Supporting Information

One-Pot Etherification of Purine Nucleosides and Pyrimidines

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General Experimental Considerations

Thin layer chromatography was performed on 250 μm silica plates and column chromatographic purifications were performed on 200–300 mesh silica gel. THF was distilled over $LiAlH_4$ and then over Na, MeCN was distilled over CaH_2. All other reagents were obtained from commercial sources and were used without further purification. ^{1}H NMR spectra were recorded at 500 MHz in $CDCl_3$ and are referenced to the residual protonated solvent resonance. ^{13}C NMR spectra were recorded at 125 MHz in $CDCl_3$ and are referenced to the solvent resonance. Chemical shifts (δ) are reported in parts per million (ppm) and coupling constants (*J*) are in hertz (Hz).

General Procedure for the One-Pot Etherification Reactions

To a solution of the substrate (0.16 mmol of protected inosine or 79 μmol of protected guanosine or 0.10 mmol of protected 2'-deoxyguanosine or 0.68 mmol of either quinazolin-4(3*H*)-one or 4-methylpyrimidin-2(1*H*)-one•HCl or 0.57 mmol of 5-bromopyrimidin-2(1*H*)-one) in dry THF (2 mL) were added 2 molar equiv each of BOP and Cs_2CO_3 under a nitrogen atmosphere. The mixture was stirred at room temperature (10 min for inosine, 1 h for guanosine and 2'-deoxyguanosine, 50 min for pyrimidinones).

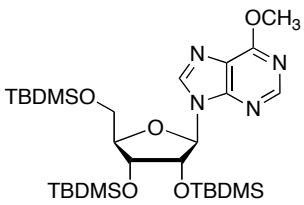
For reactions with alcohols: the resulting mixture was evaporated under reduced pressure, 2 molar equiv of Cs_2CO_3 and 20 molar equiv of alcohol were added.

For reactions with phenols: 2 molar equiv of Cs_2CO_3, and 2 molar equiv of the phenol were added without evaporation of the mixture.

In each case, the mixture was allowed to stir at the appropriate temperature (indicated in Tables 1 and 2), until TLC indicated complete reaction. The reaction mixture was diluted with water (10 mL) and extracted with EtOAc (3 x 10 mL). The organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The crude products were purified by column chromatography on silica gel using the solvent indicated under each compound heading. Any deviation from the general procedure is described under the specific compound heading.

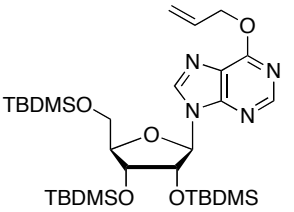
2',3',5'-Tri-*O*-(*tert*-butyldimethylsilyl)-O^6-methylinosine (2).

Purification using 20% EtOAc in hexanes gave 96 mg (94%) of **2** as a yellowish solid. R_f (SiO_2/20% EtOAc in hexanes) = 0.4. ^{1}H NMR: δ 8.52 (s, 1H, Ar-H), 8.32 (s, 1H, Ar-H), 6.08 (d, 1H, H-1', *J* = 4.8 Hz), 4.62 (t, 1H, H-2', *J* = 4.6 Hz), 4.31 (t, 1H, H-3', *J* = 4.1 Hz), 4.18 (s, 3H,



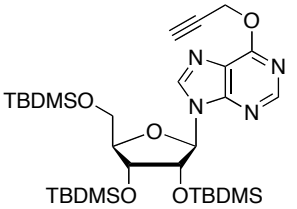
OCH_3), 4.13 (q, 1H, H-4', J = 3.1 Hz), 4.03 (dd, 1H, H-5', J = 3.9, 11.2 Hz), 3.79 (dd, 1H, H-5', J = 2.6, 11.2 Hz), 0.95, 0.93, and 0.75 (3s, 27H, *tert*-Bu), 0.14, 0.13, 0.10, 0.09, –0.04, and –0.22 (6s, 18H, $SiCH_3$). ^{13}C NMR: δ 161.2, 152.2, 151.9, 141.3, 122.2, 88.6, 85.6, 76.3, 72.1, 62.7, 54.3, 26.3, 26.0, 25.8, 18.7, 18.3, 18.0, –4.1, –4.4, –4.8, –5.1. HRMS (ESI) calcd for $C_{29}H_{57}N_4O_5Si_3$ $[M + H]^+$ 625.3631, found 625.3636.

O^6-(Allyl)-2',3',5'-tri-*O*-(*tert*-butyldimethylsilyl)inosine (4).



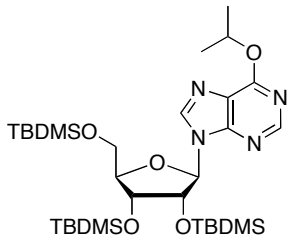
Purification using 20% EtOAc in hexanes gave 96 mg (90%) of **4** as a white solid. R_f (SiO_2/30% EtOAc in hexanes) = 0.7. ^1H NMR: δ 8.50 (s, 1H, Ar-H), 8.30 (s, 1H, Ar-H), 6.17 (m, 1H, -CH=), 6.08 (d, 1H, H-1', J = 4.8 Hz) 5.45 (d, 1H, $=CH_{trans}$, J = 17.0 Hz) 5.29 (d, 1H, $=CH_{cis}$, J = 10.2 Hz), 5.15 (br d, 2H, OCH_2, J = 5.8 Hz), 4.63 (t, 1H, H-2', J = 8.3 Hz), 4.31 (t, 1H, H-3', J = 3.4 Hz), 4.13 (br d, 1H, H-4', J = 2.4 Hz), 4.02 (dd, 1H, H-5', J = 3.4, 11.2 Hz), 3.79 (d, 1H, H-5', J = 11.2 Hz), 0.95, 0.93, and 0.78 (3s, 27H, *tert*-Bu), 0.14, 0.13, 0.10, 0.09, –0.04, and –0.23 (6s, 18H, $SiCH_3$). ^{13}C NMR: δ 160.5, 152.1, 141.3, 132.6, 122.1, 118.7, 88.5, 85.7, 76.3, 72.1, 67.7, 62.7, 26.3, 26.0, 25.8, 18.7, 18.3, 18.0, -4.1, –4.4, –4.8, –5.1. HRMS (ESI) calcd for $C_{31}H_{59}N_4O_5Si_3$ $[M + H]^+$ 651.3788, found 651.3787.

2',3',5'-Tri-*O*-(*tert*-butyldimethylsilyl)-O^6-propargylinosine (5).



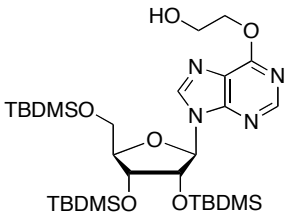
Purification using 20% EtOAc in hexanes gave 91 (86%) mg of **5** as a white solid. R_f (SiO_2/30% EtOAc in hexanes) = 0.7. ^1H NMR: δ 8.54 (s, 1H, Ar-H), 8.36 (s, 1H, Ar-H), 6.08 (d, 1H, H-1', J = 4.8 Hz), 5.23 (dd, 2H, OCH_2, J = 1.4, 2.4 Hz), 4.59 (t, 1H, H-2', J = 4.4 Hz), 4.32 (t, 1H, H-3', J = 4.1 Hz), 4.13 (q, 1H, H-4', J = 3.4 Hz), 4.00 (dd, 1H, H-5', J = 3.6, 11.4 Hz), 3.79 (dd, 1H, H-5', J = 2.4, 11.7 Hz), 2.49 (t, 1H, ≡C-H, J = 2.4 Hz), 0.95, 0.92, and 0.79 (3s, 27H, *tert*-Bu), 0.14, 0.13, 0.10, 0.09, –0.04, and –0.21 (6s, 18H, $SiCH_3$). ^{13}C NMR: δ 159.5, 152.3, 151.9, 141.7, 122.1, 88.7, 85.6, 78.3, 76.4, 75.3, 71.9, 62.6, 54.3, 26.3, 26.0, 25.8, 18.7, 18.2, 18.0, –4.1, –4.5, –4.7, –5.1. HRMS (ESI) calcd for $C_{31}H_{57}N_4O_5Si_3$ $[M + H]^+$ 649.3631, found 649.3634.

2',3',5'-Tri-*O*-(*tert*-butyldimethylsilyl)-O^6-isopropylinosine (6).[1]



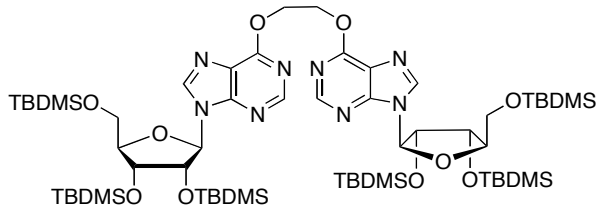
Purification 15% EtOAc in hexanes gave 80 mg (75%) of **6** as a clear gummy material. R_f (SiO_2/30% EtOAc in hexanes) = 0.7. ^{1}H NMR: δ 8.48 (s, 1H, Ar-H), 8.26 (s, 1H, Ar-H), 6.08 (d, 1H, H-1', J = 4.8 Hz), 5.66 (septet, 1H, OCH, J = 6.3 Hz), 4.63 (t, 1H, H-2', J = 4.6 Hz), 4.31 (t, 1H, H-3', J = 3.6 Hz), 4.12 (br d, 1H, H-4', J = 2.4 Hz), 4.01 (dd, 1H, H-5', J = 3.6, 11.4 Hz), 3.79 (dd, 1H, H-5', J = 1.9, 11.2 Hz), 1.47 (d, 6H, $(CH_3)_2$, J = 6.3 Hz), 0.94, 0.92, and 0.78 (3s, 27H, *tert*-Bu), 0.13, 0.12, 0.10, 0.09, –0.04, –0.22 (6s, 18H, $SiCH_3$). ^{13}C NMR: δ 160.7, 152.2, 152.1, 141.0, 122.3, 88.5, 85.6, 76.2, 72.1, 70.4, 62.7, 26.3, 26.0, 25.8, 22.1, 18.7, 18.3, 18.0, –4.1, –4.4, –4.8, –5.1.

2',3',5'-Tri-*O*-(*tert*-butyldimethylsilyl)-O^6-(2-hydroxyethyl)inosine (7).



Purification using 70% EtOAc in hexanes gave 68 mg (63%) of **7** as a white solid. R_f (SiO_2/EtOAc) = 0.3. ^{1}H NMR: δ 8.49 (s, 1H, Ar-H), 8.35 (s, 1H, Ar-H), 6.87 (d, 1H, H-1', J = 4.4 Hz), 4.72 (m, 2H, OCH_2), 4.61 (t, 1H, H-2', J = 4.4 Hz), 4.31 (m, 1H, H-3'), 4.13 (br s, 1H, H-4'), 4.02 (m, 3H, H-5'and OCH_2), 3.79 (d, 1H, H-5', J = 11.2 Hz), 3.52 (s, 1H, OH), 0.95, 0.92 and, 0.79 (3s, 27H, *tert*-Bu), 0.14, 0.13, 0.09, 0.08, –0.04, –0.22 (6s, 18H, $SiCH_3$). ^{13}C NMR: δ 160.8 152.2, 152.0, 141.6, 122.0, 88.6, 85.7, 76.4, 72.0, 69.8, 62.6, 61.9, 26.3, 26.0, 25.8, 18.7, 18.2, 18.0, –4.1, –4.5, –4.8, –5.1. HRMS (ESI) calcd for $C_{30}H_{59}N_4O_6Si_3$ $[M + H]^+$ 655.3737, found 655.3739.

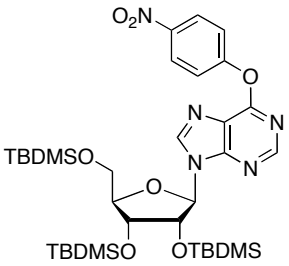
Ethylene glycol bis-O^6-[9-(2',3',5'-tri-*O*-*tert*-butyldimethylsilyl-β-D-ribofuranosyl)]purinyl ether.



Minor product obtained as a white solid (32 mg, 15%) in the reaction of **3** with ethylene glycol. R_f (SiO_2/20% EtOAc in hexanes) = 0.4. ^{1}H NMR: δ 8.49 (s, 2H, Ar-H), 8.29 (s, 2H, Ar-H), 6.09 (d, 2H, H-1', J = 4.8 Hz), 5.06 (m, 4H, OCH_2), 4.61 (t, 2H, H-2', J = 4.4 Hz), 4.31 (t, 2H, H-3', J = 2.9 Hz), 4.13 (br d, 2H, H-4', J = 2.4 Hz), 4.02 (dd, 2H, H-5', J = 3.4, 11.2), 3.79 (d, 2H, H-5', J = 11.7 Hz), 0.94, 0.92, and 0.79 (3s, 54H, *tert*-Bu), 0.13, 0.12, 0.10, 0.09, –0.04, –0.22 (6s, 36H, $SiCH_3$). ^{13}C NMR: δ 160.5, 152.2,

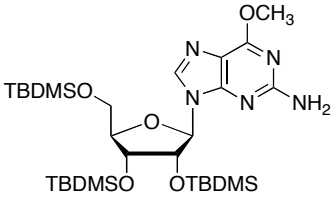
152.0, 141.3, 122.0, 88.6, 85.7, 76.3, 72.1, 64.9, 62.7, 26.3, 26.0, 25.9, 18.7, 18.3, 18.0, –4.1, –4.5, –4.7, –5.1. HRMS (ESI) calcd for $C_{58}H_{111}N_8O_{10}Si_6$ $[M + H]^+$ 1247.7033, found 1247.7032.

2',3',5'-Tri-*O*-(*tert*-butyldimethylsilyl)-O^6-(4-nitrophenyl)inosine (8).[1]



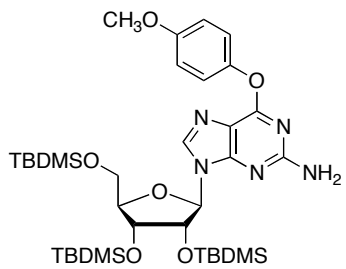
Purification using 20% EtOAc in hexanes gave 102 mg (85%) of **8** as a white solid. R_f (SiO_2/20% EtOAc in hexanes) = 0.5. ^{1}H NMR: δ 8.52 (s, 1H, Ar-H), 8.50 (s, 1H, Ar-H), 8.33 (d, 2H, Ar-H, *J* = 8.7 Hz), 7.47 (d, 2H, Ar-H, *J* = 8.7 Hz), 6.14 (d, 1H, H-1', *J* = 4.7 Hz), 4.60 (t, 1H, H-2', *J* = 4.4 Hz), 4.33 (t, 1H, H-3', *J* = 3.9 Hz), 4.17 (m, 1H, H-4'), 4.04 (dd, 1H, H-5', *J* = 3.3, 11.4), 3.82 (d, 1H, H-5', *J* = 11.3 Hz), 0.97, 0.93, and 0.81 (3s, 27H, *tert*-Bu), 0.16, 0.15, 0.11, 0.10, –0.01, and –0.18 (6s, 18H, $SiCH_3$). ^{13}C NMR: δ 159.0, 157.5, 153.5, 151.9, 145.3, 143.0, 125.6, 122.6, 122.3, 88.8, 85.7, 76.6, 71.9, 62.5, 26.3, 26.0, 25.8, 18.7, 18.2, 18.0, –4.1, –4.4, –4.7, –5.1. (The structure of this compound shown on the ^{1}H NMR spectrum in the Supporting Information to ref 1 contains a typographical error. It should have an O atom in place of the NH).

2',3',5'-Tri-*O*-(*tert*-butyldimethylsilyl)-O^6-methylguanosine (9).[2]



Purification using 20% EtOAc in hexanes gave 37 mg (73%) of **9** as an orange solid. R_f (SiO_2/20% EtOAc in hexanes) = 0.3. ^{1}H NMR: δ 7.97 (s, 1H, Ar-H), 5.91 (d, 1H, H-1', *J* = 5.3 Hz), 5.12 (s, 2H, NH_2), 4.46 (t, 1H, H-2', *J* = 4.9 Hz), 4.26 (t, 1H, H-3', *J* = 4.3 Hz), 4.08 (m, 1H, H-4'), 4.04 (s, 3H, OCH_3), 3.96 (dd, 1H, H-5', *J* = 3.9, 11.2 Hz), 3.76 (dd, 1H, H-5', *J* = 2.4, 11.2 Hz), 0.94, 0.92, and 0.79 (s, 27H, *tert*-Bu), 0.13, 0.12, 0.11, 0.09, –0.04, and –0.18 (6s, 18H, $SiCH_3$). ^{13}C NMR: δ 161.6, 159.5, 153.8, 137.9, 116.0, 87.7, 85.4, 76.4, 72.2, 62.8, 53.9, 26.2, 26.0, 25.9, 18.7, 18.2, 18.1, –4.1, –4.5, –4.8, –5.2.

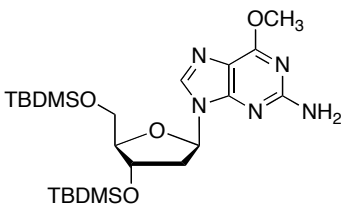
2',3',5'-Tri-*O*-(*tert*-butyldimethylsilyl)-O^6-(4-methoxyphenyl)guanosine (10).



Purification using 20% EtOAc in hexanes gave 44 mg (75%) of **10** as a white solid. R_f (SiO_2/20% EtOAc in hexanes) = 0.4. ^{1}H NMR: δ 8.08 (s, 1H, Ar-H), 7.16 (d, 2H, Ar-H, *J* = 8.8 Hz), 6.91 (d, 2H, Ar-H, *J* = 8.8 Hz), 5.92 (d, 1H, H-1', *J* = 4.5 Hz), 4.79 (s, 2H, NH_2), 4.51 (t, 1H, H-2', *J* = 4.4 Hz), 4.31 (t, 1H, H-3', *J* = 4.0 Hz), 4.16 (m, 1H, H-4'), 3.99 (dd, 1H, H-5', *J* = 3.5, 11.4 Hz), 3.82 (s, 3H, OCH_3), 3.79 (dd, 1H, H-

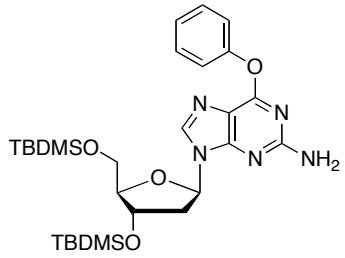
5', J = 1.9, 11.4 Hz), 0.95, 0.92, and 0.83 (3s, 27H, *tert*-Bu), 0.14, 0.13, 0.10, 0.09, 0.00, and –0.10 (6s, 18H, $SiCH_3$). ^{13}C NMR: δ 160.9, 159.3, 157.0, 154.7, 146.2, 138.9, 122.9, 116.1, 114.4, 88.2, 85.1, 76.3, 71.9, 62.6, 55.7, 26.3, 26.0, 25.9, 18.7, 18.7, 18.2, 18.1, –4.1, –4.4, –4.6, –5.1. HRMS (ESI) calcd for $C_{35}H_{62}N_5O_6Si_3$ $[M + H]^+$ 732.4002, found 732.4007.

3',5'-Di-*O*-(*tert*-butyldimethylsilyl)-O^6-methyl-2'-deoxyguanosine (11).[2]



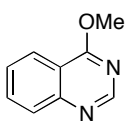
Purification using 20% EtOAc in hexanes gave 38 mg (75%) of **11** as a yellowish solid (from 0.1 mmol of 3',5'-di-*O*-TBDMS 2'-deoxyguanosine). R_f (SiO_2/40% EtOAc in hexanes) = 0.3. ^1H NMR: δ 7.70 (s, 1H, Ar-H), 6.31 (t, 1H, H-1', J = 6.3 Hz), 4.89 (s, 2H, NH_2), 4.57 (m, 1H, H-3'), 4.05 (s, 3H, OCH_3), 3.89 (m, 1H, H-4'), 3.80 (dd, 1H, H-5', J = 4.4, 11.2 Hz), 3.74 (dd, 1H, H-5', J = 2.9, 11.2 Hz), 2.55 (app quint, 1H, H-2', J_{app} ~ 6.3 Hz), 2.34 (ddd, 1H, H-2', J = 2.2, 6.0, 13.1 Hz), 0.90 (br s, 18H, *tert*-Bu), 0.08, 0.07, and 0.06 (3s, 12H, $SiCH_3$). ^{13}C NMR: δ 161.7, 159.5, 153.6, 137.7, 116.1, 87.8, 83.7, 72.0, 63.0, 53.9, 41.1, 26.1, 25.9, 18.6, 18.1, –4.4, –4.5, –5.2, –5.3.

3',5'-Di-*O*-(*tert*-butyldimethylsilyl)-O^6-phenyl-2'-deoxyguanosine (12).[2]



Purification using 20% EtOAc in hexanes gave 42 mg (73%) of **12** as a yellowish solid (from 0.1 mmol of 3',5'-di-*O*-TBDMS 2'-deoxyguanosine). R_f (SiO_2/40% EtOAc in hexanes) = 0.3. ^1H NMR: δ 8.00 (s, 1H, Ar-H), 7.40 (t, 2H, Ar-H, J = 7.7 Hz), 7.25 (m, 3H, Ar-H), 6.34 (t, 1H, H-1', J = 6.4 Hz), 4.77 (s, 2H, NH_2), 4.60 (m, 1H, H-3'), 3.99 (m, 1H, H-4'), 3.83 (dd, 1H, H-5', J = 4.1, 11.1 Hz), 3.77 (dd, 1H, H-5', J = 2.6, 11.1 Hz), 2.59 (app quint, 1H, H-2', J_{app} ~ 6.4 Hz), 2.37 (ddd, 1H, H-2', J = 1.4, 5.8, 12.9 Hz), 0.923 and 0.920 (2s, 18H, *tert*-Bu), 0.10 and 0.09 (2s, 12H, $SiCH_3$). ^{13}C NMR: δ 160.6, 159.3, 154.7, 152.7, 138.7, 129.4, 125.4, 122.1, 116.2, 87.9, 83.9, 72.1, 63.0, 41.2, 26.1, 25.9, 18.6, 18.2, –4.4, –4.5, –5.1, –5.2.

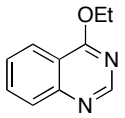
4-Methoxyquinazoline (16a).[3,4]



Purification using 15% EtOAc in hexanes gave 89 mg (81%) of **16a** as a colorless oil (solidifies at 0 °C). R_f (SiO_2/20% EtOAc in hexanes) = 0.4. ^1H NMR: δ 8.77 (s, 1H, Ar-H), 8.10 (d, 1H, Ar-H, J = 8.3 Hz), 7.89 (d, 1H, Ar-H, J = 8.5 Hz), 7.77 (dt, 1H,

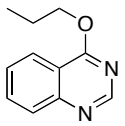
Ar-H, J = 1.3, 8.3 Hz), 7.50 (t, 1H, Ar-H, J = 7.8 Hz), 4.13 (s, 3H, OCH_3). ^{13}C NMR: δ 167.2, 154.5, 151.0, 133.6, 127.8, 127.1, 123.6, 116.7, 54.4.

4-Ethoxyquinazoline (16b).[4]



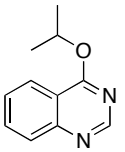
Purification using 15% EtOAc in hexanes gave 92 mg (77%) of **16b** as a colorless oil (solidifies at 0 °C). R_f (SiO_2/20% EtOAc in hexanes) = 0.4. ^1H NMR: δ 8.79 (s, 1H, Ar-H), 8.18 (d, 1H, Ar-H, J = 8.3 Hz), 7.92 (d, 1H, Ar-H, J = 8.3 Hz), 7.82 (t, 1H, Ar-H, J = 7.3 Hz), 7.55 (t, 1H, Ar-H, J = 7.5 Hz), 4.64 (q, 2H, OCH_2, J = 7.3 Hz), 1.52 (t, 3H, CH_3, J = 7.3 Hz). ^{13}C NMR: δ 166.9, 154.6, 151.1, 133.6, 127.8, 127.0, 123.7, 116.9, 63.2, 14.5.

4-Propoxyquinazoline (16c).[4]



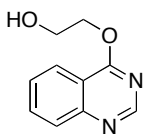
Purification using 15% EtOAc in hexanes gave 101 mg (78%) of **16c** as a colorless oil. R_f (SiO_2/30% EtOAc in hexanes) = 0.5. ^1H NMR: δ 8.78 (s, 1H, Ar-H), 8.17 (d, 1H, Ar-H, J = 8.3 Hz), 7.91 (d, 1H, Ar-H, J = 8.3 Hz), 7.81 (dt, 1H, Ar-H, J = 1.4, 7.6 Hz), 7.54 (t, 1H, Ar-H, J = 7.5 Hz), 4.52 (t, 2H, OCH_2, J = 6.8 Hz), 1.92 (sextet, 2H, CH_2, J = 7.5), 1.09 (t, 3H, CH_3, J = 7.5 Hz). ^{13}C NMR: δ 166.9, 154.6, 151.0, 133.5, 127.8, 127.0, 123.6, 116.8, 68.8, 22.2, 10.6.

4-Isopropoxyquinazoline (16d).[5]



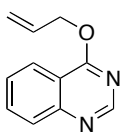
Purification using 15% EtOAc in hexanes gave 90 mg (70%) of **16d** as a yellowish oil. R_f (SiO_2/30% EtOAc in hexanes) = 0.6. ^1H NMR: δ 8.77 (s, 1H, Ar-H), 8.15 (d, 1H, Ar-H, J = 8.3 Hz), 7.89 (d, 1H, Ar-H, J = 8.3 Hz), 7.79 (t, 1H, Ar-H, J = 7.5 Hz), 7.52 (t, 1H, Ar-H, J = 7.5 Hz), 5.62 (septet, 1H, OCH, J = 6.3 Hz), 1.45 (d, 6H, $(CH_3)_2$, J = 6.3 Hz). ^{13}C NMR: δ 166.4, 154.6, 151.1, 133.4, 127.7, 126.8, 123.8, 117.2, 70.3, 22.0.

2-(4-Quinazolinyloxy)ethanol (16e).



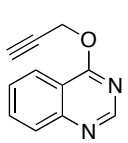
Purification using 70% EtOAc in hexanes gave 100 mg (77%) of **16e** as a white solid. R_f (SiO_2/100% EtOAc) = 0.2. ^1H NMR: δ 8.76 (s, 1H, Ar-H), 8.16 (d, 1H, Ar-H, J = 8.3 Hz), 7.93 (d, 1H, Ar-H, J = 8.3 Hz), 7.81 (td, 1H, Ar-H, J = 1.4, 8.3 Hz), 7.55 (t, 1H, Ar-H, J = 7.5 Hz), 4.75 (t, 2H, OCH_2, J = 4.4 Hz), 4.08 (br s, 2H, CH_2OH), 3.36 (br s, 1H, OH). ^{13}C NMR: δ 167.1, 154.0, 151.1, 134.0, 127.9, 127.4, 123.7, 116.6, 69.7, 61.9. HRMS (ESI) calcd for $C_{10}H_{11}N_2O_2$ $[M + H]^+$ 191.0815, found 191.0815.

4-(Allyloxy)quinazoline (16f).[6]



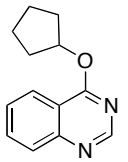
Purification using 20% EtOAc in hexanes gave 96 mg (75%) of **16f** as a yellowish liquid. R_f (SiO_2/30% EtOAc in hexanes) = 0.6. ^{1}H NMR: δ 8.79 (s, 1H, Ar-H), 8.20 (d, 1H, Ar-H, J = 8.3 Hz), 7.93 (d, 1H, Ar-H, J = 8.3 Hz), 7.82 (td, 1H, Ar-H, J = 1.4, 8.3 Hz), 7.56 (t, 1H, Ar-H, J = 7.5 Hz), 6.16 (m, 1H, -CH=), 5.48 (dd, 1H, $=CH_{trans}$, J = 1.4, 17.0 Hz), 5.33 (dd, 1H, $=CH_{cis}$, J = 1.4, 10.7 Hz), 5.10 (d, 2H, OCH_2, J = 5.3 Hz). ^{13}C NMR: δ 166.5, 154.5, 151.2, 133.6, 132.5, 127.9, 127.2, 123.7, 118.6, 116.8, 67.6.

4-(2-Propynyloxy)quinazoline (16g).[6]



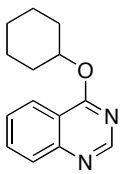
Purification using 20% EtOAc in hexanes gave 94 mg (75%) of **16g** as a white solid. R_f (SiO_2/20% EtOAc in hexanes) = 0.5. ^{1}H NMR: δ 8.81 (s, 1H, Ar-H), 8.19 (d, 1H, Ar-H, J = 7.8 Hz), 7.88 (d, 1H, Ar-H, J = 8.3 Hz), 7.83 (t, 1H, Ar-H, J = 8.3 Hz), 7.56 (t, 1H, Ar-H, J = 7.5 Hz), 5.21 (s, 2H, OCH_2), 2.55 (s, 1H, ≡CH). ^{13}C NMR: δ 165.7, 154.1, 151.2, 133.9, 127.9, 127.4, 123.6, 116.5, 78.1, 75.4, 54.5.

4-(Cyclopentyloxy)quinazoline (16h).



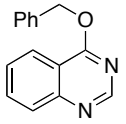
Purification using 20% EtOAc in hexanes gave 115 mg (78%) of **16h** as a colorless oil. R_f (SiO_2/30% EtOAc in hexanes) = 0.5. ^{1}H NMR: δ 8.78 (s, 1H, Ar-H), 8.12 (d, 1H, Ar-H, J = 8.3 Hz), 7.89 (d, 1H, Ar-H, J = 8.3 Hz), 7.79 (t, 1H, Ar-H, J = 8.3 Hz), 7.51 (t, 1H, Ar-H, J = 7.5 Hz), 5.72 (m, 1H, OCH), 2.08–2.01 (m, 2H, CH_2), 1.95–1.89 (m, 2H, CH_2), 1.88–1.81 (m, 2H, CH_2), 1.73–1.65 (m, 2H, CH_2). ^{13}C NMR: δ 166.7, 154.7, 151.1, 133.5, 127.8, 126.9, 123.8, 117.3, 79.9, 33.0, 24.1. HRMS (ESI) calcd for $C_{13}H_{15}N_2O$ [M + H]$^+$ 215.1179, found 215.1176.

4-(Cyclohexyloxy)quinazoline (16i).[7]



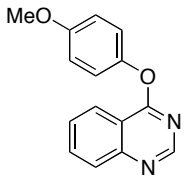
Purification using 20% EtOAc in hexanes gave 120 mg (77%) of **16i** as a colorless oil. R_f (SiO_2/30% EtOAc in hexanes) = 0.5. ^{1}H NMR: δ 8.77 (s, 1H, Ar-H), 8.17 (d, 1H, Ar-H, J = 7.8 Hz), 7.90 (d, 1H, Ar-H, J = 8.3 Hz), 7.80 (t, 1H, Ar-H, J = 8.0 Hz), 7.53 (t, 1H, Ar-H, J = 7.5 Hz), 5.41 (m, 1H, OCH), 2.08–2.05 (m, 2H, CH_2), 1.85–1.82 (m, 2H, CH_2), 1.73–1.66 (m, 2H, CH_2), 1.62–1.58 (m, 1H, CH_2), 1.54–1.46 (m, 2H, CH_2), 1.42–1.37 (m, 1H, CH_2). ^{13}C NMR: δ 166.5, 154.7, 151.2, 133.5, 127.8, 126.9, 123.8, 117.3, 75.0, 31.6, 25.7, 23.8.

4-(Benzyloxy)quinazoline (16j).[8]



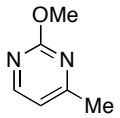
Purification using 5% MeOH in hexanes gave 155 mg (96%) of **16j** as a colorless oil. R_f (SiO_2/40% EtOAc in hexanes) = 0.4. ^{1}H NMR: δ 8.83 (s, 1H, Ar-H), 8.20 (dd, 1H, Ar-H, *J* = 0.7, 8.2 Hz), 7.94 (d, 1H, Ar-H, *J* = 8.4 Hz), 7.82 (td, 1H, Ar-H, *J* = 1.4, 7.0 Hz), 7.54 (m, 3H, Ar-H), 7.41 (t, 2H, Ar-H, *J* = 7.2 Hz), 7.36 (t, 1H, Ar-H, *J* = 7.2 Hz), 5.64 (br s, 2H, OCH_2). ^{13}C NMR: δ 166.7, 154.5, 151.2, 136.3, 133.8, 128.8, 128.5, 128.3, 127.9, 127.2, 123.7, 116.8, 68.7.

4-(4-Methoxyphenoxy)quinazoline (16k).[9]



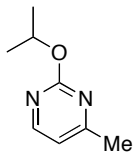
Purification using 30% EtOAc in hexanes gave 165 mg (96%) of **16k** as a white solid. R_f (SiO_2/30% EtOAc in hexanes) = 0.5. ^{1}H NMR: δ 8.77 (s, 1H, Ar-H), 8.36 (d, 1H, Ar-H, *J* = 8.1 Hz), 7.99 (d, 1H, Ar-H, *J* = 8.4), 7.89 (t, 1H, Ar-H, *J* = 7.1 Hz), 7.63 (t, 1H, Ar-H, *J* = 7.5 Hz), 7.18 (br d, 2H, Ar-H, *J* = 8.9 Hz), 6.98 (br d, 2H, Ar-H, *J* = 8.9 Hz), 3.83 (s, 3H, OCH_3). ^{13}C NMR: δ 167.4, 157.5, 154.5, 151.7, 145.8, 134.1, 128.0, 127.6, 123.7, 122.8, 116.6, 114.9, 55.7.

2-(Methoxy)-4-methylpyrimidine (17a).[10]



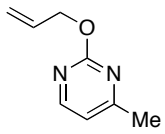
Purification using 15% EtOAc in hexanes gave 65 mg (77%) of **17a** as a clear oil. R_f (SiO_2/20% EtOAc in hexanes) = 0.2. ^{1}H NMR: δ 8.33 (d, 1H, Ar-H, *J* = 4.8 Hz), 6.78 (d, 1H, Ar-H, *J* = 4.8 Hz), 3.98 (s, 3H, OCH_3), 2.44 (s, 3H, CH_3). ^{13}C NMR: δ 170.2, 165.7, 158.7, 114.6, 54.8, 24.2.

2-(Isopropoxy)-4-methylpyrimidine (17b).



Purification using 15% EtOAc in hexanes gave 76 mg (73%) of **17b** as a yellowish oil. R_f (SiO_2/20% EtOAc in hexanes) = 0.5. ^{1}H NMR: δ 8.31 (d, 1H, Ar-H, *J* = 4.8 Hz), 6.74 (d, 1H, Ar-H, *J* = 4.8 Hz), 5.27 (septet, 1H, OCH, *J* = 6.3 Hz), 2.43 (s, 3H, CH_3), 1.38 (d, 6H, $(CH_3)_2$, *J* = 6.3 Hz). ^{13}C NMR: δ 170.1, 164.9, 158.6, 114.2, 70.0, 24.3, 22.1. HRMS (ESI) calcd for $C_8H_{13}N_2O$ $[M + H]^+$ 153.1022, found 153.1033.

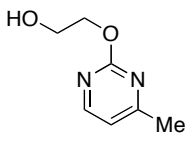
2-(Allyloxy)-4-methylpyrimidine (17c).



Purification using 20% EtOAc in hexanes gave 77 mg (75%) of **17c** as a yellowish oil. R_f (SiO_2/40% EtOAc in hexanes) = 0.5. ^{1}H NMR: δ 8.31 (d, 1H, Ar-H, *J* = 4.8 Hz), 6.76 (d, 1H, Ar-H, *J* = 4.8 Hz), 6.06 (m, 1H, -CH=), 5.39 (d,

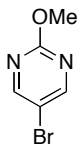
1H, =CH_{trans}, J = 17.5 Hz), 5.22 (d, 1H, =CH_{cis}, J = 10.2 Hz), 4.86 (d, 2H, OCH_2, J = 5.3 Hz), 2.43 (s, 3H, Ar-CH_3). ^{13}C NMR: δ 170.2, 165.0, 158.6, 133.0, 117.8, 114.7, 68.0, 24.2. HRMS (ESI) calcd for $C_8H_{11}N_2O$ $[M + H]^+$ 151.0866, found 151.0861.

2-[(4-Methylpyrimidin-2-yl)oxy]ethanol (17d).



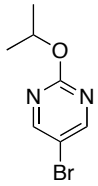
Purification using 60% EtOAc in hexanes gave 74 mg (70%) of **17d** as a pale yellow liquid. R_f (SiO_2/EtOAc) = 0.3. ^{1}H NMR: δ 8.34 (d, 1H, Ar-H, J = 4.9 Hz), 6.82 (d, 1H, Ar-H, J = 4.9 Hz), 4.49 (t, 2H, OCH_2, J = 4.3 Hz), 3.97 (t, 2H, OCH_2, J = 4.3 Hz), 2.97 (br s, 1H, OH), 2.46 (s, 3H, CH_3). ^{13}C NMR: δ 170.4, 165.2, 158.7, 115.0, 69.6, 62.0, 24.2. HRMS (ESI) calcd for $C_7H_{11}N_2O_2$ $[M + H]^+$ 155.0815, found 155.0814.

5-Bromo-2-methoxypyrimidine (18a).[11]



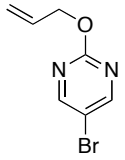
Purification using 30% EtOAc in hexanes gave 85 mg (78%) of **18a** as a colorless liquid. R_f (SiO_2/40% EtOAc in hexanes) = 0.5. ^{1}H NMR: δ 8.52 (s, 2H, Ar-H), 3.98 (s, 3H, OCH_3). ^{13}C NMR: δ 164.4, 159.7, 112.0, 55.6.

5-Bromo-2-isopropoxypyrimidine (18b).[12]



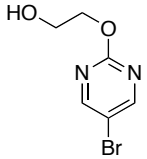
Purification using 30% EtOAc in hexanes gave 100 mg (80%) of **18b** as a clear oil. R_f (SiO_2/40% EtOAc in hexanes) = 0.6. ^{1}H NMR: δ 8.48 (s, 2H, Ar-H), 5.19 (septet, 1H, OCH, J = 6.3 Hz), 1.37 (d, 6H, $(CH_3)_2$, J = 6.3 Hz). ^{13}C NMR: δ 163.6, 159.7, 111.3, 71.3, 21.8.

2-(Allyloxy)-5-bromopyrimidine (18c).[13]



Purification using 30% EtOAc in hexanes gave 97 mg (79%) of **18c** as a yellowish solid. R_f (SiO_2/40% EtOAc in hexanes) = 0.5. ^{1}H NMR: δ 8.50 (s, 2H, Ar-H), 6.06 (m, 1H, -CH=), 5.41 (dq, 1H, =CH_{trans}, J = 1.5, 17.2 Hz), 5.39 (dd, 1H, =CH_{cis}, J = 1.2, 10.5 Hz), 4.72 (dt, 2H, OCH_2, J = 1.4, 6.8 Hz). ^{13}C NMR: δ 163.7, 159.7, 132.3, 118.4, 112.0, 68.8.

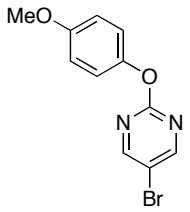
2-(5-Bromopyrimidin-2-yloxy)ethanol (18d).



Purification using 40% EtOAc in hexanes gave 94 mg (75%) of **18d** as a white solid. R_f (SiO_2/40% EtOAc in hexanes) = 0.4. ^{1}H NMR: δ 8.51 (s, 2H, Ar-H), 4.45 (t, 2H, OCH_2, J = 4.6 Hz), 3.94–3.97 (m, 2H, OCH_2), 2.98 (t, 1H, OH, J = 6.3 Hz).

^{13}C NMR: δ 164.0, 159.9, 112.3, 70.1, 61.4. HRMS (ESI) calcd for $C_6H_8BrN_2O_2$ $[M + H]^+$ 218.9764, found 218.9763.

5-Bromo-2-(4-methoxyphenoxy)pyrimidine (18e).[14]



Purification using 20% EtOAc in hexanes gave 114 mg (71%) of **18e** as a white solid. R_f (SiO_2/20% EtOAc in hexanes) = 0.4. ^{1}H NMR: δ 8.55 (s, 2H, Ar-H), 7.09 (d, 2H, Ar-H, J = 8.9 Hz), 6.93 (d, 2H, Ar-H, J = 8.9 Hz), 3.81 (s, 3H, OCH_3). ^{13}C NMR: δ 164.4, 160.1, 157.3, 146.3, 122.4, 114.8, 113.1, 55.7.

1203-hp-01-050

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Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-050
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
10 repetitions
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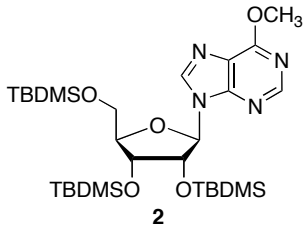
Total time 0 min, 34 sec



2

1203-hp-01-046

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Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-046
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7707222 MHz
DATA PROCESSING
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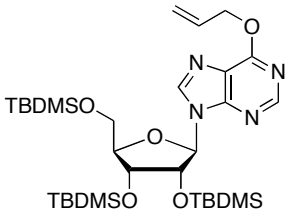
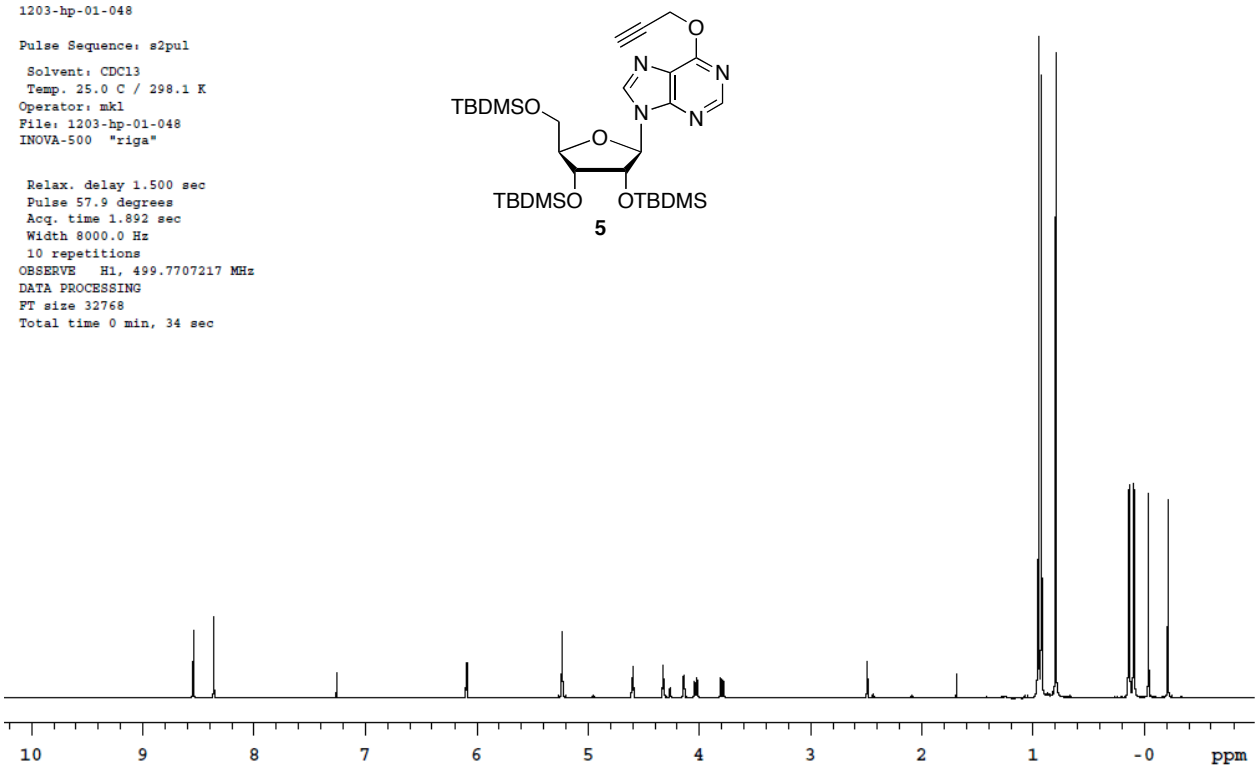
Total time 0 min, 54 sec



4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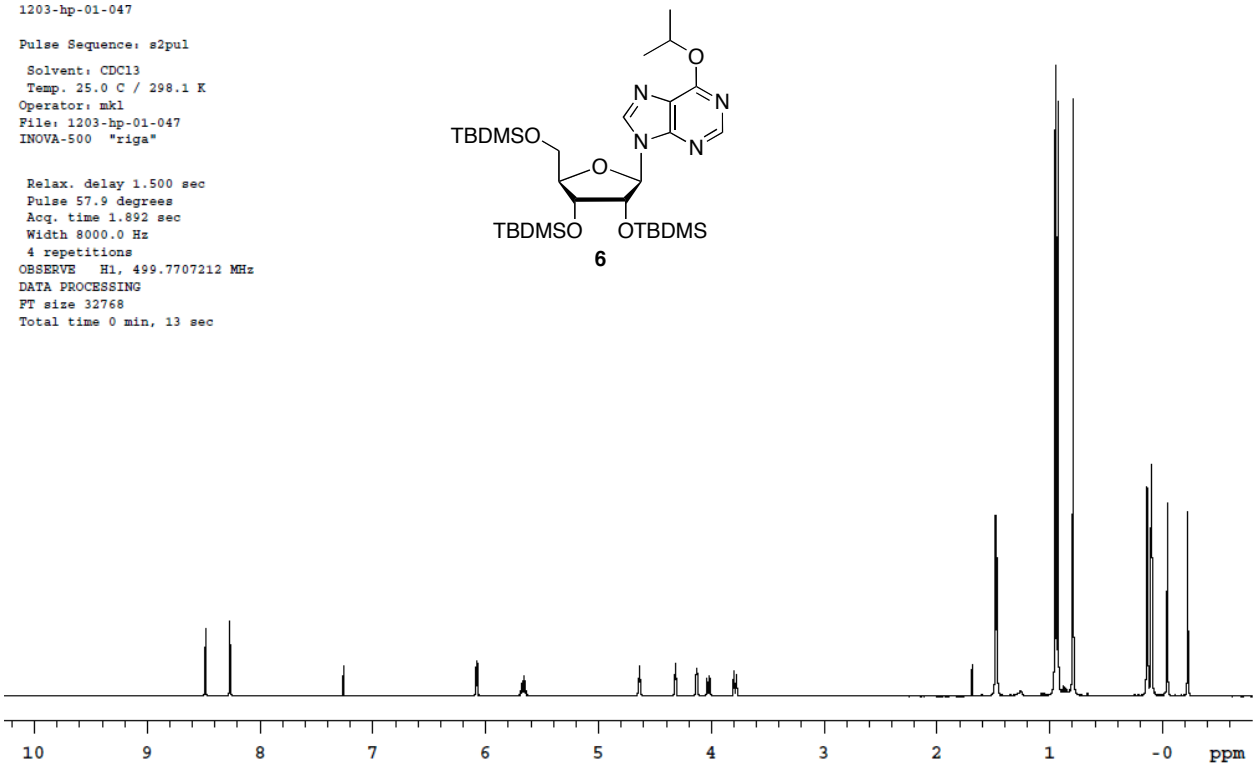
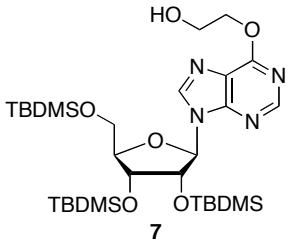
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-048
INOVA-500 "riga"
Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
10 repetitions
OBSERVE H1, 499.7707217 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 34 sec
TBDMSO
TBDMSO
OTBDMS
5
10
9
8
7
6
5
4
3
2
1
-0
ppm


1203-hp-01-047
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-047
INOVA-500 "riga"
Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
4 repetitions
OBSERVE H1, 499.7707212 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 13 sec
TBDMSO
TBDMSO
OTBDMS
6
10
9
8
7
6
5
4
3
2
1
-0
ppm

1203-HP-01-049b

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-049b
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
10 repetitions
OBSERVE H1, 499.7707222 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 34 sec

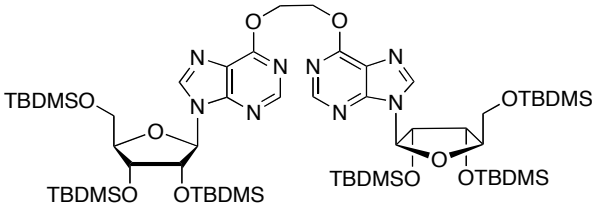


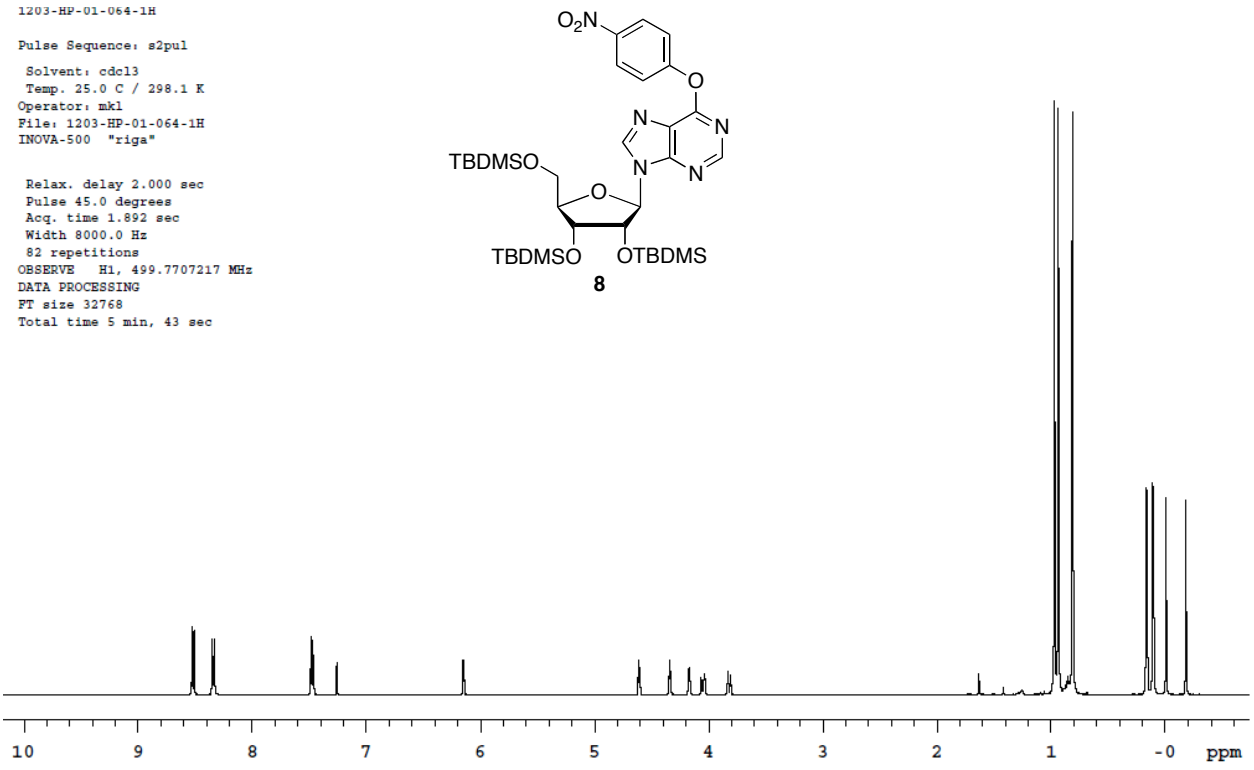
7

1203-HP-01-049t

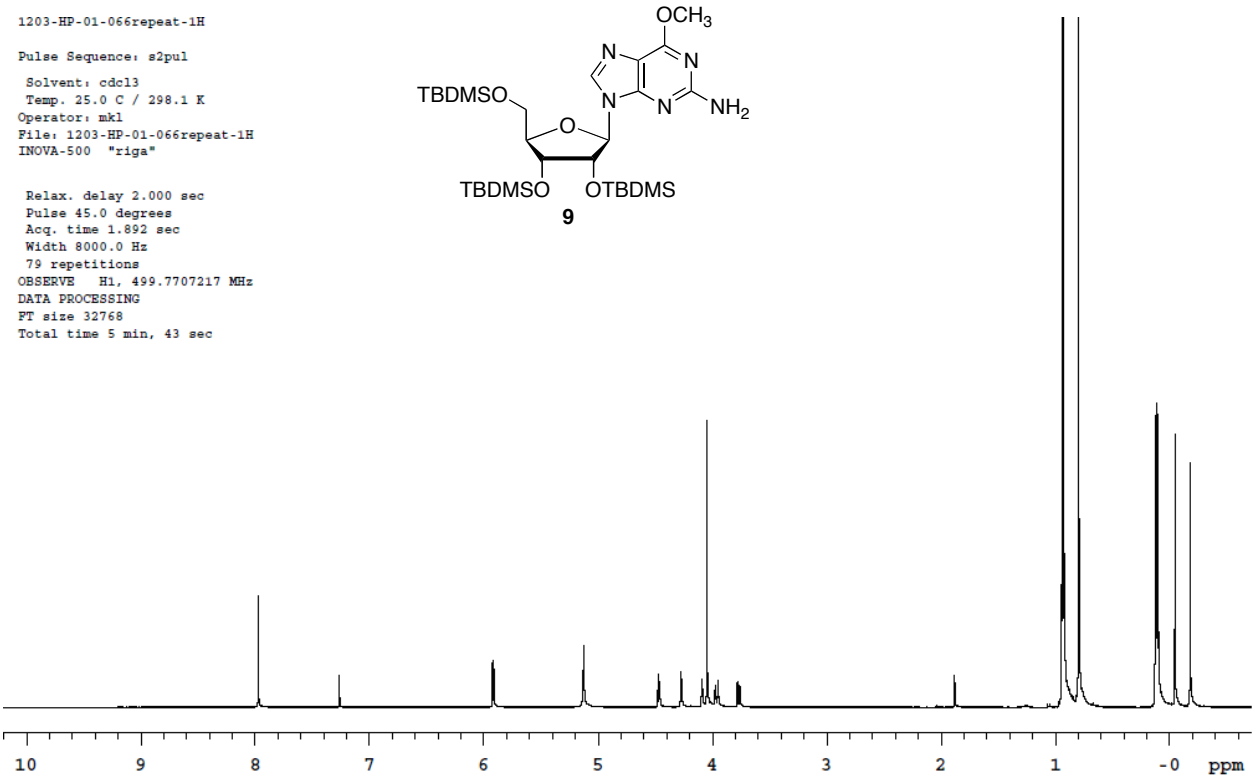
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-049t
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
10 repetitions
OBSERVE H1, 499.7707222 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 34 sec




1203-HP-01-064-1H
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-064-1H
INOVA-500 "riga"
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
82 repetitions
OBSERVE H1, 499.7707217 MHz
DATA PROCESSING
FT size 32768
Total time 5 min, 43 sec
O2N
O
N
N
N
N
TBDMSO
O
TBDMSO
OTBDMS
10
9
8
7
6
5
4
3
2
1
-0
ppm

8


1203-HP-01-066repeat-1H
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-066repeat-1H
INOVA-500 "riga"
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
79 repetitions
OBSERVE H1, 499.7707217 MHz
DATA PROCESSING
FT size 32768
Total time 5 min, 43 sec
OCH3
N
N
N
N
NH2
TBDMSO
O
TBDMSO
OTBDMS
10
9
8
7
6
5
4
3
2
1
-0
ppm

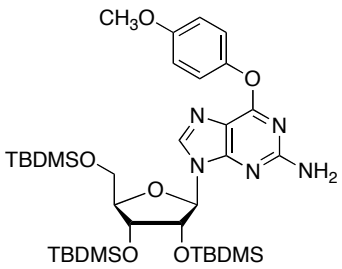
9

1203-HP-01-065-1H

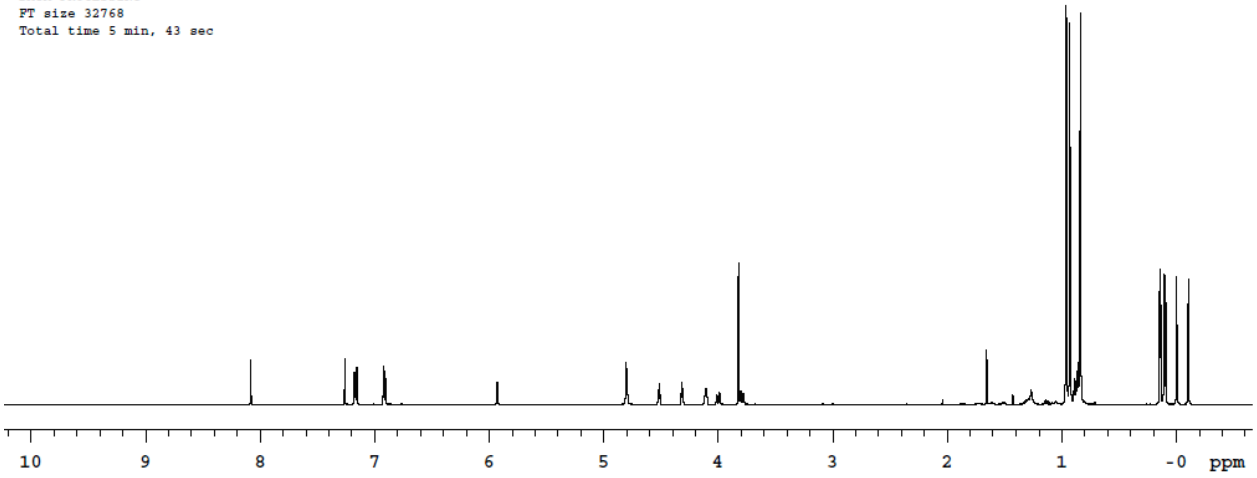
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-065-1H
INOVA-500 "riga"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
85 repetitions
OBSERVE H1, 499.7707217 MHz
DATA PROCESSING
FT size 32768
Total time 5 min, 43 sec



10

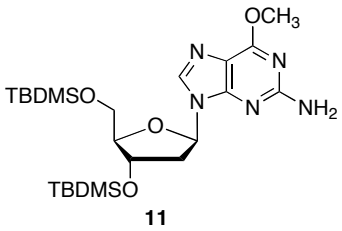


1203-HP-01-068repeat-1H

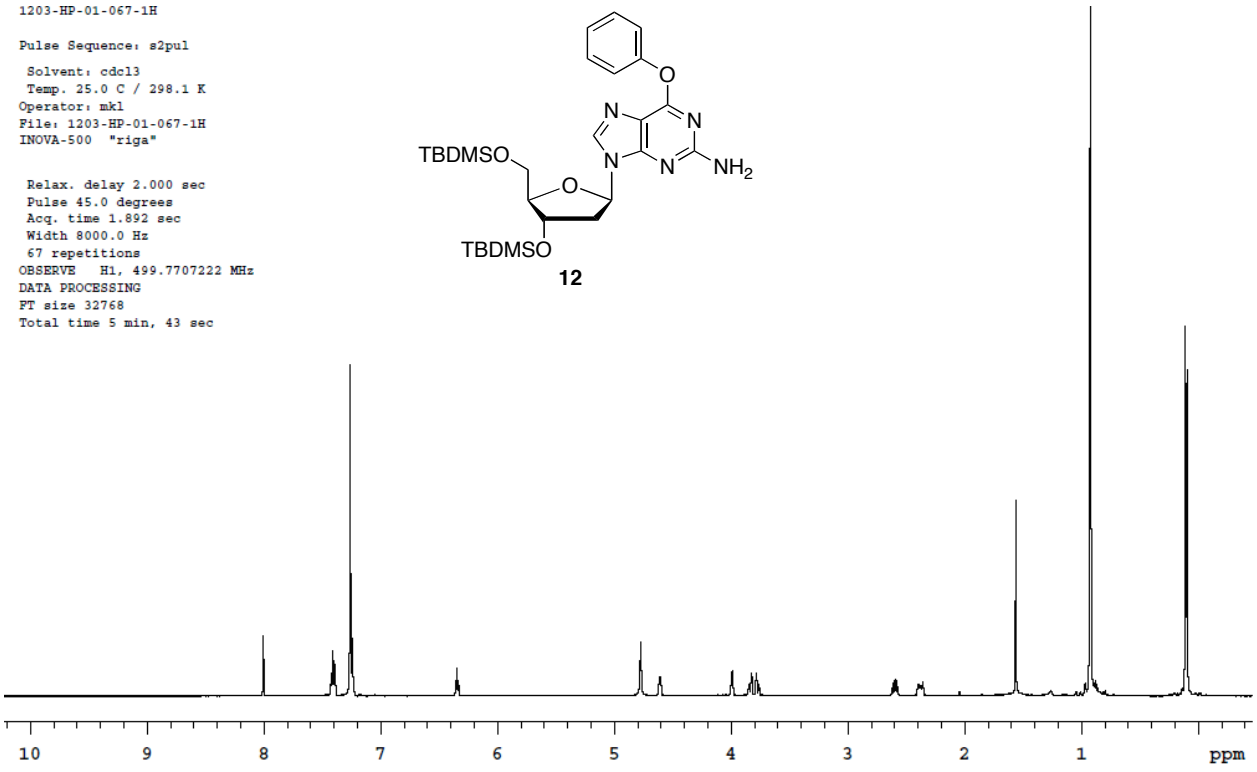
Pulse Sequence: s2pul

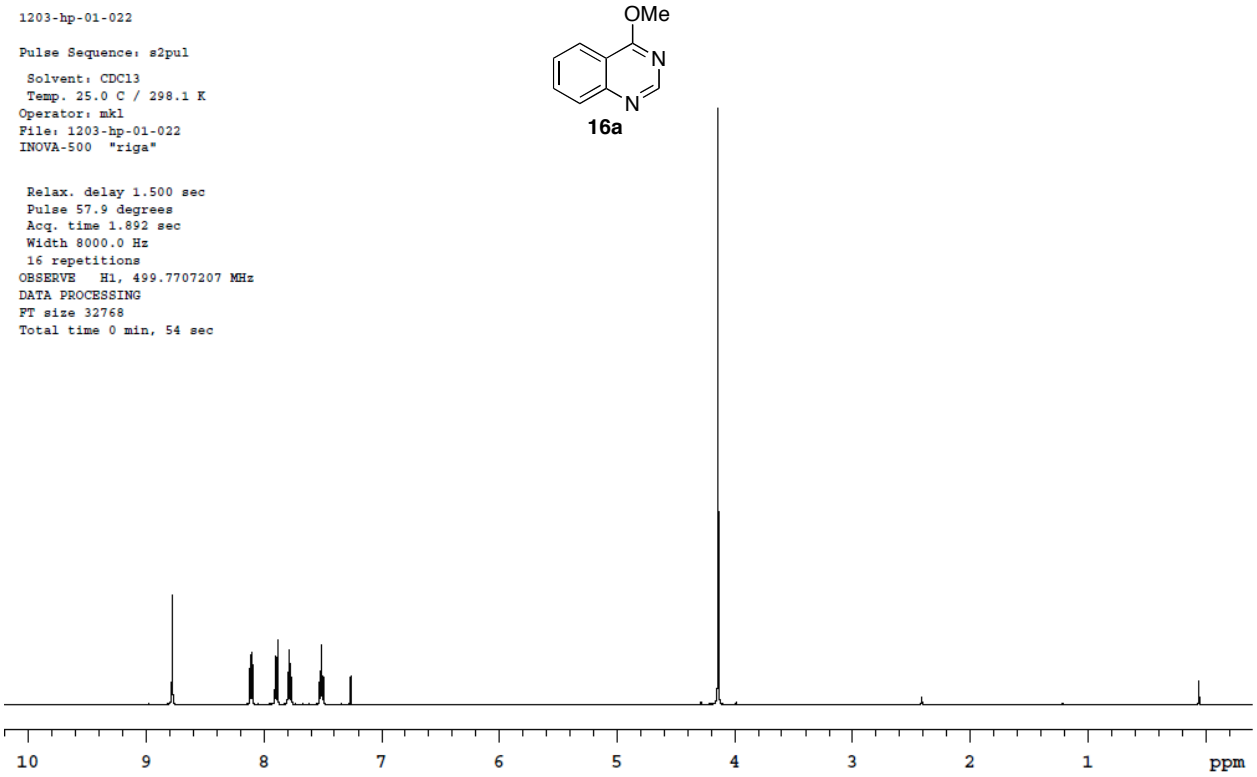
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-068repeat-1H
INOVA-500 "riga"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
88 repetitions
OBSERVE H1, 499.7707217 MHz
DATA PROCESSING
FT size 32768
Total time 5 min, 43 sec



11

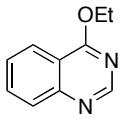

1203-HP-01-067-1H
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-067-1H
INOVA-500 "riga"
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
67 repetitions
OBSERVE H1, 499.7707222 MHz
DATA PROCESSING
FT size 32768
Total time 5 min, 43 sec
TBDMSO
TBDMSO
NH2
12
10
9
8
7
6
5
4
3
2
1
ppm


1203-hp-01-022
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-022
INOVA-500 "riga"
Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7707207 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 54 sec
OMe
16a
10
9
8
7
6
5
4
3
2
1
ppm

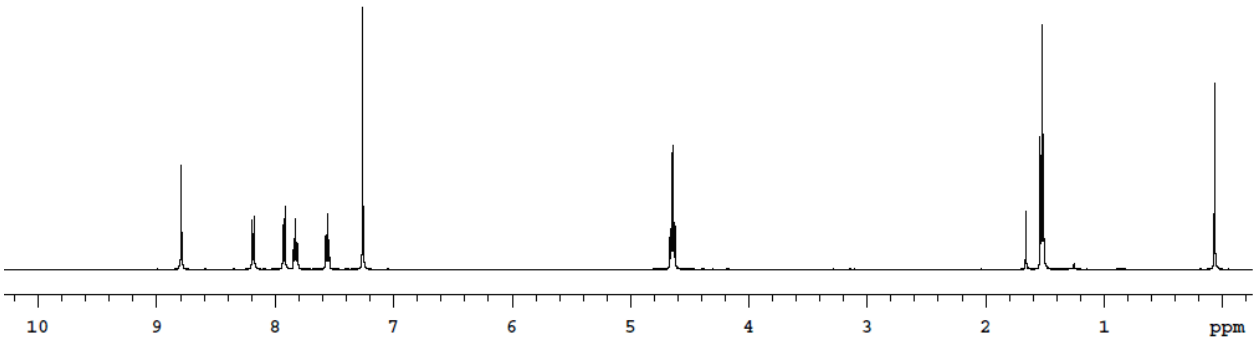
1203-HP-01-023

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-023
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
12 repetitions
OBSERVE H1, 499.7707212 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 11 min, 20 sec



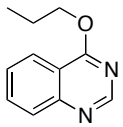
16b



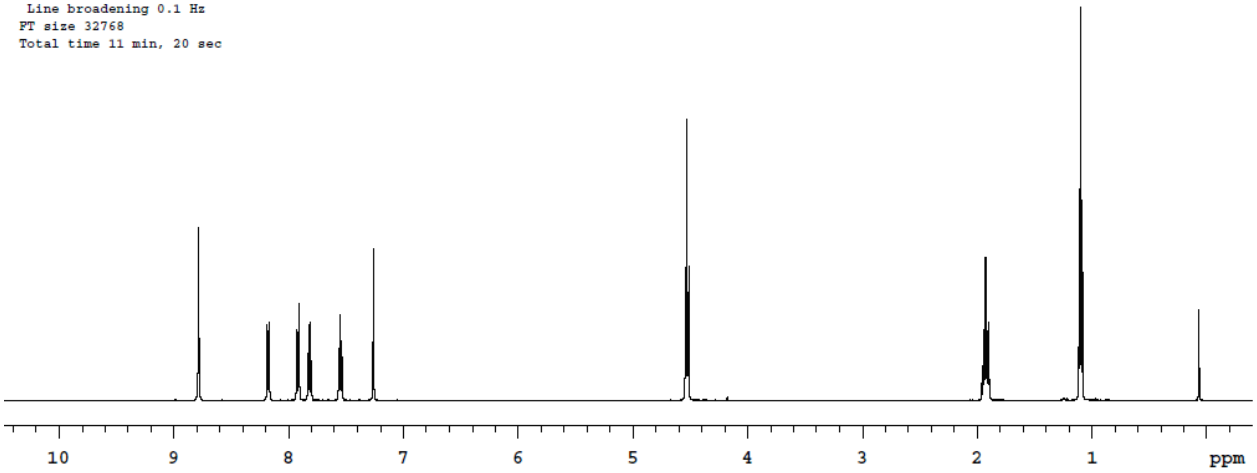
1203-HP-01-024

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-024
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7707212 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 11 min, 20 sec



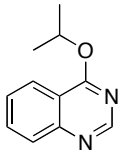
16c



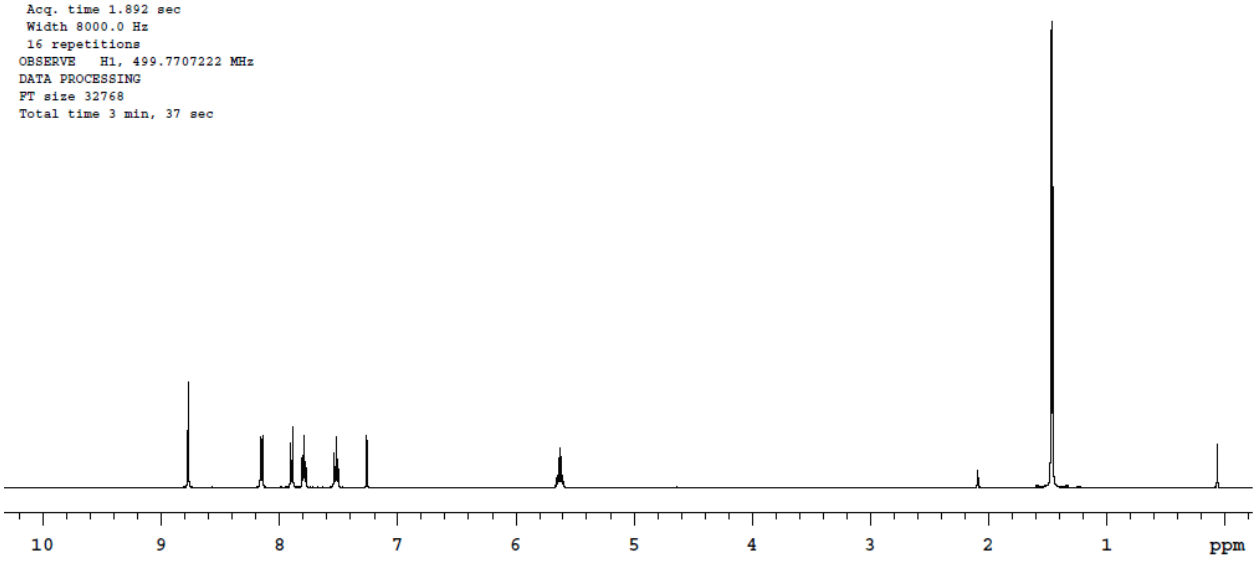
1203-hp-01-025

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-025
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7707222 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 37 sec



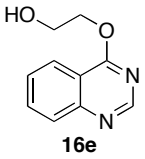
16d



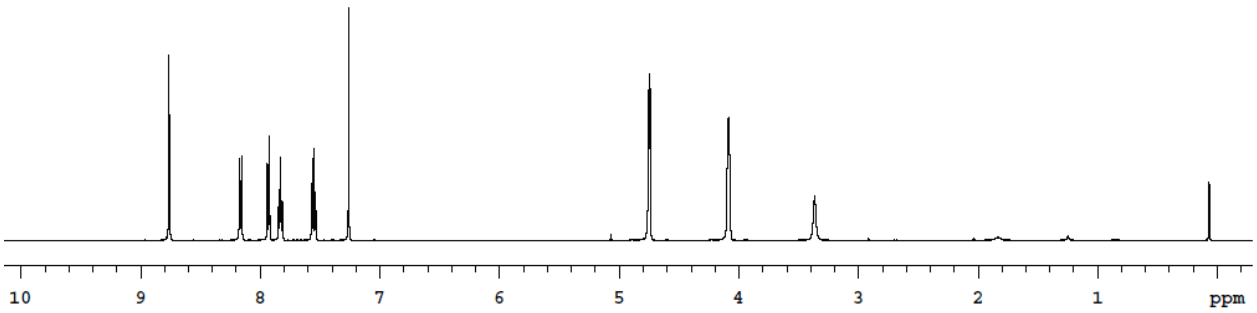
1203-HP-01-028m

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-028m
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7707212 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 37 sec



16e

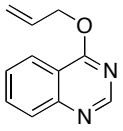


1203-HP-01-029

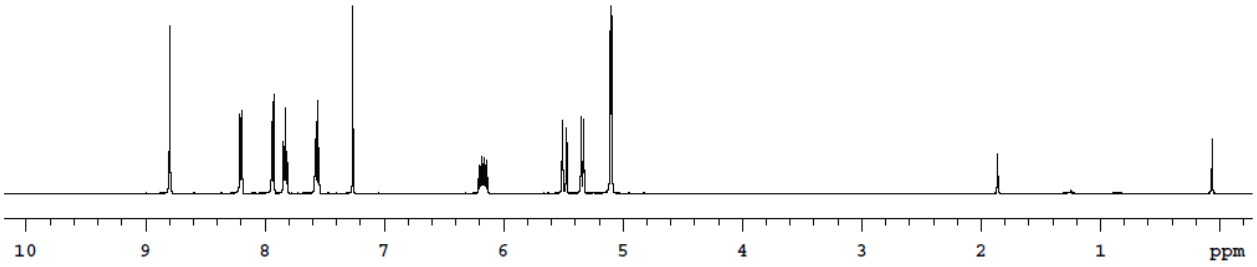
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-029
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
164 repetitions
OBSERVE H1, 499.7707212 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 11 min, 20 sec



16f

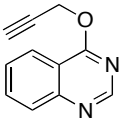


1203-HP-01-030

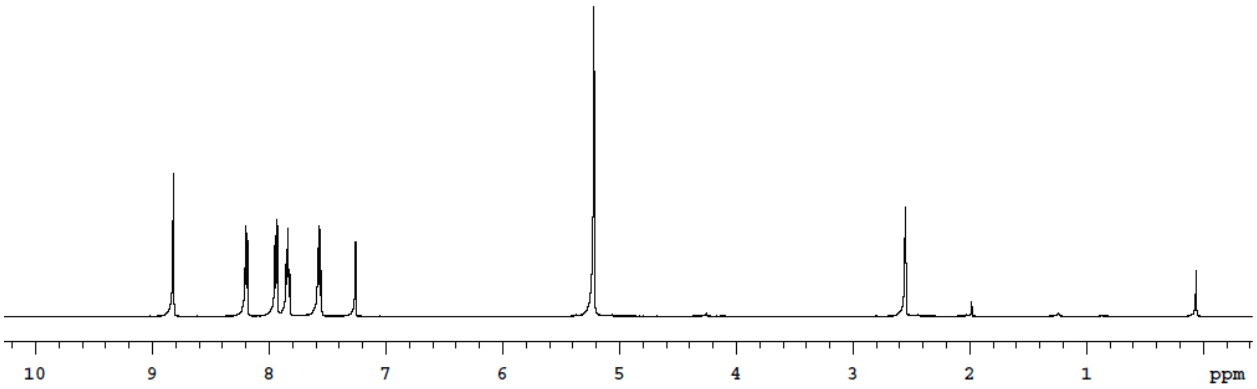
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-030
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
20 repetitions
OBSERVE H1, 499.7707212 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 11 min, 20 sec



16g

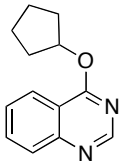


1203-hp-01-032p

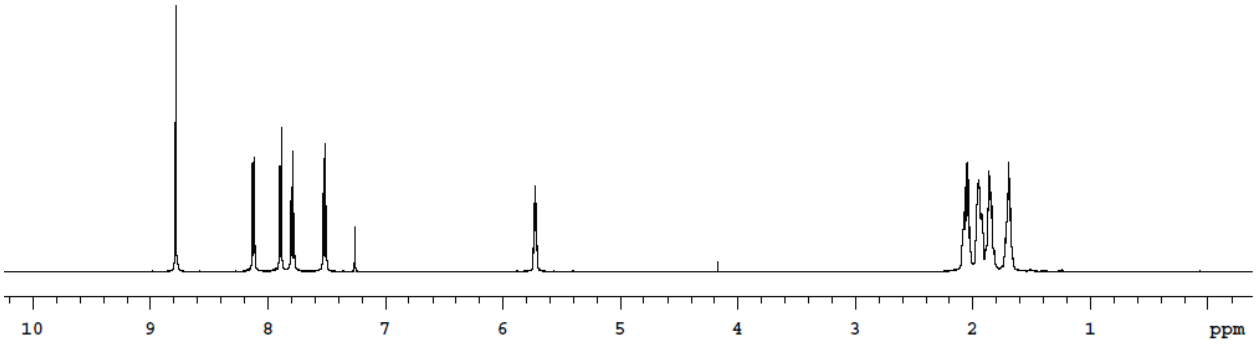
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-032p
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7707217 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 54 sec



16h

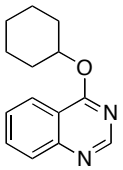


1203-hp-01-031p

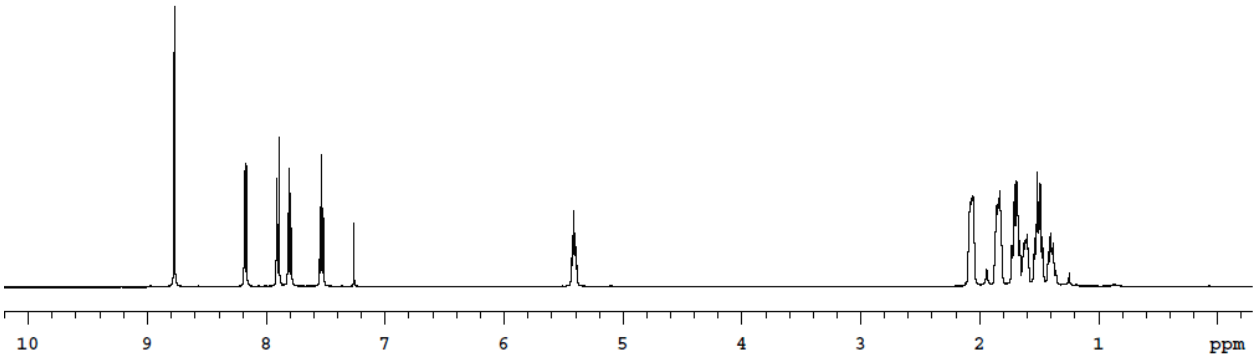
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-031p
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7707212 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 54 sec



16i

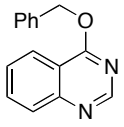


1203-HP-01-033repeat-puri1-1H

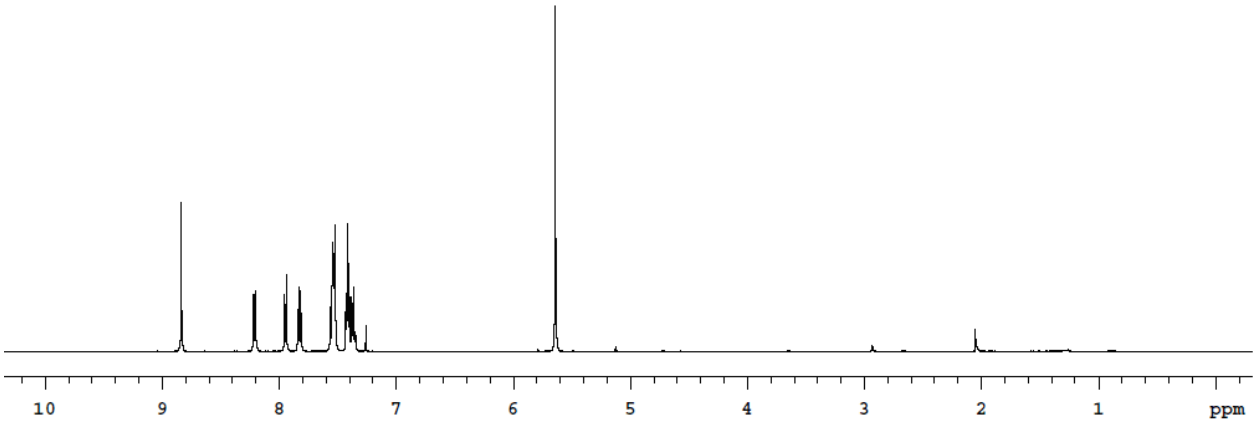
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-033repeat-puri1-1H
INOVA-500 "riga"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
99 repetitions
OBSERVE H1, 499.7707217 MHz
DATA PROCESSING
FT size 32768
Total time 13 min, 0 sec



16j

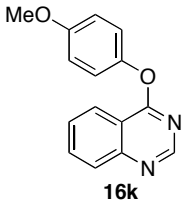


1203-HP-01-062-1h

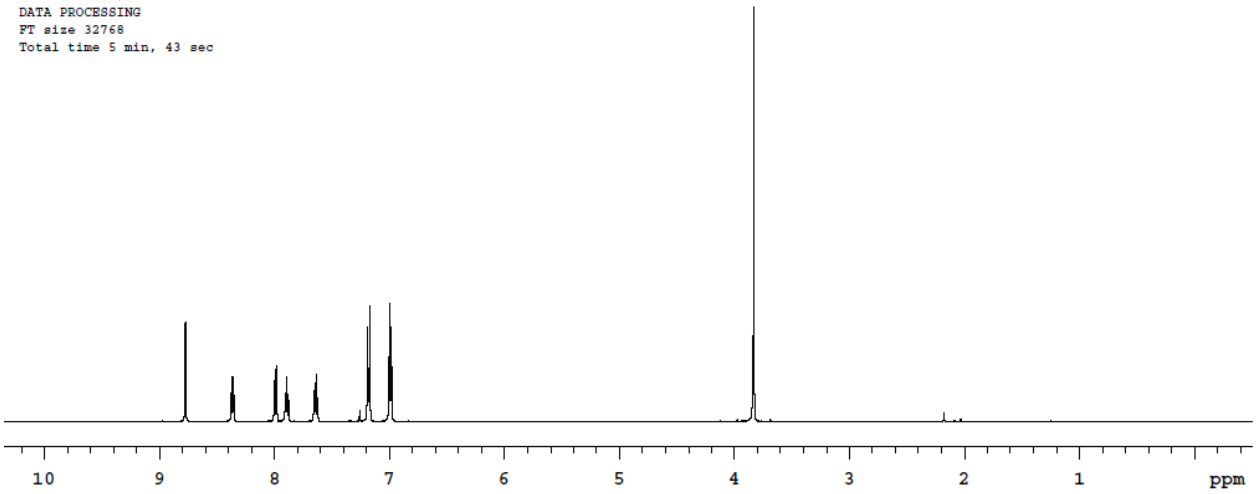
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-062-1h
INOVA-500 "riga"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
58 repetitions
OBSERVE H1, 499.7707217 MHz
DATA PROCESSING
FT size 32768
Total time 5 min, 43 sec



16k

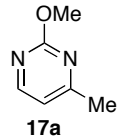


1203-hp-01-037

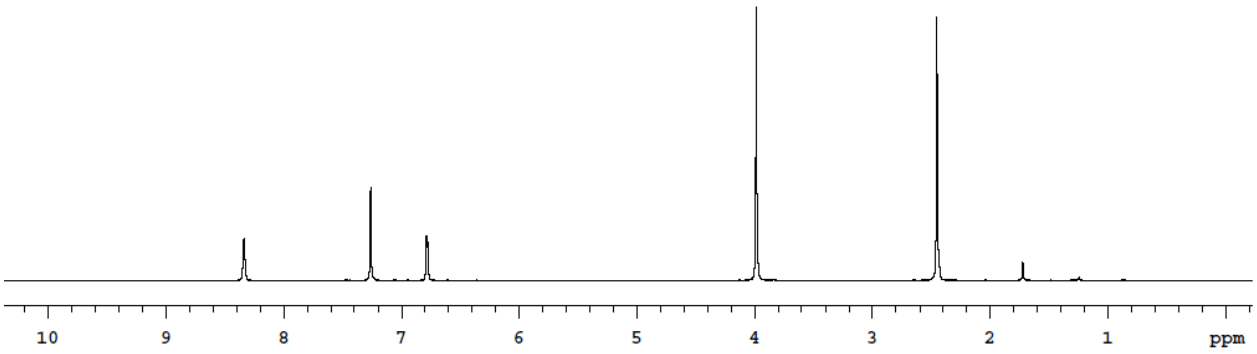
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-037
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7707197 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 37 sec



17a

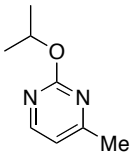


1203-hp-01-038

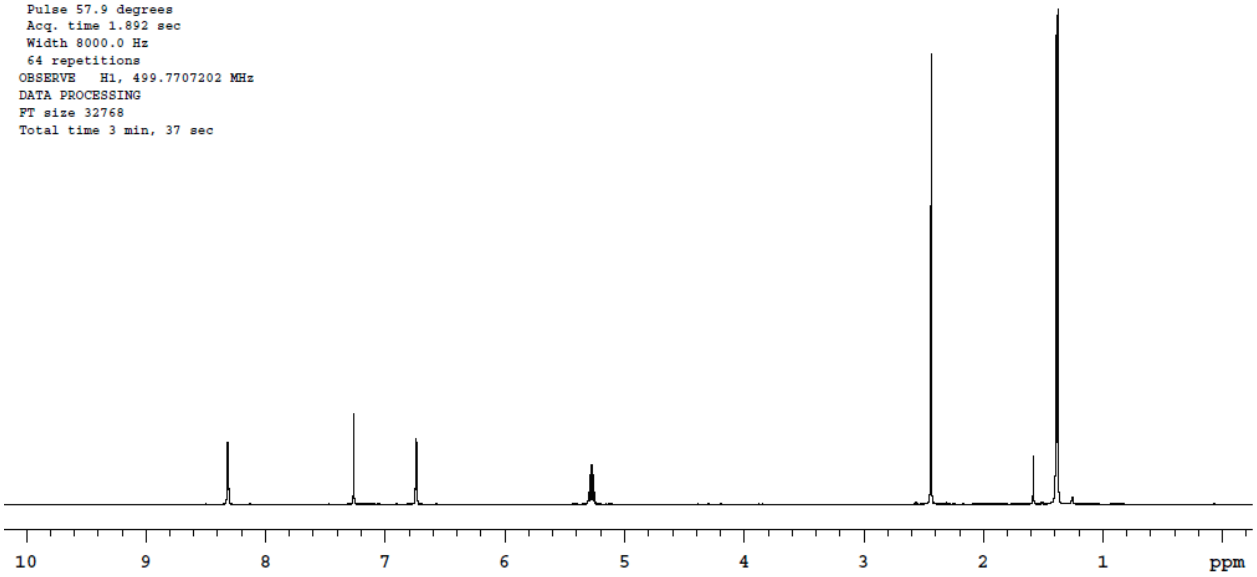
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-038
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7707202 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 37 sec



17b

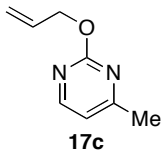


1203-hp-01-039

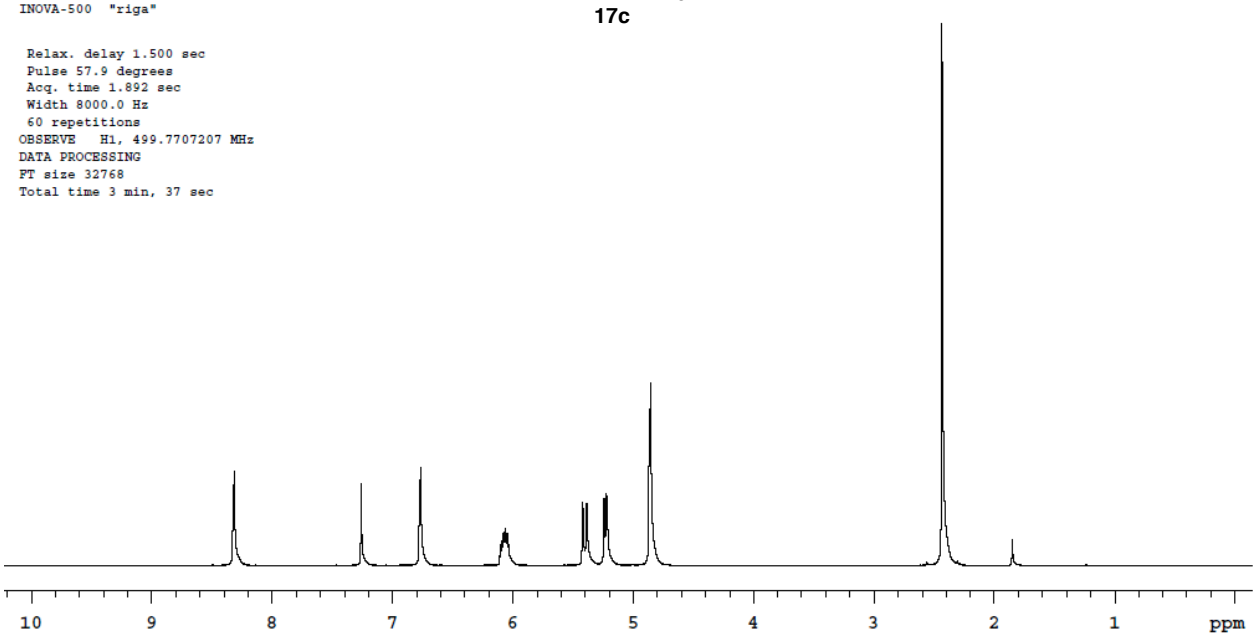
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-039
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
60 repetitions
OBSERVE H1, 499.7707207 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 37 sec



17c

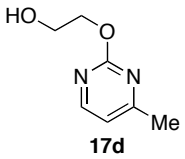


1203-HP-01-053

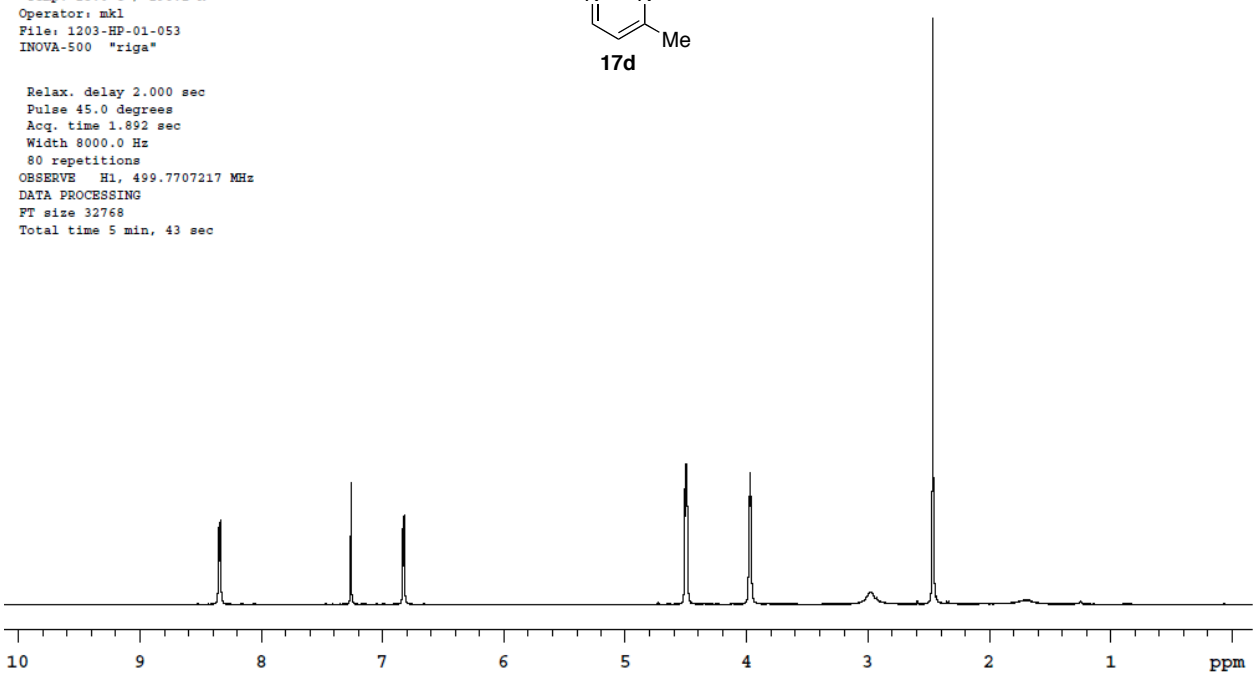
Pulse Sequence: s2pul

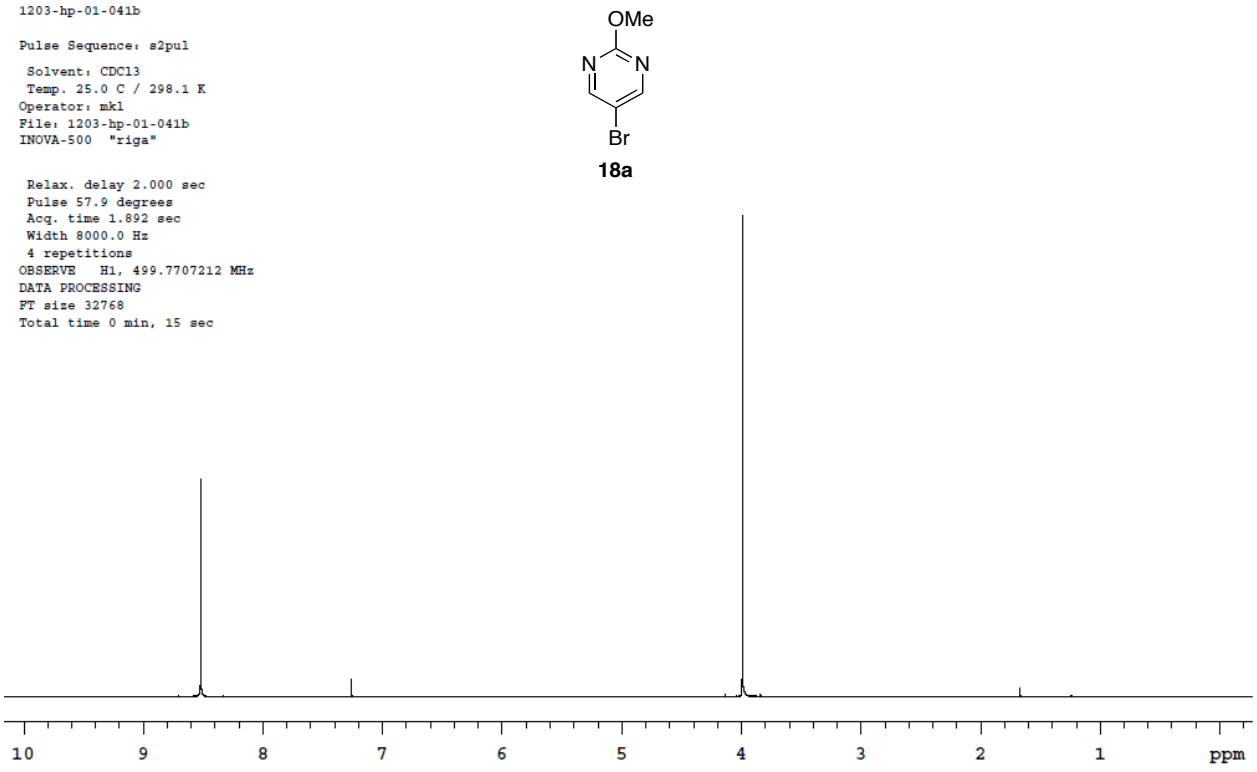
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-053
INOVA-500 "riga"

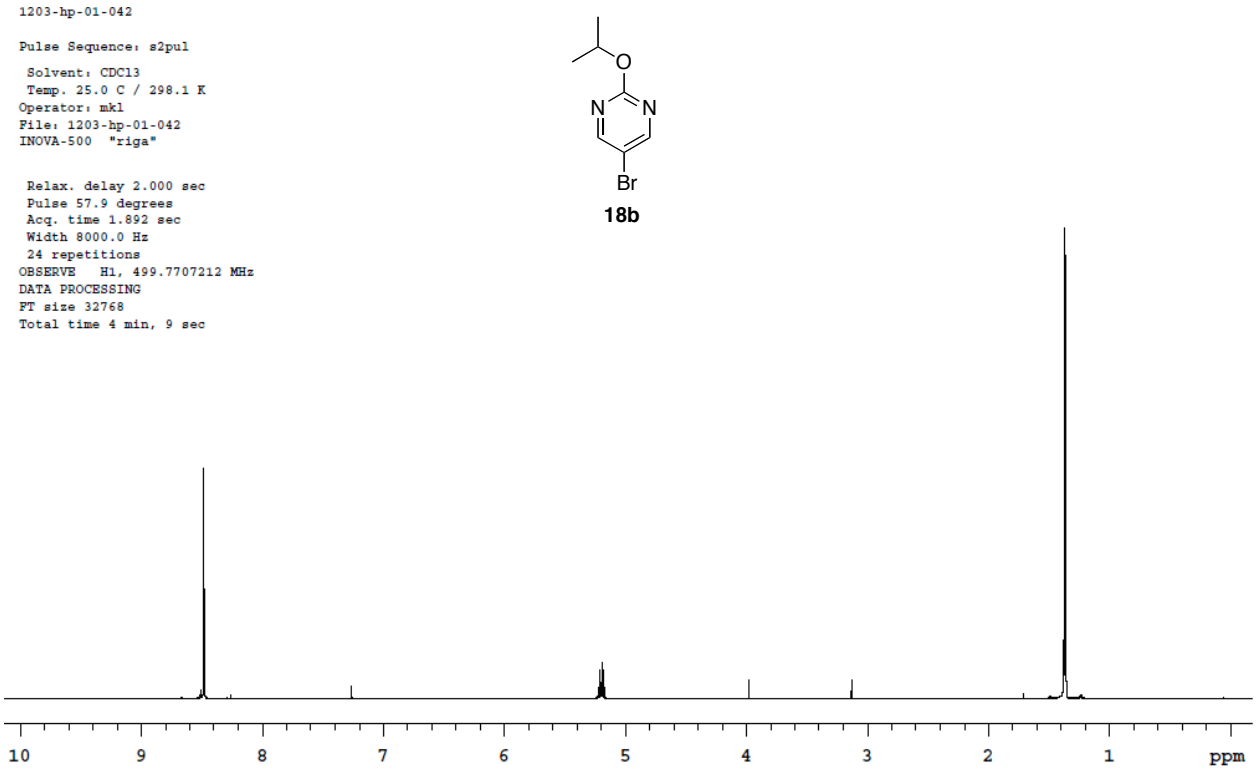
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
80 repetitions
OBSERVE H1, 499.7707217 MHz
DATA PROCESSING
FT size 32768
Total time 5 min, 43 sec



17d




1203-hp-01-041b
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-041b
INOVA-500 "riga"
Relax. delay 2.000 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
4 repetitions
OBSERVE H1, 499.7707212 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 15 sec
OMe
N
N
Br
18a
10
9
8
7
6
5
4
3
2
1
ppm

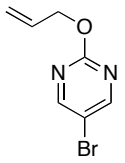

1203-hp-01-042
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-042
INOVA-500 "riga"
Relax. delay 2.000 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
24 repetitions
OBSERVE H1, 499.7707212 MHz
DATA PROCESSING
FT size 32768
Total time 4 min, 9 sec
O
N
N
Br
18b
10
9
8
7
6
5
4
3
2
1
ppm

1203-hp-01-043

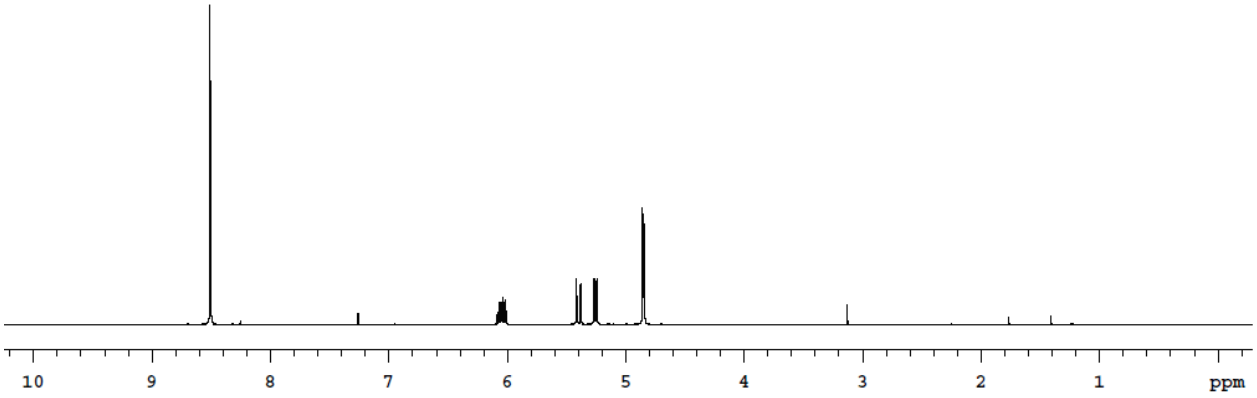
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-hp-01-043a
INOVA-500 "riga"

Relax. delay 1.500 sec
Pulse 57.9 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7707217 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 37 sec



18c

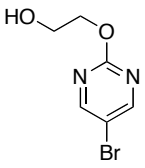


1203-HP-01044b1

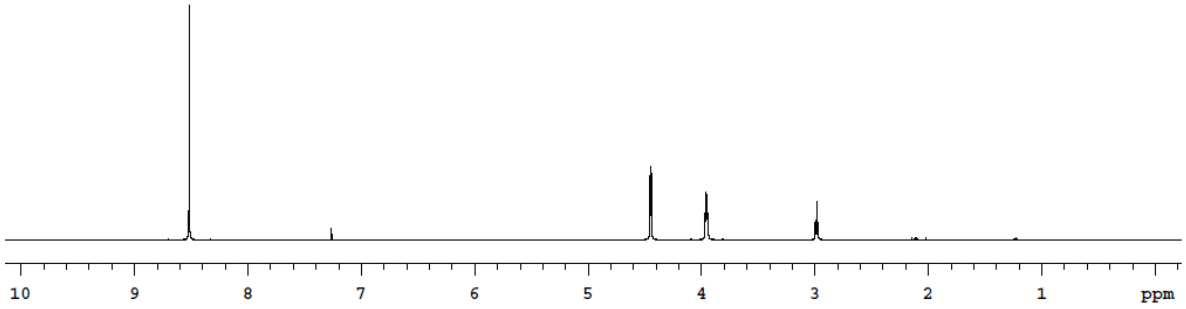
Pulse Sequence: s2pul

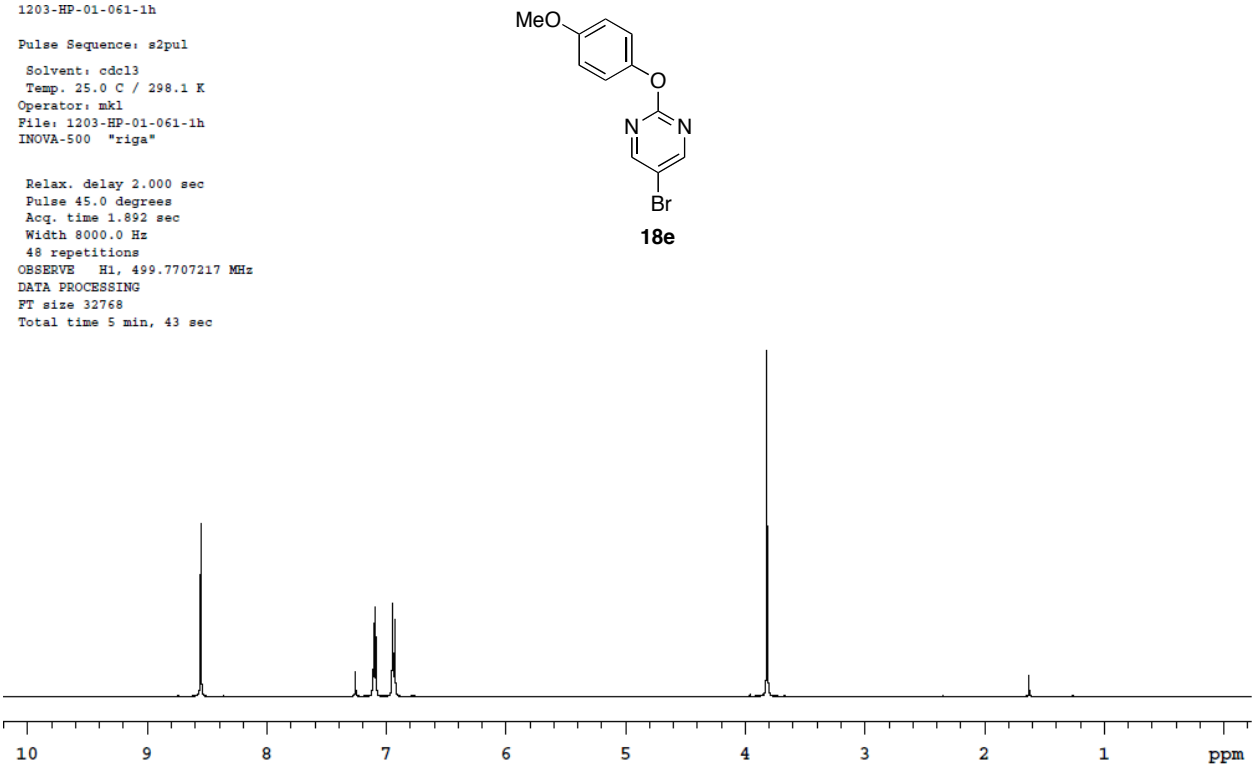
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01044b1
INOVA-500 "riga"

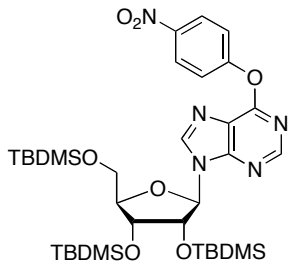
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7707217 MHz
DATA PROCESSING
FT size 32768
Total time 2 min, 4 sec



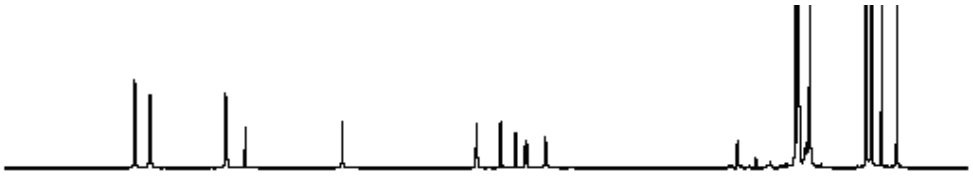
18d

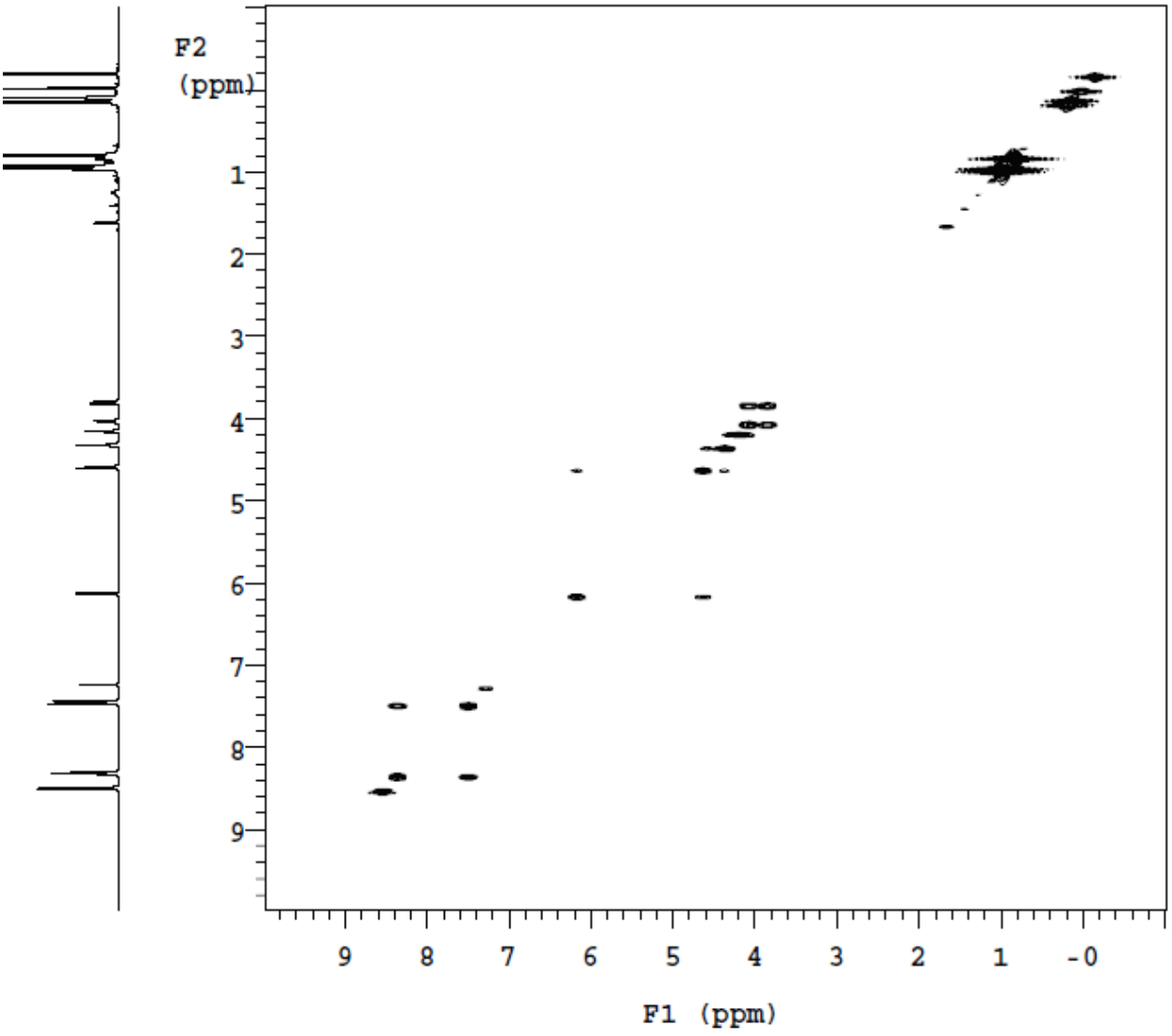


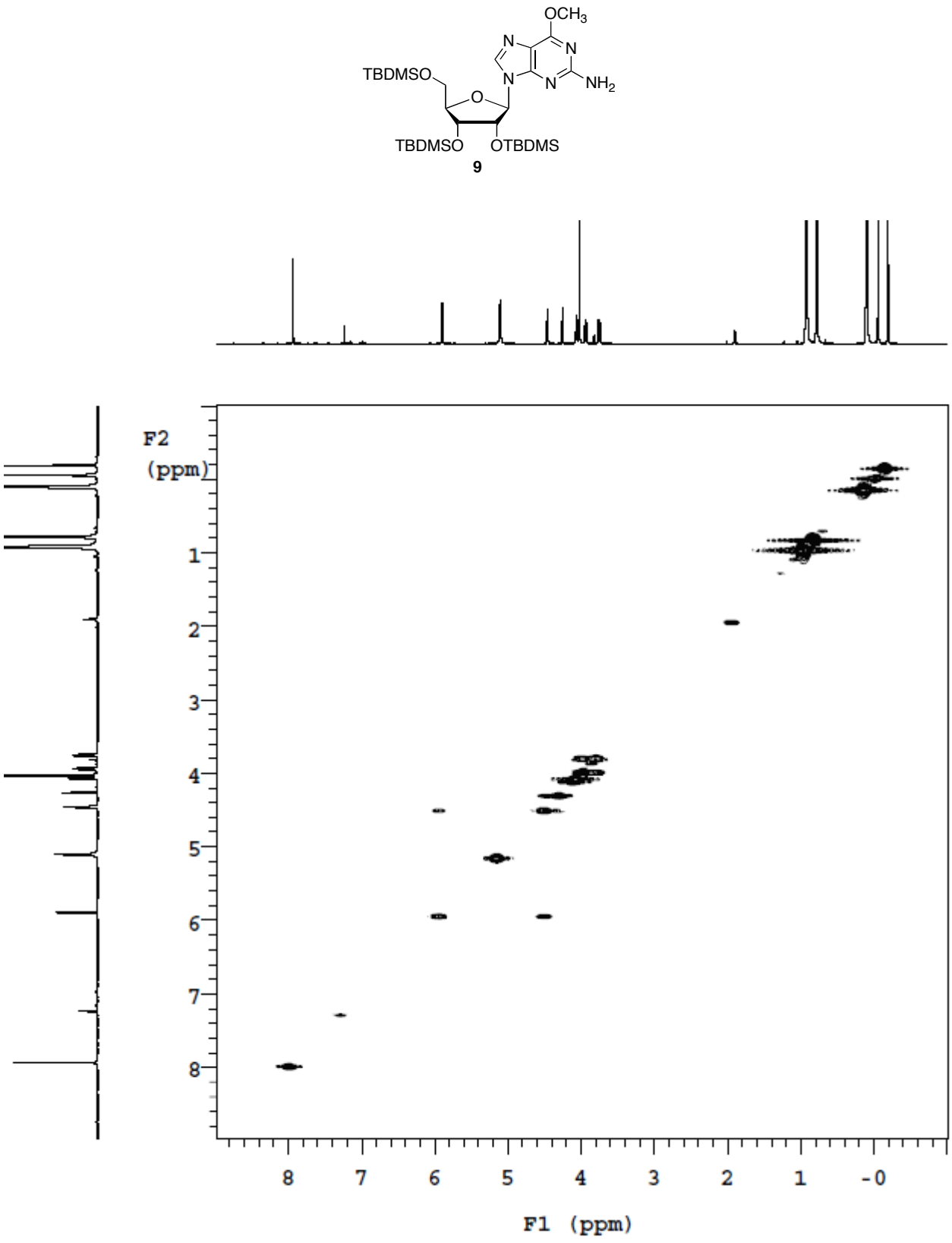

1203-HP-01-061-1h
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mkl
File: 1203-HP-01-061-1h
INOVA-500 "riga"
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
48 repetitions
OBSERVE H1, 499.7707217 MHz
DATA PROCESSING
FT size 32768
Total time 5 min, 43 sec
MeO
O
N
N
Br
18e
10
9
8
7
6
5
4
3
2
1
ppm

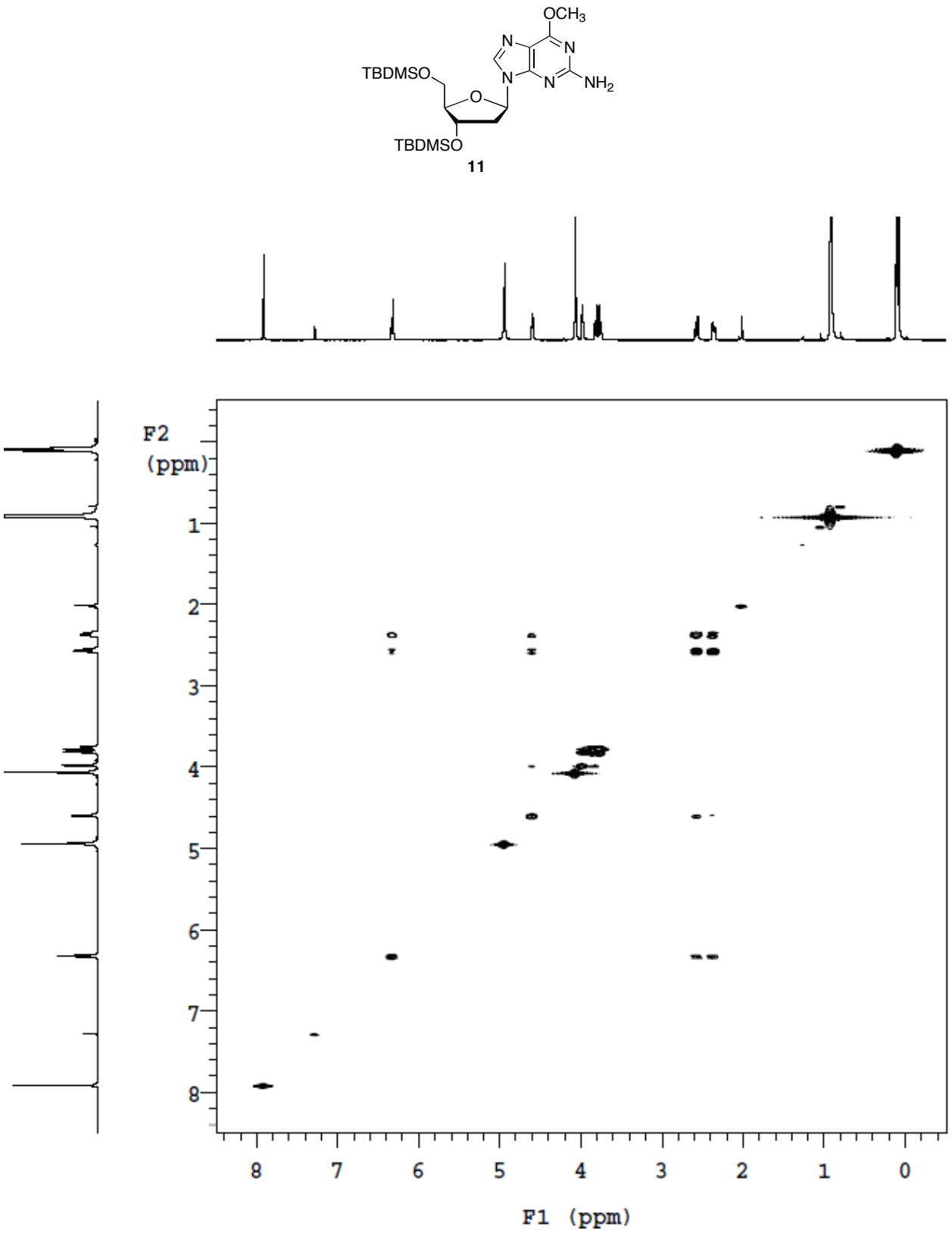

O2N
O
N
N
N
N
TBDMSO
O
TBDMSO
OTBDMS

8




F2
(ppm)
1
2
3
4
5
6
7
8
9
9
8
7
6
5
4
3
2
1
-0
F1 (ppm)


OCH3
N
N
N
N
N
NH2
TBDMSO
O
TBDMSO
OTBDMS
9
F2
(ppm)
1
2
3
4
5
6
7
8
8
7
6
5
4
3
2
1
-0
F1 (ppm)


OCH3
N
N
TBDMSO
N
N
NH2
O
TBDMSO
F2
(ppm)
1
2
3
4
5
6
7
8
8
7
6
5
4
3
2
1
0
F1 (ppm)

11

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