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A Vicinal Acyloxy Group Participation S_N2 Reaction of Thiol Nucleophiles in the Formation of Thioacetals

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Thioacetalization of acyl protected furanosides led to products with an ethanethiol group at C-2 and 3-*O*-acetyl-1,2-di-*O*-isopropylidene-*D*-furanoses were converted into corresponding thioacetals with two ethanethiol groups at both C-2 and C-3 positions under the standard thioacetalization conditions. All products were characterized by ^{1}H NMR, ^{13}C NMR and HRMS data. X-ray structure analysis indicates that the vicinal acyloxy group is stereoselectively substituted by ethanethiols. The supposed mechanisms for these two kinds of transformations were presented.

Keywords acetal, thioacetal, protecting group, acyl group

Introduction

Thioacetals are frequently employed as carbonyl protecting groups in synthetic chemistry due to their ready preparation and proper stabilities under both acidic and basic reaction conditions,[1] as well as used as precursors of acyl anion equivalents for formation of carbon-carbon bonds since the pioneer work reported by Corey.[2] In this view, a number of methods for preparation of thioacetals have been developed. Usually, they are prepared via the condensation of carbonyl compounds with thiols in the presence of protic acids or Lewis acids, such as $ZnCl_2$,[3] $NiCl_2$,[4] $InCl_3$,[5] I_2,[6] NBS[7] and SiO_2-SO_3H.[8] Herein, we wish to report a vicinal acyloxy group participation S_N2 reaction of ethanethiol under the standard thioacetalization conditions.

Experimental

General

NMR spectra were measured at 400 MHz with a Varian Mercury 400 spectrometer, operating at 400 MHz for ^{1}H NMR and 100 MHz for ^{13}C NMR spectra. Tetramethylsilane was used as an internal standard, and *J* values are given in Hz. m.p. was measured with an X-6 melting-point apparatus. High resolution electron impact mass spectra (HRMS EI) were recorded on a Bruker Daltonics Inc. APEXII-FT-ICR mass spectrometer. Dichloromethane was dried over phosphorus pentoxide and distilled.

General procedure

$ZnBr_2$ (0.27 g, 1.2 mmol) was added to a solution of sugar derivative (1 mmol) and ethanethiol (0.78 mL, 10.0 mmol) in dry CH_2Cl_2 (5.0 mL). After complete conversion, saturated aqueous solution of $NaHCO_3$ (20 mL) was added and the two layers were separated. The organic layer was washed with brine (20 mL×2), dried ($MgSO_4$) and concentrated under reduced pressure. The residue was isolated through short column chromatography on silica gel, which was eluded with EtOAc-petroleum (1∶9, *V*/*V*) to give the target compound.

5-Deoxy-3,4-di-*O*-acetyl-2-*S*-ethyl-2-thio-*D*-lyxose diethyl dithioacetal (2) m.p. 64 ℃; ^{1}H NMR (DMSO-d_6) δ: 1.08—1.23 (m, 12H), 2.04 (s, 3H), 2.14 (s, 3H), 2.58—2.70 (m, 6H), 3.22 (dd, *J*=2.0, 10.4 Hz, 1H), 4.09 (d, *J*=2.0 Hz, 1H), 5.04 (dd, *J*=2.0, 10.4 Hz, 1H), 5.41 (dd, *J*=1.6, 6.4 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ: 14.59, 14.76, 17.32, 21.21, 21.53, 26.52, 26.66, 26.48, 53.27, 55.33, 69.87, 75.57, 170.14, 170.32; HRMS (EI) calcd for $C_{15}H_{28}O_4S_3$ 368.1150, found 368.1155.

5-Deoxy-3,4-di-*O*-benzoyl-2-*S*-ethyl-2-thio-*D*-lyxose diethyl dithioacetal (6) ^{1}H NMR ($CDCl_3$) δ: 1.21—1.28 (m, 9H), 1.41 (d, *J*=6.4 Hz, 3H), 2.67—2.77 (m, 6H), 3.17—3.20 (m, 1H), 4.13 (d, *J*=5.8 Hz, 1H), 5.95 (dd, *J*=4.8, 5.5 Hz, 1H), 6.01 (t, *J*=4.0 Hz, 1H), 7.38—7.42 (m, 4H), 7.50—7.53 (m, 2H), 8.01—8.04 (m, 4H); ^{13}C NMR ($CDCl_3$) δ: 14.70, 14.77, 20.67, 25.47, 26.19, 53.65, 67.65, 75.61, 75.91, 83.73, 84.40, 127.78, 128.05, 128.40, 128.52, 128.64, 138.51, 138.74; HRMS (EI) calcd for $C_{25}H_{32}O_4S_3$ 492.1463, found 492.1467.

3,4,5-Tri-*O*-acetyl-2-*S*-ethyl-2-thio-*D*-arabinose diethyl dithioacetal (8) ^{1}H NMR ($CDCl_3$) δ: 1.24—1.30 (m, 9H), 2.06 (s, 3H), 2.09 (s, 6H), 2.68—2.75 (m, 6H), 3.12 (dd, *J*=3.2, 5.1 Hz, 1H), 4.06 (d, *J*=5.2 Hz, 1H), 4.15 (dd, *J*=5.8, 12.4 Hz, 1H), 4.40 (dd, *J*=2.7,

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12.4 Hz, 1H), 5.40—5.44 (m, 1H), 5.74 (dd, *J*=3.1, 7.2 Hz, 1H); ^{13}C NMR ($CDCl_3$) *δ*: 9.68, 10.07, 16.23, 16.41, 16.58, 21.46, 22.05, 24.09, 25.16, 48.36, 51.77, 57.33, 66.66, 66.95, 165.21, 165.32, 166.21; HRMS (EI) calcd for $C_{17}H_{30}O_6S_3$ 426.1205, found 426.1211.

3,4,5-Tri-*O*-acetyl-2-*S*-ethyl-2-thio-D-lyxose diethyl dithioacetal (10) m.p. 60 ℃; ^{1}H NMR ($CDCl_3$) *δ*: 1.19—1.30 (m, 9H), 2.05—2.14 (m, 9H), 2.63—2.77 (m, 6H), 3.22 (dd, *J*=3.2, 9.6 Hz, 1H), 4.02—4.07 (m, 2H), 4.28 (dd, *J*=5.6, 11.6 Hz, 1H), 5.35 (dd, *J*=1.2, 9.6 Hz, 1H), 5.32—5.77 (m, 1H); ^{13}C NMR ($CDCl_3$) *δ*: 14.59, 14.67, 14.70, 20.93, 21.09, 21.15, 26.46, 26.50, 29.75, 53.45, 55.24, 63.02, 70.05, 72.27, 169.81, 170.22, 170.64; HRMS (EI) calcd for $C_{17}H_{30}O_6S_3$ 426.1205, found 426.1210.

4,5,6-Tri-*O*-acetyl-2,3-di-*S*-ethyl-2,3-dithio-*D*-allose diethyl dithioacetal (13) m.p. 62 ℃; ^{1}H NMR ($CDCl_3$) *δ*: 1.22—1.34 (m, 12H), 2.00—2.11 (m, 9H), 2.60—2.89 (m, 8H), 3.07 (dd, *J*=5.2, 8.4 Hz, 1H), 3.48 (dd, *J*=5.2, 8.4 Hz, 1H), 4.17—4.22 (m, 1H), 4.49 (dd, *J*=2.4, 12.4 Hz, 1H), 4.64 (d, *J*=4.8 Hz, 1H), 5.52—5.55 (m, 1H), 5.89 (dd, *J*=1.6, 6.4 Hz, 1H); ^{13}C NMR ($CDCl_3$) *δ*: 14.60, 14.74, 14.85, 14.92, 15.02, 21.23, 21.27, 25.79, 26.39, 28.31, 30.24, 51.47, 57.30, 57.48, 62.56, 71.96, 72.78, 169.19, 169.61, 170.82; HRMS (EI) calcd for $C_{20}H_{36}O_6S_4$ 500.1395, found 500.1403.

5-Deoxy-4-*O*-acetyl-2,3-di-*S*-ethyl-2,3-dithio-*D*-ribose diethyl dithioacetal (15) ^{1}H NMR ($CDCl_3$) *δ*: 1.22—1.33 (m, 15H), 2.04 (s, 3H), 2.60—2.87 (m, 9H), 3.34 (dd, *J*=4.4, 9.6 Hz, 1H), 4.78 (d, *J*=4.4 Hz, 1H), 5.63 (dd, *J*=4.4, 6.4 Hz, 1H); ^{13}C NMR ($CDCl_3$) *δ*: 14.62, 14.76, 15.03, 15.23, 16.30, 21.63, 26.32, 26.46, 28.87, 29.90, 54.61, 56.21, 57.61, 72.34, 170.12; HRMS (EI) calcd for $C_{15}H_{30}O_2S_4$ 370.1129, found 370.1134.

4,5-Di-*O*-acetyl-2,3-di-*S*-ethyl-2,3-dithio-*D*-ribose diethyl dithioacetal (17) ^{1}H NMR ($CDCl_3$) *δ*: 1.26—1.32 (m, 12H), 2.08 (d, *J*=5.5 Hz, 6H), 2.69—2.88 (m, 8H), 3.02—3.04 (m, 1H), 3.56 (t, *J*=6.8 Hz, 1H), 4.42 (dd, *J*=6.2, 12.2 Hz, 1H), 4.49 (d, *J*=2.8 Hz, 1H), 4.54 (dd, *J*=2.8, 17.1 Hz, 1H), 5.63—5.66 (m, 1H); ^{13}C NMR ($CDCl_3$) *δ*: 14.57, 14.74, 14.86, 15.08, 21.06, 21.38, 25.82, 26.52, 28.47, 30.29, 50.55, 56.60, 57.56, 63.85, 73.63, 169.86, 170.85; HRMS (EI) calcd for $C_{17}H_{32}O_4S_4$ 428.1183, found 428.1190.

5-Deoxy-3-*O*-benzoyl-*D*-xylose diethyl dithioacetal (19) ^{1}H NMR ($CDCl_3$) *δ*: 1.21—1.33 (m, 9H), 2.70—2.86 (m, 4H), 3.36—3.40 (m, 1H), 4.38—4.50 (m, 1H), 5.32 (d, *J*=7.2 Hz, 1H), 5.60—5.66 (m, 1H), 7.43—7.51 (m, 2H), 7.56—7.62 (m, 1H), 8.11 (d, *J*=7.6 Hz, 2H).

3,5-Di-*O*-benzoyl-*D*-xylose diethyl dithioacetal (21) ^{1}H NMR ($CDCl_3$) *δ*: 1.22—1.37 (m, 6H), 2.64—2.69 (m, 2H), 2.76—2.81 (m, 2H), 3.42 (dd, *J*=5.1, 7.4 Hz, 12H), 4.50 (dd, *J*=5.7, 11.4 Hz, 1H), 4.60 (dd, *J*=6.6, 11.4 Hz, 1H), 4.69—4.72 (m, 1H), 5.37 (d, *J*=7.5 Hz, 1H), 5.91 (dd, *J*=3.4, 5.2 Hz, 1H), 7.40—7.57 (m, 6H), 7.98—8.08 (m, 4H).

5,6-Di-*O*-acetyl-3-*O*-Ts-*D*-glucose diethyl dithioacetal (23) ^{1}H NMR ($CDCl_3$) *δ*: 1.25—1.33 (m, 6H), 2.07 (s, 3H), 2.14 (s, 3H), 2.44 (s, 3H), 2.763—2.78 (m, 4H), 3.87 (dd, *J*=3.1, 8.2 Hz, 1H), 4.12—4.15 (m, 2H), 4.28 (dd, *J*=3.3, 12.4 Hz, 1H), 4.56 (dd, *J*=2.7, 12.4 Hz, 1H), 4.80—4.83 (m, 1H), 5.43 (t, *J*=2.4 Hz, 1H).

Results and discussion

In the course of our synthesis of nucleosides, we initially wished to convert compound **1** into thioacetal **3** via treatment of methyl 2,3-di-*O*-acetyl-5-deoxy-*D*-xylofuranoside (**1**) with ethanethiol (5 equiv.) in the presence of zinc bromide (1.1 equiv.) in dichloromethane solution at room temperature. In fact, thioacetal **2** with a thioether group at C-2 was obtained as crystalline in 50% yield without the desired product after a series of attempts. Furthermore, in the presence of $ZnCl_2$, Dowex 50 W, *p*-methylbenzenesulfonic acid (PTSA) or 6 mol/L HCl, no product was formed under various conditions (Entries 1—4, Table 1). Compound **2** was formed as a main product as BF_3•Et_2O, $BiCl_3$ and $ZnBr_2$ were used as catalysts, respectively (Entries 5—7, Table 1), which is similar to the results reported by Blumberg *et al*.[9]

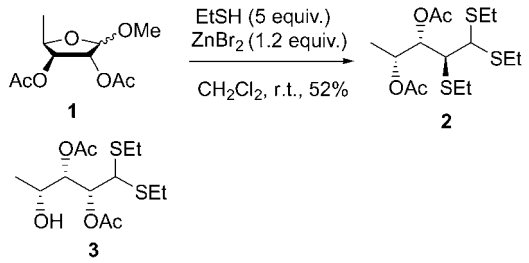


Table 1 Treatment of **1** with ethanethiol with different catalysts

Entry	Catalyst	Solvent	Time/h	Product (yield)
1	$ZnCl_2$	CH_2Cl_2	48	no
2	DowX	MeOH	48	no
3	6 mol/L HCl	MeOH	32	no
4	PTSA	CH_2Cl_2	48	no
5	BF_3•Et_2O	CH_2Cl_2 (r.t. or −10 ℃)	18	**2** (45%)
6	$BiCl_3$	CH_2Cl_2 (r.t. or −10 ℃)	32	**2** (47%)
7	$ZnBr_2$	CH_2Cl_2 (r.t. or −10 ℃)	15	**2** (50%)

When compound **4** was treated with ethanethiol under similar reaction conditions, compound **2** was also generated in 52% and 50% yields in the presence of $ZnBr_2$ and $BiCl_3$, respectively (Entry b, Table 2), which was recrystallized from petroleum ether (30—60 ℃). The molecular structure of compound **2** was fully characterized by ^{1}H NMR, ^{13}C NMR and HRMS data, and a single-crystal X-ray diffraction study (Figure 1). X-ray crystallographic analysis allowed absolute confirmation of the molecular structure and the configuration of the second carbon atom to be anticlockwise that is contrary to its precursor. These results might suggest that the 2-acetyloxy group participate this transformation and be substituted by ethanethiol via an S_N2 mechanism.

Neighboring acyloxy group participating reaction is well known[10] and has extensively been employed in stereoselective synthesis,[11] particularly, in the synthesis of glycosides[12] and nucleosides.[13] As anticipated, products with similar molecular structures, **6**, **8** and **10**, were formed when benzoyl protected compound **5**, *D*-ribose derivative **7** and *D*-xylose derivative **9** were treated with ethanethiol under the same conditions as above, respectively (Entries c, d and e, Table 2).

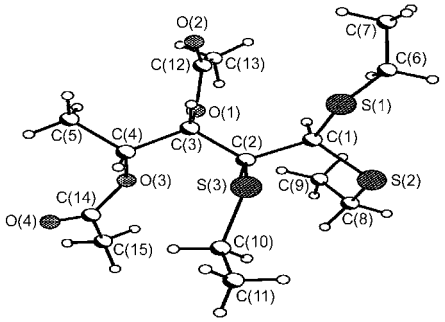
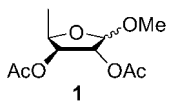
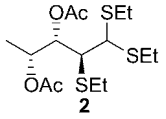
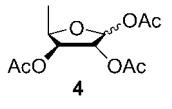
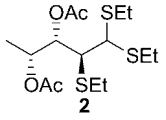
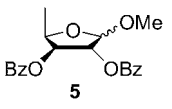
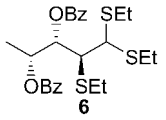
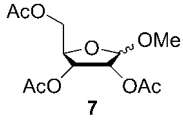
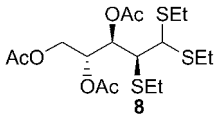
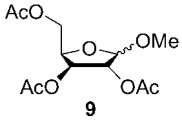
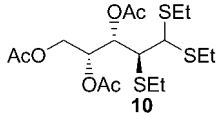


Figure 1 ORTEP view of the X-ray crystal structure of compound **2** (Crystallographic data have been deposited with the CCDC under reference no. 644093). Monoclinic, $P3_2$, a = 8.9934(13) Å, b=8.9934(13) Å, c=22.219(4) Å, β=90.00°, V= 1556.33 Å^3, Z=3, and μ=0.369 mm^{-1}.

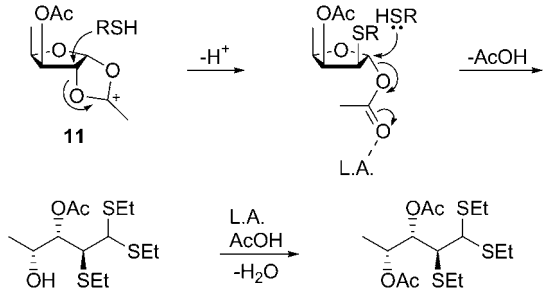
Table 2 2-Acyloxy participating thioacetalization of glycosides promoted by $ZnBr_2$

Entry	Substrate	Product	Time/h	Yield/%
a	 **1**	 **2**	15 30	50 47[a]
b	 **4**	 **2**	15 30	52 50[a]
c	 **5**	 **6**	20	60
d	 **7**	 **8**	24	84
e	 **9**	 **10**	19	47

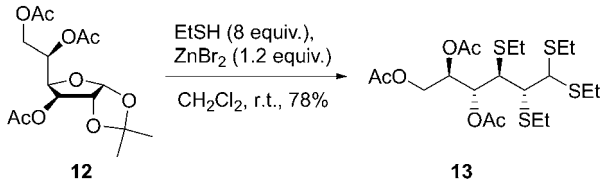
[a] Promoted by $BiCl_3$.

In view of the fact that the configurations of C-2 and C-4 of compound **2** are reversed and maintained, respectively (Figure 1), it might suggest that the 2-acyloxy group participates to disperse the positive charge of the anomeric cation generated in the presence of $ZnBr_2$ to give a corresponding more stable acetoxonium species **11** (Scheme 1), which directs the attack of nucleophile, ethanethiol, from the top face at C-2, meanwhile, the 2-acyloxy group moves to C-1 (Scheme 1).

Scheme 1 The proposed mechanism for the formation of α-thioether group in thioacetalization reaction



On the other hand, as 3,5,6-tri-*O*-acetyl-1,2-di-*O*-isopropylidene-*D*-glucofuranose (**12**) was treated with thiols under similar conditions as above, product **13** was formed, in which there are two thioether groups at both C-2 and C-3 (Entry a, Table 3), respectively.[14] Molecular structure of **13** was identified with ^{1}H NMR, ^{13}C NMR and HRMS data, and a single-crystal X-ray diffraction study (Figure 2). X-ray crystallographic analysis of **13** showed absolute confirmation of the configurations of the C-2 and C-3 to be both clockwise. Compared to its precursor **12**, the configurations of C-2 and C-3 are kept and inversed, respectively (Figure 2).



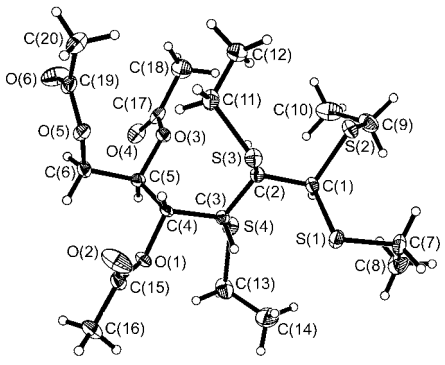
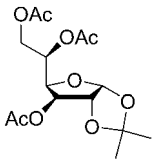
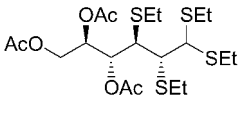
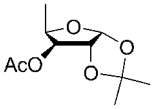
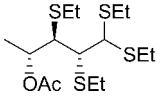
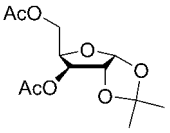
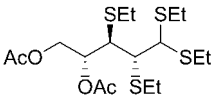
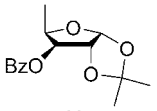
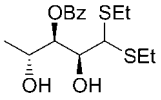
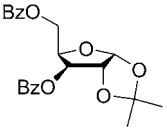
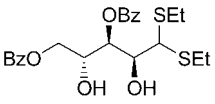
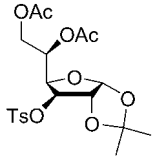
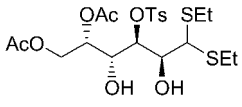


Figure 2 ORTEP view of the X-ray crystal structure of compound **13** (Crystallographic data have been deposited with the CCDC under reference no. 665474). Monoclinic, $P2_1$, a = 8.3395(12) Å, b=16.726(2) Å, c=9.2027(14) Å, α=90°, β= 95.772(5)°, γ=90°, V=1277.15 Å^3, and μ=0.403 mm^{-1}.

Table 3 Thioacetalization of 3-*O*-acyl-1,2-di-*O*-isopropylidene-*D*-furanoses promoted by $ZnBr_2$

Entry	Substrate	Product	Time/h	Yield/%
a	 **12**	 **13**	5	78
b	 **14**	 **15**	8	64
c	 **16**	 **17**	15	86
d	 **18**	 **19**	6	72
e	 **20**	 **21**	4	61
f	 **22**	 **23**	2	95

Under similar reaction conditions, **15** and **17** (Entries b and c, Table 3) were obtained in moderate to good yields as expected. Both **18** and **20** were, however, converted into thioacetal **19** and **21**, respectively, without ethanethio groups, which is similar to the thioacetalization product reported by Nicolaou as a 3-deoxy-1,2-di-*O*-isopropylidene-*D*-ribofuranose derivative was treated with ethanethiol using zinc chloride as catalysts.[15] Additionally, 3-tosylate glucose derivative **22** was converted into corresponding thioacetal **23** without any thioether group. It is well known that tosylate groups were usually used as a non-participating group in organic synthesis,[16] both the 3-acetyloxy and 1,2-di-*O*-isopropylidene groups thus might take part in thiol substitution process and the 3-acetyloxy group as a neighboring-group maybe play an important role via migration from C-3 to C-1. We could not exactly describe the mechanism for the ethanethio substitution approach due to the absence of undouted evidences although we can not thoroughly agree with the proposal of ethanethio group migration from C-1 to C-2 and then to C-3 described by Ferrier and his coworkers.[9]

Conclusion

We demonstrated that acyl protected furanosides were converted into corresponding thioacetals with a thiol ether group at C-2 and 3-*O*-acetyl-1,2-di-*O*-isopropylidene-*D*-furanoses were converted into corresponding thioacetals with two thioether groups at both C-2 and C-3 under the standard thioacetalization conditions. The molecular structure was elucidated by ^{1}H NMR, ^{13}C NMR and HRMS data, and X-ray analysis and the supposed mechanism was depicted as neighboring acyloxy group participation and substitution by ethanethiol via an S_N2 manner.

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