Synthesis and Application of *N*-Methoxy-*N*-methyl-2-phenylsulfonylacetamide as a Two-Carbon Homologating Agent

N. Satyamurthi,[a] Jaimala Singh,[b] Indrapal Singh Aidhen*[a]

[a] Department of Chemistry, Indian Institute of Technology, Madras, Chennai-600 036, India
Fax +91(44)2350509; E-mail: isingh@pallava.iitm.ernet.in
[b] Division of Organic Chemistry, Central Leather Research Institute, Chennai-600 020, India

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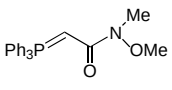
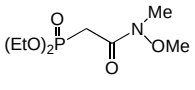
Abstract: With the well-known precedence that *N*-methoxy-*N*-methyl amides are excellent acyl cation and aldehyde equivalents, *N*-methoxy-*N*-methyl-2-phenylsulfonylacetamide **(3)**, a new reagent, synthetically equivalent to $^{\ominus}CH_2CHO$ and $^{\ominus}CH_2COR$ was synthesised. The simplicity involved in the alkylation at the active methylene site in **3**, followed by safe removal of the phenylsulfonyl group, makes **3** a versatile reagent for two-carbon homologation of alkyl halides. The method, when applied to sugar halides **6j** and **6k** led to the synthesis of 2,3-dideoxy sugars.

Key words: Weinreb amides, two-carbon homologation, higher 2,3-dideoxy sugars, (+)-aspicillin, sulfones, desulfonylation, alkylations

Since the initial report of Weinreb on the preparation and use of *N*-methoxy-*N*-methyl amides,[1] these amides, now popularly called Weinreb amides have displayed a remarkable selectivity in their reactions with organometallic reagents. Even with a large excess of organomagnesium or organolithium species, these amides exclusively yield ketones. Little or no double addition occurs due to effective chelation of the metal ion between carbonyl oxygen and *N*-methoxy oxygen atoms which prevents the collapse of the tetrahedral intermediate until aqueous acidic workup. Following Weinreb's initial report, these amides have served as valuable intermediates in variety of synthetic endeavours[2] and have been put to elegant use in synthesising key fragments of important molecules.[3] Evans[4] combining the usefulness of these amides and the phosphorane chemistry developed **1** whereas Siedel[5] and Nullizard[6] independently reported the Emmons−Horner−Wadsworth variant **2**. Although both reagents **1** and **2** have served as useful homologating agents for chain extension,[7] they have been seldom used in the homologation of sugars.



1



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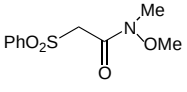
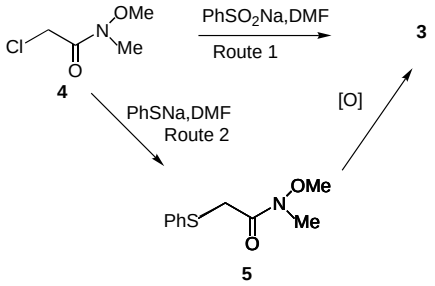


3

Herein, we report the synthesis and utility of a new Weinreb amide based synthetic equivalent **3** for $^{\ominus}CH_2CHO$ and $^{\ominus}CH_2COR$ synthons. Reagent **3** combines the usefulness of Weinreb amide functionality and the sulfone chemistry.[8] The methylene group doubly activated by phenylsulfonyl group and other electron-withdrawing groups (COR, CO_2R, CN) have been routinely alkylated under mild conditions.[9] Based on this precedence it was envisaged that clean alkylation of **3** and subsequent desulfonylation[10] would give a two-carbon homologated product with a valuable handle in the form of Weinreb amide functionality. Alkylation of **3** under mild conditions, avoiding the need of a strong base is of paramount importance as *N*-methoxy-*N*-methyl amides have been shown to be unstable in strongly basic medium liberating formaldehyde either by retro-ene reaction of their enolates or by direct E2 elimination.[11]

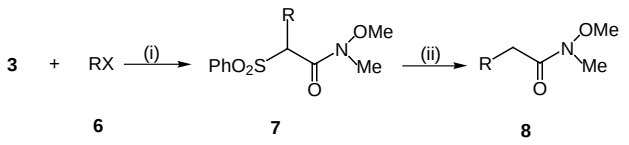
Reagent **3** is a crystalline solid easily prepared on a large scale by two different routes as depicted in Scheme 1. Route 1 involves a direct S_N2 reaction of phenyl sulfinate anion on the known *N*-methoxy-*N*-methyl-2-chloroacetamide **(4)**[12] whereas in the second route first the sulfide **5** is prepared by the reaction between sodium phenylmercaptide and **4** in DMF. The sulfide **5** is then oxidized to sulfone **3** using 30% H_2O_2 solution in a mixture of acetic anhydride and acetic acid. Both these routes are equally efficient. Reagent **3** is a stable crystalline solid and can be stored indefinitely at room temperature.



Scheme 1

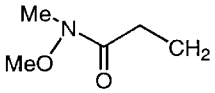
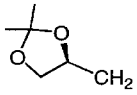
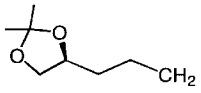
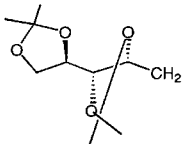
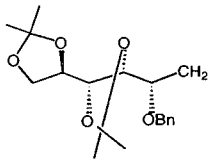
Reagent **3** undergoes clean alkylation (Scheme 2) with various electrophiles **6a−k** under mild conditions of K_2CO_3 in DMF at temperatures ranging from ambient to 80°C. Except with 1,2-dibromoethane (**6e)** wherein a cyclopropane ring was formed during alkylation, in all cases only monoalkylation occurred in good yields. The monoalkylated products after purification by flash chromatography were directly subjected to desulfonylation us-

ing Na(Hg), Na_2HPO_4 in MeOH.[13] A clean reaction ensued yielding the corresponding desulfonylated products again in good yields.



Reagents and conditions: (i) K_2CO_3/anhyd DMF (ii) Na(Hg)/Na_2HPO_4/anhyd MeOH, 0°C, 1.5 h

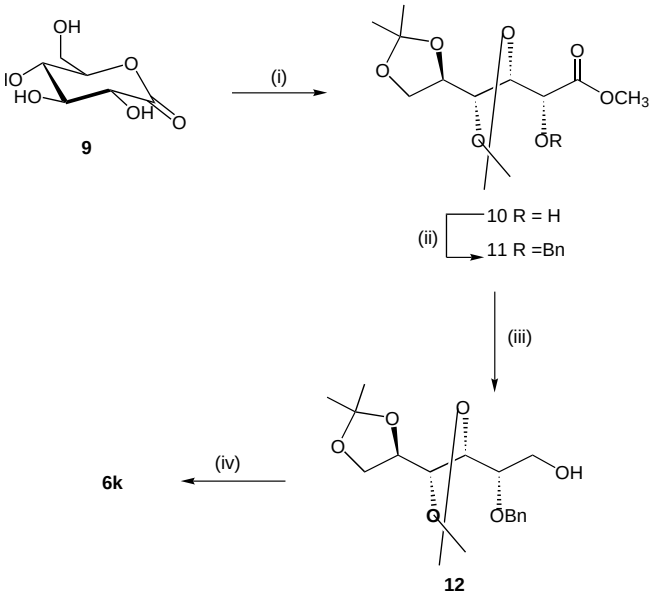
Scheme 2

6, 7, 8	R
a	C_4H_9
b	$H_2C{=}CHCH_2$
c	$HC{\equiv}CCH_2$
d	$C_6H_5CH_2$
e	CH_2CH_2
f	$PhOCH_2CH_2$
g#	
h	
i#	
j	
k	

X = Cl for **8b**, **8d**; X = Br for **8a**, **8c**, **8e**, **8f**, **8g**
X = I for **8h**, **8i**, **8j**, **8k**

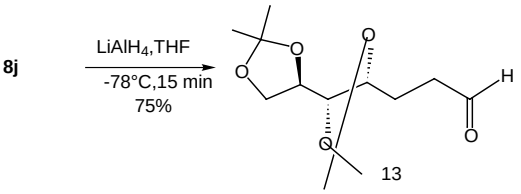
For Preparation see Ref. 20.

The successful application of two-carbon homologation is extremely useful in the light of recent failure[14] to homologate terminal halogeno deoxy sugars by two carbons. In the case of electrophiles **6h**[15],**6j**[16] and **6k** which represents alkyl iodides having a stereogenic centre at the α-carbon, no reaction occurred at room temperature, probably due to steric factors. However, clean reaction ensued as the temperature was raised. Successful reactions particularly with electrophiles **6j** (D-*arabino*-configurated) and **6k** (D-*gluco*-configurated) illustrated the application to homologation of sugars.[17] Electrophile **6k** was prepared in five steps starting from D-(+)-glucono-1,5-lactone[18] **(9)** as depicted in Scheme 3. Interestingly, reduction of the homologated product **8j** furnished the *arabino*- configurated heptose **13**, thereby (Scheme 4) providing an efficient synthesis of the C-3−C-9 fragment of a natural product (+)-aspicillin.[19]



Reagents and conditions: (i) Ref. 16 (ii) Ag_2O/BnBr in anhyd Et_2O at r.t.; 82% (iii) $LiAlH_4$ in THF at r.t.; 85% (iv) I_2/imidazole/Ph_3P/toluene, reflux, 45%

Scheme 3



Scheme 4

In summary, a new reagent **3** has been developed which undergoes clean alkylation with various primary halides in good yields. Subsequent desulfonylation leads to a two carbon homologated product containing a valuable and stable functionality for further synthetic endeavours. Further application of this method to the iodo sugar electrophiles led to the synthesis of the higher 2,3-dideoxy sugars.

All the solvents and reagents were distilled before use. Anhyd solvents were prepared using standard procedures. Melting points were determined in capillary with a Toshniwal melting point apparatus and are uncorrected. NMR spectra were recorded on Bruker (200 MHz ^{1}H, 50 MHz ^{13}C) or Jeol (400 MHz ^{1}H, 100 MHz ^{13}C) NMR spectrometers using TMS as a reference compound. IR spectra were recorded on Shimadzu IR 470 or Bruker IFS 66V FTIR spectrometers. Electron-Impact mass spectra and HRMS were accomplished at 70 eV using a Finnigan MAT 8230 spectrometer. Microanalysis were performed on a Heraeus CHN analyser. Optical rotations were measured with a Autopol II polarimeter at r. t. For monitoring the formation of the alkylated products **7h–k** and the desulfonylated products **8h–k**, TLC was performed on precoated silica gel plates (Merck 5554) by dipping in a solution prepared by adding cerium(IV) sulfate (1 g), and ammonium molybdate (21 g) to concd H_2SO_4 (31 mL) and made up to 500 mL with H_2O. The TLC plates were later heated to 100°C for development. For compounds **7a–g** and **8a–g**, the TLC were performed on glass plates coated with silica gel (7 cm × 2.5 cm) followed by staining in I_2 vapours.

***N*-methoxy-*N*-methyl-2-phenylsulfonylacetamide (3)**

Method 1: To a solution of $PhSO_2Na$ (9.02 g, 55 mmol) in anhyd DMF (20 mL) was added a solution of **4** (6.87 g, 50 mmol) in DMF (20 mL) and the mixture was stirred at r.t. for 24 h. After complete disappearance of the starting material (TLC), H_2O (50 mL) was added to the solution and extracted with EtOAc (3 × 15mL). The combined organic layers were washed with H_2O (2 × 15mL) and dried (Na_2SO_4). Concentration of the organic extract gave 9.72 g (80%) of the product **3**. The product obtained was pure and was recrystallised from CH_2Cl_2 and hexane; mp 82–84°C.

Method 2: To a solution of thiophenol (0.121 g, 1.1 mmol) in anhyd DMF (2 mL) was added NaH (0.026 g, 1.1 mmol) and was stirred at 0°C for 20 min under N_2 atm. To the above solution was added dropwise **4** (0.137 g, 1 mmol) in DMF (1 mL) at 0°C and the mixture was warmed up to r.t. After the complete consumption of **4,** as monitored by TLC (R_f 0.7 in 7:3 hexane/EtOAc), aq sat. NH_4Cl (5 mL) was added at 0°C and then diluted with H_2O (10 mL). The mixture was extracted with EtOAc (3 × 5 mL). The combined organic layers were washed with aq NaOH solution (2 × 10 mL), H_2O (2 × 10 mL), dried (Na_2SO_4) and concentrated to give 0.151 g (72%) of the product **5**. To the crude product (0.211 g, 1 mmol) in a mixture of AcOH and Ac_2O (10 mL, 8:2) was added 30% H_2O_2 (1 mL) and the mixture was stirred for 8 h at r. t. The mixture was then treated with H_2O (20 mL) and extracted with EtOAc (2 × 10 mL). The combined organic layers were washed with $NaHCO_3$ (2 × 10 mL), H_2O (2 × 10 mL) and dried (Na_2SO_4). Evaporation of the solvent gave the product **3**, 0.179 g (74%); R_f 0.24 (hexane/EtOAc, 1:1); mp 82–84°C.

Table 1 Compounds **7a–k** Prepared

Product[a]	Time (h)	Temp (°C)	Yield (%)	mp (°C)	IR ($CHCl_3$) ν (cm^{-1})	EI-MS (70 eV) *m/z* (%)
7a	24	r.t.	76	73–75	2944, 2849, 1670, 1315, 1145	299 (8), 239 (23), 141 (52), 77 (47), 61 (100), 55 (18)
7b	12	r.t.	79	108–110	3008, 1664, 1584, 1315, 1145	283 (2), 223 (11), 142 (11), 141 (50), 125 (39), 61 (100), 77 (86)
7c	2	r.t.	86	113–115	3296, 2976, 1670, 1600, 1500, 1308, 1145	281 (2), 221 (6), 141 (16), 77 (55), 61 (100), 51 (32)
7d	6	r.t.	75	104–106	2992, 1664, 1584, 1523, 1305, 1142	333 (1), 273 (7), 192 (29), 141 (5), 132 (15), 131 (28), 104 (20), 103 (25), 91 (38), 77 (100), 61 (19), 60 (15)
7e	24	r.t.	80	68–70	2944, 2864, 1670, 1584, 1520, 1315, 1139	269 (6), 209 (84), 141 (23), 77 (100), 61 (16)
7f	24	r.t.	72	73–75	2944, 1670, 1600, 1488, 1443, 1312, 1145	363 (4), 270 (100), 242 (6), 162 (10), 141 (9), 129 (22), 77 (71), 61 (28), 55 (13)
7g	24	r.t.	67	syrup	2992, 2944, 1651, 1452, 1300, 1142	358 (<1), 298 (100), 237 (16), 195 (25), 77 (25), 61 (40), 55 (52)
7h	30	60	68	syrup	2950, 2900, 1676, 1500, 1452, 1315, 1161	357 (<1), 342 (27), 297 (20), 239 (40), 141 (46), 97 (60), 77 (98), 61 (100)
7i	12	r.t.	72	syrup	2950, 2900, 1660, 1500, 1452, 1315, 1161	385 (<1), 370 (20), 325 (12), 310 (30), 267 (32), 244 (16), 143 (38), 141 (30), 125 (58), 115 (10), 109 (74), 85 (20), 77 (100), 61 (56), 55 (30)
7j	30	60	68	syrup	2960, 1660, 1440, 1366, 1145	457 (<1), 442 (10),338 (12), 98 (8), 85 (46), 81 (44), 77 (100), 61 (24), 57 (84), 55 (47)
7k	30	80	56	syrup	2944, 1651, 1595, 1440, 1369, 1300, 1142, 1075	577 (<1), 562 (2), 312 (8), 237 (6), 192 (12), 143 (20), 125 (24), 101 (9), 91 (100), 77 (15), 61 (12)

[a] The compounds **7a–k** were characterised by their ^{1}H and ^{13}C NMR spectra and were directly subjected to desulfonylation.

Table 2 ^{1}H and ^{13}C NMR of Compounds **7a–k**

Product	^{1}H NMR ($CDCl_3$/TMS); δ, *J* (Hz)	^{13}C NMR ($CDCl_3$/TMS)
7a	0.86 (t, 3 H, *J* = 6.8, C*H*$_3$), 1.18–1.30 (m, 4 H, C*H*$_2$C*H*$_2$CH$_3$), 1.60– 1.72 (m, 1 H, C*H*$_2$CHSO$_2$Ph), 1.85–1.95 (m, 1 H, C*H*$_2$CHSO$_2$Ph), 3.19 (s, 3 H, NCH$_3$), 3.84 (s, 3 H, OCH$_3$), 4.60 (dd, 1 H, *J* = 8.3, 3.4, C*H*SO$_2$Ph), 7.50–7.58 (m, 2 H, Ar-H), 7.60–7.66 (m, 1 H, Ar-H), 7.80–7.86 (m, 2 H, Ar-H)	13.63, 22.01, 27.68, 28.74, 31.99, 61.36, 65.04, 128.15, 130.11, 133.40, 136.79, 165.96
7b	2.46–2.55 (m, 1 H, C*H*$_2$CHSO$_2$Ph), 2.63 (m, 1 H, C*H*$_2$CHSO$_2$Ph), 3.07 (s, 3 H, NCH$_3$), 3.66 (s, 3 H, OCH$_3$), 4.65 (dd, 1 H, *J* = 11.8, 3.4, C*H*SO$_2$Ph), 4.95–5.05 (m, 2 H, CH=C*H*$_2$), 5.50–5.60 (m, 1 H, C*H*=CH$_2$), 7.45–7.52 (m, 2 H, *J* = 7.32, Ar-H), 7.57–7.62 (m, 1 H, Ar-H), 7.79–7.84 (m, 2 H, *J* = 7.3, Ar-H)	31.82, 32.17, 61.68, 64.67, 118.87, 128.66, 129.83, 131.79, 134.08, 136.63, 165.31
7c	2.01 (dd, 1 H, *J* = 2.8, 1.4, C≡CH), 2.84–2.95 (m, 2 H, C*H*$_2$CHSO$_2$Ph), 3.16 (s, 3 H, NCH$_3$), 3.77 (s, 3 H, OCH$_3$), 4.84 (t, 1 H, *J* = 7.3, C*H*SO$_2$Ph), 7.54–7.62 (m, 2 H, 7.81, Ar-H), 7.66–7.72 (m, 1 H, Ar-H), 7.86 – 7.90 (m, 2 H, Ar-H)	17.83, 32.31, 62.08, 63.55, 71.35, 78.29, 128.97, 129.76, 134.48, 136.38, 164.45
7d	2.96 (s, 3 H, NCH$_3$), 3.16 (t, 1 H, *J* = 12.7, C*H*$_2$Ph), 3.23 (s, 3 H, OCH$_3$), 3.33 (dd, 1 H, *J* = 12.7, 2.9, C*H*$_2$Ph), 4.85 (dd, 1 H, *J* = 12.7, 2.9, C*H*SO$_2$Ph), 7.10–7.30 (m, 5 H, Ar-H), 7.54–7.64 (m, 2 H, Ar-H), 7.68–7.74 (m, 1 H, Ar-H), 7.92–8.00 (m, 2 H, Ar-H)	32.04, 33.48, 61.35, 66.87, 127.15, 128.70, 128.76, 128.98, 129.92, 134.13, 136.01, 136.89, 165.13
7e	1.43 (dd, 2 H, *J* = 5.9, 7.8, CH*H*'CH*H*'), 1.77 (dd, 2 H, *J* = 5.4, 7.8, C*H*H'C*H*H'), 3.37 (s, 3 H, NCH$_3$), 3.76 (s, 3 H, OCH$_3$), 7.52–7.58 (m, 2 H, Ar-H), 7.62–7.80 (m, 1 H, Ar-H), 7.84–7.90 (m, 2 H, Ar-H)	13.74, 32.49, 45.20, 60.75, 128.56, 128.95, 133.84, 138.80, 164.90
7f	2.30–2.39 (m, 1 H, C*H*$_2$CH$_2$OPh), 2.48–2.56 (m, 1 H, C*H*$_2$CH$_2$OPh), 3.11 (s, 3 H, NCH$_3$), 3.64 (s, 3 H, OCH$_3$), 3.80–3.86 (m, 1 H, C*H*$_2$OPh), 4.02–4.07 (m, 1 H, C*H*$_2$OPh), 5.03 (dd, 1 H, *J* = 11.2, 3.4, C*H*SO$_2$Ph), 6.77–6.81 (m, 2 H, Ar-H), 6.89–6.95 (m, 1 H, Ar-H), 7.20–7.26 (m, 2 H, Ar-H), 7.52–7.57 (m, 2 H, Ar-H), 7.63–7.68 (m, 1 H, Ar-H), 7.87–7.91 (m, 2 H, Ar-H)	27.45, 32.28, 61.56, 62.44, 64.12, 114.26, 121.11, 128.74, 129.45, 129.73, 134.10, 136.89, 158.08, 165.32
7g	2.11–2.18 (m, 1 H, C*H*$_2$CHSO$_2$Ph), 2.31–2.43 (m, 2 H, CH$_2$CO), 2.59–2.63 (m, 1 H, C*H*$_2$CHSO$_2$Ph), 3.13 (s, 3 H, NCH$_3$), 3.15 (s, 3 H, NCH$_3$), 3.64 (s, 3 H, OCH$_3$), 3.71 (s, 3 H, OCH$_3$), 4.91 (dd, 1 H, *J* = 9.3, 4.9, C*H*SO$_2$Ph), 7.53–7.57 (m, 2 H, Ar-H), 7.64–7.68 (m, 1 H, Ar-H), 7.89–7.91 (m, 2 H, Ar-H)	22.62, 28.17, 32.13, 61.08, 61.57, 63.97, 128.64, 129.56, 133.91, 137.05, 165.60, 172.32
7h[a]	1.16 (s, 3 H, CH$_3$), 1.17 (s, 3 H, CH$_3$), 1.21 (s, 3 H, CH$_3$), 1.26 (s, 3 H, CH$_3$), 2.02–2.21 (m, 2 H, C*H*$_2$CHSO$_2$Ph), 3.05 (s, 3 H, NCH$_3$), 3.42–3.48 (m, 1 H, OCH), 3.66 (s, 3 H, OCH$_3$), 3.82–4.10 (m, 2 H, OCH$_2$), 4.72 (dd, 0.4 H, *J* = 9.2, 3.4, C*H*SO$_2$Ph), 4.86 (dd, 0.6 H, *J* = 11.7, 2.9, C*H*SO$_2$Ph), 7.45–7.52 (m, 2 H, Ar-H), 7.57–7.64 (m, 1 H, Ar-H), 7.77–7.84 (m, 2 H, Ar-H)	25.25, 26.34, 26.74, 31.28, 31.90, 32.07, 32.28, 60.09, 61.29, 61.92, 62.58, 68.54, 68.74, 71.99, 73.58, 109.13, 109.21, 128.51, 128.59, 129.26, 129.56, 133.90, 136.50, 136.79, 164.78, 165.93
7i	1.31 (s, 3 H, CH$_3$), 1.36 (s, 3 H, CH$_3$), 1.39–1.60 (m, 2H, CHCH$_2$C*H*$_2$), 1.85–1.90 (m, 2 H, OCHC*H*$_2$), 1.96–2.04 (m, 2 H, C*H*$_2$CHSO$_2$Ph), 3.18 (s, 3 H, NCH$_3$), 3.42–3.47 (m, 1 H, OCH), 3.79 (s, 3 H, OCH$_3$), 3.96–4.09 (m, 2 H, OCH$_2$), 4.67 (dd, 1 H, *J* = 11.2, 3.4, C*H*SO$_2$Ph), 7.53–7.57 (m, 2 H, Ar-H), 7.65–7.69 (m, 1 H, Ar-H), 7.80–7.88 (m, 2 H, Ar-H)	23.05, 23.26, 25.63, 26.88, 27.73, 27.89, 32.27, 33.04, 33.28, 61.73, 65.07, 65.13, 69.18, 69.26, 75.25, 75.53, 109.85, 128.70, 129.90, 134.09, 136.77, 165.77
7j[a]	1.26 (s, 3 H, CH$_3$), 1.27 (s, 3 H, CH$_3$), 1.32 (s, 3 H, CH$_3$), 1.34 (s, 3 H, CH$_3$), 2.03–2.17 (m, 1 H, C*H*$_2$CHSO$_2$Ph), 2.41–2.49 (m, 1 H, C*H*$_2$CHSO$_2$Ph), 3.14 (s, 3 H, NCH$_3$), 3.44–3.56 (m, 1 H, OCH), 3.75 (s, 3 H, OCH$_3$), 3.78 (s, 3 H, OCH$_3$), 3.88–4.11 (m, 4 H, OCH$_2$, 2 × OCH), 4.92 (dd, 0.35 H, *J* = 9.2, 3.4, C*H*SO$_2$Ph), 4.97 (dd, 0.65 H, *J* = 9.3, 2.9, C*H*SO$_2$Ph), 7.52–7.59 (m, 2 H, Ar-H), 7.64–7.67 (m, 1 H, Ar-H), 7.86–7.89 (m, 2 H, Ar-H)	25.10, 25.24, 26.70, 26.77, 26.94, 27.26, 29.70, 31.14, 31.91, 32.45, 32.74, 61.50, 62.18, 63.22, 67.42, 67.72, 76.29, 76.50, 76.83, 77.35, 78.50, 81.17, 81.52, 109.44, 109.56, 109.69, 128.62, 128.70, 129.81, 130.16, 134.06, 136.74, 136.99, 165.58, 166.68
7k[a]	1.33 (s, 3 H, CH$_3$), 1.37 (s, 3 H, CH$_3$), 1.38 (s, 3 H, CH$_3$), 1.41 (s, 3 H, CH$_3$), 2.10–2.40 (m, 1 H, C*H*$_2$CHSO$_2$Ph), 2.50–2.90 (m, 1 H, C*H*$_2$CHSO$_2$Ph), 2.96 (s, 3 H, NCH$_3$), 3.05 (s, 3 H, NCH$_3$), 3.54 (s, 3 H, OCH$_3$), 3.60–3.65 (m, 1 H, OCH), 3.69 (s, 3 H, OCH$_3$), 3.74–4.20 (m, 5 H, OCH$_2$, 3 × OCH), 4.60 (s, 2 H, OC*H*$_2$Ph), 4.94 (dd, 0.35 H, *J* = 8.8, 2.5, C*H*SO$_2$Ph), 5.28 (dd, 0.65 H, *J* = 8.8, 2.5, C*H*SO$_2$Ph), 7.26–7.40 (m, 5 H, Ar-H), 7.50–7.70 (m, 3 H, Ar-H), 7.80–7.86 (m, 2 H, Ar-H)	25.21, 26.50, 26.85, 29.37, 29.70, 32.10, 32.76, 61.20, 61.62, 61.97, 67.86, 74.39, 75.31, 77.62, 81.75, 83.86, 109.60, 109.77, 109.85, 127.68, 128.15, 128.26, 128.62, 128.70, 128.73, 129.70, 133.87, 134.09, 137.55, 138.03, 138.20, 165.58, 165.69

[a] Mixture of diastereomers.

Table 3 Compounds **8a−k** Prepared

Product[a]	Yield (%)	$[\alpha]_D$[b]	Molecular Formula	HRMS (*m*/*z* calcd)	HRMS (*m*/*z* found)	IR ($CHCl_3$) ν (cm^{-1})	EI-MS (70 eV) *m*/*z* (%)
8a[c]	69	–	$C_8H_{17}NO_2$	159.1164	159.1134	2928, 2848, 1664, 1400	159 (5), 103 (25), 99 (80), 71 (68), 61 (100), 57 (19)
8b[c]	70	–	$C_7H_{13}NO_2$	143.1046	143.1004	3008, 2932, 1643, 1664, 1446	143 (30), 115 (20), 81 (38), 61 (100), 53 (98)
8c[c]	71		$C_7H_{11}NO_2$	141.0889	141.0848	3280, 2928, 2096, 1658, 1452	141 (<1), 81 (30), 61 (100), 53 (98)
8d[c]	70	–	$C_{11}H_{15}NO_2$	193.1202	193.1241	2944, 1680, 1600, 1500, 1440	193 (41), 133 (20), 105 (94), 91 (100), 77 (20), 61 (37)
8e[c]	73	–	$C_6H_{12}NO_2$	129.0889	129.0855	2944, 2864, 1670	129 (10), 69 (100), 61 (8)
8f[c]	82	–	$C_{12}H_{17}NO_3$	223.1358	223.1337	2896, 1680, 1600, 1500, 1400, 1160, 1033	223 (4), 163 (12), 130 (100), 107 (12), 77 (22), 69 (79), 61 (10)
8g[d]	73	–	$C_9H_{18}N_2O_4$	158.0967 (M − 60)	158.0961 (M − 60)	2896, 1715, 1654, 1452	217 (<1), 202 (31), 157 (78), 100 (14), 99 (100), 71 (36), 61 (18)
8h[d]	74	−4.00	$C_{10}H_{19}NO_4$	202.1248 (M − 15)	202.1232 (M − 15)	2896, 1715, 1654, 1452	245 (<1), 230 (25), 185 (28), 170 (50), 127 (85), 109 (20), 99 (10), 97 (35), 85 (27), 81 (100), 61 (52)
8i[d]	72	+10	$C_{12}H_{23}NO_4$	230.1380 (M − 15)	230.1392 (M − 15)	2896, 1715, 1654, 1452	245 (<1), 230 (25), 185 (28), 170 (50), 127 (85), 109 (20), 99 (10), 97 (35), 85 (27), 81 (100), 61 (52)
8j[d]	73	+22	$C_{15}H_{27}NO_6$	302.1857 (M − 15)	302.1886 (M − 15)	2944, 1654, 1449, 1363	317 (<1), 302 (38), 244 (16), 241 (18), 216 (8), 201 (10), 200 (10), 199 (76), 184 (42), 158 (100), 141 (36), 123 (16), 113 (28), 101 (46), 99 (30), 85 (54), 83 (32), 61 (32), 59 (76), 55 (70)
8k[d]	70	+10	$C_{23}H_{35}NO_7$	422.2528 (M − 15)	422.2512 (M − 15)	2980, 2922, 1723, 1665, 1600, 1452, 1380, 1072	437 (<1), 422 (4), 377 (4), 236 (28), 172 (10), 152 (12), 101 (8), 91 (100), 85 (8)

[a] Satisfactory microanalyses were obtained for all new compounds **8f−h**: C, ± 0.30; H, ±0.41; N, ±0.48.
[b] Specific rotations were measured in $CHCl_3$ (*c* = 1) at r. t.
[c] liquid
[d] Syrup.

^{1}H NMR ($CDCl_3$): δ = 3.10 (s, 3 H, NCH_3), 3.70 (s, 3 H, OCH_3), 4.27 (s, 2 H, CH_2), 7.48 − 7.54 (m, 2 H, Ar-H), 7.58−7.64 (m, 1 H, Ar-H), 7.86−7.92 (m, 2 H, Ar-H).

^{13}C NMR ($CDCl_3$): δ = 32.5, 57.9, 61.7, 128.56, 129.01, 134.01, 139.23, 162.23.

IR ($CHCl_3$): ν = 2992, 1670, 1600, 1323, 1154 cm^{-1}.

MS (EI): *m*/*z* (%) = 243 (5), 141 (22), 77 (32), 61 (100), 55 (6).

HRMS: *m*/*z* calcd for $C_{10}H_{13}NO_4S$: 243.0765. Found: 243.0751.

Anal. Calcd. for $C_{10}H_{13}NO_4S$: C, 49.37; H, 5.39; N, 5.76. Found: C, 49.37; H, 5.48; N, 5.15.

Alkylation of 3 with Various Electrophiles; Preparation of 6a−k; General Procedure

Electrophile **6** (1.2 mmol) and acetamide **3** (0.243 g, 1 mmol) were stirred with anhyd K_2CO_3 (0.414 g, 3 mmol) in anhyd DMF (2 mL, 2.5 mL in the cases of **6b**, **6c**, **6d**) at temperatures ranging from ambient to 80°C. After the completion of the reaction, H_2O (10 mL) was added and the solution extracted with EtOAc (3 × 5 mL). The combined EtOAc layer was concentrated after drying (Na_2SO_4) and the crude product obtained was chromatographed to give **7** in good yields. In the case of electrophiles **6h−k**, a diastereomeric mixture was obtained which was directly subjected to desulfonylation.

Table 4 ^{1}H and ^{13}C NMR of Compounds **8a–k**

Product	^{1}H NMR ($CDCl_3$ / TMS); δ, *J* (Hz)	^{13}C NMR ($CDCl_3$ / TMS); δ
8a[a]	0.83 (t, 3 H, *J* = 6.8, CH_3), 1.20-1.28 (m, 4 H, $CH_3CH_2CH_2$), 1.56–1.60 (m, 2 H, CH_2CH_2CO), 2.34 (t, 2 H, *J* = 7.3, CH_2CO), 3.10 (s, 3 H, NCH_3), 3.61 (s, 3 H , OCH_3)	13.76, 22.29, 24.17, 29.52, 31.43, 31.67, 61.06, 174.60
8b[a]	2.33–2.57 (m, 4 H, CH_2CH_2), 3.18 (s, 3 H, NCH_3), 3.69 (s, 3 H , OCH_3), 4.96–5.11 (m, 2 H, CH=CH_2), 5.76–5.96 (m, 1 H, CH=CH_2)	28.45, 31.08, 32.12, 61.10, 114.99, 137.35, 173.59
8c[a]	1.98 (t, 1 H, *J* = 2.4, C≡CH), 2.52 (dt, 2 H, *J* = 7.8, 2.4, CH_2CH_2CO), 2.69 (t, 2 H, *J* = 7.8, CH_2CO), 3.19 (s, 3 H, NCH_3), 3.70 (s, 3 H , OCH_3)	13.69, 30.87, 32.11, 61.13, 68.52, 83.26, 172.17
8d[a]	2.65 (t, 2 H, *J* = 7.8, CH_2CO), 2.87 (t, 2 H, *J* = 7.8, CH_2Ph), 3.08 (s, 3 H, NCH_3), 3.50 (s, 3 H , OCH_3), 7.10–7.26 (m, 5 H, Ar-H)	30.53, 32.02, 33.61, 61.03, 103.86, 125.27, 128.27, 141.14, 174.63
8e[a]	0.72–0.80 (m, 2 H, CHH'CHH'), 0.88–0.93 (m, 2 H, CHH'CHH'), 2.10 (br t, 1 H, CHCO), 3.14 (s, 3 H, NCH_3), 3.69 (s, 3 H , OCH_3)	7.73, 9.69, 32.49, 61.38, 174.63
8f	2.02 (m, 2 H, $CH_2CH_2CH_2$), 2.54 (t, 2 H, *J* = 6.3, CH_2CO), 3.07 (s, 3 H, NCH_3), 3.55 (s, 3 H , OCH_3), 3.92 (t, 2 H, *J* = 6.3, CH_2OPh), 6.78–6.84 (m, 3 H, Ar-H), 7.14–7.18 (m, 2 H, Ar-H)	23.99, 28.02, 31.96, 60.95, 66.60, 114.23, 120.36, 129.20, 158.67, 173.72
8g	1.93–1.97 (m, 2 H, $CH_2CH_2CH_2$), 2.51 (br t, 4 H, *J* = 5.9, CH_2CO), 3.17 (s, 3 H, NCH_3), 3.69 (s, 3 H , OCH_3)	19.46, 31.04, 32.12, 61.22, 174.08
8h	1.27 (s, 3 H, CH_3), 1.33 (s, 3 H, CH_3), 1.75–1.96 (m, 2 H, CH_2CH_2CO), 2.40–2.60 (m, 2 H, CH_2CO), 3.11 (s, 3 H, NCH_3), 3.46–3.50 (m, 1 H, OCH), 3.62 (s, 3 H , OCH_3), 3.96–4.10 (m, 2 H, OCH_2)	25.66, 26.95, 28.08, 28.46, 32.22, 61.23, 69.24, 75.30, 108.86, 173.92
8i	1.19 (s, 3 H, CH_3), 1.24 (s, 3 H, CH_3), 1.36–1.42 (m, 6 H, $CH_2CH_2CH_2$), 2.29 (t, 2 H, *J* = 7.3, CH_2CO), 3.02 (s, 3 H, NCH_3), 3.30–3.40 (m, 1 H, OCH), 3.54 (s, 3 H , OCH_3), 3.86–3.95 (m, 2 H, OCH_2)	24.13, 25.16, 25.27, 26.49, 31.25, 31.72, 32.95, 60.74, 68.97, 75.48, 108.16, 173.97
8j	1.33 (s, 3 H, CH_3), 1.35 (s, 3 H, CH_3), 1.38 (s, 3 H, CH_3), 1.41 (s, 3 H, CH_3), 1.80–1.90 (m, 1 H, CHH'CH_2CO), 2.10–2.17 (m, 1 H, CHH'CH_2CO), 2.50–2.67 (m, 2 H, CH_2CH_2CO), 3.18 (s, 3 H, NCH_3), 3.60 (t, 1 H, *J* = 7.8, OCH), 3.69 (s, 3 H , OCH_3), 3.92–4.00 (m, 2 H, OCH_2), 4.01–4.05 (m, 1 H, OCH), 4.10–4.14 (dd, 1 H, *J* = 8.3, 6.3, OCH)	25.30, 26.69, 27.03, 27.30, 28.47, 28.63, 32.28, 61.18, 67.62, 77.06, 79.73, 81.28, 108.96, 109.61, 174.20
8k	1.25 (s, 3 H, CH_3), 1.33 (s, 3 H, CH_3), 1.37 (s, 3 H, CH_3), 1.41 (s, 3 H, CH_3), 1.94–2.12 (m, 2 H, CH_2CH_2CO), 2.46–2.68 (m, 2 H, CH_2CO), 3.15 (s, 3 H, NCH_3), 3.62 (s, 3 H , OCH_3), 3.60–3.70 (m, 1 H, OCH), 3.89 (dd, 1 H, *J* = 7.8, 2.9, OCH), 4.00–4.13 (m, 4 H,OCH_2, 2 × OCH), 4.65 (s, 2 H, OCH_2Ph), 7.24–7.37 (m, 5 H, Ar-H)	25.24, 25.78, 26.43, 27.00, 27.29, 27.54, 32.31, 61.07, 67.51, 72.35, 77.18, 77.30, 77.58, 82.37, 109.42, 109.57, 127.41, 127.70, 128.21, 138.62, 174.20

[a] Compounds known in the literature. See Ref. 21.

Desulfonylation of 7; General Procedure

A solution of **7** (1 mmol) in MeOH (10 mL) cooled to 0°C was treated with Na_2HPO_4 (0.568 g, 4 mmol) and 6% Na(Hg) (1.50g). The reaction mixture was stirred for 1.5 h after which H_2O (10 mL) was added and the solution extracted with EtOAc (3 × 10 mL). The combined organic layers were dried (Na_2SO_4) and the solvent evaporated. Further purification of the product was done by column chromatography.

Methyl 3,4:5,6-Di-*O*-isopropylidene-2-*O*-benzyl-D-gluconoate (11)

To a suspension of Ag_2O (0.232 g, 1 mmol) in anhyd Et_2O (3 mL) was added **10** (0.290 g, 1 mmol) and benzyl bromide (0.130mL, 1.1 mmol) and the mixture was stirred for 24 h at r.t. The mixture was then filtered, the filtrate evaporated and chromatographed to give **11**; yield: 0.311 g (82%); R_f 0.43 (hexane/EtOAc, 4:1); $[\alpha]_D$ +78 (*c* = 1, $CHCl_3$).

^{1}H NMR ($CDCl_3$): δ = 1.30 (s, 3 H, CH_3), 1.32 (s, 3 H, CH_3), 1.36 (s, 3 H, CH_3), 1.39 (s, 3 H, CH_3), 3.80 (s, 3 H, CO_2CH_3), 3.86−3.88 (m, 1 H, OCH), 4.05−4.15 (m, 4 H, OCH_2, 2 × OCH), 4.34−4.35 (m, 1 H, OCH), 4.41−4.44 (d, 1 H, *J* = 11.2Hz, OCH_2Ph), 4.85−4.88 (d, 1 H, *J* = 11.2Hz, OCH_2Ph), 7.26−7.38 (m, 5 H, Ar-H).

^{13}C NMR ($CDCl_3$): δ = 25.16, 26.56, 27.26, 52.15, 67.68, 73.10, 76.77, 77.12, 77.39, 80.66, 109.65, 110.33, 127.89, 127.99, 128.38, 137.23, 170.74.

IR ($CHCl_3$): ν = 2980, 2922, 1756, 1495, 1452, 1376, 1072 cm^{-1}.

MS (EI): *m*/*z* (%) = 380 (<1), 365 (4), 143 (22), 101 (40), 91 (100), 85 (9).

HRMS: *m*/*z* calcd. for $C_{19}H_{25}O_7$ (M−15):365.19503. Found: 365.199118.

Anal. Calcd. for $C_{20}H_{28}O_7$: C, 63.14; H, 7.42. Found: C, 63.01; H, 7.38.

3,4:5,6-Di-*O*-isopropylidene-2-*O*-benzyl-D-glucitol (12)

To a suspension of $LiAlH_4$ (0.038 g, 1 mmol) in anhyd THF (10 mL) was added **11** (0.380 g, 1 mmol) and stirred at r. t. for 3 h. The

excess $LiAlH_4$ was then quenched with EtOAc, and the mixture was subsequently treated with H_2O (0.1 mL), 15% aq NaOH solution (0.3 mL) and again with H_2O (0.1 mL). The mixture was then filtered through a Celite pad, the filtrate dried (Na_2SO_4) and evaporated to dryness to give **12**; yield: 0.299 g (85%). Further purification was done by column chromotography; R_f 0.20 (hexane/EtOAc, 4:1); $[\alpha]_D$ +22.0 (*c* = 1, $CHCl_3$).

^{1}H NMR ($CDCl_3$): δ = 1.33 (s, 3 H, CH_3), 1.38 (s, 6 H, 2 × CH_3), 1.40 (s, 3 H, CH_3), 3.62 (dd, 1 H, *J* = 8.3, 4.4Hz, OCH), 3.75−4.16 (m, 5 H, OCH_2, 3 × OCH), 4.65 (d, 1 H, *J* = 11.7 Hz, OC*H*$_2$Ph), 4.77 (d, 1 H, *J* = 11.7 Hz, OC*H*$_2$Ph), 7.25−7.40 (m, 5 H, Ar-H).

^{13}C NMR ($CDCl_3$): δ = 25.19, 26.44, 26.71, 27.18, 62.20, 67.83, 72.55, 77.26, 77.94, 80.85, 81.80, 109.76, 127.67, 127.74, 128.38, 138.31.

IR ($CHCl_3$): ν = 3481(rb), 2987, 2933, 2889, 1493, 1071 cm^{-1}.

MS (EI): *m*/*z* (%) = 352 (4), 337 (6), 217 (40), 143 (36), 131 (24), 108 (42), 107 (32), 101 (50), 91 (100), 77 (44), 61 (8), 59 (86), 51 (22).

HRMS: *m*/*z* calcd. for $C_{18}H_{25}O_6$ (M−15): 337.1651. Found:337.1689.

Anal. Calcd. for $C_{19}H_{28}O_6$: C, 64.75; H, 8.01. Found: C, 64.38; H, 7.87.

1-Deoxy-1-iodo-3,4:5,6-di-*O*-isopropylidene-2-*O*-benzyl-D-glucitol (6k)

To a solution of the above glucitol **12** (0.352 g, 1 mmol) in toluene (30 mL) was added Ph_3P (0.655 g, 2.5 mmol), imidazole (0.170 g, 2.5 mmol) and I_2 (0.508 g, 2 mmol) and the mixture was heated to reflux for 12 h. The mixture was then evaporated and the residue column chromatographed to give 0.207 g (45%) of **6k;** .R_f 0.14 (hexane); $[\alpha]_D$ −6.0 (*c* = 1, $CHCl_3$).

^{1}H NMR($CDCl_3$): δ = 1.34 (s, 3 H, CH_3), 1.37 (s, 3 H, CH_3), 1.38 (s, 3 H, CH_3), 1.39 (s, 3 H, CH_3), 3.34−3.41 (m, 2 H, CH_2I), 3.73−3.78 (m, 1 H, OCH), 3.84−3.88 (m, 1 H, OCH), 3.92−3.96 (m, 1 H, OCH), 4.03−4.14 (m, 2 H, OCH_2), 4.27−4.30 (m, 1 H, OCH),4.62 (d, 1 H, *J* = 11.2 Hz, OC*H*$_2$Ph), 4.77−4.80 (d, 1 H, *J* = 11.2 Hz, OC*H*$_2$Ph), 7.25−7.36 (m, 5 H, Ar-H).

^{13}C NMR($CDCl_3$): δ = 4.00, 25.14, 26.58, 26.73, 27.15, 67.72, 73.18, 77.02, 77.56, 78.32, 80.62, 109.65, 127.77, 127.99, 128.39, 137.75.

IR($CHCl_3$): ν = 2980, 2922, 1455, 1372, 1253, 1072, 844 cm^{-1}.

MS (EI): *m*/*z* (%) = 462 (<1), 447 (8), 201 (6), 143 (50), 101 (12), 91 (100), 85 (10), 57 (14).

HRMS: *m*/*z* calcd for $C_{18}H_{24}IO_5$(M−15): 447.0916. Found: 447.0948.

2,3-Dideoxy-4,5:6,7-di-*O*-isopropylidene-D-*arabino*-heptose (13)

To a suspension of $LiAlH_4$ (0.038 g, 1 mmol) in anhyd THF (10 mL) was added **8j** (0.317 g, 1 mmol) and stirred at −78°C for 15 min. The excess $LiAlH_4$ was then quenched with EtOAc (0.5 mL), and the mixture was subsequently treated with H_2O (0.1 mL), 15% aq NaOH solution (0.3 mL) and again with H_2O (0.1 mL). The mixture was then filtered through a Celite pad, the filtrate dried (Na_2SO_4) and evaporated to dryness to give 0.193 g, (75%) of the aldehyde **13;** R_f 0.37 (hexane/EtOAc, 4:1; $[\alpha]_D$ +15 (*c* = 1, $CHCl_3$).

^{1}H NMR ($CDCl_3$): δ = 1.34 (3 H, CH_3), 1.36 (s, 6 H, 2 x CH_3), 1.40 (s, 3 H, CH_3), 1.80−1.96 (m, 1 H, C*H*$_2$CH$_2$CO), 2.06−2.22 (m, 1 H, C*H*$_2$CH$_2$CO), 2.53−2.67 (m, 2 H, CH_2CO), 3.49−3.58 (m, 1 H, OCH), 3.89−4.16 (m, 4 H, OCH_2, 2 x OCH), 9.79 (s, 1 H, CHO).

^{13}C NMR ($CDCl_3$): δ = 25.12, 25.92, 26.60, 26.82, 27.08, 40.29, 67.67, 77.01, 79.41, 80.97, 108.96, 109.54, 201.76.

IR ($CHCl_3$): ν = 2986, 2924, 2879, 1724, 1454, 1062 cm^{-1}.

MS (EI): *m*/*z* (%) = 229 (10), 201 (10), 186 (15), 171 (20), 156 (20), 101 (45), 99 (50), 57 (100).

HRMS: *m*/*z* calcd for $C_{12}H_{21}O_4$(M−29): 229.1639. Found: 229.1646.

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