SHORT COMMUNICATION

EurJOC European Journal of Organic Chemistry

DOI: 10.1002/ejoc.201500186

Phenyliodine Bis(trifluoroacetate) (PIFA) as an Excellent Promoter of 2-Deoxy-2-phthalimido-1-thioglycosides in the Presence of Triflic Acid in Glycosylation Reactions

Tetsuya Kajimoto,[a] Koji Morimoto,[a] Ryosuke Ogawa,[a] Toshifumi Dohi,[a] and Yasuyuki Kita*[a]

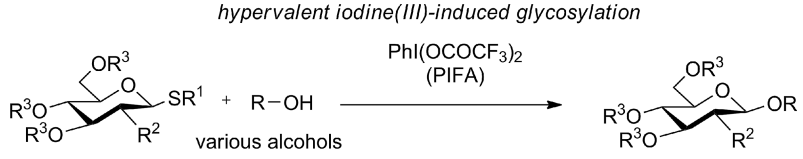
Keywords: Iodine / Phenyliodine bis(trifluoroacetate) / Glycosylation / Thioglycosides

A combination of phenyliodine bis(trifluoroacetate) (PIFA) and trifluoromethanesulfonic acid was found to be an effective activator for the glycosylation reaction, of which the glycosyl donor was *p*-(octyloxy)phenyl 3,4,6-tri-*O*-acetyl-2-deoxy-2-phthalimido-1-thio-D-glucopyranoside or *p*-(octyloxy)phenyl 3,4,6-tri-*O*-acetyl-2-deoxy-2-phthalimido-1-thio-D-galactopyranoside. The reaction proceeded with good yields when naturally occurring and derived alkanols, such as cholestanol, (–)-menthol, 1- and 2-adamantanols and norkauranol, were employed as acceptor substrates. Interestingly, the glycosylation reaction of sterically hindered terpenoid alcohols, such as (–)- and (+)-borneols and (+)-fenchyl alcohol, with *p*-(octyloxy)phenyl 3,4,6-tri-*O*-acetyl-2-deoxy-2-phthalimido-1-thio-D-glucopyranoside afforded the desired β-glycoside in quantitative yield. Moreover, the present reaction was useful to synthesize disaccharides of which the non-reducing end was composed of a D-glucosamine or D-galactosamine unit.

Introduction

The developing of glycosciences revealed that complex oligosaccharides on cell surfaces play important roles in biological events concerning cell-to-cell adhesions, and medicines having glycosidic bonds or glycosyl structures have become widely used to treat diabetes, infection by influenza viruses, and heart diseases.[1] Thus, the synthesis of biologically active oligosaccharides still attracts organic chemists.[2] Among them, the chemistry of thioglycosides[3] has been well developed in the past three decades due to their stability and ease of preparation. The conventional glycosylation of thioglycosides was performed by using a mercury salt, such as $HgSO_4$, $HgCl_2$, $Hg(OBz)_2$, or $Hg(NO_3)_2$.[4] The use of $Cu(OTf)_2$ (Mukaiyama) or $CuBr_2$/Bu_4NBr/AgOTf (Ogawa and Ito) as an activator was further explored.[5] However, these reported systems for the glycosylation of thioglycosides have relied on a toxic heavy metal oxidant for product formation. A key contribution to the glycosylation with thioglycosides was made in 1990 by van Boom and co-workers,[6] who first reported the use of a combination of a stoichiometric amount of *N*-iodosuccinimide (NIS) with a catalytic amount of triflic acid as a promoter to activate the disarmed thioglycosides. The use of strong acid salts in conjunction with *N*-bromosuccinimide to activate the thioglycosides has been investigated by Kusumoto and co-workers.[7] Recently, the alternative combinations of NIS and a Lewis acid were also exploited as the promoter of the thioglycoside activation.[8–13]

Recently, the environmentally benign hypervalent iodine oxidation has witnessed profound progress in the field of organic chemistry.[14] The low toxicity, ready availability and easy handling of hypervalent iodine compounds are expected to be useful features for the activation of the sulfur atom. We have been engaged in the development of the oxidation of the sulfur atom utilizing hypervalent iodine reagents to form an active iodonium intermediate.[15,16] Inspired by these studies, other chemists have also reported



Scheme 1. Hypervalent iodine induced metal-free glycosylation.

[a] College of Pharmaceutical Sciences, Ritsumeikan University, 1-1-1 Nojihigashi, Kusatsu, Shiga 525-8577, Japan
E-mail: kita@ph.ritsumei.ac.jp

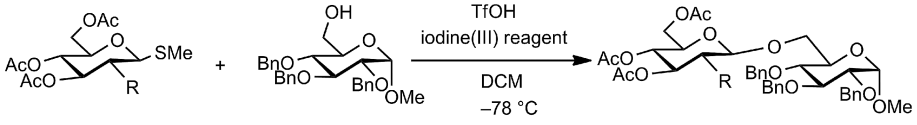
Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/ejoc.201500186.

the hypervalent iodine induced oxidation of sulfides.[17,18] In contrast, apart from the initial studies of oxidation of the sulfur atom, the I^{III}-promoted glycosylation reaction of thioglycosides has rarely been studied.[19] This oxidation ability led us to find an efficient glycosylation reaction of thioglycosides. We would now like to report the efficient hypervalent iodine induced glycosylation reaction (Scheme 1).

Results and Discussion

We initially examined the glycosylation with methyl 2,3,4,6-tetra-*O*-acetyl-1-thio-β-D-glucopyranoside (**1a**)[20] with methyl 2,3,4-tri-*O*-benzyl-α-D-glucopyranoside (**2**) using the combination of hypervalent iodine reagent and an acid. First, the reaction mixture was treated with 1 equiv. of phenyliodine diacetate (PIDA) (Table 1, Entry 1), phenyliodine bis(trifluoroacetate) (PIFA) (Entry 2), PhI(OH)OTs (HTIB) (Entry 3), and three other kinds of iodine reagents (Entries 4–6) in the presence of TfOH (2 equiv.) in dichloromethane at –78 °C. As a result, the reaction with PIFA and TfOH (Entry 2) afforded the best yield (40%) among the reactions from Entries 1 to 6. It is worth noting that the isolated yield (40%) of product **3a** in Entry 2 was two times higher than that of the previously reported reaction, in which the same substrates **1**, **2** were treated with PhI=O in the presence of Tf_2O in acetonitrile.[19a,19b] Thus, several acids were then utilized as the additive instead of TfOH under the same reaction conditions. By choosing trimethylsilyl triflate as the additive, the product **3a** was obtained in the same yield as that shown in Entry 2; however, the use of tris(pentafluorophenyl)borane, the trifluoroborane–diethyl ether complex, bis(cyclohexyl)(trifluoro-

Table 1. Glycosylation reactions of **1a**, **1b** and **2** with various hypervalent iodine reagents.[a,b]



1a: R = OAc
1b: R = NPhth

2

3a: R = OAc
3b: R = NPhth

Entry	Donor	Hypervalent iodine reagent (1 equiv.)	Product	Yield [%]
1	**1a**	PIDA	**3a**	20
2	**1a**	PIFA	**3a**	40
3	**1a**	PhI(OH)OTs	**3a**	trace
4	**1a**	p-$MeC_6H_4I(OCOCF_3)_2$	**3a**	23
5	**1a**	p-$F_3CC_6H_4I(OCOCF_3)_2$	**3a**	30
6	**1a**	$C_6F_5I(OCOCF_3)_2$	**3a**	30
7	**1b**	PIFA	**3b**	77

[a] Reactions were carried out by adding the I^{III} reagent (0.2 mmol) to a stirred solution of the starting glycosyl donor **1** (0.2 mmol) and glycosyl acceptor **2** (0.3 mmol) in CH_2Cl_2 (4 mL) in the presence of TfOH (0.4 mmol) at –78 °C. [b] Yield of isolated product.

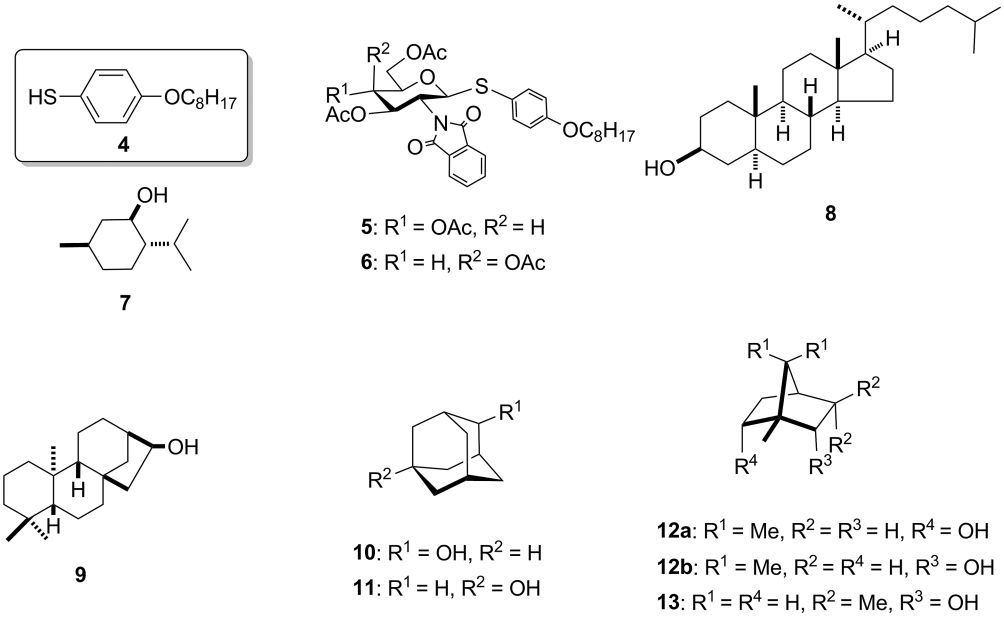


Figure 1. Structures of thioglycosides **5** and **6** and hydrophobic alcohols **7**–**13** chosen as the glycosyl acceptor.

methanesulfonyl)borane, and methanesulfonic acid produced no significant results. Thus, the combination of PIFA and TfOH was hereafter employed as the activator of the thioglycoside donor in the glycosylation reactions. Interestingly, the glycosylation reaction with methyl 2-deoxy-2-phthalimido-3,4,6-tri-*O*-acetyl-1-thio-D-glucopyranoside (**1b**) instead of compound **1a** as the glycosyl donor afforded the appropriate product **3b** in a much higher yield (Entry 7). 1-Thioglycosides bearing a phthalimido group at the C-2 position of a pyranose ring were then nominated as excellent candidates for the glycosyl donor to study the effectiveness of PIFA in the glycosylation reaction.

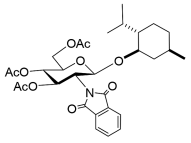
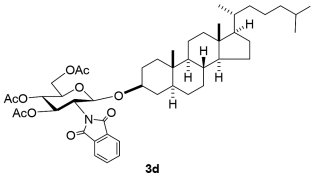
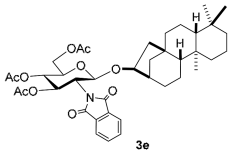
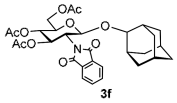
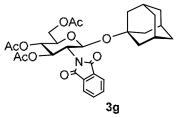
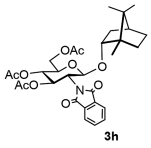
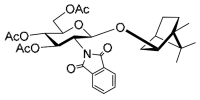
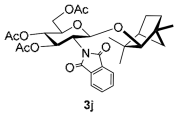
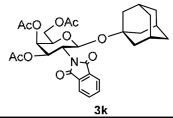
Moreover, the odorless *p*-(octyloxyl)benzenethiol (**4**)[21,22] was chosen as the source of the aglycon moiety of the 1-thioglycosides to avoid tedious handling of odoriferous organosulfur compounds like methanethiol or benzenethiol, which were generated during the conventional glycosylation reaction with methyl or phenyl 1-thioglycosides. Based on these results, *p*-(octyloxy)phenyl 2-deoxy-2-phthalimido-3,4,6-tri-*O*-acetyl-1-thio-β-D-glucopyranoside (**5**) was adopted as the first choice of the donor glycoside and synthesized by the method in the literature.[22,23] In addition, *p*-(octyloxy)phenyl 2-deoxy-2-phthalimido-3,4,6-tri-*O*-acetyl-1-thio-β-D-galactopyranoside (**6**) was also prepared from 2-deoxy-2-phthalimido-D-galactose tetraacetate[24] and **4** with good yield (83%) in the same way as the synthesis of **5**; i.e., **6** was obtained by the reaction of 2-deoxy-2-phthalimino-D-galactose tetraacetate with **4** in the presence of $BF_3{\cdot}Et_2O$ in CH_2Cl_2.

Herein, (–)-menthol (**7**), cholestanol (**8**), *ent*-17-nor-kauranol (**9**),[25] 2- and 1-adamantanols (**10**, **11**), (–)- and (+)-borneols (**12a**, **b**), and (+)-fenchyl alcohol (**13**) were chosen as the acceptors (Figure 1).

In the beginning, thioglycoside **5** and alkanols bearing the simple six-membered cyclohexane ring (**7**, **8**) were treated with PIFA in the presence of TfOH in dichloromethane at 0 °C (Table 2, Entries 1 and 2). The products **3c** and **3d** were obtained in moderate to good yields. Next, alkanols bearing a secondary alcohol on a rigid fused ring (**9**, **10**) were employed as the acceptor substrate in the same reaction to afford the appropriate glycosylated products **3e** and **3f** with almost the same yield (Entries 3 and 4). Surprisingly, the tertiary alcohol (**11**) was glycosylated in 71% under the same reaction condition in spite of the steric hindrance caused by the adamantane skeleton (Entry 5). Moreover, the substrates carrying a much more hindered alcohol, such as the (–)- and (+)-borneols (**12a**, **12b**) and (+)-fenchyl alcohol (**13**), were glycosylated with **5** in quantitative yields (Entries 6–8). As expected, 1-adamantanol (**11**) was glycosylated with **6** to afford adamantanyl 2-deoxy-2-phthalimido-β-D-galactopyranoside **3k** in a good yield (Entry 9).

Finally, the synthesis of disaccharides by the above glycosylation was also performed with methyl 2,3,4-tri-*O*-benzyl-α-D-glucopyanoside (**2**) and **5** as the acceptor and the donor substrates, respectively. Since the former was "armed" and the latter was "disarmed",[24] the combination seemed to be one of the most undesired ones; however, the reaction occurred to provide the desired disaccharide **3b** in an excellent yield (87%) (Table 3, Entry 1). Being encouraged by the above result, the glycosylation reactions with **6** as the donor and **2** as the acceptor substrate were chal-

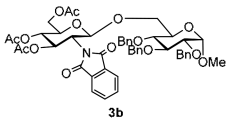
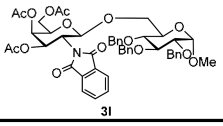
Table 2. Glycosylation of thioglycosides and several alkanols by the activation of PIFA with TfOH.[a,b]

Entry	Donor	Acceptor	Product	Yield [%]
1	**5**	**7**	 **3c**	68
2	**5**	**8**	 **3d**	76
3	**5**	**9**	 **3e**	83
4	**5**	**10**	 **3f**	70
5	**5**	**11**	 **3g**	71
6	**5**	**12a**	 **3h**	93
7	**5**	**12b**	 **3i**	84
8	**5**	**13**	 **3j**	97
9	**6**	**11**	 **3k**	65

[a] Reactions were carried out by adding PIFA (0.2 mmol) to a stirred solution of the starting glycosyl donor **5** or **6** (0.2 mmol) and glycosyl acceptors **7**–**13** (0.3 mmol) in CH_2Cl_2 (4 mL) in the presence of TfOH (0.4 mmol) at 0 °C. [b] Yield of isolated product.

lenged to obtain the disaccharides **3l**. As shown in Table 3, the appropriate disaccharides were provided in good yields.

Table 3. Disaccharide synthesis by thioglycosylation with PIFA/TfOH.[a,b]

Entry	Donor	Acceptor	Product	Yield [%]
1	**5**	**2**	 **3b**	87
2	**6**	**2**	 **3l**	63

[a] Reactions were carried out by adding PIFA (0.2 mmol) to a stirred solution of the starting glycosyl donor **5** or **6** (0.2 mmol) and glycosyl acceptor **2** (0.3 mmol) in CH_2Cl_2 (4 mL) in the presence of TfOH (0.4 mmol) at –78 °C. [b] Yield of isolated product.

Conclusions

We found that the hypervalent iodine compound PIFA is an excellent promoter of the glycosylation reaction of thioglycosides in the presence of TfOH, and the reaction afforded satisfactory results especially with the thioglycosides (**5**, **6**), which have a phthalimido group at the C-2 position. In addition, our method could allow the glycosylation of the undesirable combination of substrates, i.e., the combination of the "disarmed" donor and "armed" acceptor, in an excellent yield. Further studies are now in progress.

Experimental Section

To a stirred solution of *p*-(octyloxy)phenyl 2-deoxy-2-phthalimido-3,4,6-tri-*O*-acetyl-1-thio-β-D-glucopyranoside (**5**) (1.5 equiv.) and methyl 2,3,4-tri-*O*-benzyl-α-D-glucopyranoside (**2**) (1 equiv.) in the presence of molecular sieves (4 Å) in CH_2Cl_2 (0.05 M), TfOH (2 equiv.) was added at –78 °C. Then PIFA (1 equiv.) was subsequently added to the reaction mixture with stirring and the mixture stirred for an additional 3 h under the same conditions, while the reaction progress was monitored by TLC. Saturated aqueous sodium hydrogen carbonate solution was added to the mixture when the reaction completed. The aqueous phase was extracted with CH_2Cl_2. The combined organic phases were dried with anhydrous Na_2SO_4 and then concentrated to dryness. The crude residue was purified by column chromatography on silica gel (eluent: *n*-hexane/CH_2Cl_2) to give the pure glycosylation product **3b** in 87% yield.

Acknowledgments

This work was partially supported by Grants-in-Aid for Scientific Research (A) from the Japan Society for the Promotion of Science (JSPS), a Grant-in-Aid for Scientific Research on Innovative Areas "Advanced Molecular Transformation by Organocatalysts" from The Ministry of Education, Culture, Sports, Science and Technology (MEXT). T. K. acknowledges support from the Grant-in-Aid for Scientific Research (C). K. M. also acknowledges support from the Grant-in-Aid for Young Scientists (B) from the JSPS.

[1] See, e.g.: T. Kajimoto, M. Node, *Curr. Top. Med. Chem.* **2009**, *9*, 13–33.
[2] See, e.g.: a) A. Liptak, A. Borbas, I. Bajza in *Comprehensive Glycoscience: From Chemistry to Systems Biology* (Ed.: J. P. Kamerling), Elsevier, Amsterdam, **2007**, vol. 1, pp. 203–259; b) C.-C. Wang, J.-C. Lee, S.-Y. Luo, S. S. Kulkarni, Y.-W. Huang, C.-C. Lin, K.-L. Chang, S.-C. Hung, *Nature* **2007**, *446*, 896–899; c) C.-C. Wang, S. S. Kulkarni, J.-C. Lee, S.-Y. Luo, S.-C. Hung, *Nat. Protoc.* **2008**, *3*, 97–113; d) X. Zhu, R. R. Schmidt, *Angew. Chem. Int. Ed.* **2009**, *48*, 1900–1934; *Angew. Chem.* **2009**, *121*, 1932–1967.
[3] See, e.g.: a) S. Hanessian, Y. Guindon, *Carbohydr. Res.* **1980**, *86*, C3–C6; b) K. C. Nicolaou, M. R. Pavia, S. P. Sitz, *J. Am. Chem. Soc.* **1982**, *104*, 2027–2029; c) S. Nunomura, M. Mori, Y. Ito, T. Ogawa, *Tetrahedron Lett.* **1989**, *30*, 6713–6716; d) A. Hasegawa, H. Ohki, T. Nagahama, H. Ishida, M. Kiso, *Carbohydr. Res.* **1991**, *212*, 277–281; e) K. Toshima, K. Tatsuta, *Chem. Rev.* **1993**, *93*, 1503–1531; f) Jona, K. Takeuchi, T. Saitoh, T. Mukaiyama, *Chem. Lett.* **2000**, 1178–1179; g) D. Crich, M. Smith, *Org. Lett.* **2000**, *2*, 4067–4069; h) D. Crich, M. Smith, *J. Am. Chem. Soc.* **2001**, *123*, 9015–9020; i) J. D. C. Codee, R. E. J. N. Litjens, R. den Heeten, H. S. Overkleeft, J. H. van Boom, G. A. van der Marel, *Org. Lett.* **2003**, *5*, 1519–1522; j) S. G. Duron, T. Polat, C. H. Wong, *Org. Lett.* **2004**, *6*, 839–841; k) C. Wang, H. Wang, H. Huang, L. H. Zhang, X. S. Ye, *Synlett* **2006**, 2864–2850; l) D. Crich, H. Li, *J. Org. Chem.* **2002**, *67*, 4640–4646; m) J. Dinkelaar, J. D. C. Codee, L. van den Bos, H. S. Overkleeft, G. A. van der Marel, *J. Org. Chem.* **2007**, *72*, 5737–5742; n) J. Tatai, P. Fugedi, *Org. Lett.* **2007**, *9*, 4647–4650; o) A. H. A. Chu, S. H. Nguyen, J. A. Sisel, A. Minciunescu, C. S. Bennett, *Org. Lett.* **2013**, *15*, 2566–2569; p) H. He, X. Zhu, *Org. Lett.* **2014**, *16*, 3102–3105.
[4] a) R. J. Ferrier, R. W. Hay, N. Vethaviyaear, *Carbohydr. Res.* **1973**, *27*, 65–70; b) T. Y. R. Tsai, H. Jin, K. Wiesner, *Can. J. Chem.* **1984**, *62*, 1403–1413; c) P. J. Garegg, C. Henrichson, T. Norberg, *Carbohydr. Res.* **1983**, *116*, 162–165; d) J. W. van Cleve, *Carbohydr. Res.* **1979**, *70*, 161–164; e) S. Haneasian, C. Bacquet, N. Lehong, *Carbohydr. Res.* **1980**, *80*, C17–C22.
[5] a) T. Mukaiyama, T. Nakatauki, S. Shoda, *Chem. Lett.* **1979**, 487–488; b) S. Sato, M. Mori, Y. Ito, T. Ogawa, *Carbohydr. Res.* **1986**, *156*, C6–C10.
[6] G. H. Veeneman, S. H. van Leeuwen, J. H. van Boom, *Tetrahedron Lett.* **1990**, *31*, 1331–1334.
[7] K. Fukase, A. Hasuoka, I. Kinoshita, Y. Aoki, S. Kusumoto, *Tetrahedron* **1995**, *51*, 4923–4932.
[8] R. Perion, L. Lemee, V. Ferrieres, R. Duval, D. Plusquellec, *Carbohydr. Res.* **2003**, *338*, 2779–2792.
[9] B. Mukhopadhyay, B. Collet, R. A. Field, *Tetrahedron Lett.* **2005**, *46*, 5923–5925.
[10] C. Mukherjee, A. K. Mirsa, *Synthesis* **2007**, 683–685.
[11] K. Takeuchi, T. Tamura, T. Mukaiyama, *Chem. Lett.* **2000**, 124–125.
[12] R. M. Salmasan, Y. Manabe, Y. Kitawaki, T.-C. Chang, K. Fukase, *Chem. Lett.* **2014**, *43*, 956–958.
[13] C.-H. Yao, J.-C. Lee, *Tetrahedron* **2014**, *70*, 6757–6762.
[14] For recent reviews and publications, see: a) P. J. Stang, V. V. Zhdankin, *Chem. Rev.* **1996**, *96*, 1123–1178; b) Y. Kita, T. Takada, H. Tohma, *Pure Appl. Chem.* **1996**, *68*, 627–637; c) A. Varvoglis, *Hypervalent Iodine in Organic Synthesis*, Academic Press, San Diego, CA, **1997**; d) H. Tohma, Y. Kita, *Top. Curr. Chem.* **2003**, *224*, 209–248; e) H. Tohma, Y. Kita, *Adv. Synth. Catal.* **2004**, *346*, 111–124; f) V. V. Zhdankin, P. J. Stang, *Chem. Rev.* **2008**, *108*, 5299–5358; g) E. A. Merritt, B. Olofsson, *Angew. Chem. Int. Ed.* **2009**, *48*, 9052–9070; *Angew. Chem.* **2009**, *121*, 9214–9234; h) T. Dohi, Y. Kita, *Chem. Commun.* **2009**, 2073–2085; i) A. Duschek, S. F. Kirsch, *Angew. Chem. Int. Ed.*

2011, *50*, 1524–1552; *Angew. Chem.* **2011**, *123*, 1562–1590; j) E. A. Merritt, B. Olofsson, *Synthesis* **2011**, 517–528; k) D. F. Gonzalez, F. Benfatti, J. Waser, *ChemCatChem* **2012**, *4*, 955–958; l) M. S. Yusubov, V. V. Zhdankin, *Curr. Org. Synth.* **2012**, *9*, 247–272 and references cited therein.

[15] a) Y. Kita, M. Egi, M. Ohtsubo, T. Saiki, T. Takada, H. Tohma, *Chem. Commun.* **1996**, 2225–2226; b) Y. Kita, M. Egi, H. Tohma, *Chem. Commun.* **1999**, 143–144.

[16] The oxidation of sulfides to sulfoxides was reported by using a hypervalent iodine(III) reagent: a) H. Tohma, S. Takizawa, H. Watanabe, Y. Kita, *Tetrahedron Lett.* **1998**, *39*, 4547–4550; b) H. Tohma, S. Takizawa, H. Watanabe, Y. Fukuoka, T. Maegawa, Y. Kita, *J. Org. Chem.* **1999**, *64*, 3519–3523; c) H. Tohma, T. Maegawa, Y. Kita, *ARKIVOC* **2003**, *vi*, 62–70; d) H. Tohma, S. Takizawa, H. Morioka, T. Meagawa, Y. Kita, *Chem. Pharm. Bull.* **2000**, *48*, 445–446; the unstable hypervalent iodine reagent (phenyliodine difluoride) induced fluorination of the α-carbon atom of a sulfide has already been reported: e) S. Caddick, W. B. Motherwell, J. Wilkinson, *J. Chem. Soc., Chem. Commun.* **1991**, 674–675; f) T. Fuchigami, T. Fujita, S. Higashiya, A. Kanno, *J. Chin. Chem. Soc.* **1998**, *45*, 131–133; g) W. B. Motherwell, M. F. Greaney, J. J. Edmunds, J. W. Steed, *J. Chem. Soc. Perkin Trans. 1* **2002**, 2816–2826.

[17] a) G. F. Koser, P. B. Kokil, M. Shah, *Tetrahedron Lett.* **1987**, *28*, 5431–5434; b) D. G. III Ray, G. F. Koser, *J. Org. Chem.* **1992**, *57*, 1607–1610; c) M. Xia, Z. Chen, *Synth. Commun.* **1997**, *27*, 1315–1320; d) W. Qian, L. Pei, *Synlett* **2006**, 709–712.

[18] The PIFA-promoted deprotection of thioketals or thioacetals has been employed: a) G. Stork, K. Zhao, *Tetrahedron Lett.* **1989**, *30*, 287–290; b) F. F. Fleming, L. Funk, R. Altundas, Y. Tu, *J. Org. Chem.* **2001**, *66*, 6502–6504.

[19] a) K. Fukase, A. Hasuoka, I. Kinoshita, S. Kusumoto, *Tetrahedron Lett.* **1992**, *33*, 7165–7168; b) K. Fukase, I. Kinoshita, T. Kanoh, Y. Nakai, A. Hasuoka, S. Kusumoto, *Tetrahedron* **1996**, *52*, 3897–3904; c) A. H. A. Chu, A. Minciunescu, V. Montanari, K. Kumar, C. S. Bennett, *Org. Lett.* **2014**, *16*, 1780–1782.

[20] T. Ogawa, M. Matsui, *Carbohydr. Res.* **1977**, *54*, C17–C21.

[21] T. Kajimoto, Y. Ishioka, T. Katoh, M. Node, *Bioorg. Med. Chem. Lett.* **2006**, *16*, 5736–5739.

[22] T. Kajimoto, Y. Ishioka, T. Katoh, M. Node, *J. Carbohydr. Chem.* **2007**, *26*, 469–495.

[23] O. T. Leong, S. P. Douglas, D. M. Whitfield, H. Y. S. Pang, *New J. Chem.* **1994**, *18*, 349–363.

[24] a) D. R. Mootoo, P. Konradsson, U. Udodong, B. Fraiser-Reid, *J. Am. Chem. Soc.* **1988**, *110*, 5583–5584; b) P. Konradsson, D. R. Mootoo, R. E. McDevitt, B. R. Fraiser-Reid, *J. Chem. Soc., Chem. Commun.* **1990**, 270–272.

[25] M. Node, H. Hori, E. Fujita, *J. Chem. Soc. Perkin Trans. 1* **1976**, 2237–2240.

Received: February 9, 2015
Published Online: March 2, 2015

