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Synthesis of 1,3-Dideoxynojirimycin *via* an α-Amino Aldehyde as a Key Intermediate

by D. Gryko[1] and J. Jurczak[1,2*]

[1]*Institute of Organic Chemistry, Polish Academy of Science, Kasprzaka 44/52, 01-224 Warsaw, Poland*
[2]*Department of Chemistry, Warsaw University, Pasteura 1, 02-093 Warsaw, Poland*

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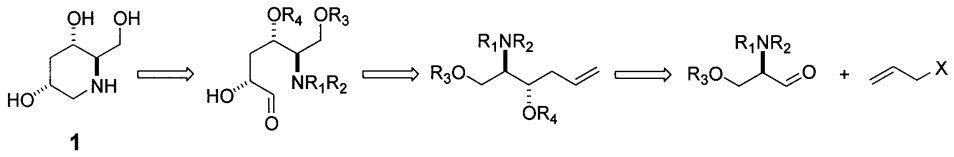
1,3-Dideoxynojirimycin was synthesized starting from a suitably protected α-amino aldehyde. The crucial step involves addition of allyl bromide to *N*-benzyl-*N*-carbobenzoxy-*O*-tert-butyldimethylsilyl-D-serinal in the presence of tin(II) chloride and sodium iodide.

Key words: 1,3-dideoxynojirimycin, α-amino aldehydes, Barbier-type addition, allyl addition

Azasugars have generated a great deal of interest due to their ability to mimic carbohydrates in a variety of biological processes. Early studies indicated that they possess substantial biological activity as potent inhibitors of glycosidase [1,2]. Therefore, they have become attractive candidates for the treatment of cancer [3,4], diabetes [5] and viral infections (HIV) [6,7]. A considerable amount of research has been devoted to the synthesis of this class of compounds. The known methods for the synthesis of azasugars are mainly based on transformations of naturally occurring D-pentoses and D-hexoses [8–11], but they can also be synthesized from nonsugar precursors. Johnson *et al*. [12] have published a new method for the synthesis of 1,3-dideoxynojirimycin (**1**) using an enzymatic dissymmetrization of cyclopentadiene. Another example was presented by Meyers and Price [13], who synthesized azasugars starting from readily available chiral lactams, obtained from cyclohexan-1,3-dione and (*S*)-phenylglycinol. During the course of our studies on the application of α-amino aldehydes as a chiral pool, we have found that they are very convenient, versatile and

Scheme 1



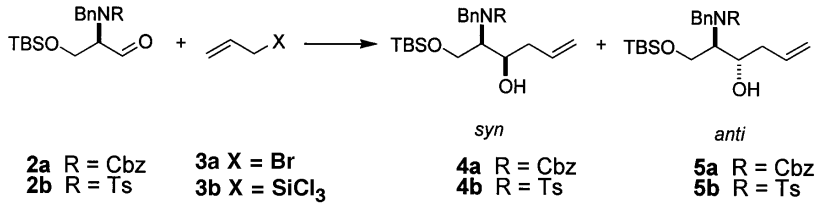
*Author for correspondence.

effective chirons [14–16]. Herein, we would like to report that they can also be applied to the synthesis of 1,3-dideoxynojirimycin (**1**). Retrosynthetic analysis shown in Scheme 1, as it was reported in the preliminary communication [17], suggested that a suitably *N*,*N*,*O*-protected D-serinal [18] and an allylation reagent are convenient starting materials.

RESULTS AND DISCUSSION

Model studies. Recently we have reported an allyl-type addition to variously *N*-mono and *N*,*N*-diprotected alaninals [19]. For allyl additions to *N*-monoprotected L-alaninals *syn*-diastereoselectivity was observed, whereas for *N*,*N*-diprotected L-alaninals *anti*-diastereoselectivity predominated. Giannis *et al.* [20] have reported that the reaction of *N*-monoprotected serinals with allyl halides proceeds smoothly to give *syn*-diastereoisomer as a major product. Since the addition of allyl reagents to *N*,*N*-diprotected alaninals leads to the substantial *anti*-diastereoselectivity, we expected that application of *N*,*N*-diprotected serinals should lead to *anti*-diastereoselectivity. Therefore, for our studies, we have chosen *N*-Bn-*N*-Cbz-*O*-TBS-D-serinal (**2a**) and *N*-Bn-*N*-Ts-*O*-TBS-D-serinal (**2b**) since they are suitably protected for further synthetic steps. On the addition of allyl bromide (**3a**) to aldehydes, **2a** and **2b** (Scheme 2) carried out in the presence of NH_4Cl_{aq} [21,22], *anti*-diastereoselectivity (8:2) and very high yield were observed (entries 1 and 5, Table 1).

Scheme 2



The addition of allylzinc bromide, generated *in situ* from allyl bromide (**3a**) and zinc dust in the presence of $AlCl_3$ [23], to **2a** and **2b** gave slightly better yield and also *anti*-adduct as a major product (entries 2 and 6). Similar results were obtained when the addition of allyl bromide (**3a**) was carried out in the presence of $SnCl_2 \cdot H_2O$ and NaI [24] (entries 3 and 7). In the case of additions of allyltrichlorosilane (**3b**) to **2a** and **2b**, performed in DMF [25], in both cases yields were very low due to the side-products formation, and the level of diastereoselectivity decreased (entries 4 and 8). The determination of a *syn/anti* ratio was based on ^{1}H NMR spectra namely on the integration of separated signals derived from one of the diastereotopic protons at the double bond. Having determined the asymmetric induction, we studied its direction

by establishing the configuration of products. The structure of *syn*-**4a** and *anti*-**5a** was univocally determined by a comparison with the literature data available for their enantiomers [26]. Direction of the asymmetric induction can be explained by the non-chelating Felkin-Anh model [27,28]. The attack of allyl reagents occurs from the less hindered side of the nonchelating Felkin-Anh model leading to *anti*-products. Taking into consideration that aldehyde **2b** differs from **2a** only in the nature of the second *N*-protecting group we can assume with a high probability that the direction of asymmetric induction will be retained. So upon the addition of allyl reagents (presented in this paper) to aldehyde **2b**, *anti*-**5b** was formed as a major product.

Table 1. Addition of allyl reagents to aldehyde **2a** and **2b**.

Entry	Aldehyde	Allyl reagent	Modifying additives	Solvent	Temp.	Time [h]	Yield [%]	Ratio *syn*:*anti*
1	**2a**	AllBr	Zn	NH_4C_{aq}/THF	r.t.	5	96	18:82
2	**2a**	AllBr	Zn, $AlCl_3$	THF	r.t.	0.5	99	20:80
3	**2a**	AllBr	$SnCl_2 \cdot H_2O$, NaI	DMF	r.t.	1	81	11:89
4	**2a**	$AllSiCl_3$	–	DMF	0°C	3	45	21:79
5	**2b**	AllBr	Zn	NH_4Cl_{aq}/THF	r.t.	3	78	15:85
6	**2b**	AllBr	Zn, $AlCl_3$	THF	r.t.	3	90	12:88
7	**2b**	AllBr	$SnCl_2 \cdot H_2O$, NaI	DMF	r.t.	1.5	67	17:83
8	**2b**	$AllSiCl_3$	–	DMF	0°C	5	8	36:64

Total synthesis (Scheme 3). *syn*-Dihydroxylation of *anti*-adduct, **5a** with NMO and osmium(IV) oxide [29] afforded a mixture of diastereoisomeric triols **6** (1:1 as determined by HPLC). We thought that the protection of the hydroxy group in **5** could influence the asymmetric induction of the dihydroxylation process. Therefore, we chose the triphenylsilyl protecting group as it could be cleaved in the same way as the *tert*-butyldimethylsilyl group, already present in **5**. Unfortunately, we did not obtained acceptable diastereoselectivity in this case as well 1.2:1 for **7a** and 2:1 for **7b**. Being unable to obtain satisfactory 1,3-asymmetric induction in this process, we turned our attention to an asymmetric *syn*-dihydroxylation process discovered by Sharpless [30]. Treatment of adduct **5a** with commercially available AD-mix α, even at room temperature, did not give any product, unreacted **5a** was fully recovered. We have also checked this reaction adding all components separately. In this case diols **8a** were formed but the diastereoselectivity was very poor. Unable to obtain desired pure diastereoisomeric diols **8a**, we decided to pursue our synthesis using a diastereoisomeric mixture of diols **8a**. The primary hydroxy group in diols **8a** was oxidized to aldehyde **9** using the TEMPO method [31]. Compound **9** was not purified due to the possibility of isomerization. Therefore, it was hydrogenated, as obtained, in the presence of the catalytic amount of palladium. Upon treatment of hydrogen, *N*-Bn and *N*-Cbz protections were cleaved, an imine was formed, and finally it was reduced to cyclic amines **10** and **11**. Column chromatography on silica afforded both diastereoisomers in enantiometrically pure form.

Scheme 3

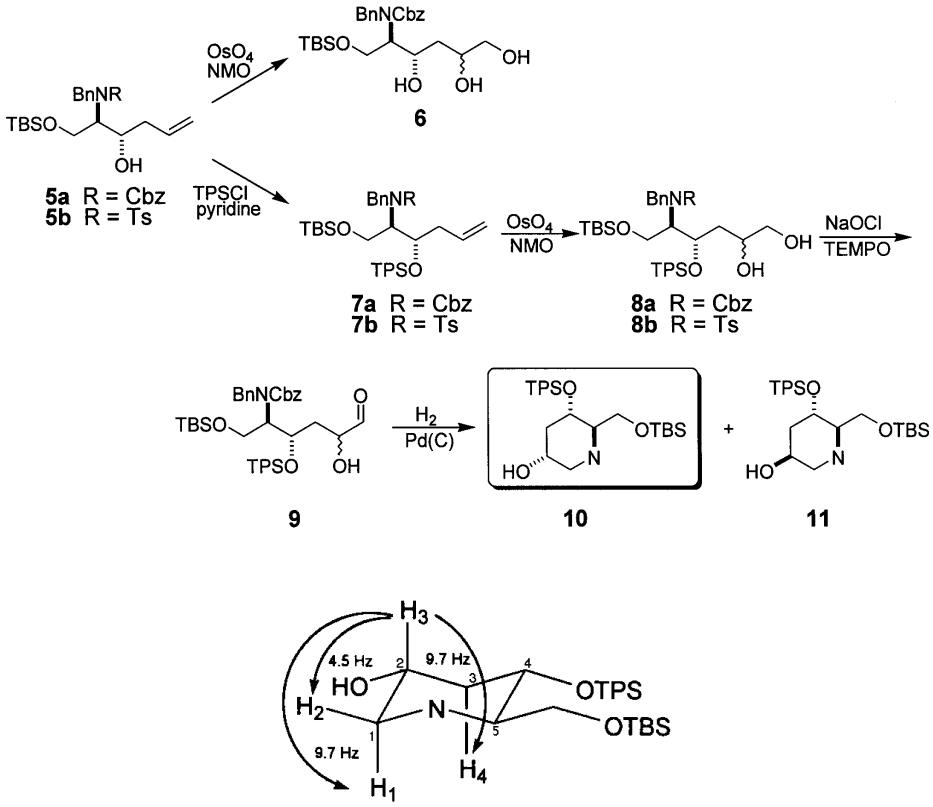


Figure 1.

The diastereoisomeric assignments for *O*-protected 1,3-dideoxynojirimycin **10** was made by ^{1}H NMR spectroscopy (Figure 1). The signal originated from the proton H-2 is a doublet of doublets of triplets and exhibits three coupling constants $J_1 = J_2 =$ 9.7 Hz and $J_3 = 4.5$ Hz. The coupling constant ($J = 9.7$ Hz) between H-3 and H-1 and H-4 protons indicates axial : axial interactions, while the coupling constant ($J = 4.5$ Hz) between H-3 and H-2 protons indicates axial : equatorial interactions, leading to the conclusive (*R*) configuration of carbon C-2.

CONCLUSIONS

In summary we have developed a facile and effective synthesis of 1,3-*O*-protected dideoxynojirimycin (**10**) starting from *N,N,O*-protected serinal **2a** using allylation reaction as a key step with overall yield 19.9%. We were able to obtain the

desired *anti*-allyl adduct with good yield and high optical purity, using allylation reactions presented in this paper. This approach should be general for the synthesis of substituted piperidines if started from various α-amino aldehydes.

EXPERIMENTAL

General remarks: All chemicals were used as received unless otherwise noted. Reagent grade solvents ($CHCl_3$, CH_2Cl_2, hexanes, AcOEt) were distilled prior use. All reported NMR spectra were recorded with a Bruker spectrometer at 500 (^{1}H NMR) and 125 (^{13}C NMR) MHz or Varian Gemini spectrometer at 200 (^{1}H NMR) and 50 (^{13}C NMR) MHz. Chemical shifts are reported as δ values relative to TMS peak defined at δ = 0.00 (^{1}H NMR) or δ = 0.0 (^{13}C NMR). IR spectra were obtained on a Perkin-Elmer 1640 FTIR. Mass spectra were obtained on an AMD-604 Intectra instrument using the EI or LSIMS technique. Chromatography was performed on silica (Kieselgel 60, 200–400 mesh). Optical rotations were recorded using a JASCO DIP-360 polarimeter with a thermally jacketed 10 cm cell.

Compounds 4b and 5b as a mixture *anti:syn* = 9:1: ^{1}H NMR (200 MHz, $CDCl_3$) 0.03 (s, 3H), 0.12 (s, 3H), 0.90 (s, 9H), 2.0–2.2 (m, 1H), 2.2–2.4 (m, 1H), 2.46 (s, 3H), 2.56 (bd, 1H), 3.6–3.9 (m, 4H), 4.54 (AB, *J* = 15.4, 2H), 4.9–5.1 (m, 2H), 5.4–5.6 (m, 1H), 7.2–7.4 (m, 6H), 7.8–7.9 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) –5.8, –5.7, 18.1, 21.4, 25.7, 38.4, 39.3, 49.8, 50.2, 61.9, 62.1, 63.4, 64.1, 68.9, 72.0, 117.6, 117.7, 127.3, 127.4, 127.5, 127.7, 128.4, 128.5, 128.6, 129.5, 129.6, 134.3, 134.6, 137.8, 138.0, 143.2.

***syn*-Dihydroxylation OsO_4/NMO**. To a solution of allyl adduct (1 mmol) in the mixture of THF-*tert*-BuOH-H_2O (8 ml : 0.65 ml : 0.1 ml), *N*-methylmorpholin (320 mg, 2.3 mmol) and the solution of OsO_4 in *tert*-BuOH (2.2 ml, 0.11 mmol, 0.05 M) were added. The reaction mixture was stirred for 6 h, and then saturated solution of $Na_2S_2O_3$ (50 ml) and methanol (50 ml) were added. Extraction with ether (organic phase was washed with brine and dried over $MgSO_4$), followed by chromatography (silica, $CHCl_3$/methanol) gave the desired product.

Compound 6 as a mixture of diastereoisomers 1:1: Yield 90%, ^{1}H NMR (500 MHz, 90°C, toluen-d_8) –0.02 (s, 3H), –0.01 (s, 3H), 0.88 (s, 9H), 1.5–1.3 (m, 3H), 3.3–3.1 (m, 5H), 3.7–3.4 (m, 2H), 4.2–4.0 (m, 2H), 4.5–4.3 (m, 1H), 4.7–4.6 (m, 1H), 5.1–5.0 (m, 2H), 7.2–7.0 (m, 10H); ^{13}C NMR (125 MHz, 90°C, toluen-d_8) –5.3, 15.5, 17.4, 18.5, 26.2, 38.1, 53.1, 53.3, 62.1, 62.4, 65.9, 67.0, 67.1, 67.9, 70.2, 70.8, 72.5, 73.4, 96.7, 127.6, 127.7, 128.3, 128.5, 128.6, 128.7, 128.8, 129.1, 129.2, 137.2, 137.3, 137.6, 145.3, 157.4; HR LSIMS $C_{27}H_{41}NO_6SiNa$ $(M+Na)^+$ calcd 526.2601, found 526. 2610.

Protection of the hydroxy group with triphenylsilylchloride. To a solution of allyl adduct (1 mmol) in a mixture of dry CH_2Cl_2 (40 ml) and dry pyridine (40 ml), triphenylsilylchloride (570 mg, 2 mmol) was added. The reaction mixture was stirred for 20 h and then diluted with water. Extraction with ether (organic phase was washed with saturated aqueous $NaHCO_3$ and brine, dried over $MgSO_4$), followed by chromatography (silica, hexane/ethyl acetate) gave afforded crystals.

Compound 7a: Yield 80%, ^{1}H-NMR (200 MHz, $CDCl_3$) 0.0–(–0.9) (m, 6H), 0.85 (s, 9H), 2.4–2.0 (m, 2H), 3.8–3.5 (m, 1H), 4.6–4.0 (m, 5H), 5.0–4.6 (m, 2H), 5.2–5.1 (m, 2H), 5.8–5.3 (m, 1H), 7.9–7.1 (m, 25H); IR (film): 708, 711, 837, 1116, 1251, 1428, 1703, 2828, 2855, 2954, 3069; $[\alpha]_D^{20}$ +5.26 (*c* 1.4) Anal. Calcd for $C_{45}H_{53}NO_4Si_2$: C, 73.96; H, 7.17; N, 1.90. Found: C, 74.28; H, 7.29; N, 1.93.

Compound 7b: ^{1}H NMR (500 MHz, $CDCl_3$) –0.05 (s, 3H), 0.72 (s, 3H), 0.96 (s, 9H), 2.6–2.5 (m, 2H), 2.59 (s, 3H), 3.76 (dd, *J* = 11.1, 9.0 Hz, 1H), 4.03 (AB, *J* = 5.9 Hz, 1H), 4.21 (dd, *J* = 11.1, 4.8 Hz, 1H), 4.35 (d, *J* = 15.9 Hz, 2H), 4.73 (AB/2, *J* = 15.9 Hz, 1H), 4.93 (m, 1H), 5.1–5.0 (m, 1H), 5.56 (ddt, *J* = 17.1, 10.2, 7.0 Hz, 1H), 7.4–7.3 (m, 6H,), 7.54 (m, 6H), 7.63 (m, 4H), 7.73 (m, 6H), 7.82 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) 18.3, 21.4, 25.9, 39.2, 48.7, 61.1, 63.1, 74.5, 117.7, 127.2, 127.6, 127.7, 127.8, 128.2, 128.5, 129.3, 129.9, 134.1, 134.2, 135.2, 135.4, 135.6, 137.7, 138.3, 142.6; IR (film) 701, 710, 837, 997, 1092, 1161, 1338, 1429, 2855, 2927, 2954, 3049, 3068; $[\alpha]_D^{20}$ –17.7 (*c* 0.9); HR LSIMS $C_{44}H_{53}NO_4Si_2SNa$ $(M+Na)^+$ calcd 770.3132, found 770.3138.

Compound 8a as a mixture of diastereoisomers 1.2:1: Yield 83%, ^{1}H NMR (500 MHz, 90°C, toluen-d_8) –0.0–(–0.1) (m, 6H), 0.87 (s, 9H), 1.4–1.3 (m, 1H), 1.7–1.6 (m, 2H), 3.0–2.9 (m, 1H), 3.2–3.1 (m, 1H), 3.8–3.7 (m, 1H), 3.9–3.8 (m, 1H), 4.2–4.1 (m, 0.3H), 4.3–4.2 (m, 0.7H), 4.5–4.3 (m, 2H), 4.7–4.5

(m, 2H), 5.1–4.9 (m, 2H), 7.2–7.0 (m, 20H), 7.68 (m, 5H); ^{13}C NMR (125 MHz, 90°C, toluen-d_8) –5.3, –5.2, 18.5, 19.6, 19.7, 19.9, 20.1, 20.3, 20.4, 20.6, 20.7, 20.9, 26.3, 29.6, 38.6, 39.1, 51.3, 51.8, 62.2, 62.5, 65.1, 65.7, 67.3, 67.5, 67.7, 69.1, 69.3, 72.3, 72.4, 96.7, 127.3, 127.4, 128.1, 128.2, 128.3, 128.5, 128.6, 128.8, 128.9, 130.4, 130.5, 135.1, 136.0, 136.2, 136.3, 137.4, 137.6, 139.6, 157.1, 157.5; IR (film) 508, 700, 837, 1115, 1252, 1428, 1462, 1698, 2855, 2959, 3445; Anal. Calcd for $C_{45}H_{54}NO_6Si_2$: C, 70.96; H, 7.23; N, 1.84. Found: C, 71.08; H, 7.34; N, 1.75.

Compound 8b as a mixture of diastereoisomers 2:1: Yield 85%, ^{1}H NMR (500 MHz, $CDCl_3$) –0.32 (s, 0.9H), –0.26 (s, 2.1H), –0.21 (s, 0.9H), –0.18 (s, 2.1H), 0.69 (s, 2.7H), 0.71 (s, 6.3H), 1.8–1.4 (m, 4H), 2.37 (s, 0.9H), 2.38 (s, 2.1H), 3.01 (m, 0.6H), 3.1–3.0 (m, 1.4H), 3.32 (dd, J = 11.1, 10.0 Hz, 0.3H), 3.39 (m, 0.3H), 3.54 (dd, J = 11.2, 9.5 Hz, 0.7H), 3.72 (m, 0.7H), 3.9 (dd, J = 11.3, 4.0 Hz, 0.3H), 4.00 (m, 0.7H), 4.1–4.0 (m, 2H), 4.3–4.2 (m, 1H), 4.42 (d, J = 15.5 Hz, 0.3H), 4. 50 (d, J = 15.7 Hz, 0.7H), 7.3–7.2 (m, 6H), 7.4–7.3 (m, 6H), 7.44 (m, 3H), 7.50 (m, 3H), 7.54 (m, 3H), 7.61 (m, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$) –5.57, –5.58, 18.2, 18.3, 21.3, 21.4, 25.8, 36.6, 36.9, 61.2, 61.4, 63.8, 64.4, 66.5, 66.9, 68.4, 68.7, 71.9, 72.0, 127.5, 127.6, 127.7, 127.8, 127.9, 128.0, 128.4, 128.7, 129.2, 129.3, 129.4, 129.8, 130.0, 130.1, 130.2, 133.7, 135.0, 135.2, 135.3, 135.4, 135.5, 135.6, 137.2, 137.5, 137.8, 138.2, 142.8, 143.1; HR LSIMS $C_{44}H_{55}NO_6SSi_2Na$ $(M+Na)^+$ calcd 804.3186, found 804.3199.

Oxidation of diastereoisomeric diols 8 . To a solution of diols **8** (500 mg, 0.63 mmol) in a mixture of toluene (31 ml) and ethyl acetate (31 ml) at 0°C, NaBr (6.3 mg; 0.06 mmol), TEMPO (catalytic amount) and water (350 µl) was added. Then the reaction mixture were vigorously stirred, $NaHCO_3$ (153 mg, 1.8 mmol) and NaOCl (2 ml, 5% active chlorine) were added portionwise (progress of the reaction was controlled by TLC). When the reaction was completed, a reaction mixture was diluted with water and then extracted with ether. Organic phase was dried over Na_2SO_4, evaporated and the residue was used without purification for the next reaction.

Hydrogenation of aldehyde 9. Crude aldehyde **9** was dissolved in methanol (7 ml), palladium (Degussa) was added and the reaction mixture was hydrogenated (*Parr* apparatus) for 28 h. Filtration through a Celite pad, followed by column chromatography (silica, chloroform/methanol) gave two diastereoisomers in 75% yield.

Compound 10: ^{1}H NMR (500 MHz, $CDCl_3$ with D_2O) –0.75 (s, 3H), –0.40 (s, 3H), 0.81 (s, 9H), 1.54 (dt, J_d = 11.6 Hz, J_t = 10.7 Hz, 1H), 2.20 (m, 1H), 2.40 (dd, J = 11.3, 9.7 Hz, 1H), 2.62 (ddd, J = 16.1, 7.7, 3.3 Hz, 1H), 2.94 (m, 1H), 3.10 (ddd, J = 11.3, 4.5, 1.7 Hz, 1H), 3.48 (m, 1H), 3.62 (ddd, J = 10.4, 8.6, 4.4 Hz, 1H), 3.88 (dd, J = 9.7, 3.3 Hz, 1H), 7.4–7.3 (m, 10H), 7.59 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) –5.6, –5.5, 18.2, 25.9, 42.5, 51.4, 62.5, 62.9, 65.8, 68.5, 127.9, 128.0, 130.2, 134.2, 135.5; IR (film) 710, 778, 837, 1067, 1115, 1255, 1428, 1462, 1665, 2855, 2927, 3069, 3356; HR LSIMS $C_{30}H_{42}NO_3Si_2$ $(M+H)^+$ calcd 520.2703, found 520.2702; $[\alpha]_D^{20}$ +16.28 (*c* 1.2).

Compound 11: ^{1}H NMR (500 MHz, $CDCl_3$) –0.05 (s, 3H), 0.00 (s, 3H), 0.83 (s, 9H), 1.60 (m, 1H), 1.94 (bs, 1H), 2.2–2.1 (m, 1H), 2.77 (m, 1H), 2.84 (m, 1H), 3.2–3.1 (m, 1H), 3.50 (m, 1H), 3.97 (m, 2H), 4.2–4.1 (m, 1H), 7.5–7.4 (m, 10H), 7.59 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) –5.6, –5.5, 18.2, 25.9, 40.1, 50.2, 62.4, 63.4, 64.8, 66.0, 127.9, 130.2, 134.1, 135.5; HR LSIMS $C_{30}H_{42}NO_3Si_2$ $(M+H)^+$ calcd 520.2703, found 520.2707; $[\alpha]_D^{20}$ +0.34 (*c* 1.2).

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