10790 reflections measured, 10790 independent reflections. Final *R* = 0.042, *wR* = 0.040, *GoF* = 0.968 for 9618 reflections with $I > 2\sigma(I)$ and 650 parameters. CCDC 1014820. The asymmetric unit consists of two molecules of **7a.** Furthermore, it contains solvent molecules – disordered ethyl acetate and partially occupied water molecules. These were not included in the refinement and the disordered density was taken into account using the SQUEEZE procedure (from PLATON[4]).

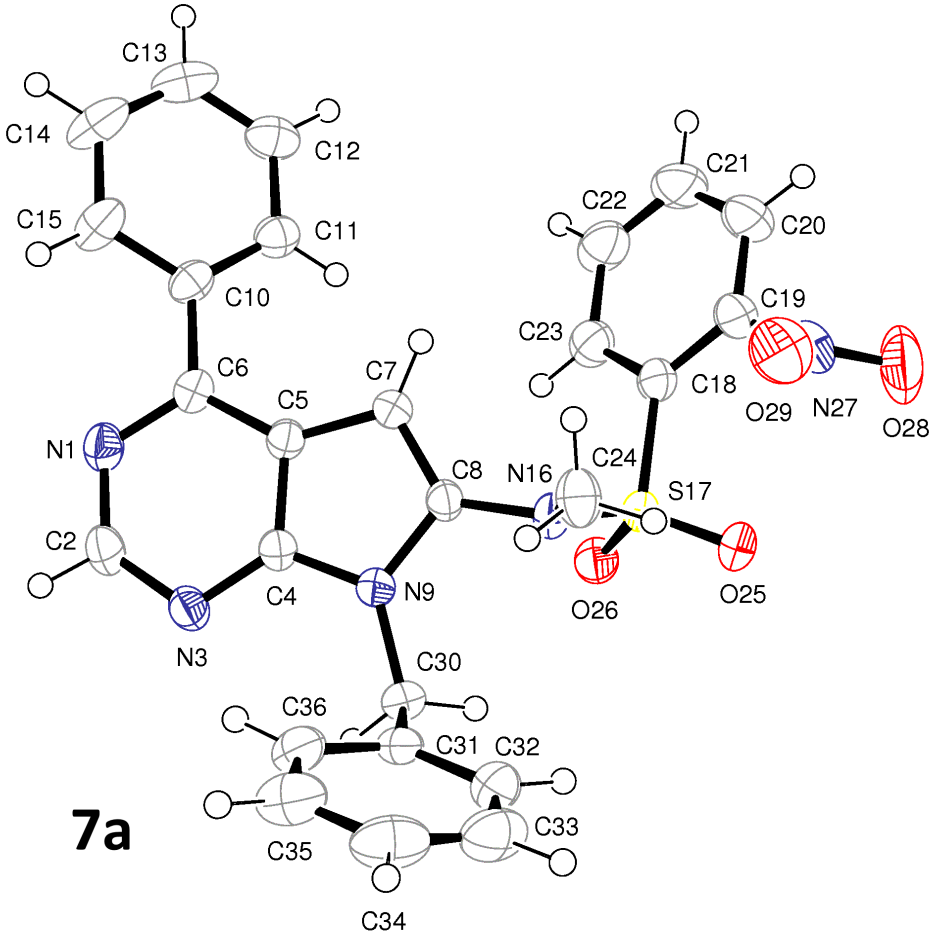


Figure 2. An ORTEP view of compounds **7a** shown with 50 % probability displacement ellipsoids.

Crystal data for 8a (colourless, 0.48 x 0.53 x 0.79 mm)**:**

$C_{26}H_{20}Cl_1N_5O_4S_1$, monoclinic, space group $P2_1/n$, a = 10.3204(3) Å, b = 10.7781(2) Å, c = 22.4546(7) Å, β = 103.112(3)°, V = 2432.59(12) Å^3, Z = 4, M = 533.99, 17639 reflections measured, 4993 independent reflections. Final R = 0.035, wR = 0.039, GoF = 1.033 for 4759 reflections with $I > 2\sigma(I)$ and 335 parameters. CCDC 1014817.

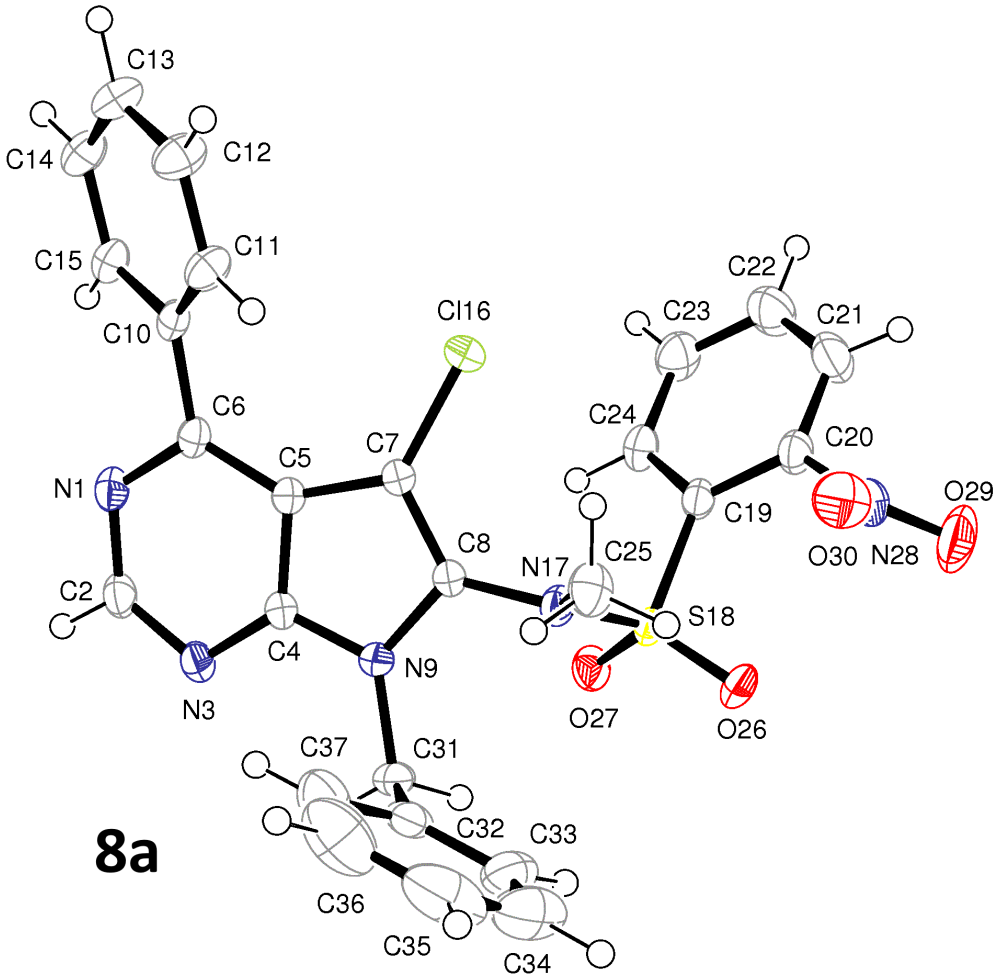


Figure 3. An ORTEP view of compounds **8a** shown with 50 % probability displacement ellipsoids.

Crystal data for 7b (colourless, 0.21 x 0.30 x 0.83 mm)**:**

$C_{21}H_{19}N_5O_5S_1$, triclinic, space group *P*-1, *a* = 8.0254(2) Å, *b* = 8.5175(2) Å, *c* = 16.5553(4) Å, α = 76.069(2)°, *β* = 76.692(2)°, γ = 76.024(2)°, *V* = 1047.92(5) Å^3, *Z* = 2, *M* = 453.48, 18491 reflections measured, 4263 independent reflections. Final *R* = 0.036, *wR* = 0.042, *GoF* = 0.820 for 3984 reflections with $I > 2\sigma(I)$ and 290 parameters. CCDC 1014818.

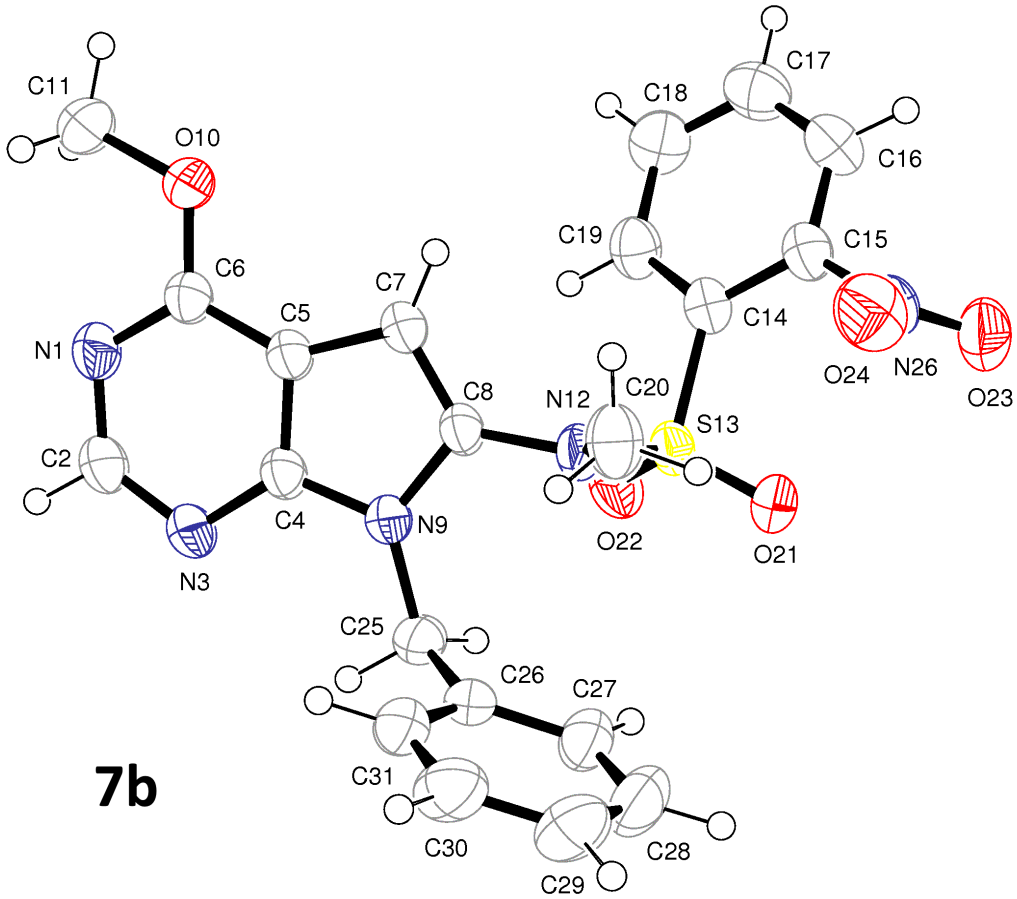
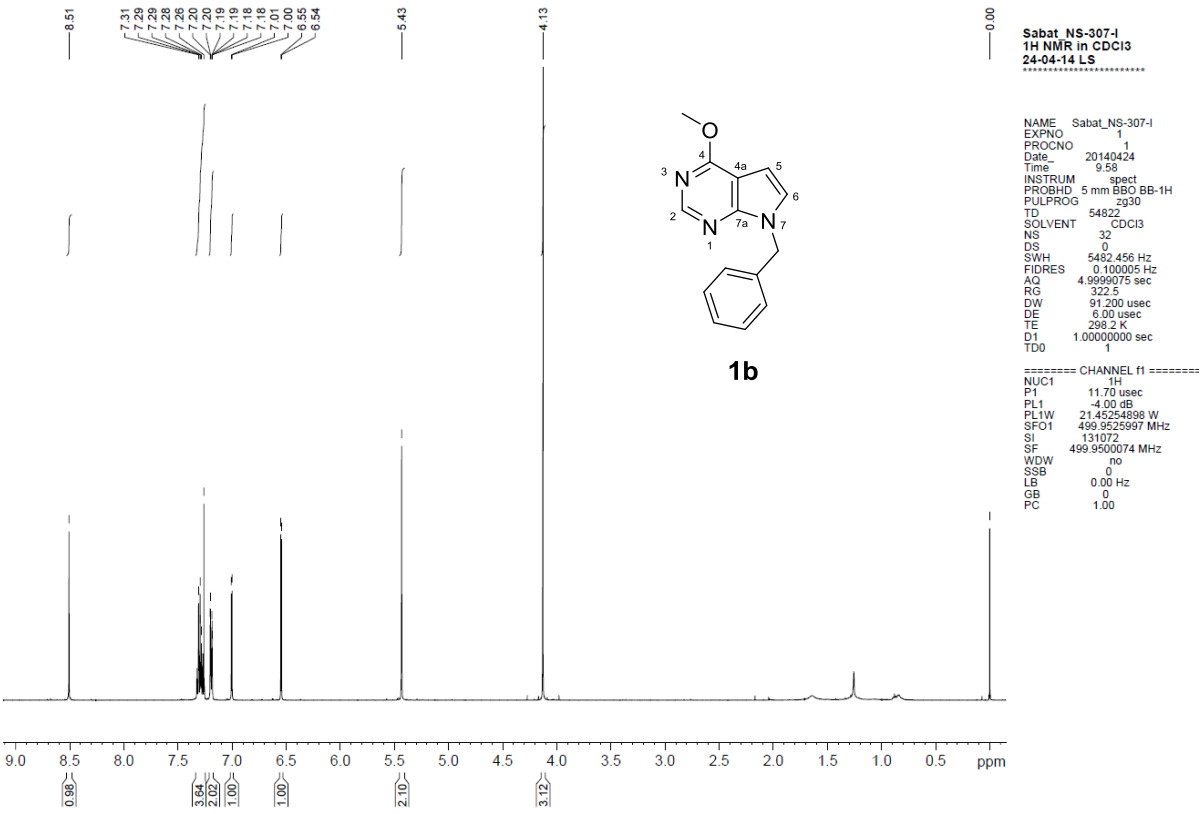
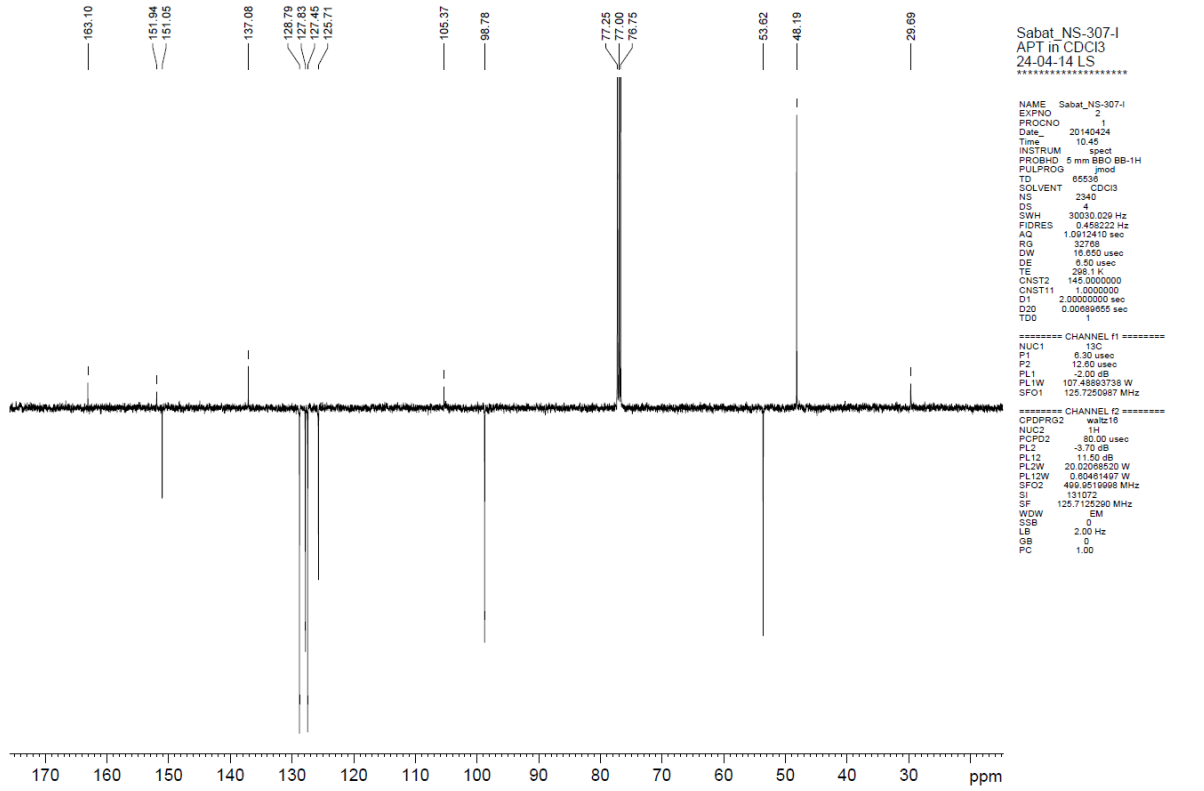
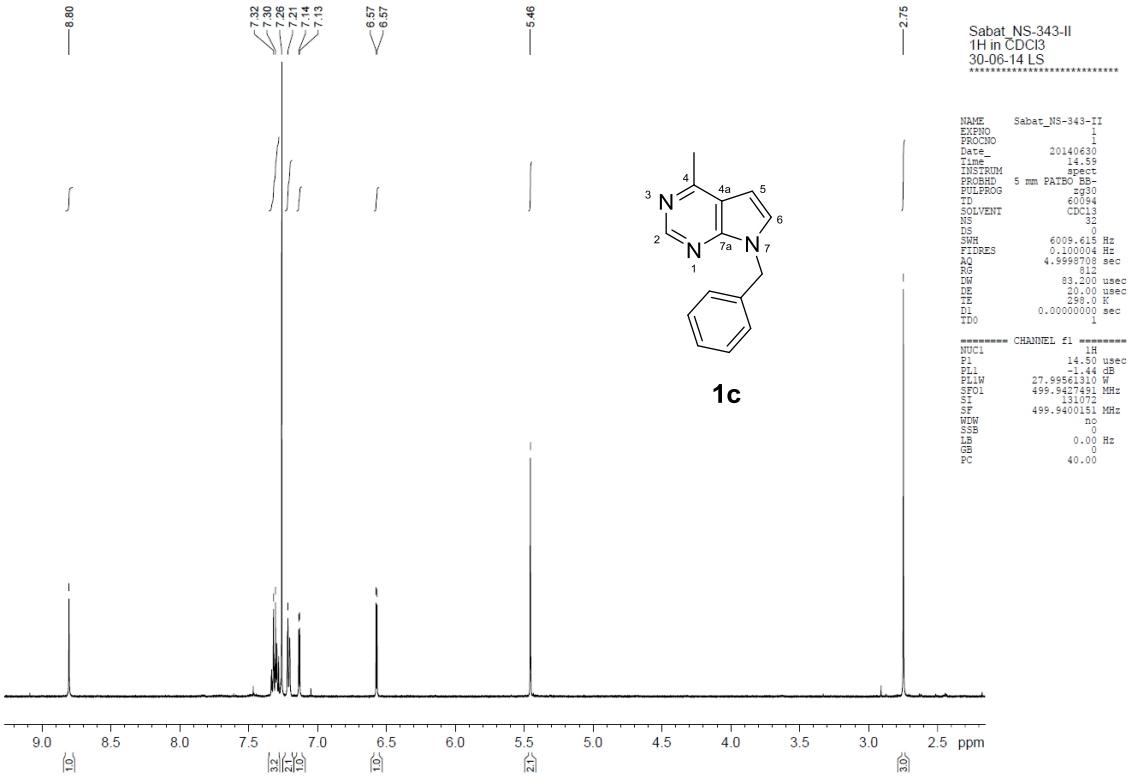


Figure 4. An ORTEP view of compounds **7b** shown with 50 % probability displacement ellipsoids.

3. Copies of NMR spectra



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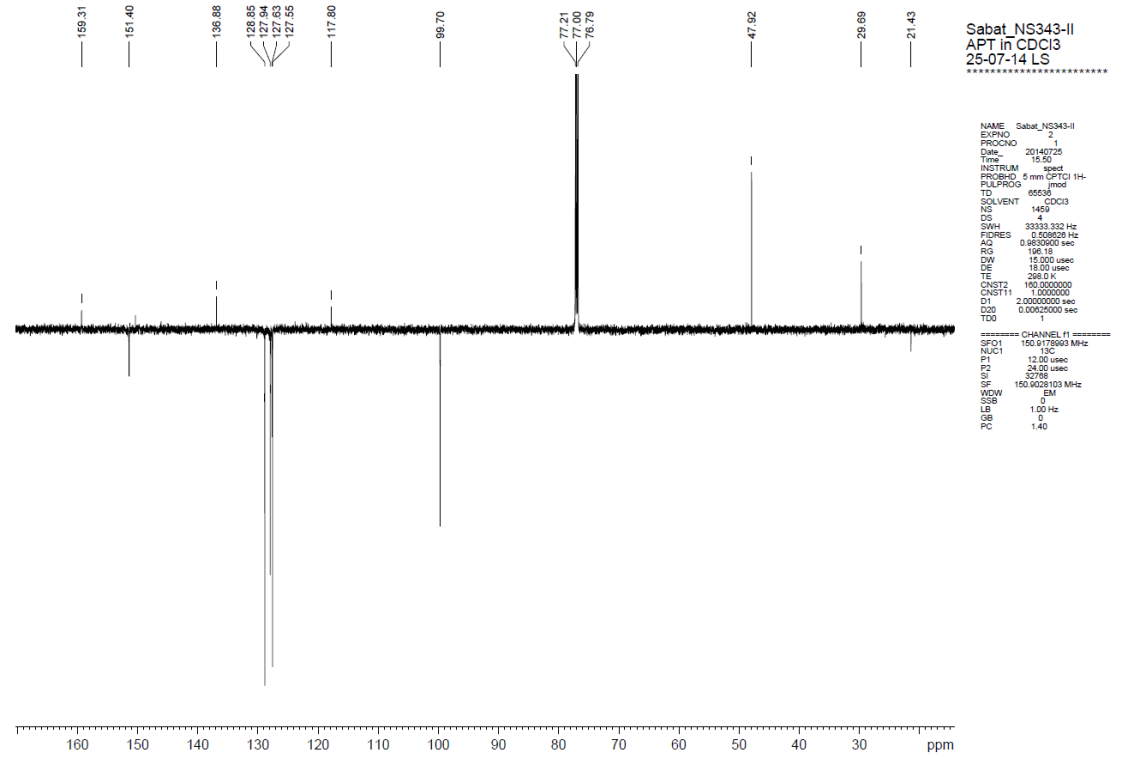
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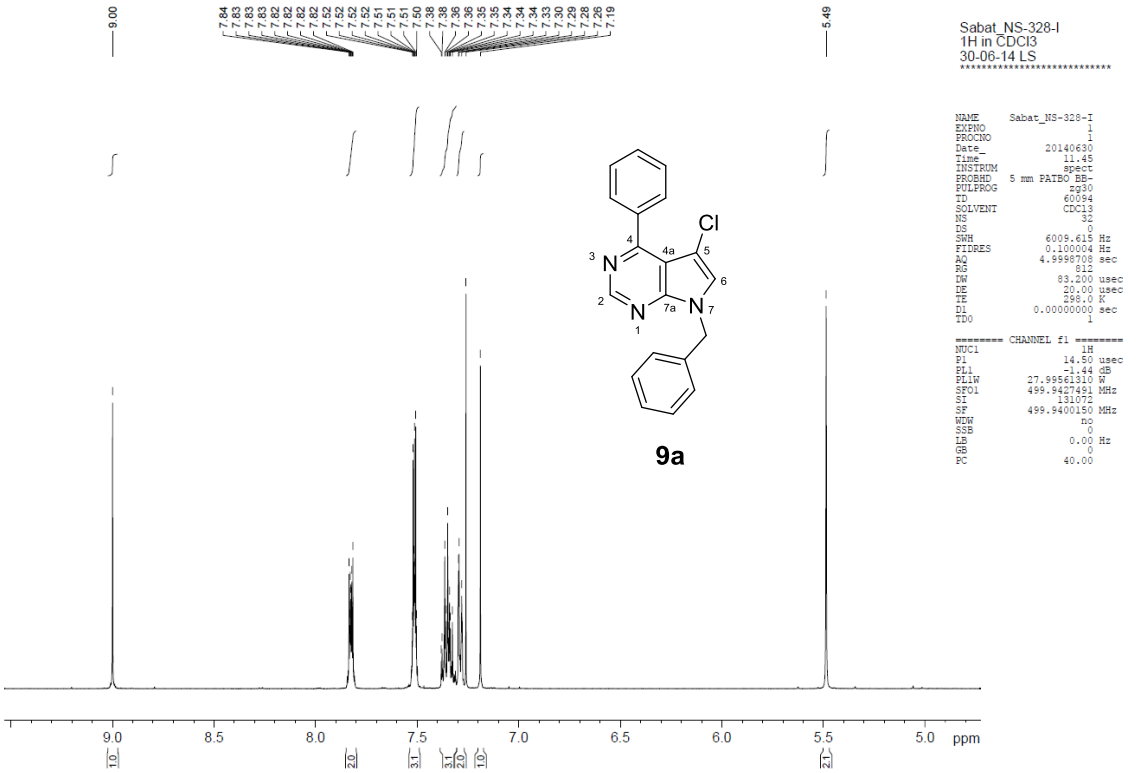
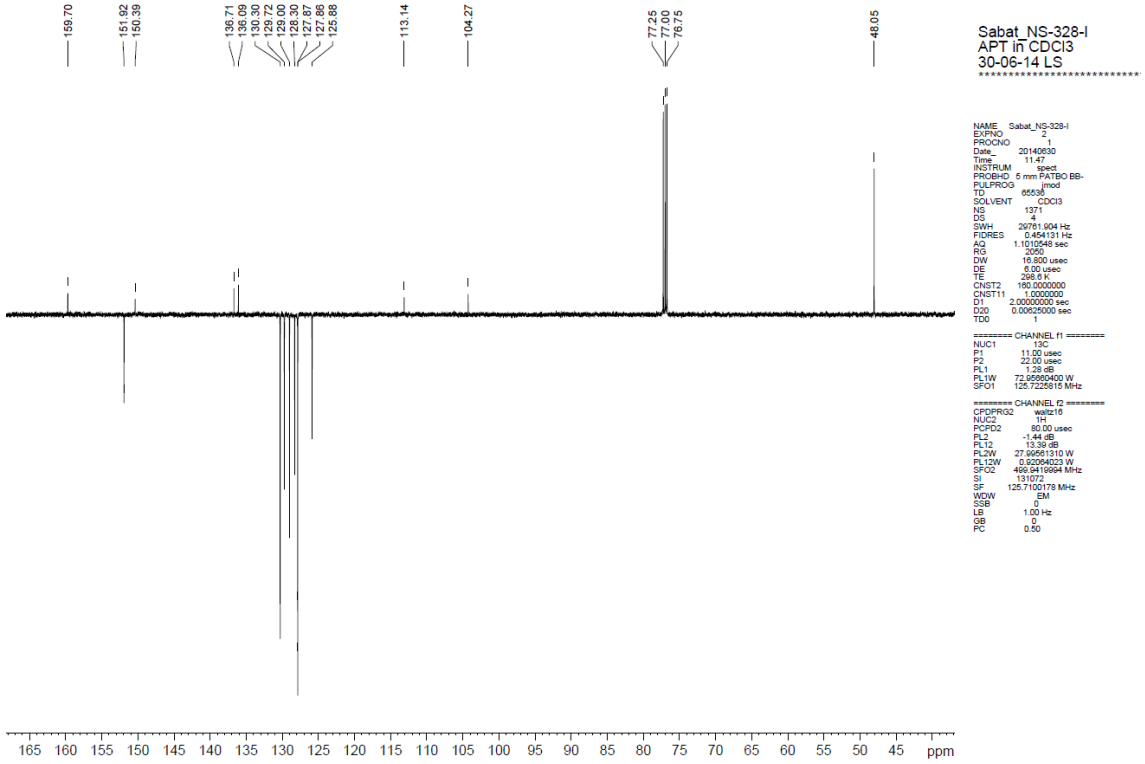
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm



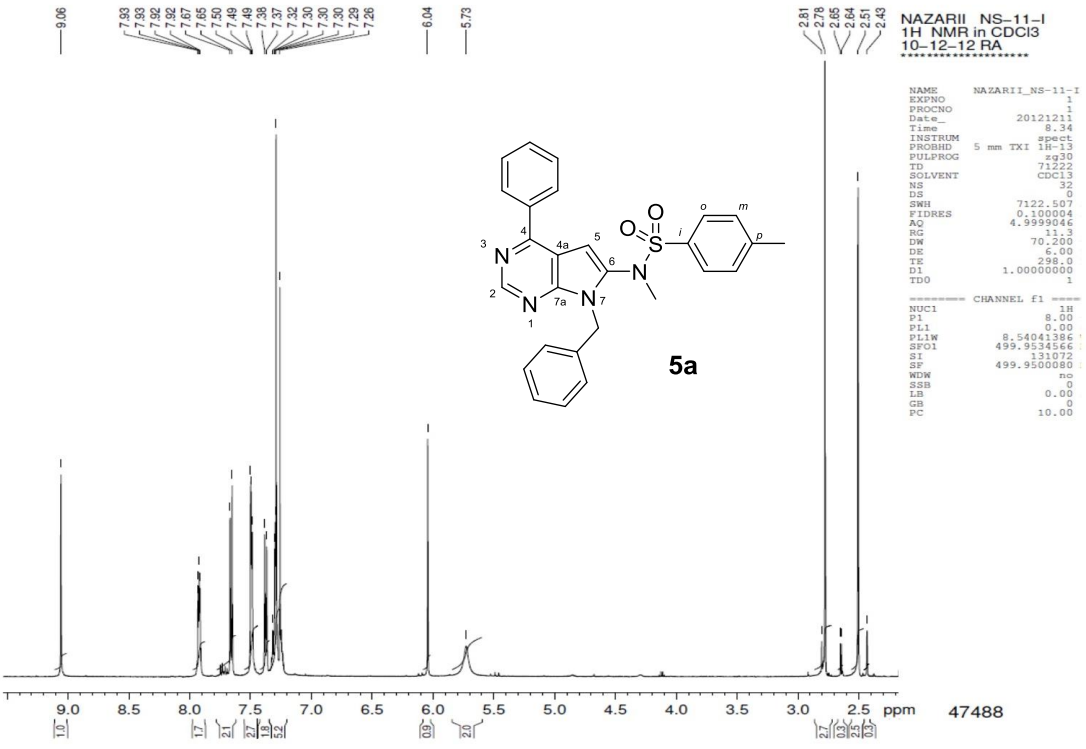
Sabat_NS-328-I
1H in CDCl3
30-06-14 LS

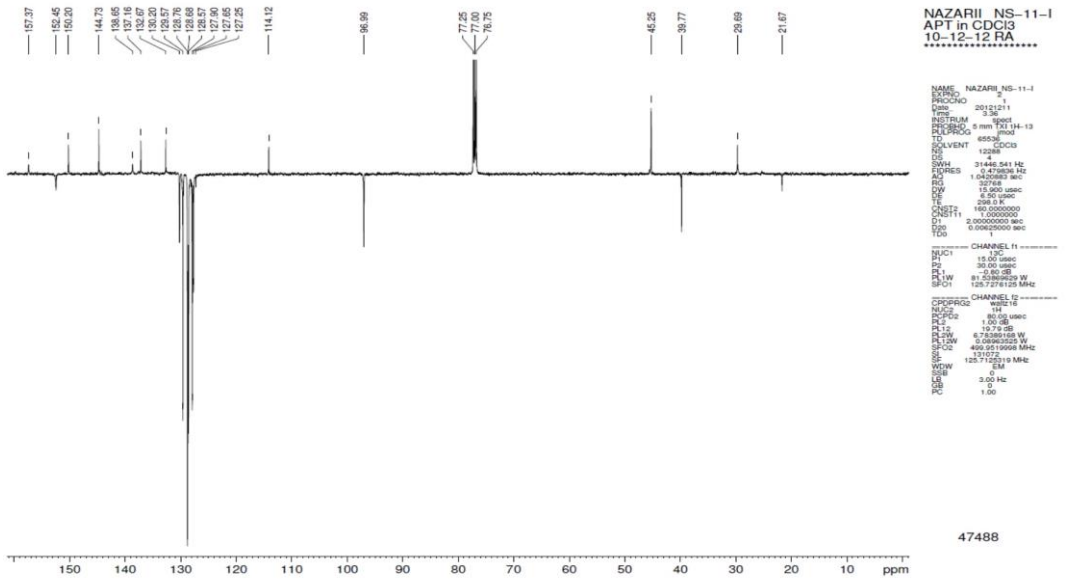
```
NAME        Sabat_NS-328-I
EXPNO                    1
PROCNO                   1
Date_             20140630
Time                 11.45
INSTRUM              spect
PROBHD  5 mm PATBO BB-
PULPROG              zg30
TD                  60094
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              6009.615 Hz
FIDRES           0.100004 Hz
AQ               4.9998708 sec
RG                    812
DW                 83.200 usec
DE                 20.00 usec
TE                298.0 K
D1             0.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.50 usec
PL1                 -1.44 dB
PL1W          27.99561310 W
SFO1          499.9427491 MHz
SI                  131072
SF            499.9400150 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                  40.00
```

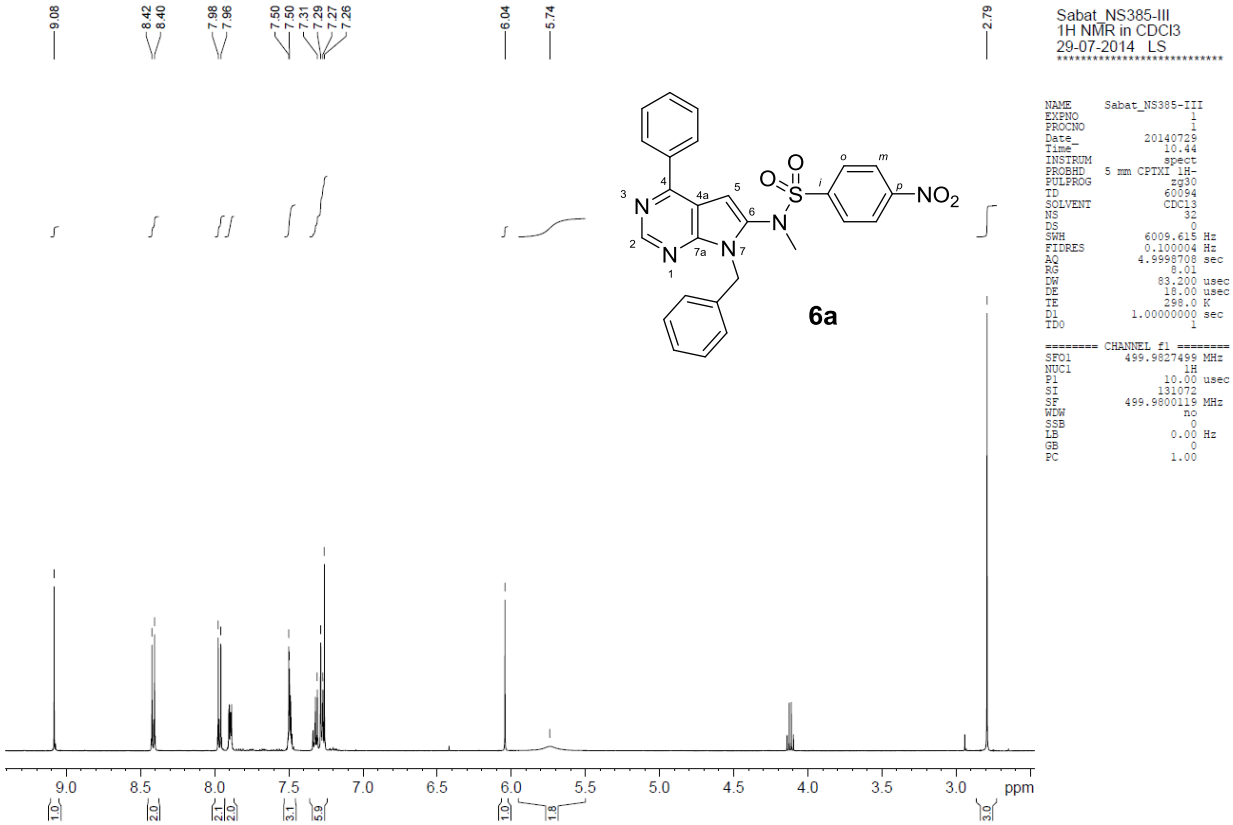


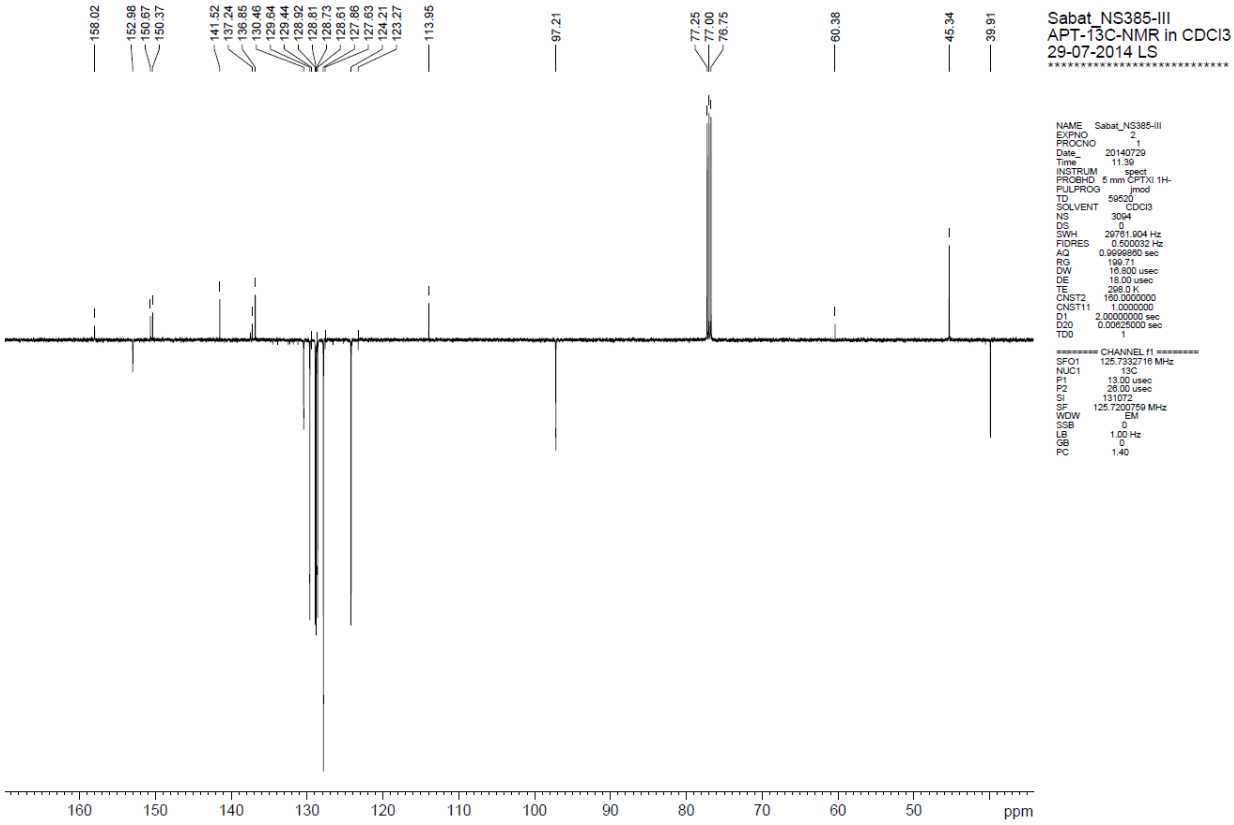
Sabat_NS-328-I
APT in CDCl3
30-06-14 LS

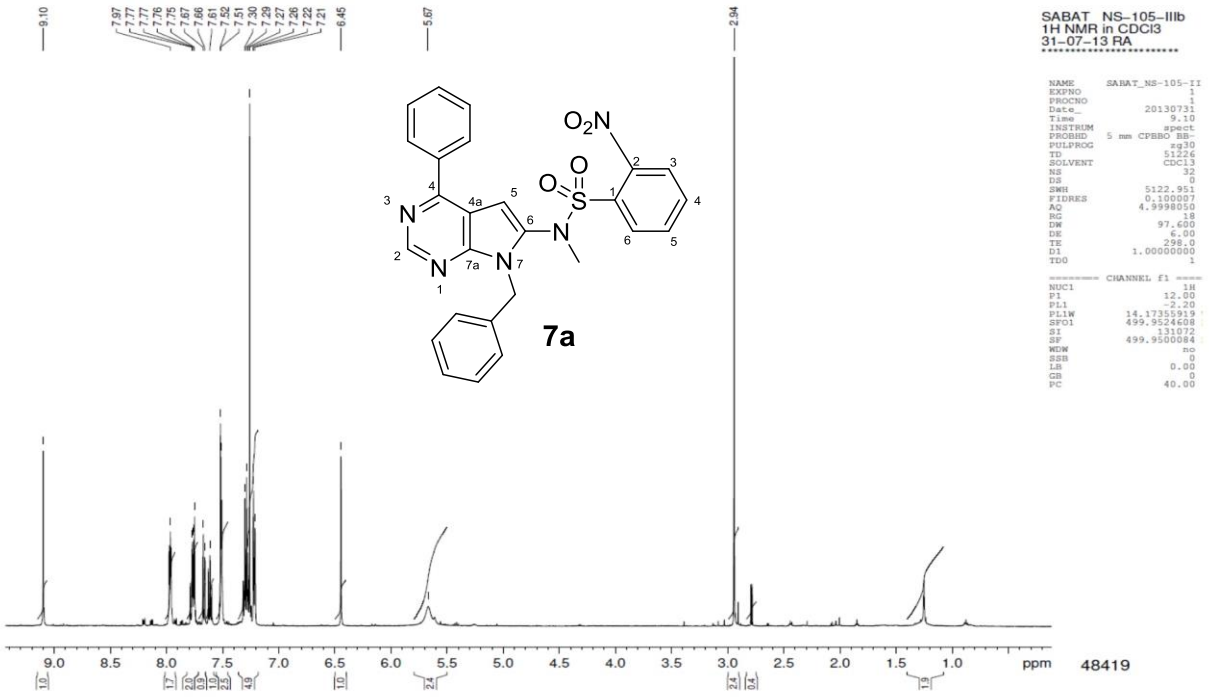
```
NAME    Sabat_NS-328-I
EXPNO               2
PROCNO              1
Date_        20140630
Time            11.47
INSTRUM         spect
PROBHD  5 mm PATBO BB-
PULPROG         mod
TD              65536
SOLVENT         CDCl3
NS              1371
DS                 4
SWH         29761.904 Hz
FIDRES      0.454131 Hz
AQ          1.1010548 sec
RG              2050
DW            16.800 usec
DE             6.00 usec
TE            298.6 K
CNST2       160.0000000
CNST11        1.0000000
D1          2.00000000 sec
D20         0.00625000 sec
TD0                1

======== CHANNEL f1 ========
NUC1               13C
P1             11.00 usec
P2             22.00 usec
PL1             1.28 dB
PL1W       72.95680400 W
SFO1       125.7225815 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2               1H
PCPD2          80.00 usec
PL2            -1.44 dB
PL12           13.39 dB
PL2W       27.99561310 W
PL12W       0.92094023 W
SFO2       499.9419994 MHz
SI              131072
SF         125.7100178 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC               0.50
```

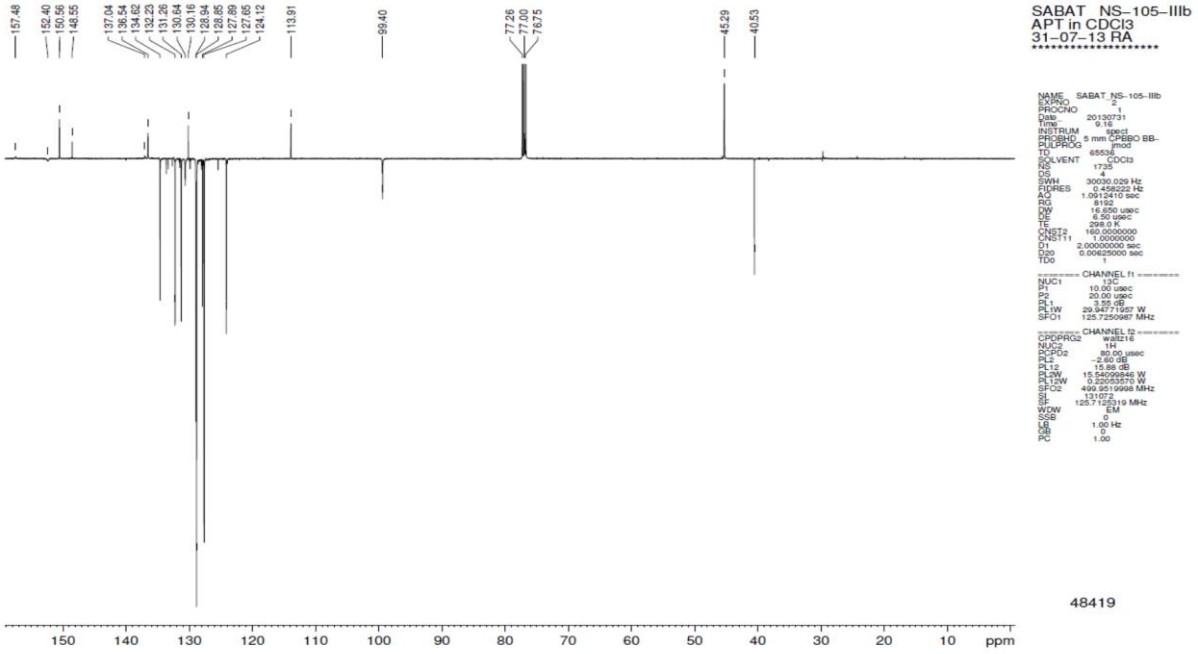

NAZARII NS-11-I
1H NMR in CDCl3
10-12-12 RA
5a
47488

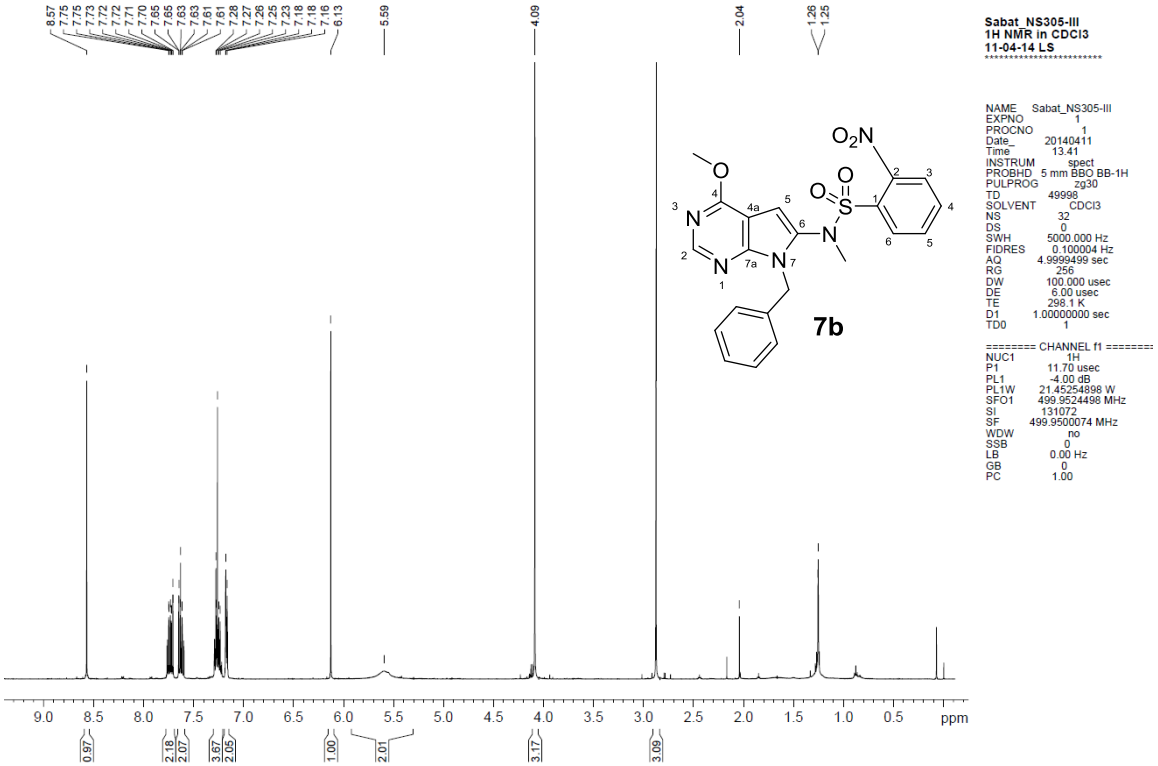

NAZARII NS-11-I
APT in CDCl3
10-12-12 RA
47488

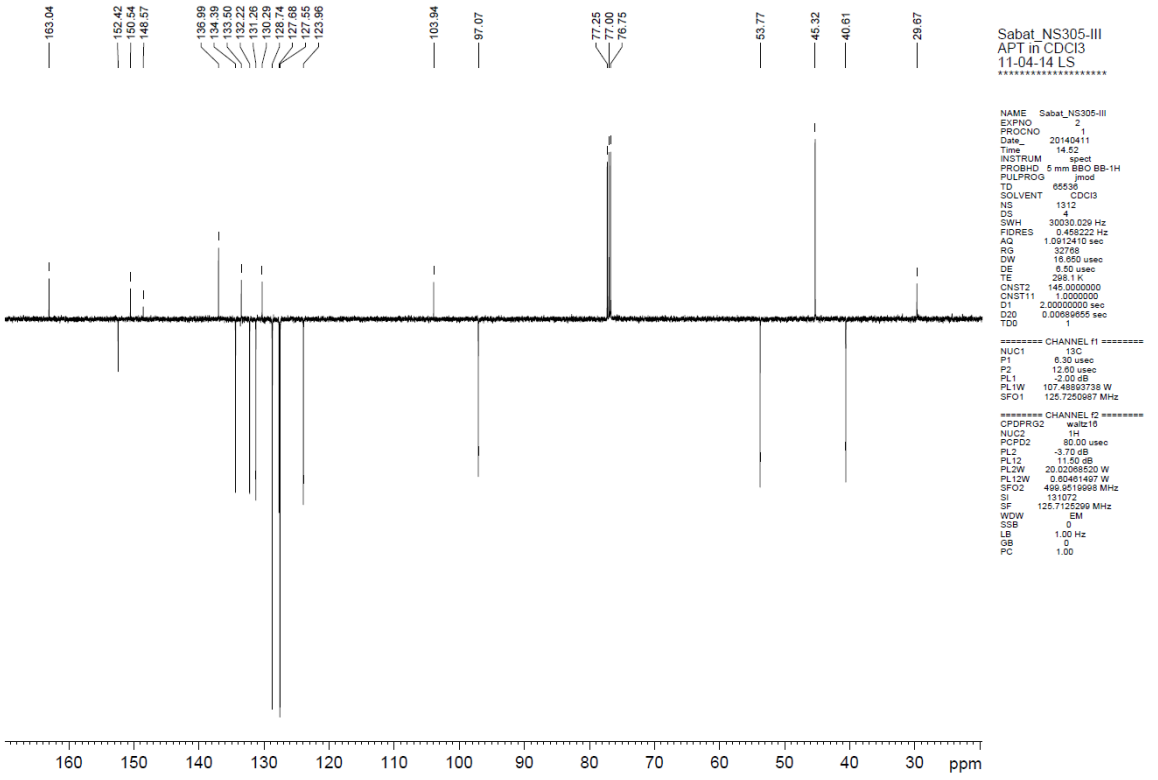

9.08
8.42
8.40
7.98
7.96
7.50
7.50
7.31
7.29
7.27
7.26
6.04
5.74
2.79
Sabat_NS385-III
1H NMR in CDCl3
29-07-2014 LS
6a
ppm

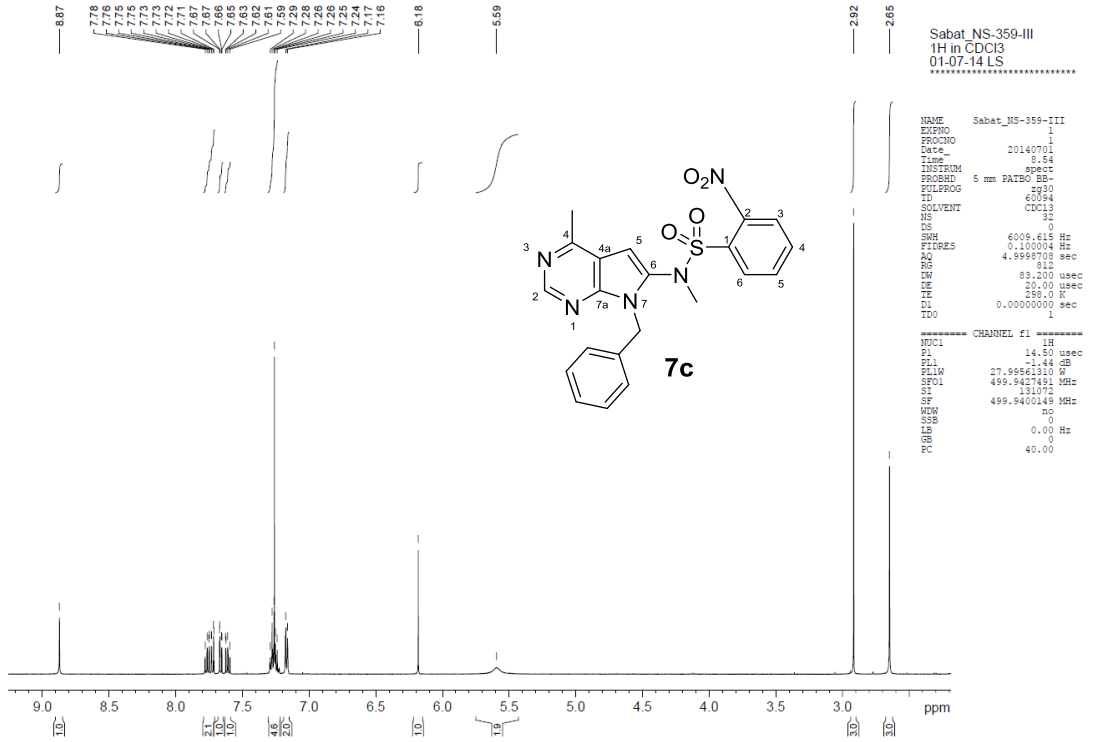

158.02
152.98
150.67
150.37
141.52
137.24
136.85
130.46
129.64
129.44
128.92
128.81
128.73
128.61
127.86
127.63
124.21
123.27
113.95
97.21
77.25
77.00
76.75
60.38
45.34
39.91
Sabat_NS385-III
APT-13C-NMR in CDCl3
29-07-2014 LS
ppm

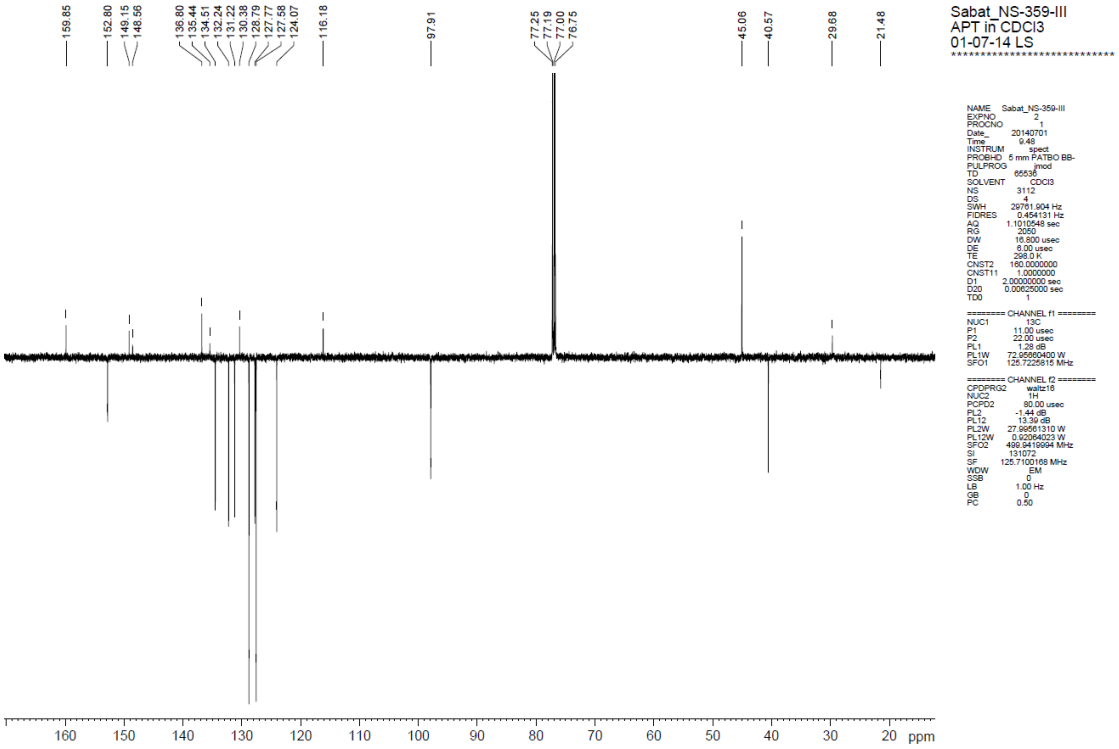

9.10
7.97
7.77
7.77
7.76
7.75
7.67
7.66
7.61
7.52
7.51
7.30
7.29
7.27
7.26
7.22
7.21
6.45
5.67
2.94
O_2N
O
O
S
N
N
N
7a
SABAT NS-105-IIIb
1H NMR in CDCl3
31-07-13 RA
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
48419

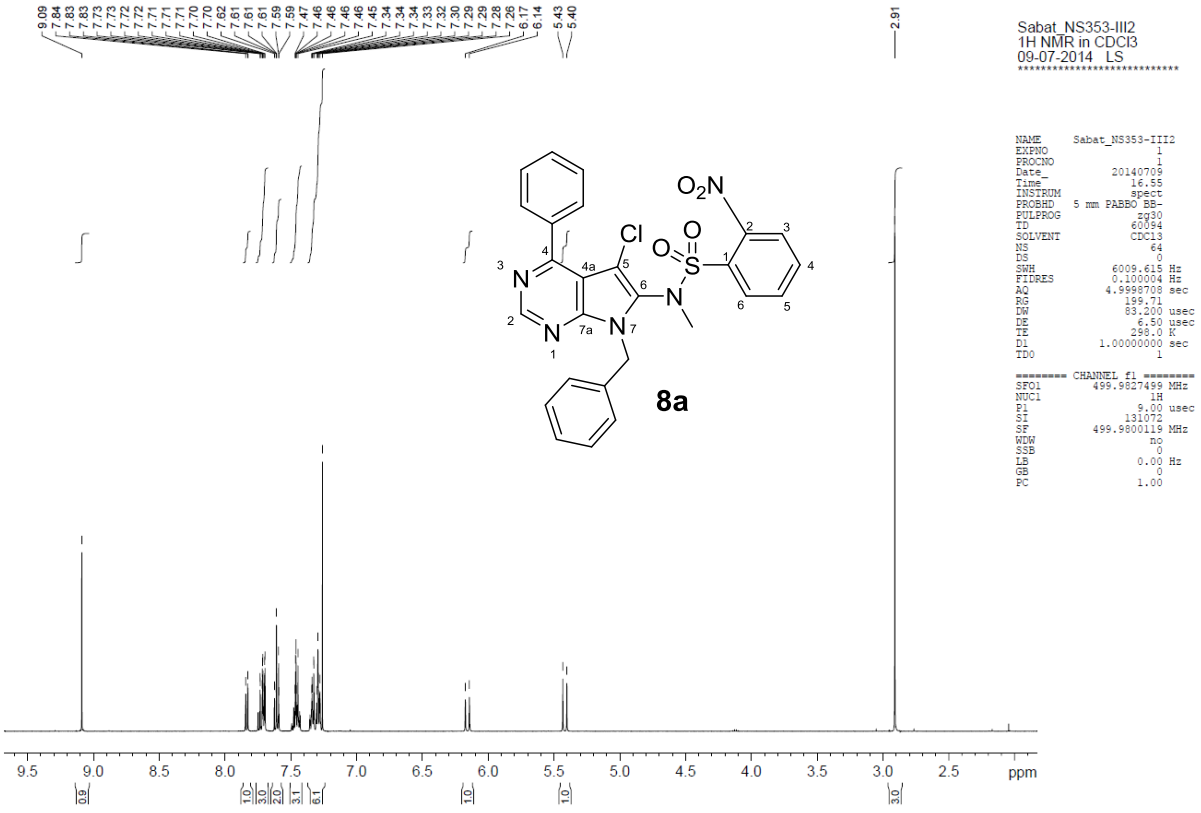

157.48
152.40
150.56
148.55
137.04
136.54
134.62
134.23
131.26
130.64
130.16
128.94
128.85
127.89
127.65
124.12
113.91
99.40
77.26
77.00
76.75
45.29
40.53
SABAT NS-105-IIIb
APT in CDCl3
31-07-13 RA
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
48419

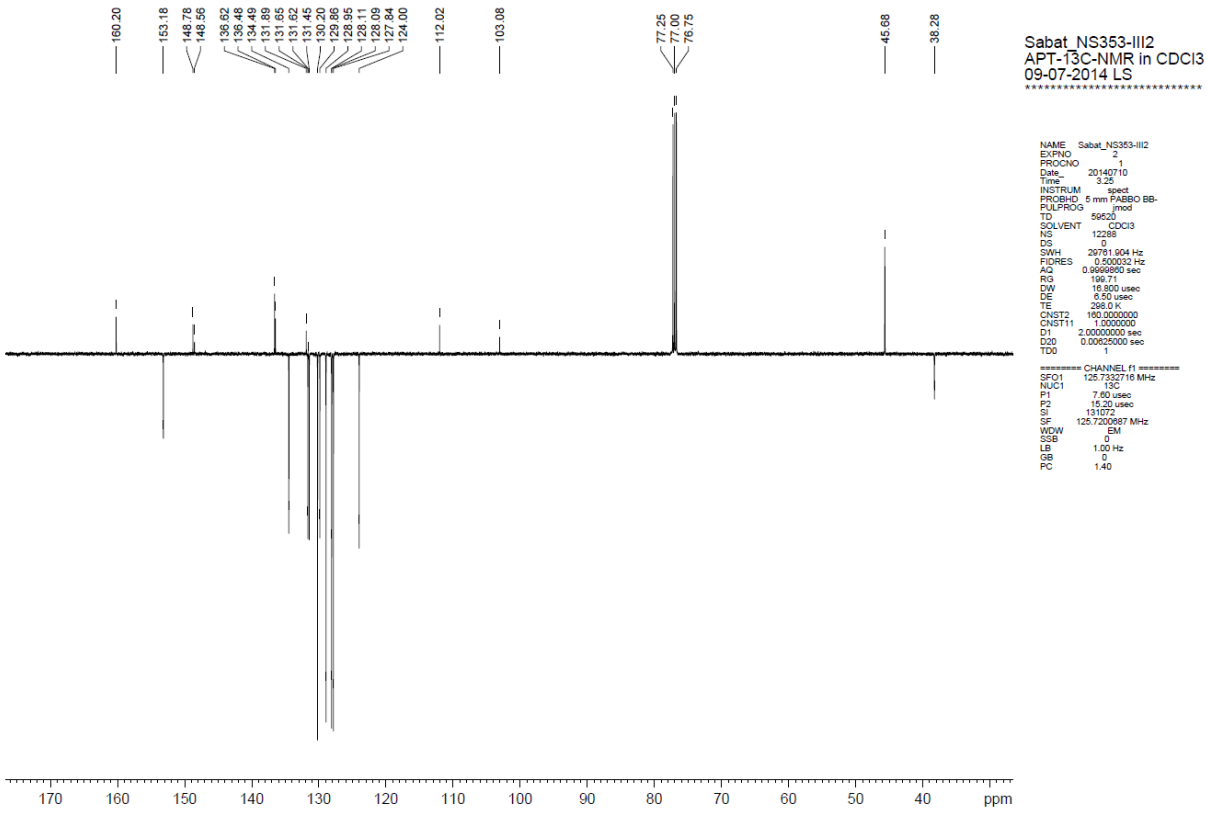

8.57 7.75 7.75 7.73 7.72 7.72 7.71 7.70 7.65 7.65 7.63 7.63 7.61 7.61 7.28 7.27 7.26 7.25 7.23 7.18 7.18 7.16 6.13 5.59 4.09 2.04 1.26 1.25
Sabat_NS305-III
1H NMR in CDCl3
11-04-14 LS
NAME Sabat_NS305-III
EXPNO 1
PROCNO 1
Date_ 20140411
Time 13.41
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 49998
SOLVENT CDCl3
NS 32
DS 0
SWH 5000.000 Hz
FIDRES 0.100004 Hz
AQ 4.9999499 sec
RG 256
DW 100.000 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 499.9524498 MHz
SI 131072
SF 499.9500074 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 2.0 1.5 1.0 0.5 ppm
0.97 2.18 2.07 3.67 2.05 1.00 2.01 3.17 3.09

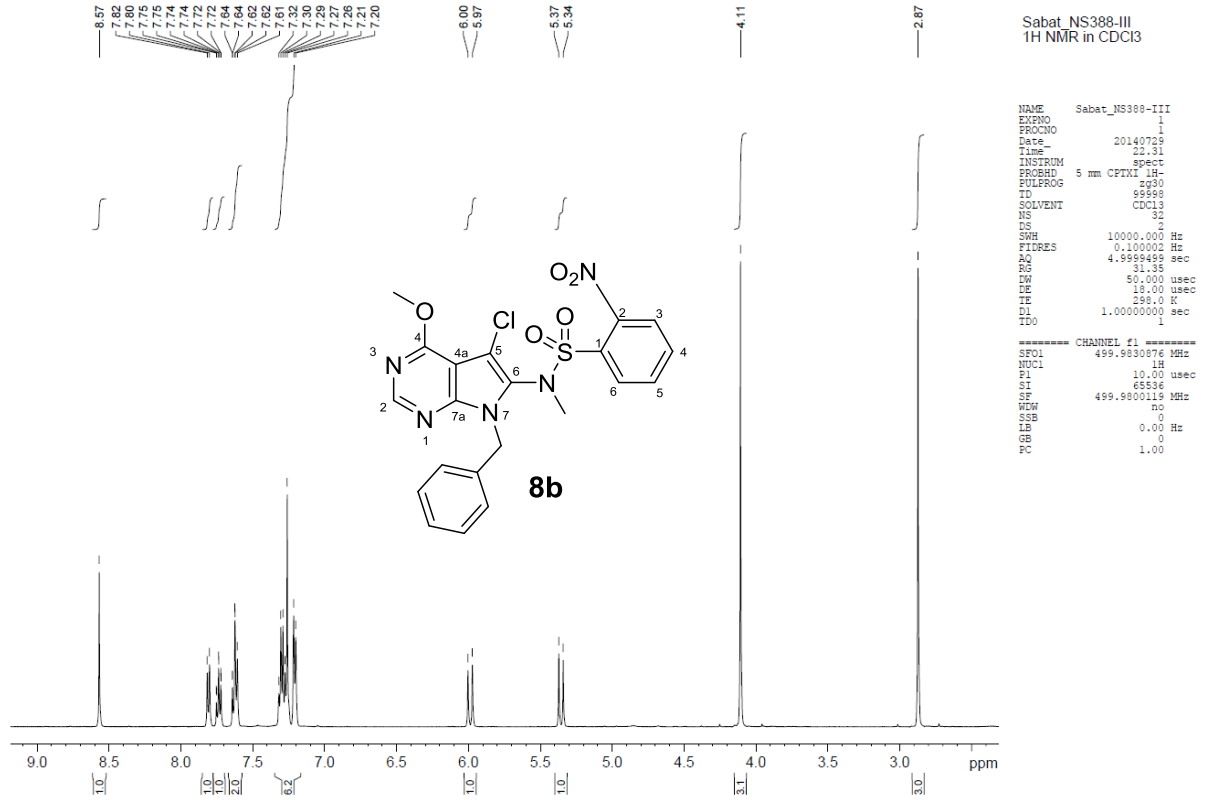

163.04 152.42 150.54 148.57 136.99 134.39 133.50 132.22 131.26 130.29 128.74 127.68 127.55 123.96 103.94 97.07 77.25 77.00 76.75 53.77 45.32 40.61 29.67
Sabat_NS305-III
APT in CDCl3
11-04-14 LS
NAME Sabat_NS305-III
EXPNO 2
PROCNO 1
Date_ 20140411
Time 14.52
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 1312
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.09124410 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 125.7256987 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.70 dB
PL12 11.50 dB
PL2W 20.02068520 W
PL12W 0.65461497 W
SFO2 499.9519998 MHz
SI 131072
SF 125.7125299 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

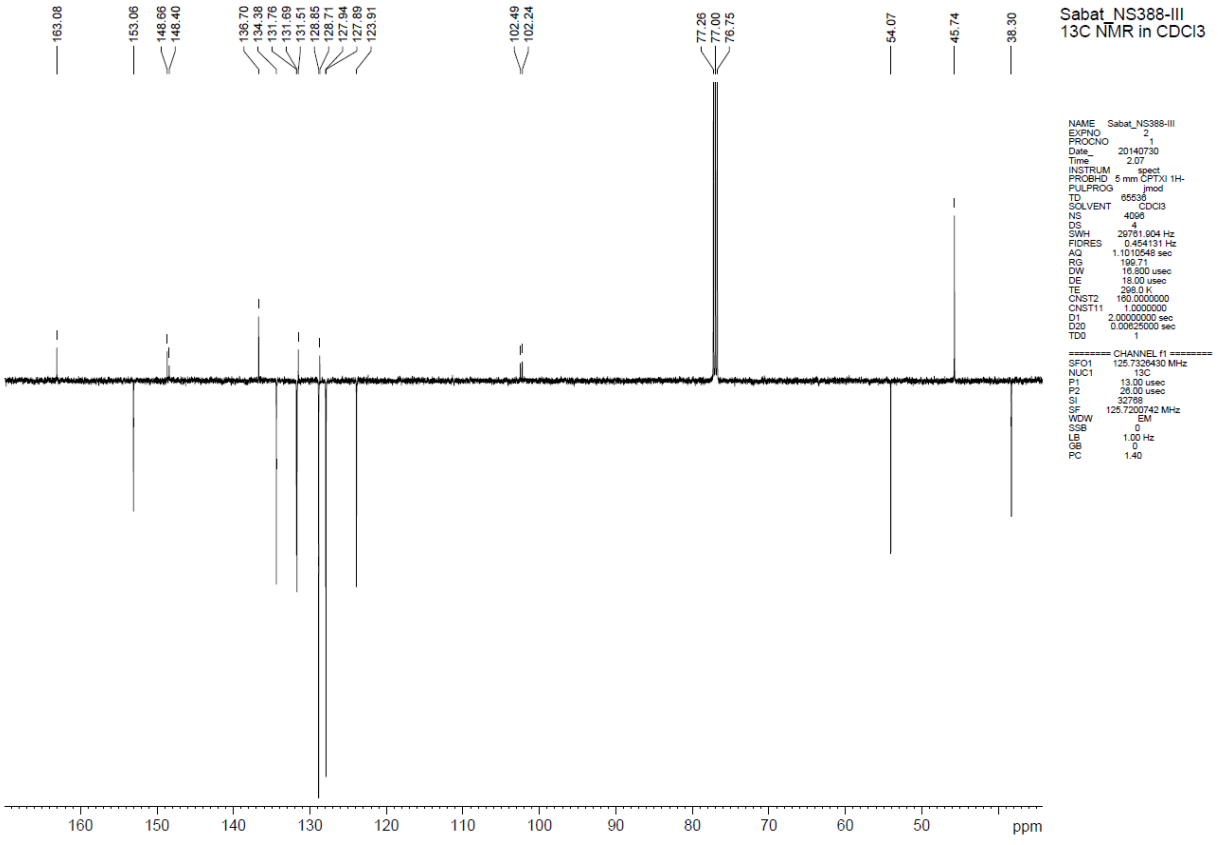

8.87
7.78
7.76
7.75
7.73
7.72
7.71
7.67
7.67
7.66
7.65
7.63
7.62
7.61
7.59
7.29
7.28
7.26
7.25
7.24
7.17
7.16
6.18
5.59
2.92
2.65
Sabat_NS-359-III
1H in CDCl3
01-07-14 LS
O_2N
7c
ppm

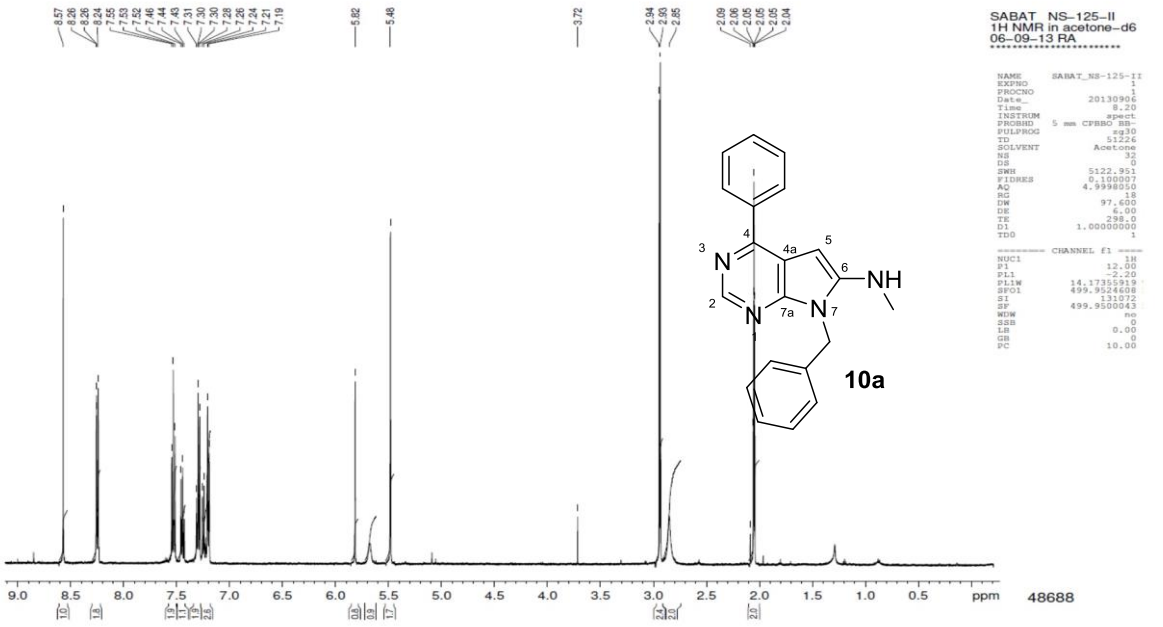

159.85
152.80
149.15
146.56
136.80
135.44
134.51
132.24
131.22
130.38
128.79
127.77
127.58
124.07
116.18
97.91
77.25
77.19
77.00
76.75
45.06
40.57
29.68
21.48
Sabat_NS-359-III
APT in CDCl3
01-07-14 LS
ppm

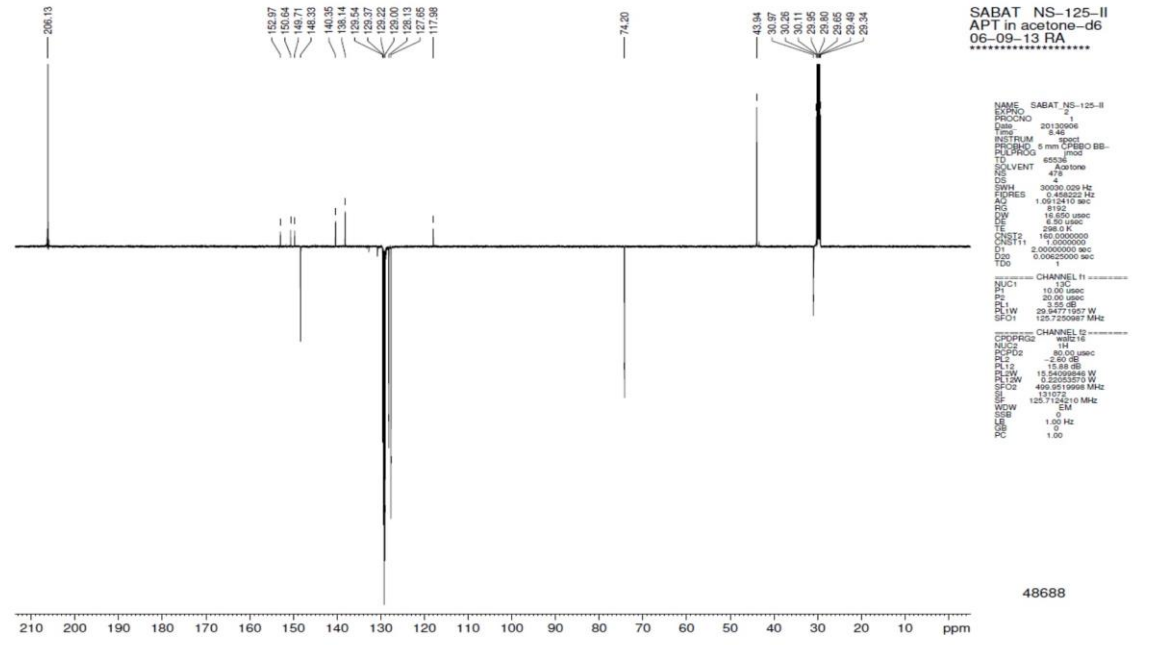

9.09 7.84 7.83 7.83 7.73 7.73 7.72 7.72 7.71 7.71 7.70 7.70 7.62 7.61 7.61 7.61 7.59 7.59 7.47 7.46 7.46 7.46 7.46 7.45 7.34 7.34 7.34 7.33 7.32 7.30 7.29 7.29 7.28 7.26 6.17 6.14 5.43 5.40
2.91
Sabat_NS353-III2
1H NMR in CDCl3
09-07-2014 LS
NAME Sabat_NS353-III2
EXPNO 1
PROCNO 1
Date_ 20140709
Time 16.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 60094
SOLVENT CDCl3
NS 64
DS 0
SWH 6009.615 Hz
FIDRES 0.100004 Hz
AQ 4.9998708 sec
RG 199.71
DW 83.200 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 499.9827499 MHz
NUC1 1H
P1 9.00 usec
SI 131072
SF 499.9800119 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Cl
O2N
8a
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
0.9 1.0 3.0 2.0 3.1 6.1 1.0 1.0 3.0


160.20 153.18 148.78 148.56 136.62 136.48 134.49 131.89 131.65 131.62 131.45 130.20 129.86 128.95 128.11 128.09 127.84 124.00 112.02 103.08 77.25 77.00 76.75 45.68 38.28
Sabat_NS353-III2
APT-13C-NMR in CDCl3
09-07-2014 LS
NAME Sabat_NS353-III2
EXPNO 2
PROCNO 1
Date_ 20140710
Time 3.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 59520
SOLVENT CDCl3
NS 12288
DS 0
SWH 29761.904 Hz
FIDRES 0.500032 Hz
AQ 0.9999850 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 125.7200687 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 ppm


8.57
7.82
7.80
7.75
7.75
7.74
7.74
7.72
7.72
7.64
7.64
7.62
7.62
7.61
7.32
7.30
7.29
7.27
7.26
7.21
7.20
6.00
5.97
5.37
5.34
4.11
2.87
Sabat_NS388-III
1H NMR in CDCl3
NAME Sabat_NS388-III
EXPNO 1
PROCNO 1
Date_ 20140729
Time 22.31
INSTRUM spect
PROBHD 5 mm CPTXI 1H-
PULPROG zg30
TD 99998
SOLVENT CDCl3
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.100002 Hz
AQ 4.9999489 sec
RG 31.35
DW 50.000 usec
DE 18.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 499.9830876 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 499.9800119 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
O2N
Cl
O
O
S
N
O
N
N
N
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
ppm
1.0
1.0
1.0
2.0
6.2
1.0
1.0
3.1
3.0


163.08
153.06
148.66
148.40
136.70
134.38
131.76
131.69
131.51
128.85
128.71
127.94
127.89
123.91
102.49
102.24
77.26
77.00
76.75
54.07
45.74
38.30
Sabat_NS388-III
13C NMR in CDCl3
NAME Sabat_NS388-III
EXPNO 2
PROCNO 1
Date_ 20140730
Time 2.07
INSTRUM spect
PROBHD 5 mm CPTXI 1H-
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 4096
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 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 125.7326430 MHz
NUC1 13C
P1 13.00 usec
P2 26.00 usec
SI 32768
SF 125.7200742 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
ppm


SABAT NS-125-II
1H NMR in acetone-d6
06-09-13 RA
10a
48688


SABAT NS-125-II
APT in acetone-d6
06-09-13 RA
48688

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