DOI: 10.1002/ejoc.200600126

New Reagent for Convenient Access to the α,β-Unsaturated *N*-Methoxy-*N*-methyl-amide Functionality by a Synthesis Based on the Julia Olefination Protocol

Beedimane Narayana Manjunath,[a] Neeraj P. Sane,[a] and Indrapal Singh Aidhen*[a]

Dedicated to Professor N. S. Narasimhan

Keywords: Julia olefination / Weinreb amides / Heterocycles / Sulfones

A new reagent for the synthesis of the α,β-unsaturated *N*-methoxy-*N*-methyl-amide structural unit has been developed. 2-(Benzo[*d*]thiazol-2-ylsulfonyl)-*N*-methoxy-*N*-methylacetamide, a crystalline solid with an indefinite shelf life that can be easily prepared in two convenient steps from 2-chloro-*N*-methoxy-*N*-methylacetamide, reacted with a variety of aldehydes under Julia conditions to furnish the α,β-unsaturated *N*-methoxy-*N*-methyl-amide functionality.

In contrast to the α,β-unsaturated ester structural unit which boasts a rich chemistry and has often been a common target in organic synthesis, the interest in the α,β-unsaturated *N*-methoxy-*N*-methyl-amide structural unit has found a significant surge only in recent times. This unit has served as a valuable functionality in many synthetic endeavours[1] because of the presence of a versatile *N*-methoxy-*N*-methyl-amide functionality, popularly known as Weinreb amide (WA).[2] WA has been an excellent carbonyl equivalent with large applications in organic synthesis exclusively because of its ease of preparation and its versatile reactivity, for example, in nucleophilic addition and selective reduction reactions to form aldehydes.[3] Among the many applications of the α,β-unsaturated *N*-methoxy-*N*-methyl-amide functionality, this structural unit has shown unique distinction on two occasions. In the light of the fact that α,β-unsaturated aldehydes and ketones have been poor substrates for the asymmetric dihydroxylation (AD) process, the facile and convenient AD process at the α,β-unsaturated Weinreb amide has brought to the surface their importance for indirect access to these functionalities.[4] A similar advantage has been observed during cyclopropanation of α,β-unsaturated *N*-methoxy-*N*-methyl-amide for indirect access to α,β-cyclopropyl ketones, particularly when well-documented direct cyclopropanation of α,β-unsaturated ketones failed.[5]

Currently, two approaches are available in the literature to obtain the α,β-unsaturated *N*-methoxy-*N*-methyl-amide structural unit. The first approach, based on Wittig carbonyl olefination, uses *N*-methoxy-*N*-methyl-2-(triphenylphosphoranylidene)acetamide[6] or its Horner–Wadsworth–Emmons (HWE) variant,[7] whereas the second approach makes use of *N*-methoxy-*N*-methyl-2-(phenylsulfinyl)acetamide[8] as a reagent for reaction with alkyl halides to furnish the same target. Although the former approach shows significant potential for general use, the latter has been severely restricted to nonfunctionalized and simple substrates. None of the approaches have been used to extend the chain on aldehydes from the carbohydrate domain. Our own

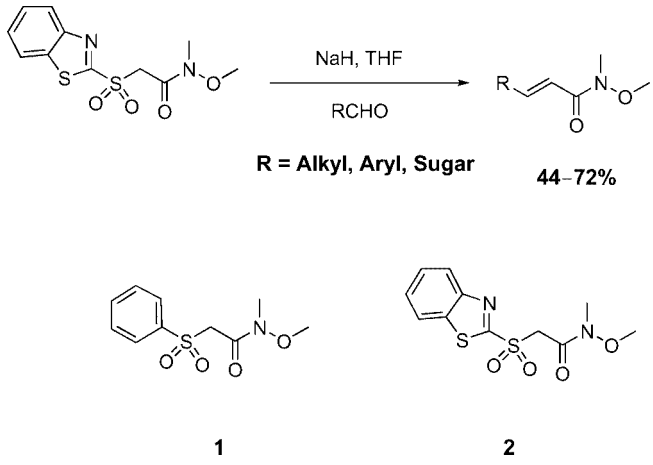
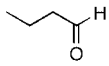
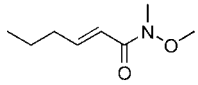
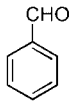
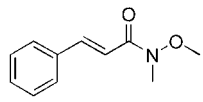
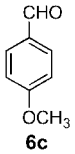
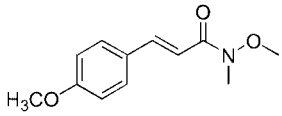
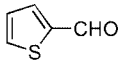
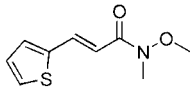
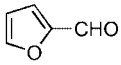
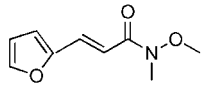
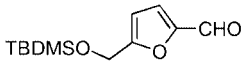
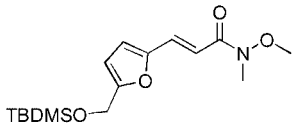
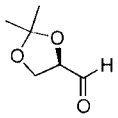
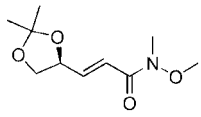
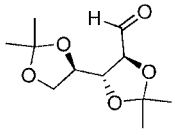
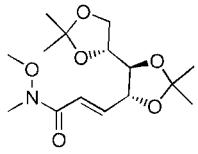
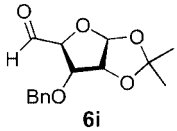
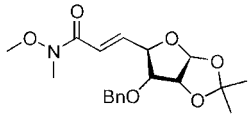
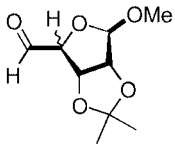
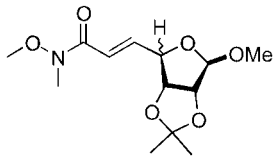
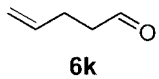
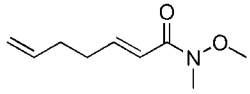


Figure 1. Reaction of reagent **2** with various aldehydes to give α,β-unsaturated Weinreb amides.

[a] Department of Chemistry, Indian Institute of Technology Madras,
Chennai 600036, India
Fax: +91-44-22574202
E-mail: isingh@iitm.ac.in

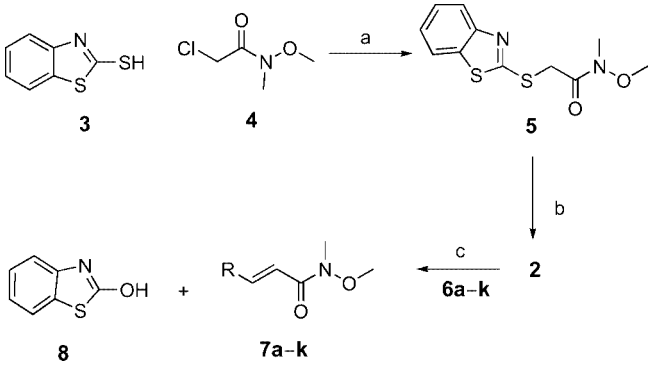
interest[9a,9b] in developing synthetic equivalents based on the Weinreb amide functionality for two-carbon homologation in the area of carbohydrates has initiated this study. Reagent **1**, developed in our group,[9c] has been successfully used for two-carbon homologation of sugar halides; however, it failed to react with aldehydes under Knoevenagel conditions for chain extension. Simple replacement of the phenylsulfonyl moiety in **1** with benzo[*d*]thiazol-2-ylsulfonyl

Table 1. Reaction of reagent **2** with various aldehydes (**6a**–**k**) to furnish α,β-unsaturated *N*-methoxy-*N*-methyl-amides (**7a**–**k**).

Entry	Aldehyde (**6a**–**k**)	Product (**7a**–**k**)	Yield (%)
1	 **6a**	 **7a**	48
2	 **6b**	 **7b**	67
3	 **6c**	 **7c**	44
4	 **6d**	 **7d**	63
5	 **6e**	 **7e**	52
6	 **6f**	 **7f**	59
7	 **6g**	 **7g**	50
8	 **6h**	 **7h**	72
9	 **6i**	 **7i**	55
10	 **6j**	 **7j**	53
11	 **6k**	 **7k**	57

led to the proposal of **2** as a potential new reagent for the synthesis of α,β-unsaturated Weinreb amides, which is based on the well-established Julia olefination concept.[10] Conspicuous absence of the reagent **2** in the literature prompted this study. Presented herein is a new reagent **2** whose soft carbanion can easily be formed and successfully reacted with aldehydes to afford α,β-unsaturated *N*-methoxy-*N*-methyl-amides (Figure 1).

Analytically pure reagent **2** can be easily prepared as a crystalline solid on a 10-g scale and in an overall 70% yield from inexpensive benzo[*d*]thiazole-2-thiol (**3**) by base-mediated *S*-alkylation with 2-chloro-*N*-methoxy-*N*-methylacetamide (**4**),[11] followed by clean oxidation of sulfide **5** with hydrogen peroxide in the presence of catalytic amounts of sodium tungstate.[12] The carbanion from reagent **2** obtained by using NaH in THF at room temperature readily reacts with a variety of aldehydes given in Table 1 to give exclusively the expected *E* stereochemistry about the newly formed double bond in the α,β-unsaturated *N*-methoxy-*N*-methyl-amides **7a**–**k** (Scheme 1).



Scheme 1. Reagents and conditions: a: Na_2CO_3, H_2O 14 h; b: Na_2WO_4, H_2O_2, MeOH, 16 h; c: NaH, THF then RCHO (**6a**–**k**), room temp., 24 h.

The side product 2-hydroxy benzothiazole **8** can be easily removed by extracting the organic phase during workup with a cold and dilute solution of NaOH. This purification offers a significant advantage over the removal of triphenylphosphane oxide in the Wittig reaction. The ^{1}H NMR spectroscopic data for the products **7g**–**i** clearly indicate no sign of any epimerization at the α-stereocenter in aldehydes **6g**–**i**.[13,14] The usual threat of possible epimerization at the α-stereocenter in chiral aldehydes under basic conditions was completely averted by ensuring the complete formation of the soft carbanion from **2** in a dry THF environment, prior to the addition of the requisite aldehyde. The obtainment of product **7k** in 57% yield serves as proof that this is an excellent application. This compound **7k**, recently reported in the literature, has served as a key building block in the first synthesis[15] of Pochonin D, a potent inhibitor of the heat shock protein 90 and an attractive target for chemotherapy.

In summary, a shelf-stable and crystalline solid reagent has been synthesized and used for olefination of functionalised aldehydes under Julia conditions. In light of the facts that α,β-unsaturated *N*-methoxy-*N*-methyl-amides undergo facile asymmetric dihydroxylation and that the amide is a robust equivalent of the aldehyde, the present disclosure opens up vistas for a possible iterative strategy for higher sugars.

Experimental Section

All solvents were distilled before use. Anhydrous THF was prepared by using standard procedures that involved drying over sodium followed by distillation. Melting points were determined in capillaries and are uncorrected. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded with deuteriochloroform ($CDCl_3$) as the solvent and tetramethylsilane (TMS) as a reference. IR spectra were recorded with KBr pellets for solid samples and neat for liquid samples. Mass spectra were recorded on a MICRO-Q TOF mass spectrometer by using the ESI technique at 10 eV. Optical rotations were measured with an Autopol IV polarimeter at room temperature. All the reactions were monitored by TLC on precoated silica gel plates by developing the spots with iodine vapor or under UV-light absorbance. For monitoring reactions of compounds **6g**–**6j**, TLC was performed on precoated silica gel plates by dipping in a solution that was prepared by adding ammonium ceric sulfate (1 g) and ammonium molybdate (21 g) to concentrated sulfuric acid (31 mL) and making the volume up to 500 mL with distilled water. The TLC plates were later heated up to 100 °C to develop.

Commercially available samples of aldehydes **6a**–**6e** were directly used after drying. The aldehydes **6f**,[16a] **6g**,[16b] **6h**,[16c] and **6i**[16b] were prepared as reported in the literature. Aldehydes **6j** and **6k** were prepared by Swern oxidation of methyl 2,3-*O*-isoproylidene-β-D-ribofuranoside[13] and 4-penten-1-ol respectively. The latter was purchased from Aldrich. The known products **7a**,[17a] **7b**,[17b] **7c**,[17c] **7d**,[17d] **7e**,[17d] and **7k**[15] gave satisfactory analytical and spectroscopic data.

2-(Benzo[*d*]thiazol-2-ylthio)-*N*-methoxy-*N*-methylacetamide (5): To a suspension of 2-mercaptobenzothiazole (**3**, 1.75 g, 0.0105 mol) in water (20 mL) was added Na_2CO_3 (2.015 g, 0.019 mol), and the solution was stirred for 15 min. To this reaction mixture was added chloroamide **4** (1.313 g, 0.0095 mol), and the stirring was continued overnight at room temperature. On completion of the reaction, the product was extracted with ethyl acetate (2×25 mL). The combined organic layers were washed with water and brine and then dried with anhydrous Na_2SO_4 and concentrated. The product, sulfide **5**, was purified by silica gel column chromatography with ethyl acetate/hexane (2:5) as eluent. The pure product was obtained as a yellowish oil (2.38 g, 93% yield). R_f (EtOAc/Hexane, 1:1) 0.40. ^{1}H NMR ($CDCl_3$/TMS, 400 MHz): δ = 3.28 (s, 3 H), 3.86 (s, 3 H),4.47 (s, 2 H), 7.29–7.46 (m, 2 H), 7.77 (d, J = 8.0 Hz, 1 H), 7.87 (d, J = 8.0 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$/TMS, 100 MHz): δ = 32.6, 35.1, 61.6, 121.1, 121.4, 124.4, 126.1, 135.4, 152.7, 165.8 ppm.

2-(Benzo[*d*]thiazol-2-ylsulfonyl)-*N*-methoxy-*N*-methylacetamide (2): To a solution of sulfide **5** (2.38 g, 0.0089 mol) in methanol (20 mL) at 0 °C was added sodium tungstate dihydrate (1.47 g, 0.0045 mol). Hydrogen peroxide (3.62 mL, 0.0355 mol, 30%) was added 5 min later. The reaction mixture was stirred overnight at room temperature. On completion of the reaction, methanol was removed under vacuum. To the residue was added an aqueous solution of sodium metabisulfite (20 mL, 25% solution). The product was extracted with ethyl acetate (2×25 mL). The combined organic layers were washed with water and brine and then dried with anhydrous Na_2SO_4 and concentrated. The crude product was purified by silica

gel column chromatography with ethyl acetate/hexane (2:5) as eluent. The pure product was obtained as a white solid (2.0 g, 75% yield). R_f = 0.27 (EtOAc/hexane, 1:1) M.p. 128 °C. ^{1}H NMR ($CDCl_3$/TMS, 400 MHz): δ = 3.18 (s, 3 H), 3.79 (s, 3 H), 4.80 (s, 2 H), 7.49–7.65 (m, 2 H), 7.99 (d, J = 8.0 Hz, 1 H), 8.20 (d, J = 8.0 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$/TMS, 100 MHz): δ = 32.1, 56.4, 61.9, 122.4, 125.5, 127.6, 128.0, 137.1, 152.5, 161.5, 165.6 ppm. HRMS (ES): calcd. for $C_{11}H_{12}N_2S_2O_4$ $[M+H]^+$ 301.0238; found 301.0330. $C_{11}H_{12}N_2S_2O_4$ (300.36): calcd. C 43.99, H 4.03, N 9.33; found C 44.30, H 4.15, N 8.90.

A Representative Procedure for the Reaction of the Aldehyde with the Reagent 2: A solution of sulfone **2** (0.155 g, 0.52 mmol) in dry THF (5 mL) was added to oil-free sodium hydride (0.016 g, 0.65 mmol) under an inert atmosphere and stirred for 2 min at room temperature. To this reaction mixture was added a solution of aldehyde **6h** (0.100 g, 0.43 mmol) dissolved in dry THF (2 mL). The stirring was continued for a further 24 hr. On completion of the reaction, which was indicated by TLC, THF was removed under reduced pressure. To the residue was added ammonium chloride (5 mL, 20% solution), and the product was extracted with diethyl ether (3 × 5 mL). The combined ether extract was washed with an ice-cold aqueous solution of sodium hydroxide (10 mL, 20%). On evaporation of the ether layer after drying over sodium sulfate, the crude product was obtained and was subjected to column chromatography on silica gel (hexane/ethyl acetate, 8:2) to furnish compound **7h** as a colorless syrup (0.099 g, 72%).

(*E*)-3-[5-(*tert*-Butyl-dimethyl-silanyloxymethyl)furan-2-yl]-*N*-methoxy-*N*-methylpropenamide (7f): Yield 59%. R_f = 0.20 (EtOAc/hexane, 1:5). IR 3119, 2955, 2931, 1657, 1618, 1584, 1381 cm^{-1}. ^{1}H NMR ($CDCl_3$/TMS, 400 MHz): δ = 0.06 (s, 3 H), 0.12 (s, 3 H), 0.93 (s, 9 H), 3.29 (s, 3 H), 3.75 (s, 3 H), 4.69 (s, 2 H), 6.3 (d, J = 3.6 Hz, 1 H), 6.53 (d, J = 3.6 Hz, 1 H), 6.89 (d, J = 15.0 Hz, 1 H), 7.44 (d, J = 15.0 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$/TMS,100 MHz): δ = –5.2, 18.4, 25.8, 58.4, 32.6, 61.9, 109.6, 113.0, 115.3, 130.0, 151.1, 156.4, 167.2 ppm. HRMS (ES): calcd. for $C_{16}H_{27}SiNO_4$ $[M+H]^+$ 326.1788; found 326.1795.

(*E*)-*N*-Methoxy-*N*-methyl-2,3-dideoxy-4,5-isopropylidene-D-*glycero*-pent-2-enamide (7g): Yield 50%. $[a]_D^{25}$ = –1.22 (c = 1, $CHCl_3$). R_f = 0.22 (EtOAc/hexane, 3:7). IR 2983, 2938, 1716, 1663, 1622, 1386 cm^{-1}. ^{1}H NMR ($CDCl_3$/TMS, 400 MHz): δ = 1.35 (s, 3 H), 1.38 (s, 3 H), 3.18 (s, 3 H), 3.67 (s, 3 H), 4.12 (m, 2 H), 4.65 (m, 1 H), 6.62 (d, J = 15.0 Hz, 1 H), 6.82 (dd, J = 6.1 Hz, 15.0 Hz, 1 H) ppm; ^{13}CNMR ($CDCl_3$/TMS, 100 MHz): δ = 25.7, 26.4, 32.3, 61.7, 68.8, 75.3, 109.9, 119.8, 143.2, 165.9 ppm. HRMS (ES): calcd. for $C_{10}H_{17}NO_4$ $[M+H]^+$ 216.1236; found 216.1238.

(*E*)-*N*-Methoxy-*N*-methyl-2,3-dideoxy-4,5:6,7-di-*O*-isopropylidene-D-*arabino*-hept-2-enamide (7h): Yield 73%. $[a]_D^{25}$ = +8.97 (c = 1, $CHCl_3$); R_f = 0.25 (EtOAc/hexane, 1:5). IR 2986, 2928,1665, 1637, 1378 cm^{-1}. ^{1}H NMR ($CDCl_3$/TMS, 400 MHz): δ = 1.35 (s, 3 H), 1.41 (s, 3 H), 1.43 (s, 3 H), 1.45 (s, 3 H), 3.26 (s, 3 H), 3.71 (s, 3 H), 3.73–3.75 (m, 1 H), 3.93–3.96 (m, 1 H), 4.11–4.15 (m,2 H), 4.58–4.61(m,1 H), 6.73 (d, J = 16 Hz, 1 H), 7.02 (dd, J = 16 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$/TMS, 100 MHz): δ = 25.2, 26.7, 26.7, 26.9, 32.3, 61.7, 67.4, 76.9, 79.3, 81.2, 109.8, 109.9, 119.2, 143.6, 166.2 ppm. HRMS (ES): calcd. for $C_{15}H_{25}NO_6$ $[M+Na]^+$ 338.1580; found 338.1567.

(*E*)-*N*-Methoxy-*N*-methyl-3-*O*-benzyl-5,6-dideoxy-1,2-*O*-isopropylidene-α-D-*xylo*-hept-5-enofuranuronamide (7i): Yield 56%. $[a]_D^{25}$ = –11.79 (c = 1, $CHCl_3$); R_f = 0.3 (EtOAc/hexane, 3:7). IR 2984, 2937, 1667, 1634, 1384 cm^{-1}. ^{1}H NMR ($CDCl_3$/TMS, 400 MHz): δ = 1.33 (s, 3 H), 1.49 (s, 3 H), 3.26 (s, 3 H), 3.68 (s, 3 H), 3.99 (d, J = 3.0 Hz, 1 H), 4.52 (d, J = 12 Hz, 1 H), 4.60 (d, J = 12 Hz, 1 H), 4.64 (d, J = 3.7 Hz), 4.84–4.87 (m, 1 H), 6.01 (d, J = 3.8 Hz, 1 H), 6.77 (d, J = 16.0 Hz, 1 H), 6.99–7.05 (dd, J = 5.7, 16.0 Hz, 1 H), 7.27–7.35 (m, 5 H) ppm. ^{13}C NMR ($CDCl_3$/TMS, 100 MHz): δ = 26.2, 26.8, 32.4, 61.8, 72.3, 79.8, 82.8, 104.9, 111.8, 120.7, 127.8, 127.9, 128.5, 137.2, 139.7, 166.1 ppm. HRMS (ES): calcd. for $C_{19}H_{25}NO_6$ $[M+H]^+$ 364.1760; found 364.1739.

(*E*)-*N*-Methoxy-*N*-methyl-[methyl-5,6-dideoxy-2,3-*O*-(1-methylethylidene)-β-D-*ribo*-hept-5-enofuranosid]uronamide (7j): Yield 53%. $[a]_D^{25}$ = –17.60 (c = 1, $CHCl_3$); R_f = 0.24 (EtOAc/hexane, 1:2). IR 2937, 1668, 1635, 1384 cm^{-1}. ^{1}H NMR ($CDCl_3$/TMS, 400 MHz): δ = 1.33 (s, 3 H), 1.51 (s, 3 H), 3.25 (s, 3 H), 3.38 (s, 3 H), 3.71(s, 3 H), 4.59–4.64 (m, 1 H), 4.70 (d, J = 5.0 Hz, 1 H), 4.83 (d, J = 5.7 Hz, 1 H), 5.04 (s, 1 H), 6.59 (d, J = 15.0 Hz, 1 H), 6.89–6.95 (dd, J = 5.7, 15.0 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$/TMS, 100 MHz): δ = 25.1, 26.5, 55.0, 32.3, 61.8, 85.2, 85.4, 86.7, 109.8,112.7, 120.2, 144.4,165.9 ppm. HRMS (ES): calcd. for $C_{13}H_{21}NO_6$ $[M+H]^+$ 288.1447; found 288.1464.

Acknowledgments

We thank CSIR New Delhi for the funding of the new project in the year 2005 [01(1971)/05/EMR-II]. We thank DST New Delhi, for the funding towards a 400 MHz NMR machine under the IRPHA Scheme and a ESI-MS facility under the DST-FIST program. B. N. M. is thankful to the University Grants Commission for a Junior Research Fellowship.

[1] a) R. Shintani, T. Kimura, T. Hayashi, *Chem. Commun.* **2005**, 3213–3214 and references cited therein; b) L. Blackburn, H. Kanno, R. J. K. Taylor, *Tetrahedron Lett.* **2003**, *44*, 115–118; c) S. G. Davies, K. Iwamoto, C. A. P. Smethurst, A. D. Smith, H. Rodriguez-Solla, *Synlett* **2002**, 1146–1148.
[2] S. Nahm, S. M. Weinreb, *Tetrahedron Lett.* **1981**, *22*, 3815–3818.
[3] For reviews on Weinreb amide chemistry see: a) J. Singh, N. Satyamurthi, I. S. Aidhen, *J. Prakt. Chem.* **2000**, *342*, 340–347; b) M. Mentzel, H. M. R. Hoffmann, *J. Prakt. Chem.* **1997**, *339*, 517–524; c) M. P. Sibi, *Org. Prep. Proced. Int.* **1993**, *25*, 15–40.
[4] Y. L. Bennani, K. B. Sharpless, *Tetrahedron Lett.* **1993**, *34*, 2079–2082 and references cited therein.
[5] K. E. Rodriques, *Tetrahedron Lett.* **1991**, *32*, 1275–1278.
[6] D. A. Evans, S. W. Kaldor, T. K. Jones, J. Clardy, T. J. Stout, *J. Am. Chem. Soc.* **1990**, *112*, 7001.
[7] a) J.-M. Nuzillard, A. Boumendjel, G. Massiot, *Tetrahedron Lett.* **1989**, *30*, 3779–3780; b) D. F. Netz, J. L. Seidel, *Tetrahedron Lett.* **1992**, *33*, 1957–1958; c) S. Fortin, F. Dupont, P. Deslongchamps, *J. Org. Chem.* **2002**, *67*, 5437.
[8] C. Beney, A. Boumendjel, A. M. Mariotte, *Tetrahedron Lett.* **1998**, *39*, 5779–5780.
[9] a) N. Satyamurthi, J. Singh, I. S. Aidhen, *Synthesis* **2000**, 375; b) N. Satyamurthi, I. S. Aidhen, *Carbohydr. Lett.* **1999**, *3*, 355; c) Reagent **1**: *N*-Methoxy-*N*-methyl-2-phenylsulfonylacetamide is now available from Aldrich (Catalogue No: 56, 108–8).
[10] For a comprehensive review on Julia olefination, see: P. R. Blakemore, *J. Chem. Soc., Perkin Trans. 1* **2002**, 2563.
[11] R. Tillyer, L. F. Frey, D. M. Tshaen, U. H. Dolling, *Synlett* **1996**, 225.
[12] a) H. S. Schultz, H. B. Freyermuth, S. R. Buc, *J. Org. Chem.* **1963**, *28*, 1140; b) T. J. Blacklock, P. Sohar, J. W. Butcher, T. Lamanec, E. J. J. Grabowski, *J. Org. Chem.* **1993**, *58*, 1672.
[13] Epimerization at the α-stereocenter in the aldehyde **6j** was observed during preparation by the Swern oxidation of the corresponding alcohol. Product **7j** was obtained in the same diastereoisomeric ratio after reaction with the carbanion from **2**. The use of the Swern oxidation method became inevitable as the

reported procedure (see ref.[14]) based on Collins oxidation for the obtainment of **6j** failed.

[14] A. G. M. Barrett, S. A. Lebold, *J. Org. Chem.* **1990**, *55*, 3853.

[15] E. Moulin, V. Zoete, S. Barluenga, M. Karplus, N. Winssinger, *J. Am. Chem. Soc.* **2005**, *127*, 6999–7004.

[16] a) B. J. Mc Nelis, D. D. Sternbach, A. T. MacPhail, *Tetrahedron* **1994**, *50*, 6767–6782; b) M. Daumas, Y. Vo-Quang, L. Vo-Quang, F. Le Goffic, *Synthesis* **1989**, 64–65; c) H. Regeling, E. D. Rouville, G. J. F. Chittenden, *Recl. Trav. Chim. Pays-Bas* **1987**, *106*, 46.

[17] a) A. M. Chippindale, S. G. Davies, K. Iwamoto, R. M. Parkin, C. A. P. Smethurst, A. D. Smith, H. Rodriguez-Solla, *Tetrahedron* **2003**, *59*, 3253–3265; b) K. Hioki, H. Kobayashi, R. Ohkihara, S. Tani, M. Kunishima, *Chem. Pharm. Bull.* **2004**, *52*, 470–472; c) E. N. Durantini, *Synth. Commun.* **1999**, *29*, 4201–4222; d) K. E. Rodriques, *Tetrahedron Lett.* **1991**, *32*, 1275–1278.

Received: February 13, 2006
Published Online: April 18, 2006

