Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Practical synthesis of natural plant-growth regulator 2-azahypoxanthine, its derivatives, and biotin-labeled probes

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Content

I. Experimental procedure

Analysis instruments

Nuclear magnetic resonance, [^{1}H NMR (500 MHz), ^{13}C NMR (125 MHz)] spectra were determined on JEOL ECA-500 and JEOL α-500 instruments, and [^{1}H NMR (400 MHz), ^{13}C NMR (100 MHz)] spectra were determined on JEOL LA-400 instruments. Chemical shifts for ^{1}H NMR were reported in parts per million downfields from tetramethylsilane (δ) as the internal standard and coupling constants are in hertz (Hz). The following abbreviations are used for spin multiplicity: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Chemical shifts for ^{13}C NMR were reported in ppm relative to the centerline of a triplet at 77.0 ppm for deuteriochloroform.

High-resolution mass spectra (HRMS) were obtained on a JEOL MStation 700. Fast atom bombardment (FAB) mass spectra were obtained with 3-nitrobenzylalcohol as the matrix and ESI-MS was taken with a BRUKER DALTONICS micrOTOF.

Analytical thin layer chromatography (TLC) was performed on Merck precoated analytical plates, 0.25 mm thick, silica gel 60 F_{254}. Preparative TLC separations were made on 7 x 20 cm plates prepared with a 0.25 mm or 0.50 mm layer of Merck silica gel 60 F_{254}.

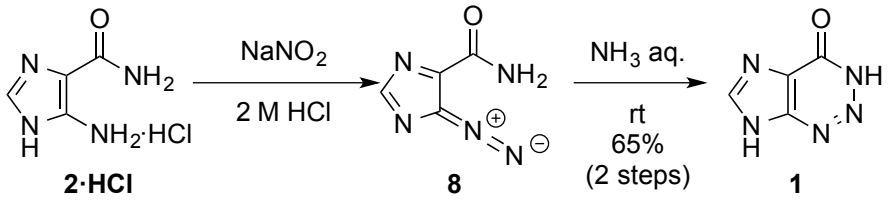
Column chromatography separations were performed on KANTO CHEMICAL Silica Gel 60 (spherical) 40 - 50 μm, Silica Gel 60 (spherical) 63 - 210 μm or Silica Gel 60 N (spherical, neutral) 63 - 210 μm.

Reagents and solvents were commercial grades and were used as supplied with following exceptions:

Dichloromethane, diethyl ether, *n*-hexane, tetrahydrofuran toluene: dried over molecular sieves 4A.

Methanol, acetonitrile: dried over molecular sieves 3 A.

3,7-dihydro-4*H*-imidazo[4,5-d][1,2,3]triazin-4-one, 2-azahypoxhanthine (AHX; **1**) (Method A)



To a stirred solution of $NaNO_2$ (400 g, 5.80 mol) in H_2O (1.5 L) was added dropwise 5-amino-*1H*-imidazole-4-carboxamide hydrochloride (AICA·HCl; **2**·HCl) (124 g, 0.762 mol) at 0 °C for 30 minutes. After being stirred for 1 hour, the reaction mixture was filtered, and the residue was washed with THF. The residue was dried in vacuum at room temperature to afford 4-diazo-*4H*-imidazole-5-carboxamide (DICA) as crude material.

To a stirred solution of the crude material including DICA in H_2O (300 mL) was added 25% aqueous NH_3 (400 mL) at room temperature. After being stirred for 30 minutes, the reaction mixture was evaporated under reduced pressure. The residue was added to MeOH, then filtered to remove insoluble red solid. To a stirred of the filtrate was added activate carbon at room temperature. After being stirred for 12 hours, the mixture was filtered, evaporated under reduced pressure to afford 2-azahypoxanthine (AHX; **1**) (68 g, 0.495 mol, 65%, 2 steps) as a white solid.

Analytical data for compound **1**

m.p.: 150 ºC (decomp.)

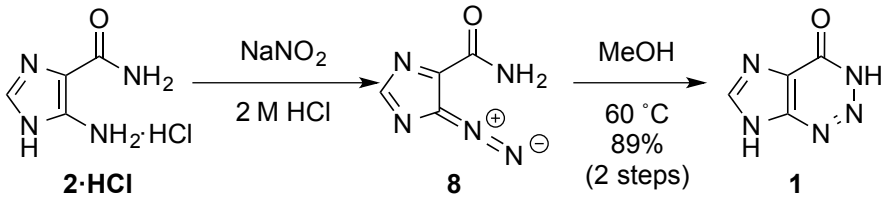
IR (KBr, cm^{-1}): 3350, 1701, 1664.

^{1}H NMR (500 MHz, d_6-DMSO): δ 8.33 (s, 1H).

^{13}C NMR (125 MHz, d_6-DMSO): δ 153.5, 153.3, 145.5, 120.9.

HRMS (ESI): Calculated for $C_4H_3N_5NaO$, 160.0235 [$(M+Na)^+$], found 160.0233.

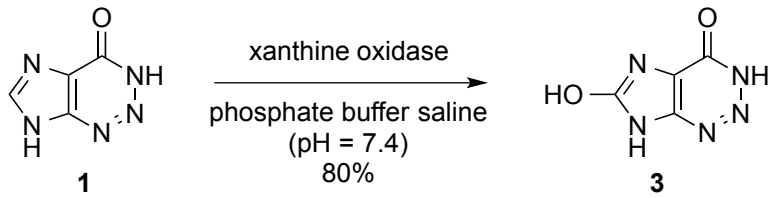
3,7-dihydro-4*H*-imidazo[4,5-d][1,2,3]triazin-4-one, 2-azahypoxhanthine (AHX; **1**) (Method B)



To a stirred solution of $NaNO_2$ (400 g, 5.80 mol) in H_2O (1.5 L) was added dropwise 5-amino-*1H*-imidazole-4-carboxamide hydrochloride (AICA·HCl; **2**·HCl) (124 g, 0.762 mol) in 6 M HCl (500 mL) at 0 °C for 30 minutes. After being stirred for 1 hour, the reaction mixture was filtered, and the residue was washed with THF. The residue was dried in vacuum at room temperature to afford 4-diazo-*4H*-imidazole-5-carboxamide (DICA; **8**) as crude material.

A suspension of the crude material DICA (**8**) (19.7 g, 0.144 mmol) in MeOH (300 mL) was stirred at 60 ºC for 10 hours. After cooling, the reaction mixture was added activate carbon until the color of the red solution was changed to the dark. After being stirred for 12 hours at room temperature, the mixture was filtered, evaporated under reduced pressure to afford 2-azahypoxanthine (AHX; **1**) (17.5 g, 0.128 mol, 89%, 2 steps) as a white solid.

6-Hydroxy-3,7-dihydro-4H-imidazo[4,5-d][1,2,3]triazin-4-one (AOH; **3**)



1 **3**

To a stirred of solution of AHX (**1**) (68.5 mg, 0.500 mmol) in 10 mM phosphate buffered saline (pH 7.4, 1 L) was added to XOD (20 mg). After being leaving to stand at 30°C for 12 h, XOD (10 mg) was repeatedly added to the reaction mixture every 12 h twice. Then, the resulting mixture was evaporated to dryness under reduced pressure. The residue was recrystallization with H_2O at 4 ºC to afford AOH (**3**) (61.2 mg, 0.400 mmol, 80%) as a white solid.

Analytical data for compound **3**

m.p.: 170 ºC (decomp.)

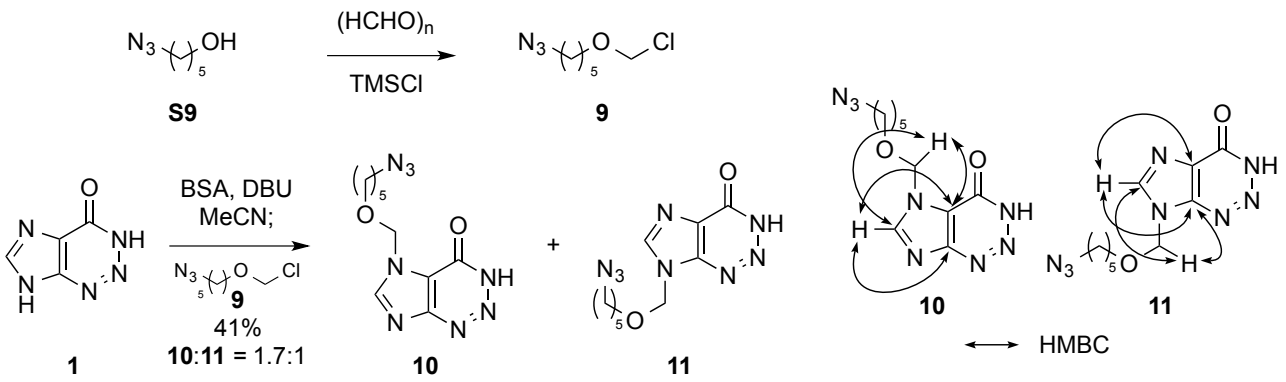
IR (KBr, cm^{-1}): 3057, 1668, 1455, 929.

^{13}C NMR (125 MHz, d_6-DMSO): δ 156.4, 154.6, 147.4, 110.1.

HRMS (ESI): Calculated for $C_4H_2N_5O_2$, 152.0209 [$(M-H)^-$], found 152.0206.

5-(((5-azidopentyl)oxy)methyl)-3,5-dihydro-*4H*-imidazo[4,5-d][1,2,3]triazin-4-one (**10**)

7-(((5-azidopentyl)oxy)methyl)-3,7-dihydro-*4H*-imidazo[4,5-d][1,2,3]triazin-4-one (**11**)



1-azido-5-(chloromethoxy)pentane (**9**) was prepared according to ref 1). To a stirred solution of **S9** (87.2 mg, 0.676 mmol) in TMSCl (2 mL) was added $(HCHO)_n$ (17.3 mg, 0.563 mmol) at 0 ºC under an argon atmosphere. After being stirred for 1 hour at room temperature, the reaction mixture was concentrated under reduced pressure to afford **9** as the crude material. The crude material including **9** was applied to the following reaction without further purification.

To a stirred suspension of **1** (235 mg, 1.69 mmol) in MeCN (5 mL) were added *N*,*O*-bis(trimethylsilyl)acetamide (BSA, 0.42 mL, 1.69 mmol) and DBU (0.15 mL, 1.00 mmol) at room temperature under an argon atmosphere. After being stirred for 20 minutes, the reaction mixture was added to a solution of the crude material of **9** (about 0.563 mmol) in MeCN (1 mL). The reaction mixture was stirred at room temperature for 3 hours. Then, the resulting mixture was poured into MeOH and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAc = 1:1 to 1:3) to afford **10** (40.4 mg, 0.145 mmol, 26%) as a colorless oil and **11** (23.4 mg, 85.5 μmol, 15%) as a colorless oil.

Structure determination of **10** and **11** was carried out by HMBC correlation.

Analytical data for compound **10**

IR (film, cm^{-1}): 3106, 2944, 2097, 1706, 1379, 1339, 1207, 1100.

^1H NMR (500 MHz CD_3OD): δ 8.57 (s, 1H), 5.84 (s, 2H), 3.59 (t, *J* = 6.3 Hz, 2H), 3.21 (t, *J* = 6.6 Hz, 2H), 1.59-1.47 (m, 4H), 1.38-1.32 (m, 2H).

^{13}C NMR (125 MHz, CD_3OD): δ 155.6, 153.2, 147.0, 118.3, 77.6, 70.3, 52.3, 29.8, 29.4, 24.2.

HRMS (ESI): Calculated for $C_{10}H_{13}N_8O_2$ 277.1156 [$(M-H)^-$], found 277.1151.

Analytical data for compound **11**

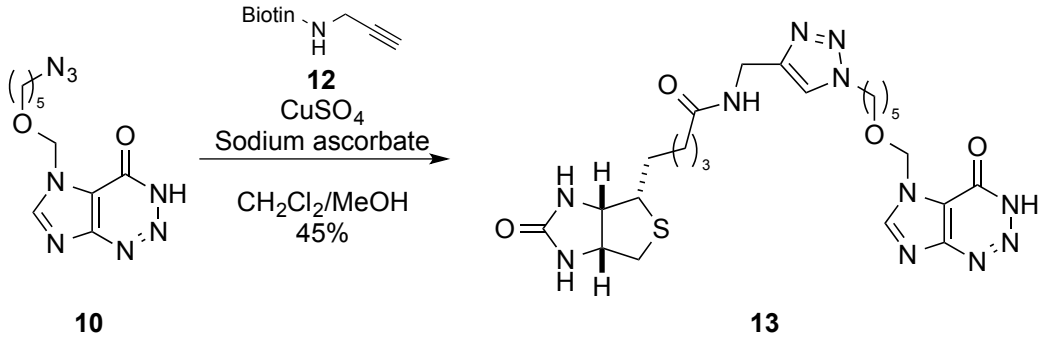
IR (film, cm^{-1}): 3106, 2934, 2093, 1720, 1386, 1315, 1207, 1100.

^1H NMR (500 MHz CD_3OD): δ 8.44 (s, 1H), 5.80 (s, 2H), 3.61 (t, *J* = 6.3 Hz, 2H), 3.23 (t, *J* = 6.6 Hz, 2H), 1.62-1.50 (m, 4H), 1.41-1.36 (m, 2H).

^{13}C NMR (125 MHz, CD_3OD): δ 158.6, 147.5, 145.2, 126.8, 75.4, 70.8, 52.3, 29.9, 29.5, 24.2.

HRMS (ESI): Calculated for $C_{10}H_{13}N_8O_2$ 277.1156 [$(M-H)^-$], found 277.1147.

N-((1-(5-((4-oxo-3,4-dihydro-*5H*-imidazo[4,5-d][1,2,3]triazin-5-yl)methoxy)pentyl)-*1H*-1,2,3-triazol-4-yl)methyl)-5-((*3aS,4S,6aR*)-2-oxohexahydro-*1H*-thieno[3,4-d]imidazol-4-yl)pentanamide (**13**)



10 **13**

To a solution of **10** (33 mg, 0.11 mmol) and **12**[2] (32 mg, 0.11 mmol) in a 1 : 1 mixture of CH_2Cl_2 and MeOH (total 6 mL) were added $CuSO_4$ (11 mg, 56 μmol) and sodium ascorbate (12 mg, 56 μmol) at room temperature. The reaction mixture was stirred for 24 hours. Then, the resulting mixture was filtered through a pad of Celite and concentrated under reduced pressure. The residue was purified by silica gel column chromatography ($CHCl_3$/MeOH = 10:1 to 7:1) to afford **13** (26 mg, 50 μmol, 45%) as a colorless oil.

Analytical data for compound **13**

$[\alpha]^{20}_D$ +22.1 (*c* 0.62, CH_3OH).

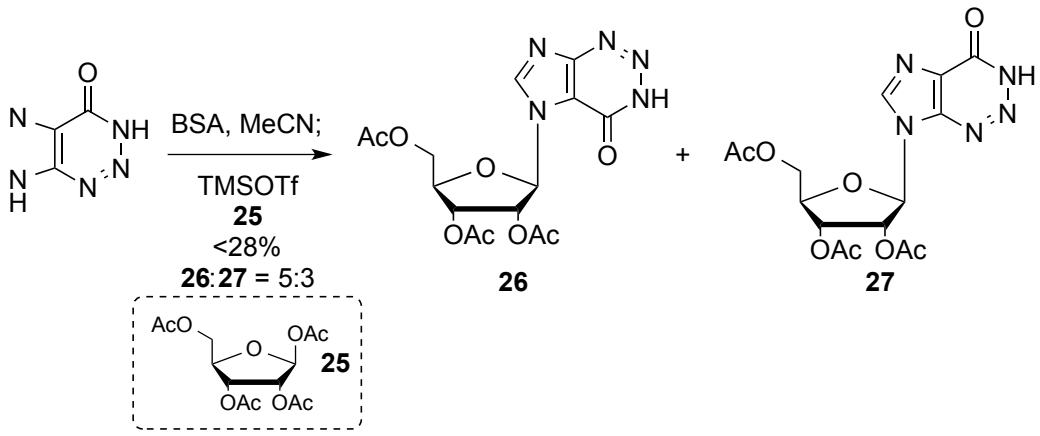
IR (film, cm^{-1}): 3200, 2900, 1700, 1447, 1340, 1214, 1118.

^1H NMR (500 MHz CD_3OD): δ 8.56 (s, 1H), 7.81 (s, 1H), 5.81 (s, 2H), 4.49 (dd, *J* = 8.0, 4.0 Hz, 1H), 4.43 (s, 2H), 4.34 (t, *J* = 6.9 Hz, 2H), 4.29 (dd, *J* = 8.0, 4.6 Hz, 1H), 3.56 (t, *J* = 6.3 Hz, 2H), 3.21-3.16 (m, 1H), 2.91 (dd, *J* = 12.6, 5.2 Hz, 1H), 2.70 (d, *J* = 12.6 Hz, 1H), 2.25 (t, *J* = 7.5 Hz, 2H), 1.89-1.81 (m, 2H), 1.75-1.54 (m, 6H), 1.44-1.39 (m, 2H), 1.34-1.29 (m, 2H).

^{13}C NMR (125 MHz CD_3OD): δ 176.3, 166.1, 155.8, 153.4, 147.1, 146.1, 124.2, 118.1, 77.7, 70.1, 63.3, 61.6, 57.0, 51.2, 41.1, 36.6, 35.6, 30.8, 29.7, 29.6, 29.4, 26.7, 24.0.

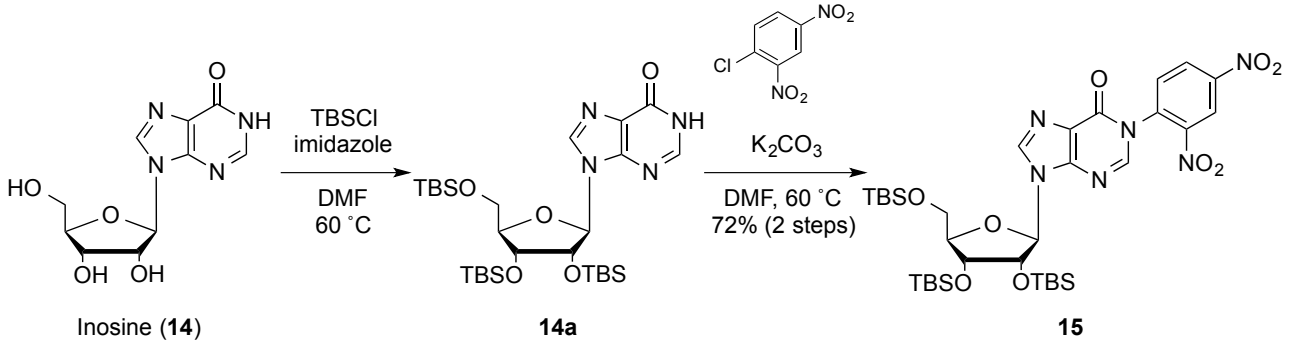
HRMS (ESI): Calculated for $C_{23}H_{33}N_{11}O_4SNa$ 582.2330 [$(M+Na)^+$], found 582.2352.

Attempt to ribosylation of AHX (**1**)



We first tried to prepare AHXr (**4**) by the direct condensation reaction of AHX (**1**) and tetraacetyl ribose (**25**) proceeded by treatment with TMSOTf and BSA.[3] But, the yield and regioselectivity were furnished in unsatisfacutly results. Additionally, separation of both isomers was difficult. Therefore, we planned the synthesis of **4** and its derivatives from inosine (**14**).

9-((*2R,3R,4R,5R*)-3,4-bis((*tert*-butyldimethylsilyl)oxy)-5-(((*tert*-butyldimethylsilyl)oxy)methyl)tetrahydrofuran-2-yl)-1-(2,4-dinitrophenyl)-1,9-dihydro-*6H*-purin-6-one (**15**)

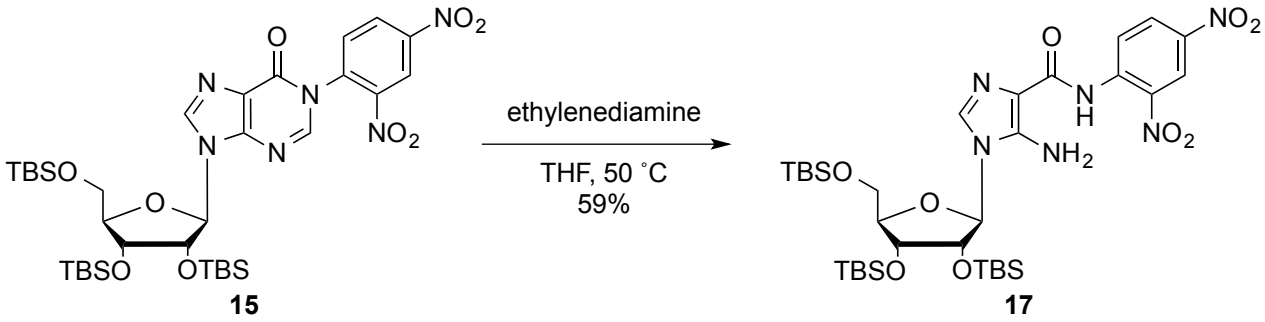


To a stirred solution of inosine (**14**) (20.9 g, 73.5 mmol) in DMF (260 mL) were added imidazole (26.4 g, 0.388 mol) and TBSCl (38.7 g, 0.257 mol) at room temperature under an argon atmosphere. After being stirred at 60 ºC for 48 hours, the resulting mixture was poured into H_2O at 0 ºC. The white precipitate was filtered and dried in vacuum at 60 ºC to afford a crude material including **14a**.

To a stirred solution of the crude material including **14a** and 1-chloro 2,4-dinitrobenzene (16.4 g, 80.9 mmol) in DMF (500 mL) was added K_2CO_3 (20.3 g, 0.147 mol) at room temperature under an argon atmosphere. After being stirred at 60 ºC for 48 hours, the resulting mixture was poured into H_2O at 0 ºC. The red precipitate was filtered and washed with H_2O. Then, the red solid was dissolved with EtOAc (1 L), and washed with brine for three times. The organic layer was dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by recrystallization with Et_2O/*n*-hexane to afford **15** (41.4 g, 53.3 mmol, 72%, 2 steps) as a brown solid.

The spectra data of **15** was in good agreement of reference 4).

5-amino-1-((*2R,3R,4R,5R*)-3,4-bis((*tert*-butyldimethylsilyl)oxy)-5-(((*tert*-butyldimethylsilyl)oxy)methyl)tetrahydrofura n-2-yl)-*N*-(2,4-dinitrophenyl)-*1H*-imidazole-4-carboxamide (**17**)



To a stirred solution of **15** (6.78 g, 8.72 mmol) in THF (20 mL) was added ethylenediamine (3.2 mL, 52.3 mmol) at room temperature under an argon atmosphere. After being stirred at 50 ºC for 3 hours, the resulting mixture was poured into H_2O at 0 °C, and extracted with *n*-hexane. The organic layer was washed with 2 M HCl, brine, dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by trituration with petroleum Et_2O to afford **17** (3.94 g, 5.14 mmol, 59%) as an orange solid.

Analytical data for compound **17**

m.p.: 165-167 ºC

$[\alpha]^{20}_D$ –84.2 (*c* 1.00, $CHCl_3$).

IR (film, cm^{-1}): 3442, 3327, 2954, 2930, 1676, 1595, 1557, 1505, 1339, 1260, 1107, 835, 779.

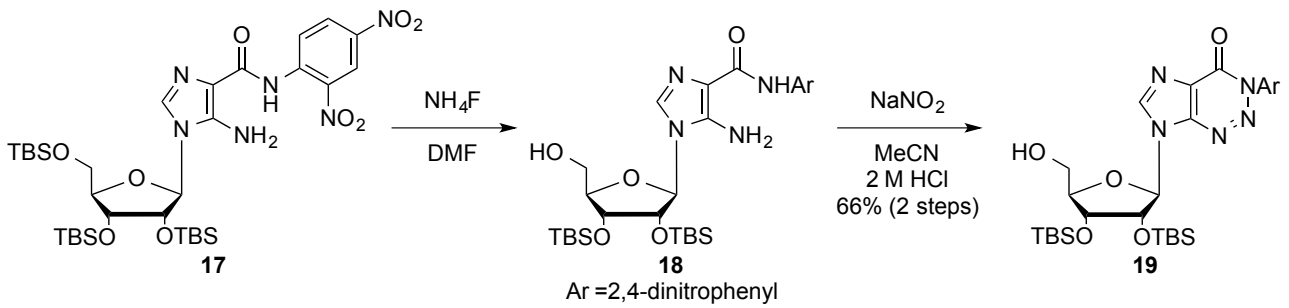
^1H NMR (500 MHz, $CDCl_3$): δ 11.8 (brs, 1H), 9.45 (d, *J* = 9.5 Hz, 1H), 9.13 (d, *J* = 2.3 Hz, 1H), 8.39 (dd, *J* = 9.5, 2.3 Hz, 1H), 7.03 (s, 1H), 6.03 (brs, 2H), 5.49 (d, *J* = 8.0 Hz, 1H), 4.52-4.48 (m, 1H), 4.12 (d, *J* = 5.2 Hz, 1H), 4.08 (brs, 1H), 3.93 (dd, *J* = 11.7, 1.7 Hz, 1H), 3.81 (dd, *J* = 11.7, 1.7 Hz, 1H), 0.96 (s, 9H), 0.93 (s, 9H), 0.82 (s, 9H), 0.17 (s, 3H), 0.16 (s, 3H), 0.12 (s, 3H), 0.09 (s, 3H), -0.04 (s, 3H), -0.27 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 163.1, 145.6, 141.2, 140.4, 134.2, 131.0, 129.5, 122.4, 121.3, 113.7, 89.5, 87.4, 72.8, 71.8, 63.6, 26.0 (3C), 25.8 (3C), 25.7 (3C), 18.6, 18.0, 17.8, -4.46 (2C), -4.55, -5.23, -5.43, -5.66

HRMS (ESI): Calculated for $C_{33}H_{58}N_6O_9Si_3Na$ 789.3465 [$(M+Na)^+$], found 789.3442.

7-((*2R,3R,4R,5R*)-3,4-bis((*tert*-butyldimethylsilyl)oxy)-5-(hydroxymethyl)tetrahydrofuran-2-yl)

-3-(2,4-dinitrophenyl)-3,7-dihydro-*4H*-imidazo[4,5-d][1,2,3]triazin-4-one (**19**)



To a stirred solution of **17** (1.76 g, 2.29 mmol) in DMF (10 mL) was added NH_4F (425 mg, 11.5 mmol) at room temperature. After being stirred for 24 hours, the resulting mixture was poured into H_2O at 0 °C, and extracted with Et_2O. The organic layer was washed with brine, dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure. The crude material including **18** was applied to the following reaction without further purification.

To a stirred solution of the crude material including **18** in a 3 : 1 mixture of MeCN and 2 M HCl (total 8 mL) was added $NaNO_2$ (453 mg, 6.57 mmol) at 0 °C. After being stirred at the same temperature for 1 hour, the resulting mixture was poured into H_2O, and extracted with CH_2Cl_2. The organic layer was dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by recrystallization from EtOAc/*n*-hexane to afford **19** (963 mg, 1.45 mmol, 66%, 2 steps) as a colorless solid.

Analytical data for compound **19**

m.p.: 155 ºC (decomp.)

$[\alpha]^{20}_{D}$ −18.0 (*c* 1.02, $CHCl_3$).

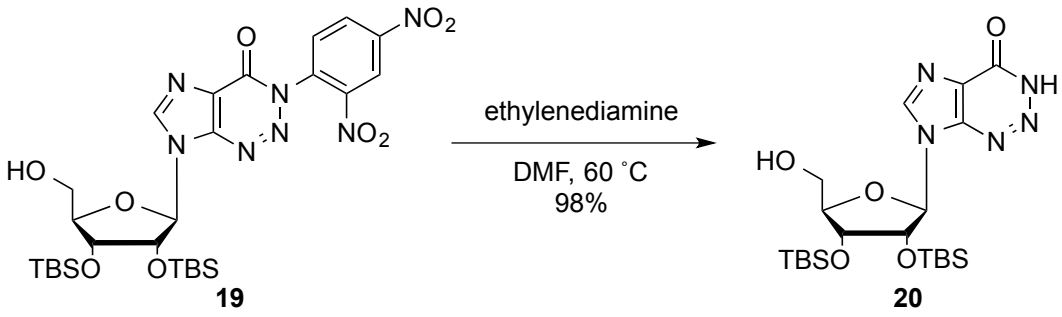
IR (film, cm^{-1}): 3497, 2930, 2858, 1716, 1544, 1354, 1240, 1183, 1085, 835, 790.

^{1}H NMR (500 MHz, $CDCl_3$): δ 9.08 (d, *J* = 2.3Hz, 1H), 8.69 (dd, *J* = 8.6, 2.3 Hz, 1H), 8.33 (s, 1H), 7.92 (d, *J* = 8.6 Hz, 1H), 6.02 (d, *J* = 6.9 Hz, 1H), 4.79 (dd, *J* = 6.9, 4.6 Hz, 1H), 4.36 (dd, *J* = 4.6, 1.7 Hz, 1H), 4.25 (brs, 1H), 4.04-3.98 (m, 1H), 3.83-3.76 (m, 2H), 0.95 (s, 9H), 0.80 (s, 9H), 0.14 (s, 3H), 0.13 (s, 3H), -0.03 (s, 3H), -0.36 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 153.0, 148.2, 145.3, 144.1, 136.6, 131.7, 128.7, 128.4, 121.2 (2C), 91.1, 88.7, 76.3, 72.9, 62.3, 25.8 (3C), 25.6 (3C), 18.1, 17.8, -4.5, -4.6 (2C), -5.7.

HRMS (ESI): $C_{27}H_{41}N_7O_9Si_2Na$ 686.2397 [$(M+Na)^+$], found 686.2368.

7-((*2R,3R,4R,5R*)-3,4-bis((*tert*-butyldimethylsilyl)oxy)-5-(hydroxymethyl)tetrahydrofuran-2-yl)-3,7-dihydro-*4H*-imidazo[4,5-d][1,2,3]triazin-4-one (**20**)



To a stirred solution of **19** (301 mg, 0.453 mmol) in DMF (3 mL) was added ethylenediamine (1 mL) at room temperature. After being stirred at 60 ºC for 2 hours, the resulting mixture was poured into H_2O at 0 °C, and extracted with EtOAc. The organic layer was washed with 2 M HCl, brine, dried over anhydrous $MgSO_4$, filtered. The filtrate was concentrated under reduced pressure to afford **20** (221 mg, 0.444 mmol, 98%) as a colorless solid.

Analytical data for compound **20**

m.p.: 115-117 ºC

$[\alpha]^{20}_D$ −57.0 (*c* 1.00, $CHCl_3$).

IR (film, cm^{-1}): 3494, 2950, 2857, 1720, 1472, 1260, 1165, 1099, 836, 777.

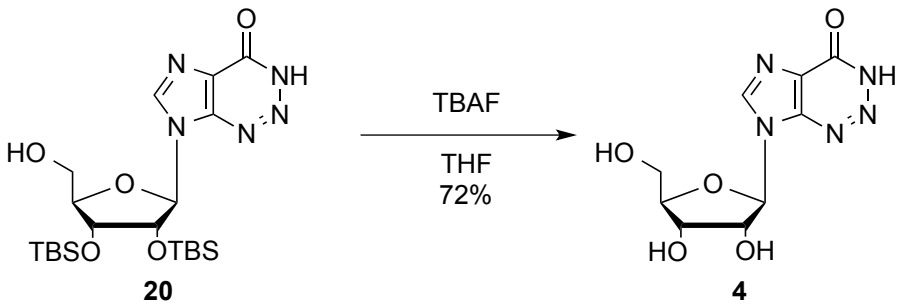
^1H NMR (500 MHz, $CDCl_3$): δ 8.19 (s, 1H), 5.95 (d, *J* = 7.5 Hz, 1H), 4.82 (dd, *J* = 7.5, 4.6 Hz, 1H), 4.43 (brs, 1H), 4.35 (dd, *J* = 4.6, 1.2 Hz, 1H), 4.23-4.21 (m, 1H), 3.99 (dd, *J* = 12.6, 1.7 Hz, 1H), 3.77 (d, *J* = 12.6 Hz, 1H), 0.95 (s, 9H), 0.77 (s, 9H), 0.13 (s, 3H), 0.12 (s, 3H), -0.08 (s, 3H), -0.49 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 154.3, 144.6, 143.3, 129.1, 91.2, 89.1, 76.8, 73.2, 62.5, 25.8 (3C), 25.6 (3C), 18.0, 17.8, -4.54, -4.61, -4.64, -5.77.

HRMS (ESI): Calculated for $C_{21}H_{39}N_5O_5Si_2Na$ 520.2382 [$(M+Na)^+$], found 520.2369.

7-((*2R,3R,4S,5R*)-3,4-dihydroxy-5-(hydroxymethyl)tetrahydrofuran-2-yl)-

3,7-dihydro-*4H*-imidazo[4,5-d][1,2,3]triazin-4-one (AHXr; **4**)



20 **4**

To a stirred solution of **20** (109 mg, 0.219 mmol) in THF (3 mL) was added TBAF (1.0 M solution of THF, 0.5 mL, 0.482 mmol) at 0 °C under an argon atmosphere. After being stirred for 2 hours, the resulting mixture was concentrated under reduced pressure. The residue was purified by silica gel column chromatography ($CHCl_3$/MeOH = 15/1 to 5/1) to afford **4** (42.7 mg, 0.159 mmol, 72%) as a colorless solid.

Analytical data for compound **4**

m.p.: 170 ºC (decomp.)

$[\alpha]^{20}_D$ -59.7 (*c* 0.56, H_2O)

IR (film, cm^{-1}): 3344, 3122, 2947, 1712, 1418, 1323, 1222, 1082, 935.

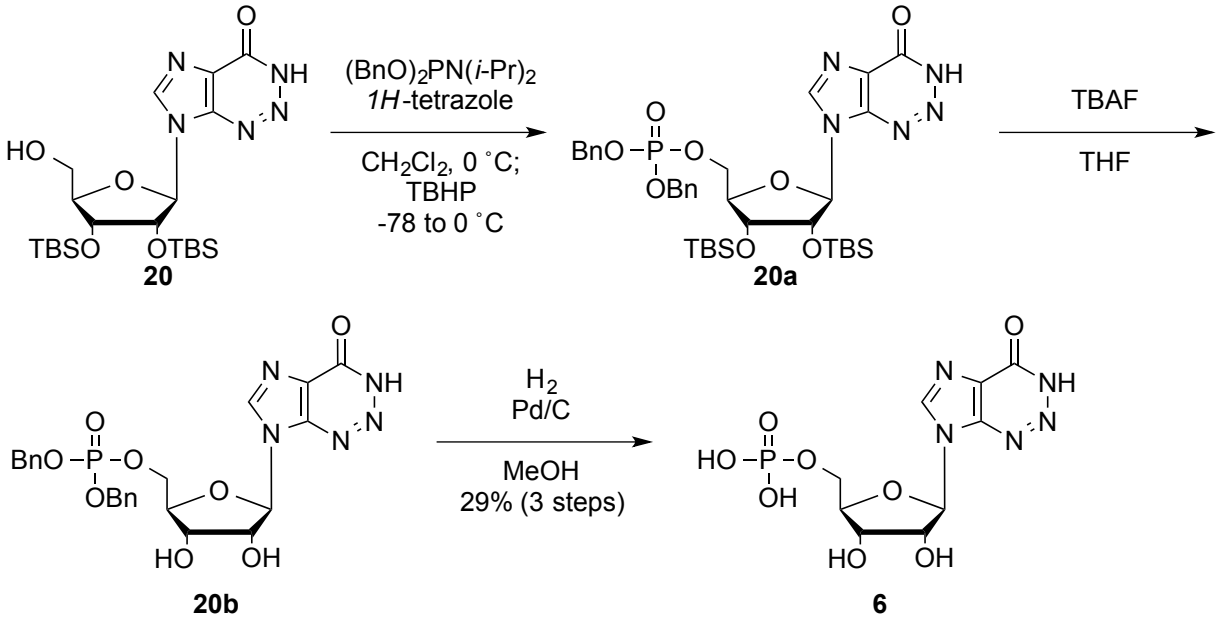
^1H NMR (500 MHz, CD_3OD): δ 8.54 (s, 1H), 6.16 (d, *J* = 5.1 Hz, 1H), 4.71 (dd, *J* = 5.1, 5.1 Hz, 1H), 4.36 (dd, *J* = 5.1, 4.0 Hz, 1H), 4.19-4.17 (m, 1H), 3.91 (dd, *J* = 12.5, 2.8 Hz, 1H), 3.78 (dd, *J* = 12.5, 3.4 Hz, 1H).

^{13}C NMR (125 MHz, CD_3OD): δ 160.1, 147.3, 143.4, 126.7, 91.4, 87.8, 76.4, 72.1, 62.9.

HRMS (ESI): Calculated for $C_9H_{10}N_5O_5$ 268.0674 [$(M-H)^-$], found 268.0674.

((*2R,3S,4R,5R*)-3,4-dihydroxy-5-(4-oxo-3,4-dihydro-*7H*-imidazo[4,5-d][1,2,3]triazin-7-yl)

tetrahydrofuran-2-yl)methyl dihydrogen phosphate (AHXR; **6**)



To a stirred solution of **20** (87.0 mg, 0.175 mmol) in CH_2Cl_2 (3 mL) were added *1H*-tetrazole (49 mg, 0.70 mmol) and dibenzyl *N*,*N*-diisopropylphosphoramidite (0.13 mL, 0.385 mmol) at 0 °C under an argon atmosphere. After being stirred at the same temperature for 20 minutes, the reaction mixture was added TBHP (5.5 M solution of *n*-decane, 0.13 mL, 0.70 mmol) at -78 ºC. After being warmed to 0 ºC for 1 hour, the resulting mixture was poured into saturated aqueous $Na_2S_2O_3$ and extracted with EtOAc. The organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The residue was passed through a short silica gel column chromatography (*n*-hexane/EtOAc = 3/1 to 1/3) to afford a crude material including **20a**.

To a stirred solution of the crude material including **20a** in THF (3 mL) was added TBAF (1.0 M solution of THF, 0.4 mL, 0.403 mmol) at 0 °C under an argon atmosphere. After being stirred for 40 minutes, the resulting mixture was concentrated under reduced pressure. The residue was passed through a short silica gel column chromatography ($CHCl_3$/MeOH = 10/1 to 3/1) to afford a crude material including **20b**.

A mixture of the crude material including **20b** and 5% Pd/C (19 mg, 8.93 mmol) in MeOH (2 mL) was stirred under ordinary hydrogen pressure (balloon) at room temperature for 30 minutes. The resulting mixture was filtered through a pad of Celite and concentrated under reduced pressure. The residue was purified by preparative TLC ($CHCl_3$/MeOH = 2/1 to 0/1) to afford **6** (17.8 mg, 51.0 μmol, 29%, 3 steps) as a colorless solid.

Analytical data for compound **6**

m.p.: 170 ºC (decomp.)

$[\alpha]^{20}_D$ -139 (*c* 0.21, H_2O).

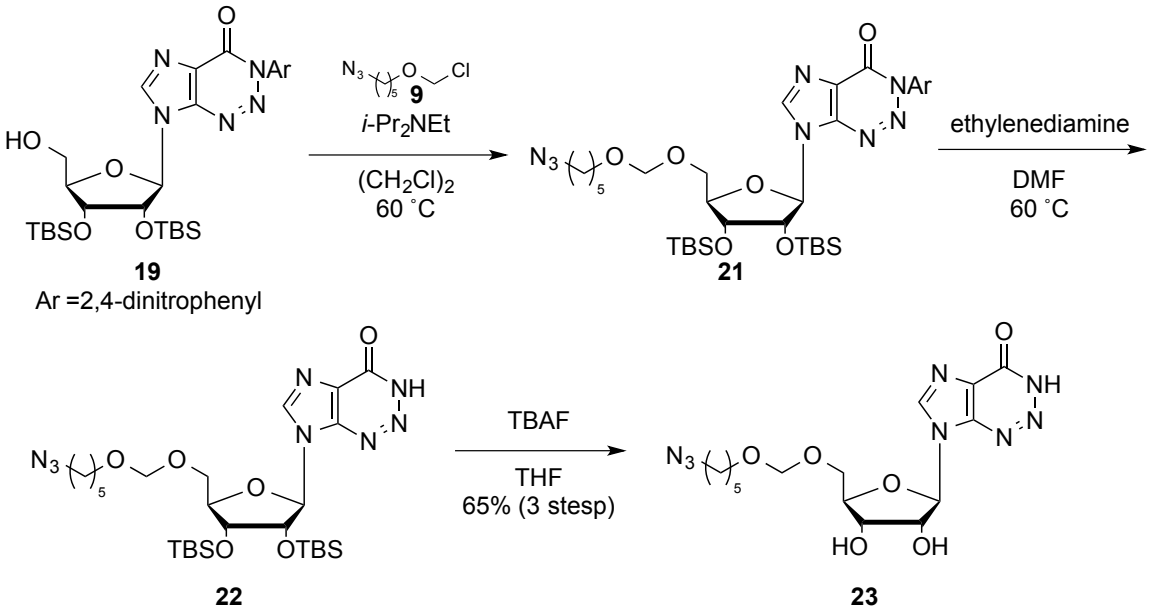
IR (film, cm^{-1}): 3250, 3120, 2945, 2885, 1708, 1545, 1522, 1420, 1196, 1067, 932.

^{1}H NMR (500 MHz, CD_3OD): δ 8.76 (s, 1H), 6.29 (d, *J* = 5.7 Hz, 1H), 4.72 (dd, *J* = 5.7, 5.1 Hz, 1H), 4.43 (dd, *J* = 5.1, 3.4 Hz, 1H), 4.29-4.26 (m, 1H), 4.16-4.08 (m, 2H).

^{13}C NMR (125 MHz, CD_3OD): δ 158.4, 147.5, 143.5, 126.8, 90.1, 86.6, 77.0, 72.4, 65.6.

HRMS (ESI): Calculated for $C_9H_{11}N_5O_8P$ 348.0400 [$(M-H)^-$], found 348.0341.

7-((*2R*,*3R*,*4S*,*5R*)-5-((((5-azidopentyl)oxy)methoxy)methyl)-3,4-dihydroxytetrahydrofuran-2-yl)-3,7-dihydro-*4H*-imidazo[4,5-d][1,2,3]triazin-4-one (**23**)



To a stirred solution of **19** (150 mg, 0.226 mmol) in $(CH_2Cl)_2$ (3 mL) were added *i*-Pr_2NEt (3.0 mL, 17.6 mmol) and 1-azido-5-(chloromethoxy)pentane (**9**) (300 mg, about 1.69 mmol) at 0 ºC under an argon atmosphere. The reaction mixture was stirred at 60 ºC for 12 hours. Then, the resulting mixture was poured into saturated aqueous NH_4Cl at 0 °C, and extracted with CH_2Cl_2. The organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The residue was passed through a short silica gel column chromatography (*n*-hexane/EtOAc = 3/1 to 1/1) to afford a crude material including **21**.

To a stirred solution of the crude material including **21** in DMF (3 mL) was added ethylenediamine (1 mL) at room temperature. After being stirred at 60 ºC for 4 hours, the resulting mixture was poured into 1 M HCl at 0 °C, and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure to afford a crude material including **22**.

To a stirred solution of the crude material including **22** in THF (3 mL) was added TBAF (1.0 M solution of THF, 0.55 mL, 0.550 mmol) at 0 °C under an argon atmosphere. After being stirred for 40 minutes, the resulting mixture was concentrated under reduced pressure. The residue was purified by silica gel column chromatography ($CHCl_3$/MeOH = 20/1 to 10/1) to afford **23** (60.0 mg, 0.146 mmol, 65%, 3 steps) as a colorless oil.

Analytical data for compound **23**

$[\alpha]^{20}_D$ −14.2 (*c* 0.96, CH_3OH).

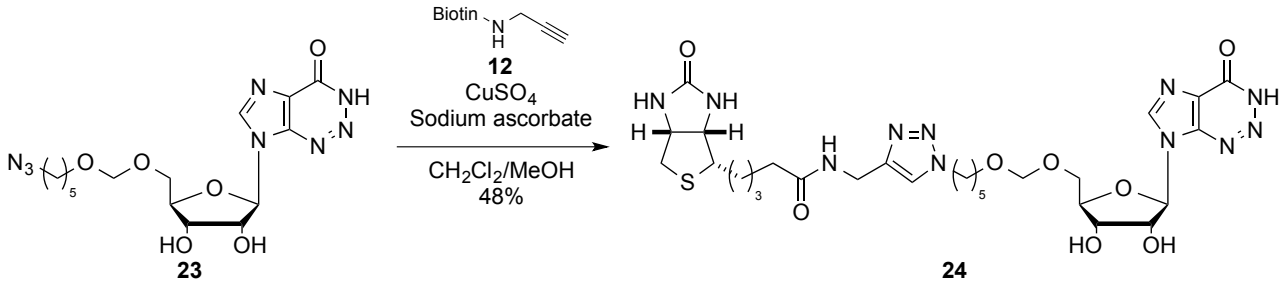
IR (film, cm^{-1}): 3410, 2477, 2257, 1650, 1049, 1026, 1001, 826.

^1H NMR (500 MHz, d_6-acetone): δ 8.62 (s, 1H), 6.27 (d, *J* = 4.0 Hz, 1H), 4.81-4.75 (m, 3H), 4.49 (dd, *J* = 4.5, 4.0 Hz, 1H), 4.29 (dt, *J* = 7.9, 4.0 Hz, 1H), 3.90 (dd, *J* = 11.1, 4.0 Hz, 1H), 3.83 (dd, *J* = 11.1, 4.0 Hz, 1H), 3.59-3.52 (m, 2H), 3.32 (t, *J* = 6.8 Hz, 2H), 1.63-1.55 (m, 4H), 1.47-1.39 (m, 2H).

^{13}C NMR (125 MHz, d_6-acetone): δ 155.0, 146.0, 142.6, 128.3, 96.2, 90.5, 85.1, 76.3, 71.7, 68.4, 67.8, 51.9, 30.0, 29.3, 24.2.

HRMS (ESI): Calculated for $C_{15}H_{21}N_8O_6$ 409.1579 [$(M-H)^-$], found 409.1578.

N-((1-(5-((((*2R,3S,4R,5R*)-3,4-dihydroxy-5-(4-oxo-3,4-dihydro-*7H*-imidazo[4,5-d][1,2,3]triazin-7-yl)tetrahydrofuran-2-yl)methoxy)methoxy)pentyl)-*1H*-1,2,3-triazol-4-yl)methyl)-5-((*3aS,4S,6aR*)-2-oxohexahydro-*1H*-thieno[3,4-d]imidazol-4-yl)pentanamide (**24**)



To a solution of **23** (26 mg, 90 μmol) and **12** (25 mg, 90 μmol) in a 1 : 1 mixture of CH_2Cl_2 and MeOH (total 5 mL) was added $CuSO_4$ (7.2 mg, 45 μmol) and sodium ascorbate (9.0 mg, 45 μmol) at room temperature. The reaction mixture was stirred for 24 hours. Then, the resulting mixture was filtered through a pad of Celite and concentrated under reduced pressure. The residue was purified by silica gel column chromatography ($CHCl_3$/MeOH = 10:1 to 7:1) to afford **24** (30 mg, 43 μmol, 48%) as a colorless oil.

Analytical data for compound **24**

$[\alpha]^{20}_D$ +4.06 (*c* 0.27, CH_3OH).

IR (film, cm^{-1}): 3450, 2950, 2746, 1678, 1512, 1450, 1120.

^1H NMR (500 MHz CD_3OD): δ 8.51 (s, 1H), 7.86 (s, 1H), 6.21 (d, *J* = 4.6 Hz, 1H), 4.74-4,71 (m, 3H), 4.48 (dd, *J* = 8.0, 4.0 Hz, 1H), 4.43 (s, 2H), 4.40 (dd, *J* = 5.2, 4.8 Hz, 1H), 4.35 (t, *J* = 6.9 Hz, 2H), 4.30-4.25 (m, 2H), 3.89 (dd, *J* = 11.2, 2.9 Hz, 1H), 3.78 (dd, *J* = 11.2, 2.9 Hz, 1H), 3.52-3.47 (m, 2H), 3.21-3.16 (m, 1H), 2.91 (dd, *J* = 12.6, 4.6 Hz, 1H), 2.69 (d, *J* = 12.6 Hz, 1H), 2.24 (t, *J* = 7.5 Hz, 2H), 1.88-1.82 (m, 2H), 1.75-1.54 (m, 6H), 1.44-1.39 (m, 2H), 1.34-1.29(m, 2H).

^{13}C NMR (125 MHz CD_3OD): δ 176.0, 166.1, 154.9, 147.6, 146.3, 142.9, 126.4, 124.2, 119.8, 91.0, 85.4, 76.2, 71.9, 68.8, 68.0, 63.3, 61.6, 57.0, 51.3, 41.0, 36.6, 35.6, 31.0, 30.4, 29.7, 29.4, 26.7, 24.2.

HRMS (ESI): Calculated for $C_{28}H_{41}N_{11}O_8SNa$ 714.2752 [$(M+Na)^+$], found 714.2762.

II. Bioassay

Sterilized rice seeds (*Oryza sativa* L. *cv*. Nipponbare) were germinated in the dark at 28 °C for 2 days. Five germinated seeds were transferred in each test tube (30 × 200 mm) containing 3 mL of nutrient solution (0.5 mM NH_4NO_3, 0.3 mM Na_2HPO_4, 0.15 mM K_2SO_4, 0.2 mM $MgCl_2$, 0.1 mM $CaC1_2$, 23 μM Fe-ethylenediaminetetraacetic acid, 25 μM H_3BO_3, 4.5 μM $MnSO_4$, 150 nM $CuSO_4$, 350 nM $ZnSO_4$ and 50 nM Na_2MoO_4) and incubated for 3 days and then treated with 3 mL of 0.1 mM of each samples. The seedlings were grown under 18-hr photoperiod at 28 °C for 8 days and the samples were replaced every two days. All the procedures were performed in sterile environments. The effect of AHX derivatives on plant elongation was measured using a ruler.

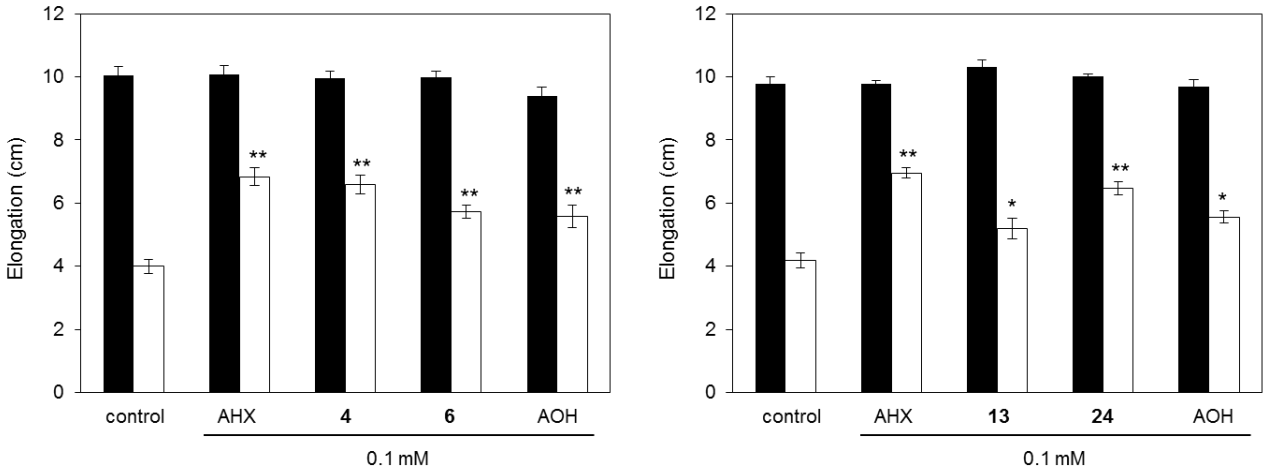


Figure S1. The growth promotion of rice by AHX and its analogues.

Closed and open columns indicate shoot and root, respectively. Asterisk indicates significant difference from the control (Student's *t*-test, $*P < 0.05$, $**P < 0.01$). Results are the mean ± SEM ($n = 15$).

III. Reference

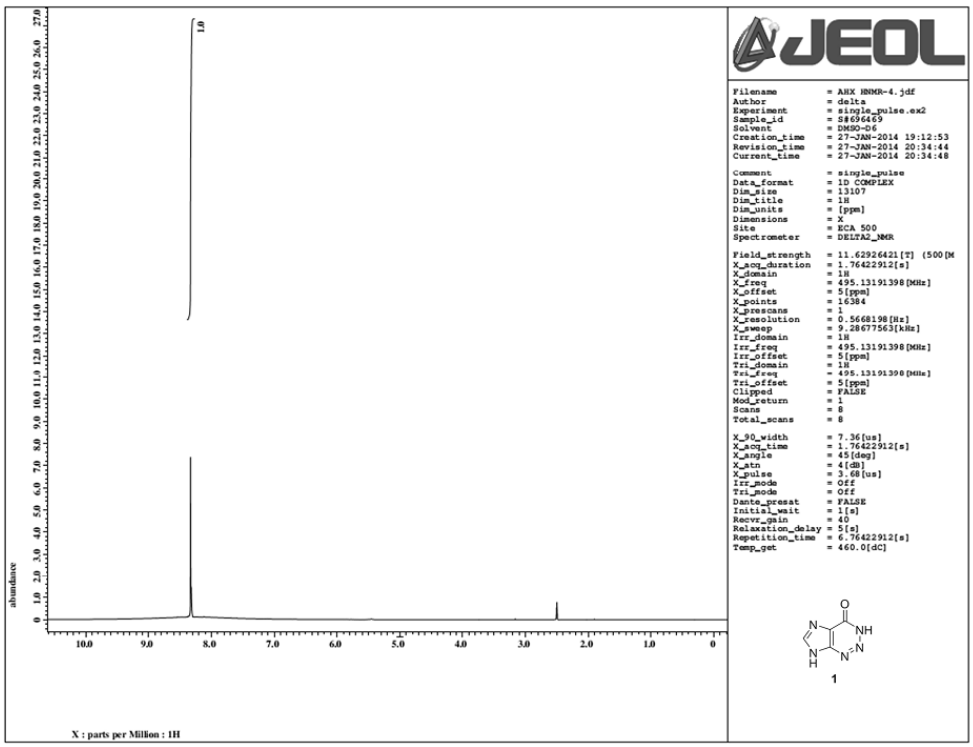
1) C.-V. T. Vo, A. Mitchell, J. W. Bode, *J. Am. Chem. Soc.,* 2011, **133**, 14082.

2) J. I. Gavrilyuk, U. Wuellner, C. F. Barbas III, *Bio. Med. Chem. Lett*., 2009, **19**, 1421.

3) (a) A. M. Kawasaki, L. B. Townsend, *Nucleic acid Chem*., 1991, 298; (b) M. Iwasaki, F. Eckstein, *J. Org. Chem*., 1978, **43**, 3044.

4) M. B. Erlacher, L. Kathrin, B. Wotzel, R. Rieder, R. Micura, N. Polacek, *J. Am. Chem. Soc.,* 2006, **128**, 4453.

IV. ^{1}HNMR and ^{13}CNMR spectral data



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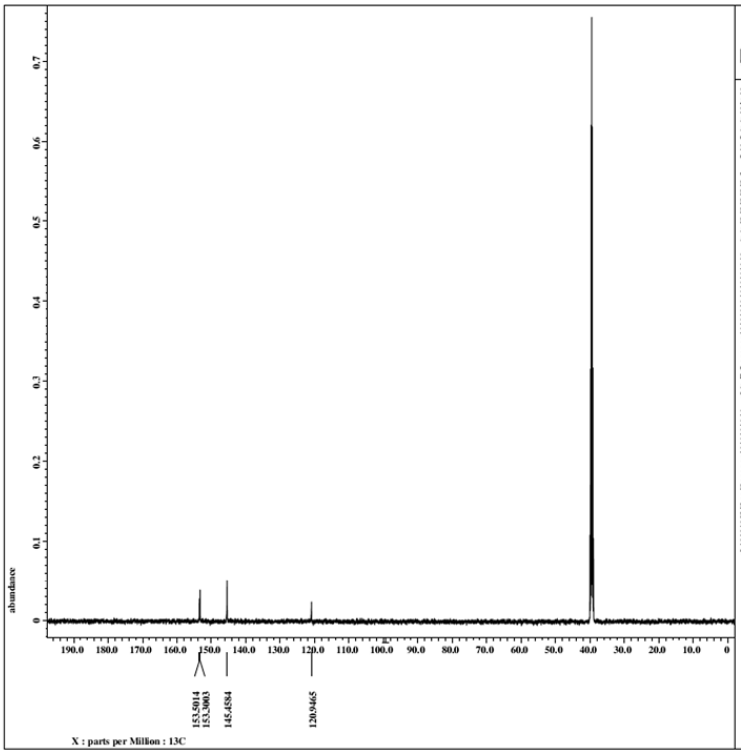

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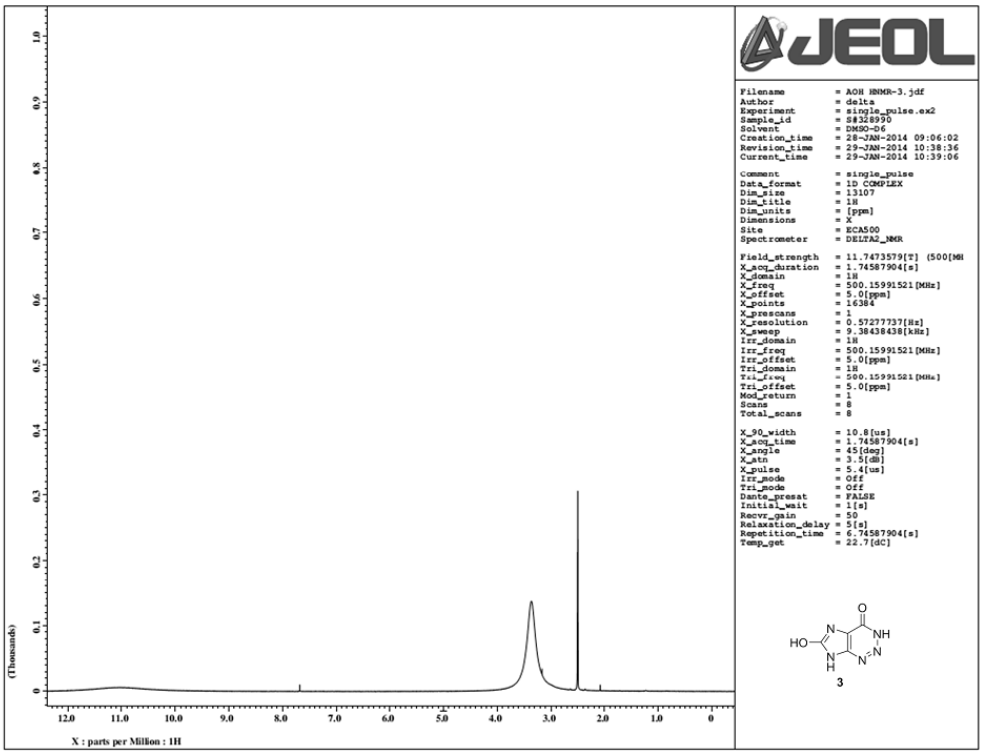
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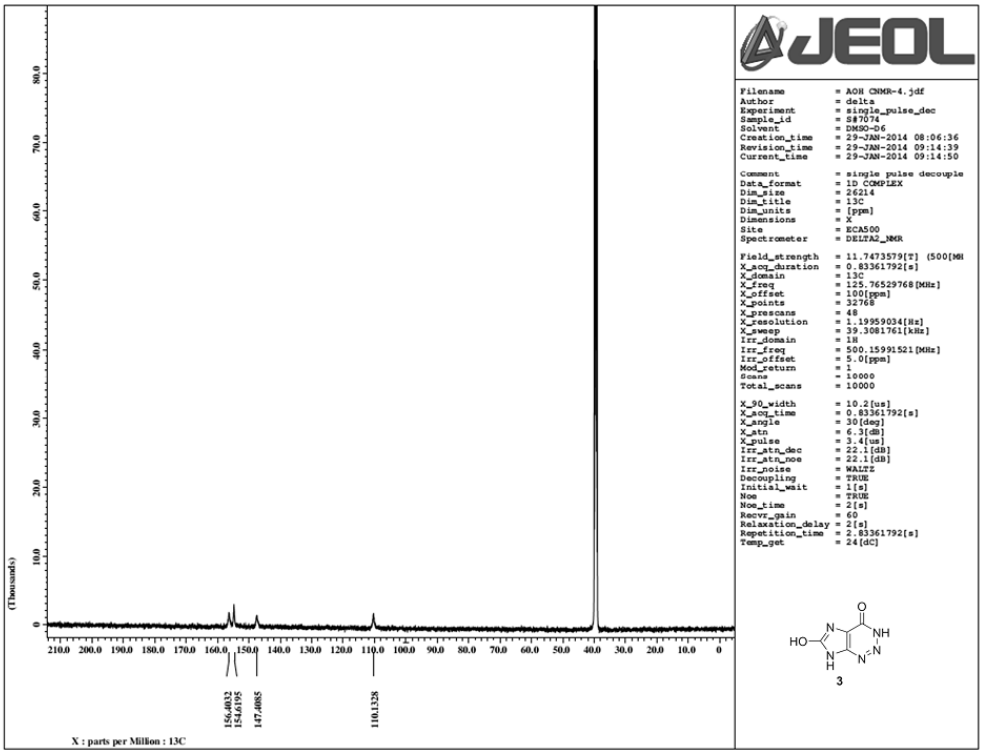
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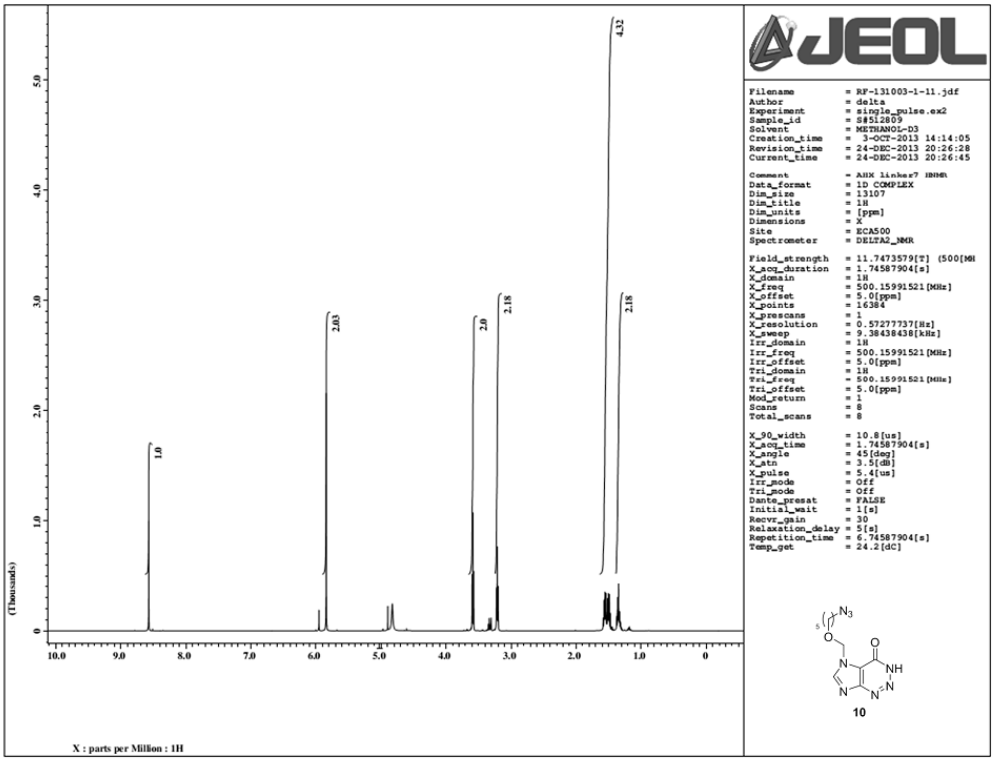
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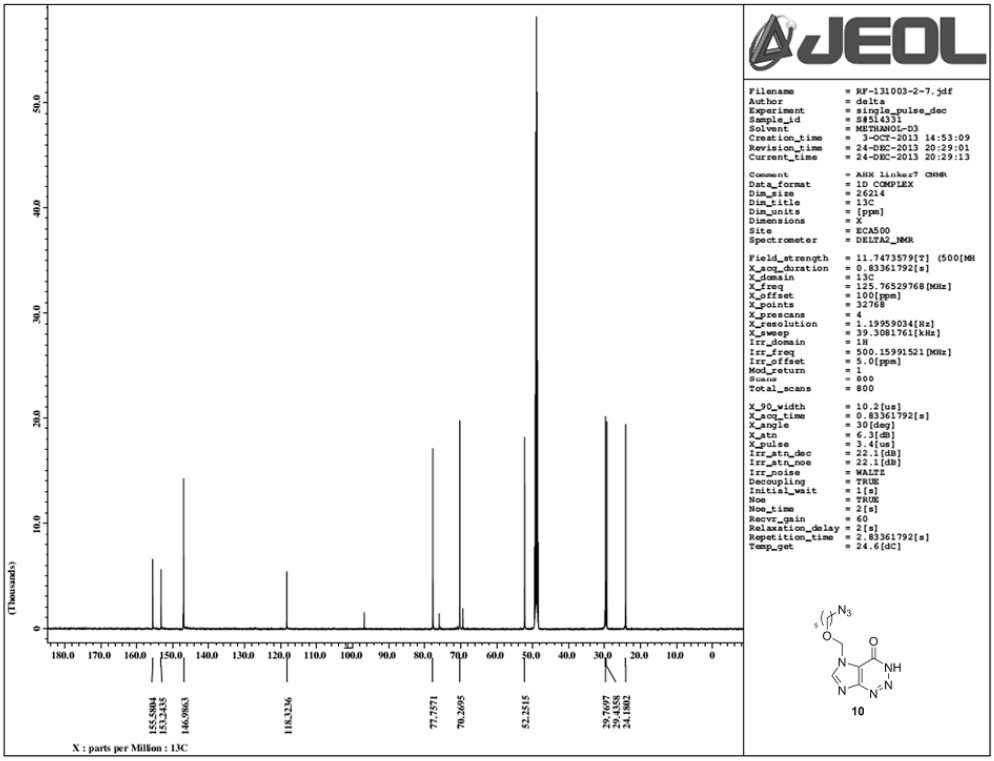
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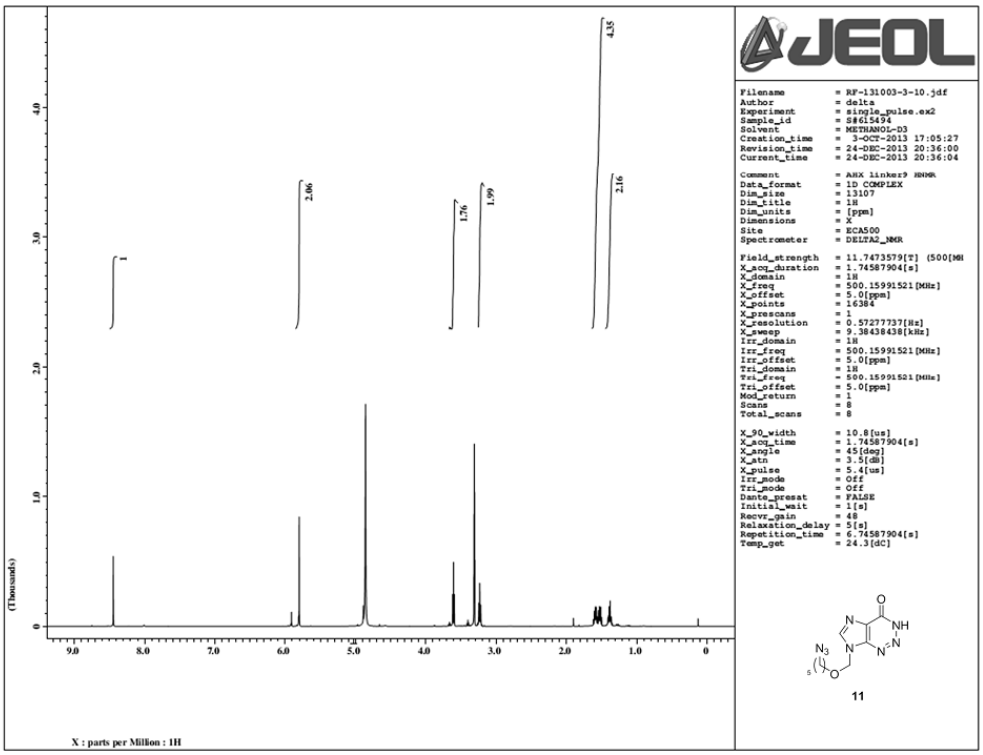
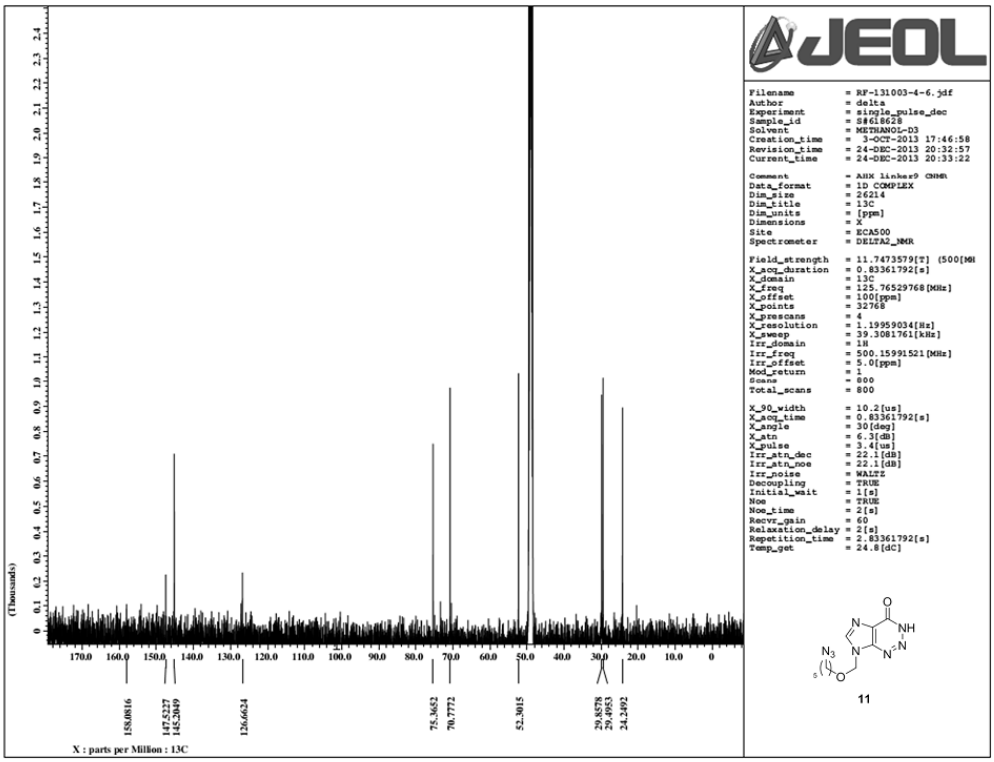
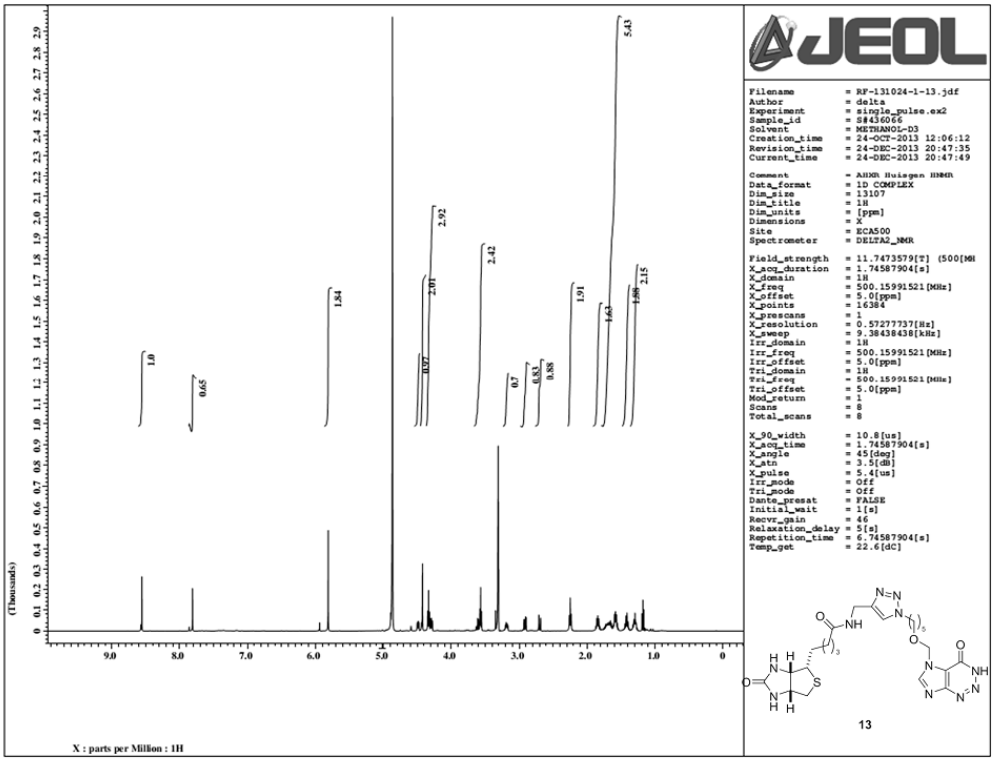
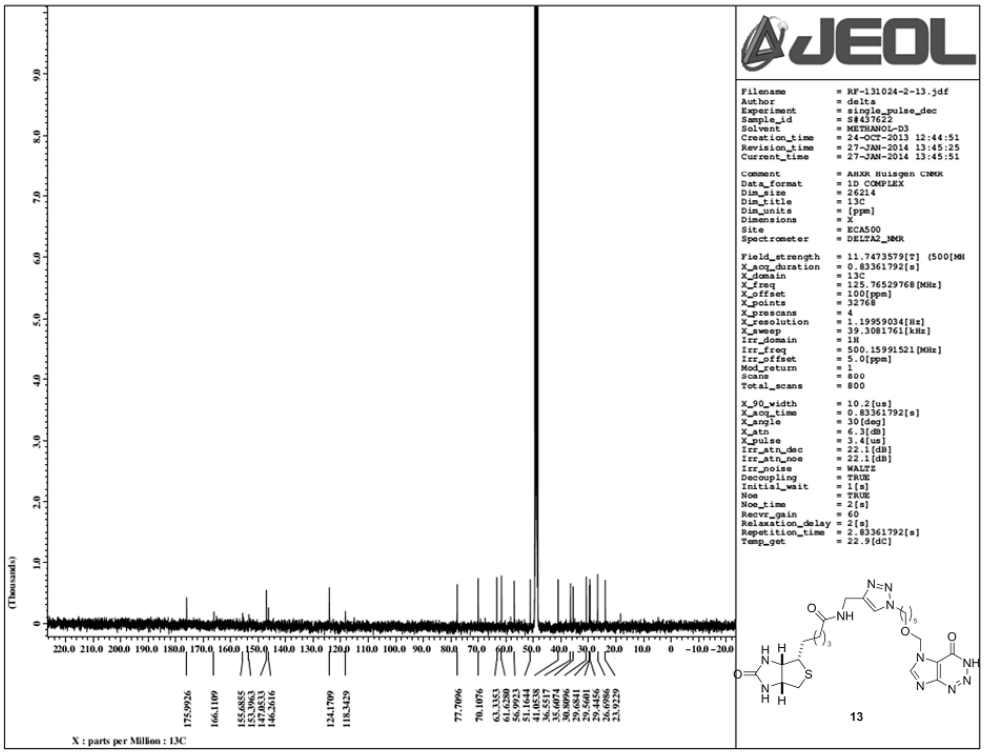
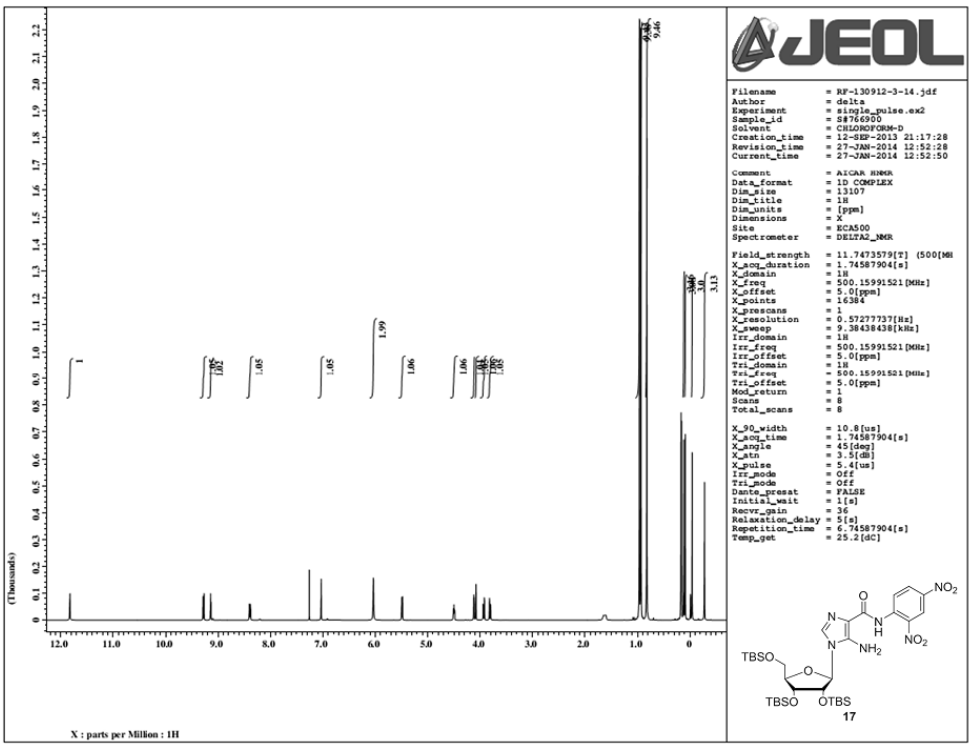
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Mod_return = 1
Scans = 800
Total_scans = 800
X_90_width = 10.2[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 6.3[dB]
X_pulse = 3.4[us]
Irr_atn_dec = 22.1[dB]
Irr_atn_noe = 22.1[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 24.6[dC]
10

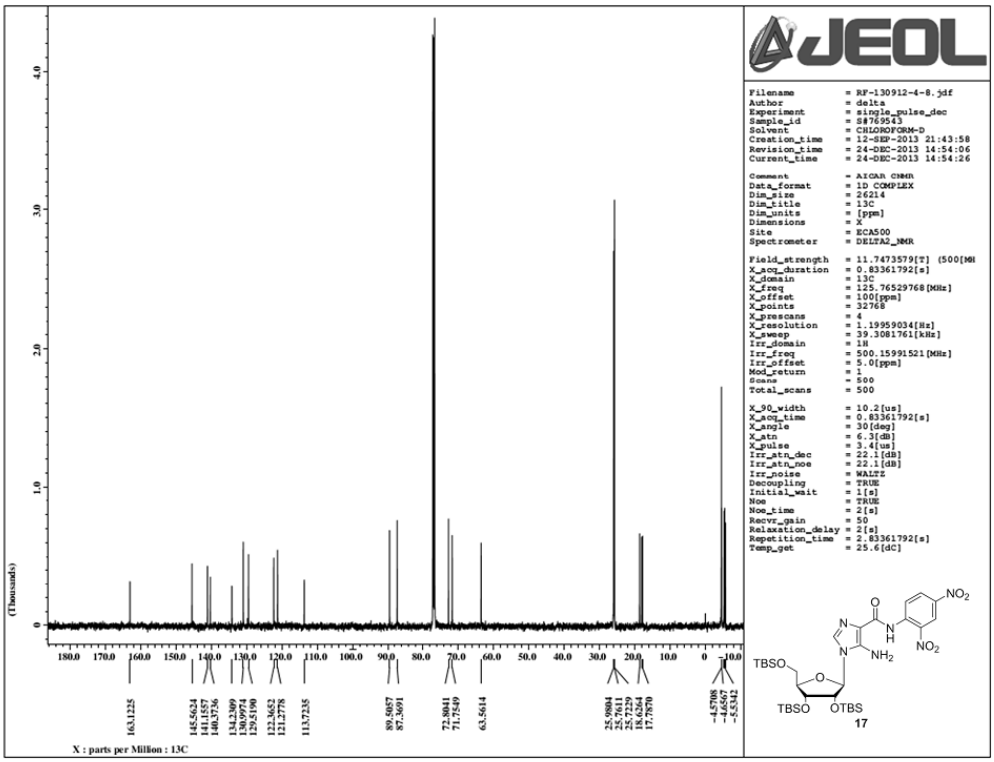

4.35
2.16
2.06
1.76
1.99
1
(Thousands)
X : parts per Million : 1H
Filename = RF-131003-3-10.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#615494
Solvent = METHANOL-D3
Creation_time = 3-OCT-2013 17:05:27
Revision_time = 24-DEC-2013 20:36:00
Current_time = 24-DEC-2013 20:36:04
Comment = AHX linker9 HNMR
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 10.8[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.5[dB]
X_pulse = 5.4[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 24.3[dC]
11

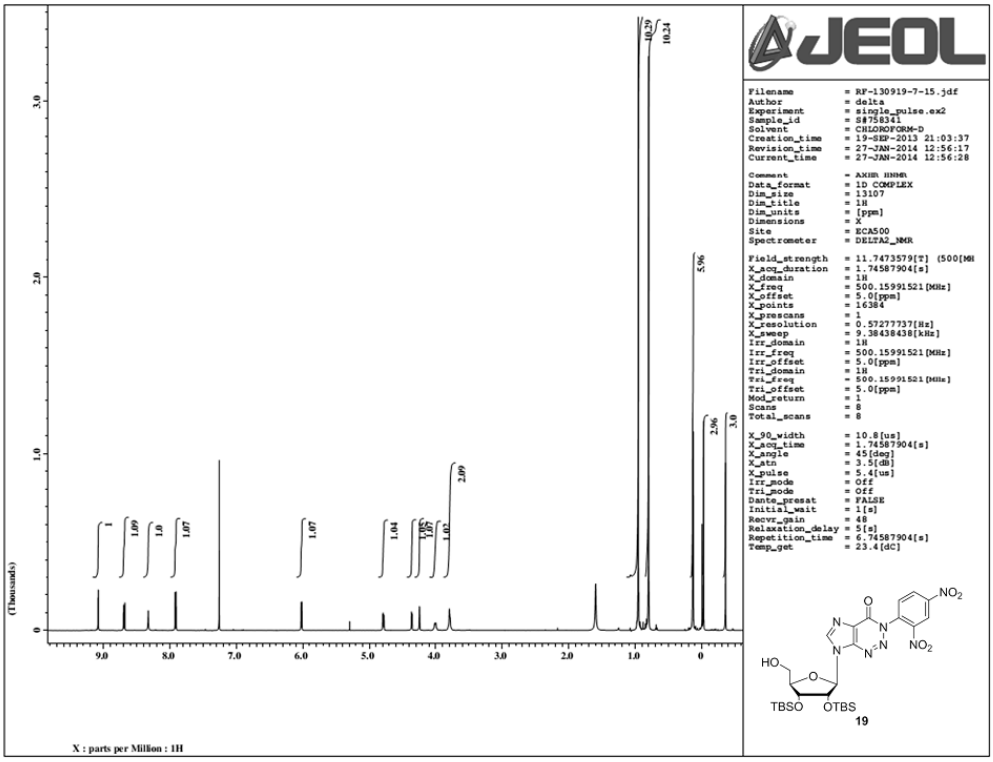

(Thousands)
158.0816
147.5227
145.2049
126.6624
75.3652
70.7772
52.3015
29.8578
29.4953
24.2492
X : parts per Million : 13C
Filename = RF-131003-4-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#618628
Solvent = METHANOL-D3
Creation_time = 3-OCT-2013 17:46:58
Revision_time = 24-DEC-2013 20:32:57
Current_time = 24-DEC-2013 20:33:22
Comment = AHX linker9 CNMR
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Mod_return = 1
Scans = 800
Total_scans = 800
X_90_width = 10.2[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 6.3[dB]
X_pulse = 3.4[us]
Irr_atn_dec = 22.1[dB]
Irr_atn_noe = 22.1[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 24.8[dC]
11

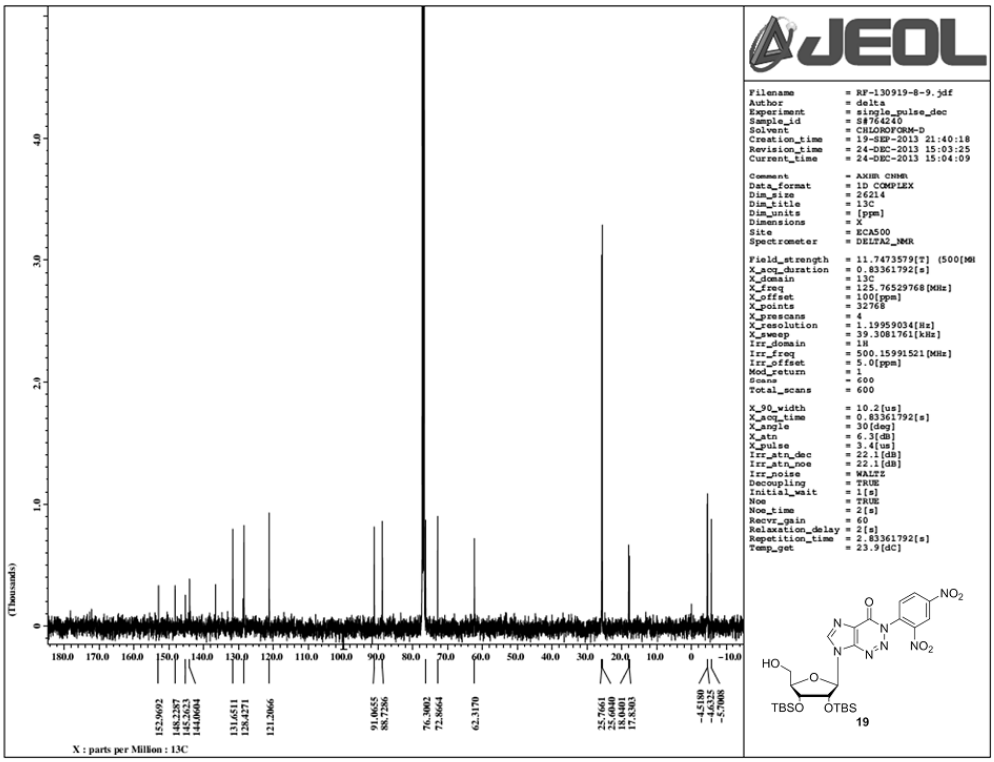

(Thousands)
X : parts per Million : 1H
1.0
0.65
1.84
2.01
2.07
2.92
2.42
0.7
0.83
0.88
1.91
1.63
1.98
2.15
5.43
Filename = RF-131024-1-13.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#436066
Solvent = METHANOL-D3
Creation_time = 24-OCT-2013 12:06:12
Revision_time = 24-DEC-2013 20:47:35
Current_time = 24-DEC-2013 20:47:49
Comment = AHXR Huisgen HNMR
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 10.8[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.5[dB]
X_pulse = 5.4[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.6[dC]
13

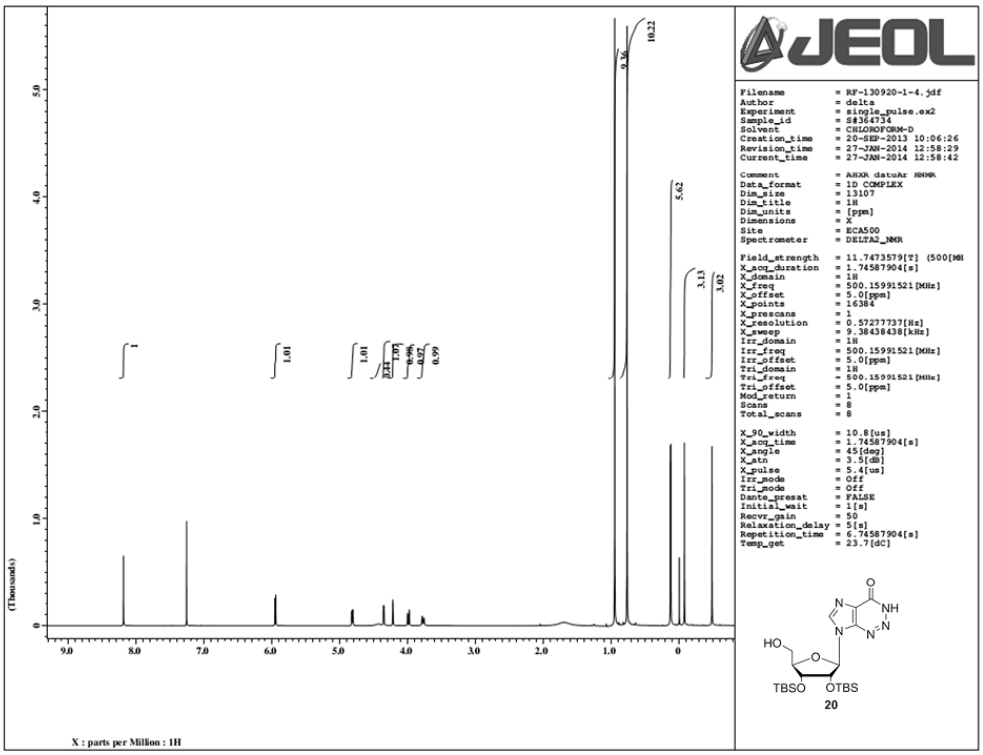

(Thousands)
X : parts per Million : 13C
175.9926
166.1109
155.6855
153.3963
147.4581
146.2616
124.1709
118.3429
77.7096
70.1076
63.3353
61.6280
56.9923
51.1614
41.0838
36.5517
35.6071
30.8096
29.6841
29.5481
29.1456
26.6986
23.9229
Filename = RF-131024-2-13.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#437622
Solvent = METHANOL-D3
Creation_time = 24-OCT-2013 12:44:51
Revision_time = 27-JAN-2014 13:45:25
Current_time = 27-JAN-2014 13:45:51
Comment = AHXR Huisgen CNMR
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Mod_return = 1
Scans = 800
Total_scans = 800
X_90_width = 10.2[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 6.3[dB]
X_pulse = 3.4[us]
Irr_atn_dec = 22.1[dB]
Irr_atn_noe = 22.1[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 22.9[dC]
13

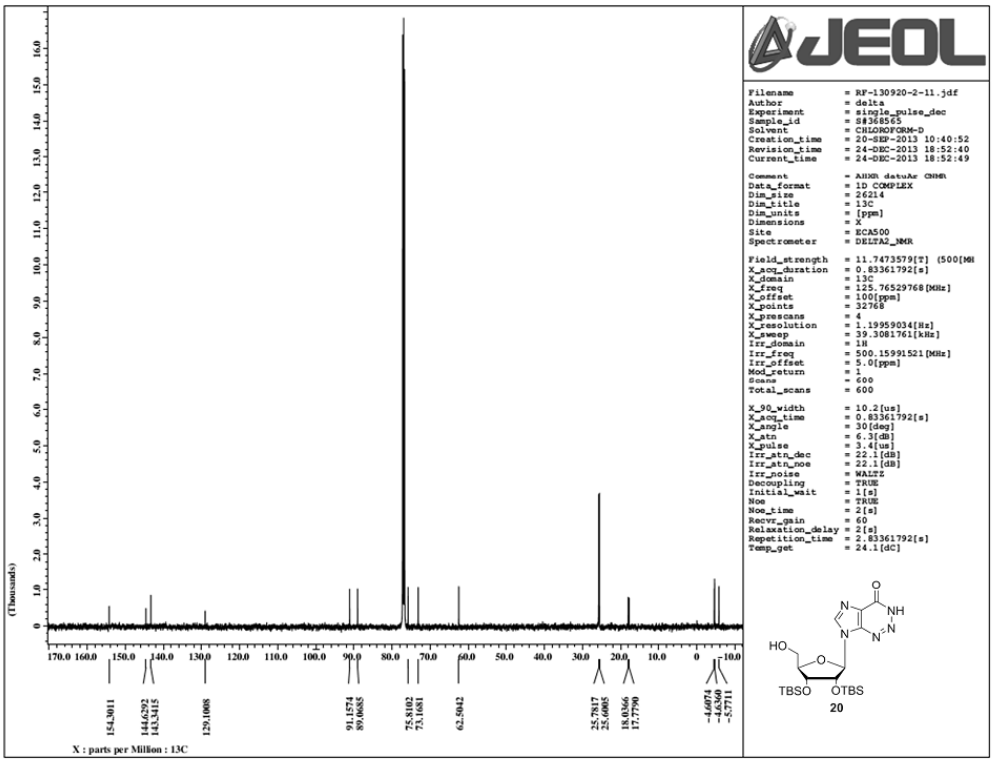

JEOL
Filename = RF-130912-3-14.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S8769800
Solvent = CHLOROFORM-D
Creation_time = 12-SEP-2013 21:17:28
Revision_time = 27-JAN-2014 12:52:28
Current_time = 27-JAN-2014 12:52:50
Comment = AICAR NMR
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737[Hz]
X_sweep = 9.3843843[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 10.8[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.5[dB]
X_pulse = 5.4[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 36
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 25.2[dC]
NO2
NO2
TBSO
TBSO
OTBS
17
X : parts per Million : 1H

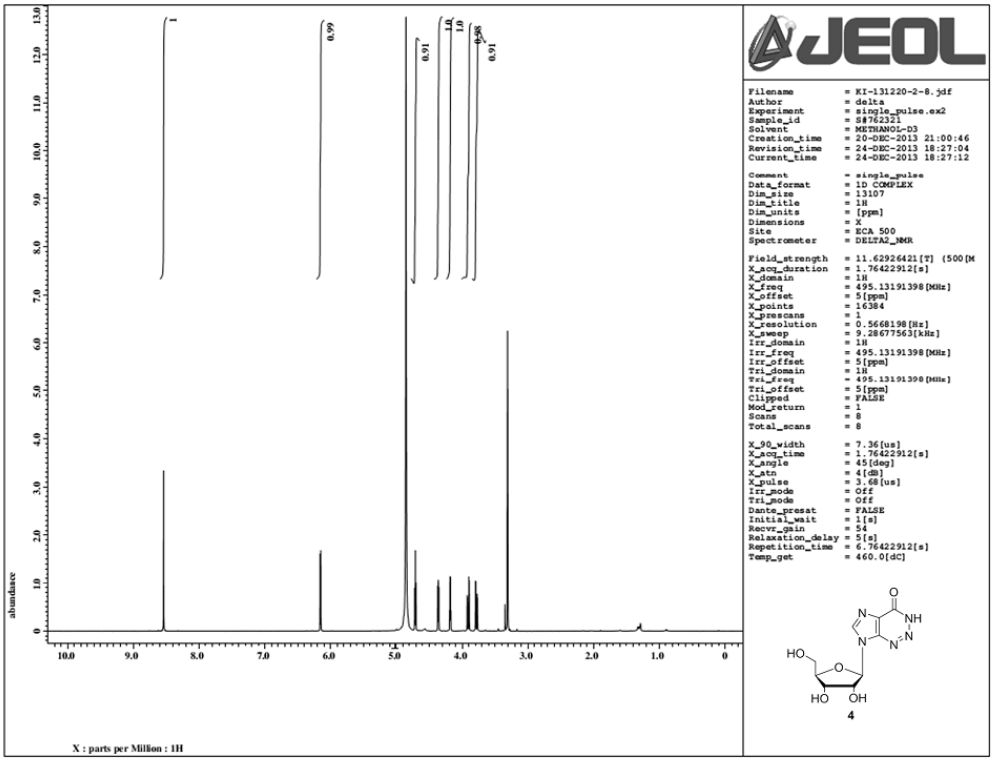

JEOL
Filename = RF-130912-4-8.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S8769543
Solvent = CHLOROFORM-D
Creation_time = 12-SEP-2013 21:43:58
Revision_time = 24-DEC-2013 14:54:06
Current_time = 24-DEC-2013 14:54:26
Comment = AICAR CNMR
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Mod_return = 1
Scans = 500
Total_scans = 500
X_90_width = 10.2[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 6.3[dB]
X_pulse = 3.4[us]
Irr_atn_dec = 22.1[dB]
Irr_atn_noe = 22.1[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 25.6[dC]
NO2
NO2
TBSO
TBSO
OTBS
17
163.1225
145.5624
141.1557
140.3536
134.2389
130.9974
129.5190
122.3652
121.2778
113.7235
89.5057
87.3091
72.8041
71.7549
63.5614
25.9884
25.7611
25.7229
18.6264
17.7870
-4.5708
-4.6587
-5.5542
X : parts per Million : 13C

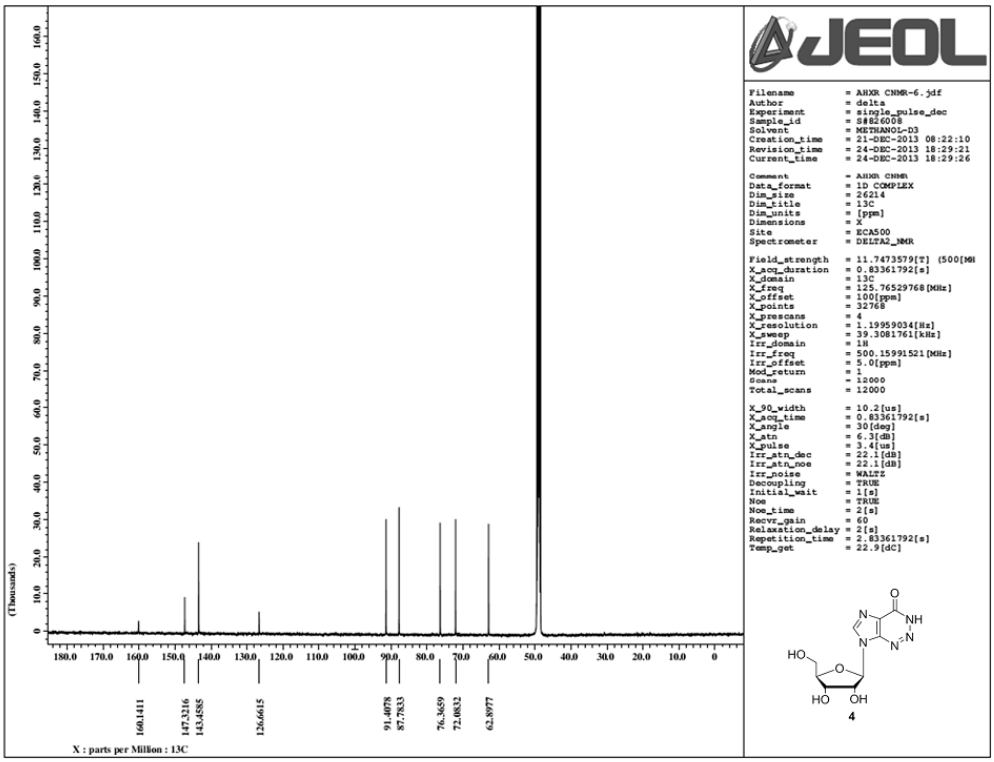

48.29
10.24
5.96
2.96
3.0
2.09
1.07
1.04
1.03
1.02
1.09
1.0
1
(Thousands)
X : parts per Million : 1H
Filename = RF-130919-7-15.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#758341
Solvent = CHLOROFORM-D
Creation_time = 19-SEP-2013 21:03:37
Revision_time = 27-JAN-2014 12:56:17
Current_time = 27-JAN-2014 12:56:28
Comment = AXHR HNMR
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 10.8[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.5[dB]
X_pulse = 5.4[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 23.4[dC]
NO2
NO2
HO
TBSO
OTBS
19

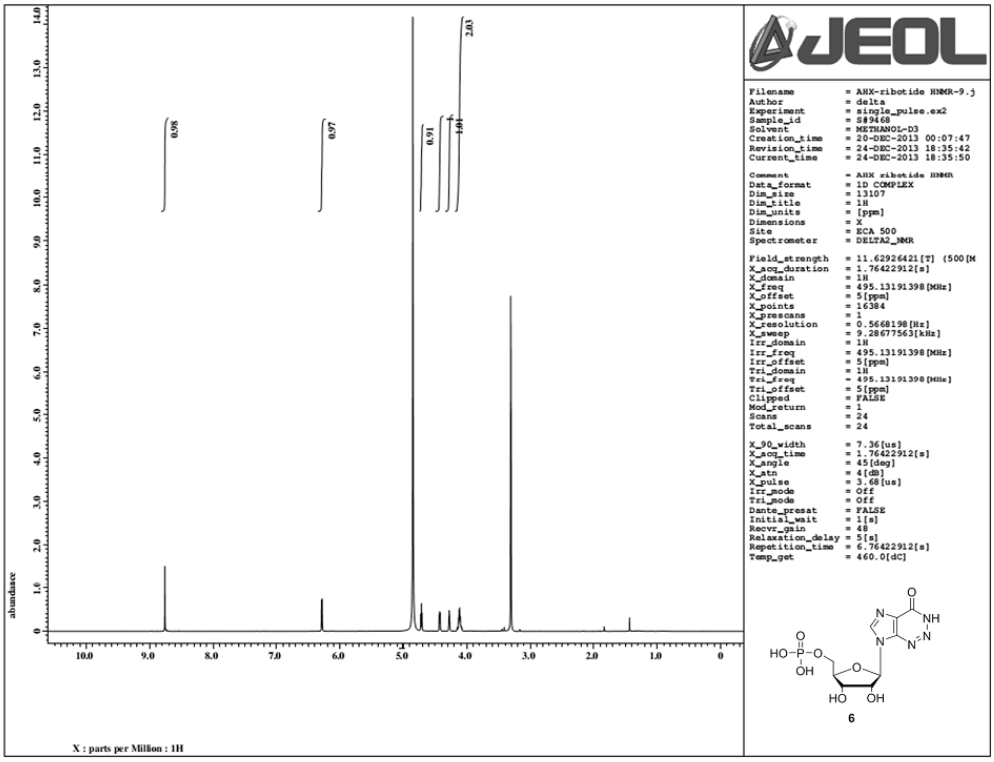

(Thousands)
X : parts per Million : 13C
152.9692
148.2282
148.2023
144.0684
131.6511
128.4271
121.2066
91.0655
88.7286
76.3002
72.3664
62.3170
25.7661
25.6040
18.1681
17.8303
-4.5180
-4.6325
-5.7008
Filename = RF-130919-8-9.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#764240
Solvent = CHLOROFORM-D
Creation_time = 19-SEP-2013 21:40:18
Revision_time = 24-DEC-2013 15:03:25
Current_time = 24-DEC-2013 15:04:09
Comment = AXHR CNMR
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Mod_return = 1
Scans = 600
Total_scans = 600
X_90_width = 10.2[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 6.3[dB]
X_pulse = 3.4[us]
Irr_atn_dec = 22.1[dB]
Irr_atn_noe = 22.1[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 23.9[dC]
NO2
NO2
HO
TBSO
OTBS
19

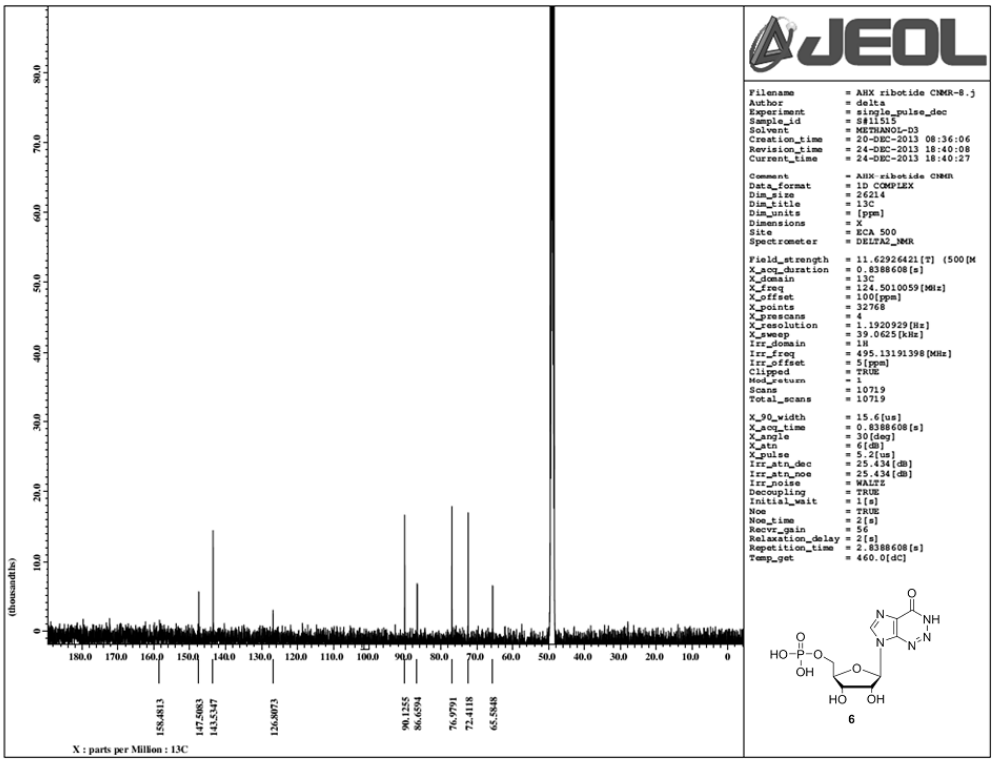

JEOL
Filename = RF-130920-1-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#364734
Solvent = CHLOROFORM-D
Creation_time = 20-SEP-2013 10:06:26
Revision_time = 27-JAN-2014 12:58:29
Current_time = 27-JAN-2014 12:58:42
Comment = AHXR datuAr HNMR
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 10.8[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.5[dB]
X_pulse = 5.4[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 23.7[dC]
HO
TBSO
OTBS
20
X : parts per Million : 1H

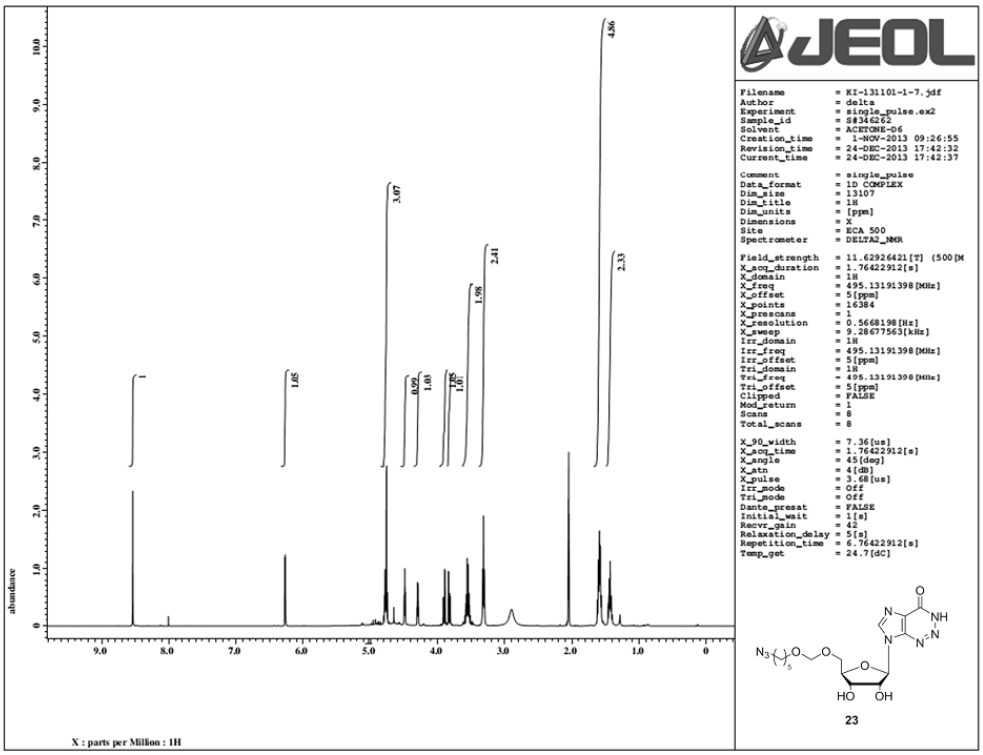

JEOL
Filename = RF-130920-2-11.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#368565
Solvent = CHLOROFORM-D
Creation_time = 20-SEP-2013 10:40:52
Revision_time = 24-DEC-2013 18:52:40
Current_time = 24-DEC-2013 18:52:49
Comment = AHXR datuAr CNMR
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Mod_return = 1
Scans = 600
Total_scans = 600
X_90_width = 10.2[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 6.3[dB]
X_pulse = 3.4[us]
Irr_atn_dec = 22.1[dB]
Irr_atn_noe = 22.1[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 24.1[dC]
HO
TBSO
OTBS
20
X : parts per Million : 13C

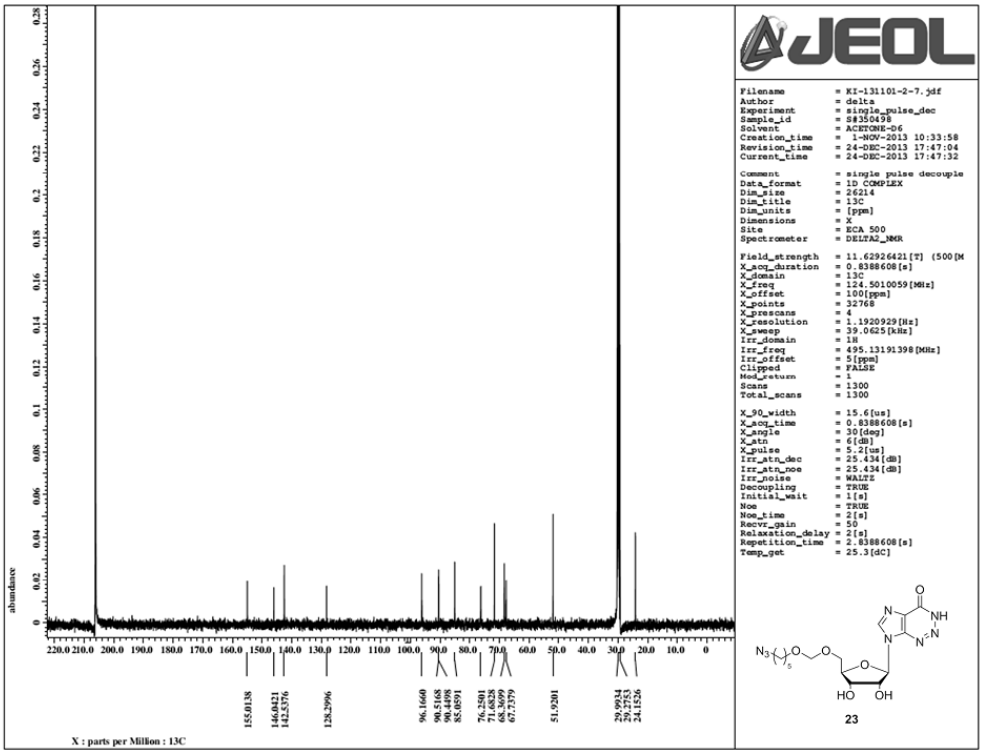

1
0.99
0.91
1.0
1.0
0.98
0.91
abundance
X : parts per Million : 1H
JEOL
Filename = KI-131220-2-8.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#762321
Solvent = METHANOL-D3
Creation_time = 20-DEC-2013 21:00:46
Revision_time = 24-DEC-2013 18:27:04
Current_time = 24-DEC-2013 18:27:12
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 7.36[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 3.68[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 54
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 460.0[dC]
HO
O
N
NH
HO
OH
4

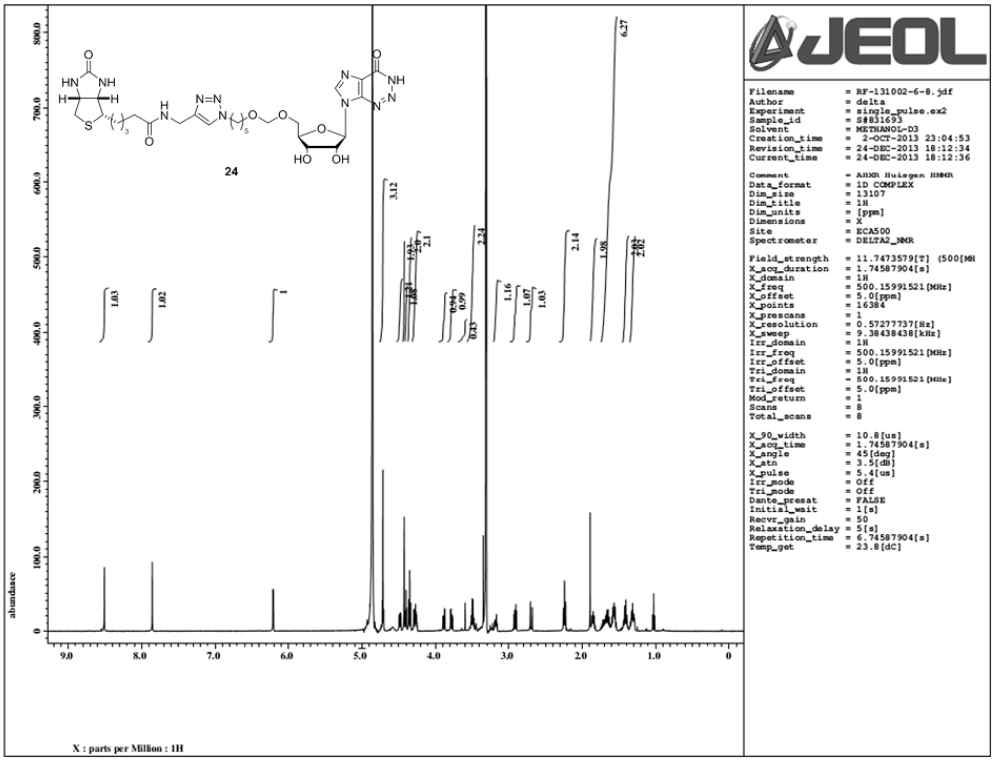

(Thousands)
160.1411
147.3216
143.4585
126.6615
91.4078
87.7833
76.3659
72.0832
62.8977
X : parts per Million : 13C
JEOL
Filename = AHXR CNMR-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#826008
Solvent = METHANOL-D3
Creation_time = 21-DEC-2013 08:22:10
Revision_time = 24-DEC-2013 18:29:21
Current_time = 24-DEC-2013 18:29:26
Comment = AHXR CNMR
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Mod_return = 1
Scans = 12000
Total_scans = 12000
X_90_width = 10.2[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 6.3[dB]
X_pulse = 3.4[us]
Irr_atn_dec = 22.1[dB]
Irr_atn_noe = 22.1[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 22.9[dC]
HO
O
N
NH
HO
OH
4

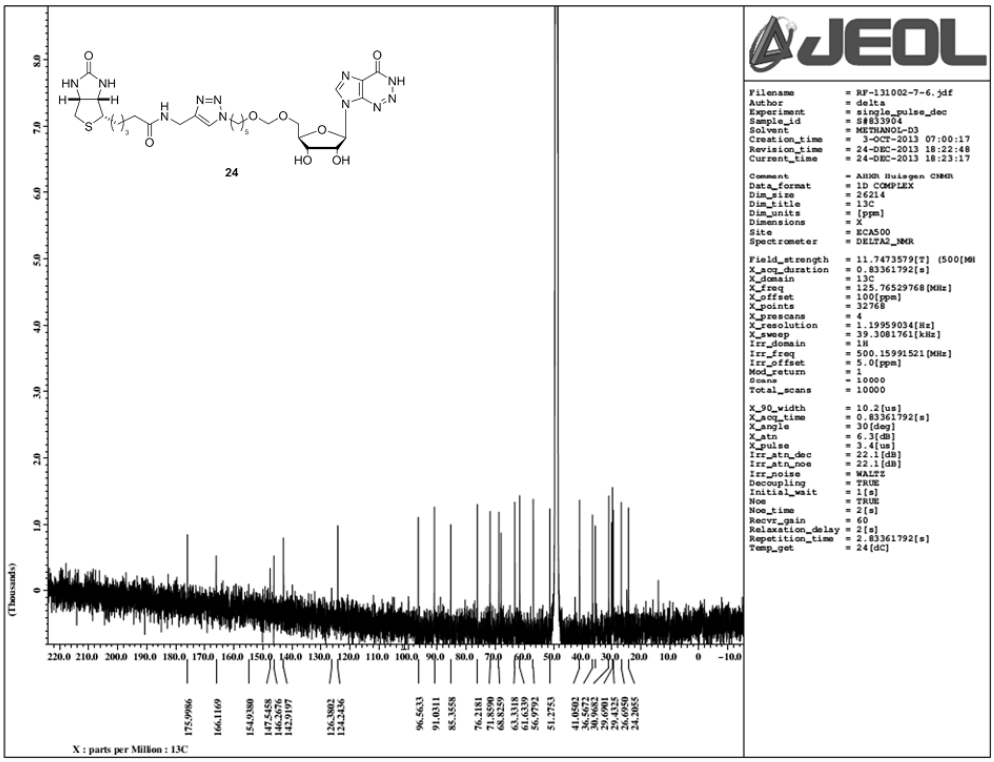

0.98
0.97
0.91
1.01
2.03
abundance
X : parts per Million : 1H
JEOL
Filename = AHX-ribotide HNMR-9.j
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#9468
Solvent = METHANOL-D3
Creation_time = 20-DEC-2013 00:07:47
Revision_time = 24-DEC-2013 18:35:42
Current_time = 24-DEC-2013 18:35:50
Comment = AHX ribotide HNMR
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 7.36[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 3.68[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 460.0[dC]
6


(thousandths)
158.4813
147.5083
143.5347
126.8073
90.1255
86.6594
76.9791
72.4118
65.5848
X : parts per Million : 13C
JEOL
Filename = AHX ribotide CNMR-8.j
Author = delta
Experiment = single_pulse_dec
Sample_id = S#1515
Solvent = METHANOL-D3
Creation_time = 20-DEC-2013 08:36:06
Revision_time = 24-DEC-2013 18:40:08
Current_time = 24-DEC-2013 18:40:27
Comment = AHX ribotide CNMR
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 10719
Total_scans = 10719
X_90_width = 15.6[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 5.2[us]
Irr_atn_dec = 25.434[dB]
Irr_atn_noe = 25.434[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 56
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 460.0[dC]
6


4.86
3.67
2.41
2.33
1.98
1.65
1.03
1.02
1.01
1.00
1
abundance
X : parts per Million : 1H
Filename = KI-131101-1-7.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#346262
Solvent = ACETONE-D6
Creation_time = 1-NOV-2013 09:26:55
Revision_time = 24-DEC-2013 17:42:32
Current_time = 24-DEC-2013 17:42:37
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.566819[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 7.36[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 3.68[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 24.7[dC]
HO OH
23


155.0138
146.0421
142.5376
128.2996
96.1660
90.5168
90.1488
85.0591
76.2501
71.1628
68.3099
67.7379
51.9201
29.9934
29.2753
24.1526
abundance
X : parts per Million : 13C
Filename = KI-131101-2-7.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#350498
Solvent = ACETONE-D6
Creation_time = 1-NOV-2013 10:33:58
Revision_time = 24-DEC-2013 17:47:04
Current_time = 24-DEC-2013 17:47:32
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 1300
Total_scans = 1300
X_90_width = 15.6[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 5.2[us]
Irr_atn_dec = 25.434[dB]
Irr_atn_noe = 25.434[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 25.3[dC]
HO OH
23


24
abundance
X : parts per Million : 1H
Filename = RF-131002-6-8.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#831693
Solvent = METHANOL-D3
Creation_time = 2-OCT-2013 23:04:53
Revision_time = 24-DEC-2013 18:12:34
Current_time = 24-DEC-2013 18:12:36
Comment = ANKR Huisgen HNMR
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 10.8[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.5[dB]
X_pulse = 5.4[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 23.8[dC]


24
(Thousands)
X : parts per Million : 13C
Filename = RF-131002-7-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#833904
Solvent = METHANOL-D3
Creation_time = 3-OCT-2013 07:00:17
Revision_time = 24-DEC-2013 18:22:48
Current_time = 24-DEC-2013 18:23:17
Comment = ANKR Huisgen CNMR
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Mod_return = 1
Scans = 10000
Total_scans = 10000
X_90_width = 10.2[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 6.3[dB]
X_pulse = 3.4[us]
Irr_atn_dec = 22.1[dB]
Irr_atn_noe = 22.1[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 24[dC]