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# **ORIGINAL ARTICLE**

# Synthesis of some novel sulfonyl ester derivatives derived from *D*-mannitol

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# **KEYWORDS**

1. Introduction

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Sulfonyl ester; p-Mannitol; Chiral compounds; Bistosylate; Chiral bidentate ligand; Nucleophilic substitution **Abstract** The preparation of sulfonate-derivatives of D-mannitol i.e. 1,2:3,4-di-*O*-isopropylidene-3,4-di-*O*-*p*-toluenesulfonate-D-mannitol (**3a**), 1,2:3,4-di-*O*-isopropylidene-3,4-di-*O*-methanesulfonate-D-mannitol (**3b**), and 1, 2:3,4-di-*O*-isopropylidene-3,4-di-*O*-trifluoromethanesulfonate-D-mannitol (**3c**) is described. Full characterization and methodologies of these sulfonate-D-mannitol derivatives have been described as well.

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The chiral compounds which possess  $C_2$ -symmetry (Orsini<sup>\*</sup> and Pelizzoni, 1996; Whitesell, 1989) such as D-mannitol (2) with terminal protected moieties is quite useful starting material to prepare different types of chiral ligands (Padmakumari Amma and Stille, 1982; Schmid et al., 1991; Hertel et al., 1991). Many features are found in such sugar compounds, for instance: availability, cheap, chirality and being non-toxic (Al-Majid et al., 2003). It is expected that such ligands will chelate to metal complexes which will prevent the dissociation of these metals during the reaction course (Kagan, 1985). The main objective of this work is to prepare new intermediate sulfonate-deriva-

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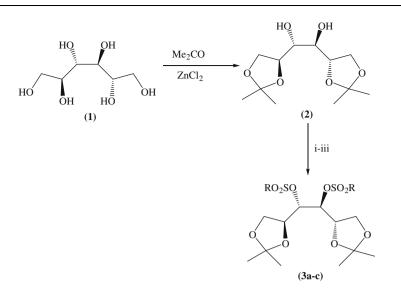
tives  $3(\mathbf{a-c})$  as shown in Scheme 1. It is expected that such sulfonate intermediates at non-protected chiral centres will be good leaving groups to obtain a valuable chiral ligands by various nucleophiles *e.g.*  $R_2P$  (Littke and Fu, 1999; Wolfe et al., 2000; Wolfe, 1999), NH<sub>2</sub> (Nakadai et al., 2002; Rasappan and Reiser<sup>\*</sup>, 2009) and SH (Moore et al., 1990; Wei et al., 2005) groups. These sulfonate intermediates were obtained *via*  $S_N2$  pathway where the nucleophilic substitution was achieved at an oxygen atom rather than a chiral carbon atom.

# 2. Experimental

Melting points were determined on Tottoli capillary melting point apparatus and are uncorrected. IR spectra were recorded on Perkin Elmer FT Spectrophotometer, 1000. <sup>1</sup>H and <sup>13</sup>C NMR spectra were taken on JEOL ECP 400 NMR spectrometer operating at 400 MHz in CDCl<sub>3</sub> and DMSO-d<sup>6</sup> with TMS as internal standard. Mass spectra were carried out on Shimadzn GCMSQP5050A spectrometer, ionization energy 70 eV at college of Science, King Saud University. Polarimeter PO-LAX-2L (ATAGO), LED + inter reference filter (589 nm). Silica gel column chromatography and Sephadex LH-25-100 (micro miter) were used for purification of the some prepared

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i) *p*-CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>Cl (2.0 mmol), CH<sub>2</sub>Cl<sub>2</sub>, Pyridine, N<sub>2</sub>
ii) CH<sub>3</sub>SO<sub>2</sub>Cl (2.5 mmol), Pyridine, N<sub>2</sub>
iii) CF<sub>3</sub>SO<sub>2</sub>Cl (2.4 mmol), CH<sub>2</sub>Cl<sub>2</sub>, Pyridine, N<sub>2</sub>

a) R= *p*-CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub><sup>-</sup> b) R= CH<sub>3</sub> c) R= CF<sub>3</sub>

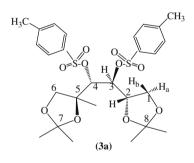
#### Scheme 1

compounds. TLC with UV 254-indicator was used. Reactions involving moisture sensitive compounds (3a-b) were carried out under argon atmosphere. D-Mannitol, anhydrous pyridine, methane sulfonyl chloride, *p*-toluenesulfonyl chloride and trifluoromethanesulfonic anhydride were supplied from Aldrich Chemical Company and used without further purification.

### 2.1. Preparation of 3,4-bis-o-tosyl-1,2:5,6-di-o-isopropylidine-Dmannitol (**3a**)

In a 3-necked round-bottomed flask were added 1,2:5,6-di-oisopropylidine-D-mannitol (Schmid et al., 1991) (10.0 g, 38.16 mmol) and dry dichloromethane (15.0 ml), the mixture was stirred at 25 °C under an inert atmosphere of nitrogen. A mixture of the p-toluenesulfonyl chloride (14.55 g, 76.32 mmol) and freshly-distilled pyridine (50.0 ml) was then added drop wise over a period of 15 min and the reaction mixture was allowed to react at 70 °C for 8 h and then cooled down to the room temperature. The reaction mixture was then extracted with dichloromethane  $(3 \times 70.0 \text{ ml})$  and the organic extracts were washed with 10% CuSO<sub>4</sub> ( $2 \times 50.0$  ml), saturated NaHCO<sub>3</sub> (100.0 ml), H<sub>2</sub>O (100.0 ml) followed by brine (100.0 ml), dried over anhydrous MgSO<sub>4</sub> and filtered off and finally evaporated to yield an oily crude product which when dissolved in a cold diethyl ether and evaporated repeatedly gave the desired bistosylate (3a) as colorless crystals (16.30 g, 28.60 mmol, 75%), m.p 118 °C, [α]<sub>589</sub> –128° (c 0.023, CHCl<sub>3</sub>), IR(KBr): v<sub>max</sub>(cm<sup>-1</sup>): 3090 w, 3040 w, 3015 (C-H, aromatic, str.), 2990 s, 2974 s (C-H alkane, str.), 1371 (SO<sub>2</sub>, asym.str.), 1211 (SO<sub>2</sub>, symm. str.) cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta_{\rm H}$ : 1.27 (6H, s, 2×CH<sub>3</sub>), 1.37 (6H, s, 2×CH<sub>3</sub>), (6H, s, 2×CH3 aromatic), 3.80-3.83 (2H, m, 2×H2), 3.86-3.90 (2H, dd,  ${}^{3}J$  5.58 Hz,  ${}^{3}J$  5.8 4 Hz, 2 × H<sub>a</sub>), 3.97–4.00 (2H, dd,  ${}^{3}J$ 4.4 Hz,  ${}^{3}J$  5.12 Hz, 2×<sub>b</sub>), 4.93–4.95 (2H, d,  ${}^{3}J$  8.08 Hz, 2×H-3), 7.78-7.8 (4H, d, <sup>3</sup>J 8.08 Hz, aromatic) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta_{\rm C}$ : 21.8 (2×CH<sub>3</sub> aromatic), 25.3 (2×CH<sub>3</sub>), 26.7 (2×CH<sub>3</sub>), 66.7 (C-1 and C-6), 72.9 (C-2 and C-5), 78.9 (C-3

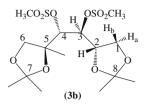
and C-4), 110.2 (C-7 and C-8), 128 (C-11 and C-13), 129.8 (C-10 and C-14), 133.8 (C-12), 145.2 (C-9) ppm; MS: m/z: 571 (M+H)<sup>+</sup> 2.4%, 555 (M-CH<sub>3</sub>)<sup>+</sup> 62.9%.



2.2. Preparation of 3,4-bis-o-mesyl-1,2:5,6-di-o-isopropylidine-*D*-mannitol (**3b**)

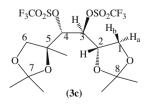
In a 3-necked round-bottomed flask equipped with equalizing funnel were added 1,2:5,6-di-o-isopropylidine-D-mannitol (5.0 g, 19.08 mmol) in dry pyridine (25.0 ml), the mixture was stirred at 0.0 °C under an inert atmosphere of nitrogen. Methanesulfonyl chloride (3.5 ml, 5.20 g, 45.5 mmol) was then added drop wise over a period of 15 min and the reaction mixture was allowed to stir at 0.0 °C for 90 min and kept in the fridge overnight, Water (3.0 ml) was then added, extracted with chloroform  $(3 \times 50.0 \text{ ml})$  and the organic extracts were washed with 10% HCl followed by H<sub>2</sub>O (1×100.0 ml), 5% Na<sub>2</sub>CO<sub>3</sub> (1× 100.0 ml),  $H_2O$  (1 × 100.0 ml) followed by brine (100.0 ml), dried over anhydrous MgSO4 and filtered off and evaporated to yield yellow oily crude product which upon dissolved in a cold diethyl ether and evaporated repeatedly gave colorless crystals of the desired bismesylate (3b) (7.11 g, 17.01 mmol, 88%), m.p 130 °C,  $[\alpha]_{589}$  –97.0° (c 0.023, CHCl<sub>3</sub>), IR(KBr):  $v_{max}$ (cm<sup>-1</sup>): 2990 s, 2953 s, 2940 m (C-H alkane, str.), 1418, 1455, 1424 (C-

H alkane, def.), 1347 (SO<sub>2</sub>, asym. str.), 1219 (SO<sub>2</sub>, symm. str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta_{\rm H}$ : 1.34 (6H, s, 2 × CH<sub>3</sub>): 1.42 (6H, s, 2 × CH<sub>3</sub>), 3.16 (6H, s, 2 × SCH<sub>3</sub>), 4.16 (2H, d, <sup>3</sup>J 5.12 Hz, 2 × H<sub>a+b</sub>), 4.26 (2H, m, 2 H-2), 4.95 (2H, <sup>3</sup>J 7.36 Hz, 2 H-3) ppm; <sup>13</sup>C NMR(CDCl<sub>3</sub>):  $\delta_{\rm C}$ : 25.2 (2 × CH<sub>3</sub>), 26.6 (2 × CH<sub>3</sub>), 39.0 (2 × SCH<sub>3</sub>), 66.5 (C-1 and C-6), 73.5 (C-2 and C-5), 79.0 (C-3 and C-4), 110.3 (C-7 and C-8) ppm MS: *m/z*: 419 (M+H)<sup>+</sup> 0.2%, 403 (M-CH<sub>3</sub>)<sup>+</sup> 44.2%.



2.3. Preparation of 3,4-bis-o-trifyl-1,2:5,6-di-o-isopropylidine-*D*-mannitol (**3c**)

In a 3-necked round-bottomed flask equipped with equalizing funnel were added 1,2:5,6-di-o-isopropylidine-D-mannitol (3.0 g, 11.45 mmol) and dichloromethane (25 ml), the mixture was stirred for 10 min at 0.0 °C under an inert atmosphere of nitrogen, dry pyridine (5.0 ml) was then added. A mixture of trifluoromethanesulfonic anhydride (4.8 ml. 8.0 g. 28.60 mmol) in dry pyridine (10.0 ml) was added drop wise over a period of 15 min and the reaction mixture was allowed to stir at 25.0 °C for 90 min and then poured into cold water (30 ml), extracted with dichloromethane  $(3 \times 50.0 \text{ ml})$  and the organic extracts were washed by  $H_2O$  (1 × 100.0 ml), dried over anhydrous MgSO4 and filtered off and evaporated to yield an oily crude product, kept in the fridge for 48 h. The crude product was then dissolved in cold diethyl ether and evacuated repeatedly gave the desired ditriflate (3c) as colorless crystals (3.70 g, 7.03 mmol, 62%), m.p 94 °C, IR(KBr): v<sub>max</sub>(cm<sup>-1</sup>): 2992 s, 2937 s, 2903 m (C-H alkane, str.), 1406 (C-H alkane, def.), 1390 (SO<sub>2</sub>, asym. str.), 1242 (CF<sub>3</sub>, str.), 1215 (SO<sub>2</sub>, symm. str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta_{\rm H}$ 1.35(6H, s, 2×CH<sub>3</sub>); 1.43 (6H, s, 2×CH<sub>3</sub>), 4.03–4.06 (2H, d,  ${}^{3}J$  4.4 Hz, 2 × H<sub>a</sub>), 4.16–4.20 (2H,dd,  ${}^{3}J$  5.84 Hz,  ${}^{3}J$  5.88 Hz,  $2 \times H_{\rm b}$ ), 4.25–4.30 (2H, <sup>3</sup>J 7.36 Hz, 2H-2), 5.20–5.23 (2H,d,  $^{3}J$  8.04 Hz, 2H-3) ppm;  $^{13}C$  NMR (CDCl<sub>3</sub>):  $\delta_{C}$ : 24.7 (2×CH<sub>3</sub>), 26.6 (2×CH<sub>3</sub>), 66.7 (C-1 and C-6), 72.0 (C-2 and C-5), 82.2 (C-3 and C-4), 111.0 (C-7 and C-8), 116.7-120.0 (2CF<sub>3</sub>) ppm; <sup>19</sup>F NMR (CDCl<sub>3</sub>):  $\delta_{\rm F}$  –74.36 ppm, *m/z* (MS):  $527 (M + H)^+ 0.6\%, 511 (M - CH_3)^+ 100\%.$ 



# 3. Results and discussion

In order to prepare chiral sulfonate-intermediates derived from D-mannitol (**3a**-c) the both two terminal hydroxyl groups of D-mannitol (**1**) need to be protected. The resulting terminal ke-

tal-protected diol (2) is a well-known compound (Padmakumari Amma and Stille, 1982; Schmid et al., 1991; Hertel et al., 1991) and will be the key step to prepare different types of chiral bidentate ligands (Schmid et al., 1991; Hertel et al., 1991; Al-Majid et al., 2003; Kagan, 1985; Littke and Fu, 1999; Wolfe, 1999; Nakadai et al., 2002; Rasappan and Reiser<sup>\*</sup>, 2009), so the starting material *i.e.* diol (2) was prepared according to the procedure described in the literature (Schmid et al., 1991).

One of the sulfonate-derivatives derived from D-mannitol is the preparation of tosylate ester (3a). It is known that the tosyl group p-CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>SO<sub>2</sub> is electron-withdrawing (Todd Whitaker et al., 2006). Hence, it is an excellent leaving group, and toluenesulfonyl chloride activates the alcohol (2) by nucleophilic attack at positively sulfur atom of the sulfonyl group. Thus, when the diol (2) was treated with two equivalents of *p*-toluenesulfonvl chloride in the presence of a moderate base such as pyridine (Kazemi et al., 2007) and heated at 70 °C for 8 h under an inert atmosphere conditions, the crystalline colorless product of the bistosylates (3a) was obtained in a reasonable yield (75%). The resulting product showed an optically activity  $[\alpha]_{589}$  -128°, in chloroform maintaining the direction of the rotation which confirmed that the substitution has taken place at an oxygen atom rather than at the chiral carbon atom. The spectral data of (3a) supported the formation of the desired product. IR spectrum showed the familiar stretching bands of the phenyl ring at 3170 and 3090 cm<sup>-1</sup> and the stretching band of the new formed sulfonyl group was observed at 1371 cm<sup>-1</sup>. Additionally IR showed the absence of absorption band for the OH group. In the <sup>1</sup>H NMR spectrum, the four methyl groups of the ketal moieties appeared as two singlets at  $\delta$  1.27 and 1.37 ppm while the absorption of the methyl group of the aromatic ring was seen as singlet at  $\delta$  2.43 comparisons with those seen of the ketal moieties. A multiplet signal of  $\delta$  3.8– 3.83 ppm represent the H-2 was obtained, while the H-3 was observed as doublet (d) at  $\delta$  4.93–4.95 ppm. The characterized peaks of the aromatic ring were shown as two doublets at  $\delta$ 7.29–7.31 and at  $\delta$  7.78–7.80 ppm following the pattern  $AA^{-}XX^{-}$ . Finally the mass spectrum of confirmed the proposed structure of bistosylate (3a) by obtaining the m/z: 555 for the molecular ion  $[M-CH_3]^+$  with 63% intensity and m/z: 571 for the molecular ion  $[M + H]^+$  with 2.4% intensity. Addition molecular fragmental ions [CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>O<sub>2-</sub> SO]<sup>+</sup> and  $[C_6H_4CH_3]$  were obtained at m/z: 155 with 60% intensity and m/z: 91 with intensity 73% respectively. The ionization fragment of the ketal moiety was given at m/z: 101 with 100% intensity.

The second sulfonate-derivative *i.e.* bismesylate ester (**3b**) is considered too as an excellent leaving group in nucleophilic substitution reactions. Thus, bismesylate (**3b**) (Coates and Chen, 1969; Crossland and Servis, 1970) was prepared from the reaction of the diol (**2**) with an excess amount of methane sulfonyl chloride at 0.0 °C in the presence of freshly-distilled pyridine for 90 min under an anhydrous conditions to avoid any possible hydrolysis might be resulted. The precise mechanism for the reaction has not been fully determined. It appears that the mechanism is dependent on the substrate alcohol and the reaction conditions. A good yield (88%) was achieved, and the compound showed an optically activity maintaining the direction of optical rotation as reported earlier. All analytical data have supported the desired product: IR showed completely disappeared of the familiar peak of OH group, it is also showed the stretching band of the asymmetric SO<sub>2</sub> at 1374  $\text{cm}^{-1}$  in addition to the symmetric one at  $1219 \text{ cm}^{-1}$ . The formation of bismesylate (3b) has been confirmed by <sup>1</sup>H NMR spectrum, thus, the four methyl groups of the ketal moieties were seen as two singlets at  $\delta$  1.34 and 1.42 ppm. The methyl group of the mesylate was also obtained as singlet with more down field at  $\delta$  3.16 ppm. The methylene protons have followed a ABX pattern *i.e.* the  $H_a$  was seen as doublet at  $\delta$  4.16 with J 8.3 Hz and the  $H_b$ was also seen as doublet at d 4.174 at the same J value. A multiplet peak was given for assignment of the H-2 and H-5, while the H-3 and H-4 absorption were obtained as a doublet at 4.95 with J 7.36 Hz. The  $^{13}$ C NMR in denaturated chloroform showed all the required seven carbon lines for  $C_2$ -symmetry of the molecule as follows:  $\delta_{\rm C}$ : 25.2 and d 26.6 (all four methyl groups of the terminal ketals), 38.9 (SCH<sub>3</sub>) 66.5 (C-1 and C-6), 73.5 (C-2 and C-5), 79.0 (C3 and C4) and 110.3 for C-7 and C-8 ppm. Finally the desired product of the mesylate (3b) was confirmed by mass spectrum by giving the molecular ion  $[M-CH_3]^+$  at m/z: 403 with 44% intensity.

The third sulfonate group is the preparation of the trifluoromethanesulfonate (triflate) (**3c**). A triflate group  $CF_3SO_3$  is an excellent leaving group used in certain organic reactions such as *Suzuki coupling* (Miyaura and Suzuki, 1995; Saito and Fu, 2007) and *Heck reaction* (Heck, 1982). Since alkyl triflate is extremely reactive in  $S_N2$  reactions, they must be stored in cold–dry conditions to preventing from nucleophiles such as

H<sub>2</sub>O. The anion 
$$||_{F_3C} = 0^{-1}$$
 owes stability to reserve stability

ization which causes the negative charges to be spread over the three oxygen atoms and the sulfur atom. An additional stabilization is achieved by the trifluoromethyl group as strong electron-withdrawing group (Kobayashi, 1999; Dubreail et al., 1999; Netscher and Bohrer, 1996).

When 3,4-diol-D-mannitol (2) was treated with an excess of triflic anhydride in the presence of dichloromethane and pyridine at 0.0 °C. Because of highly moisture sensitivity of the triflic anhydride and the expected product (3c), the reaction was carried out in an inert atmosphere of argon. After a period of 90 min a colorless crystalline product of the bistriflates (3c) was obtained in 61%. The resulting product was confirmed by standard analytical methods; Infra red spectrum showed a strong band at 1242 cm<sup>-1</sup> represents the stretching vibration of the trifyl group, a stretching band of the asymmetric  $S(=O)_2$  at 1390 cm<sup>-1</sup> was observed. The OH group peak was absent confirming that the nucleophilic substitution has taken place. <sup>1</sup>H NMR spectrum showed two singlet peaks of the terminal isoproplidene moieties at  $\delta$  1.35 and 1.43 ppm. A doublet of doublet (dd) at  $\delta$  4.03–4.06 was observed which represent H<sub>a</sub>. Another doublet of doublet (dd) was also seen at  $\delta$  4.16–4.20 for H<sub>b</sub>. A multiplet was obtained in the region of  $\delta$  4.25–4.30 representing the H-2, while H-3 was seen as duplet (d) in the range of  $\delta$  5.20–5.23. The triflate was also confirmed by <sup>13</sup>C NMR by obtaining the expected chemical shifts as follows:  $\delta_{\rm C}$ : 24.7 and 26.6 represent the four methyl groups of the two terminal ketals. The two methylene carbons (C-1 and C-6) were appeared at  $\delta_{\rm C}$  66.7 and the asymmetric carbons (C-2 and C-5) were given at  $\delta_{\rm C}$  72.0. The other asymmetric methine carbons (C-3 and C-4) were observed at  $\delta_{\rm C}$  82.2. The peak of C-7 and C-8 was seen at  $\delta_{\rm C}$  111.0. The remaining two lines at  $\delta_{\rm C}$  74.4 were attributed to the CF<sub>3</sub> group. Additionally the <sup>19</sup>F NMR spectrum showed a single line at  $\delta_{\rm F}$  74.4 ppm confirming that the formation of bistriflates (**3c**) has been taken place.

Finally the resulting bistriflates product (3c) was confirmed by mass spectrum by giving the molecular ion  $[M-CH_3]^+$  at m/z: 511 with 100% integration.

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