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Carbohydrate Research 317 (1999) 204–209

CARBOHYDRATE RESEARCH

Note

Convenient synthesis of 2,3-*O*-isopropylidene-5-thio-D-ribose and 5-thio-D-ribose; synthesis of 1,4-anhydro-2,3-*O*-isopropylidene-α-D-ribopyranose and 1,4-anhydro-2,3-*O*-isopropylidene-5-thio-α-D-ribopyranose☆

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Received 1 December 1998; accepted 26 February 1999

Abstract

Sequential mesylation–acetylation of 2,3-*O*-isopropylidene-D-ribofuranose gave 1-*O*-acetyl-2,3-*O*-isopropylidene-5-*O*-methanesulfonyl-β-D-ribofuranose that was converted into 1-*O*-acetyl-5-(*S*)-acetyl-2,3-*O*-isopropylidene-5-thio-β-D-ribose, deacetylation of which gave 2,3-*O*-isopropylidene-5-thio-D-ribose as the β-pyranose form, which was hydrolysed to 5-thio-D-ribose. 1,4-Anhydro-2,3-*O*-isopropylidene-α-D-ribopyranose was obtained by sodium methoxide treatment of 1-*O*-acetyl-2,3-*O*-isopropylidene-5-*O*-methanesulfonyl-β-D-ribofuranose and 1,4-anhydro-2,3-*O*-isopropylidene-5-thio-α-D-ribopyranose was similarly synthesised via 1-(*S*)-acetyl-2,3-*O*-isopropylidene-5-*O*-methanesulfonyl-5-thio-α-D-ribofuranose.

Keywords: Anydro sugar; Furanose–pyranose equilibria; Methanesulfonate esters; 5-Thio-D-ribose derivatives; Thio sugars

When D-ribose is treated with acetone in the presence of an acid catalyst the major products are of the furanose form: the 2,3-acetal **1** together with smaller amounts of the isomeric 1,2-acetal and the anhydro compound **13**; a small amount of the pyranose 1,2:3,4 diacetal **12** is also formed [2]. In contrast, similar treatment of 5-thio-D-ribose (**11**) gives the 1,2:3,4-diacetal **9** as a major product, demonstrating the strong preference for the sulfur-in-the-ring pyranose form [3]. The bicyclic system in the acetal **1** is particularly stable and it was of interest to see (i) if it discouraged the furanose to pyranose conversion in the corresponding 5-thio analogue **5** and (ii) if it favoured the formation of the thiopyranose compound **10** from **5** in acid conditions.

Selective mesylation of the primary hydroxy group in **1** gave the mesylate **2b** ($J_{1,2} = 0$ Hz),

☆ 5-Thiopyranoses, Part 14. For Part 13, see Ref. [1].

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0008-6215/99/$ - see front matter
PII: S0008-6215(99)00064-6

Table 1
^{1}H NMR data: chemical shifts (ppm) and coupling constants (Hz)

Compd	H-1	H-2	H-3	H-4	H-5a	H-5b	Other signals	$J_{1,2}$	$J_{2,3}$	$J_{3,4}$	$J_{4,5a}$	$J_{4,5b}$	$J_{5a,5b}$	
1[a]	5.41	4.69	4.85	4.28	←3.66→		1.48, 1.35 (CMe_2)	0	5.9	1.0	5.8	5.8		
2	5.51	←4.2–4.8→					1.49, 1.34 (CMe_2); 3.09, ($MeSO_2$)	0						
3a	6.20	4.84	4.73	4.50	←4.38→		1.57. 1.37 (CMe_2); 3.06 ($MeSO_2$); 2.14(AcO)	4.4	6.9	2.9	3.2	3.2		
3b	6.20	←4.74→		4.52	4.33	4.21	1.54, 1.35 (CMe_2); 3.08 ($MeSO_2$); 2.10 (AcO)	0		0	7.0	6.0	10.8	
4a	6.17	4.81	4.45	4.43	3.22	3.17	1.51, 1.34(CMe_2); 2.39 (AcS); 2.10 (AcO)	4.5	6.7	3.1	5.0	5.5	13.8	
4b	6.22	4.63	4.75	4.30	3.11	3.04	1.48, 1.32 (CMe_2); 2.38 (AcS); 2.10 (AcO)	0	5.9	0	8.5	7.3	13.8	
6a[a]	4.96				2.99	2.66	1.52, 1.38 (CMe_2)	4.2			7.6	3.2	12.6	
6b[a]	4.90	4.30	4.47	4.28	2.86	2.62	1.52, 1.38 (CMe_2)	6.8	6.0	3.5	10.8	4.5	12.4	1.0 ($J_{3,5b}$)
7b	4.56	4.47	4.39	4.22	2.82	2.66	1.59, 1.40 (CMe_2); 3.45 (MeO); 2.35 (OH)	4.9	6.9	3.1	9.8	4.6	11.7	0.9 ($J_{3,5b}$) 8.3 ($J_{1,OH}$)
8b[b]	6.03	4.15	4.20	5.43	2.97	2.43	1.50, 1.11 (CMe_2); 1.62, 1.57 (AcO)	4.1	7.0	2.3	11.3	4.7	10.9	1.0 ($J_{3,5b}$)
10	5.49	4.28	4.56	4.87	2.88	2.64	1.46, 1.28 (CMe_2)	0	5.4	0	4.9	0	9.8	
13	5.45	4.29	4.33	4.71	3.43	3.32	1.46,1.29 (CMe_2)	0	5.5	0	3.7	0	7.2	
15a	6.19	4.87	4.76	4.57	←4.45→		1.67, 1.40 (CMe_2); 3.07 (Me SO_2)	4.5	7.3	3.9	3.0	3.0		
15b	6.17	5.03	4.88	4.58	←4.43→		1.49, 1.35 (CMe_2); 3.10 ($MeSO_2$)	0	5.7	1.5	6.0	7.6		
16a	6.00	4.95	4.79	←4.2–4.5→			1.55, 1.36 (CMe_2);3.13 ($MeSO_2$); 2.39 (AcS)	4.7	6.3	1.9				

[a] In D_2O.
[b] In C_6D_6.

which on acetylation gave the acetate–mesylate **3b**; more conveniently and more efficiently **3b** could be obtained by a one-pot sequential mesylation–acetylation of **1**. Displacement of the mesylate group by potassium thioacetate proceeded smoothly to give the thioacetate **4b**. All the above compounds were obtained in crystalline form. Deacetylation of **4b** gave a readily crystalline product whose ^{1}H NMR spectrum clearly showed it to be the β-pyranose form **6b** by the similarity of the coupling constants with those of the ^{1}H NMR spectrum of the known [3] methyl 2,3-*O*-isopropylidene-5-thio-β-D-ribopyranoside (**7b**) (see Table 1). Hydrolysis of **6b** with aqueous acid gave 5-thio-D-ribose (**11**) [4].

Compound **6b** underwent mutarotation and its final ^{1}H and ^{13}C NMR spectra (D_2O solution) (Table 2) showed the presence of 12% of the α-form **6a**, but no indication of any furanose forms **5**. However, when **6b** was acetylated in pyridine, while the major product was the pyranose diacetate **8b**, the ^{1}H NMR spectrum of the mother liquor material indicated the presence of the furanose diacetate **4b** (5% of the total product), suggesting that in pyridine a small amount of the furanose acetal **5** was present in the equilibrium. Clearly the preference for the sulfur-in-the-ring pyranose form **6** is stronger than that for the bicyclic system of the furanose form **5**.

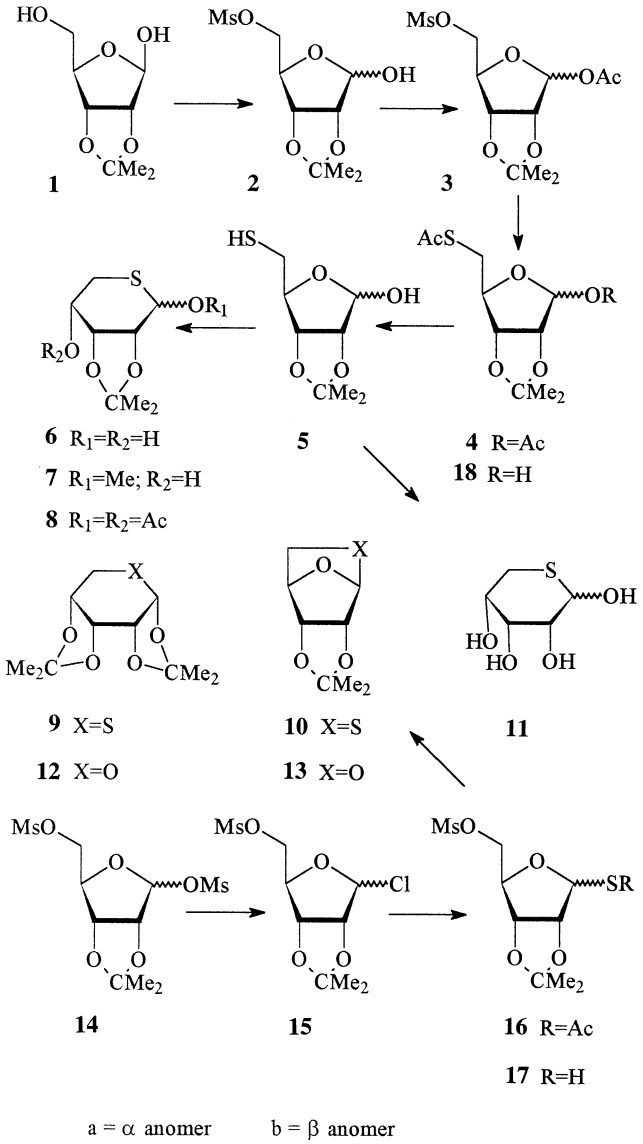
When **6b** was treated with acetone containing sulfuric acid, a small quantity of material was obtained that was identified as the diacetal **9** from its ^{1}H NMR spectrum [3]. Similar treatment of **6b** with sulfuric acid in ether/toluene instead of acetone also resulted in a small quantity of material identified as the anhydro compound **10** (q.v.) from its ^{1}H NMR spectrum. Evidently the replacement of oxygen by sulfur at C-5 had not increased the tendency for cyclisation to occur.

It was observed that treatment of either **2b** or **3b** with sodium methoxide resulted in clean conversion to the anhydro compound **13** by intramolecular displacement of the mesylate group by the C-1 oxyanion. A similar approach to the anhydro compound **10** required the synthesis of the thioacetate **16** (Scheme 1).

Treatment of **1** with an excess of methanesulfonyl chloride in dichloromethane in the presence of triethylamine [5] was expected to give the dimesylate **14**. However work-up gave instead the chloro compounds **15**, identified from their ^{1}H NMR and mass spectra, and the monomesylate **2b**. Clearly the dimesylate **14** was formed but the more reactive mesylate group at C-1 underwent a displacement reaction by the chloride ions present in the reaction mixture or by water during the aqueous work-up. The mesylation was repeated and potassium thioacetate was then added to the reaction mixture in the expectation that it would react with the dimesylate **14** or the chloro compound **15** to give the required thioacetate **16**. A complex mixture of products

Table 2
^{13}C NMR data

Compd	Chemical shifts (ppm)					
	C-1	C-2, C-3, C-4	C-5	CMe$_2$	CMe_2	Other signals
1	102.9	87.8, 86.85, 81.7	63.7	112.2	26.4, 24.7	
4a	96.2	82.2, 82.1, 80.5	31.2	116.0	26.0, 25.6	194.6 (Me*C*OS); 169.3 (Me*C*OO); 30.6 (*Me*COS); 21.1 (*Me*COO)
4b	102.4	86.7, 85.2, 83.0	32.0	113.2	26.5, 25.1	
6a	75.7	73.7, 70.3, 66.6	26.9	114.3	26.3, 25.5	194.6 (Me*C*OS); 169.3 (Me*C*OO); 30.6 (*Me*COS); 21.3 (*Me*COO)
6b	80.4	77.1, 73.6, 68.3	27.9	111.3	27.8, 25.9	
10	86.0	84.2, 82.4, 81.8	31.7	111.4	26.0, 25.2	
13	99.8	81.3, 79.3, 77.6	63.0	112.2	25.9, 25.3	
16a	85.6	81.6, 81.6, 81.5	69.6	114.9	26.2, 25.0	194.0 (Me*C*OS); 37.6 ($MeSO_2$); 30.9 (*Me*COS)



Scheme 1.

was formed from which the α-thioacetate **16a**, identified by its ^{1}H NMR spectrum, was obtained in low yield. Treatment of **16a** with sodium methoxide gave the required anhydro compound **10**; evidently the intermediate thiol **17a**, or its thiolate anion, underwent mutarotation to the β-form **17b** necessary for the cyclisation. The assignment of the structure of **10** was supported by the close similarity of its ^{1}H NMR spectrum with that of the oxygen analogue **13**.

The sequence of reactions leading to **6b** can also be carried out using the α-compounds **3a** and **4a** so that it is possible to obtain **6b** without separation of anomeric forms of the intermediates **3** and **4**. As **6b** is readily hydrolysed to 5-thio-D-ribose (**11**), this results in a particularly convenient synthesis of this thio sugar.

In an effort to simplify further the synthesis of **6b**, a Mitsunobu reaction with thioacetic acid [6] was carried out on **1** in the hope of obtaining the 5-thioacetate **18** which would yield **6b** on deacetylation. However, the only carbohydrate product identified was the anhydro compound **13**, apparently formed by intramolecular dehydration.

1. Experimental

General methods.—Melting points are uncorrected. Optical rotations were measured at 22 °C. NMR spectra were recorded at 200 MHz (^{1}H) or 50 MHz (^{13}C) for solutions in $CDCl_3$ unless otherwise stated. The petroleum ether (PE) used had boiling range 60–80 °C. Kieselgel 60 was used for TLC (E. Merck 5554) and column chomatography (Prolabo, 200–400 mesh); elution was with EtOAC–PE (1:1 or 1:2). Carbohydrates were visualised on TLC plates with 2% H_2SO_4 in EtOH and heating to 100 °C.

2,3-O-Isopropylidene-5-O-methanesulfonyl-β-D-ribofuranose (**2b**).—Methanesulfonyl chloride (2.14 mL, 3.18 g, 27.0 mmol) was added slowly, with stirring, to a solution of **1** (5.09 g, 26.8 mmol) in pyridine (20 mL) at 0 °C. The mixture was left overnight at 10 °C and then partitioned between H_2O and CH_2Cl_2. The organic extract was washed with 2 M HCl (2 ×), 1 M $KHCO_3$, dried and concentrated to a solid (3.10 g). Recrystallisation from CH_2Cl_2–Et_2O gave the mesylate **2b** (1.05 g, 39 mmol, 15%), mp 112–114 °C, $[\alpha]_D$ − 19° (− 16° final) (*c* 0.25, CH_2Cl_2), (Lit., for L-enantiomer, mp 111–112 °C, $[\alpha]_D$ + 9.2° (MeOH) [7]). Anal. Calcd for $C_9H_{16}O_7S$: C, 40.25; H, 6.01. Found: C, 40.17; H, 5.93.

1-O-Acetyl-2,3-O-isopropylidene-5-O-methanesulfonyl-D-ribofuranose (**3**)

(a) From **2b**. The mesylate **2b** (0.21 g, 0.78 mmol) was left in pyridine (1.0 mL) and Ac_2O (0.5 mL) for 2 days at room temperature (rt). Work-up in the usual way gave the β-acetate **3b** (0.24 g, 0.74 mmol, 95%), mp 91–93 °C (from EtOH), $[\alpha]_D$ − 44° (*c* 1.2, CH_2Cl_2).

Anal. Calcd for $C_{11}H_{18}0_8S$: C, 42.57; H, 5.85. Found: C, 42.22; H, 6.06.

(b) From **1**. The acetal **1** (2.45 g, 12.9 mmol) was treated with methanesulfonyl chloride (1.0 mL, 1.48 g, 12.8 mmol) in pyridine (9 mL) as in the earlier experiment. After 6 h, Ac_2O (2 mL, 2.16 g, 21.2 mmol) was added, and after a further 15 h at rt the reaction mixture was worked-up in the usual way to give a syrup (3.6 g) which, crystallised from EtOH, gave **3b** (1.96 g, 6.3 mmol, 49%), mp 91–93 °C. The mother liquors (1.4 g) were chromatographed on silica and eluted with EtOAc–PE (1:1). Later fractions gave the α-acetate **3a** (0.45 g, 11.5 mmol, 11%), mp 91–93 °C (mixed mp with **3b** 78–82 °C), $[\alpha]_D + 10°$ (*c* 0.6, CH_2Cl_2). Found: C, 42.45; H, 5.85.

1-O-*Acetyl-5*-S-*acetyl-2,3*-O-*isopropylidene*-D-*ribofuranose* (**4**).—A mixture of the mesylate **3b** (0.93 g, 3.0 mmol) and KSAc (0.69 g, 6.0 mmol) in DMF (6 mL) was heated at 70 °C for 2 h. The mixture was cooled and partitioned between Et_2O and 1 M Na_2CO_3. The ether extract was dried and concentrated, crystallisation of the residue from PE gave the thioacetate **4b** (0.61 g, 2.1 mmol, 70%), mp 61–63 °C, $[\alpha]_D - 55.5°$ (*c* 2.0, CH_2Cl_2). Anal. Calcd for $C_{12}H_{18}O_6S$: C, 49.64; H, 6.25. Found: C, 49.52; H, 6.32. Similar treatment of the α-mesylate **3a** gave the α-thioacetate (**4a**), mp 67–68 °C, $[\alpha]_D + 14°$ (*c* 0.5, CH_2Cl_2) Found: C, 49.57; H, 6.09.

2,3-O-*Isopropylidene-5-thio-β*-D-*ribopyranose* (**6b**).—A solution of the thioacetate **4b** ((0.50 g, 1.7 mmol) in MeOH 5 mL) was flushed with N_2 then NaOMe [from Na (75 mg)] in MeOH (3 mL), also N_2 flushed, was added. After 30 min at rt, the mixture was neutralised (CO_2), concentrated to dryness, triturated with EtOAc, and filtered through silica. The filtrate was concentrated to a syrup (0.29 g) which crystallised from ether to give **6b** (0.25 g, 1.2 mmol, 71%), mp 136–138 °C, $[\alpha]_D - 37°$ ($-30°$ final) (*c* 1.44, MeOH). Anal. Calcd for $C_8H_{14}O_4S$: C, 46.58; H, 6.84. Found: C, 46.58, H, 6.81. Similar treatment of the α-thioacetate **4a** also gave **6b**.

Acetylation of 2,3-O-*isopropylidene-5-thio-β*-D-*ribopyranose* (**6b**).—The acetal **6b** (0.11 g, 0.53 mmol) was dissolved in pyridine (1 mL) and Ac_2O (0.75 mL) was added. After 15 h at rt, solvents were evaporated and the residue was dissolved in CH_2Cl_2 and filtered through silica. Concentration and crystallisation of the residue from PE gave 1,4-di-*O*-acetyl-2,3-*O*-isopropylidene-5-thio-β-D-ribopyranose (**8b**). (107 mg, 0.37 mmol, 70%), mp 95–97 °C, $[\alpha]_D - 64.5°$ (*c* 0.8, CH_2Cl_2). Anal. Calcd for $C_{12}H_{18}O_6S$: C, 49.64; H, 6.25. Found: C, 44.43; H, 6.25. The ^1H NMR spectrum of the mother liquor material (46 mg, 0.16 mmol, 30%) showed only peaks for **8b** and **4b** in the ratio 84:16.

Action of 1% sulphuric acid on 2,3-O-*isopropylidene-5-thio-β*-D-*ribopyranose* (**6b**)

(a) In acetone. A solution of **6b** (90 mg, 0.44 mmol) in acetone (2 mL) containing H_2SO_4 (0.02 mL) was left at rt for 2 h. The mixture was neutralised (Na_2CO_3), filtered and concentrated to give a residue (80 mg) which was chromatographed on silica. Elution with EtOAc–PE (1:2) gave a fraction (9 mg) whose ^1H NMR spectrum was identical with that of the diacetal **9** [3].

(b) In toluene–Et_2O. A mixture of **6b** (100 mg, 0.49 mmol), toluene (5 mL), Et_2O (5 mL) and H_2SO_4 (0.1 mL) was stirred at rt. After 15 h the mixture was washed with 1 M Na_2CO_3. The organic extract was dried, filtered, and concentrated to a residue which was dissolved in CH_2Cl_2 and passed through silica to give, after concentration, a residue (10 mg) whose ^1H NMR spectrum indicated the presence of the anhydro compound **10**.

5-Thio-D-*ribose* (**11**).—A solution of **6b** (0.47 g, 2.28 mmol) in H_2O (15 mL) was stirred with Zerolit-225-H^+ (3 mL) for 30 min at rt. The resin was filtered off and the filtrate was concentrated, final traces of H_2O were removed by co-distillation with EtOH. Crystallisation of the product from EtOH gave **11** (0.24 g, 1.44 mmol, 63%) mp and mixed mp 143–145 °C.

1,4-Anhydro-2,3-O-*isopropylidene-α*-D-*ribopyranose* (**13**)

(a) From **3b**. The acetate **3b** (0.22 g, 0.71 mmol) was treated with sodium methoxide (1.30 mmol) [from sodium (30 mg)] in methanol (8 mL). After 1 h at rt the mixture was neutralised (CO_2) and concentrated to dryness. The product was extracted with CH_2Cl_2, and purified by chromatography [EtOAc–PE (1:2)] to give, after sublimation

(100 °C/15 mm Hg), **13** (74 mg, 0.43 mmol, 61%), mp and mixed mp 60–61 °C.

(b) From **2b**. Similar treatment of **2b** (0.35 g, 11.31 mmol) also gave **13** (0.15 g, 0.87 mmol, 66%), mp 60–61 °C.

(c) From **1**. A mixture of **1** (0.12 g, 0.63 mmol) and thioacetic acid (0.07 mL, 75 mg, 0.98 mmol) in THF (2 mL) was added, with stirring at 0 °C, to a suspension formed by mixing Ph_3P (0.25 g, 0.95 mmol) and diisopropyl azodicarboxylate (0.19 mL, 0.20 g, 0.97 mmol) in THF (2 mL) at 0 °C. After 30 min, solvents were removed in vacuo and the residue was chromatographed on silica, eluting with EtOAc–PE (1:6). The ^1H NMR spectrum of the only fraction (40 mg) which contained carbohydrate material, showed it to be composed mainly of anhydro compound **13**.

*Methanesulfonylation of 2,3-*O*-isopropylidene-*D*-ribose* (**1**).—A solution of methanesulfonyl chloride (0.55 mL, 0.81 g, 7.10 mmol) in ice-cold CH_2Cl_2 (5 mL) was slowly added with stirring to a solution of **1** (0.60 g, 3.16 mmol) in CH_2Cl_2 (6 mL) containing Et_3N (1.2 mL, 0.88 g, 7.70 mmol) at 0 °C. After 30 min, Et_3N (1.0 mL) and H_2O (1.0 mL) were added and stirring was continued for 15 min. The mixture was washed with ice-cold 2 M HCl (2 ×) and 1 M $KHCO_3$. After filtering and drying, concentration of the organic extract left a residue which was chromatographed on silica and eluted with EtOAc–PE (1:2) to give, in order of elution: 1-chloro-1-deoxy-2,3-*O*-isopropylidene-5-*O*-methanesulfonyl-β-D-ribofuranose (**15b**) (20 mg, 0.07 mmol, 2%), $[\alpha]_D - 43.5°$ (*c* 1.5, CH_2Cl_2), mass spectrum: *m/z* 271.0012 ($C_9H_{15}O_6ClS$ Calcd 271.0043 for $M^+ - CH_3$); the α-chloro anomer **15a** (40 mg, 0.14 mmol, 4%), $[\alpha]_D + 36°$ (*c* 1.9, CH_2Cl_2), mass spectrum: *m/z* 271.0026; and the mesylate **2b** (0.17 g, 0.63 mmol, 20%), mp and mixed mp 110–112 °C.

*1-*S*-Acetyl-2,3-*O*-isopropylidene-5-*O*-methanesulfonyl-α-*D*-ribofuranose* (**16a**).—As in the previous experiment, the acetal **1** (0.95 g, 5.00 mmol) in CH_2Cl_2 (10 mL), containing Et_3N (2.0 mL, 1.46 g, 14.5 mmol) was treated with methanesulfonyl chloride (0.82 mL, 1.21 g, 10.6 mmol). After 15 min, KSAc (0.60 g, 5.0 mmol) in DMF (10 mL) was added and the CH_2Cl_2 carefully removed in vacuo. More DMF (5 mL) was added and the mixture was left for 24 h at rt. Ether (50 mL) was added and the organic layer was washed successfully with 2 M HCl (2 ×), 1 M Na_2CO_3 (2 ×), dried, filtered, and concentrated to a syrup (0.78 g). This was chromatographed on silica, eluting with EtOAc–PE (1:1) to yield the thioacetate **16b** (0.25 g, 0.79 mmol, 16%), $[\alpha]_D + 20°$ (*c* 1.2, CH_2Cl_2), mass spectrum: *m/z* 311.0230 ($C_{11}H_{18}O_7S$ Calcd 311.0259 for $M^+ - CH_3$).

*1,4-Anhydro-2,3-*O*-isopropylidene-5-thio-α-*D*-ribopyranose* (**10**).—The thioacetate **16a** (0.21 g, 0.64 mmol) was dissolved in methanol (5 mL) flushed with N_2, and sodium methoxide (0.65 mmol) [from sodium(15 mg)] was added under N_2. After 1 h the reaction mixture was worked-up as described for the anhydro compound **13** to give **10** (61 mg, 0.32 mmol, 50%), mp 82–84 °C, $[\alpha]_D - 20°$ (*c* 1.0, CH_2Cl_2). Anal. Calcd for $C_8H_{12}O_3S$: C, 51.04; H, 6.43. Found: C, 50.84; H, 6.42.

Acknowledgements

The authors thank L. Cook for the NMR data, D. Dunbar for the elemental analyses and S. Addison for the MS data.

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