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Note

The cyanation reaction of O-acetylated *S*-glycosyl phosphorothioates

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Abstract

The reaction of α- and β-1-*S*-phosphorothioates of per-O-acetylated monosaccharides (galactose, xylose, glucose) with trimethylsilyl cyanide in the presence of boron trifluoride etherate has been investigated. It has been established that in dichloromethane 1,2-*O*-cyanoethylidene derivatives of aldoses are formed in high yields. When the reaction was carried out in acetonitrile, peracetylated galactosyl cyanide was formed as the major product.

Keywords: *S*-Glycosyl phosphorothioate; Cyanation; 1,2-*O*-[1-(*exo*- and *endo*-Cyano)ethylidene]-glycopyranose; Glycosyl cyanide

1,2-*O*-(1-Cyanoethylidene)glycopyranoses and isomeric glycosyl cyanides (anhydroaldononitriles) are valuable intermediates in organic synthesis. Methods for the synthesis of 1,2-*O*-cyanoethylidene sugar derivatives involve transformations of glycosyl halides in the reaction: (a) with sodium or potassium cyanide and tetra-*n*-butylammonium halide [1–3] and (b) with silver cyanide [4–6]. However, the corresponding glycosyl isocyanides have also been generated in the latter approach [7]. Russian authors, who primarily examined this class of compounds, not only elaborated the appropriate methods of synthesis [1–3,5,6], but applied these reagents to the stereo- and regiospecific synthesis of di- [3,8] and polysaccharides [9–13].

Glycosyl cyanides have been prepared from glycosyl halides and mercuric(II) cyanide [14–18], from acetylated sugars and trimethylsilyl cyanide [19,20], by conversion of anhydro-1-deoxy-1-nitroalditols [21] and by dehydration of anhydroaldonoamides [22]. A novel method for the synthesis of O-benzylated glycosyl cyanides has previously been described [23], which is based on the reaction of corresponding *S*-glycosyl phosphorothioate with trimethylsilyl cyanide in the presence of a Lewis acid.

We report herein an alternative and efficient procedure for the synthesis of per-O-acetylated 1,2-*O*-1-cyanoethylidene glycopyranoses in the reaction of appropriate *S*-glycosyl phosphorothioate with trimethylsilyl cyanide in the presence of a Lewis acid as a catalyst. The cyanation of *S*-galactosyl phosphorothioate with trimethylsilyl cyanide in the presence of a Lewis acid in acetonitrile produced the corresponding glycosyl cyanide in high yield.

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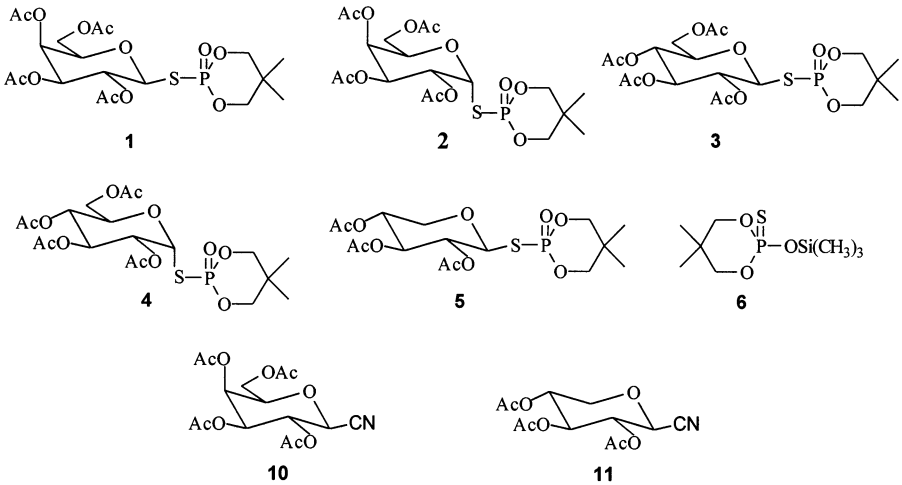
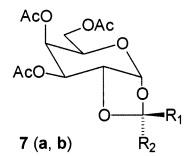
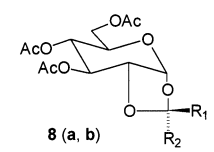
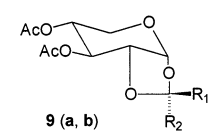


Fig. 1. *S*-Glycosyl phosphorothioates **1**–**5** applied to the cyanation reaction and products formed **6**, **10**, **11**.

The per-O-acetylated *S*-1,2-*trans* and 1,2-*cis*-glycosyl phosphorothioate derivatives of D-galactose **1**, **2**, D-glucose **3**, **4** and D-xylose **5** used in these investigations are listed in the Fig. 1. They have been prepared in the reaction of trialkylammonium salt of *O*,*O*-dialkyl phosphorothioic acid with acetylated glycosyl halides [24] or 1-*O*-acetyl sugars [25]. These glycosyl donors are stable, non-toxic and odorless reagents. In earlier studies, they have been applied to the synthesis of glycosyl disulfides [24] and glycosyl 1-*O*-acyl esters [26].

The cyanation reactions of **1**–**5** (1 molar equivalent) were carried out in aprotic solvents (dichloromethane or acetonitrile) using trimethylsilyl cyanide and the required catalytic amounts of boron trifluoride etherate. The time necessary to complete the reaction was determined by ^{31}P NMR monitoring. The products were isolated and separated by column chromatography. The results of cyanation of **1**–**5** in dichloromethane are summarized in the Table 1. Per-O-acetylated 1,2-*O*-[1-*exo* and *endo*-cyano)ethylidene]-α-D-glycopyranoses **7(a, b)**–**9(a, b)** were obtained, with high preponderance of the *exo* isomers, in yields ranging from 41 to 75%. Low temperature ^{31}P and ^{1}H NMR monitoring (-60 ± 25 °C) revealed that 1,2-*trans*-*S*-glycosyl phosphorothioates were more reactive than the corresponding 1,2-*cis* anomers. The former started to react around -40 °C and the latter above 0 °C. Substrate **1** proved to be the most reactive of D-pyranoses investigated from amongst 1,2-*trans* isomers **1**, **3**, **5** cyanated. The physical properties of **7(a, b)**–**9(a, b)** were in agreement with those reported for 1,2-*O*-[1-(*exo*- and *endo*-cyano)-ethylidene]-α-D-galacto- [1,2], α-D-*gluco*- [1,2] and α-D-xylopyranoses [3]. The forma-tion of a mixture of isomeric nitriles **7(a, b)** and 2,3,4,6 - tetra - *O* - acetyl - β - D - galactopyra-

Table 1
Synthesis of 1,2-*O*-cyanoethylidene sugar derivatives

Reactant	Conditions time (min)	Conditions temp.	Product	*Exo, endo* ratio	Yield (%)
1	5	-10°C		~2:1	63.0
2	40	room temp.		~2:1	41.0
3	15	-10°C		~13:1	58.5
4	30	room temp.		~8:1	53.5
5	10	-10°C		~6:1	75.0

7 (a, b) **8 (a, b)** **9 (a, b)**

a $R_1 = CN$ $R_2 = CH_3$
b $R_1 = CH_3$ $R_2 = CN$

nosyl cyanide (**10**) [2,18,27] (Fig. 1) was observed in 34 and 26% yield, respectively, when at room temperature the time necessary to complete the cyanation in dichloromethane was extended to 25 h. Under these conditions (dichloromethane, 25 h, room temperature) the cyanation of **3** yielded only **8**(**a**, **b**). The cyanation of **1**–**5** was also investigated using acetonitrile as a solvent. Treatment of **1** with trimethylsilyl cyanide in the presence of boron trifluoride etherate in acetonitrile for 4 h at room temperature generated the cyanide **10** and **7**(**a**, **b**) in 60 and 6% yield, respectively. There was a marked contrast in the course of the cyanation between the gluco and galacto series. Diastereomeric **8**(**a**, **b**) were the major products isolated in 58% yield when acetonitrile was applied in cyanation of **3** or **4** (72 h at room temperature). The reaction of **5** with trimethylsilyl cyanide in the presence of boron trifluoride etherate in acetonitrile at room temperature for 25 h yielded the mixture of isomeric nitriles: 2,3,4-tri-*O*-acetyl-β-D-xylopyranosyl cyanide (**11**) [16,28] (36% yield) (Fig. 1) and **9**(**a**, **b**) (13.3% yield). The quantitative formation of *O*-trimethylsilyl phosphorothioate **6** (δ ^{31}P NMR CD_2Cl_2: ~50.5 ppm) (Fig. 1) was observed in all the examined reactions (according to the ^{31}P NMR data).

In summary, depending on the stereochemistry of *S*-glycosyl phosphorothioates employed, as well as the reaction conditions either cyanoethylidene derivatives of sugars or glycosyl cyanides are formed. In comparison with hitherto described methods, the reaction conditions described herein are mild, the reaction time is shortened considerably and the temperature decreased. Thus, this cyanation reaction provides a useful approach to the cyanated sugars.

1. Experimental

General methods.—Melting points were determined with Boetius PHMK 05 apparatus and are uncorrected. Optical rotations were determined with the Perkin–Elmer 241 MC apparatus. ^{1}H, ^{13}C and ^{31}P NMR spectra were measured in $CDCl_3$ solutions on a Bruker DPX spectrometer operating at 250.13, 62.9 and 101.25 MHz, respectively. Solvents were dried and distilled prior to use. Trimethylsilyl cyanide 98% and $BF_3{\cdot}Et_2O$ were purchased from Aldrich

Starting materials.—O-Acetylated 1-*S*-phosphorothioates **1**, **3**, **5** were prepared by the procedure described in Ref. [24] and substrates **2** and **4** by the procedure described in Ref. [25]. Column chromatography was performed on Silica Gel 60 (E. Merck) (70–230 mesh, ASTM) with 3:1 (v/v) toluene–EtOAc (Solvent A), benzene (Et_2O (Solvent B). Thin-layer chromatography (TLC) was conducted on layers (0.2 mm) of Silica Gel 60 F_{254} precoated on plastic sheets (E. Merck, Cat. No 5748) with 3:1 (v/v) Et_2O–petroleum ether. Components on TLC plates were detected by spraying 10% (v/v) H_2SO_4 in CH_3OH, and heating for several minutes at 140 °C.

The reaction of S-*glycosyl phosphorothioates with Me_3SiCN in the presence of $BF_3{\cdot}Et_2O$ (^{31}P and ^{1}H NMR low temperature analysis in CD_2Cl_2).*—To the cooled solution (~ −70 °C) of **1**–**4** (0.051 g, 0.1 mmol) or **5** (0.044 g, 0.1 mmol) in 0.5 mL of CD_2Cl_2 placed in 5 mm NMR tube, Me_3SiCN (0.04 mL, 0.3 mmol) and one drop of $BF_3{\cdot}Et_2O$ were added by syringe and the reaction course was followed by ^{31}P and ^{1}H NMR spectroscopy at time intervals and temperatures ranging from −60 to 25 °C.

*General procedure 1. 1,2-*O*-[(1-(*exo*)- and (*endo*)-cyano)ethylidene]-aldopyranoses* [**7**(**a**, **b**), **8**(**a**, **b**), **9**(**a**, **b**)].—To the cooled solution (−10 °C) of thiophosphate **1** or **3** (0.512 g, 1 mmol) or **5** (0.44 g, 1 mmol) in dry CH_2Cl_2 (5 mL), Me_3SiCN (0.297 g, 0.4 mL, 3 mmol), then 4 Å MS and finally $BF_3{\cdot}Et_2O$ (0.015 mL, 0.12 mmol) was added. The reaction of substrate **2** or **4**: to the solution of **2** or **4** (0.512 g, 1 mmol) in dry CH_2Cl_2 (5 mL), Me_3SiCN (0.4 mL, 3 mmol) and 4 Å MS and finally $BF_3{\cdot}Et_2O$ (0.015 mL, 0.12 mmol) were added. The resulting solution was stirred for the time and temperature indicated in Table 1. The solution was then filtered (Celite) and washed with CH_2Cl_2 (~30 mL). The organic layer was washed with satd $NaHCO_3$ (2 × 15 mL), water (15 mL), and dried ($MgSO_4$). The solvent was evaporated in vacuo and the syrupy residue was subjected to column chromatography.

3,4,6-Tri-O-*acetyl-1,2*-O-*[1-(*exo- *and* endo-*cyano)ethylidene]-α*-D-*galactopyranose* [**7**(**a**, **b**)].—From substrate **1**. ^{13}C NMR spectra of the crude reaction mixture [**7**(**a**, **b**) and **6′** (from **6**)]: δ 169.9, 169.4, 169.3, 169.1 (*C*=O), 117.2 (CN, endo), 116.4 (CN, exo), 98.6 (*C*CN, exo), 98.2 (*C*CN, endo), 96.8 (C1), 72.3, 70.7, 69.4, 65.0, 61.0 (C2-C6, exo), 75.6, 69.6, 69.0, 65.6, 60.7 (C2–C6, endo), 26.5 (C(CN)*C*H$_3$, endo), 25.5 (C(CN)*C*H$_3$, exo), 20.9, 20.2, 20.0 (CO*C*H$_3$, exo, endo); **6′** (76.3, 76.2, 53.3, 31.9).

Isolation products by column chromatography, (solvent B). Total yield **7**(**a**, **b**) (0.225 g, 63%).

7a (0.1 g, 28%): colourless syrup; $[\alpha]_D^{20}$ + 91.4° (*c* 1.34, $CHCl_3$), lit. + 92.7° (*c* 1.10, $CHCl_3$) [1,2]; ^{1}H NMR (selected): δ 5.87 (d, $J_{1,2}$ 5.0 Hz, 1 H, H-1), 2.08, 2.07 (3s, 3 × OAc), 1.86 [s, CH_3(CCN)] in agreement with the lit. [1] ^{1}H NMR spectrum.

7(**a**, **b**) (0.125 g, 35%). **7b**, ^{1}H NMR (selected): δ 5.75 (d, $J_{1,2}$ 4.5 Hz, 1 H, H-1), 2.03, 2.06 (3s, 3 × OAc), 1.79 [s, CH_3(CCN)] in agreement with the lit. [1] ^{1}H NMR spectrum.

From substrate **2**. Isolation of products by column chromatography (solvent A), **7**(**a**, **b**) (0.146 g, 40.9%).

3,4,6-Tri-O-*acetyl-1,2*-O-*[1-(*exo- *and* endo-*cyano)ethylidene]-α*-D-*glucopyranose* **8**(**a**, **b**).—From substrate **3**. ^{13}C NMR of the crude reaction mixture [**8**(**a**, **b**) and **6′** (from **6**)]: δ 170.6, 169.6, 169.1 (*C*=O), 116.6 (CN), 100.1 (*C*CN, endo), 98.9 (*C*CN, exo), 98.2 (C1, endo), 97.5 (C1, exo), 74.2, 70.0, 69.3, 67.4, 62.2 (C2–C6), 24.5 (C(CN)*C*H$_3$), 21.5, 20.8 (CO*C*H$_3$); **6′** (76.9, 76.7, 53.8, 32.5).

Isolation of products by column chromatography (solvent A). Total yield **8**(**a**, **b**) (0.209 g, 58.5%).

8a (0.189 g, 52.9%): colourless crystals, mp 76–77 °C, lit. 77–78 °C [1,2,4]; $[\alpha]_D^{18}$ + 14.3° (*c* 1.86, $CHCl_3$), lit. + 13.6° (*c* 2.90, $CHCl_3$) [1,2], lit. + 13.8° (*c* 1.02, $CHCl_3$) [4]; ^{1}H NMR (selected): δ 5.81 (d, $J_{1,2}$ 5.1 Hz, 1 H, H-1), 2.14, 2.10 (2s, 9 H, 3 × OAc). 1.92 [s, CH_3(CCN)] in agreement with lit. [1] ^{1}H NMR spectrum.

8(**a**, **b**) (0.02 g, 5.6%). **8b**: ^{1}H NMR (selected): δ 5.72 (d, $J_{1,2}$ 5.0 Hz, 1 H, H-1), 2.04, 2.03, 2.02 (3s, 9 H, 3 × OAc), 1.69 [s, CH_3(CCN)] (in agreement with lit. [1] ^{1}H NMR spectrum).

From substrate **4**. Isolation of products by column chromatography (solvent A), **8**(**a**, **b**), (0.19 g, 53.2%).

3,4-Di-O-*acetyl-1,2*-O-*[(1*-exo *and* endo-*cyano)ethylidene]-α*-D-*xylopyranose* **9**(**a**, **b**).—From substrate **5**. ^{13}C NMR of the crude reaction mixture [**9**(**a**, **b**) and **6′** (from **6**)]: δ 170.4, 169.8, 169.2, 168.9 (*C*=O), 117.7 (CN, endo), 116.9 (CN, exo), 100.0 (*C*CN, endo), 99.3 (*C*CN, exo), 97.4 (C1, endo), 96.9 (C1, exo), 76.0 (C2, endo), 74.9 (C2, exo), 68.3 (C3, exo), 67.2 (C4, exo), 66.9 (C3, endo), 66.0 (C4, endo), 62.0 (C5, endo), 59.7 (C5, exo), 26.9 (C(CN)*C*H$_3$, endo), 25.1 (C(CN)*C*H$_3$, exo), 21.6, 21.5, 20.9 (CO*C*H$_3$) (in agreement with lit. [3] ^{13}C NMR spectrum); **6′** (76.9, 76.8, 53.8, 32.5).

Isolation of products by column chromatography (solvent A). Total yield **9**(**a**, **b**) (0.214 g, 75%): **9a** (0.185 g, 65%), **9**(**a**, **b**) (0.02 g, 7%), **9b** (0.008 g, 3%).

9a: colourless syrup, $[\alpha]_D^{18}$ − 22.1° (*c* 2.4, $CHCl_3$), lit. − 23.2° (*c* 1.0, $CHCl_3$) [3]; ^{1}H NMR: δ 5.66 (d, $J_{1,2}$ 4.5 Hz, 1 H, H-1), 5.26 (t, $J_{2,3}$ 2.75, $J_{3,4}$ 3.0 Hz, 1 H, H-3), 4.89 (m, 1 H, H-4), 4.30 (ddd, $J_{2,3}$ 2.75, $J_{1,2}$ 4.5 Hz, 1 H, H-2), 3.97 (dd, $J_{4,5'}$ 6.0, $J_{5,5'}$ 12.0 Hz, 1 H, H-5′), 3.59 (dd, $J_{4,5}$ 8.0, $J_{5,5'}$ = 12.0 Hz, 1 H, H-5), 2.13, 2.09 (2s, 2 × OAc), 1.91 [s, CH_3(CCN)] (in agreement with lit. [3] ^{1}H NMR spectrum).

9b: colourless syrup, $[\alpha]_D^{18}$ + 30.9° (*c* 0.93, $CHCl_3$), lit. + 30.4° (*c* 1.6, $CHCl_3$) [3]. ^{1}H NMR: δ 5.52 (d, $J_{1,2}$ 3.25 Hz, 1 H, H-1), 5.38 (m, 1 H, H-3), 4.72 (m, 1 H, H-4), 4.10–4.01 (m, 2 H, H-2, H-5′), 3.88 (dd, $J_{4,5}$ 2.5 Hz, $J_{5,5'}$ = 13.0 Hz, 1 H, H-5), 2.16, 2.13 (2s, 2 × OAc), 1.79 [s, CH_3(CCN)] (in agreement with lit. [3] ^{1}H NMR spectrum).

General procedure 2. Per-O-*acetylaldopyranosyl cyanides* **10** *and* **11**.—To a stirred solution of glycosyl phosphorothioate **1** (0.512 g, 1 mmol) or **5** (0.44 g, 1 mmol) in CH_3CN (5 mL), 4 Å MS, Me_3SiCN (0.297 g, 0.4 mL, 3 mmol) and $BF_3{\cdot}Et_2O$ (0.015 mL, 0.12 mmol) were added. The mixture was stirred at rt for time indicated. The reaction mixture was then

diluted with EtOAc (50 mL) and filtered through Celite 535. The filtrate was washed with satd $NaHCO_3$ (3×40 mL), water (20 mL), dried ($MgSO_4$) and evaporated in vacuo. The syrupy or semi-crystalline mixture was subjected to column chromatography (solvent A or solvent B).

1-Deoxy-2,3,4,6-tetra-O-*acetyl-β*-D-*galacto-pyranosyl cyanide (3,4,5,7-tetra*-O-*acetyl-2,6-anhydro*-D-*glycero*-L-manno-*heptononitrile)* (**10**).—From substrate **1**, time 4 h, rt. Isolation of products by column chromatography (solvent A) gave **7**(**a**, **b**) (0.022 g, 6.2%) and **10** (0.213 g, 59.7%).

10: colourless crystals, mp 169–170 °C, lit. 169–170 °C [18]; $[\alpha]_D^{18}$ +34.1° (*c* 1.33, $CHCl_3$), lit. +35.7° (*c* 3.74, $CHCl_3$) [18] lit. +37.8° (*c* 0.98, $CHCl_3$) [27]; ^{1}H NMR: δ 5.54 (t, $J_{2,3}$ 10.0 Hz, 1 H, H-2), 5.44 (dd, $J_{4,5}$ 1.25, $J_{3,4}$ 3.5 Hz, 1 H, H-4), 5.00 (dd, $J_{2,3}$ 10.0, $J_{3,4}$ 3.5 Hz, 1 H, H-3), 4.28 (d, $J_{1,2}$ 10.2 Hz, 1 H, H-1), 4.12 (d, 2 H, H-6, 6′), 3.94 (dt, $J_{5,6} = J_{5,6'}$ 6.5 Hz, 1 H, H-5), 2.19, 2.12, 2.06, 2.00 (4s, 12 H, 4(OAc) (in agreement with the lit. [18] ^{1}H NMR spectrum). ^{13}C NMR: δ 170.2, 169.8, 169.7, 168.7 (*C*=O), 114.3 (CN), 75.2 (C5), 70.6 (C3), 66.7 (C4), 66.5 (C2), 65.9 (C1), 61.1 (C6), 20.4-20.3 (CO*C*H$_3$) in agreement with the lit. [18] ^{13}C NMR spectrum.

From substrate **2**. Time 25 h, rt. Isolation of products by column chromatography (solvent A) gave **7**(**a**, **b**) (0.01 g, 2.8%) and **10** (0.153 g, 42.8%).

1-Deoxy-2,3,4-tri-O-*acetyl-β*-D-*xylopyranosyl cyanide (3,4,5-tri*-O-*acetyl-2,6-anhydro*-D-*gulononitrile* (**11**).—From substrate **5**, time 25 h, rt. Isolation of products by column chromatography (solvent A). Total yield **9**(**a**, **b**) and **11** (0.165 g, 57.9%): **9**(**a**, **b**) (0.038 g, 13.3%), **9**(**a**, **b**) and **11** (0.025 g, 8.8%), **11** (0.102 g, 35.8%).

11: colourless crystals, mp 130–131 °C, lit. 132–133 °C [28]; $[\alpha]_D^{18}$ −57.1° (*c* 1.0, $CHCl_3$), lit. −57.9° (*c* 0.8, $CHCl_3$) [28]; ^{1}H NMR: δ 5.2–5.0 (m, 2 H, H-3, H-4), 4.92 (m, $J_{5,6}$ 3.9, $J_{5,6'}$ 6.7 Hz, 1 H, H-5), 4.50 (d, $J_{1,2}$ 6.7 Hz, 1 H, H-2), 4.24 (dd, $J_{5,6}$ 3.9, $J_{6,6'}$ 12.5 Hz, 1 H, H-6), 3.60 (dd, $J_{5,6}$ 6.65, $J_{6,6'}$ 12.5 Hz, 1 H, H-6′), 2.12, 2.11, 2.08 (3s, $3 \times$ OAc) (in agreement with lit. [28] ^{1}H NMR spectrum).

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