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Electronic Supplementary Information

A general method for *N*-glycosylation of nucleobases promoted by (p-Tol)$_2$SO/Tf$_2$O with thioglycoside as donor

Guang-jian Liu, Xiao-tai Zhang and Guo-wen Xing*

Department of Chemistry, Beijing Normal University, Beijing 100875, China.
Email: gwxing@bnu.edu.cn

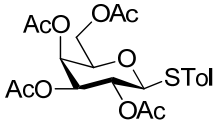
Contents

1. General information

All the starting materials were purchased as reagent grade and used without further purification. Reactions were monitored with analytical thin-layer chromatography (TLC) on silica gel F_{254} glass plates and visualized under UV light (254nm) or by staining with acidic ceric ammonium molybdate or EtOH-H_2SO_4 (3%). All the *N*-glycosylation reactions were performed in flame-dried glass vessel under an inert argon atmosphere. Purification of products was performed by flash column chromatography on silica gel (200-300 mesh). ^{1}H NMR(400MHz) and ^{13}C NMR(100MHz) spectra were recorded on a Bruker Avanced III spectrometer. Chemical shifts for ^{1}H were reported in δ-values (ppm) with tetramethylsilane as an internal standard. The δ values for ^{13}C were calibrated with deuterated solvents ($CDCl_3$ δ = 77.16 ppm). Mass spectra were recorded using electrospray ionization (ESI).

2. Characterization data for glycosyl donors

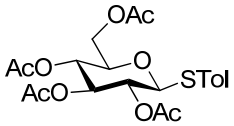
***p*-Tolyl 2,3,4,6-tetra-*O*-acetyl-1-thio-β-D-galactopyranoside (1a)**



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.1 Hz, 2H, ArH), 7.13 (d, *J* = 7.9 Hz, 2H, ArH), 5.41 (d, *J* = 2.5 Hz, 1H, H-4), 5.22 (t, *J* = 10.0 Hz, 1H, H-2), 5.04 (dd, *J* = 9.9, 3.3 Hz, 1H, H-3), 4.65 (d, *J* = 10.0 Hz, 1H, H-1), 4.19 (dd, *J* = 11.3, 6.9 Hz, 1H, H-6), 4.11 (dd, *J* = 11.3, 6.3 Hz, 1H, H-6), 3.91 (t, *J* = 6.6 Hz, 1H, H-5), 2.35 (s, 3H, STol CH_3), 2.12 (s, 3H, Ac), 2.10 (s, 3H, Ac), 2.04 (s, 3H, Ac), 1.97 (s, 3H, Ac).

The spectroscopic data coincide with the previous report[1]

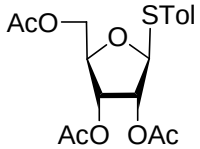
***p*-Tolyl 2,3,4,6-tetra-*O*-acetyl-1-thio-β-D-glucopyranoside (1b)**



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.1 Hz, 2H, ArH), 7.12 (d, *J* = 7.9 Hz, 2H, ArH), 5.21 (t, *J* = 9.4 Hz, 1H, H-3), 5.02 (t, *J* = 9.8 Hz, 1H, H-4), 4.93 (t, *J* = 9.7 Hz, 1H, H-2), 4.63 (d, *J* = 10.1 Hz, 1H, H-1), 4.20 (m, 2H, H-6), 3.70 (ddd, *J* = 10.1, 4.8, 2.7 Hz, 1H, H-5), 2.35 (s, 3H, STol CH_3), 2.09 (s, 3H, Ac), 2.08 (s, 3H, Ac), 2.01 (s, 3H, Ac), 1.98 (s, 3H, Ac).

The spectroscopic data coincide with the previous report[2]

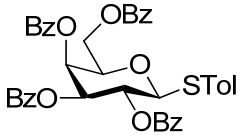
***p*-Tolyl 2,3,5-tri-*O*-acetyl-1-thio-β-D-ribofuranoside (1c)**



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.1 Hz, 2H, ArH), 7.14 (d, *J* = 7.9 Hz, 2H, ArH), 5.27 – 5.17 (m, 3H), 4.27 – 4.21 (m, 2H), 4.10 – 4.04 (m, 1H), 2.34 (s, 3H), 2.10 (s, 3H), 2.07 (s, 3H), 2.04 (s, 3H).

The spectroscopic data coincide with the previous report[3]

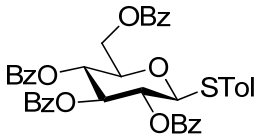
***p*-Tolyl 2,3,4,6-tetra-*O*-benzoyl-1-thio-β-D-galactopyranoside (1d)**



^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 – 8.01 (m, 2H), 8.00 – 7.97 (m, 2H), 7.93 – 7.89 (m, 2H), 7.77 – 7.73 (m, 2H), 7.64 – 7.50 (m, 3H), 7.49-7.38 (m, 9H), 7.23 (t, *J* = 7.8 Hz, 2H), 7.07 (d, *J* = 7.9 Hz, 2H), 5.99 (d, *J* = 3.2 Hz, 1H), 5.74 (t, *J* = 9.9 Hz, 1H), 5.59 (dd, *J* = 9.9, 3.3 Hz, 1H), 4.98 (d, *J* = 9.9 Hz, 1H), 4.65 (dd, *J* = 11.3, 6.8 Hz, 1H), 4.43 (dd, *J* = 11.3, 5.8 Hz, 1H), 4.37 (t, *J* = 6.4 Hz, 1H), 2.37 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 166.18, 165.65, 165.52, 165.30, 138.78, 134.54, 133.67, 133.45, 133.39, 130.16, 129.98, 129.96, 129.90, 129.75, 129.62, 129.50, 129.08, 128.89, 128.66, 128.57, 128.40, 127.54, 86.27, 75.22, 73.17, 68.51, 68.07, 62.63, 21.47.

A similar experimental procedure was applied as described in the literature. [*Carbohydr. Res.* 384 (2014) 1–8]

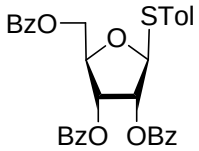
***p*-Tolyl 2,3,4,6-tetra-*O*-benzoyl-1-thio-β-D-glucopyranoside (1e)**



^{1}H NMR (500 MHz, $CDCl_3$) δ 8.04 (d, *J* = 7.3 Hz, 2H, ArH), 7.98 (d, *J* = 7.4 Hz, 2H, ArH), 7.89 (d, *J* = 7.4 Hz, 2H, ArH), 7.79 (d, *J* = 7.4 Hz, 2H, ArH), 7.60 (t, *J* = 7.4 Hz, 1H, ArH), 7.57 – 7.31 (m, 11H, ArH), 7.30 – 7.23 (m, 2H, ArH), 6.94 (d, *J* = 7.9 Hz, 2H, ArH), 5.89 (t, *J* = 9.5 Hz, 1H, H-3), 5.59 (t, *J* = 9.8 Hz, 1H, H-4), 5.45 (t, *J* = 9.7 Hz, 1H, H-2), 4.98 (d, *J* = 10.0 Hz, 1H, H-1), 4.68 (dd, *J* = 12.1, 2.5 Hz, 1H, H-6'), 4.47 (dd, *J* = 12.1, 5.8 Hz, 1H, H-6), 4.21 – 4.13 (m, 1H, H-5), 2.28 (s, 3H, $PhCH_3$).

The spectroscopic data coincide with the previous report[4]

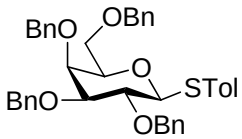
***p*-Tolyl 2, 3, 5-tri-*O*-benzoyl-1-thio-β-D-ribofuranoside (1f)**



^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.0 Hz, 2H), 7.99 (d, *J* = 8.0 Hz, 2H), 7.90 (d, *J* = 7.9 Hz, 2H), 7.59 – 7.49 (m, 3H), 7.48 – 7.30 (m, 8H), 7.05 (d, *J* = 7.8 Hz, 2H), 5.71 (t, *J* = 4.7 Hz, 1H), 5.63 (t, *J* = 5.2 Hz, 1H), 5.54 (d, *J* = 5.2 Hz, 1H), 4.67 – 4.59 (m, 2H), 4.48 (dd, *J* = 12.4, 4.5 Hz, 1H), 2.22 (s, 3H).

The spectroscopic data coincide with the previous report[3]

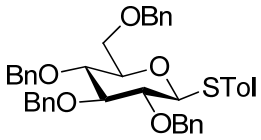
***p*-Tolyl 2,3,4,6-tetra-*O*-benzyl-1-thio-β-D-galactopyranoside (1g)**



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.1 Hz, 2H, SPh), 7.40 – 7.23 (m, 20H,Bn), 6.98 (d, *J* = 8.0

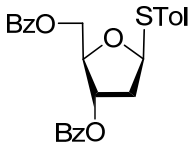
Hz, 2H, SPh), 4.95 (d, *J* = 11.5 Hz, 1H, H-1), 4.79 (d, *J* = 10.2 Hz, 1H, Bn),4.76 – 4.67 (m, 3H, Bn), 4.59 (dd, *J* = 10.6, 4.1 Hz, 2H, Bn), 4.43 (q, *J* = 11.7 Hz, 2H, Bn), 3.97 (d, *J* = 2.5 Hz, 1H, H-4), 3.89 (t, *J* = 9.4 Hz, 1H, H-2), 3.65 (d, *J* = 6.2 Hz, 2H), 3.59 (dd, *J* = 9.2, 2.7 Hz, 2H), 2.28 (s, 3H, Me).
The spectroscopic data coincide with the previous report[5]

***p*-Tolyl 2,3,4,6-tetra-*O*-benzyl-1-thio-β-D-glucopyranoside (1h)**



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.1 Hz, 2H, ArH), 7.42 – 7.37 (m, 2H, ArH), 7.36 – 7.23 (m, 16H, ArH), 7.21 – 7.17 (m, 2H, ArH), 7.02 (d, *J* = 8.0 Hz, 2H, ArH), 4.89 (dd, *J* = 10.6, 3.6 Hz, 2H, Bn), 4.83 (dd, *J* = 10.8, 8.6 Hz, 2H, Bn), 4.72 (d, *J* = 10.3 Hz, 1H, Bn), 4.63 – 4.56 (m, 3H, Bn, H-1), 4.53 (d, *J* = 11.9 Hz, 1H, Bn), 3.78 (dd, *J* = 10.9, 1.9 Hz, 1H,H-6), 3.75 – 3.66 (m, 2H, H-4, H-6), 3.63 (t, *J* = 9.3 Hz, 1H, H-3), 3.48 (ddd, *J* = 9.5, 4.4, 2.0 Hz, 2H, H-2. H-5), 2.30 (s, 3H, $PhCH_3$).
The spectroscopic data coincide with the previous report[4]

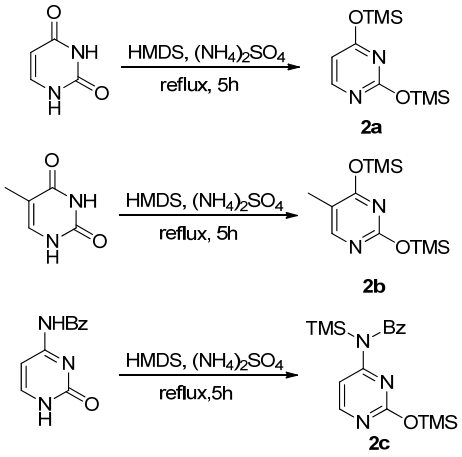
***p*-Tolyl 3,5-di-*O*-benzoyl-1-thio-2-deoxy-β-D-ribofuranoside (1i)**



^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 – 8.10 (m, 2H), 8.04 – 8.00 (m, 2H), 7.62 – 7.53 (m, 2H), 7.50 – 7.40 (m, 6H), 7.11 (d, *J* = 7.9 Hz, 2H), 5.79 (dd, *J* = 7.7, 2.5 Hz, 1H), 5.50 (ddd, *J* = 7.5, 3.8, 2.5 Hz, 1H), 4.78 (q, *J* = 4.0 Hz, 1H), 4.62 (dd, *J* = 4.0, 2.2 Hz, 2H), 2.94 (dt, *J* = 15.0, 7.6 Hz, 1H), 2.36 (dt, *J* = 14.7, 2.5 Hz, 1H), 2.32 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.35, 166.28, 137.63, 133.51, 133.28, 132.18, 131.63, 130.02, 129.92, 129.86, 129.78, 129.70, 128.63, 128.56, 88.40, 81.26, 75.00, 64.28, 39.75, 21.24. ESI-MS for $C_{26}H_{24}O_5SNa$ $[M+Na]^+$: 471.1, found: 471.3.

3. Preparation of acceptors

(1) General procedure for silylation of pyrimidines



A suspension of nucleobase (5 mmol), $(NH_4)_2SO_4$ (20 mg) in hexamethyldisilazane (HMDS) (7 mmol, 1.5 mL) was refluxed until the reaction mixture became clear. The reaction mixture was then concentrated in vacuo and the crude was kept under high vacuum overnight to remove traces of solvent.

(2) Preparation of Boc-protected purines.

The Boc-protected purines **2e** and **2f** were prepared according to the published procedures. [*J. Org. Chem.*, 65 (2000) 7697–7699]

4. General procedure for *N*-glycosylations of nucleobases

(1) General procedure for *N*-glycosylations of pyrimidines

To a mixture of thioglycoside donor (1.0 equiv, 0.069 mmol), (*p*-Tol)$_2$SO (2 – 6 equiv), and activated 3Å powdered sieves in flame-dried glass vessel was added anhydrous dichloromethane (CH_2Cl_2, 2.5 mL), which was freshly distilled over calcium hydride. The resulting mixture was stirred at preactivation temperature (-70ºC – -40ºC) for 20 min, followed by the addition of trifluoromethanesulfonic anhydride(13.6 μL, 1.2 equiv). After activation for 30 min (for donor **1e**, the activation time was 1 h), a solution of silylated nucleobase (3.0 equiv) in anhydrous CH_3CN (0.7 ml) was added, and the reaction mixture was maintained at this temperature for 2 h and then raise 20ºC higher for another 2 h. The reaction mixture was quenched with saturated $NaHCO_3$ solution (1 ml), diluted with CH_2Cl_2, filtered through Celite, washed with saturated brine, dried and concentrated to leave a residue which was purified by column chromatography on silica gel to afford the pure nucleosides **3-20** and **29-37**.

(2) General procedure for *N*-glycosylations of purines

To a mixture of thioglycoside donor (1.0 equiv, 0.069 mmol), (*p*-Tol)$_2$SO (2.0 equiv), and activated 3Å powdered sieves in flame-dried glass vessel was added anhydrous dichloromethane (CH_2Cl_2, 2.5 mL), which was freshly distilled over calcium hydride. The resulting mixture was stirred at preactivation temperature (-50ºC – -40ºC) for 20 min, followed by the addition of trifluoromethanesulfonic anhydride(13.6 μL, 1.2 equiv). After activation for 30 min (for donor **1e**, the activation time was 1 h), a solution of accepotr (3.0 equiv) in anhydrous CH_3CN (0.7 ml) was added, and the reaction mixture was allowed warming to rt gradually. After which, the reaction mixture was quenched with Et_3N (0.1 ml), diluted with CH_2Cl_2, filtered through Celite, washed with saturated brine, dried and concentrated to leave a residue which was purified by column chromatography on silica gel to afford the pure nucleosides **21** – **28**.

5. NMR data for coupling products

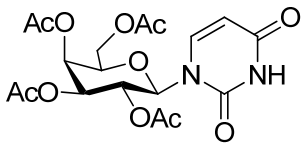
(1) Reference for known compounds

Compounds	References	^{1}H NMR	Compounds	References	^{1}H NMR
3	[6]	★	**22**	[8]	★
4	[7]	★	**23**	[8]	★
5	[8]	★	**25**	[8]	★
6	[6]	★	**26**	[8]	★
7	[9]	★	**29**	[16]	★
8	[8]	★	**30**	[16]	★
9	[10]	★	**31**	[16]	★
10	[11]	★	**32**	[13]	★
11	[12]	★	**33**	[13]	★
15	[8]	★	**34**	[16]	★
16	[13]	★	**35**	[10]	★
18	[14]	★	**36**	[17]	★
19	[14]	★	**37**	[17]	★
20	[14]	★	**S1**	[8]	★
21	[15]	★			

(2) New compounds

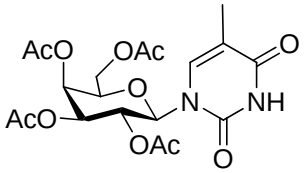
Compounds	^{1}H NMR	^{13}C NMR	HRMS
12	★	★	★
13	★	★	★
14	★	★	★
17	★	★	★
24	★	★	★
27	★	★	★
28	★	★	★
S2	★	★	★

1-(2',3',4',6'-Tetra-*O*-acetyl-β-D-galactopyranosyl)-uracil (3)



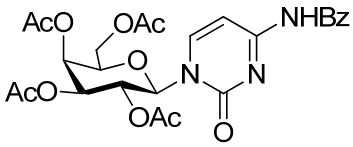
(R_f = 0.40，petroleum：ethyl acetate = 1:3) ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.16 (s, broad, 1H, NH), 7.40 (d, *J* = 8.2 Hz, 1H, H-6), 5.90 – 5.83 (m, 2H, H-1'and H-5), 5.51 (d, *J* = 2.7 Hz, 1H), 5.31 (t, *J* = 9.6 Hz, 1H), 5.24 (dd, *J* = 10.2, 3.2 Hz, 1H), 4.10-4.18 (m, 3H), 2.20 (s, 3H, -OAc), 2.06 (s, 3H, -OAc), 2.02 (s, 3H, -OAc), 2.01 (s, 3H, -OAc).

1-(2′,3′,4′,6′-Tetra-*O*-acetyl-β-D-galactopyranosyl)-thymine (4)



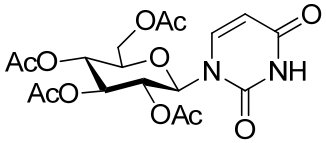
(R_f =0.21，petroleum：ethyl acetate = 2:3) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.86 (s, 1H, NH), 7.16 (d, *J* = 1.2 Hz, 1H, H-6), 5.85 (d, *J* = 9.2 Hz, 1H, H-1'), 5.51 (d, *J* = 3.2 Hz, 1H, H-4'), 5.32 (t, *J* = 9.7 Hz, 1H, H-2'), 5.22 (dd, *J* = 10.2, 3.3 Hz, 1H, H-3'), 4.22 – 4.05 (m, 3H, H-5' and H-6'), 2.22 (s, 3H, OAc), 2.06 (s, 3H, OAc), 2.01 (s, 3H, OAc), 2.00 (s, 3H, OAc), 1.98 (d, *J* = 1.0 Hz, 3H, CH_3).

***N*^{4}-benzoyl-1-(2',3',4',6'-tetra-*O*-acetyl-β-D-galactopyranosyl)-cytosine (5)**



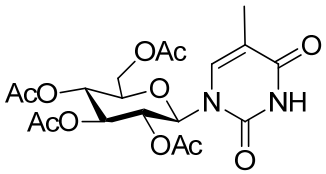
(R_f =0.40，petroleum：ethyl acetate = 1:4) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 7.5 Hz, 2H), 7.84 (d, *J* = 7.6 Hz, 1H), 7.58 (dt, *J* = 15.3, 7.4 Hz, 4H), 6.11 (d, *J* = 8.8 Hz, 1H, H-1'), 5.54 (d, *J* = 2.5 Hz, 1H, H-4'), 5.32 (dd, *J* = 12.7, 6.3 Hz, 1H, H-2'), 5.26 (dd, *J* = 10.2, 3.2 Hz, 1H, H-3'), 4.23 – 4.09 (m, 3H, H-5' and H-6'), 2.22 (s, 3H, OAc), 2.06 (s, 3H, OAc), 2.01 (s, 6H, OAc).

1-(2',3',4',6'-Tetra-*O*-acetyl-β-D-glucopyranosyl)-uracil (6)



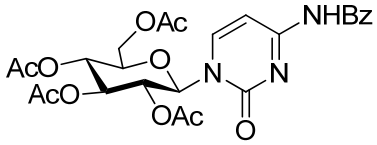
(R_f = 0.34，petroleum：ethyl acetate = 1:3) ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.35 (s, broad, 1H, NH), 7.35 (d, *J* = 8.2 Hz, 1H, H-6), 5.90 (d, *J* = 9.5 Hz, 1H, H-1'), 5.84 (dd, *J* = 8.2, 2.0 Hz, 1H, H-5), 5.41 (t, *J* = 9.5 Hz, 1H, H-2'), 5.17 (dt, *J* = 13.5, 9.6 Hz, 2H, H-3' and H-4'), 4.28 (dd, *J* = 12.6, 4.9 Hz, 1H, H-6'), 4.13 (dd, *J* = 12.6, 2.0 Hz, 1H, H-6'), 3.96 (ddd, *J* = 10.2, 4.9, 2.1 Hz, 1H, H-5'), 2.10 (s, 3H, Ac), 2.06 (s, 3H, Ac), 2.03 (s, 3H, Ac), 2.01 (s, 3H, Ac).

1-(2',3',4',6'-Tetra-*O*-acetyl-β-D-glucopyranoside)-thymine (7)



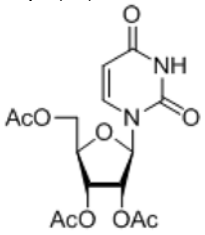
(R_f = 0.49，petroleum：ethyl acetate = 1:3)^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (s, 1H, NH), 7.14 (d, *J* = 1.2 Hz, 1H, H-6), 5.87 (d, *J* = 9.5 Hz, 1H, H-1'), 5.38 (t, *J* = 9.5 Hz, 1H, H-3'), 5.24 – 5.11 (m, 2H, H-2', H-4'), 4.28 (dd, *J* = 12.6, 5.1 Hz, 1H, H-6'), 4.12 (dd, *J* = 12.6, 2.0 Hz, 1H, H-6'), 3.93 (ddd, *J* = 10.2, 5.1, 2.1 Hz, 1H, H-5'), 2.10 (s, 3H, OAc), 2.06 (s, 3H, OAc), 2.02 (s, 3H, OAc), 2.00 (s, 3H, OAc), 1.96 (d, *J* = 1.1 Hz, 3H, CH_3).

N^4-benzoyl-1-(2',3',4',6'-tetra-*O*-acetyl-β-D-glucopyranosly)-cytosine (8)



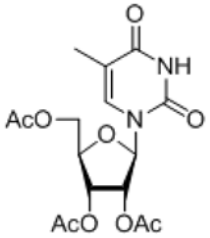
(R_f = 0.30，petroleum：ethyl acetate = 1:3) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.76 (s, 1H), 7.90 (d, *J* = 6.6 Hz, 2H), 7.81 (d, *J* = 7.0 Hz, 1H), 7.66 – 7.50 (m, 4H), 6.14 (d, *J* = 9.3 Hz, 1H), 5.44 (t, *J* = 9.4 Hz, 1H), 5.25 – 5.13 (m, 2H), 4.30 (dd, *J* = 12.6, 4.6 Hz, 1H), 4.14 (d, *J* = 12.5 Hz, 1H), 4.02-3.94 (m, 1H), 2.10 (s, 3H), 2.07 (s, 3H), 2.03 (s, 3H), 1.99 (s, 3H).

1-(2',3',5'-Tri-*O*-acetyl-β-D-ribofuranosyl)-uracil (9)



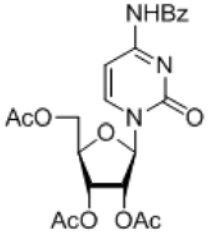
(R_f = 0.28，petroleum：ethyl acetate = 1:3) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (s, 1H, NH), 7.39 (d, *J* = 8.2 Hz, 1H, H-6), 6.04 (d, *J* = 4.9 Hz, 1H, H-1'), 5.79 (dd, *J* = 8.2, 1.8 Hz, 1H, H-5), 5.37 – 5.31 (m, 2H, H-2' and H-3'), 4.40 – 4.32 (m, 3H, H-4' and H-5'), 2.15 (s, 3H, Ac), 2.13 (s, 3H, Ac), 2.11 (s, 3H, Ac).

1-(2',3',5'-Tri-*O*-acetyl-β-D-ribofuranosyl)-thymine (10)



(R_f = 0.41，petroleum：ethyl acetate = 2:5) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.96 (br s, 1H, N-H), 7.18 (d, *J* = 1.2 Hz, 1H, H-6), 6.12 – 6.06 (m, 1H, H-1'), 5.38 – 5.31 (m, 2H, H-2' and H-3'), 4.41 – 4.31 (m, 3H, H-4' and H-5'), 2.16 (s, 3H, -OAc), 2.13 (s, 3H, -OAc), 2.10 (s, 3H, -OAc), 1.95 (d, *J* = 1.1 Hz, 3H, $-CH_3$).

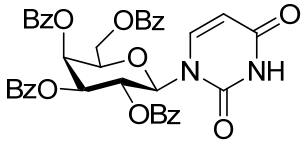
N^4-benzoyl-1-(2',3',5'-tri-*O*-acetyl-β-D-ribofuranosyl)- cytosine (11)



(R_f = 0.22，petroleum：ethyl acetate = 2:7)^{1}H NMR (400 MHz, DMSO) δ 11.35 (s, 1H, ex, NH), 8.19 (d, *J* = 7.5 Hz, 1H, H-6), 8.01 (d, *J* = 7.3 Hz, 2H, Ph), 7.63 (t, *J* = 7.4 Hz, 1H, Ph), 7.52 (t, *J* = 7.7 Hz, 2H, Ph), 7.40 (bs, 1H, H-5), 5.94 (d, *J* = 3.8 Hz, 1H, H-1'), 5.54 (dd, *J* = 6.1, 3.9 Hz, 1H, H-2'), 5.40

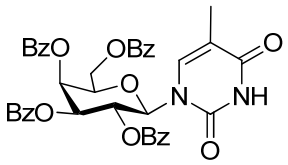
(apt, *J* = 6.3 Hz, 1H, H-3'), 4.41 – 4.30 (m, 2H), 4.25 (dd, *J* = 11.6, 5.3 Hz, 1H), 2.08 (s, 3H), 2.08 (s, 3H), 2.06 (s, 3H).

1-(2',3',4',6'-Tetra-*O*-benzoyl-β-D-galactopyranosyl)-uracil (12)



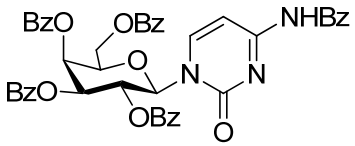
(R_f = 0.20，petroleum：ethyl acetate = 1:1) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (s, 1H, NH), 8.08 – 8.03 (m, 2H), 8.00 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.91 – 7.87 (m, 2H), 7.77 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.70-7.65(m, 1H), 7.61 – 7.40 (m, 8H), 7.36 (t, *J* = 7.8 Hz, 2H), 7.29 – 7.23 (m, 2H), 6.21 (d, *J* = 9.3 Hz, 1H), 6.10 (d, *J* = 2.7 Hz, 1H), 5.95 – 5.87 (m, 2H), 5.82 (dd, *J* = 10.1, 3.3 Hz, 1H), 4.64 (dd, *J* = 10.9, 6.7 Hz, 1H), 4.61 – 4.56 (m, 1H), 4.45 (dd, *J* = 10.9, 5.2 Hz, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 166.09, 165.62, 165.42, 165.35, 162.62, 150.39, 139.25, 133.98, 133.93, 133.58, 133.48, 130.03, 129.94, 129.90, 129.85, 129.28, 129.02, 128.97, 128.65, 128.57, 128.47, 128.14, 104.06, 81.04, 74.62, 71.79, 68.37, 68.20, 62.16; HRMS (ESI) calcd for $C_{38}H_{30}N_2O_{11}Na$ $[M+Na]^+$: 713.1742, found: 713.1741

1-(2',3',4',6'-Tetra-*O*-benzoyl-β-D-galactopyranosyl)-thymine (13)



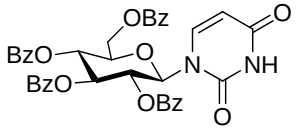
(R_f = 0.51，petroleum：ethyl acetate = 1:1) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (s, 1H), 8.10 – 8.04 (m, 2H), 8.03 – 7.98 (m, 2H), 7.91 – 7.86 (m, 2H), 7.79 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.71 – 7.65 (m, 1H), 7.55 (ddd, *J* = 7.6, 4.8, 3.3 Hz, 3H), 7.51 – 7.31 (m, 7H), 7.26 (t, *J* = 7.8 Hz, 2H), 6.25 (d, *J* = 9.3 Hz, 1H), 6.10 (d, *J* = 3.2 Hz, 1H), 5.93 (t, *J* = 9.7 Hz, 1H), 5.82 (dd, *J* = 10.1, 3.3 Hz, 1H), 4.67 – 4.57 (m, 2H), 4.51 – 4.43 (m, 1H), 2.03 (d, *J* = 0.9 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 166.12, 165.61, 165.41, 165.37, 163.18, 150.43, 134.75, 134.02, 133.94, 133.61, 133.53, 130.05, 129.95, 129.91, 129.88, 129.30, 129.10, 128.98, 128.68, 128.60, 128.49, 128.22, 112.41, 81.00, 74.53, 71.91, 68.36, 68.23, 62.24, 12.97;HRMS (ESI) calcd for $C_{39}H_{32}N_2O_{11}Na$ $[M+Na]^+$: 727.1898, found: 727.1897.

***N*^{4}-benzoyl-1-(2',3',4',5'-tetra-*O*-benzoyl-β-D-galactopyranosyl)-cytosine (14)**



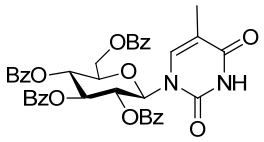
(R_f = 0.40，petroleum：ethyl acetate = 2:3) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 – 7.85 (m, 9H), 7.81 – 7.52 (m, 7H), 7.52 – 7.39 (m, 6H), 7.34 (t, *J* = 7.8 Hz, 2H), 7.29 – 7.22 (m, 2H), 6.53 (d, *J* = 9.1 Hz, 1H), 6.13 (d, *J* = 3.2 Hz, 1H), 5.96 (t, *J* = 9.7 Hz, 1H), 5.84 (dd, *J* = 10.1, 3.3 Hz, 1H), 4.68 – 4.60 (m, 2H), 4.48 (q, *J* = 9.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.06, 165.73, 165.43, 165.32, 162.62, 144.39, 133.98, 133.81, 133.53, 133.45, 133.40, 130.11, 129.93, 129.86, 129.29, 129.12, 129.03, 128.98, 128.67, 128.60, 128.56, 128.44, 127.79, 81.86, 74.86, 71.94, 69.05, 68.44, 62.19; HRMS (ESI) calcd for $C_{45}H_{35}N_3O_{11}Na$ $[M+Na]^+$:816.2164, found: 816.2165.

1-(2',3',4',6'-Tetra-*O*-benzoyl-β-D-glucopyranosyl)-uracil (15)



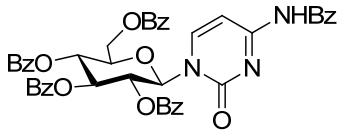
(R_f = 0.30，petroleum：ethyl acetate = 1:1) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (s, 1H), 8.04 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.93 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.90 – 7.87 (m, 2H), 7.82 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.62 – 7.56 (m, 1H), 7.56 – 7.33 (m, 10H), 7.32 – 7.26 (m, 2H), 6.25 (d, *J* = 9.5 Hz, 1H), 6.09 (t, *J* = 9.6 Hz, 1H), 5.83 (dd, *J* = 8.2, 2.2 Hz, 1H), 5.78 (t, *J* = 9.8 Hz, 1H), 5.66 (t, *J* = 9.5 Hz, 1H), 4.67 (dd, *J* = 12.4, 2.7 Hz, 1H), 4.50 (dd, *J* = 12.5, 5.0 Hz, 1H), 4.40 (ddd, *J* = 10.0, 5.0, 2.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.15, 165.61, 165.40, 165.29, 161.99, 150.10, 139.13, 134.05, 133.86, 133.61, 133.48, 130.15, 130.05, 129.94, 129.87, 129.52, 128.71, 128.67, 128.63, 128.52, 128.00, 103.96, 80.89, 75.63, 73.00, 70.31, 68.99, 62.72.

1-(2',3',4',6'-Tetra-*O*-benzoyl-β-D-glucopyranosyl)-thymine (16)



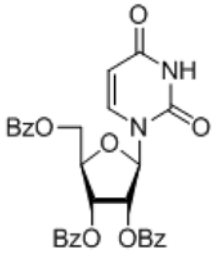
(R_f = 0.41，petroleum：ethyl acetate = 1:1) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H), 8.06 – 8.00 (m, 2H), 7.94 – 7.89 (m, 2H), 7.89 – 7.85 (m, 2H), 7.84 – 7.79 (m, 2H), 7.57 (dd, *J* = 10.6, 4.3 Hz, 1H), 7.51 (q, *J* = 7.4 Hz, 2H), 7.44 (dd, *J* = 10.9, 4.4 Hz, 3H), 7.36 (q, *J* = 8.0 Hz, 4H), 7.31 – 7.25 (m, 3H), 6.24 (d, *J* = 9.4 Hz, 1H), 6.07 (t, *J* = 9.6 Hz, 1H), 5.77 (t, *J* = 9.8 Hz, 1H), 5.67 (t, *J* = 9.5 Hz, 1H), 4.67 (dd, *J* = 12.4, 2.7 Hz, 1H), 4.48 (dd, *J* = 12.4, 5.1 Hz, 1H), 4.38 (ddd, *J* = 10.0, 5.0, 2.7 Hz, 1H), 1.94 (d, *J* = 0.9 Hz, 3H).

***N*^{4}-benzoyl-1-(2',3',4',6'-tetra-*O*-benzoyl-β-D-glucopyranosyl)-cytosine (17)**



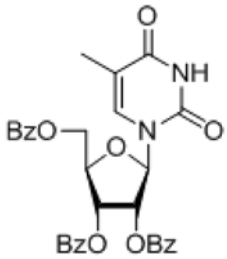
(R_f = 0.42, petroleum: ethyl acetate = 2:3) ^{1}H NMR (400 MHz, DMSO) δ 11.24 (s, 1H), 8.69 (d, *J* = 6.3 Hz, 1H), 8.00 (dd, *J* = 29.9, 7.5 Hz, 4H), 7.82 (dd, *J* = 22.7, 7.5 Hz, 4H), 7.76 – 7.36 (m, 18H), 6.67 (d, *J* = 9.1 Hz, 1H), 6.27 (t, *J* = 9.4 Hz, 1H), 6.09 (t, *J* = 9.3 Hz, 1H), 5.96 (t, *J* = 9.7 Hz, 1H), 4.87 (d, *J* = 9.7 Hz, 1H), 4.53 (s, 2H). ^{13}C NMR (126 MHz, DMSO) δ 167.39, 165.42, 165.10, 164.80, 164.72, 163.54, 154.25, 146.35, 133.99, 133.84, 133.79, 133.47, 132.94, 132.82, 129.52, 129.27, 128.95, 128.80, 128.55, 128.43, 128.13, 97.24, 80.47, 73.34, 71.60, 68.43, 62.48; HRMS (ESI) calcd for $C_{45}H_{35}N_3O_{11}Na$ $[M+Na]^+$:816.2164, found: 816.2164

1-(2',3',5'-Tri-*O*-benzoyl-β-D-ribofuranosyl)-uracil (18)



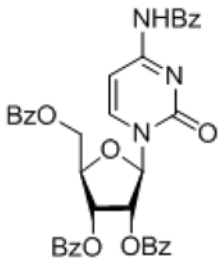
(R_f = 0.52, petroleum: ethyl acetate = 2:3) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (bs, 1H, NH), 8.10 (dd, *J* = 5.2, 3.3 Hz, 2H), 7.98 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.94 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.65 – 7.53 (m, 3H), 7.49 (t, *J* = 7.7 Hz, 2H), 7.44 – 7.34 (m, 5H), 6.32 (d, *J* = 5.6 Hz, 1H, H-1'), 5.89 (dd, *J* = 5.9, 4.5 Hz, 1H, H-3'), 5.75 (t, *J* = 5.8 Hz, 1H, H-2'), 5.61 (dd, *J* = 8.1, 2.1 Hz, 1H, H-5), 4.84 (dd, *J* = 12.0, 2.7 Hz, 1H, H-5'), 4.74 – 4.70 (m, 1H, H-4'), 4.67 (dd, *J* = 12.1, 3.8 Hz, 1H, H-5').

1-(2',3',5'-Tri-*O*-benzoyl-β-D-ribofuranosyl)-thymine (19)



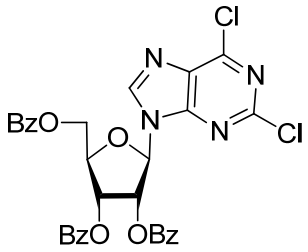
(R_f = 0.58, petroleum: ethyl acetate = 4:5) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (bs, 1H, NH), 8.17 – 8.11 (m, 2H), 7.99 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.95 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.66 – 7.47 (m, 5H), 7.42-7.35 (m, 4H), 7.16 (d, *J* = 1.2 Hz, 1H, H-6), 6.43 (d, *J* = 6.4 Hz, 1H, H-1'), 5.92 (dd, *J* = 6.0, 3.7 Hz, 1H, H-3'), 5.76 (t, *J* = 6.2 Hz, 1H, H-2'), 4.89 (dd, *J* = 12.1, 2.6 Hz, 1H, H-5'), 4.70 (dd, *J* = 6.3, 3.4 Hz, 1H), 4.65 (dd, *J* = 12.1, 3.5 Hz, 1H,), 1.60 (d, *J* = 1.0 Hz, 3H).

***N*^{4}-benzoyl-1-(2',3',5'-tri-*O*-benzoyl-β-D-ribofuranosyl)-cytosine (20)**



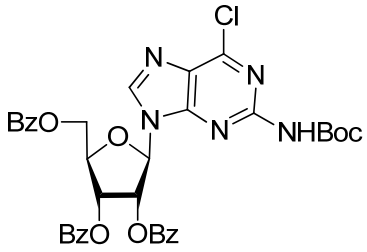
(R_f = 0.28, petroleum: ethyl acetate = 2:3) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 – 8.09 (m, 2H), 8.06 – 7.89 (m, 7H), 7.65-7.45 (m, 9H), 7.41 - 7.35 (m, 4H), 6.47 (d, *J* = 4.6 Hz, 1H, H-1'), 5.92 (t, *J* = 5.5 Hz, 1H), 5.88 – 5.82 (m, 1H), 4.87 (dd, *J* = 12.2, 2.7 Hz, 1H), 4.80 (dd, *J* = 8.3, 3.3 Hz, 1H), 4.74 (dd, *J* = 12.2, 3.9 Hz, 1H).

2',3',5'-Tri-*O*-benzoyl-β-D-ribofuranosyl-2,6-dichloropurine (21)



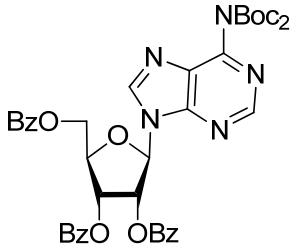
(R_f = 0.39, petroleum: ethyl acetate = 2:1) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (s, 1H, H-8), 8.08 – 8.00 (m, 4H, -OBz), 7.93 (d, *J* = 7.3 Hz, 2H, -OBz), 7.63 – 7.53 (m, 3H, -OBz), 7.45 (dd, *J* = 15.1, 7.5 Hz, 4H, -OBz), 7.37 (t, *J* = 7.8 Hz, 2H, -OBz), 6.49 (d, *J* = 5.4 Hz, 1H, H-1'), 6.19 (t, *J* = 5.6 Hz, 1H, H-3'), 6.16 – 6.12 (m, 1H, H-2'), 4.93 (dd, *J* = 12.1, 3.2 Hz, 1H, H-5'), 4.88 (dd, *J* = 7.5, 3.9 Hz, 1H, H-4'), 4.73 (dd, *J* = 12.1, 4.0 Hz, 1H, H-5'). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.16, 165.41, 165.25 (3 Bz), 153.57, 152.73, 152.42,(C-6, C-2, C-4), 143.98(C-8), 134.12, 134.01, 133.76, 131.46, 130.02, 129.98, 129.73, 129.15, 128.85, 128.75, 128.72, 128.65, 128.19,(3Bz, C-5) 87.12(C-1'), 81.61(C-4'), 74.41(C-3'), 71.66(C-2'), 63.60(C-5').

2-*tert*-Butoxycarbonylamino-6-chloro-9-(2',3',5'-tri-*O*-benzoyl-β-D-ribofuranosyl)purine (22)



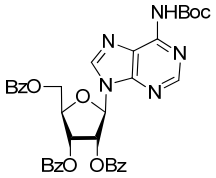
(R_f = 0.44, petroleum: ethyl acetate = 2:1) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (s, 1H), 8.04 – 7.98 (m, 4H), 7.96 – 7.91 (m, 2H), 7.62 – 7.52 (m, 4H), 7.44 – 7.34 (m, 6H), 6.61 (t, *J* = 5.9 Hz, 1H), 6.32 – 6.26 (m, 2H), 4.97 (dd, *J* = 11.9, 3.8 Hz, 1H), 4.92 – 4.87 (m, 1H), 4.79 (dd, *J* = 11.9, 5.4 Hz, 1H), 1.48 (s, 9H).

***N*,*N*-Di-*tert*-butoxycarbonyl-9-(2',3',5'-tri-*O*-benzoyl-β-D-ribofuranosyl)adenine (23)**



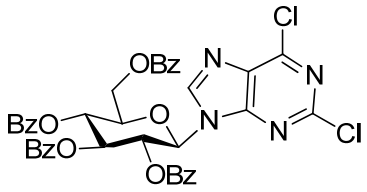
(R_f = 0.52, petroleum: ethyl acetate = 1:1) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.76 (s, 1H), 8.25 (s, 1H), 8.11 (d, *J* = 7.3 Hz, 2H), 8.02 (d, *J* = 7.3 Hz, 2H), 7.92 (d, *J* = 7.3 Hz, 2H), 7.63 – 7.52 (m, 3H), 7.49 – 7.33 (m, 6H), 6.51 (d, *J* = 5.3 Hz, 1H), 6.41 (t, *J* = 5.5 Hz, 1H), 6.27 (t, *J* = 5.2 Hz, 1H), 4.92 (dd, *J* = 12.1, 3.2 Hz, 1H), 4.86 (dd, *J* = 7.9, 4.1 Hz, 1H), 4.73 (dd, *J* = 12.1, 4.2 Hz, 1H), 1.44 (s, 18H).

***N*-tert-Butoxycarbonyl-9-(2',3',5'-tri-*O*-benzoyl-β-D-ribofuranosyl)adenine (S1)**



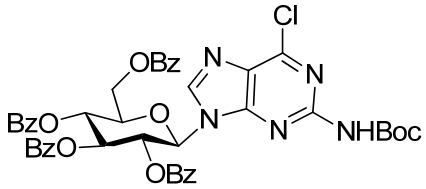
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (s, 1H), 8.10 (d, *J* = 6.8 Hz, 4H), 8.01 (d, *J* = 7.2 Hz, 2H), 7.92 (d, *J* = 7.2 Hz, 2H), 7.62 – 7.52 (m, 3H), 7.48 – 7.33 (m, 6H), 6.47 (d, *J* = 5.2 Hz, 1H), 6.38 (t, *J* = 5.5 Hz, 1H), 6.25 (t, *J* = 5.3 Hz, 1H), 4.93 (dd, *J* = 12.2, 3.2 Hz, 1H), 4.84 (dd, *J* = 7.9, 4.1 Hz, 1H), 4.70 (dd, *J* = 12.2, 4.2 Hz, 1H), 1.55 (s, 9H).

2',3',4',6'-Tetra-*O*-benzoyl-β-D- glucopyranosyl -2,6-dichloropurine (24)



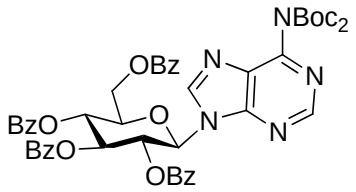
(R_f = 0.32, petroleum: ethyl acetate = 2:1) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (s, 1H, H-8), 8.02 (d, *J* = 7.4 Hz, 2H), 7.94 (d, *J* = 7.4 Hz, 2H), 7.82 (d, *J* = 7.4 Hz, 2H), 7.74 (d, *J* = 7.4 Hz, 2H), 7.60 - 7.50 (m, 2H), 7.48 - 7.34 (m, 6H), 7.32 - 7.26 m, 4H), 6.28 (d, *J* = 9.3 Hz, 1H, H-1'), 6.19 (t, *J* = 9.6 Hz, 1H), 6.05 (t, *J* = 9.5 Hz, 1H), 5.92 (t, *J* = 9.4 Hz, 1H), 4.70 (dd, *J* = 14.5, 4.8 Hz, 1H), 4.54 (d, *J* = 9.4 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.09, 165.62, 165.21, 164.96, (4 Bz), 153.67, 153.10, 152.37(C-2, C-4, C-6), 143.45(C-8), 134.13, 133.90, 133.69, 133.50, 130.72(C-5), 130.02, 129.97, 129.89, 129.86, 129.34, 128.67, 128.61, 128.53, 128.43, 128.39, 127.44, 81.39(C-1'), 76.01, 72.83, 71.21, 68.80, 62.53; HRMS (ESI) calcd for $C_{39}H_{29}N_4O_9Cl_2$ $[M+H]^+$:767.1312, found: 767.1307.

2-*tert*-Butoxycarbonylamino-6-chloro-9-(2',3',4',6'-tetra-*O*-benzoyl-β-D-glucopyranosyl)purine (25)



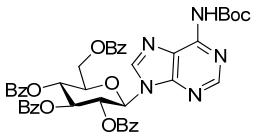
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (s, 1H), 8.05 – 7.99 (m, 2H), 7.97 – 7.93 (m, 2H), 7.85 – 7.79 (m, 2H), 7.78 – 7.71 (m, 2H), 7.58 – 7.36 (m, 9H), 7.31 – 7.22 (m, 4H), 6.27 (d, *J* = 9.3 Hz, 1H, H-1'), 6.18 (t, *J* = 9.6 Hz, 1H), 6.02 (t, *J* = 9.5 Hz, 1H), 5.89 (t, *J* = 9.5 Hz, 1H), 4.68 (q, *J* = 4.4 Hz, 1H), 4.57 – 4.48 (m, 2H), 1.58 (s, 9H, Boc).

***N*,*N*-Di-*tert*-butoxycarbonyl-9-(2',3',4',6'-tetra-*O*-benzoyl-β-D-glucopyranosyl)adenine (26)**



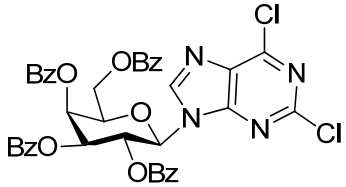
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.79 (s, 1H), 8.42 (s, 1H), 8.04 – 7.99 (m, 2H), 7.97 – 7.93 (m, 2H), 7.84 – 7.80 (m, 2H), 7.71 – 7.67 (m, 2H), 7.58 – 7.51 (m, 2H), 7.47 – 7.36 (m, 6H), 7.32 – 7.22 (m, 4H), 6.34 (d, *J* = 8.9 Hz, 1H), 6.19 – 6.10 (m, 2H), 5.93 (t, *J* = 9.5 Hz, 1H), 4.70 (dd, *J* = 12.9, 3.3 Hz, 1H), 4.56 – 4.48 (m, 2H), 1.33 (s, 18H).

_N_-*tert*-Butoxycarbonyl-9-(2',3',4',6'-tetra-_O_-benzoyl-β-D-glucopyranosyl)adenine (S2)



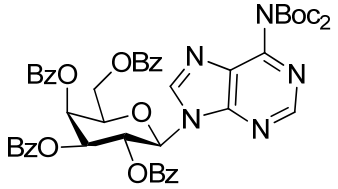
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (s, 1H), 8.31 (s, 1H), 8.02 (d, *J* = 7.2 Hz, 2H), 7.96 – 7.91 (m, 2H), 7.84 – 7.79 (m, 2H), 7.70 (d, *J* = 7.3 Hz, 2H), 7.59 – 7.50 (m, 2H), 7.47 – 7.35 (m, 6H), 7.31 – 7.23 (m, 4H), 6.31 (d, *J* = 9.2 Hz, 1H), 6.16 (t, *J* = 9.6 Hz, 1H), 6.08 (t, *J* = 9.4 Hz, 1H), 5.92 (t, *J* = 9.6 Hz, 1H), 4.69 (d, *J* = 9.8 Hz, 1H), 4.56 – 4.47 (m, 2H), 1.53 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.14, 165.67, 165.27, 165.02, 153.10, 151.22, 149.92, 149.50, 140.21, 133.97, 133.85, 133.62, 133.46, 130.05, 129.92, 129.89, 129.47, 128.67, 128.59, 128.55, 128.52, 127.70, 121.14, 82.70, 81.08, 75.86, 73.11, 71.33, 69.02, 62.66, 28.21. HRMS (ESI) calcd for $C_{44}H_{40}N_5O_{11}$ $[M+H]^+$:814.2724, found: 814.2717.

2',3',4',6'-Tetra-_O_-benzoyl-β-D- galactopyranosyl -2,6-dichloropurine (27)



(R_f = 0.51, petroleum: acetone = 2:1) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (s, 1H, H-8), 8.17 (d, *J* = 7.4 Hz, 2H), 7.98 (d, *J* = 7.4 Hz, 2H), 7.77 (dd, *J* = 18.5, 7.5 Hz, 4H), 7.68 (d, *J* = 7.4 Hz, 1H), 7.55 (dd, *J* = 14.4, 7.2 Hz, 3H), 7.49 – 7.38 (m, 4H), 7.34 – 7.23 (m, 4H), 6.40 (t, *J* = 9.7 Hz, 1H, H-2'), 6.26 – 6.15 (m, 2H, H-1' and H-4'), 5.92 (dd, *J* = 10.1, 3.1 Hz, 1H, H-3'), 4.76 – 4.62 (m, 2H, H-5' and H-6'), 4.52 (dd, *J* = 11.4, 5.4 Hz, 1H, H-6'). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.09, 165.45, 165.43, 165.05(4 Bz), 153.67, 153.11, 152.40(C-2, C-4 and C-6), 143.82(C-8), 134.12, 134.09, 133.72, 133.57, 130.96(C-5), 130.12, 129.91, 129.88, 129.18, 129.04, 128.81, 128.64, 128.60, 128.53, 128.45, 127.62, 82.25(C-1'), 75.03(C-5'), 71.72(C-3'), 68.87(C-2'), 68.06(C-4'), 62.12(C-6'). HRMS (ESI) calcd for $C_{39}H_{29}N_4O_9Cl_2$ $[M+H]^+$:767.1312, found: 767.1305.

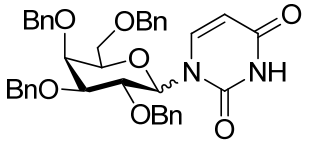
_N_,_N_-Di-*tert*-butoxycarbonyl-9-(2',3',4',6'-tetra-_O_-benzoyl-β-D-galactopyranosyl)adenine (28)



^{1}H NMR (400 MHz, $CDCl_3$) δ 8.85 (s, 1H, H-2), 8.49 (s, 1H, H-8), 8.22 – 8.16 (m, 2H), 8.02 – 7.97 (m, 2H), 7.83 – 7.77 (m, 2H), 7.69 (dd, *J* = 7.2, 4.7 Hz, 3H), 7.56 (dd, *J* = 16.1, 7.9 Hz, 3H), 7.48 – 7.38 (m, 4H), 7.29 – 7.22 (m, 4H), 6.49 (t, *J* = 9.7 Hz, 1H, H-2'), 6.31 (d, *J* = 9.4 Hz, 1H, H-1'), 6.19 (d, *J* = 3.0

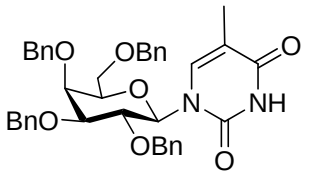
Hz, 1H, H-4'), 5.90 (dd, *J* = 10.1, 3.2 Hz, 1H, H-3'), 4.72-4.62 (m, 2H, H-5' and H-6'), 4.51 (dd, *J* = 11.1, 5.7 Hz, 1H , H-6'), 1.35 (s, 18H, Boc). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.10, 165.45, 165.42, 164.98(4Bz), 153.26(C-4), 152.56(C-2), 150.82(C-6), 150.17(Boc), 142.51(C-8), 134.06, 133.86, 133.65, 133.52, 130.12, 129.93, 129.91, 129.79, 129.25, 128.99, 128.96, 128.76, 128.58, 128.49, 128.46, 127.85(C-5), 83.85(Boc), 81.68(C-1'), 74.70(C-5'), 72.05(C-3'), 68.87(C-2'), 68.12(C-4'), 62.04(C-6'), 27.76(Boc). HRMS (ESI) calcd for $C_{49}H_{48}N_5O_{13}$ $[M+H]^+$:914.3249, found: 914.3247.

1-(2',3',4',6'-Tetra-*O*-benzyl-D-galactopyranosyl)-uracil (29)



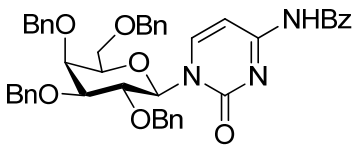
For the α-isomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (s, 1H, NH), 7.59 (d, *J* = 8.2 Hz, 1H, H-6), 7.39 – 7.24 (m, 18H, benzyl), 7.02 (dd, *J* = 6.6, 2.9 Hz, 2H, benzyl), 6.04 (d, *J* = 1.8 Hz, 1H, H-1'), 5.60 (dd, *J* = 8.2, 2.1 Hz, 1H, H-5), 4.65 – 4.47 (m, 7H), 4.32 (d, *J* = 11.7 Hz, 1H), 4.16 (dt, *J* = 11.9, 4.6 Hz, 2H), 4.05 (dd, *J* = 6.4, 2.8 Hz, 1H), 3.83 – 3.77 (m, 2H), 3.68 (dd, *J* = 3.9, 1.9 Hz, 1H).
For the β-isomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (s, 1H, NH), 7.41 – 7.22 (m, 18H, benzyl), 7.17 (dd, *J* = 6.5, 3.1 Hz, 2H, benzyl), 6.94 (d, *J* = 8.2 Hz, 1H, H-6), 5.58 (d, *J* = 8.9 Hz, 1H, H-1'), 5.34 (dd, *J* = 8.1, 2.1 Hz, 1H, H-5), 4.95 (d, *J* = 11.3 Hz, 1H, CH_2-Ph), 4.84 – 4.72 (m, 3H, CH_2-Ph), 4.57 (dd, *J* = 16.6, 11.6 Hz, 2H, CH_2-Ph), 4.44 (q, *J* = 11.9 Hz, 2H, CH_2-Ph), 3.99 (d, *J* = 2.0 Hz, 1H, H-4'), 3.87 (t, *J* = 9.2 Hz, 1H, H-3'), 3.78 – 3.70 (m, 2H, H-2', H-5'), 3.58 – 3.47 (m, 2H, H-6'a, H-6'b).

1-(2',3',4',6'-Tetra-*O*-benzyl-β-D-galactopyranosyl)-thymine (30)



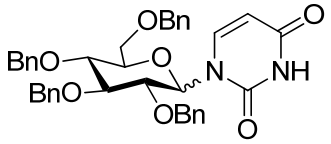
(R_f = 0.31, petroleum: ethyl acetate = 1:1) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (s, 1H, NH), 7.42 – 7.13 (m, 20H, benzyl), 6.65 (d, *J* = 1.2 Hz, 1H, H-6), 5.58 (d, *J* = 8.9 Hz, 1H, H-1'), 4.97 (d, *J* = 11.4 Hz, 1H, CH_2Ph), 4.83 – 4.72 (m, 3H, CH_2Ph), 4.58 (dd, *J* = 20.0, 11.7 Hz, 2H, CH_2Ph), 4.45 (q, *J* = 11.9 Hz, 2H, CH_2Ph), 4.00 (d, *J* = 1.9 Hz, 1H), 3.85 (t, *J* = 9.1 Hz, 1H), 3.79 – 3.70 (m, 2H), 3.53 (ddd, *J* = 15.0, 9.2, 6.6 Hz, 2H, H-6'a , H-6'b), 1.63 (d, *J* = 1.1 Hz, 3H, CH_3-thymine).

***N*^{4}-benzoyl-1-(2',3',4',6'-tetra-*O*-benzyl-β-D-galactopyranosyl)-cytosine (31)**



(R_f = 0.49, petroleum: ethyl acetate = 1:2) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (s, 2H), 7.62 (t, *J* = 7.4 Hz, 1H), 7.53 (t, *J* = 7.6 Hz, 2H), 7.42 – 7.10 (m, 22H), 5.88 (d, *J* = 8.2 Hz, 1H, H-1'), 4.98 (d, *J* = 11.3 Hz, 1H, CH_2Ph), 4.84 – 4.72 (m, 3H, CH_2Ph), 4.61 (d, *J* = 11.3 Hz, 1H, CH_2Ph), 4.55 – 4.38 (m, 3H, CH_2Ph), 4.01 (d, *J* = 2.0 Hz, 1H), 3.91 (t, *J* = 9.2 Hz, 1H), 3.84 – 3.74 (m, 2H), 3.61 – 3.48 (m, 2H).

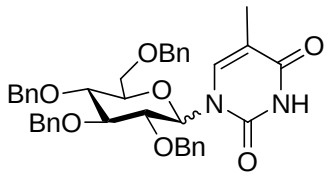
1-(2',3',4',6'-Tetra-*O*-benzyl-D-glucopyranosyl)-uracil (32)



For the α-isomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (s, 1H), 7.62 (d, *J* = 8.2 Hz, 1H), 7.39 – 7.27 (m, 16H), 7.19 (dd, *J* = 7.0, 2.4 Hz, 2H), 7.14 (dd, *J* = 6.6, 2.7 Hz, 2H), 6.03 (d, *J* = 3.2 Hz, 1H), 5.62 (dd, *J* = 8.2, 2.2 Hz, 1H), 4.59 – 4.40 (m, 7H), 4.35 – 4.28 (m, 2H), 4.05 (t, *J* = 3.0 Hz, 1H), 3.83 (t, *J* = 3.1 Hz, 1H), 3.73 (dd, *J* = 10.6, 2.7 Hz, 1H), 3.70 – 3.62 (m, 2H).

For the β-isomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, *J* = 1.5 Hz, 1H), 7.38 – 7.26 (m, 16H), 7.17 (td, *J* = 8.1, 3.1 Hz, 4H), 6.89 (d, *J* = 8.2 Hz, 1H), 5.61 (d, *J* = 9.1 Hz, 1H), 5.37 (dd, *J* = 8.1, 2.2 Hz, 1H), 4.93 (s, 2H), 4.82 (dd, *J* = 22.9, 11.3 Hz, 2H), 4.60 (d, *J* = 10.8 Hz, 1H), 4.56 – 4.45 (m, 3H), 3.86 (t, *J* = 9.0 Hz, 1H), 3.75 – 3.58 (m, 4H), 3.47 (t, *J* = 9.0 Hz, 1H)

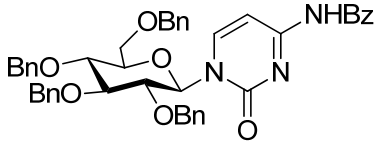
1-(2',3',4',6'-Tetra-*O*-benzyl-D-glucopyranosyl)-thymine (33)



For the α-isomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (s, 1H), 7.45 (d, *J* = 1.1 Hz, 1H), 7.39 – 7.27 (m, 16H), 7.20 (dd, *J* = 7.0, 2.4 Hz, 2H), 7.12 (dd, *J* = 7.3, 2.0 Hz, 2H), 6.04 (d, *J* = 3.2 Hz, 1H), 4.59 – 4.49 (m, 5H), 4.44 (t, *J* = 11.1 Hz, 2H), 4.36 – 4.27 (m, 2H), 4.03 (t, *J* = 3.0 Hz, 1H), 3.83 (t, *J* = 3.1 Hz, 1H), 3.74 (dd, *J* = 10.5, 3.0 Hz, 1H), 3.69 – 3.63 (m, 2H), 1.81 (d, *J* = 0.9 Hz, 3H).

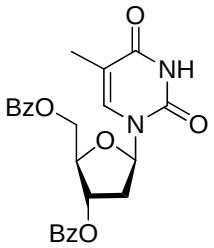
For the β-isomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (s, 1H), 7.38 – 7.20 (m, 16H), 7.17 (dd, *J* = 5.7, 3.7 Hz, 4H), 6.65 (d, *J* = 1.1 Hz, 1H), 5.59 (d, *J* = 9.2 Hz, 1H), 4.94 (s, 2H), 4.81 (dd, *J* = 21.7, 11.4 Hz, 2H), 4.59 (d, *J* = 10.7 Hz, 1H), 4.55-4.45 (m, 3H), 3.86 (t, *J* = 9.0 Hz, 1H), 3.77 – 3.68 (m, 2H), 3.65 (dd, *J* = 11.0, 1.7 Hz, 1H), 3.62 – 3.56 (m, 1H), 3.46 (t, *J* = 9.0 Hz, 1H), 1.67 (d, *J* = 0.9 Hz, 3H).

***N*^{4}-benzoyl-1-(2',3',4',6'-tetra-*O*-benzyl-β-D-glucopyranosyl)-cytosine (34)**



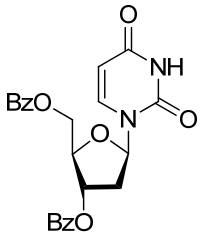
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (s, 2H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 2H), 7.38 – 7.27 (m, 14H), 7.23 – 7.10 (m, 8H), 5.91 (d, *J* = 7.7 Hz, 1H, H-1'), 4.97 – 4.89 (m, 2H, CH_2Ph), 4.95-4.80 (m, 1H, CH_2Ph), 4.74 (d, *J* = 11.8 Hz, 1H, CH_2Ph), 4.61 (d, *J* = 10.7 Hz, 1H, CH_2Ph), 4.54 (d, *J* = 12.2 Hz, 1H, CH_2Ph), 4.47 (dd, *J* = 12.0, 3.2 Hz, 2H, CH_2Ph), 3.91 (t, *J* = 8.9 Hz, 1H, H-4'), 3.82 – 3.72 (m, 2H, H-3' and H-2'), 3.70 – 3.62 (m, 2H), 3.52 (t, *J* = 8.7 Hz, 1H).

1-(3',5'-Di-*O*-benzoyl-2'-deoxy-β-D-ribofuranosyl)-thymine (35)



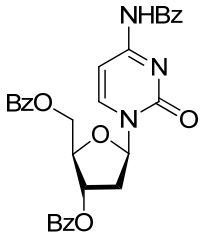
(R_f = 0.50, petroleum: ethyl acetate = 1:2) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.77 (s, 1H), 8.06 (t, *J* = 8.2 Hz, 4H), 7.62 (t, *J* = 7.4 Hz, 2H), 7.48 (t, *J* = 7.0 Hz, 4H),7.26 (s,1H, H-6), 6.47 (dd, *J* = 8.6, 5.6 Hz, 1H, H-1'), 5.66 (d, *J* = 6.4 Hz, 1H, H-3'), 4.80 (dd, *J* = 12.2, 2.8 Hz, 1H, H-5'), 4.69 (dd, *J* = 12.2, 3.3 Hz, 1H, H-5'), 4.54 (d, *J* = 2.2 Hz, 1H, H-4'), 2.72 (dd, *J* = 14.2, 5.2 Hz, 1H, H-2'), 2.41 – 2.29 (m, 1H, H-2'), 1.62 (s, 3H, -CH_3).

1-(3',5'-Di-*O*-benzoyl-2'-deoxy-β-D-ribofuranosyl)-uracil (36)



(R_f = 0.43, petroleum: ethyl acetate = 1:2) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 – 8.05 (m, 3H), 8.02 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.65 – 7.59 (m, 2H), 7.54 – 7.45 (m, 5H), 6.40 (dd, *J* = 8.4, 5.6 Hz, 1H), 5.63 (dt, *J* = 6.5, 2.1 Hz, 1H), 5.59 (dd, *J* = 8.2, 2.3 Hz, 1H), 4.77 – 4.68 (m, 2H), 4.56 (dd, *J* = 6.0, 3.4 Hz, 1H), 2.76 (ddd, *J* = 14.4, 5.7, 2.0 Hz, 1H), 2.33 (ddd, *J* = 14.6, 8.3, 6.7 Hz, 1H).

***N*^{4}-benzoyl-1-(3',5'-Di-*O*-benzoyl-2'-deoxy-β-D-ribofuranosyl)-cytosine (37)**



(R_f = 0.32, petroleum: ethyl acetate = 1:2) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 - 8.05 (m, 3H), 8.03 – 7.97 (m, 2H), 7.89 (d, *J* = 7.5 Hz, 2H), 7.65 - 7.42 (m, 10H), 6.41 (dd, *J* = 7.9, 5.6 Hz, 1H, H-1'), 5.64 (dt, *J* = 6.4, 1.9 Hz, 1H), 4.76 (qd, *J* = 12.2, 3.5 Hz, 2H), 4.67 (dd, *J* = 5.8, 3.3 Hz, 1H), 3.16 – 3.06 (m, 1H, H-2'), 2.31 (ddd, *J* = 14.5, 7.8, 6.6 Hz, 1H, H-2').

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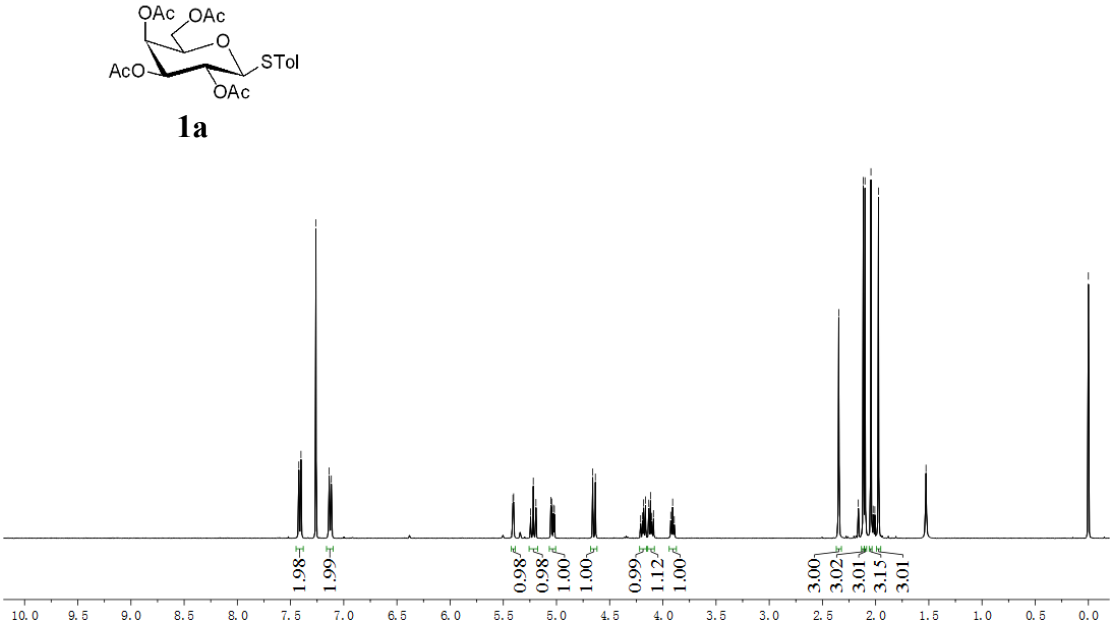
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5.03
5.02
4.66
4.63
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4.16
4.13
4.12
4.09
3.91
2.35
2.16
2.12
2.10
2.04
2.02
1.97
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STol
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OAc
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1.99
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1.00
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3.01

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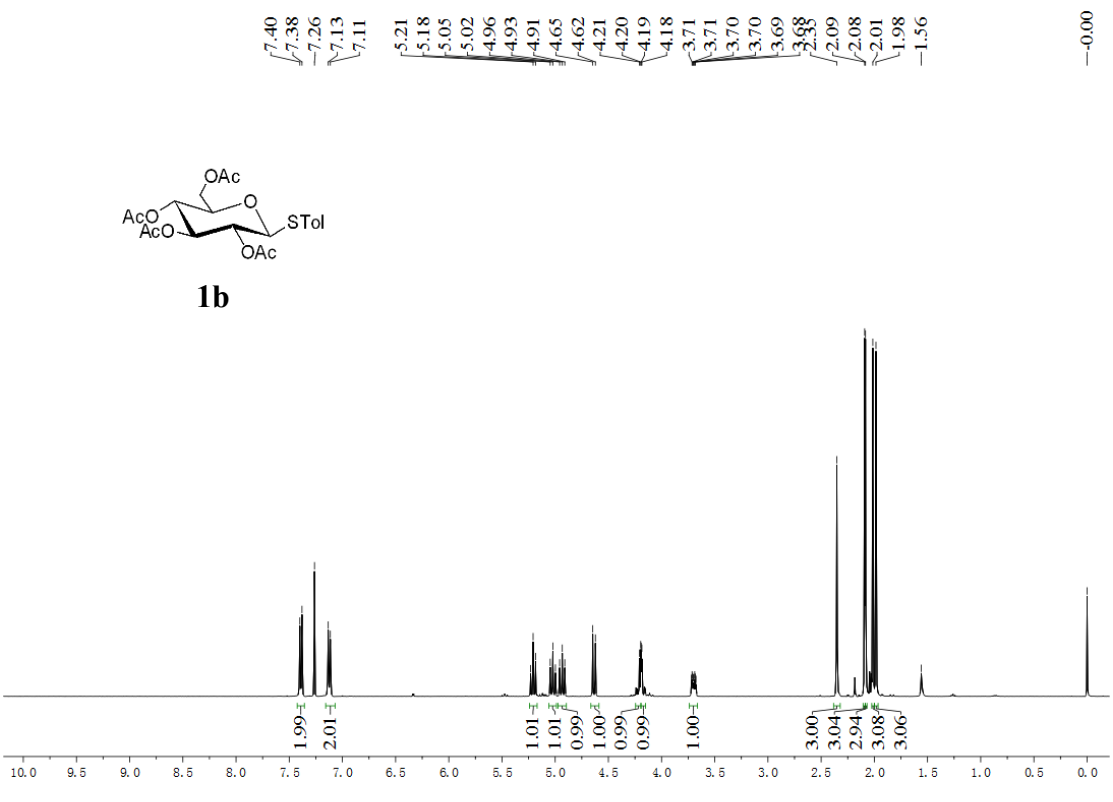
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3.70
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2.08
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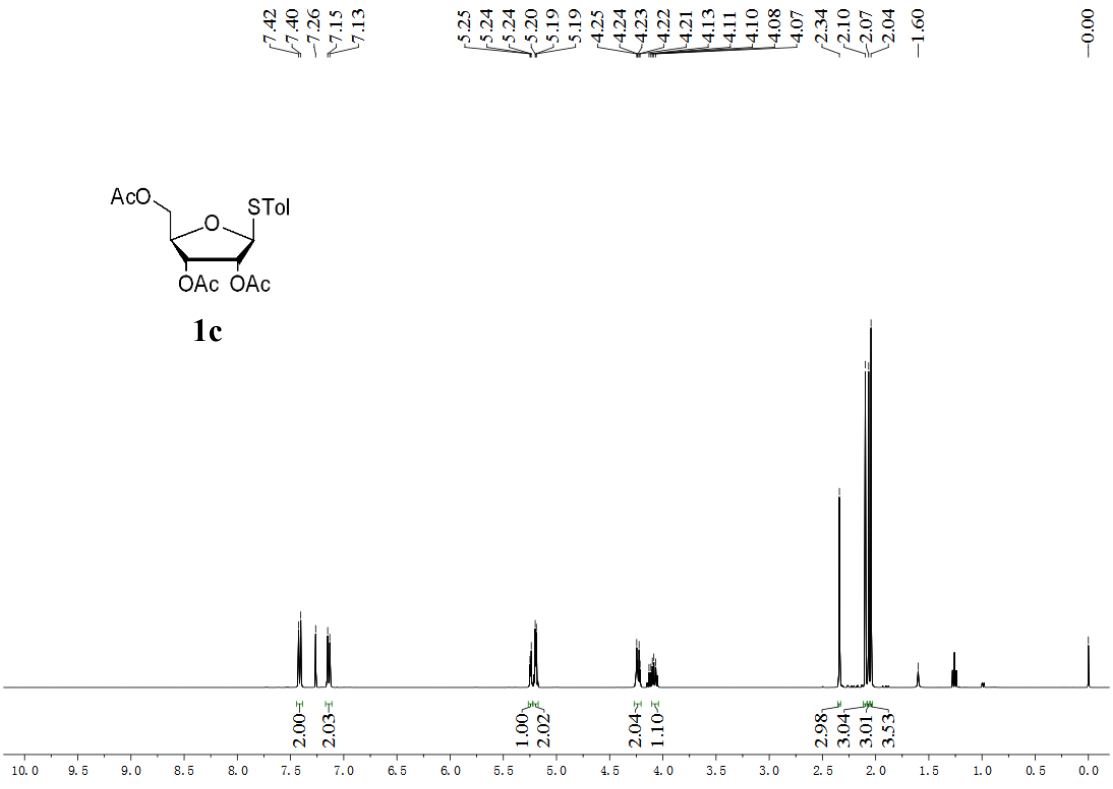
1b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4.07
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2.07
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STol
O
OAc OAc
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1.00
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3.04
3.01
3.53
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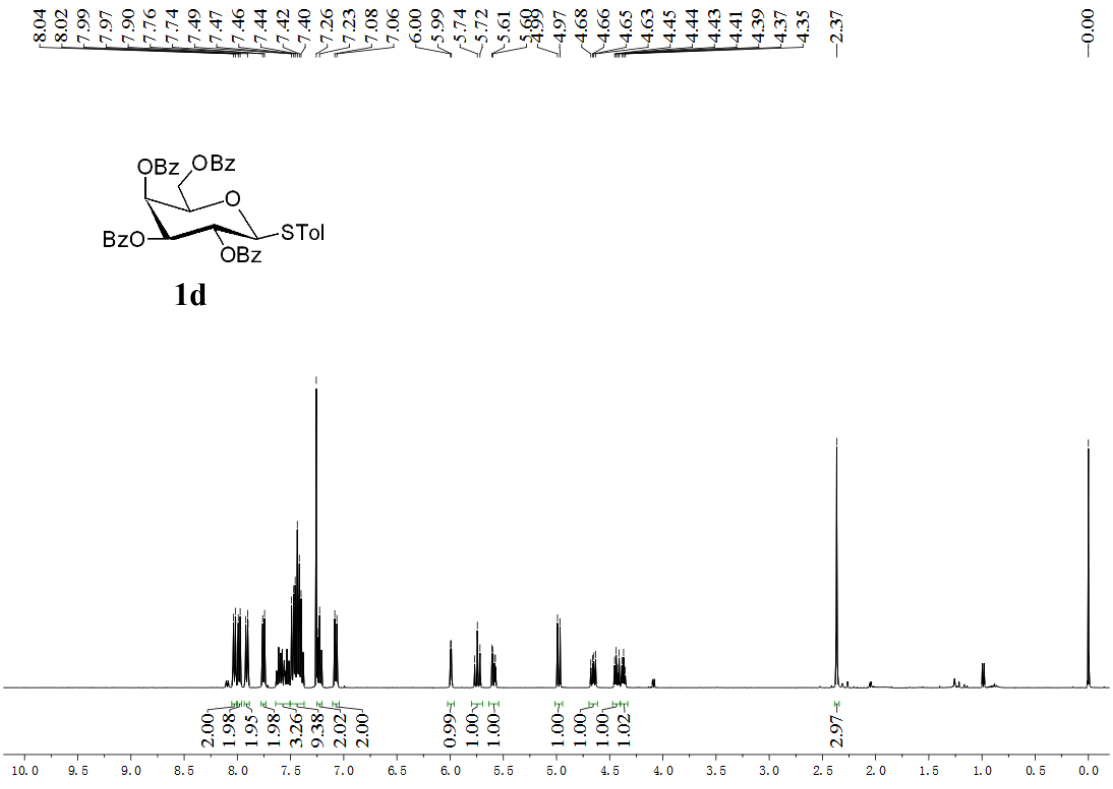
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7.26
7.23
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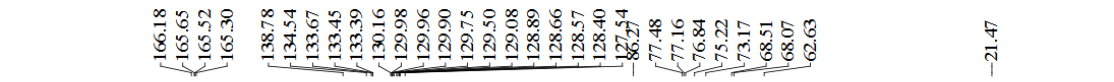
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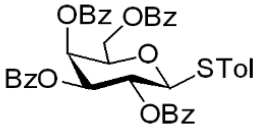
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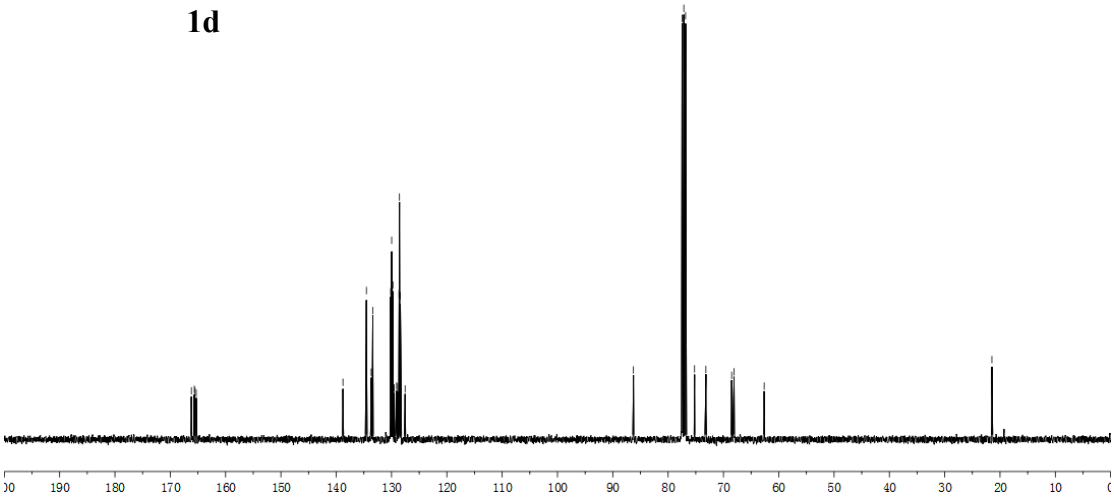
1d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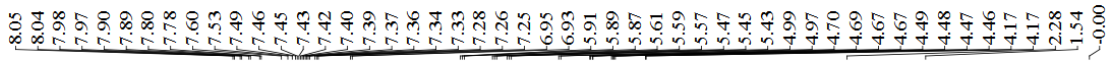
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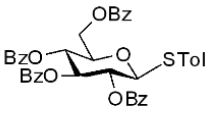
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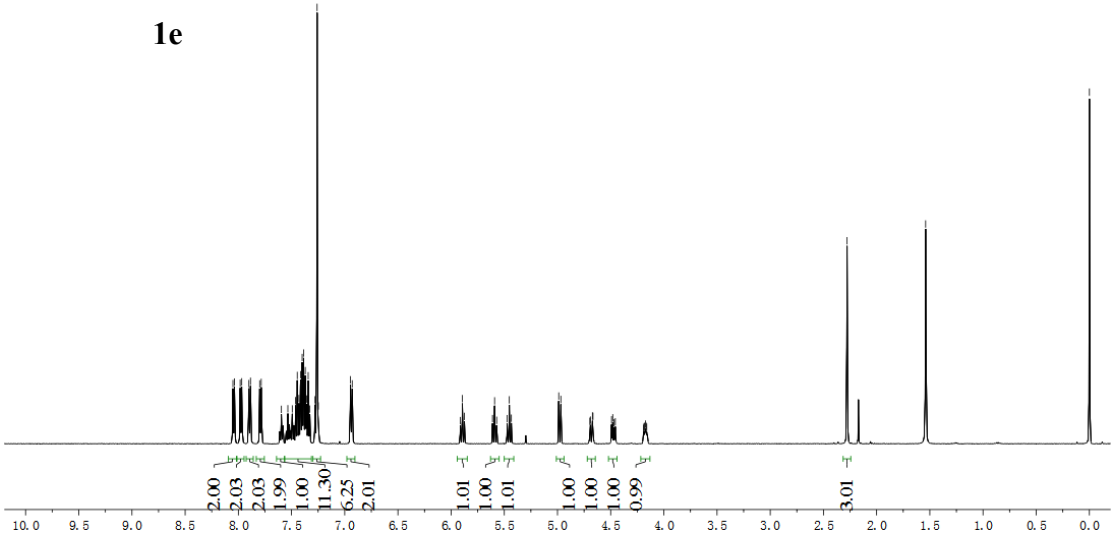
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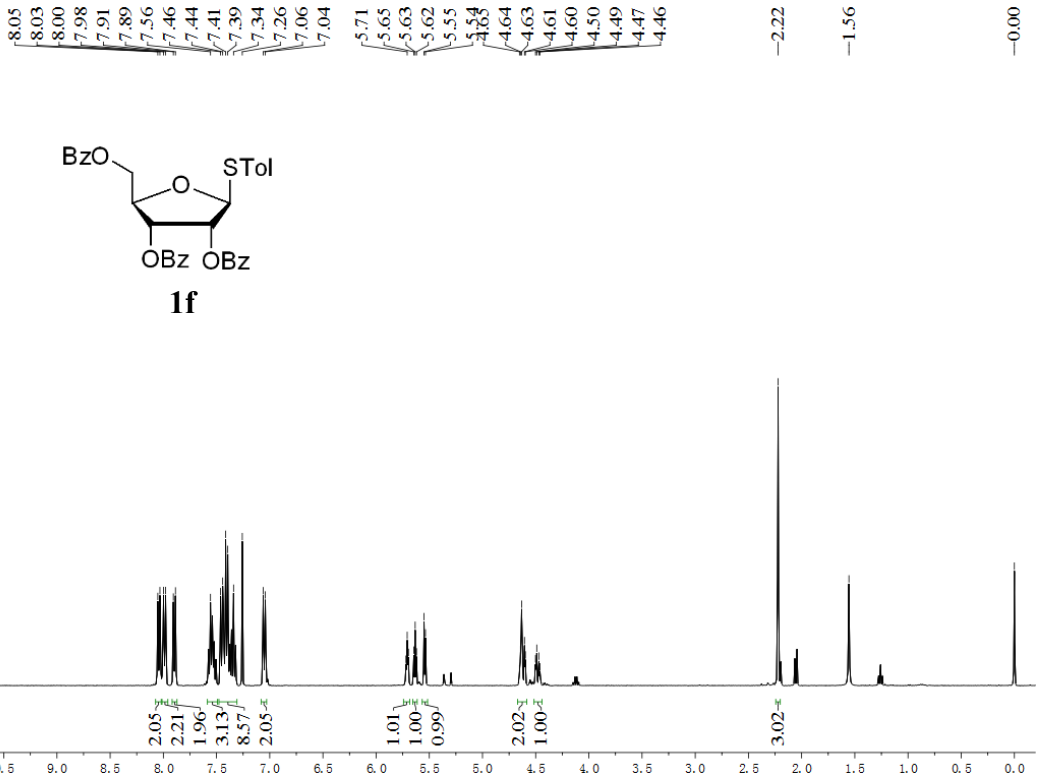
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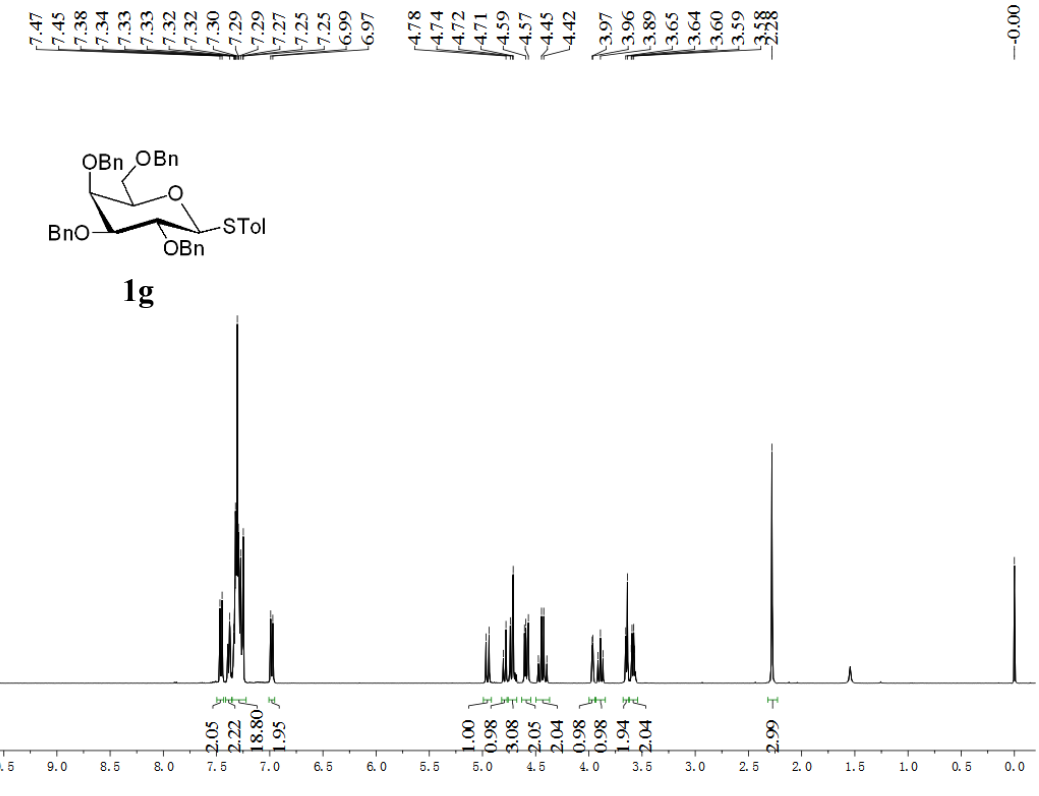
1f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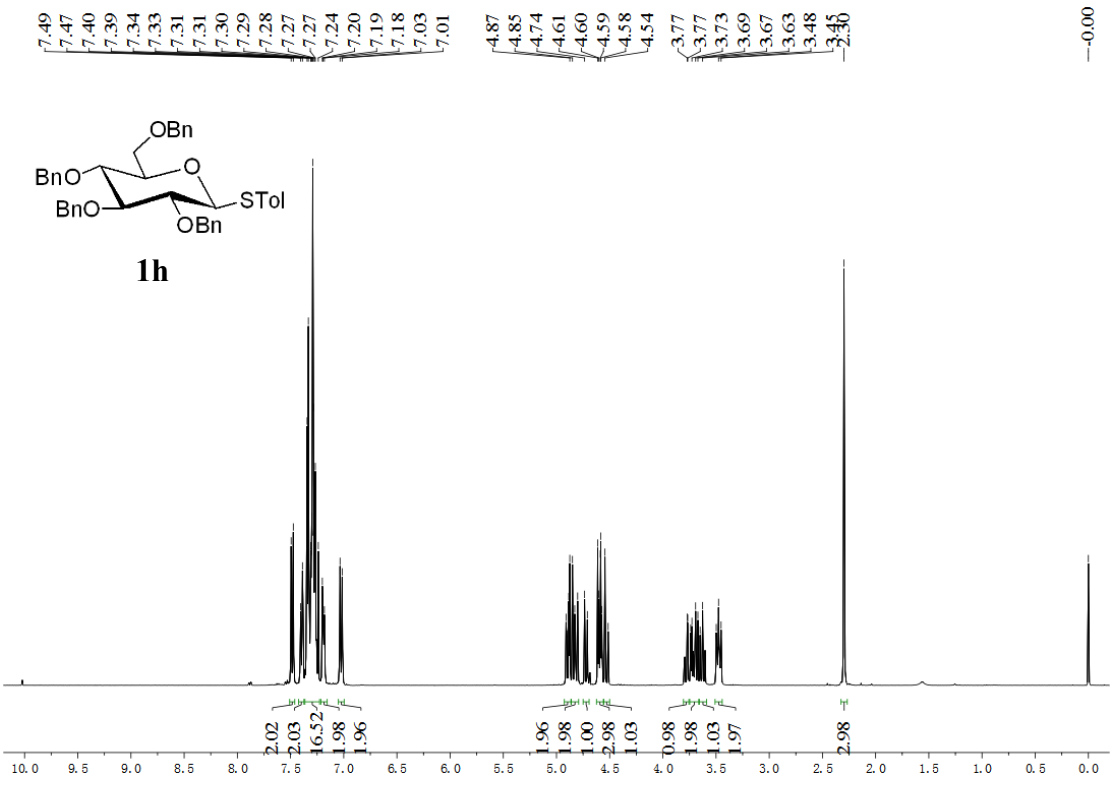
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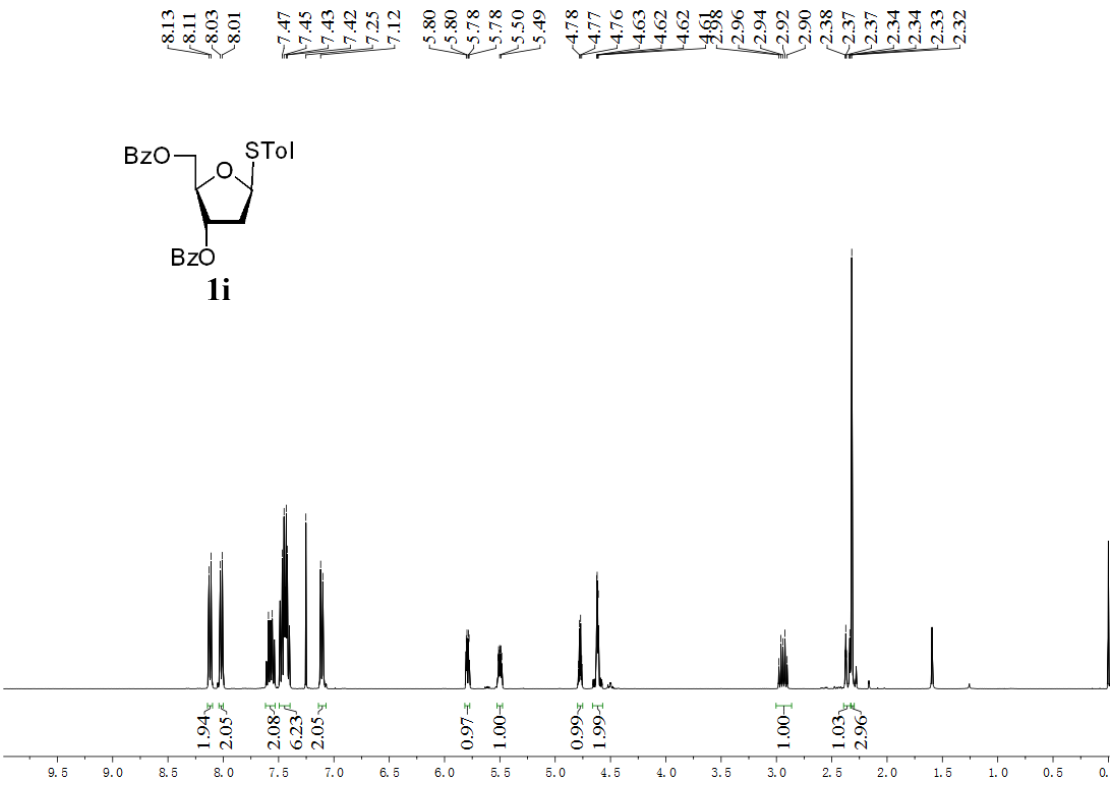
1h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O
STol
BzO
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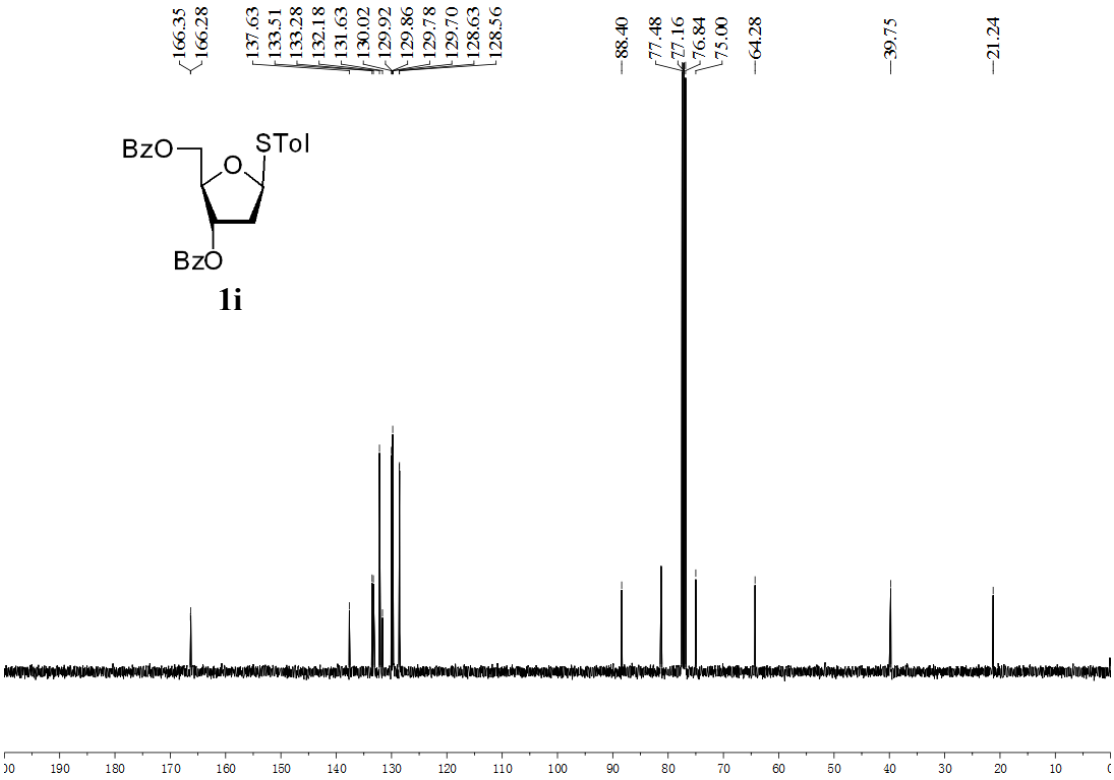
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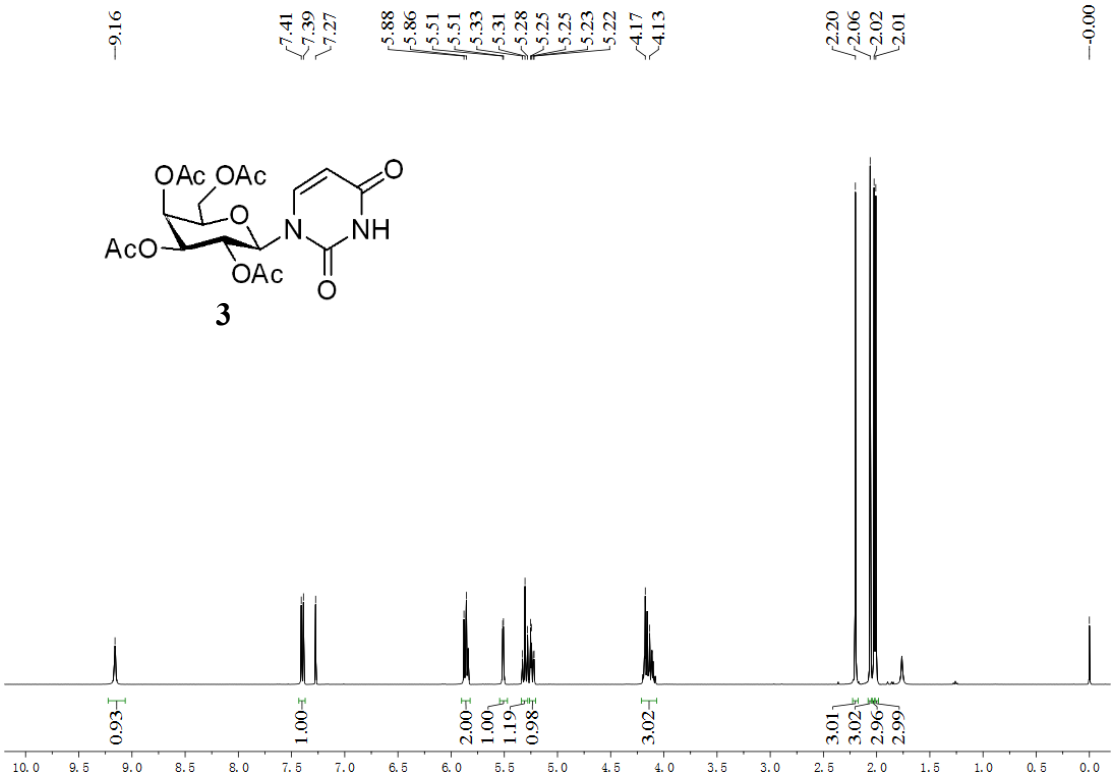
1i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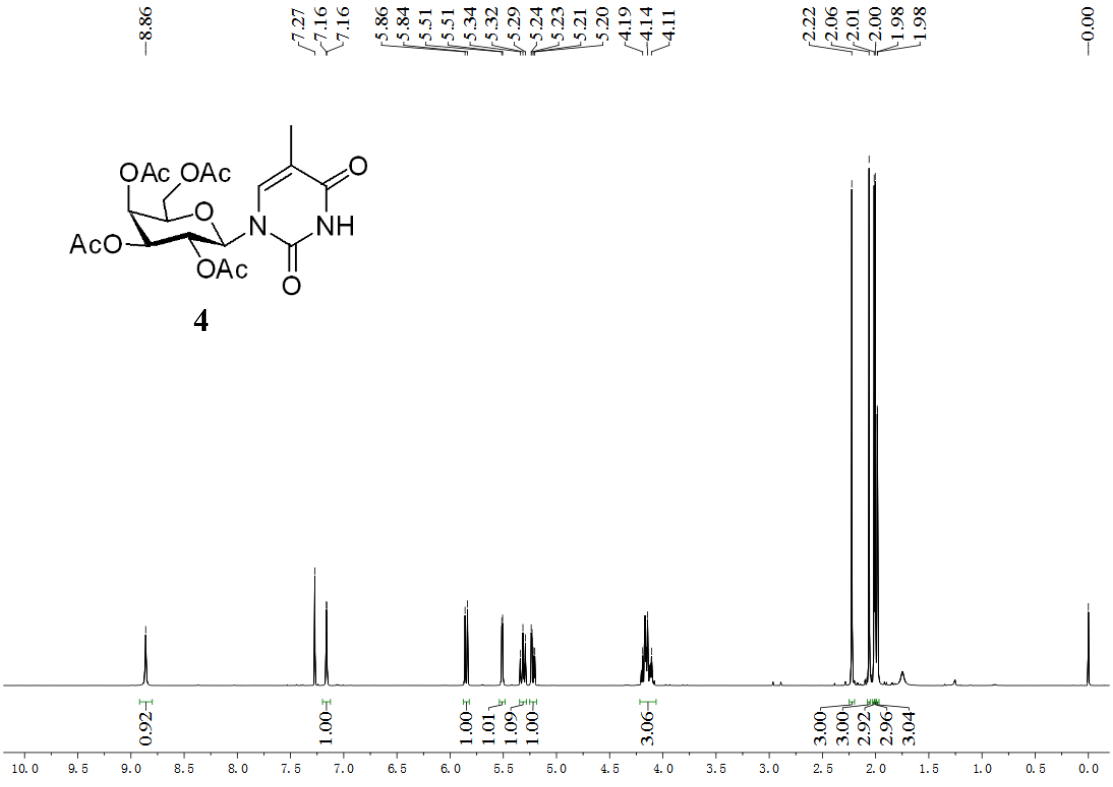
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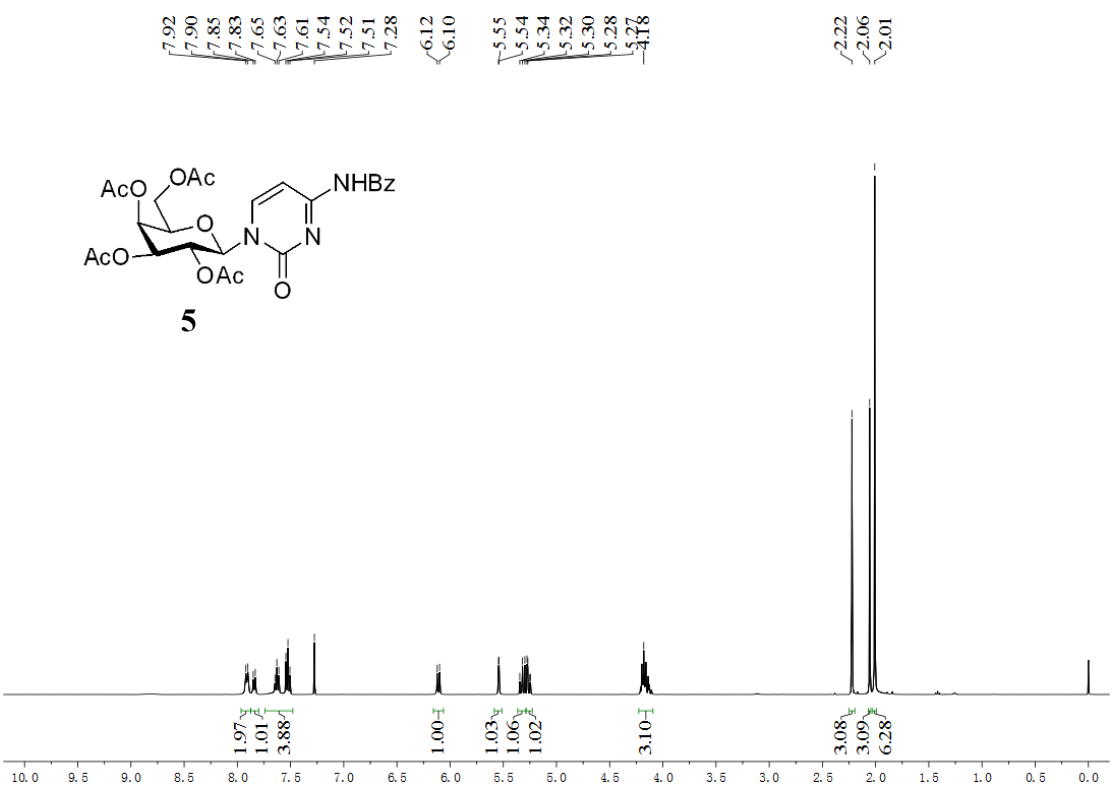
4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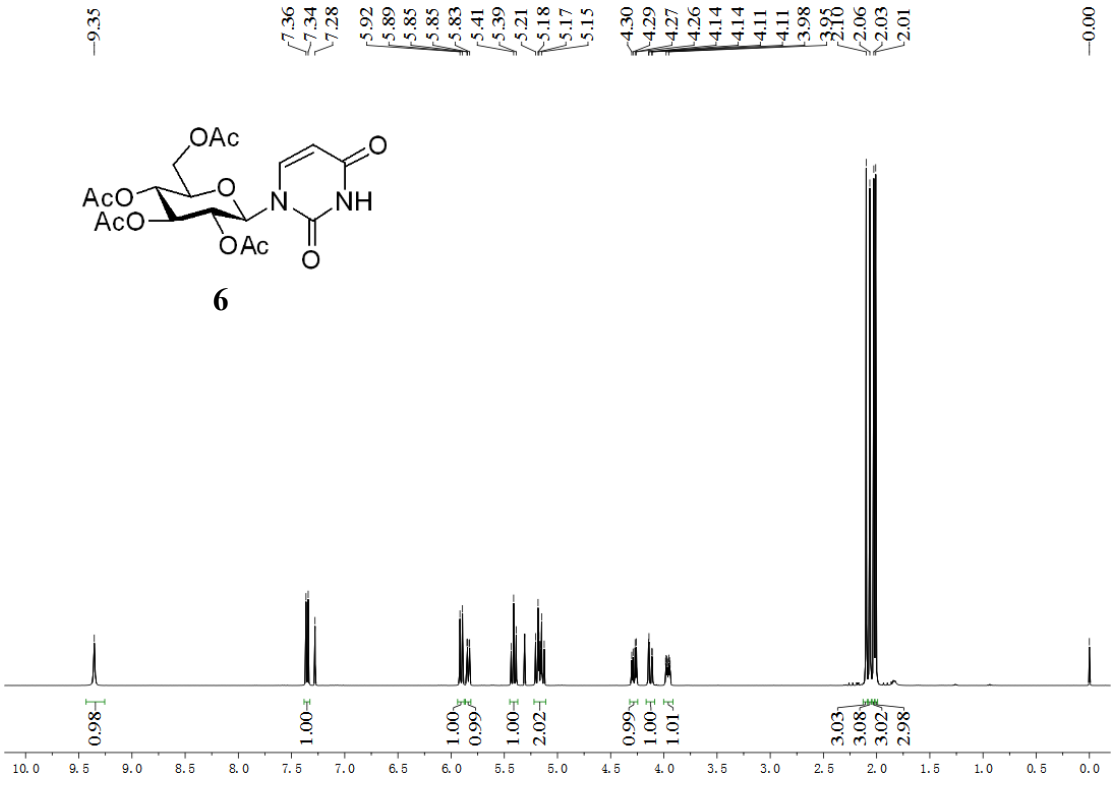
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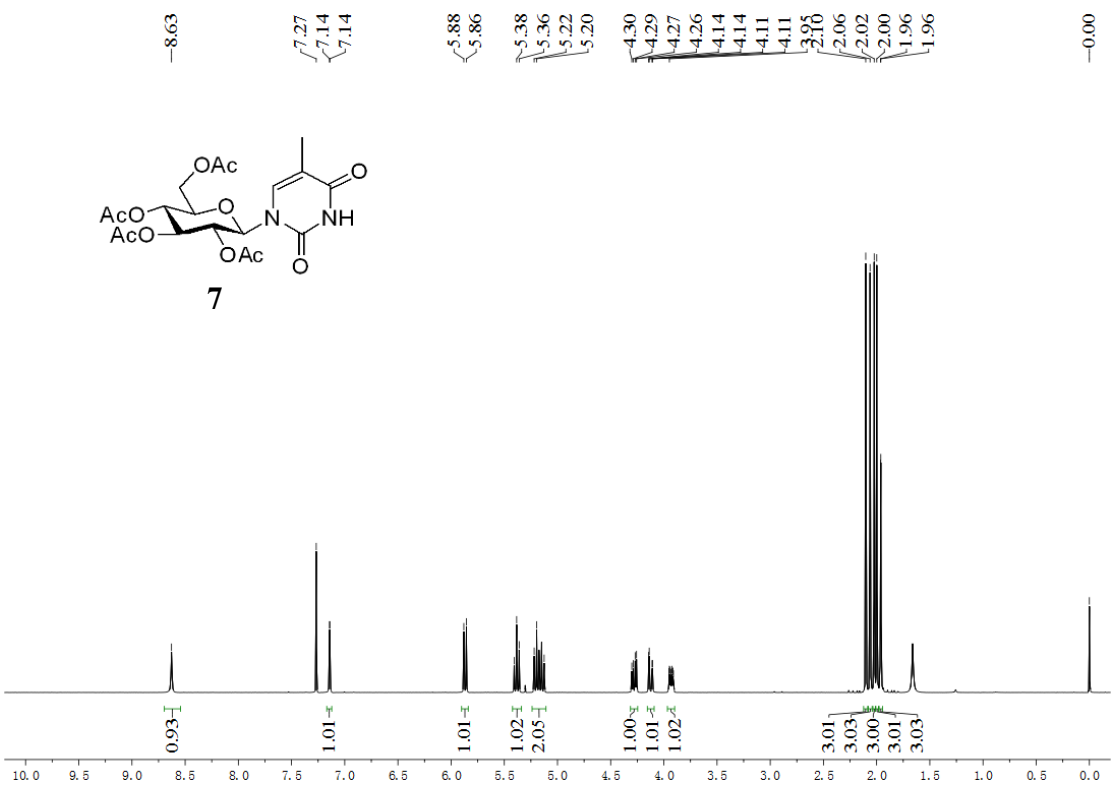
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AcO
AcO
OAc
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NH
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O
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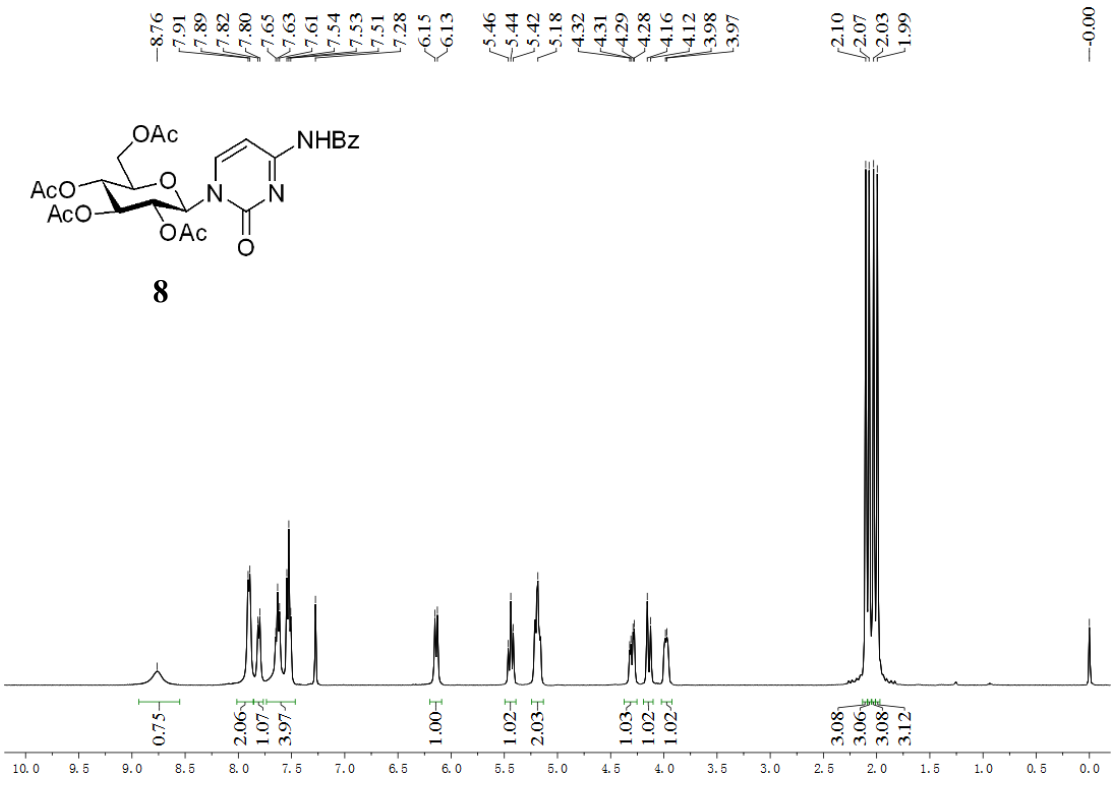
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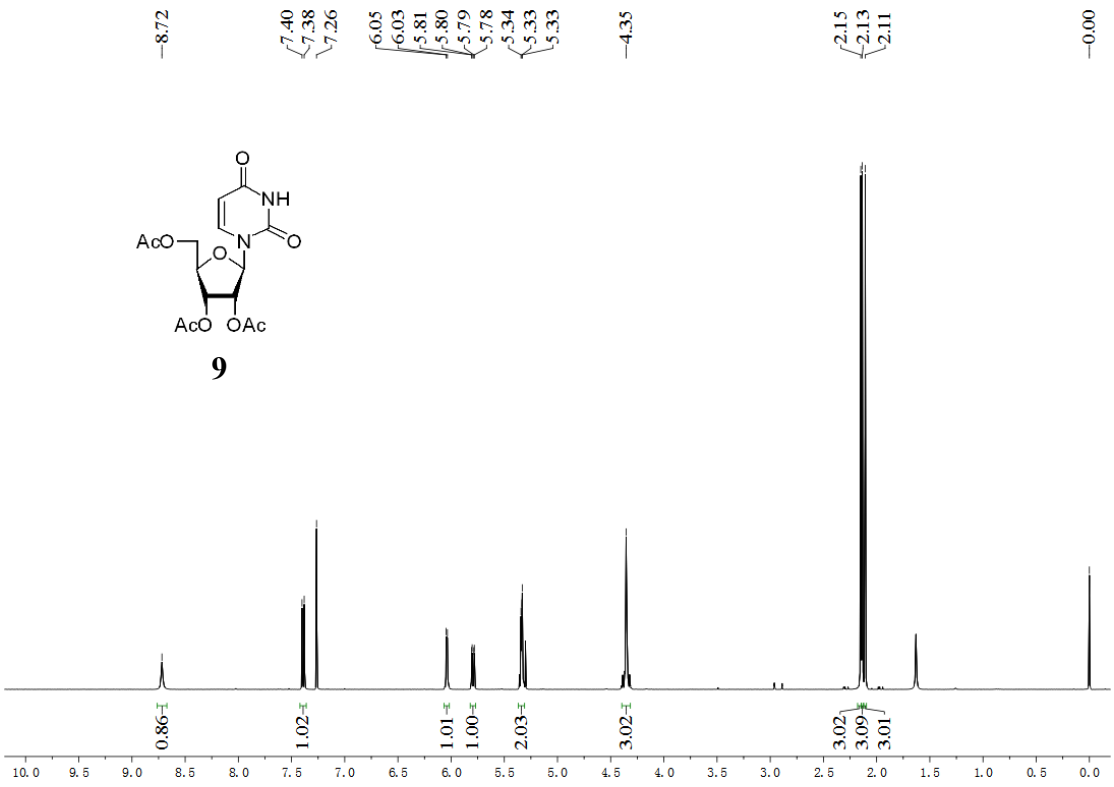
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3.01

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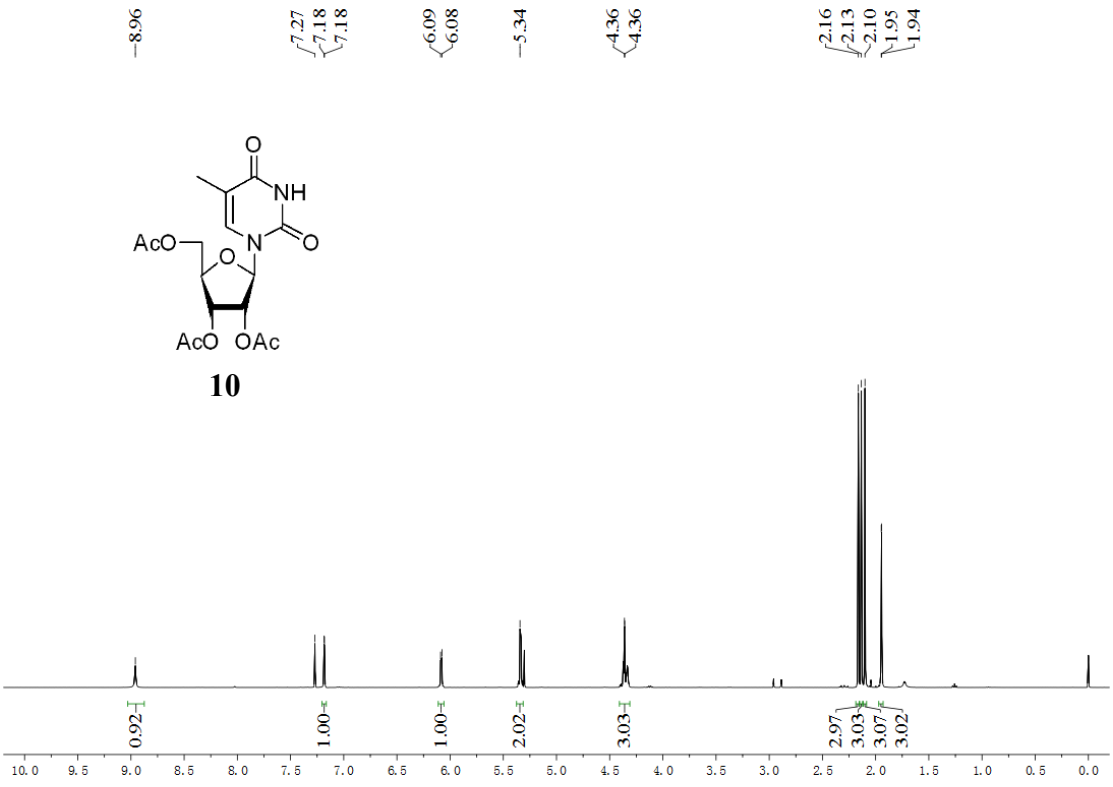
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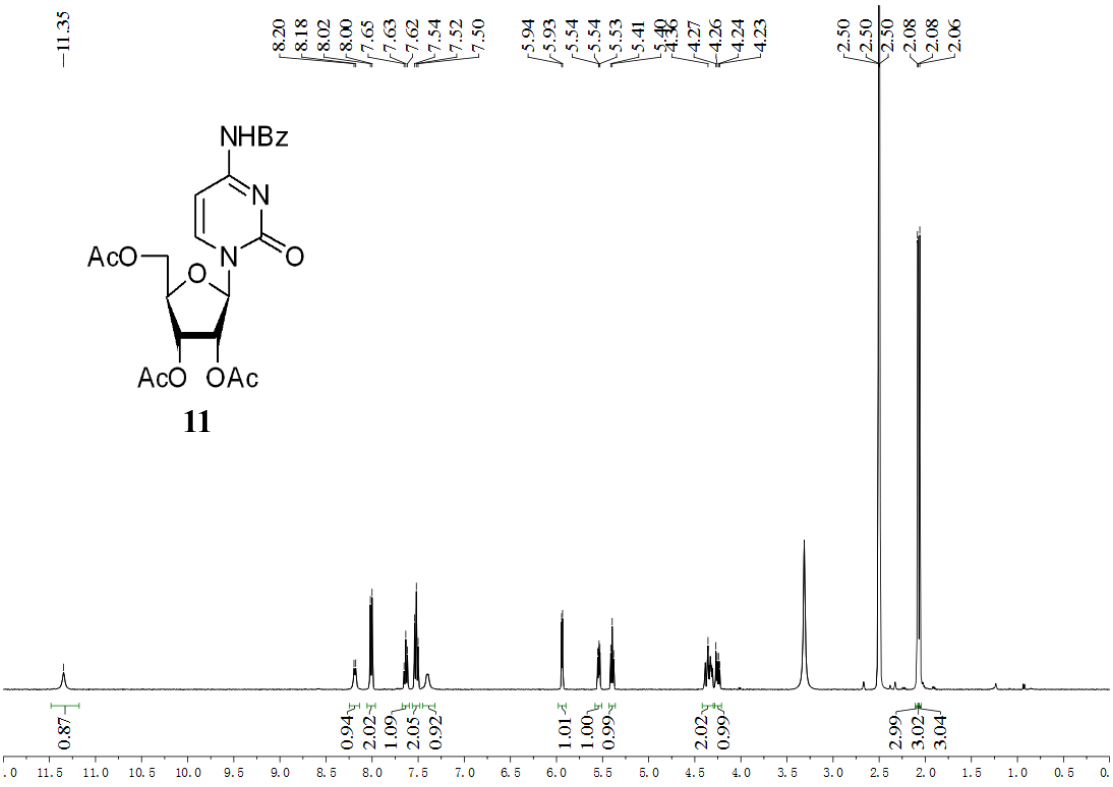
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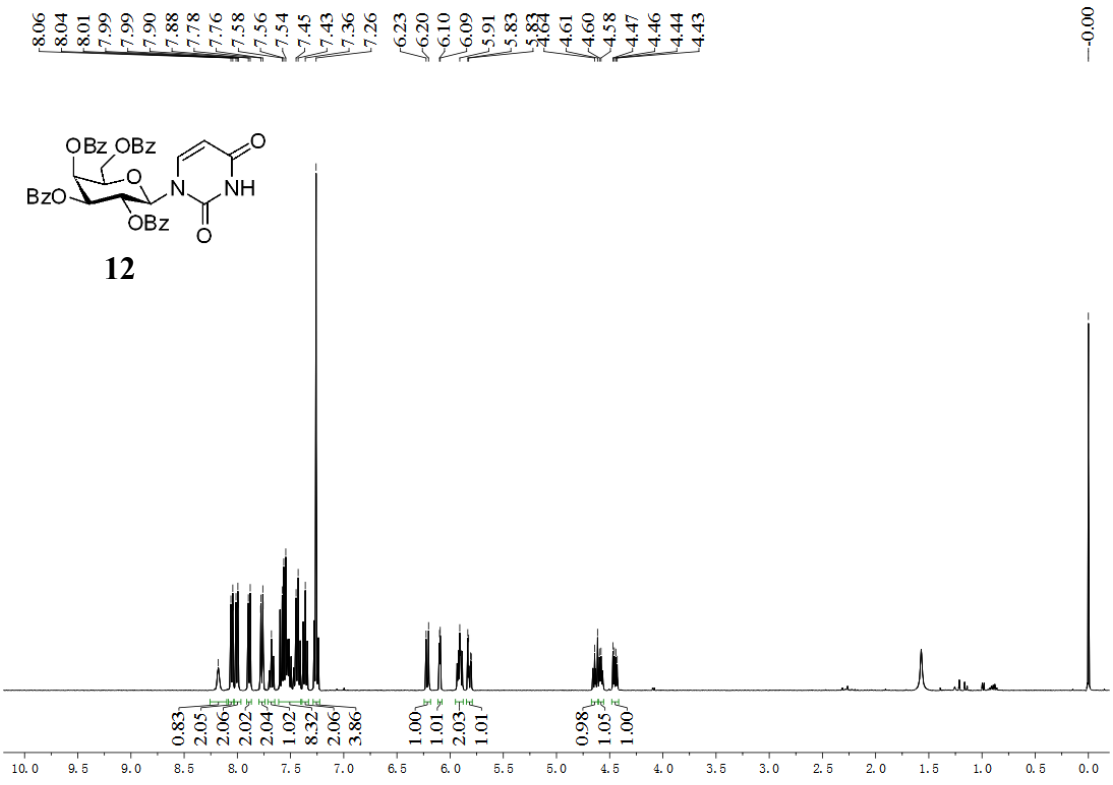
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7.26
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4.44
4.43
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BzO
OBz
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NH
O
O
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2.02
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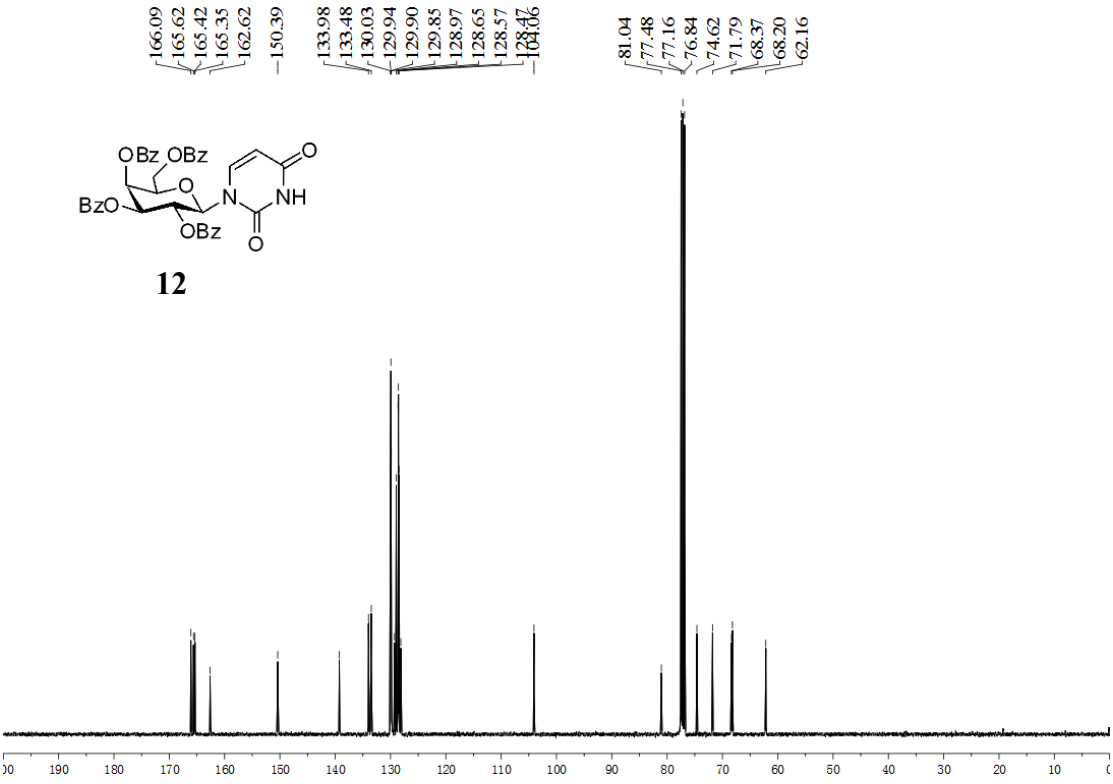
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OBz
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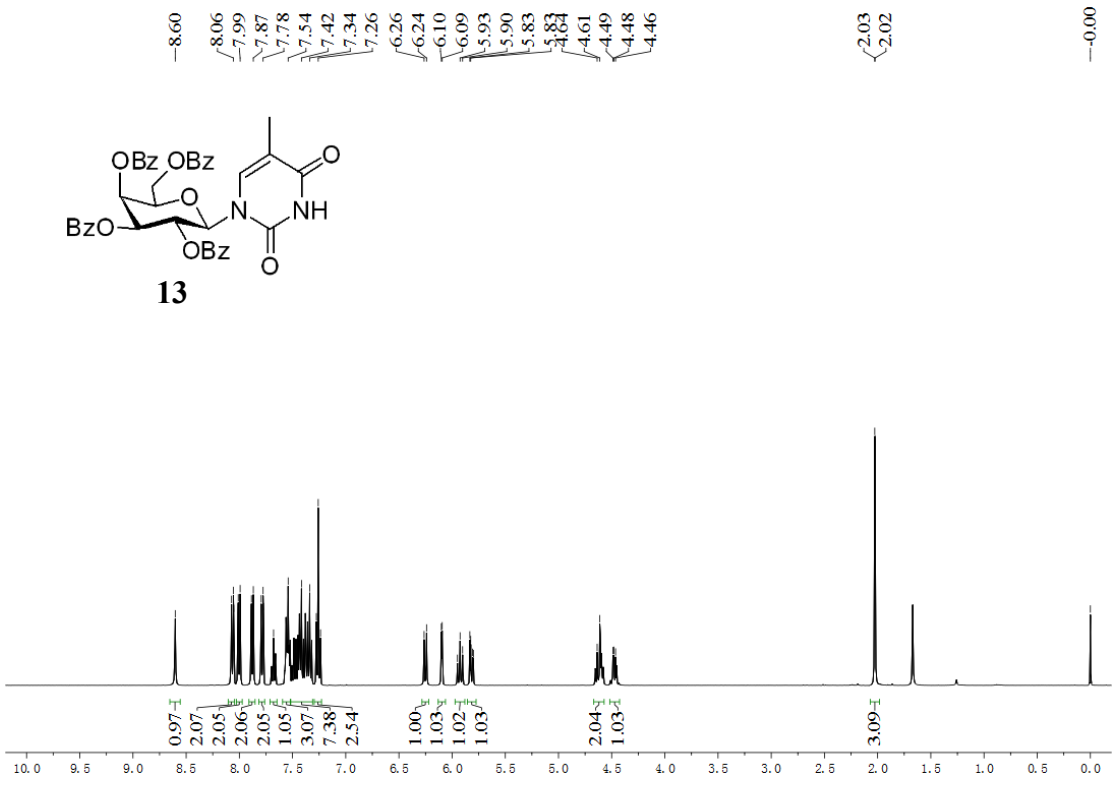
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4.61
4.49
4.48
4.46
2.03
2.02
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OBz
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3.07
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1.02
1.03
2.04
1.03
3.09
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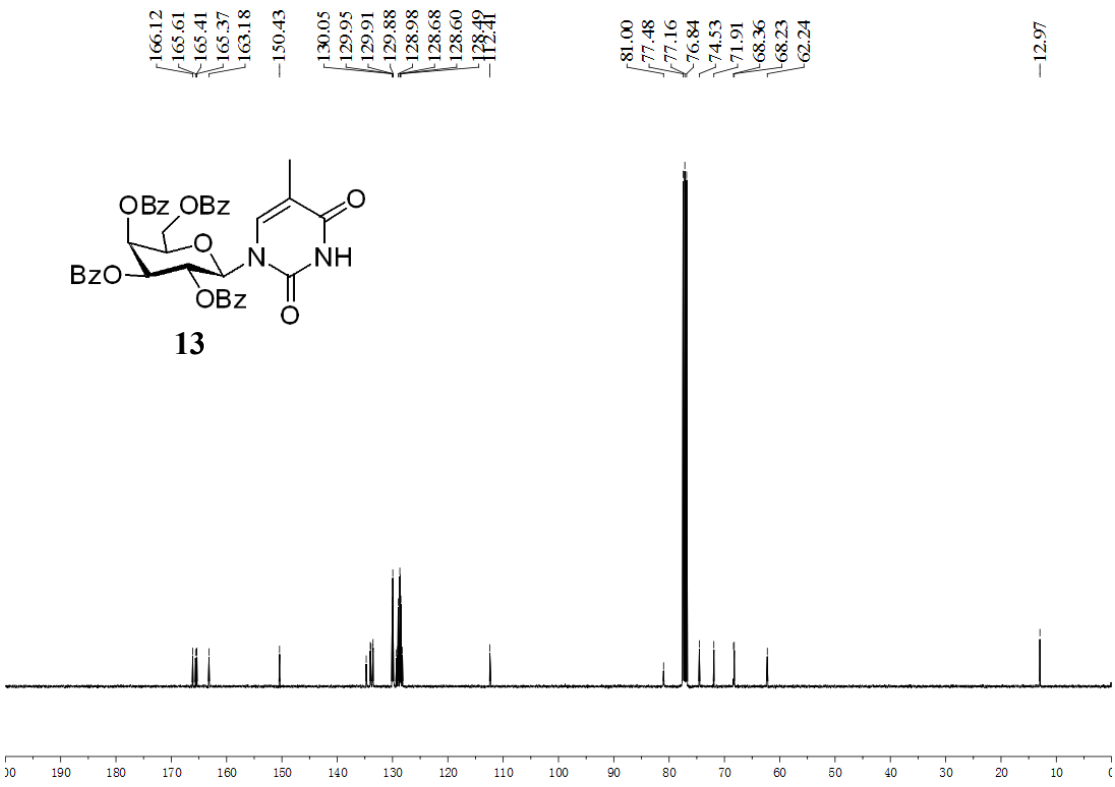
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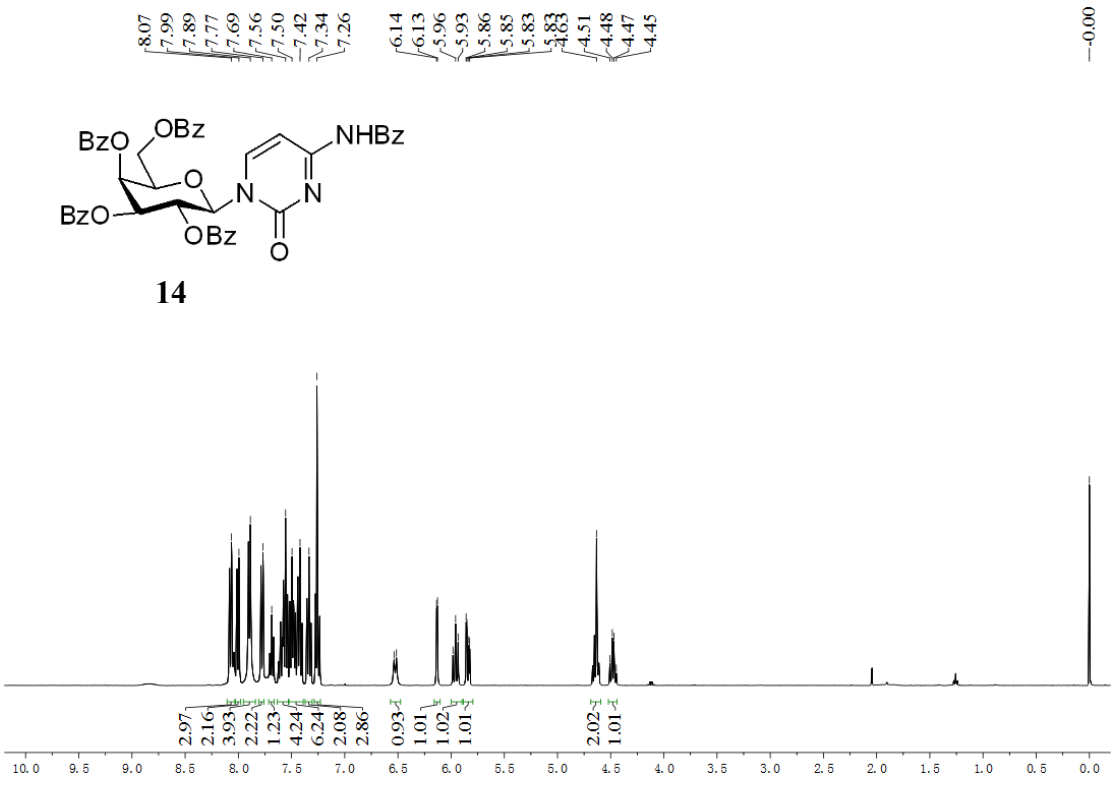
13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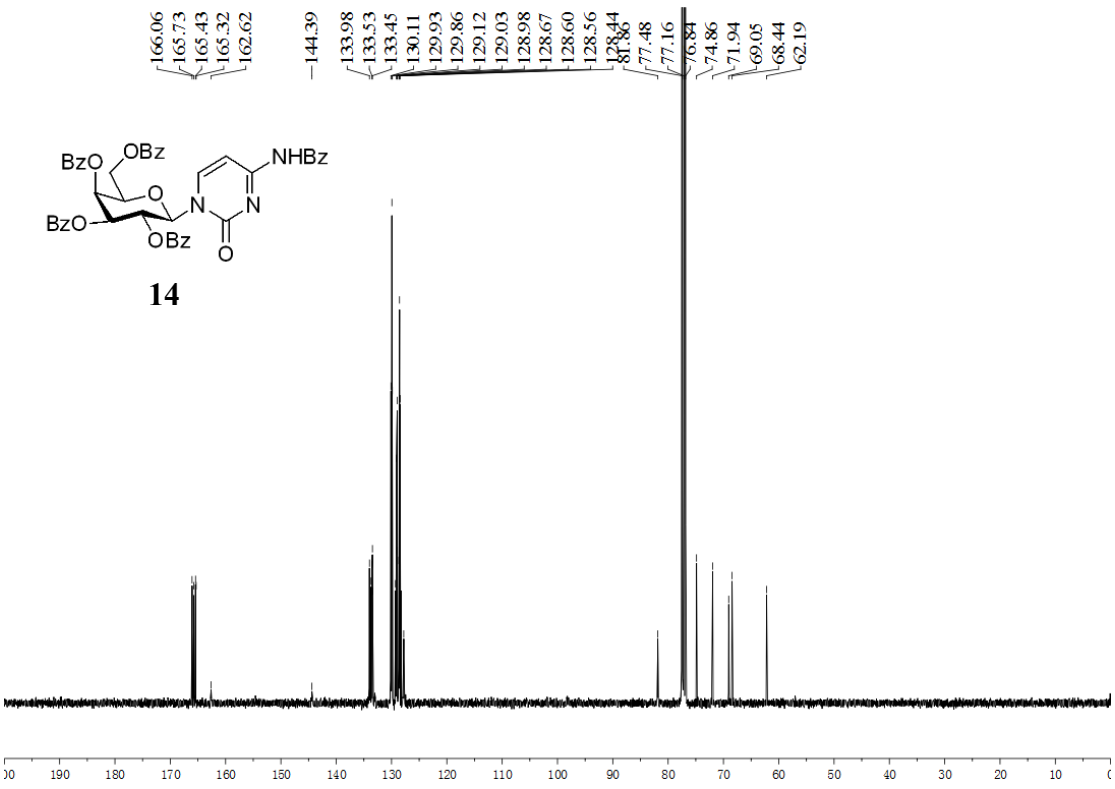
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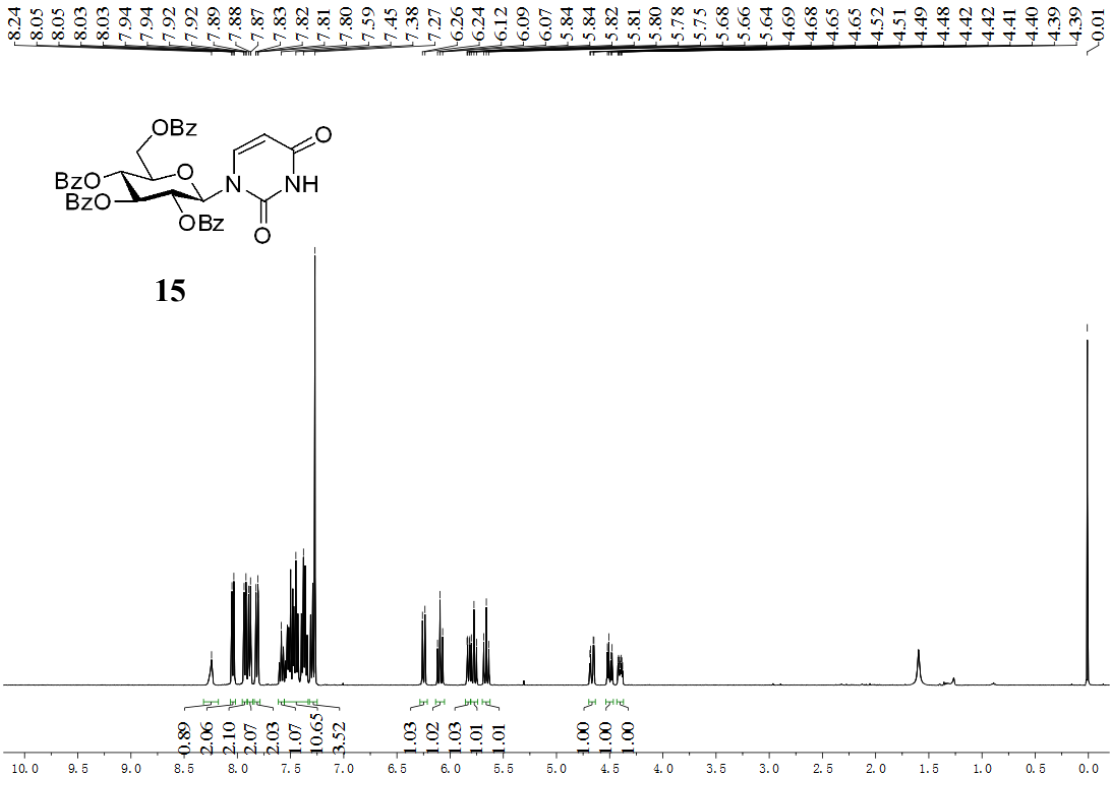
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BzO
OBz
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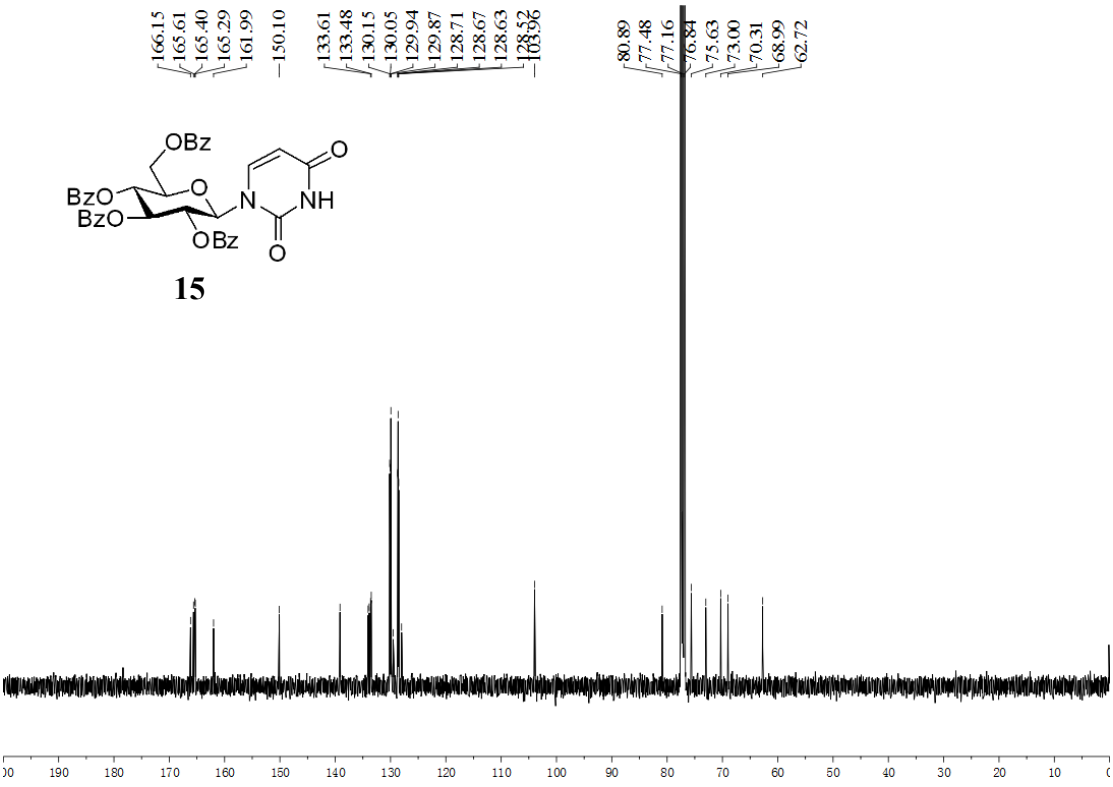
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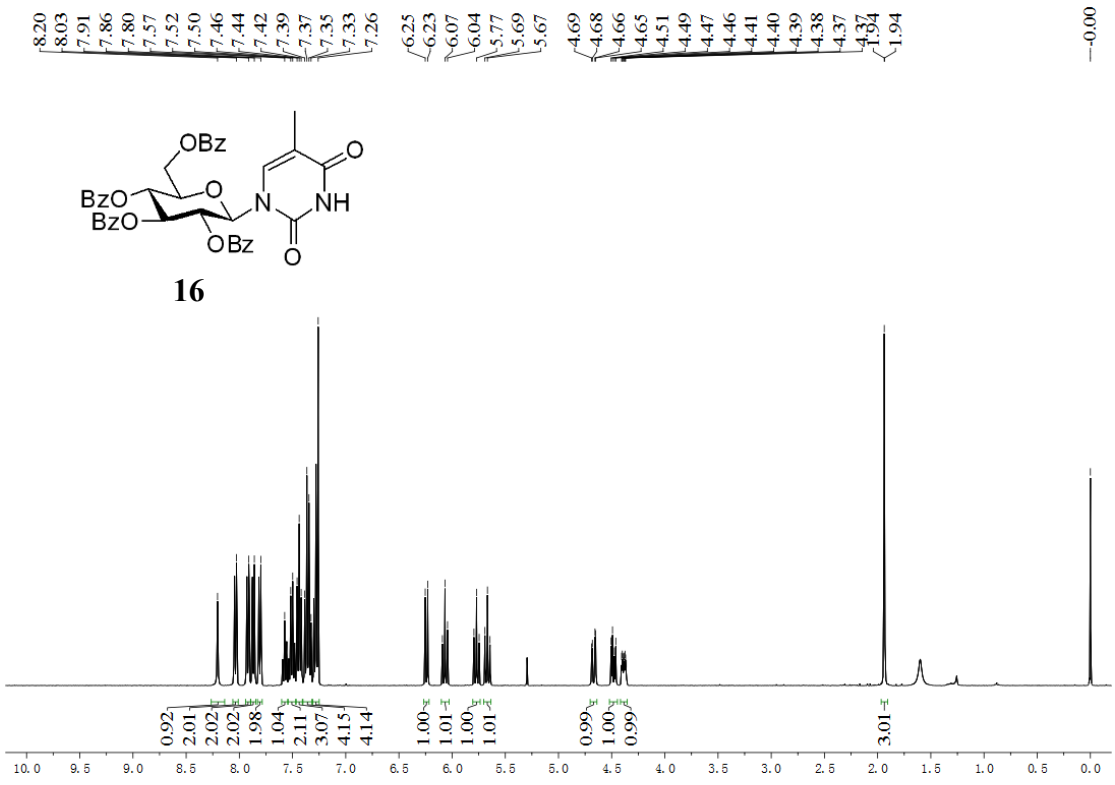
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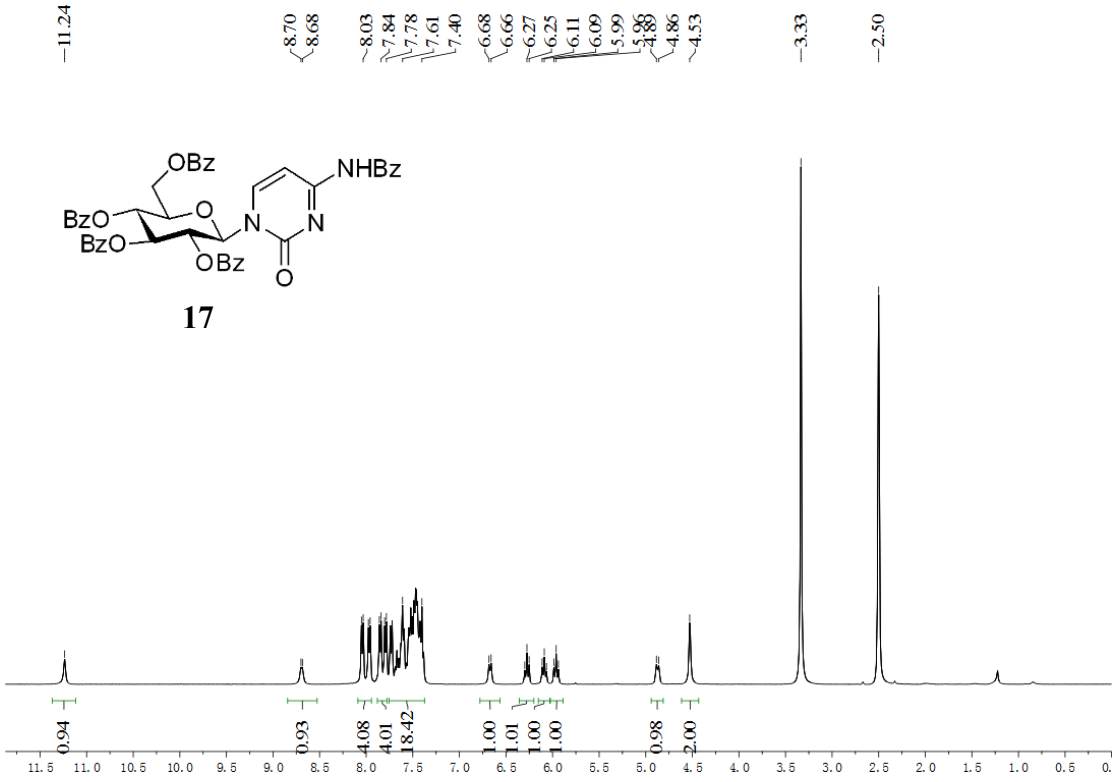
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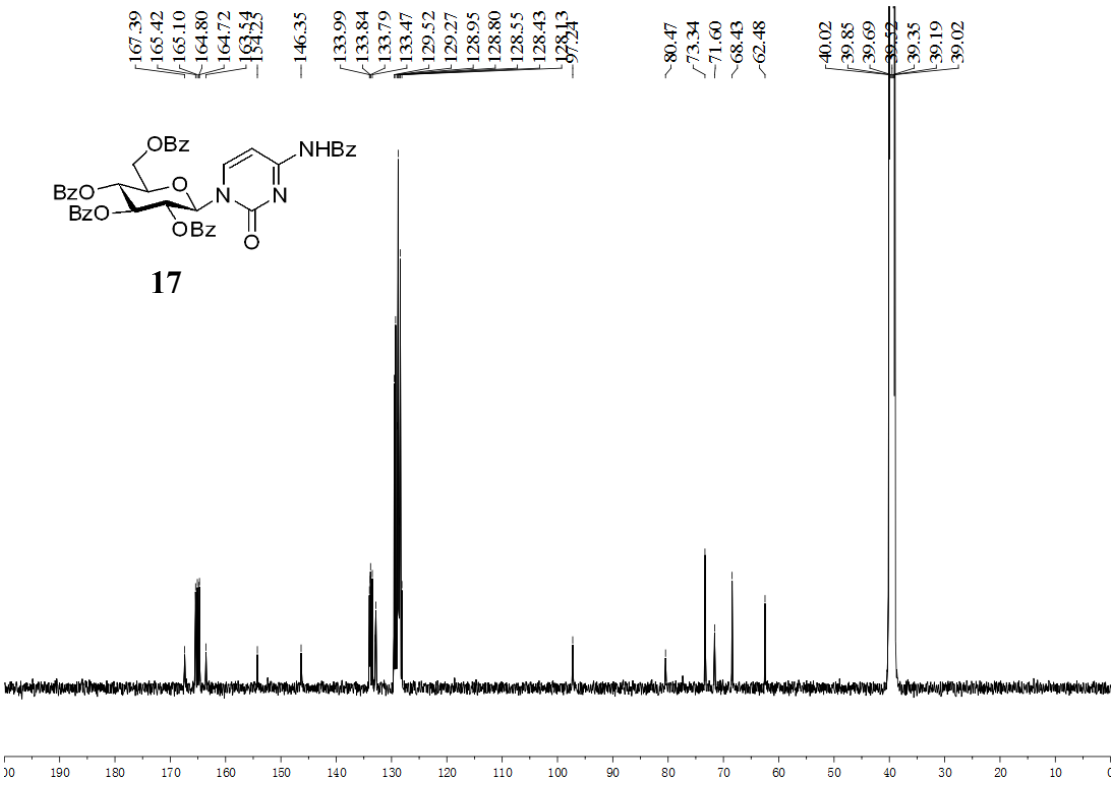
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O

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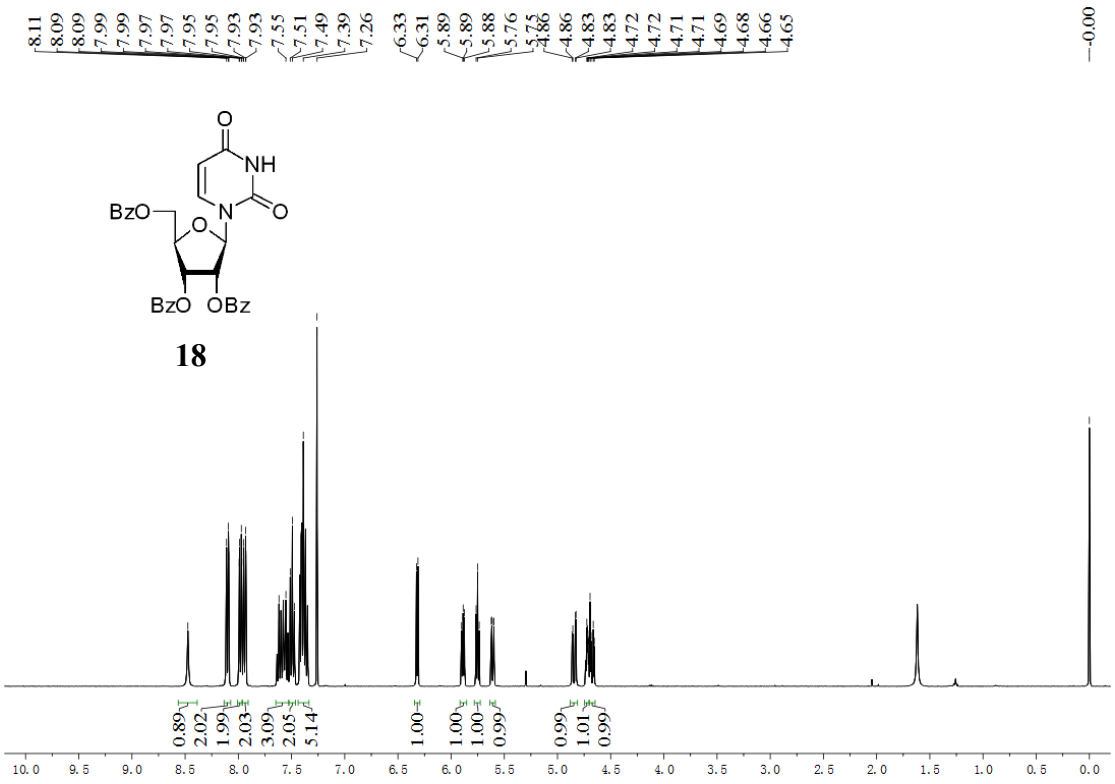
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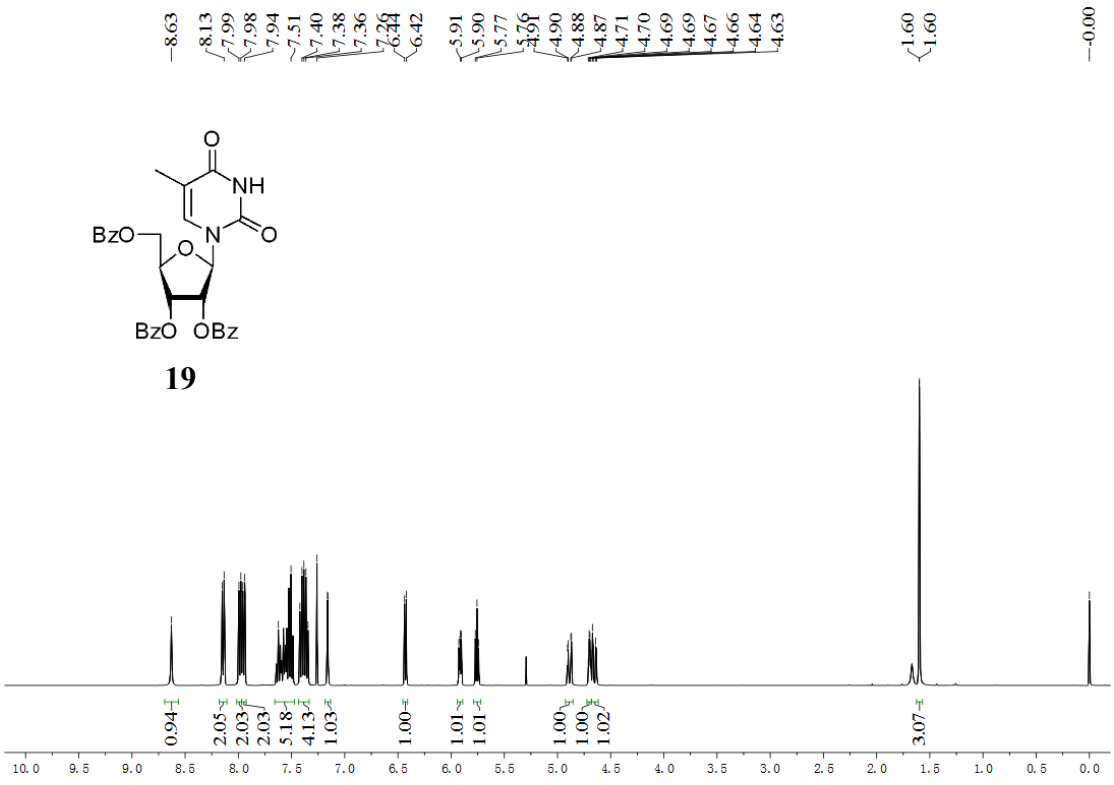
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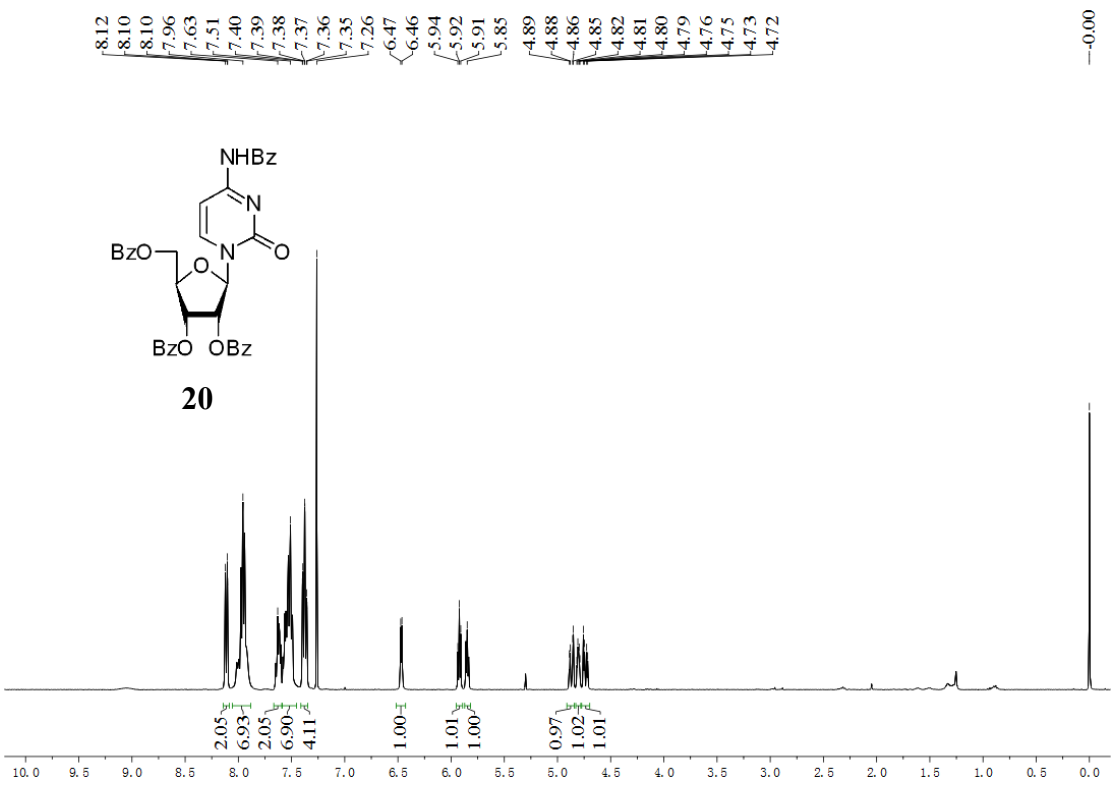
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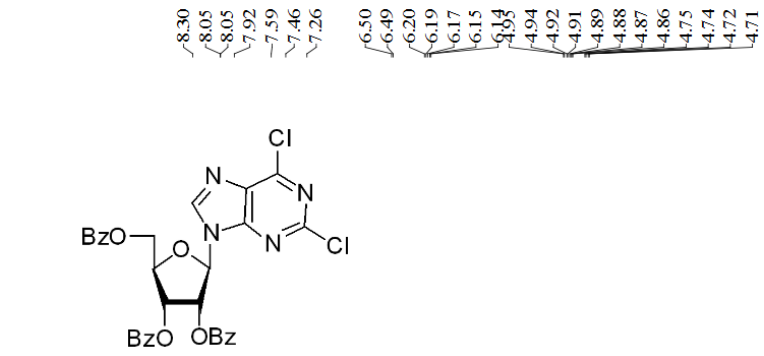
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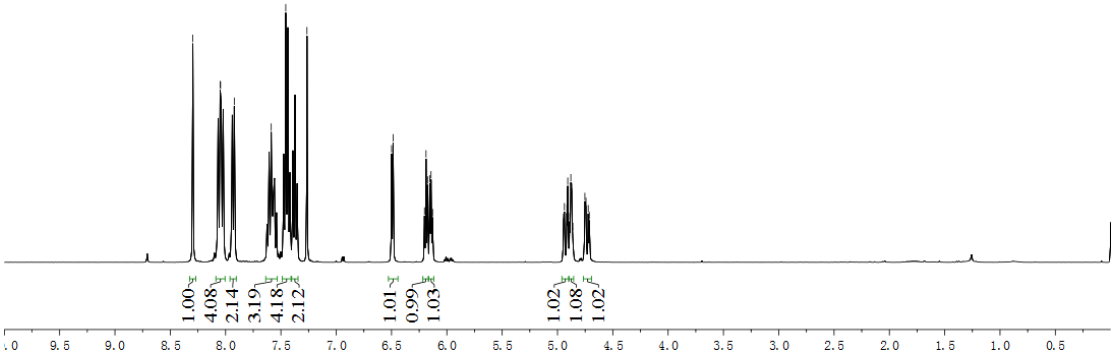
OBz

21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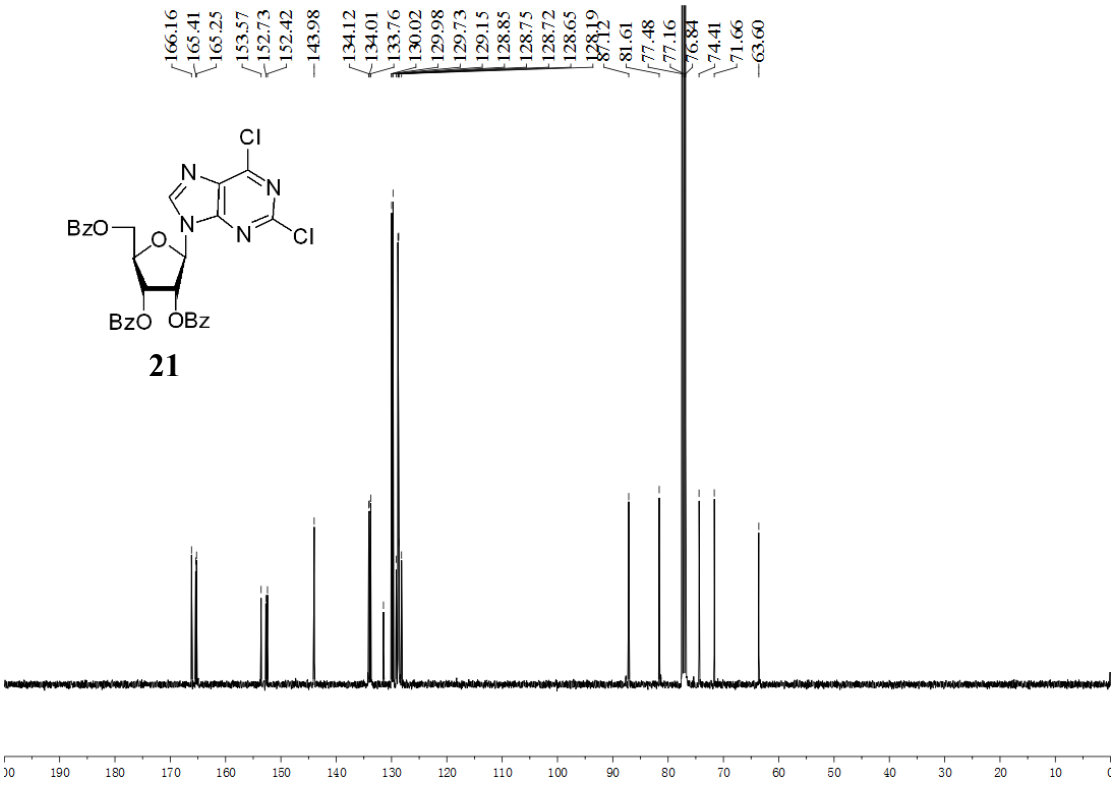
4.08
2.14
3.19
4.18
2.12
1.01
0.99
1.03
1.02
1.08
1.02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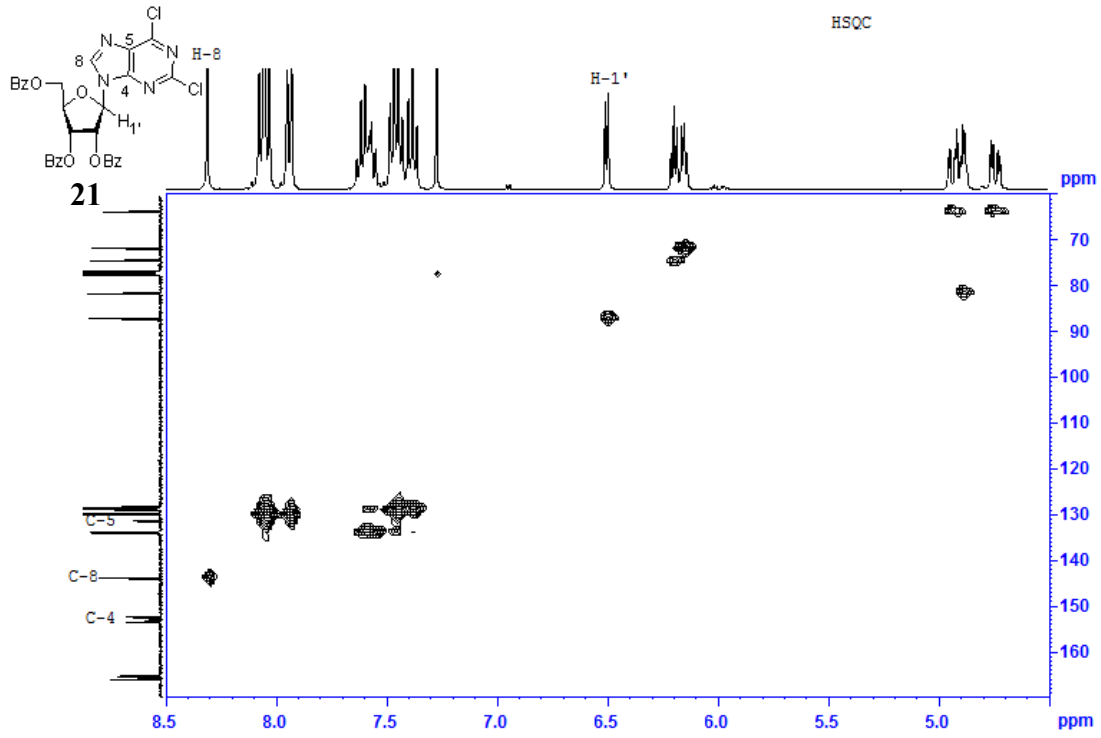
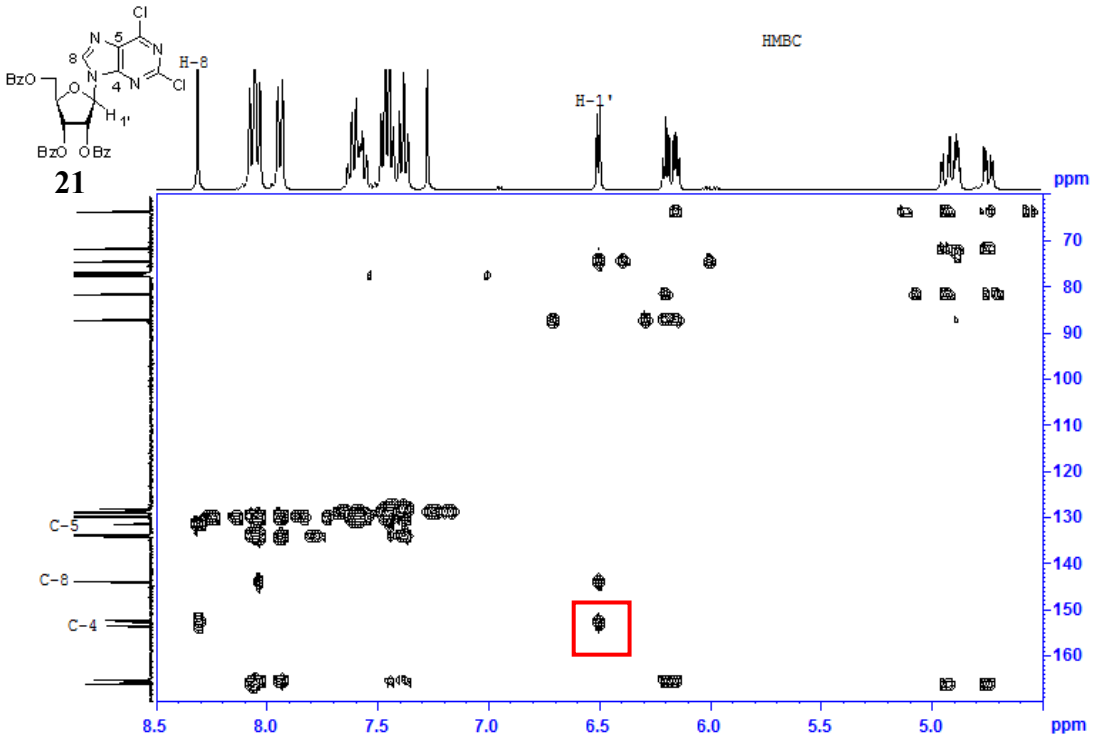
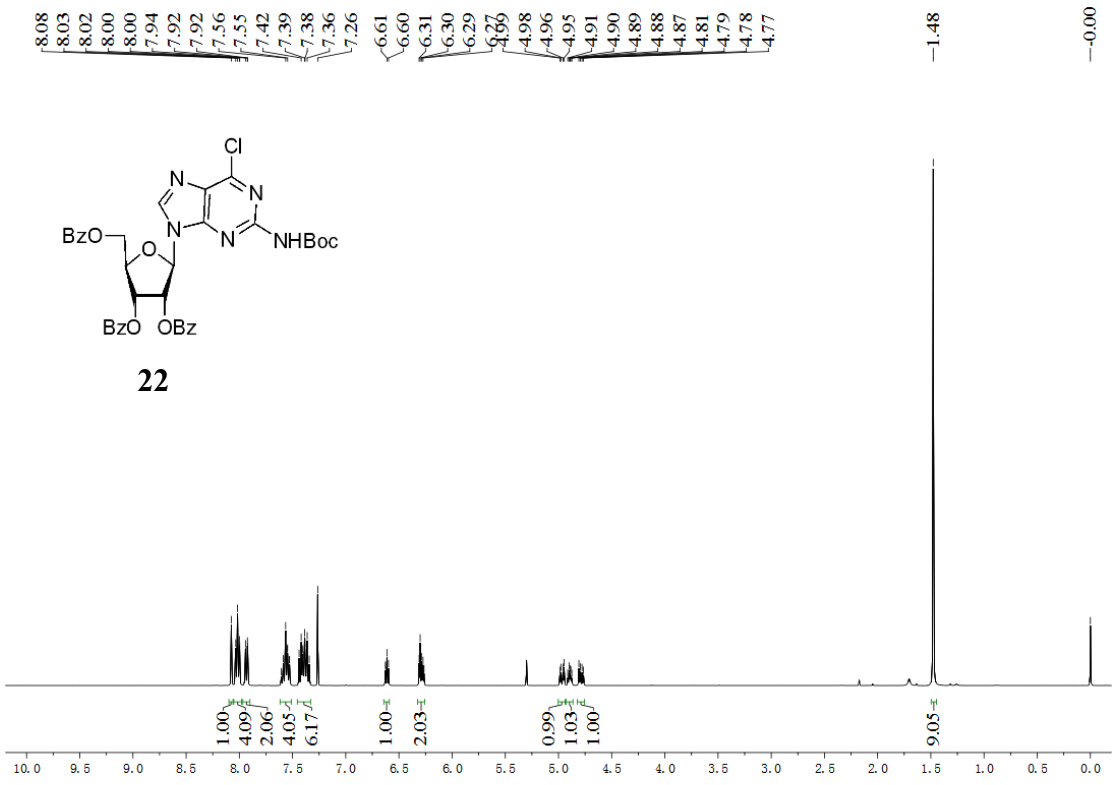
165.25
153.57
152.73
152.42
143.98
134.12
134.01
133.76
130.02
129.98
129.73
129.15
128.85
128.75
128.72
128.65
128.19
87.12
81.61
77.48
77.16
76.84
74.41
71.66
63.60
Cl
N
N
N
N
Cl
BzO
O
BzO
OBz

21


HSQC
H-8
H-1'
BzO
BzO
OBz
21
ppm
C-5
C-8
C-4
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
70
80
90
100
110
120
130
140
150
160


HMBC
H-8
H-1'
BzO
BzO
OBz
21
ppm
C-5
C-8
C-4
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
70
80
90
100
110
120
130
140
150
160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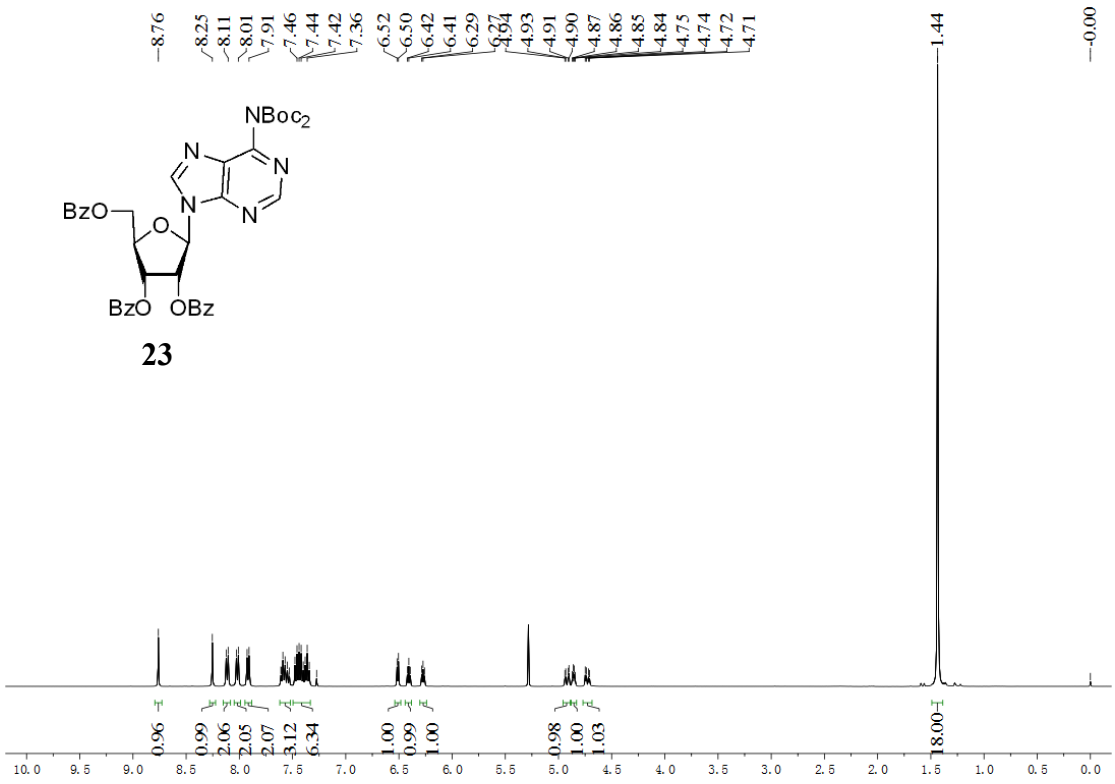
8.03
8.02
8.00
8.00
7.94
7.92
7.92
7.56
7.55
7.42
7.39
7.38
7.36
7.26
6.61
6.60
6.31
6.30
6.29
6.27
4.98
4.96
4.95
4.91
4.90
4.89
4.88
4.87
4.81
4.79
4.78
4.77
1.48
0.00
Cl
N
N
N
N
NHBoc
BzO
O
BzO
OBz
1.00
4.09
2.06
4.05
6.17
1.00
2.03
0.99
1.03
1.00
9.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

22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8.25
8.11
8.01
7.91
7.46
7.44
7.42
7.36
6.52
6.50
6.42
6.41
6.29
6.27
4.94
4.93
4.91
4.90
4.87
4.86
4.85
4.84
4.75
4.74
4.72
4.71
1.44
0.00
NBoc2
N
N
N
N
BzO
O
BzO
OBz
0.96
0.99
2.06
2.05
2.07
3.12
6.34
1.00
0.99
1.00
0.98
1.00
1.03
18.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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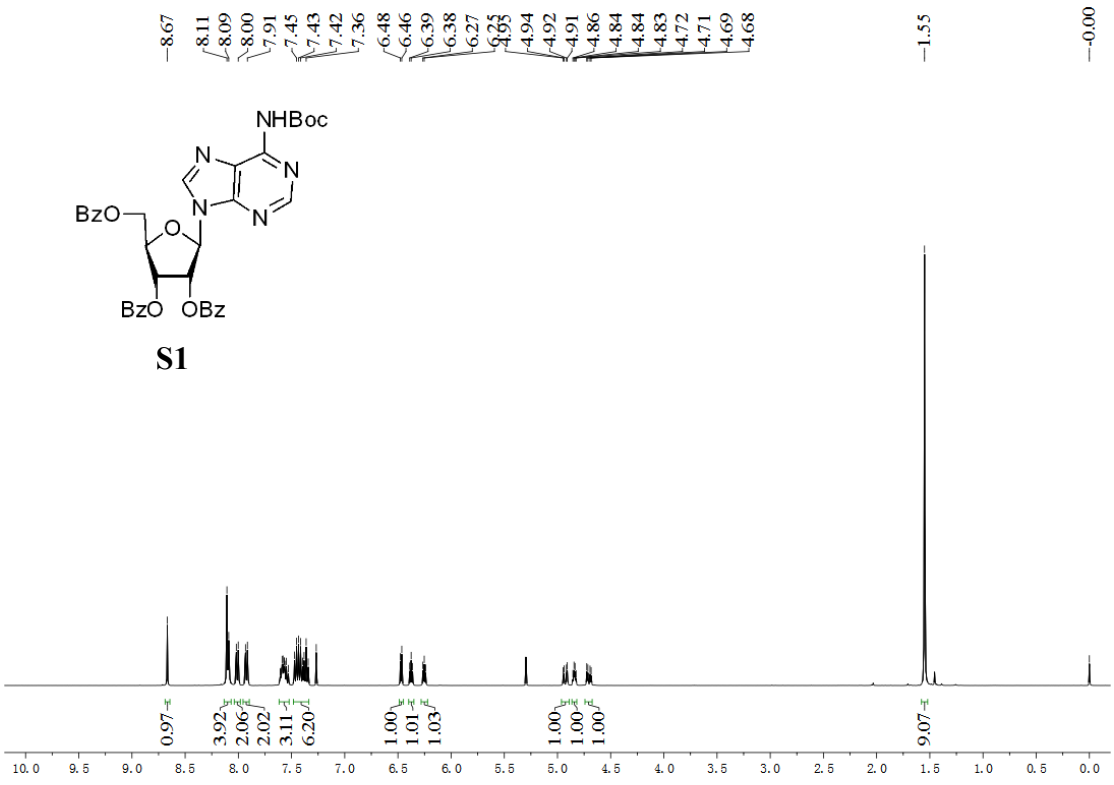
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

23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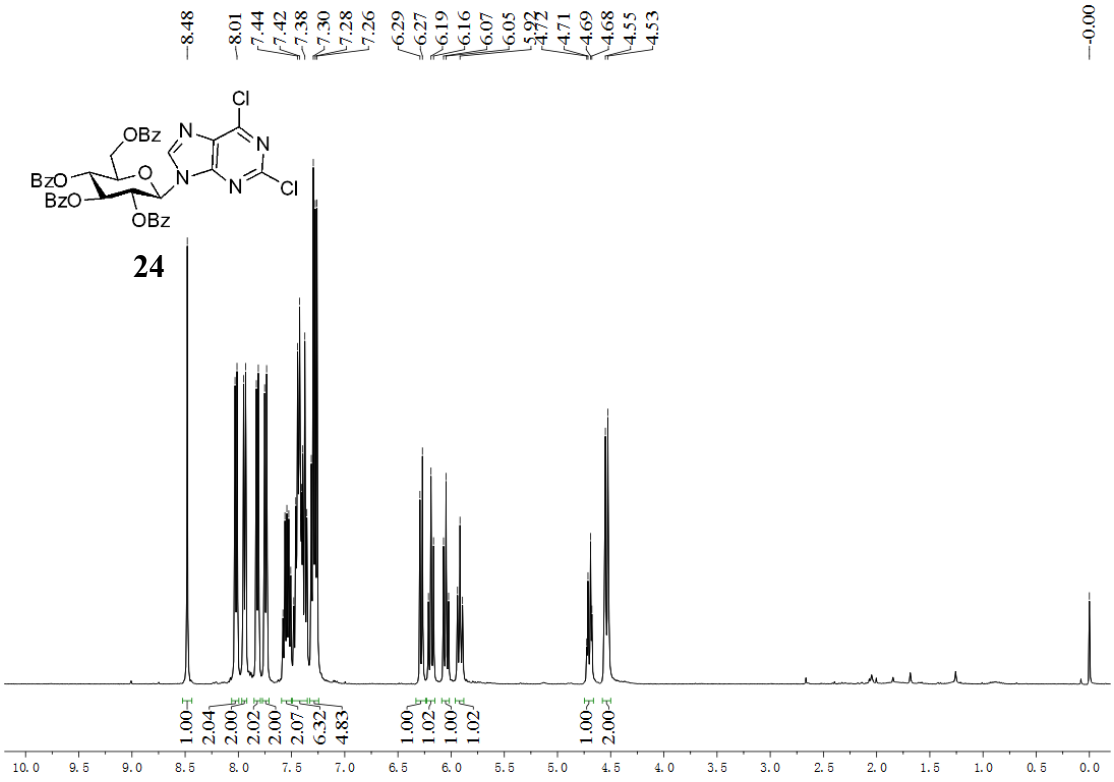
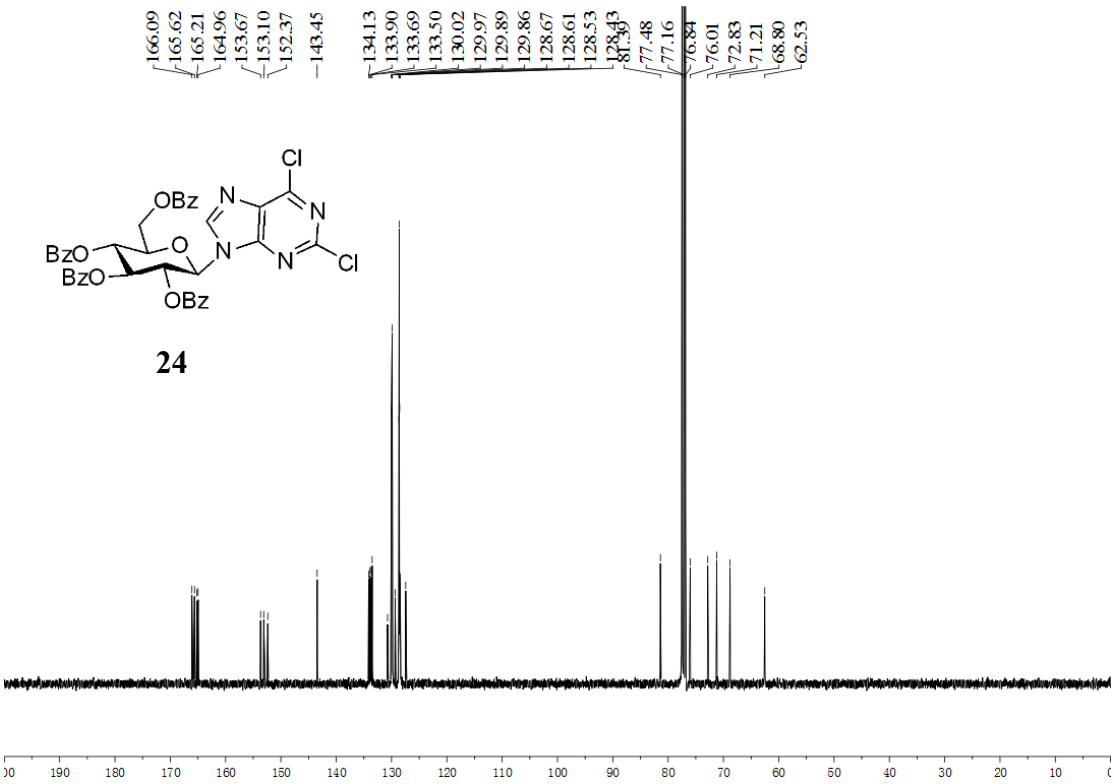
8.11
8.09
8.00
7.91
7.45
7.43
7.42
7.36
6.48
6.46
6.39
6.38
6.27
6.25
4.94
4.92
4.91
4.86
4.84
4.84
4.83
4.72
4.71
4.69
4.68
1.55
0.00
NHBoc
BzO
BzO
OBz
0.97
3.92
2.06
2.02
3.11
6.20
1.00
1.01
1.03
1.00
1.00
1.00
9.07

S1


8.48
8.01
7.44
7.42
7.38
7.30
7.28
7.26
6.29
6.27
6.19
6.16
6.07
6.05
5.92
4.72
4.71
4.69
4.68
4.55
4.53
0.00
Cl
OBz
BzO
BzO
OBz
Cl
1.00
2.04
2.00
2.02
2.00
2.07
6.32
4.83
1.00
1.02
1.00
1.02
1.00
2.00

24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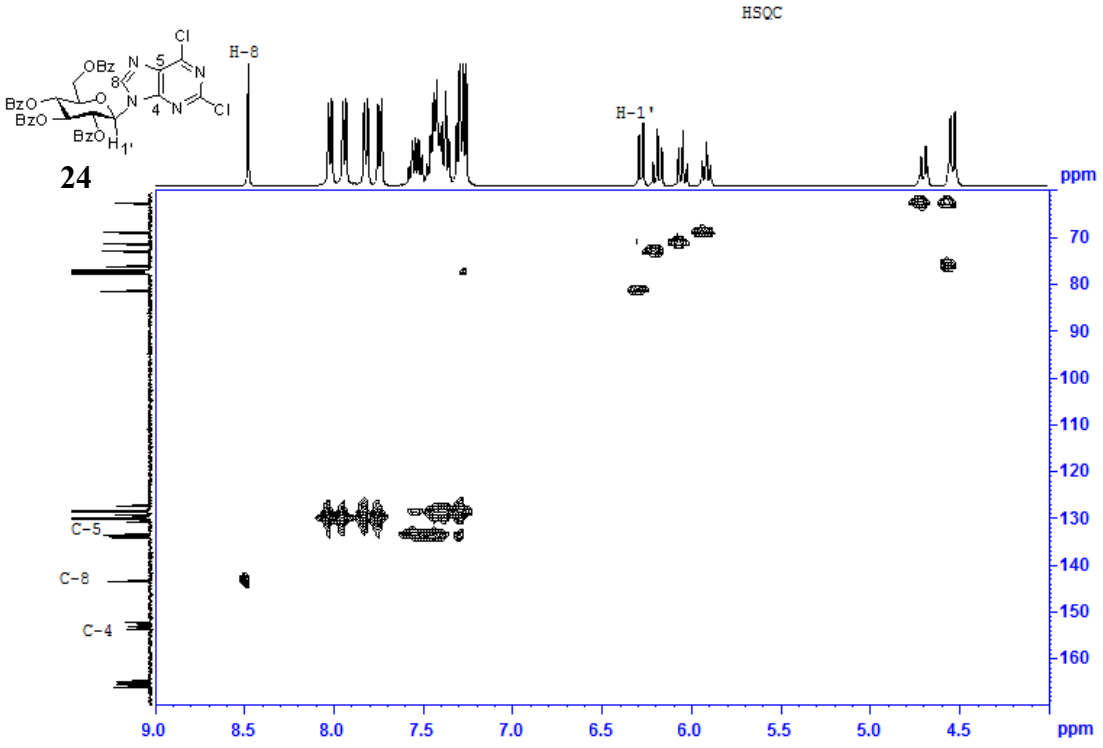
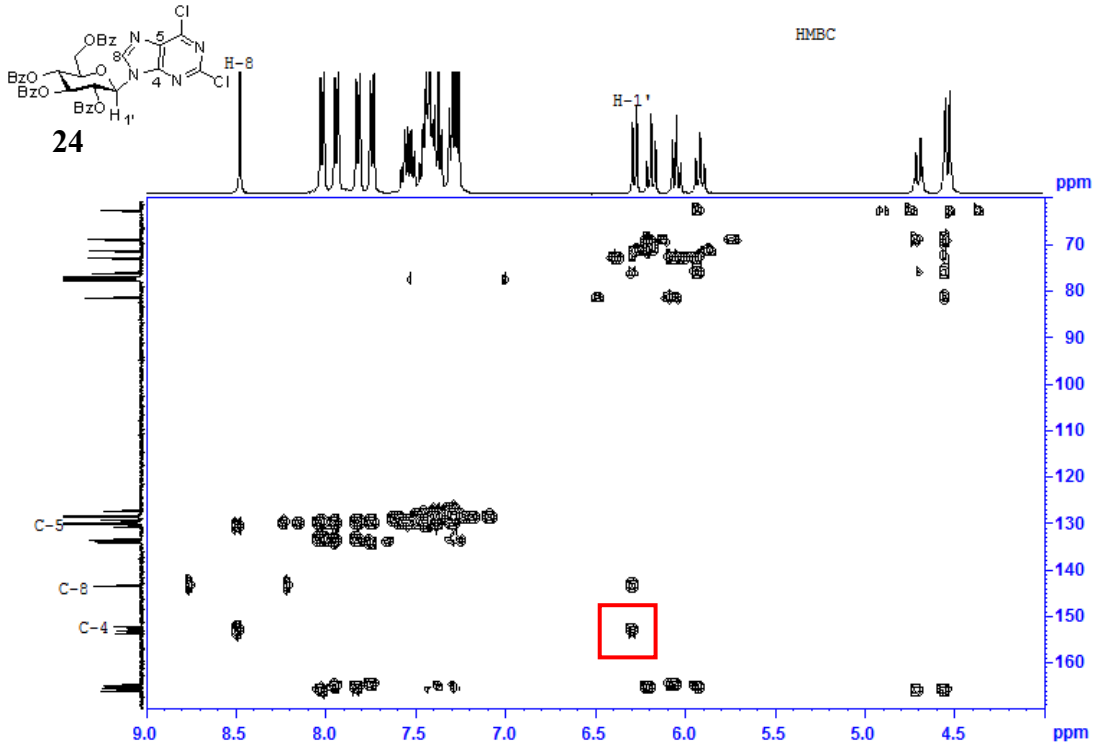
165.62
165.21
164.96
153.67
153.10
152.37
143.45
134.13
133.90
133.69
133.50
130.02
129.97
129.89
129.86
128.67
128.61
128.53
128.43
81.30
77.48
77.16
76.84
76.01
72.83
71.21
68.80
62.53
Cl
OBz
N
N
BzO
O
N
N
Cl
BzO
OBz

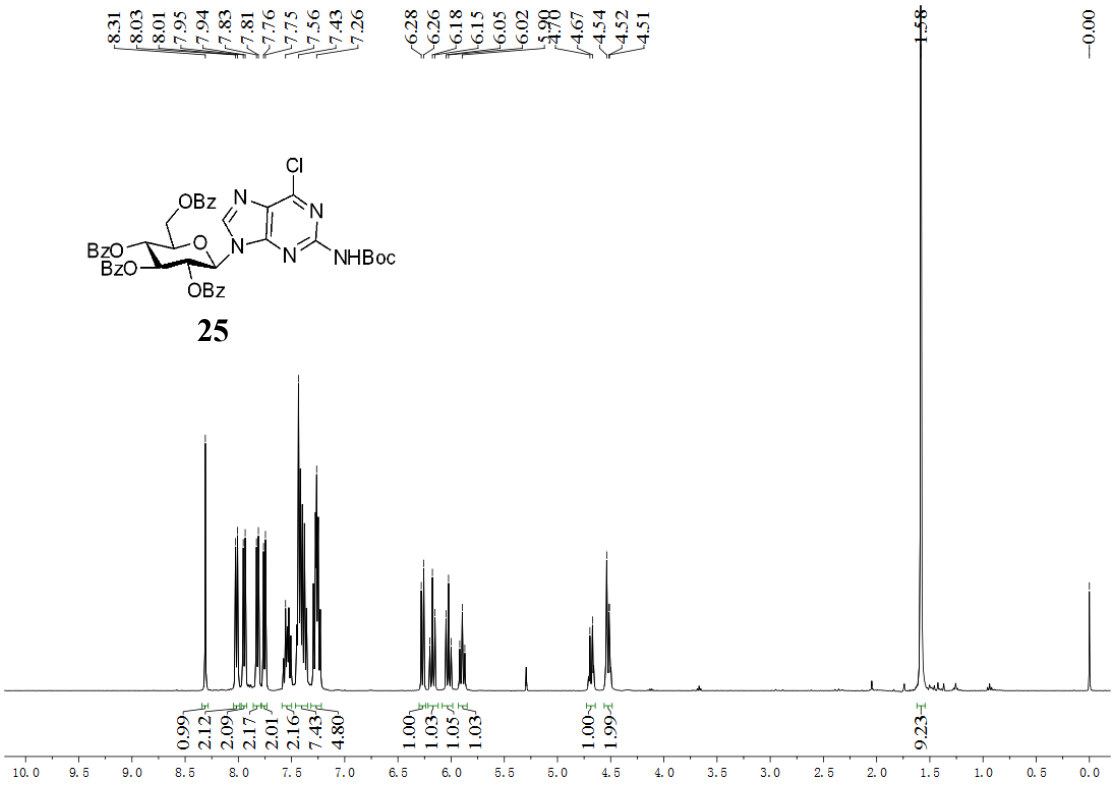
24


HSQC
Cl
OBz
N
5
N
H-8
8
BzO
O
N
4
N
Cl
BzO
H1'
H-1'
ppm
70
80
90
100
110
120
130
140
150
160
C-5
C-8
C-4
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
ppm

24


HMBC
H-8
H-1'
ppm
C-5
C-8
C-4
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
ppm

24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8.03
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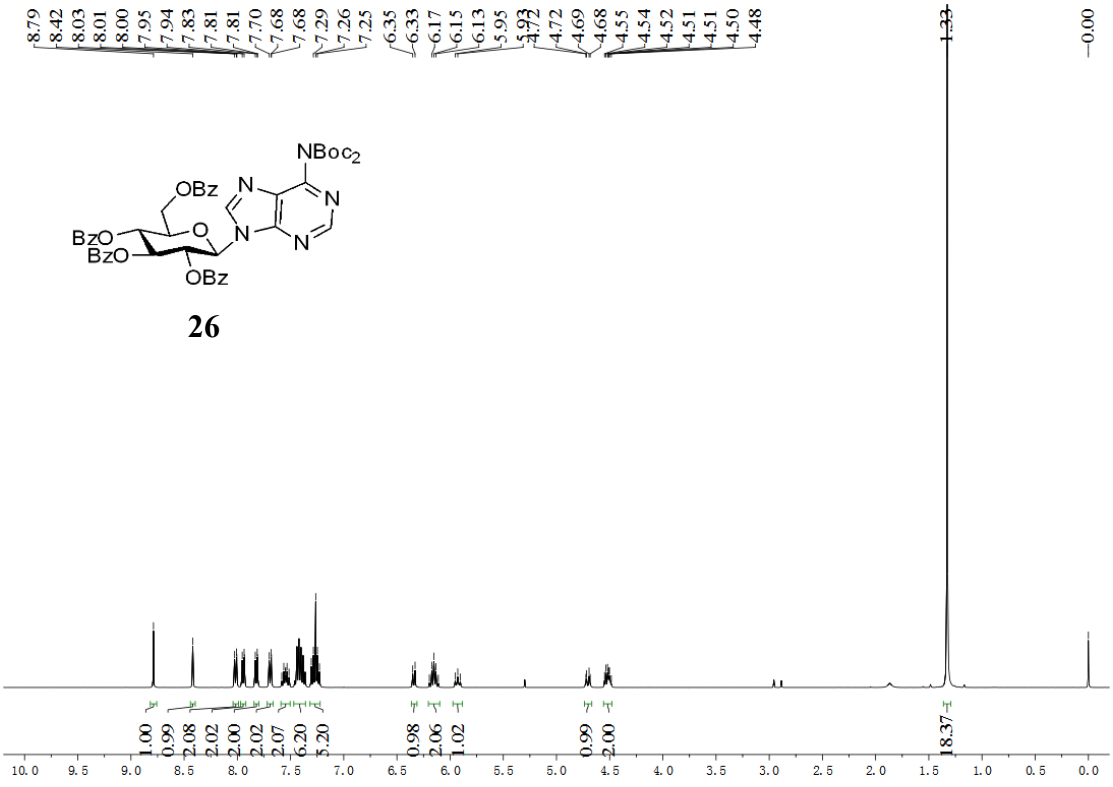
7.94
7.83
7.81
7.76
7.75
7.56
7.43
7.26
6.28
6.26
6.18
6.15
6.05
6.02
5.90
4.70
4.67
4.54
4.52
4.51
1.58
0.00
NHBoc
0.99
2.12
2.09
2.17
2.01
2.16
7.43
4.80
1.00
1.03
1.05
1.03
1.00
1.99
9.23

25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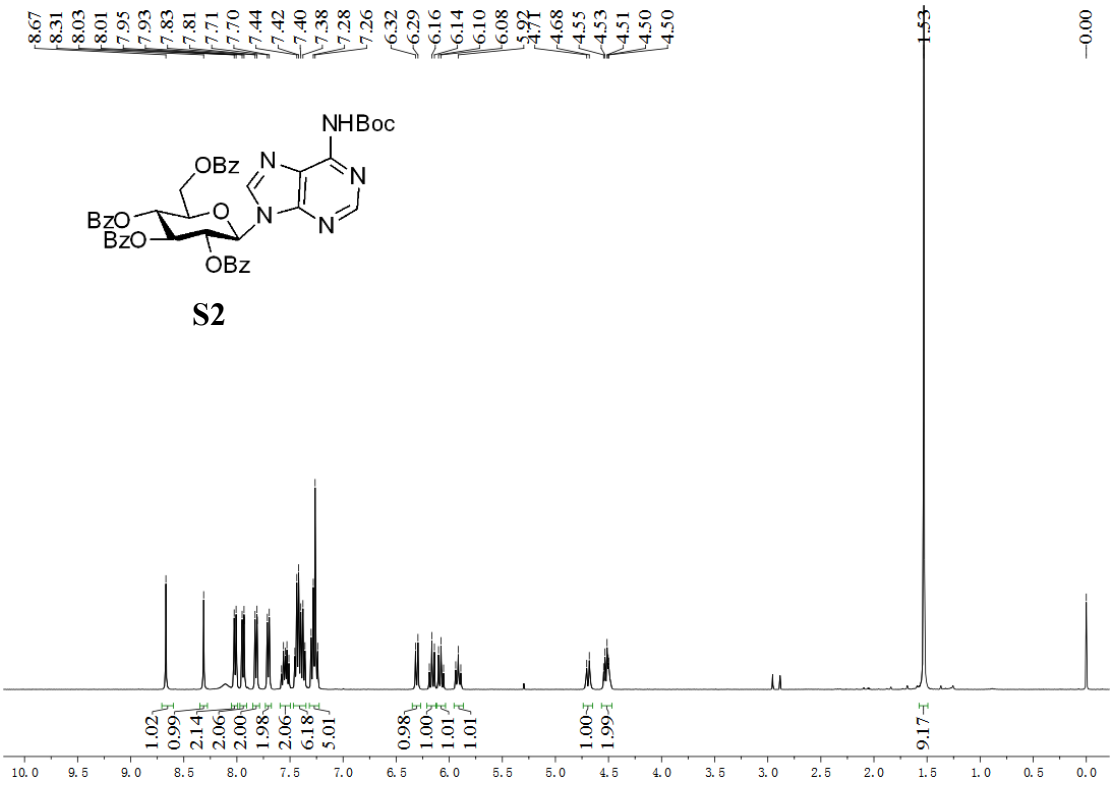
7.94
7.83
7.81
7.81
7.70
7.68
7.68
7.29
7.26
7.25
6.35
6.33
6.17
6.15
6.13
5.95
5.93
4.72
4.69
4.68
4.55
4.54
4.52
4.51
4.51
4.50
4.48
1.32
0.00
NBoc2
OBz
BzO
BzO
OBz
1.00
0.99
2.08
2.02
2.00
2.02
2.07
6.20
5.20
0.98
2.06
1.02
0.99
2.00
18.37

26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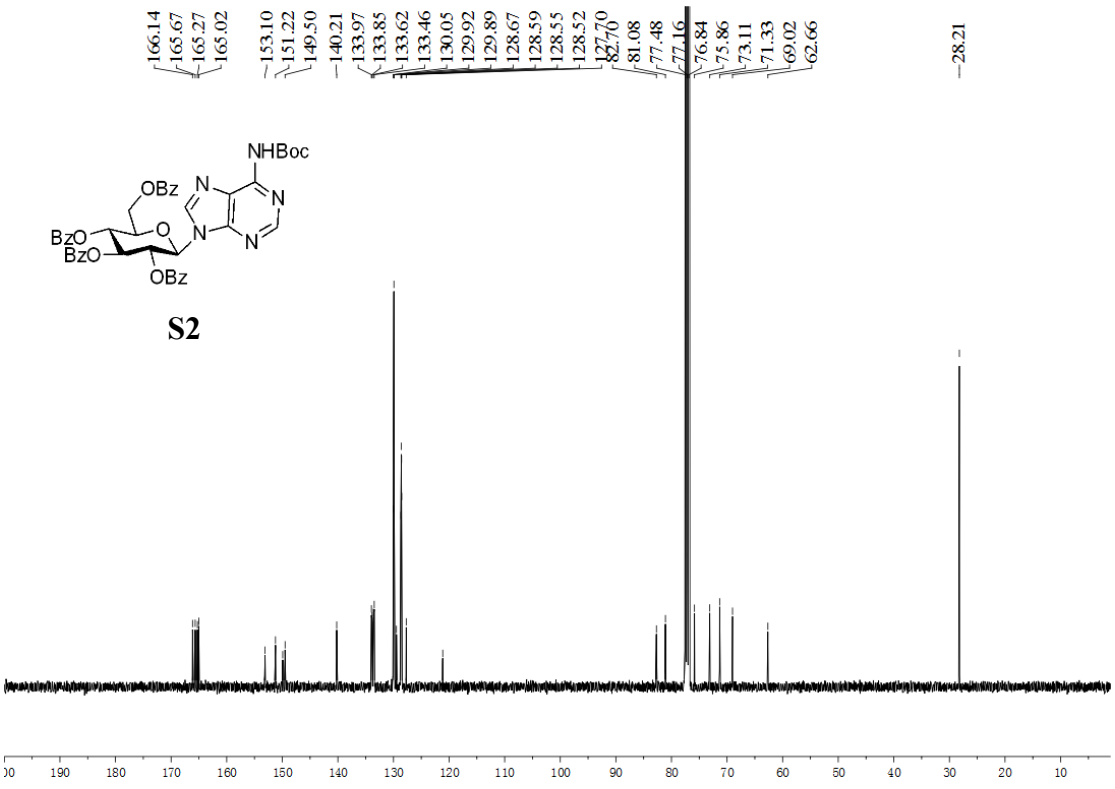
8.03
8.01
7.95
7.93
7.83
7.81
7.71
7.70
7.44
7.42
7.40
7.38
7.28
7.26
6.32
6.29
6.16
6.14
6.10
6.08
4.71
4.68
4.55
4.53
4.51
4.50
4.50
1.53
0.00
NHBoc
OBz
BzO
BzO
OBz
1.02
0.99
2.14
2.06
2.00
1.98
2.06
6.18
5.01
0.98
1.00
1.01
1.01
1.00
1.99
9.17

S2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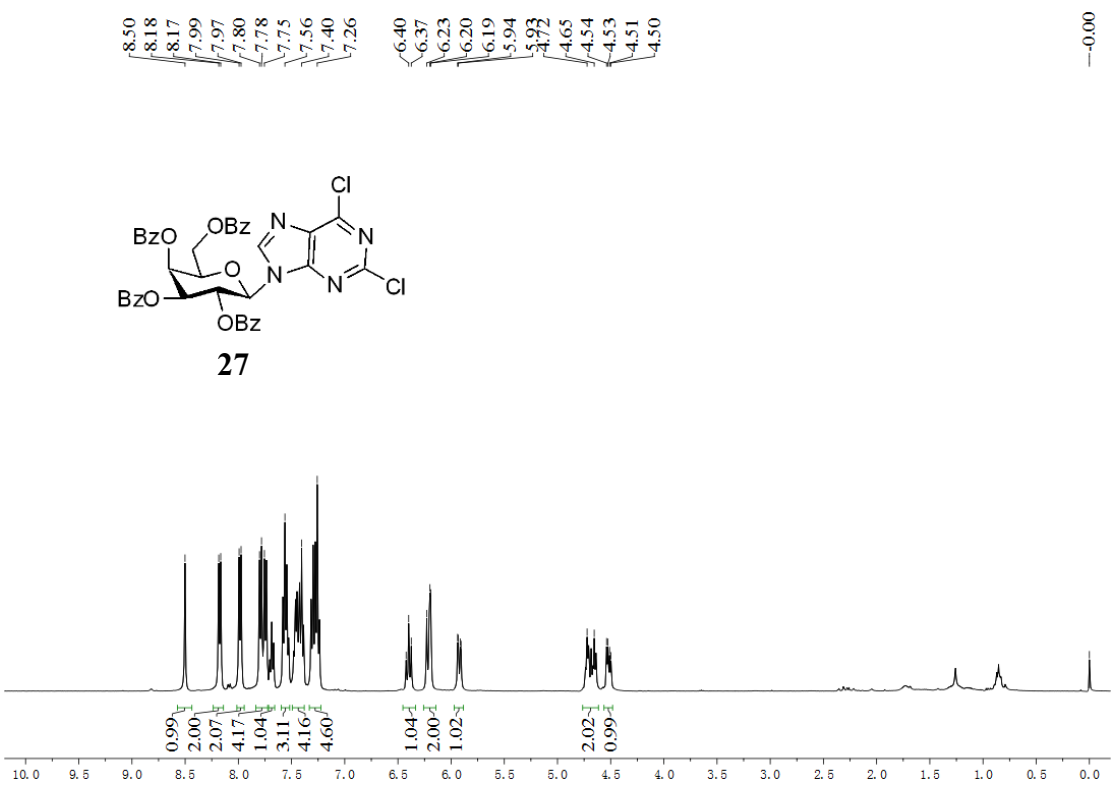
165.67
165.27
165.02
153.10
151.22
149.50
140.21
133.97
133.85
133.62
133.46
130.05
129.92
129.89
128.67
128.59
128.55
128.52
127.70
82.70
81.08
77.48
77.16
76.84
75.86
73.11
71.33
69.02
62.66
28.21
NHBoc
OBz
BzO
BzO
OBz

S2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8.17
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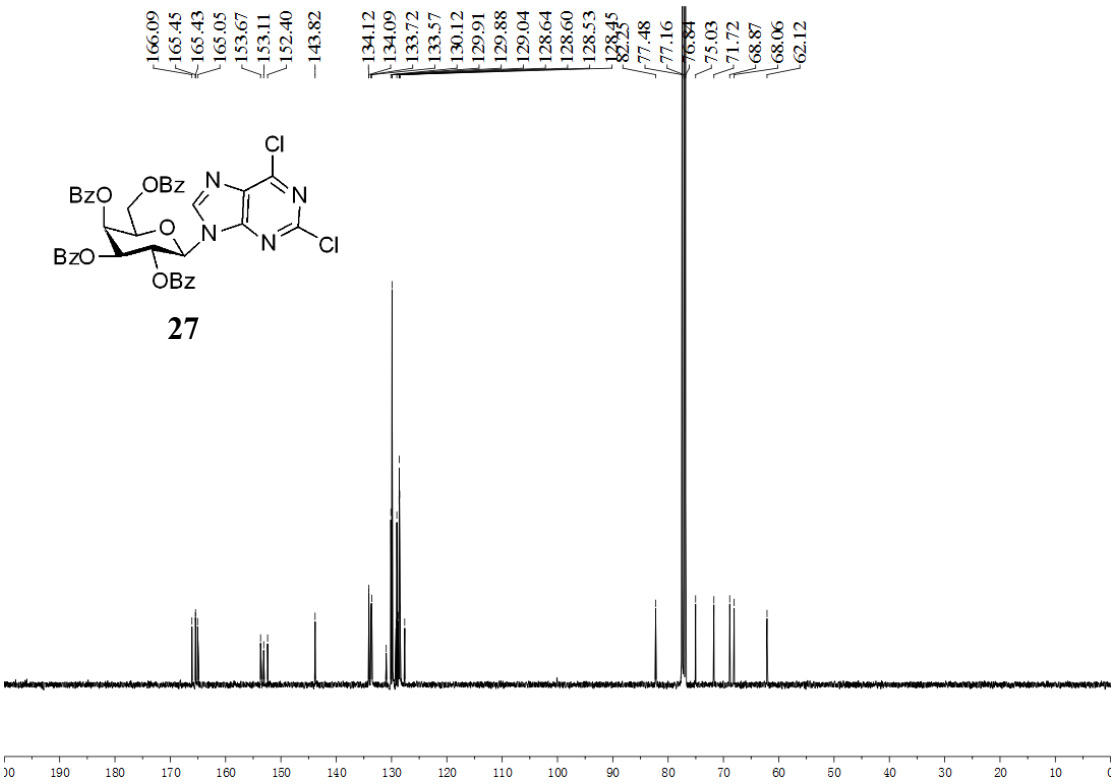
7.97
7.80
7.78
7.75
7.56
7.40
7.26
6.40
6.37
6.23
6.20
6.19
5.94
5.93
5.72
4.65
4.54
4.53
4.51
4.50
0.00
Cl
OBz
BzO
BzO
OBz
Cl
0.99
2.00
2.07
4.17
1.04
3.11
4.16
4.60
1.04
2.00
1.02
2.02
0.99

27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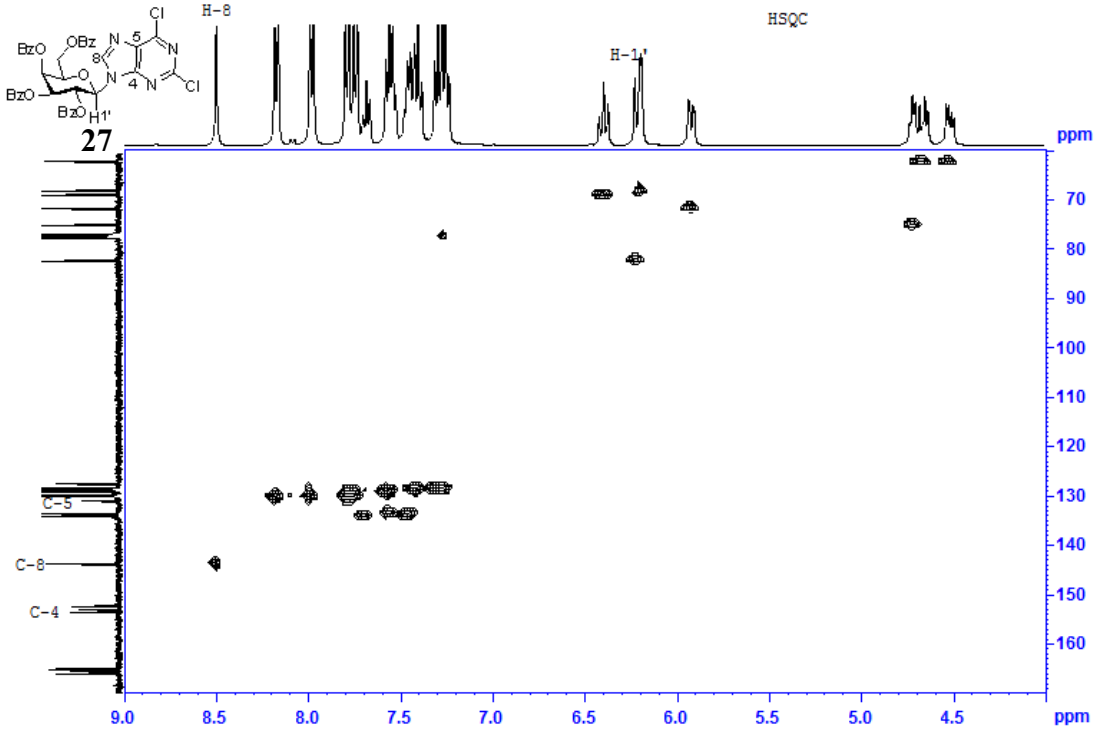
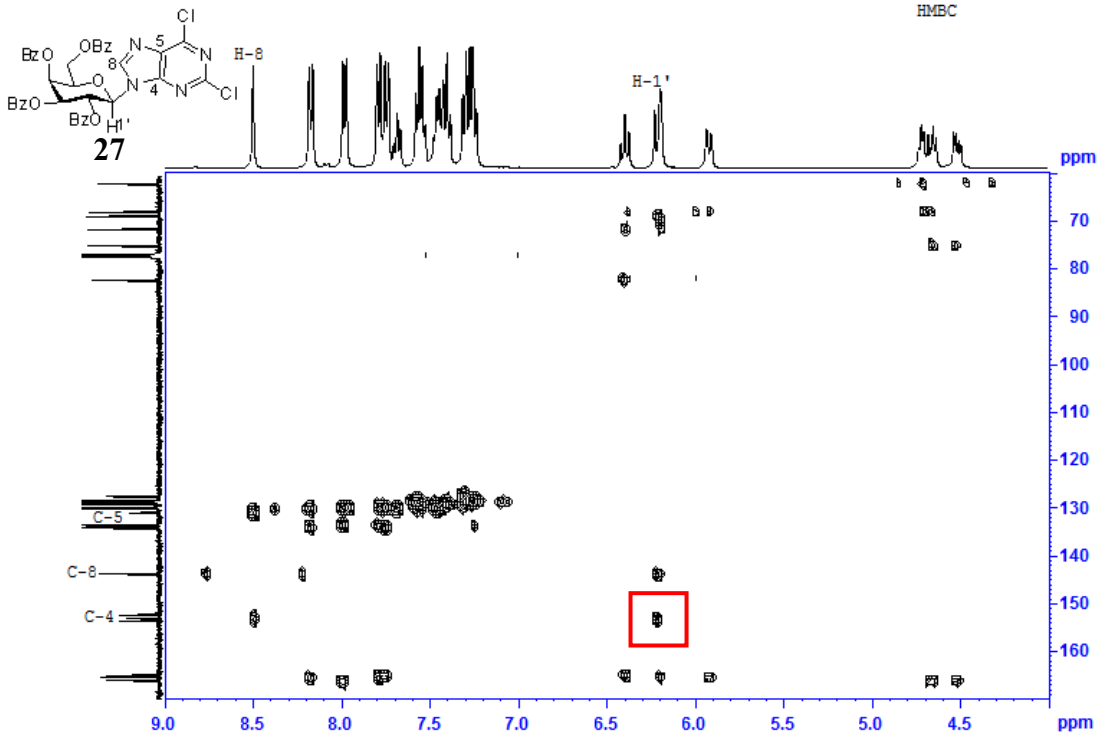
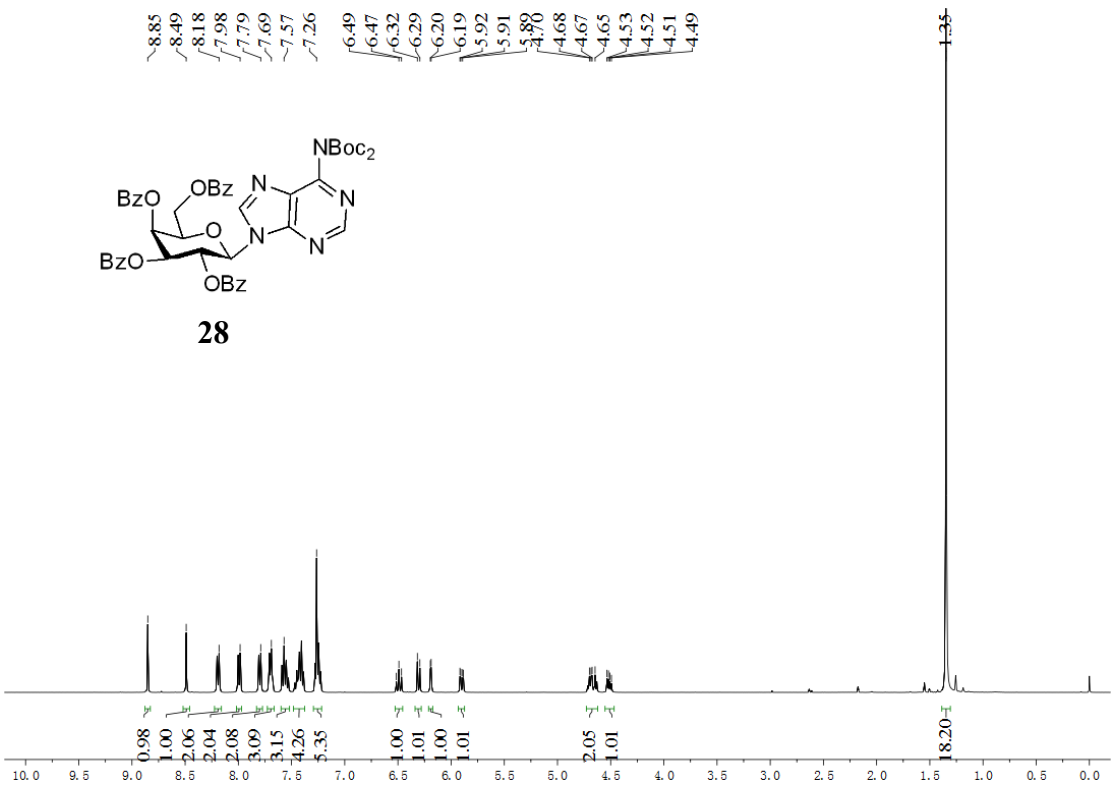
165.43
165.05
153.67
153.11
152.40
143.82
134.12
134.09
133.72
133.57
130.12
129.91
129.88
129.04
128.64
128.60
128.53
128.45
128.23
77.48
77.16
76.84
75.03
71.72
68.87
68.06
62.12
Cl
BzO
OBz
N
N
O
N
N
Cl
BzO
OBz
27


Cl
BzO
OBz
N
5
8
N
4
N
Cl
BzO
O
BzO
H1'
27
H-8
H-1'
HSQC
ppm
70
80
90
100
110
120
130
140
150
160
C-5
C-8
C-4
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
ppm


HMBC
H-8
H-1'
ppm
C-5
C-8
C-4
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
ppm

27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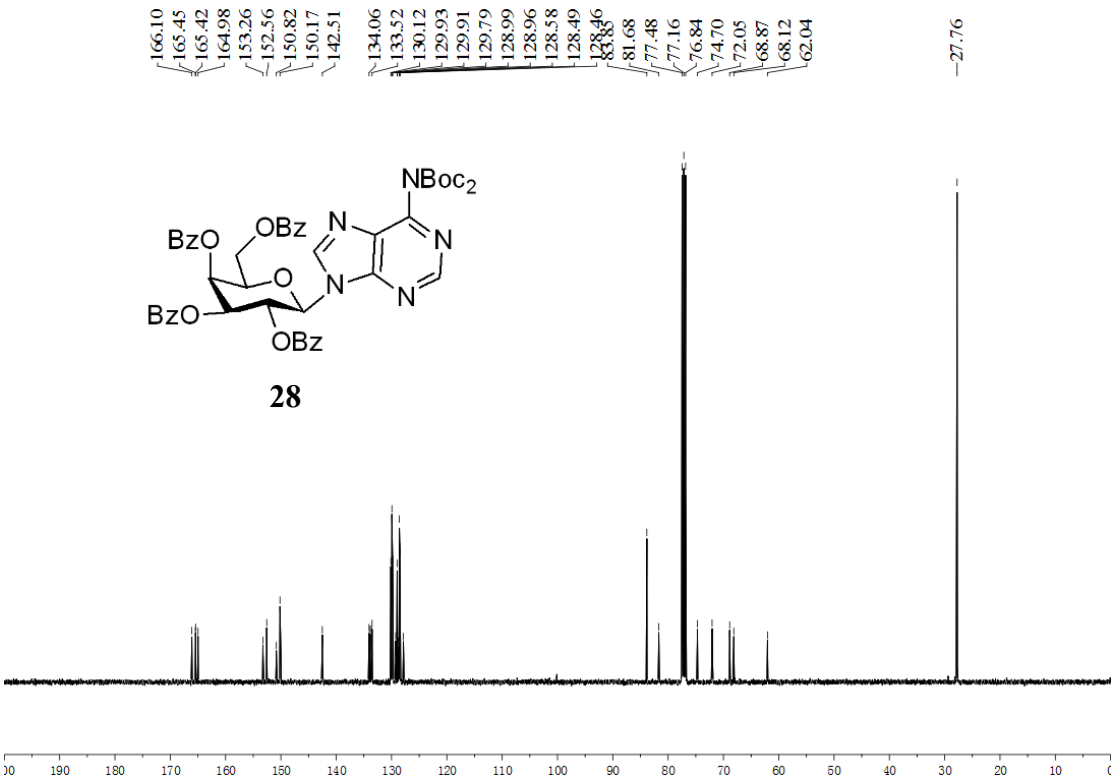
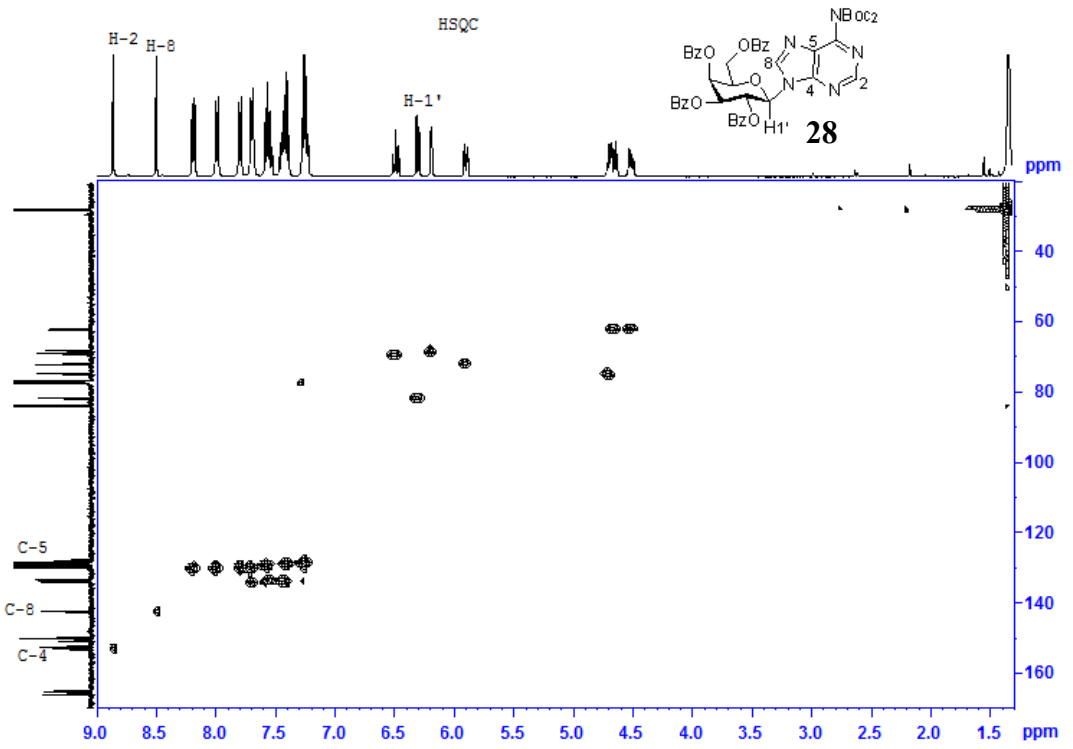
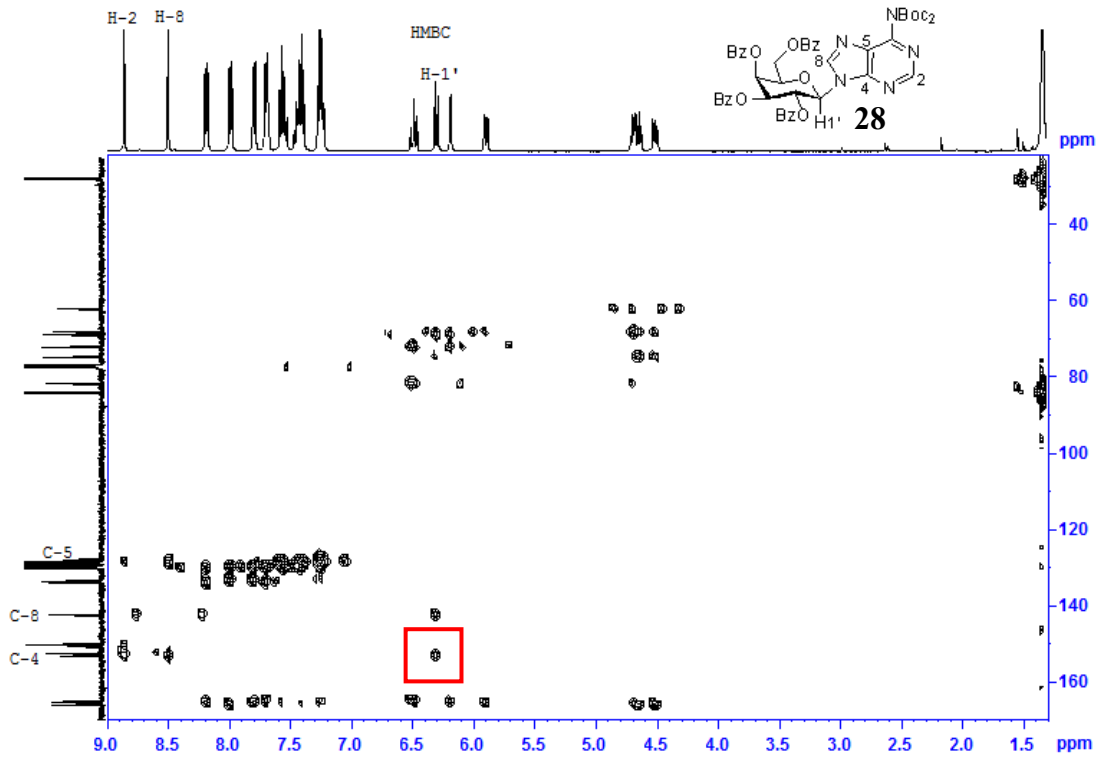
7.79
7.69
7.57
7.26
6.49
6.47
6.32
6.29
6.20
6.19
5.92
5.91
5.88
4.70
4.68
4.67
4.65
4.53
4.52
4.51
4.49
1.35
NBoc2
BzO
OBz
BzO
OBz
0.98
1.00
2.06
2.04
2.08
3.09
3.15
4.26
5.35
1.00
1.01
1.00
1.01
2.05
1.01
18.20

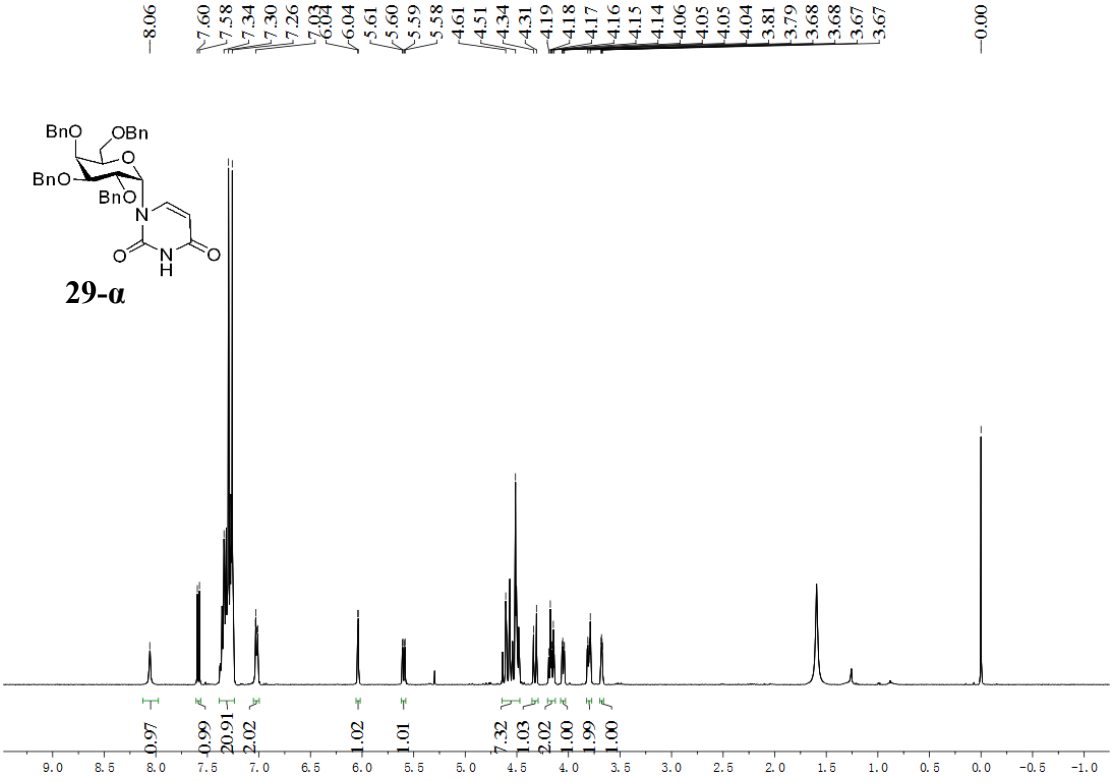
28

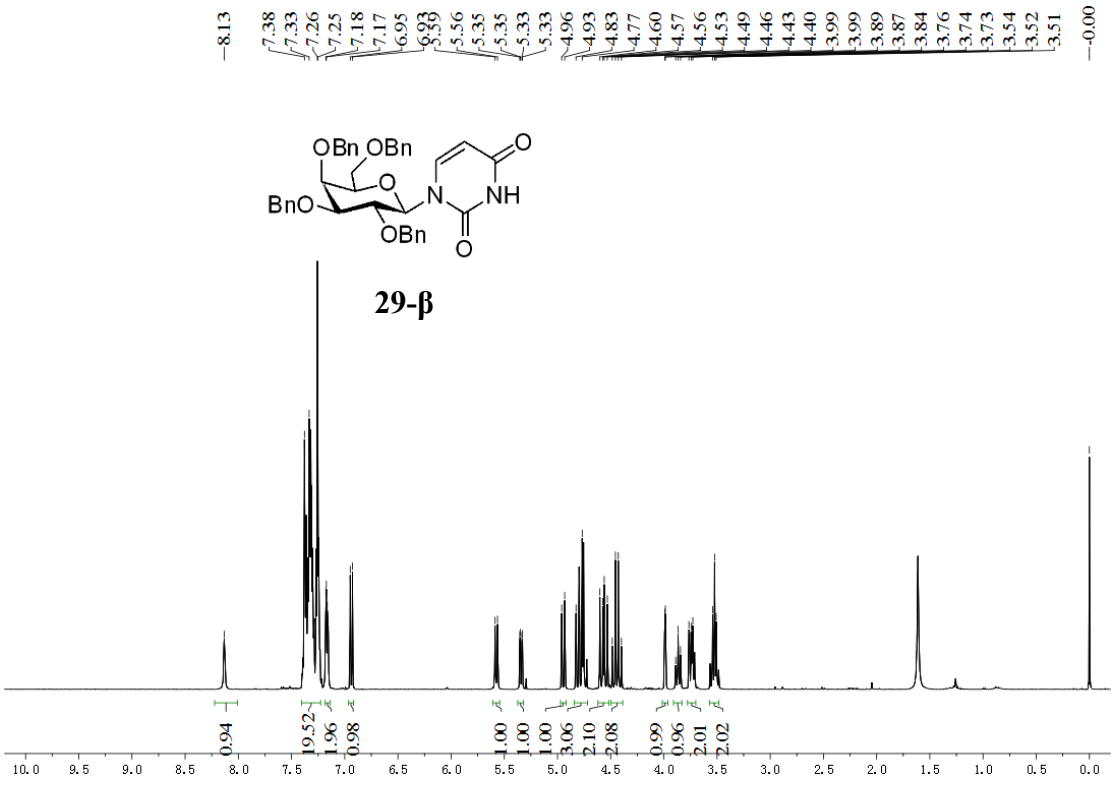

166.10
165.45
165.42
164.98
153.26
152.56
150.82
150.17
142.51
134.06
133.52
130.12
129.93
129.91
129.79
128.99
128.96
128.58
128.49
128.46
83.88
81.68
77.48
77.16
76.84
74.70
72.05
68.87
68.12
62.04
27.76
NBoc2
BzO
OBz
BzO
OBz
N
N
N
N
O

28


HSQC
H-2
H-8
H-1'
NBoc2
BzO
OBz
BzO
BzO
H1'
28
ppm
C-5
C-8
C-4
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
40
60
80
100
120
140
160


H-2
H-8
HMBC
H-1'
NBoc2
BzO
OBz
BzO
BzO
H1'
28
ppm
C-5
C-8
C-4
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm


8.06
7.60
7.58
7.34
7.30
7.26
7.03
6.04
5.61
5.60
5.59
5.58
4.61
4.51
4.34
4.31
4.19
4.18
4.17
4.16
4.15
4.14
4.06
4.05
4.05
4.04
3.81
3.79
3.68
3.68
3.67
3.67
0.00
BnO
OBn
BnO
BnO
N
O
N
H
O
29-α
0.97
0.99
20.91
2.02
1.02
1.01
7.32
1.03
2.02
1.00
1.99
1.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0


8.13
7.38
7.33
7.26
7.25
7.18
7.17
6.95
5.59
5.56
5.35
5.35
5.33
5.33
4.96
4.93
4.83
4.77
4.60
4.57
4.56
4.53
4.49
4.46
4.43
4.40
3.99
3.99
3.89
3.87
3.84
3.76
3.74
3.73
3.54
3.52
3.51
0.00
OBn OBn
BnO
OBn
O
N
NH
O
29-β
0.94
19.52
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1.00
1.00
3.06
2.10
2.08
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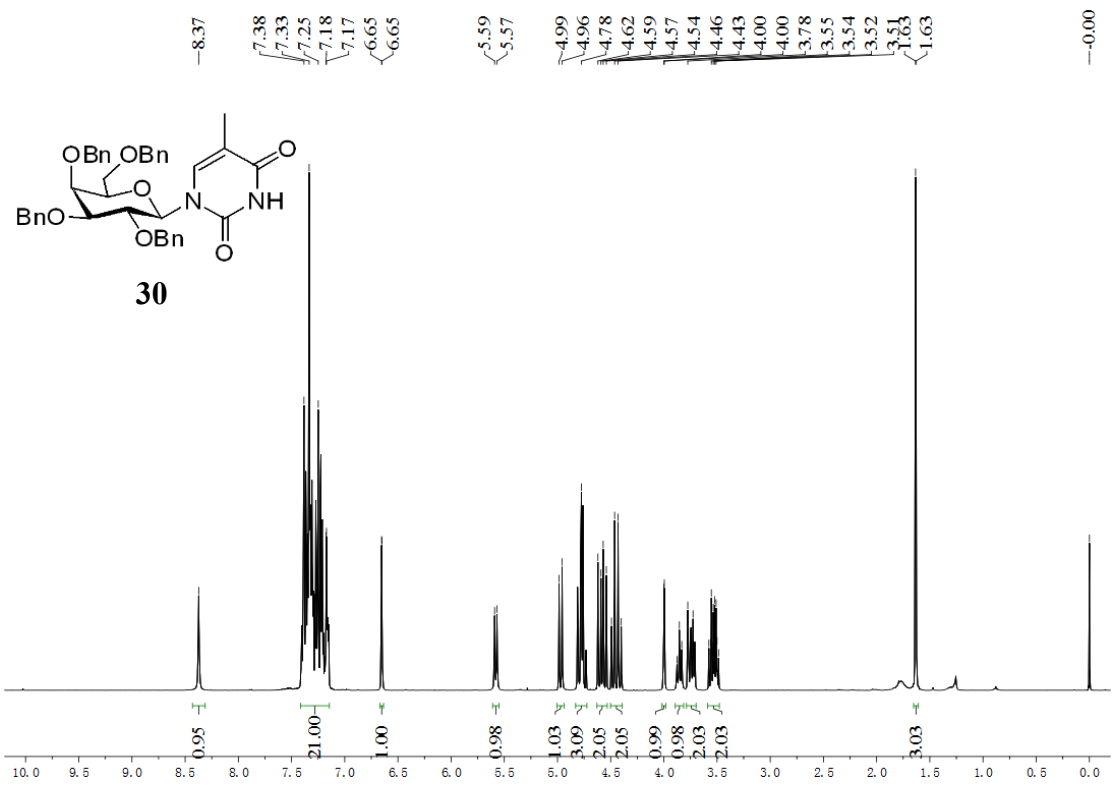
0.96
2.01
2.02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4.78
4.62
4.59
4.57
4.54
4.46
4.43
4.00
4.00
3.78
3.55
3.54
3.52
3.51
1.63
1.63
0.00
OBn OBn
BnO
OBn
O
N
NH
O
30
0.95
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1.03
3.09
2.05
2.05
0.99
0.98
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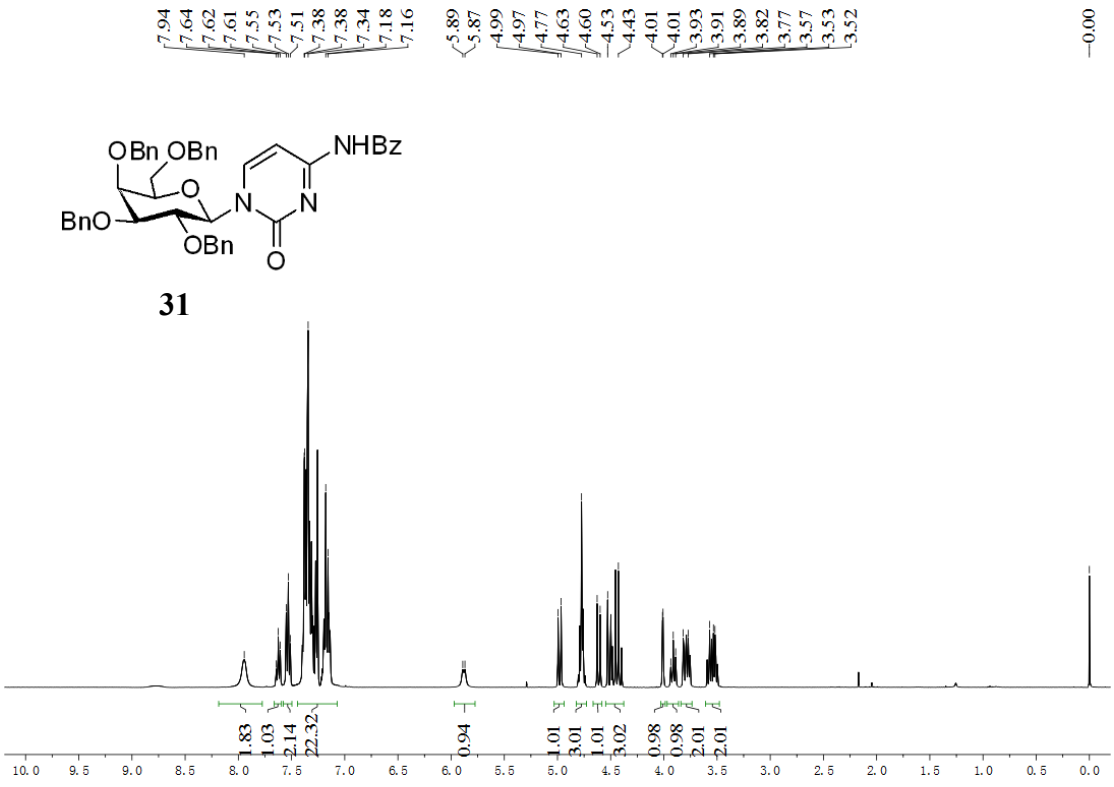
2.03
3.03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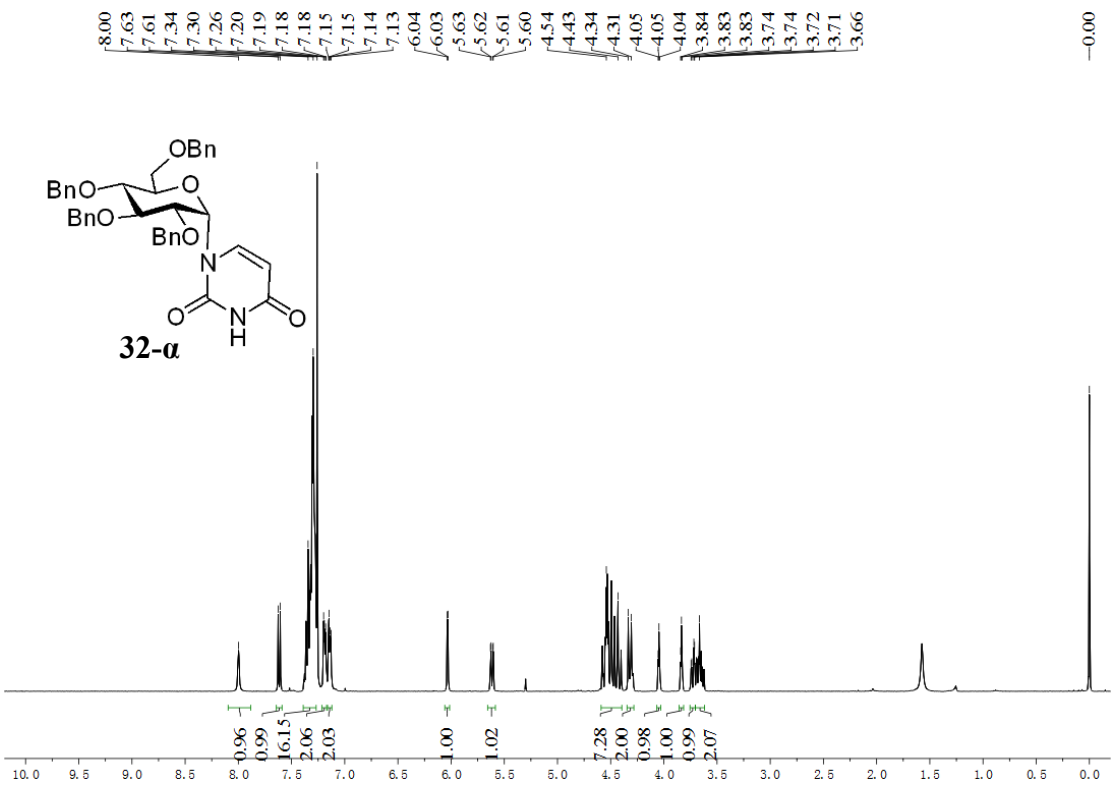
5.87
4.99
4.97
4.77
4.63
4.60
4.53
4.43
4.01
4.01
3.93
3.91
3.89
3.82
3.77
3.57
3.53
3.52
0.00
OBn OBn
NHBz
BnO
OBn
N
N
O
O
1.83
1.03
2.14
22.32
0.94
1.01
3.01
1.01
3.02
0.98
0.98
2.01
2.01

31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7.14
7.13
6.04
6.03
5.63
5.62
5.61
5.60
4.54
4.43
4.34
4.31
4.05
4.04
3.84
3.83
3.74
3.74
3.72
3.71
3.66
0.00
OBn
BnO
BnO
BnO
N
O
N
H
O
0.96
0.99
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1.00
1.02
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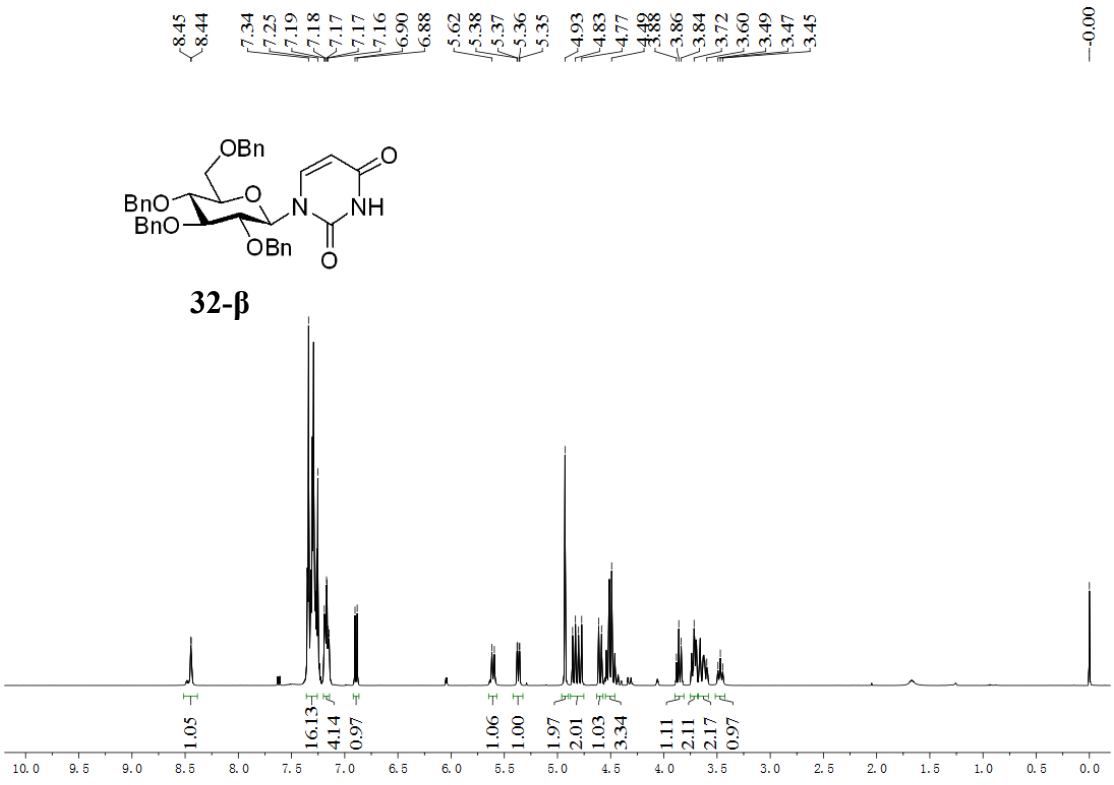
0.98
1.00
0.99
2.07

32-α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5.38
5.37
5.36
5.35
4.93
4.83
4.77
4.49
4.48
3.88
3.86
3.84
3.72
3.60
3.49
3.47
3.45
0.00
OBn
BnO
BnO
OBn
O
N
NH
O
O
1.05
16.13
4.14
0.97
1.06
1.00
1.97
2.01
1.03
3.34
1.11
2.11
2.17
0.97
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
4.0
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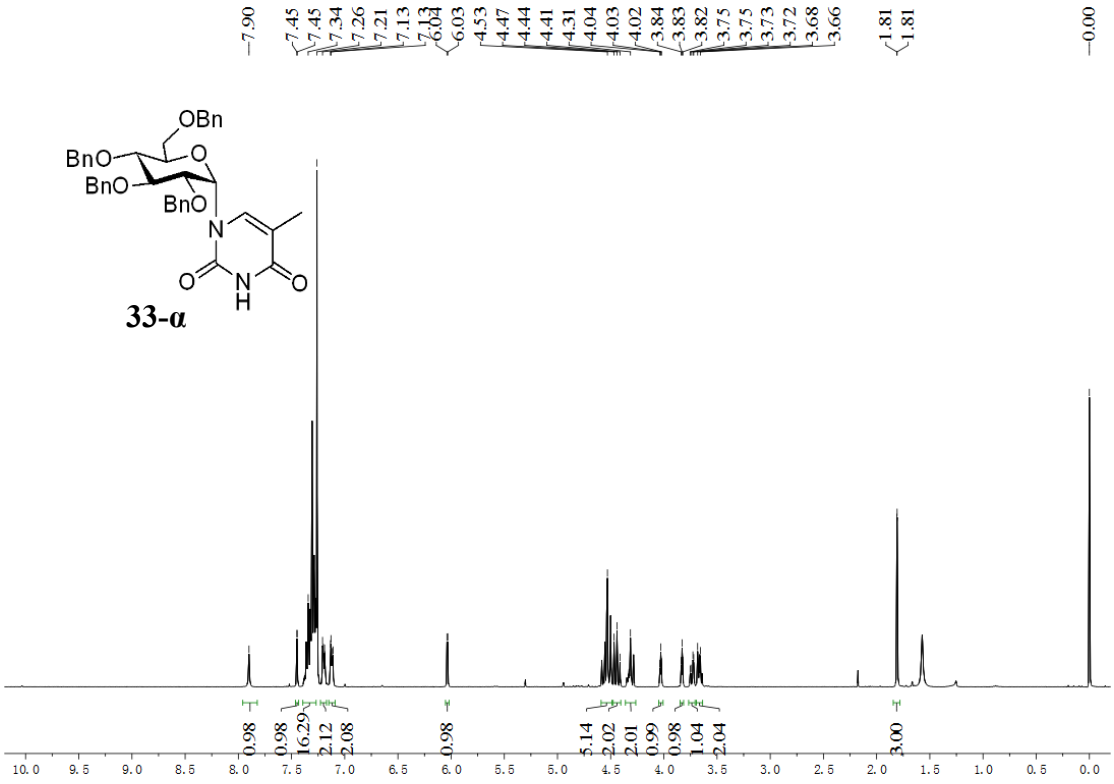
3.0
2.5
2.0
1.5
1.0
0.5
0.0

32-β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4.47
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4.41
4.31
4.04
4.03
4.02
3.84
3.83
3.82
3.75
3.75
3.73
3.72
3.68
3.66
1.81
1.81
0.00
OBn
BnO
BnO
BnO
N
O
N
H
O
0.98
0.98
16.29
2.12
2.08
0.98
5.14
2.02
2.01
0.99
0.98
1.04
2.04
3.00
10.0
9.5
9.0
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8.0
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6.5
6.0
5.5
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4.0
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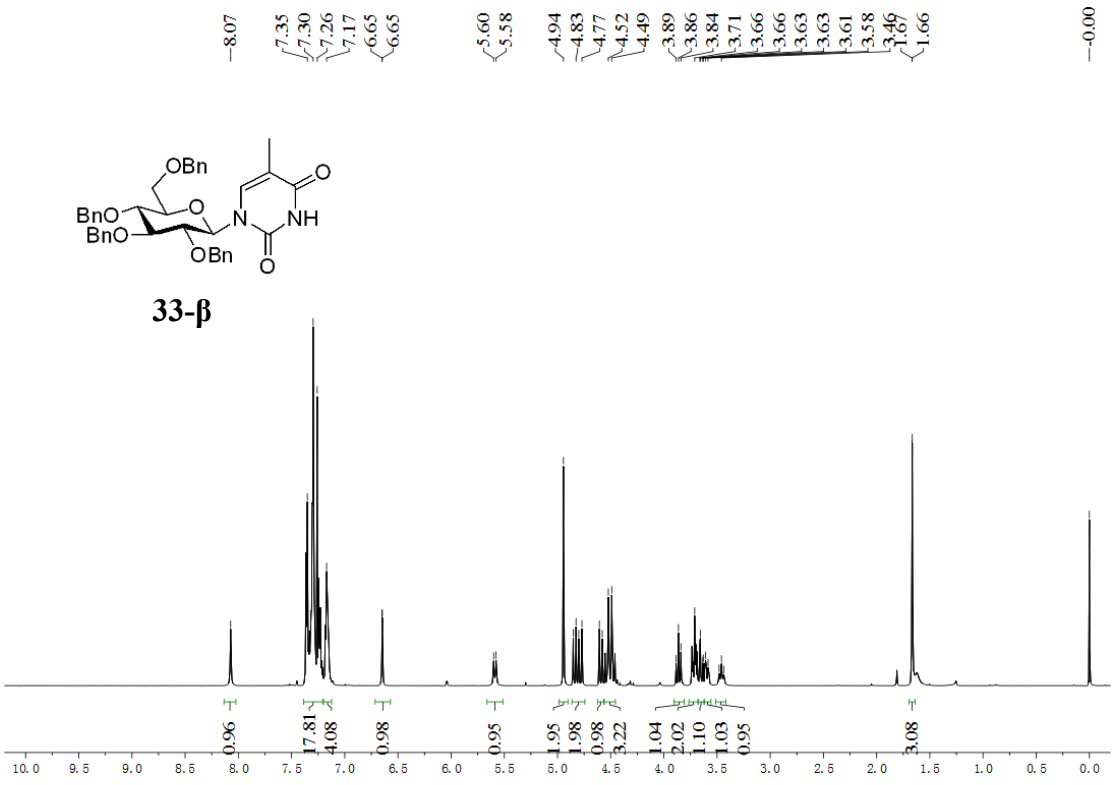
3.0
2.5
2.0
1.5
1.0
0.5
0.0

33-α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4.77
4.52
4.49
3.89
3.86
3.84
3.71
3.66
3.66
3.63
3.63
3.61
3.58
3.46
1.67
1.66
0.00
OBn
BnO
BnO
OBn
O
NH
N
O
0.96
17.81
4.08
0.98
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0.98
3.22
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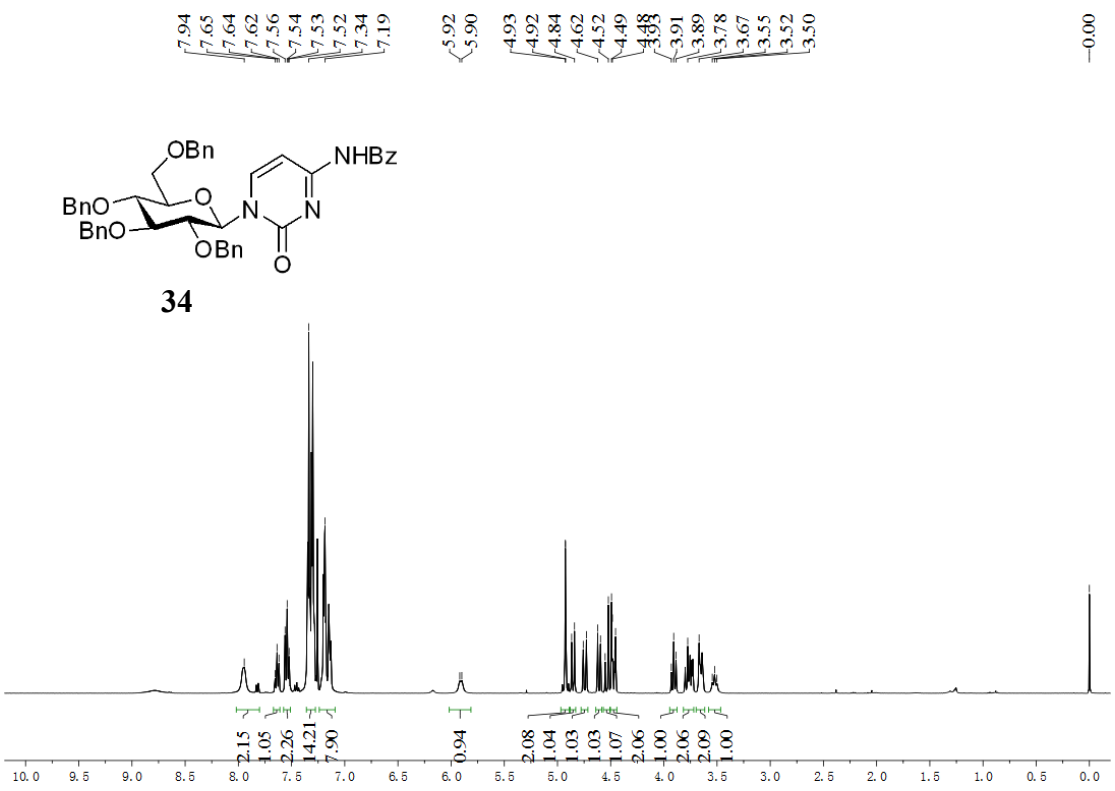
1.10
1.03
0.95
3.08

33-β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4.92
4.84
4.62
4.52
4.49
3.91
3.89
3.78
3.67
3.55
3.52
3.50
0.00
OBn
BnO
BnO
OBn
O
NHBz
N
N
O
2.15
1.05
2.26
14.21
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1.04
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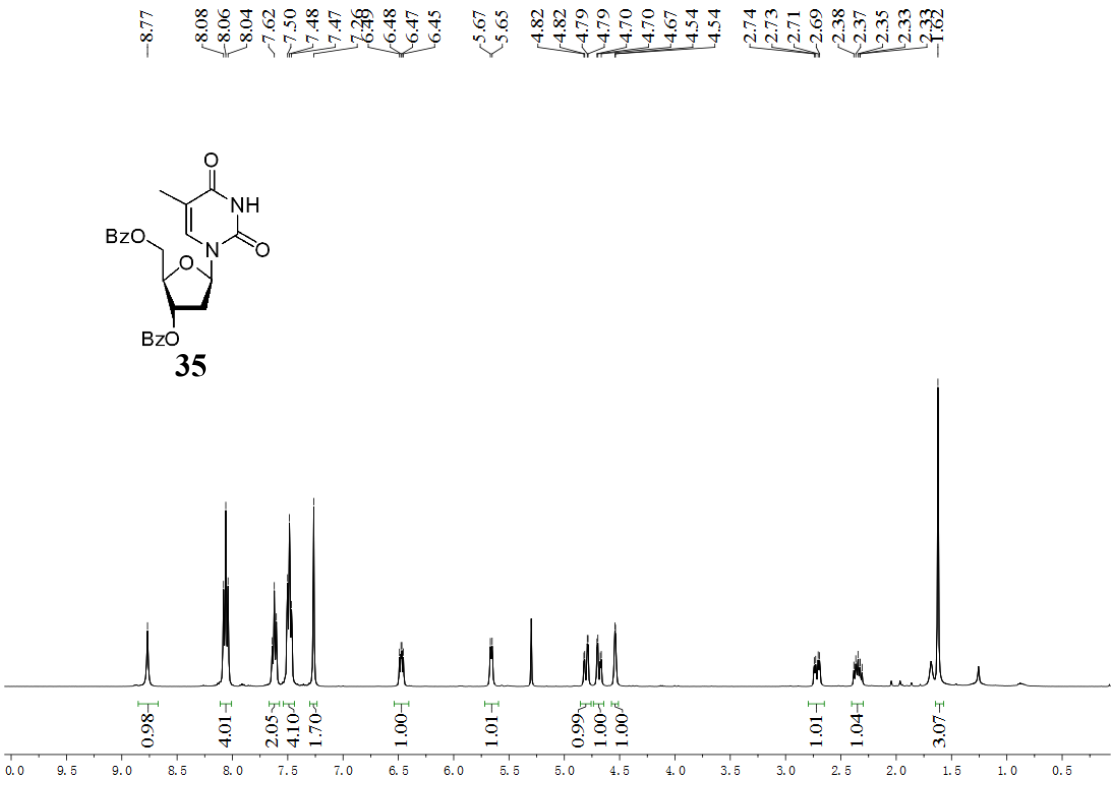
1.03
1.07
2.06
1.00
2.06
2.09
1.00

34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6.47
6.45
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5.65
4.82
4.82
4.79
4.79
4.70
4.70
4.67
4.54
4.54
2.74
2.73
2.71
2.69
2.38
2.37
2.35
2.33
2.33
1.62
NH
BzO
BzO
35
0.98
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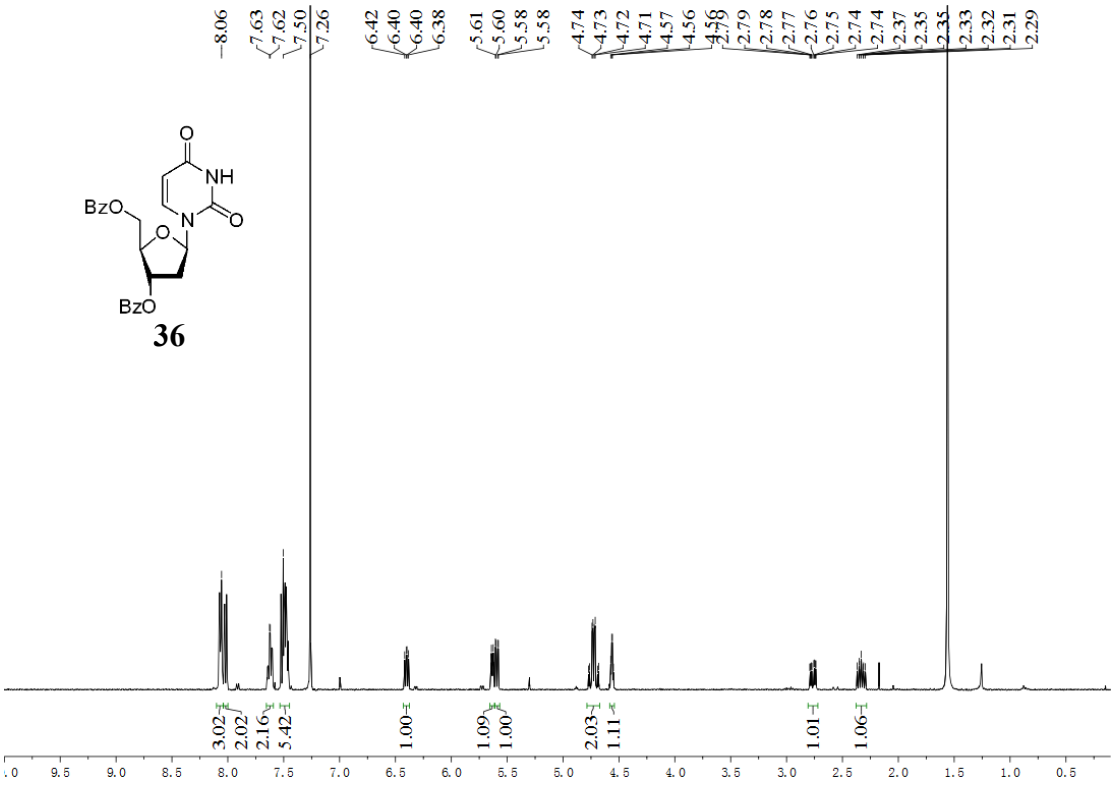
4.10
1.70
1.00
1.01
0.99
1.00
1.00
1.01
1.04
3.07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2.78
2.77
2.76
2.75
2.74
2.74
2.37
2.35
2.35
2.33
2.32
2.31
2.29
NH
BzO
BzO
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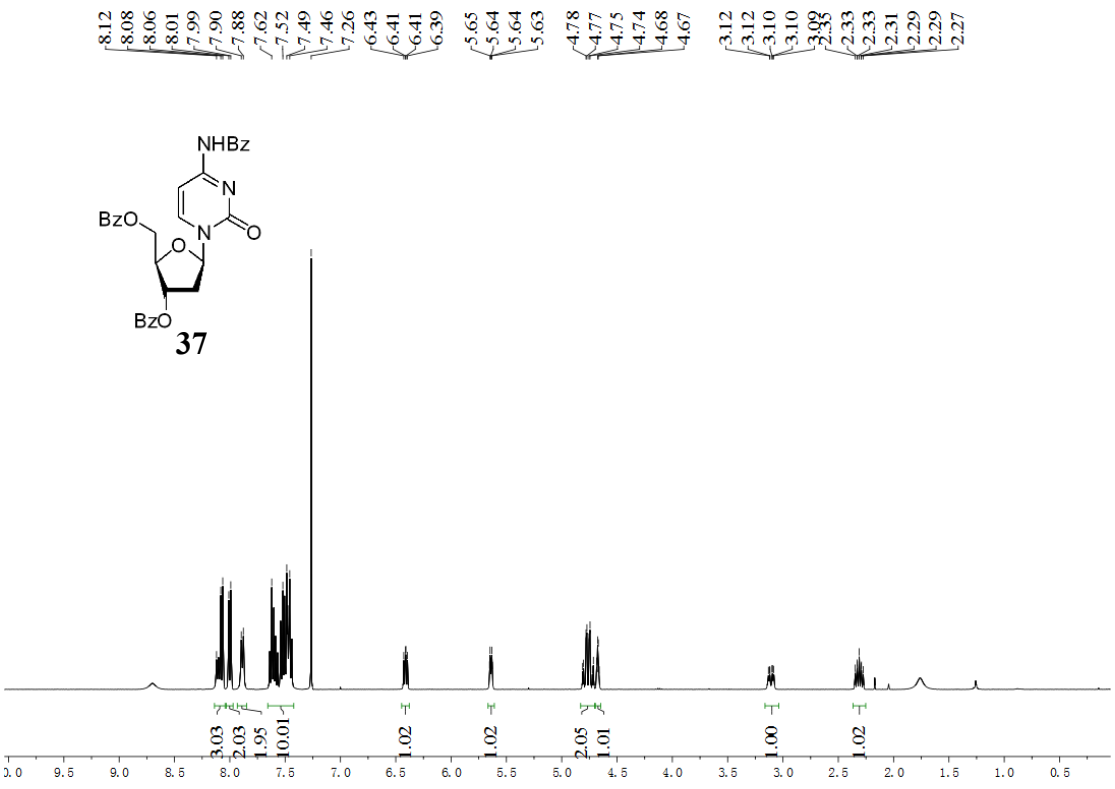
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6.41
6.41
6.39
5.65
5.64
5.64
5.63
4.78
4.77
4.75
4.74
4.68
4.67
3.12
3.12
3.10
3.10
3.09
2.35
2.33
2.33
2.31
2.29
2.29
2.27
NHBz
N
BzO
O
N
O
BzO
37
3.03
2.03
1.95
10.01
1.02
1.02
2.05
1.01
1.00
1.02