Supporting Information

Total Synthesis of 3',5'-C Branched Nucleosides

Eriks Rozners* and Qun Xu

**Department of Chemistry and Chemical Biology, Northeastern University, Boston,
Massachusetts 02115**

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Experimental Procedures

Methylene chloride, pyridine, acetonitrile, and toluene were dried by refluxing with CaH₂ followed by distillation. Tetrahydrofuran was distilled from sodium/ benzophenone ketyl. Reactions were carried out in oven (150 °C) dried glassware under an atmosphere of dry nitrogen. Infrared spectra were recorded on Mattson Satellite FT-IR spectrometer potassium bromide. NMR spectra were recorded on a Varian Mercury 300 MHz spectrometer at ambient temperature. Thin layer chromatography (TLC) was performed on Scientific Adsorbents Inc. 0.25 mm silica gel 60-F₂₅₄ plates. Column chromatography was done on 230-400 mesh silica gel (60 Å, Scientific Adsorbents).

1-(*tert*-Butyldiphenylsilanyloxy)-5-trityloxy-3-pentyn-2-ol (**8**)

n-Butyllithium (6.74 mL, 1.6 M solution in hexanes, 10.78 mmol) was added dropwise over 10 min to a solution of 3-trityloxypropyne¹ **5** (2.93 g, 9.80 mmol) in anhydrous THF (80 mL) at -78 °C and the resulting solution was stirred for additional 2h at -78 °C. A solution of (*tert*-butyldiphenylsilanyloxy)-acetaldehyde² **6** (2.92 g, 9.80 mmol) in dry THF (20 mL) was added dropwise via cannula and the mixture was stirred at -78 °C for additional 4 h. Saturated aqueous solution of NH₄Cl (70 mL) was added, the mixture was warmed up to room temperature and extracted with ethyl acetate (3 × 100mL). The organic phase was washed with aqueous NaHCO₃ solution (3 × 50mL), brine (50mL), and dried (Na₂SO₄). The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (hexanes/ethyl acetate, 9:1) to afford **8** as a colorless oil. Yield: 4.72 g, 81%. TLC R_f = 0.25 (hexanes/ethyl acetate, 9:1). MS (FAB-) *m/z*: 595 (M-1, 77%). ¹H NMR (CDCl₃, 300 MHz) δ 7.68 (m, 4H), 7.45-7.22 (m, 21H), 4.46 (m, 1H), 3.78 (s, 2H), 3.78-3.65 (m, 2H), 2.56 (d, 1H), 1.07 (s, 9H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 143.80, 136.00, 135.93, 133.38, 133.23, 130.31, 130.29, 129.00, 128.31, 128.26, 127.55, 87.79, 83.76, 82.61, 67.87, 63.63, 53.42, 27.25, 19.69.

Benzoyloxyacetic acid 1-(*tert*-butyldiphenylsilanyloxymethyl)-4-trityloxy-2-butynyl ester (**4**)

1-(*tert*-Butyldiphenylsilanyloxy)-5-trityloxy-3-pentyn-2-ol **8** (2.71 g, 4.55 mmol) was dissolved in methanol (65 mL) and Lindlar catalyst (5% palladium on calcium carbonate, poisoned with lead, Fluka, 407 mg) was added. The suspension was stirred under an atmosphere of hydrogen (rubber balloon) at room temperature for 2h and 15min. The catalyst was filtered off and the filtrate was concentrated to give the *Z*-allylic alcohol as a colorless oil, which was used in the next step without further purification. Benzoyloxyacetic acid (0.67 mL, 4.55 mmol) in dry CH₂Cl₂ (10 mL) was slowly added to a solution of dicyclohexylcarbodiimide (937 mg, 4.55 mmol) and dimethylaminopyridine (56 mg, 0.455 mmol) in dry CH₂Cl₂ (15mL) at 0 °C. The resulting cloudy solution was stirred for 5 min and a solution of the crude alcohol (after Lindlar) in dry CH₂Cl₂ (25 mL) was added dropwise via cannula. Stirring was continued for 5 min at 0 °C, the mixture was allowed to warm up to room temperature, and the stirring was continued for 3 h. The solid dicyclohexylurea was filtered off and the solution was washed with water (30 mL), aqueous NH₄Cl solution (30 mL), and brine (30 mL). The organic layers were dried (Na₂SO₄), concentrated, and the residue was purified by silica gel column chromatography (hexanes/ethyl acetate, 9:1) to afford **4** as a colorless oil. Yield: 3.2 g, 92%. TLC R_f = 0.36

¹ Yasukouchi, T.; Kanematsu, K. *Tetrahedron Letters* **1989**, 30, 6559-6562. Spitzer, J. L.; Kurth, M. J.; Schore, N. E.; Najdi, S. D. *Tetrahedron* **1997**, 53, 6791-6808.

² Shiao, M. J.; Yang, C. Y.; Lee, S. H.; Wu, T. C. *Synth. Commun.* **1988**, 18, 359-366.

(hexanes/ethyl acetate, 9:1). The product contained minor impurity that could not be separated by silica gel column chromatography, but was efficiently removed in the next step. ^1H NMR (CDCl_3 , 300 MHz) δ 7.65-7.20 (m, 30H), 5.81 (m, 1H), 5.55 (m, 1H), 5.46 (m, 1H), 4.62 (s, 2H), 3.97 (s, 2H), 3.97-3.56 (m, 4H), 0.99 (s, 9H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 169.77, 144.83, 144.51, 137.73, 136.06, 136.00, 133.67, 133.55, 132.88, 130.31, 130.28, 129.16, 128.94, 128.48, 128.39, 128.24, 127.53, 126.42, 87.60, 73.66, 72.00, 67.59, 65.68, 61.10, 27.29, 19.69.

3-Benzyloxy-5-[2-(*tert*-butyldiphenylsilyloxy)-1-iodoethyl]-4-trityloxymethyl-2-dihydrofuranone (9)

n-Butyllithium (2.85 mL, 1.6 M in hexanes, 4.56 mmol) was added to a solution of 1,1,1,3,3,3-hexamethyldisilazane (0.97 mL, 4.56 mmol) in dry THF (10 mL) at -78°C . The mixture was stirred at -78°C for 10 min, trimethylsilyl chloride (0.58 mL, 4.56 mmol) was added followed by the ester **4** (2.61 g, 3.5 mmol) in dry THF (40 mL). The mixture was stirred at -78°C for 30 min and was allowed to warm up to room temperature and stirred for another 3h. The reaction mixture was then transferred to a rapidly stirred mixture of iodine (2.67 g, 10.5 mmol) and NaHCO_3 (8.82 g, 105 mmol) in H_2O (120 mL) at 0°C . The resulting mixture was stirred for 3h at 0°C . Ethyl ether (70 mL) was added, the aqueous phase was separated and washed with ethyl ether (3 $\times$ 30 mL). The organic layers were combined and washed with aqueous $\text{Na}_2\text{S}_2\text{O}_4$ solution (100mL), brine (100mL), and dried over Na_2SO_4 . Solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (hexanes/ethyl acetate, 7:1) to afford *trans* **9** as white crystals. Yield: 1.302 g, 42% TLC R_f = 0.23 (hexanes/ethyl acetate, 7:1) MS (FAB-) m/z : 871 (M-1, 5%), ^1H NMR (CDCl_3 , 300 MHz) δ 7.64-7.17 (m, 30H, TBDPS, Tr, Bn), 4.95 (d, J = 11.7 Hz, 1H Bn), 4.62 (m, 2H, Bn, H-5), 4.34 (d, J = 8.4 Hz, 1H, H-3), 4.10 (m, 1H, CHI), 3.85 (m, 2H, CH_2OTBDPS), 3.46 (dd, 1H, CH_2OTr), 3.27 (dd, 1H, CH_2OTr), 2.90 (m, 1H, H-4), 1.03 (s, 9H, TBDPS). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 174.08, 143.94, 137.15, 135.95, 135.88, 133.02, 132.95, 130.26, 130.23, 129.00, 128.77, 128.32, 128.15, 128.11, 127.31, 87.77, 80.30, 73.19, 73.00, 65.59, 60.33, 43.948, 36.33, 27.11, 19.59. *Cis* **9**: Yield: 1.064 g, 35%. TLC R_f = 0.24 (hexanes/ethyl acetate, 7:1). ^1H NMR (CDCl_3 , 300 MHz) δ 7.75-7.22 (m, 30H, TBDPS, Tr, Bn), 5.20 (d, J = 11.7 Hz, 1H, Bn), 5.19 (d, J = 11.7 Hz, 1H, Bn), 4.70 (dd, 1H, H-5), 4.79 (d, J = 8.1 Hz, 1H, H-3), 3.98 (dd, 1H, CHI), 3.90 (m, 3H, CH_2OTBDPS , TrOCH_2), 3.44 (dd, 1H, TrOCH_2), 2.90 (m, 1H, H-4), 1.13 (s, 9H, TBDPS).

Recycling of *cis* 9: Saturated aqueous solution of NH_4Cl (24 mL) and Zn dust (397 mg, 6.10 mmol) were added to a solution of *cis* **9** (1.064 g, 1.22 mmol) in ethyl acetate (6 mL). The mixture was stirred

for 2.5 h at room temperature, then the solid was filtered off. The organic layer was separated and the aqueous layer was extracted with ethyl ether (3×30 mL). The organic layers were combined and the solvent was evaporated. The residue was dissolved in THF (20 mL) and the solution was cooled to 0 °C. Iodine and saturated NaHCO₃ aqueous solution (30 mL) were added. The mixture was stirred for 3 h at 0 °C. Ethyl ether (30 mL) was added, the aqueous phase was separated and washed with ethyl ether (3×20 mL). The organic layers were combined and washed by aqueous Na₂S₂O₄ solution (30 mL), brine (30 mL), and dried over Na₂SO₄. Solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (hexanes/ethyl acetate, 7:1) to afford *trans* **9** 519 mg and *cis* **9** 427 mg. *Cis* **9** was recycled again according to the procedure described above to afford additional 186 mg of *trans* **9**. After recycling, total yield of *trans* **9**: 2.007 g, 66% (two steps from compound **4**).

3-Benzyloxy-5-[2-(*tert*-butyldiphenylsilyloxy)ethyl]-4-trityloxymethyl-2-dihydrofuranone (2**)**

Iodofuranone **9** (1.034 g, 1.19 mmol), azo-bis-isobutyronitrile (19.7 mg, 0.12 mmol), tributyltin hydride (0.96 mL, 3.56 mmol), and 15 mL toluene were mixed at room temperature in a Schlenk flask under an atmosphere of nitrogen. The mixture was heated, under nitrogen, at 95 °C for 1 h and 25 min. Toluene was evaporated under reduced pressure and the residue was purified by silica gel chromatography (hexanes/ethyl acetate, 4:1) to afford **2** as a colorless oil. Yield: 0.85 g, 96%. TLC R_f = 0.27 (hexanes/ethyl acetate, 4:1). ¹H NMR (CDCl₃, 300 MHz) δ 7.63-7.20 (m, 30H), 4.90 (d, *J* = 12 Hz, 1H), 4.64 (m, 2H), 4.18 (d, *J* = 7.2 Hz, 1H), 3.76 (m, 2H), 3.37 (m, 2H), 2.38 (m, 1H), 1.91 (m, 1H), 1.73 (m, 1H), 1.01 (s, 9H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 174.35, 144.19, 137.44, 135.90, 135.86, 133.89, 133.72, 130.13, 129.07, 128.81, 128.26, 128.15, 127.47, 87.75, 78.95, 73.94, 72.70, 60.44, 60.28, 45.66, 38.19, 27.29, 19.56.

3-Benzyloxy-5-[2-(*tert*-butyldiphenylsilyloxy)ethyl]-4-hydroxymethyl-2-dihydrofuranone (10**)**

A solution of furanone **2** (820 mg, 1.1 mmol) in dry CH₂Cl₂ (15 mL) was cooled to 0 °C, triethylsilane (0.7 mL, 4.4 mmol) was added followed by the addition of trifluoroacetic acid (0.34 mL, 4.4 mmol). The mixture was stirred for 30 min at 0 °C. Aqueous NaHCO₃ solution (15 mL) was added to the mixture, and the aqueous phase was extracted with CH₂Cl₂ (3×5 mL). The organic layers were combined and dried over Na₂SO₄. Solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (hexanes/ethyl acetate, 4:1) to afford **10** as a colorless oil. Yield: 450 mg, 81%. TLC R_f = 0.21 (hexanes/ethyl acetate, 3:1). ¹H NMR (CDCl₃, 300 MHz) δ 7.66-7.36 (m, 15H), 5.05 (d, *J* = 12 Hz, 1H), 4.76 (m, 2H), 4.26 (d, *J* = 7.5 Hz, 1H), 3.83 (m, 4H), 2.47 (m, 1H), 2.22

(m, 1H), 1.88 (m, 2H), 1.04 (s, 9H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 174.04, 136.83, 135.75, 135.74, 133.64, 133.51, 130.02, 128.93, 128.59, 128.38, 127.99, 78.33, 74.69, 72.85, 60.21, 60.13, 46.34, 37.65, 27.07, 19.37.

Methanesulfonic acid 4-benzyloxy-2-[2-(*tert*-butyldiphenylsilanyloxy)ethyl]-5-oxo-3-tetrahydrofuranymethyl ester (11)

Methanesulfonyl chloride (0.10 mL, 2.0 mmol) was added to a solution of furanone **10** (328 mg, 0.65 mmol) and triethylamine (0.17 mL, 1.2 mmol) in dry CH_2Cl_2 (5 mL) at 0°C . After 5 min the mixture was allowed to warm up to room temperature and stirred overnight. Aqueous NH_4Cl solution (2 mL) was added to the reaction mixture and the organic layer was washed by brine (2 mL) and dried over Na_2SO_4 . Solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (hexanes/ethyl acetate, 4:1) to afford **11** as a colorless oil. Yield: 352 mg, 93%. TLC R_f = 0.25 (hexanes/ethyl acetate, 3:1). IR 1738 cm^{-1} . ^1H NMR (CDCl_3 , 300 MHz) δ 7.66-7.33 (m, 15H), 4.99 (d, J = 11.4 Hz, 1H), 4.72 (m, 2H), 4.28 (dd, 1H), 4.26 (dd, 1H), 4.22 (d, J = 7.2 Hz, 1H), 3.83 (m, 2H), 2.91 (s, 3H), 2.75 (m, 1H), 1.92 (m, 1H), 1.83 (m, 1H), 1.05 (s, 9H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 137.12, 136.70, 135.76, 135.74, 133.55, 133.41, 130.13, 128.89, 128.63, 128.56, 128.09, 77.84, 72.82, 65.63, 59.95, 44.44, 37.60, 27.12, 19.42.

4-Azidomethyl-3-benzyloxy-5-[2-(*tert*-butyldiphenylsilanyloxy)ethyl]-2-dihydrofuranone (12)

A solution of **11** (1.30 g, 2.23 mmol) and sodium azide (435 mg, 6.69 mmol) in dry DMF (20 mL) was heated at 60°C for 4 h. The reaction solution was allowed to cool down to room temperature and CH_2Cl_2 (10 mL) was added. The organic layer was separated and was washed with brine (3 $\times$ 20 mL) and dried over Na_2SO_4 . Solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (hexanes/ethyl acetate, 7:1) to afford **12** as a colorless oil. Yield: 1.108 g, 94%. TLC R_f = 0.69 (hexanes/ethyl acetate, 3:1). IR $2100, 1778\text{ cm}^{-1}$. ^1H NMR (CDCl_3 , 300 MHz) δ 7.66-7.33 (m, 15H), 4.97 (d, J = 12, 1H), 4.69 (d, J = 12, 1H), 4.60 (m, 1H), 4.14 (d, J = 7.2, 1H), 3.79 (m, 2H), 3.68 (m, 1H), 2.44 (m, 1H), 1.89 (m, 1H), 1.79 (m, 1H), 1.05 (s, 9H). ^{13}C NMR (CDCl_3 , 75.4 MHz) δ 173.40, 137.00, 135.84, 135.83, 133.67, 133.55, 130.17, 128.87, 128.57, 128.13, 78.74, 73.57, 72.66, 60.08, 48.04, 45.23, 37.51, 27.18, 19.48.

4-Azidomethyl-5-[2-(*tert*-butyldiphenylsilyloxy)ethyl]-3-hydroxy-2-dihydrofuranone (13**)**

Boron trichloride (3.19 mL, 3.19 mmol, 1 M in hexanes) was added dropwise to a solution of **12** (765 mg, 1.45 mmol) in dry CH₂Cl₂ (30 mL) at -78 °C. The reaction solution was allowed to warm up slowly to -20 °C and stirred for 3 h. The solution was cooled to -78 °C and saturated aqueous NaHCO₃ (50 mL) was added. The mixture was warmed up to room temperature, the organic layer was separated and aqueous layer was extracted with CH₂Cl₂ (3 × 30 mL). The organic layers were combined and dried (Na₂SO₄). Solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (hexanes/ethyl acetate, 3:1) to afford **13** as a colorless oil. Yield: 581 mg, 91%. TLC R_F = 0.36 (hexanes/ethyl acetate, 3:1). IR 2105, 1772 cm⁻¹. MS (FAB+) *m/z*: 439 (M, 5%), 461 (M+Na, 11%). ¹H NMR (CDCl₃, 300 MHz) δ 7.67-7.40 (m, 10H), 4.70 (m, 1H), 4.57 (dd, 1H), 3.80 (m, 2H), 3.66 (dd, 1H), 3.47 (dd, 1H), 3.44 (s, 1H), 2.60 (m, 1H), 1.88 (m, 2H), 1.06 (s, 9H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 176.89, 135.76, 135.75, 133.53, 133.41, 130.10, 128.05, 79.15, 67.74, 59.95, 48.95, 43.80, 37.63, 27.07, 19.40.

Racemic 1,2-di-*O*-acetyl-3-azidomethyl-5-[(*tert*-butyldiphenylsiloxy)methyl]-3, 5-dideoxyribofuranose (15rac**)**

Diisobutylaluminum hydride (0.72 mL, 1.08 mmol, 1.5 M solution in toluene) was added over 10 min. to a solution of **13** (237 mg, 0.54 mmol) in dry THF (54 mL) at -85°C. The solution was stirred at -85°C for 15h and saturated aqueous NH₄Cl solution was added. The mixture was allowed to warm up to room temperature. The organic layer was separated and the aqueous layer was extracted with ethyl acetate (3×30 mL). Organic layers were combined and dried (Na₂SO₄). Solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (hexanes/ethyl acetate, 2:1) to afford **14** (64 mg) and unreacted **13** (141mg). The reduction of recovered **13** was repeated twice as described above to give a total of 155 mg of **13** as a colorless oil. Yield: 65%. The diol **13** was dissolved in acetic anhydride/pyridine (1:1, 2 mL). The mixture was stirred for 24 h and concentrated under reduced pressure. The residue was purified by silica gel chromatography (hexanes/ethyl acetate, 3:1) to afford **15rac** as a colorless oil. Yield: 164 mg, 94% (61% for two steps). IR 2100, 1745 cm⁻¹. MS (FAB+) *m/z*: 548 (M+Na, 30%). ¹H NMR showed the expected mixture of two diastereomers that was used in the next step without separation. ¹H NMR (CDCl₃, 300 MHz) δ (major diastereomer) ¹H NMR (CDCl₃, 300 MHz) δ 7.67-7.37 (m, 10H), 6.07 (s, 1H), 5.27 (d, 1H), 4.21 (m, 1H), 3.79 (m, 2H), 3.83-3.37 (m, 2H), 2.55 (m, 1H), 2.12 (s, 3H), 1.97 (s, 3H), 1.88-1.73 (m, 2H), 1.06 (s, 9H).

Racemic 2'-O-acetyl-3'-azidomethyl-5'-[(*tert*-butyldiphenylsiloxy)methyl]-3',5'-dideoxyuridine (16rac)

A solution of **15rac** (110 mg, 0.21 mmol), 2,4-*O,O'*-bis(trimethylsilyl)uracil (161 mg, 0.63 mmol) and trimethylsilyl trifluoromethanesulfone (93 mg, 0.42 mmol) in dry CH₂Cl₂ (4 mL) was stirred for 2 h. The reaction solution was cooled to 0 °C. CH₂Cl₂ (3 mL) and saturated aqueous NaHCO₃ (4 mL) were added. The organic layer was separated and the aqueous layer was extracted with ethyl acetate (3 × 4 mL). The organic layers were combined and dried (Na₂SO₄). Solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (hexanes/ethyl acetate, 1:1) to afford **16rac** as a colorless oil. Yield: 103 mg, 86%. TLC R_f = 0.25 (hexanes/ethyl acetate, 1:1). IR 2101, 1692 cm⁻¹. MS (FAB+) *m/z*: 578 (M+1, 12%), 600 (M+Na, 12%). ¹H NMR (CDCl₃, 300 MHz) δ 8.69 (s, 1H), 7.66-7.36 (m, 10H), 7.12 (d, *J* = 8.1 Hz, 1H), 5.69 (dd, 1H), 5.55 (d, *J* = 2.4 Hz, 1H), 5.37 (dd, 1H), 4.11 (m, 1H), 3.84 (m, 2H), 3.52 (dd, 1H), 3.34 (dd, 1H), 2.51 (m, 1H), 2.16 (s, 3H), 1.99 (m, 1H), 1.81 (m, 1H), 1.03 (s, 9H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 170.24, 163.77, 150.32, 140.78, 135.80, 133.77, 133.74, 130.04, 130.00, 127.99, 127.97, 103.04, 91.78, 79.23, 77.33, 60.52, 48.05, 45.47, 37.00, 27.13, 21.01, 19.42.

6-O-*tert*-Butyldiphenyl-3,5-dideoxy-3-hydroxymethyl-1,2-O-isopropylidene-α-D-allofuranose (19)

3,5-Dideoxy-3-[(benzoyloxy)methyl]-1,2-O-isopropylidene-α-D-allofuranose **17**³ (12.72 g, 39.5 mmol) and imidazole (6.72 g, 98.75 mmol) were dried by evaporation with dry toluene (60 mL) and dissolved in dry DMF (350 mL). DMF was evaporated to leave approximately 80% of volume and *tert*-butylchlorodiphenylsilane (11.8 mL, 45.4 mmol) was added. The mixture was stirred at room temperature for 24 hours, diluted with toluene (700 mL), and extracted with saturated aqueous NaHCO₃ (700 mL) and saturated aqueous NaCl (3 × 500 mL). The organic layer was dried (Na₂SO₄), evaporated, and dissolved in isopropyl alcohol (800 mL). A solution of NaOH (2.37 g, 59.25 mmol) in water (250 mL) was added and the mixture was heated at 70 °C for 30 minutes. The mixture was cooled to room temperature and neutralized with acetic acid (11 mL). The mixture was concentrated to almost dry. The residue was dissolved in ethyl acetate (700 mL) and extracted with saturated aqueous NaHCO₃ (700 mL) and saturated aqueous NaCl (700 mL). The organic layer was dried (Na₂SO₄), evaporated, and the residue was purified by silica gel chromatography using stepwise gradient of ethyl acetate in hexanes (10 to 50%, 10% steps) to afford **19** as clear oil. Yield 13.87 g 77%. IR 2102, 1681 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz) δ 7.69-7.66 (m, 4H), 7.42-7.26 (m, 6H), 5.79 (d, *J* = 3.5 Hz, 1H),

4.75 (t, 1H), 4.27 (m, 1H), 3.87-3.80 (m, 4H), 2.01-1.90 (m, 2H), 1.79-1.70 (m, 1H), 1.51 (s, 3H), 1.33 (s, 3H), 1.04 (s, 9H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 135.87, 135.82, 134.09, 133.89, 129.89, 129.88, 127.95, 111.89, 105.09, 82.12, 75.60, 60.96, 59.17, 51.12, 36.36, 27.13, 26.78, 26.61, 19.44.

6-O-*tert*-Butyldiphenyl-3,5-dideoxy-3-azidomethyl-1,2-O-isopropylidene-α-D-allofuranose (21)

Methanesulfonyl chloride (2.64 mL, 34.39 mmol) was added to a solution of **19** (7.84 g, 17.19 mmol) and triethylamine (4.54 mL, 32.66 mmol) in dry CH₂Cl₂ (120 mL) at 0 °C. After 10 min. the mixture was allowed to warm up to room temperature and stirred overnight. Aqueous NH₄Cl solution (50 mL) was added to the reaction mixture and the organic layer was washed with brine (100 mL) and dried over Na₂SO₄. Solvent was removed under reduced pressure and the residue was coevaporated with dry toluene (3×10 mL). The crude product was treated with sodium azide (3.35 g, 51.57 mmol) in dry DMF (110 mL) at 60 °C for 3 h. The reaction solution was allowed to cool to room temperature. CH₂Cl₂ (100 mL) and water (60 mL) were added. The organic layer was separated, washed with brine (100 mL), and dried over Na₂SO₄. Solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (hexanes/ethyl acetate, 3:1) to give **21** as a colorless oil. Yield: 6.26 g, 76% for two steps. TLC R_f = 0.48 (hexanes/ethyl acetate, 5:1). IR 2100, 1778 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz) δ 7.75-7.67 (m, 4H), 7.48-7.37 (m, 6H), 5.81 (d, *J* = 3.9 Hz, 1H), 4.70 (t, 1H), 4.07 (m, 1H), 3.83 (m, 2H), 3.62 (m, 1H), 3.31 (m, 1H), 2.03-1.87 (m, 2H), 1.75-1.64 (m, 1H), 1.51 (s, 3H), 1.35 (s, 3H), 1.07 (s, 9H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 135.86, 135.80, 135.06, 134.00, 133.80, 129.91, 129.88, 127.94, 111.99, 104.95, 80.81, 76.18, 60.66, 49.26, 47.75, 36.20, 27.08, 26.81, 26.72, 26.63, 19.41.

1,2-Di-O-acetyl-3-azidomethyl-5-[(*tert*-butyldiphenylsiloxy)methyl]-3, 5-dideoxyribofuranose (15)

Boron trichloride (15.91 mL, 15.91 mmol, 1 M in hexanes) was added dropwise to a solution of **21** (7.65 g, 15.91 mmol) in dry CH₂Cl₂ (95 mL) at -78 °C. The mixture was stirred for 20 min -78 °C and saturated aqueous NaHCO₃ (90 mL) solution was added at -78 °C. The mixture was slowly warmed to room temperature, the organic layer was separated and the aqueous layer was extracted with CH₂Cl₂ (3×60 mL). The organic layers were combined and dried over Na₂SO₄. Solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (hexanes/ethyl acetate, 3:1) to give diol **14**. Yield: 2.82 g, 70% based on recovered starting material **21** (3.26 g). The diol was dissolved in acetic anhydride/ pyridine (1:1, 12 mL). The mixture was stirred for 24 h and concentrated under reduced pressure. The residue was purified by silica gel chromatography (hexanes/ethyl acetate,

³ Huang, Z.; Schneider, K. C.; Benner, S. A. *J.Org.Chem.* **1991**, *56*, 3869-3882.

3:1) to afford **15** as a colorless oil. Yield: 2.69 g, 87% (61% for two steps). TLC R_f = 0.36 (hexanes/ethyl acetate, 3:1).

2'-O-Acetyl-3'-azidomethyl-5'-[(*tert*-butyldiphenylsiloxy)methyl]-3',5'-dideoxyuridine (16**)**

A solution of **15** (1.063 g, 2.02 mmol), 2,4-*O,O'*-bis(trimethylsilyl)uracil (1.52 mg, 6.07 mmol) and trimethylsilyl trifluoromethanesulfone (897 mg, 4.04 mmol) in dry CH_2Cl_2 (30 mL) was stirred for 1.5 h at room temperature. The reaction solution was cooled to 0 °C. CH_2Cl_2 (30 mL) and saturated aqueous NaHCO_3 (40 mL) were added. The organic layer was separated and the aqueous layer was extracted with ethyl acetate (3×4 mL). Organic layers were combined and dried (Na_2SO_4). Solvent was removed under reduced pressure and the residue was purified by silica gel chromatography (hexanes/ethyl acetate, 1:1) to afford **16** as a white solid. Yield: 925 mg, 86%. TLC R_f = 0.25 (hexanes/ethyl acetate, 1:1). The ^1H and ^{13}C NMR spectra matched those of **16** prepared via total synthesis route.

3'-Azidomethyl-3',5'-dideoxy-5'-hydroxymethyluridine (22**)**

A solution of **16** (120 mg, 0.21 mmol) in 5 mL methanol containing 0.15 mL of concentrated HCl was stirred at room temperature for 5 days. The solution was cooled on ice, diluted with 3 mL of water, and neutralized with aqueous NaHCO_3 solution. Solvent was evaporated and the residue was purified by silica gel chromatography (methylene chloride/methanol, 10:1) to afford **22** as a white solid. Yield: 54 mg, 91% based on recovered starting material **16** (5 mg). TLC R_f = 0.37 (methylene chloride/methanol, 10:1). IR 2102, 1681 cm^{-1} . ^1H NMR (D_2O , 300 MHz) δ 7.51 (d, J = 8.1 Hz, 1H), 5.67 (d, J = 8.1 Hz, 1H), 5.59 (s, 1H), 4.33 (m, 1H), 4.04 (m, 1H), 3.62 (m, 2H), 3.43 (m, 1H), 3.33 (m, 1H), 2.10 (m, 1H), 1.90 (m, 2H). ^{13}C NMR ($(\text{CD}_3)_2\text{CO}$, 75.4 MHz) δ 164.07, 151.01, 140.64, 101.73, 93.13, 80.06, 76.01, 58.92, 47.63, 46.80, 37.06.

3'-Azidomethyl-5'-carboxy-3',5'-dideoxy-2'-O-triethylsilyluridine (1a**)**

2,2,6,6-tetramethylpiperidin-*N*-oxyl (TEMPO) (5 mg, 0.03 mmol) and diacetoxyiodobenzene (120 mg, 0.37 mmol) were added to a solution of **22** (50 mg, 0.17 mmol) in $\text{CH}_3\text{CN}/\text{H}_2\text{O}$ (1:1, 2 mL). The mixture was stirred for 27 h at room temperature. Solvent was evaporated and the residue was loaded onto reverse phase silica gel cartridge (1 g Discovery DSC –18, Supelco). The product was eluted with water (6 mL) and freeze-dried to afford a light yellow powder that was dissolved in dry pyridine (1.5 mL). Triethylsilyl chloride (77 mg, 0.51 mmol) was added. After stirring for 24 h, the reaction was

quenched by adding 0.1 ml of water. The organic layer was separated and aqueous layer was extracted with ethyl acetate (3×4 mL). The organic layers were combined and dried (Na₂SO₄). Solvent was evaporated and the residue was purified by silica gel chromatography (methylene chloride/methanol, 10:1) to afford **22** as a white solid. Yield: 47 mg, 65% for two steps. Analytical sample was obtained by further crystallization from toluene. TLC R_f = 0.42 (methylene chloride/methanol, 10:1), elementary analysis: calcd C, 47.99; H, 6.40; N, 16.46, found C, 47.85; H, 6.39; N, 16.28. IR 2101, 1693 cm⁻¹. MS (ESI+) *m/z*: 448 (M+Na); (ESI-) *m/z*: 424 (M-H). ¹H NMR (CDCl₃, 300 MHz) δ 10.06 (s, 1H), 7.58 (d, *J* = 8.1 Hz, 1H), 5.83 (d, *J* = 8.1 Hz, 1H), 5.78 (s, 1H), 4.47 (m, 2H), 3.63 (m, 1H), 3.42 (m, 1H), 2.94 (dd, 1H), 2.70 (dd, 1H), 2.19 (m, 1H), 0.96 (t, 9H), 0.67 (q, 6H). ¹³C NMR (CDCl₃, 75.4 MHz) δ 175.06, 164.84, 150.41, 140.77, 102.16, 93.61, 79.38, 77.11, 48.10, 45.83, 38.30, 6.97, 4.80.

0X-1-214-1, 11/27/02

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: 0X-1-214-1

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec

File: QX-10214-1
Mercury-300 "NMR300"

PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
16 repetitions
OBSERVE H1. 299.9504945 MHz
DATA PROCESSING
FT size 16384
Total time 0 min, 49 sec

8
OH

4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 ppm

1.00

4.43

0.91

OT

QX-1-214-1, 11/27/02
Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
File: QX-1-214-1-G
Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 1867.9 Hz

208 Repetitions

OBSERVE C13, 75.4225338 MHz

DECOUPLE H1, 299.9519364 MHz

Power 39 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 84 hr, 3 min, 29 sec

0X-1-219-1, 12/07/02

Pulse Sequence: szpu1

Solvent: CDCl3

Ambient temperature

File: 0X-1-219-1

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1 299.9504345 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec

TBDPSO

1

 $\sim$ $\text{C}=\text{C}$
$$=$$

40

Fl size 1310/2
Total time 8 hr, 25 min, 10 sec

0X-1-223-2, 12/21/02

TBDPSO

Pulse Sequence: szpul
Solvent: CDCl3
Ambient temperature
File: 0X-1-223-2-CTY
Mercury-300 "NMR300"

PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
64 repetitions
OBSERVE H1, 299.9504945 MHz
DATA PROCESSING
Ft size 16384
Total time 3 min, 18 sec

0X-1-223-2, 12/21/02

TBDPSO

Pulse Sequence: szput
Solvent: CDCl3
Ambient temperature
File: 0X-1-223-2-CTY
Mercury-300 "NMR300"

PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec

Width 3898.6 Hz
64 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384
Total time 3 min, 18 sec

0X-1-223-2, 12/21/02

TBDPSO

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: 0X-1-223-C
Mercury-300 "NMR300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 64.3 degrees
Acq. time 1.738 sec
Width 18667.9 Hz
256 repetitions
OBSERVE C13, 75.4225938 MHz
DECOUPLE H1, 299.9519364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 81 hr, 3 min, 29 sec

0X-1-078-2, 05/10/02

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: 0X-1-078-2pure

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 Repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec

0X-1-078-2, 05/10/02

Pulse Sequence: szpu1

Solvent: CDCl3

Ambient temperature

File: 0X-1-078-2pure

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec

cis-9

0X-1-226-1, 12/24/02

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

64 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 3 min, 18 sec

TBDPSO

0X-1-226-1, 12/24/02

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

64 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 3 min, 18 sec

TBDPSO

0X-1-226-1, 12/24/02

Pulse Sequence: szpul

Solvent: CDCl₃

Ambient temperature

File: 0X-1-226-1-C

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 18867.9 Hz

160 repetitions

OBSERVE C13, 75.4225938 MHz

DECOUPLE H1, 299.9519364 MHz

Power 39 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 84 hr, 3 min, 29 sec

TBDPSO

0X-1-227-1, 12/20/02

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: 0X-1-227-1

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec

TBDPSO

0X-1-227-1, 12/20/02

TBDPSO

Pulse Sequence: szpu1

Solvent: CDCl3

Ambient temperature

File: 0X-1-227-1

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FI size 16384

Total time 0 min, 49 sec

QX-1-228-1, 01/03/03

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: QX-1-228-1

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec

TBDPSO

QX-1-228-1, 01/03/03

TBDPSO

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: QX-1-228-1
Mercury-300 "NMR300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.984 sec
Width 3898.6 Hz
16 repetitions
OBSERVE H1, 299.9501945 MHz
DATA PROCESSING
F1 size 16384
Total time 0 min, 49 sec

TBPSO

0X-1-228-1, 01/03/03
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 0X-1-228-1-C
Mercury-300 "NMR300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 64.3 degrees
Acq. time 1.738 sec
Width 18867.9 Hz
720 repetitions
OBSERVE C13, 75.4225938 MHz
DECOUPLE H1, 299.9519364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 84 hr, 3 min, 29 sec

0X-1-230-1, 01/16/03

Pulse Sequence: s2pul1

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.99 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec

OTBDPS

OTBDPS

Solvent: CDCl₃

O=C1C=CC(=O)C=C1

Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz

Width 3898.6 Hz

16 repetitions
ORSEFV H1 2

OBSEVNL 11, 200.0004040 MHz
DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec

0X-1-230-1, 01/17/03

OTBDPS

Pulse Sequence: s2pul1

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 18867.9 Hz

288 repetitions

OBSERVE C13, 75.4225938 MHz

DECOUPLE H1, 299.9519364 MHz

Power 39 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 84 hr, 3 min, 29 sec

0X-1-233-1, 01/21/03

OTBDPS

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.934 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec

QX-1-239-1, 01/21/03

OTBDPS

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec

OTBDPS

0X-1-239-1, 01/21/03
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300 "NMR300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 64.3 degrees
Acq. time 1.738 sec
Width 18867.9 Hz
20544 repetitions
OBSERVE C13, 75.4225938 MHz
DECUPLE H1, 299.9519364 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 131072
Total time 84 hr, 3 min, 29 sec

0x-1-247-1, 02/11/03

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec

OTBDPS

0x-1-247-1, 02/11/03

Pulse Sequence: szpul

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FI size 16384

Total time 0 min, 49 sec

OTBDPS

15rac

Qx-1-251-1, 02/18/03

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec

16rac

0x-1-251-1, 02/18/03

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

File size 16384

Total time 0 min, 49 sec

0X-1-251-1, 02/18/03

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 18867.9 Hz

736 repetitions

OBSERVE C13, 75.4225938 MHz

DECOUPLE H1, 299.9519364 MHz

Power 39 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

line broadening 1.0 Hz

FT size 131072

Total time 84 hr, 3 min, 29 sec

0X-1-139-1, 05/22/02

Pulse Sequence: s2pul1

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FI size 16384

Total time 0 min, 49 sec

ER-107-1, 03/18/03

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSEVE H1, 299.9504945 MHz

DATA PROCESSING

File size 16384

Total time 0 min, 49 sec

OTBDPS

ER-107-1, 03/18/03

OTBDPS

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient Temperature
Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3888.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 49 sec

ER-107-1, 03/18/03

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax: delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 18667.9 Hz

912 repetitions

OBSERVE C13' 75.4225938 MHz

DECOUPLE H1' 299.9519364 MHz

Power 39 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

line broadening 1.0 Hz

FT size 131072

Total time 84 hr, 3 min, 29 sec

OTBDPS

STANDARD 1H OBSERVE

OTBDPS

Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 Mercury-300 "NMR300"
 PULSE SEQUENCE
 Relax: delay 1.000 sec
 Pulse: 55.6 degrees
 Acq: time 1.394 sec
 Width 3898.6 Hz
 64 repetitions
 OBSERVE H1, 299.9504995 MHz
 DATA PROCESSING
 F1 size 16384
 Total time 3 min, 18 sec

STANDARD 1H OBSERVE

OTBDPS

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
Mercury-300 "NMR300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
64 repetitions
OBSERVE H1 299.9504995 MHz
DATA PROCESSING
FT size 16384
Total time 3 min, 18 sec

QX-1-284-1, 05/26/03

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

64 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FT size 16384

Total time 3 min, 18 sec

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
Mercury-300 "NMR300"

PULSE SEQUENCE
Relax. delay

Relax. delay 1.000 sec
Pulse 65.6 degrees

Acq. time 1.994 sec

width 3030.0 m
64 repetitions

OBSERVE H1, 299.9504945 MHZ
DATA PROCESSING

FT size 16384
Total time 3 min. 18 sec

0X-1-284-1, 05/26/03

Pulse Sequence: szpul

Solvent: CDCl3

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 18667.9 Hz

338 repetitions

OBSERVE C13, 75.4225938 MHz

DECOUPLE H1, 299.9519364 MHz

Power 39 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 84 hr, 3 min, 29 sec

0X-1-293-A, 05/22/03

Pulse Sequence: szpul

Solvent: D2O

Ambient temperature

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

64 repetitions

OBSERVE H1, 299.9512654 MHz

DATA PROCESSING

FT size 16384

Total time 3 min, 18 sec

QX-1-293-2, 05/22/03
Pulse Sequence: szpul
Solvent: D2O
Ambient temperature
Mercury-300 "NMR300"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 65.6 degrees
Acq. time 1.994 sec
Width 3898.6 Hz
64 repetitions
OBSERVE H1, 299.9512654 MHz
DATA PROCESSING
F1 size 16384
Total time 3 min, 18 sec

0X-1-293-2, 05/19/03

Pulse Sequence: szpul

Solvent: Acetone
Ambient temperature
Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 18867.9 Hz

528 repetitions

OBSERVE C13, 75.4229853 MHz

DECOUPLE H1, 299.9334932 MHz

Power 39 dB

continuously ON

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FI size 131072

Total time 84 hr, 3 min, 29 sec

0X-1-293-1, 05/12/03

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: 0X-1-293-1

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

15 Repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FI size 16384

Total time 0 min, 49 sec

OX-1-293-1, 05/12/03

Pulse Sequence: szpul

Solvent: CDCl3

Ambient temperature

File: OX-1-293-1

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 65.6 degrees

Acq. time 1.994 sec

Width 3898.6 Hz

16 repetitions

OBSERVE H1, 299.9504945 MHz

DATA PROCESSING

FI size 16384

Total time 0 min, 49 sec

0X-1-293-1, 05/12/03

Pulse Sequence: szpu1

Solvent: CDCl3

Ambient temperature

File: 0X-1-292-1-C

Mercury-300 "NMR300"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 64.3 degrees

Acq. time 1.738 sec

Width 18667.9 Hz

18832 repetitions

OBSERVE C13, 75.4225938 MHz

DECOUPLE H1, 299.9519364 MHz

Power 39 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 84 hr, 3 min, 29 sec