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Synthesis of 4-thio-D-glucopyranose and interconversion to 4-thio-D-glucofuranose

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A B S T R A C T

Sulfur containing glycosides offer an exciting prospect for inclusion within noncanonical glycan sequences, particularly as enabling probes for chemical glycobiology and for carbohydrate-based therapeutic development. In this context, we required access to 4-thio-D-glucopyranose and sought its chemical synthesis. Unable to isolate this material in homogenous form, we observed instead a thermodynamic preference for interconversion of the pyranose to 4-thio-D-glucofuranose. Accordingly, we present an improved method to access both bis(4-thio-D-glucopyranoside)-4,4′-disulfide and 4-thio-D-glucofuranose from a single precursor, demonstrating that the latter compound can be accessed from the former using a dithiothreitol controlled reduction of the disulfide. The dithiothreitol-mediated interconversion between pyranose (monomer and disulfide) and furanose forms for this thiosugar is monitored by ^{1}H NMR spectroscopy over a 24-h period. Access to these materials will support accessing sulfur-containing mimetics of glucose and derivatives therefrom, such as sugar nucleotides.

1. Introduction

The requirement for sulfur containing glycosides, wherein the interglycosidic or ring chalcogen is switched from oxygen to sulfur, has increased in recent years. This is due to the ability of *S*-glycosides to act as chemical tools or probes to study carbohydrate active enzymes, alongside their capability as mimetics to explore regulatory processes governed by carbohydrate-protein interactions [1]. Due to the resulting close structural similarity to natural *O*-glycosides and because of the stability of thioglycosidic linkages to enzymatic cleavage, *S*-glycosides have also been considered as promising candidates for the preparation of carbohydrate-based therapeutics [2–4]. The choice of sulfur (over carbon) to replace oxygen gives a similar conformational preference about the (now longer) thioglycosidic and aglyconic bonds, both when in solution and when complexed with a protein [5]. This, combined with sulfur's lower affinity for protons, confers a reduced susceptibility to hydrolysis for *S*-glycosides [6].

Synthetic chemistry has remained a cornerstone capability to deliver *S*-glycosides, with important achievements including thio-oligosaccharide vaccine candidates [7], alongside wide-ranging examples of thiodisaccharides [8], as glycomimetics for multivalent constructs [9,10], and application towards conformational studies [11,12]. Indeed, within the capabilities for synthesis of defined heparan sulfate (HS) fragments [13–17], a recent example of *O- vs. S*-glycosidic linkage change within an HS disaccharide highlighted key conformational differences, granting access to previously untapped chemical space [18]. Additionally, there are emergent examples of enzymes capable of synthesising *S*-glycoconjugates [19–21]. As part of a wider program seeking to establish chemical access to modified hexoses for chemoenzymatic synthesis [22–24], we were interested to synthesise 4-thio-D-glucopyranose **5** (Fig. 1). Specifically, we sought thiosugar **5** as an intended precursor for subsequent enzymatic transformation into an artificial sugar nucleotide, 4-thio-UDP-GlcA; a related 4-thio UDP-GlcNAc donor has recently been exploited for incorporation into heparosan polysaccharide chains [25].

Reported herein is our attempted synthesis of 4-thio-D-glucopyranose **5** and a study of an observed interconversion to its furanose form **6** *via* a dithiothreitol (DTT) controlled reduction of the pyranose disulfide **7**.

2. Results and discussion

Synthetic access to 4-thiohexofuranose derivatives possessing D/L-*ribo* [26], D-*xylo* [27], D-*ido* [28], 6-deoxy-D-*gluco* [29], and D-*galacto* [30] configurations have been reported using acetolytic ring contraction of the corresponding methylpyranosides (Fig. 2, red highlight). In contrast, there are examples for protected D-*manno* [31] and D-*gluco* [32] configured systems where acetolysis delivers peracetylated pyranosides as the major products (Fig. 2, blue highlight). In 1973, Vegh and

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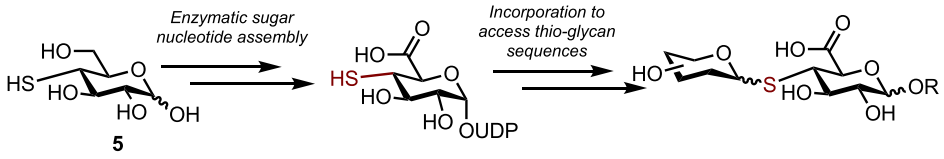


Fig. 1. Intended utilisation of 4-thio-D-glucopyranose **5** as a substrate for trialling enzymatic conversion to 4-thio-UDP-glucuronic acid.

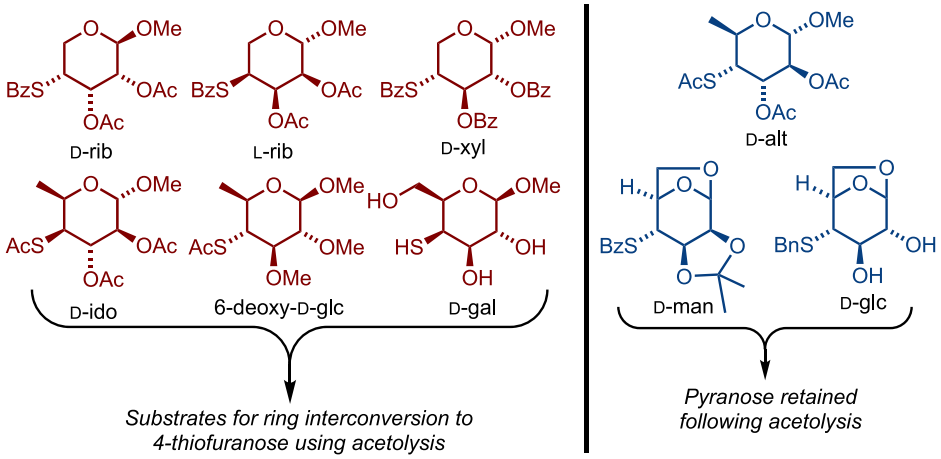
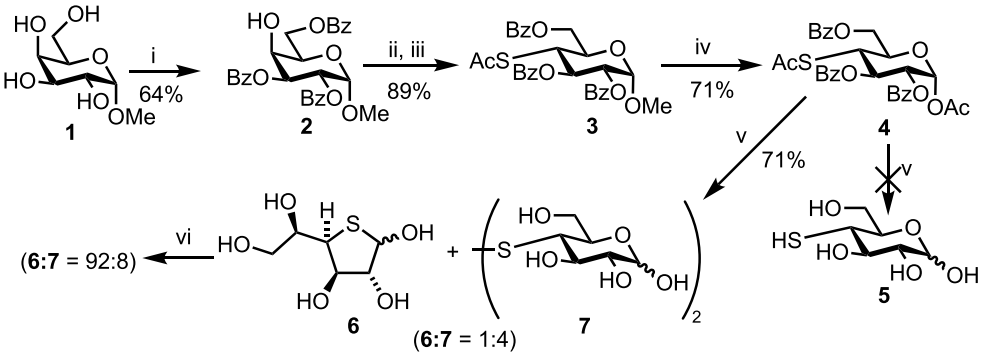


Fig. 2. Prior examples of substrates undergoing 4-thiohexopyranose ring contraction using acetolysis (in red) alongside examples of 4-thiohexopyranoses where ring contraction was not observed during acetolysis (in blue).



Scheme 1. Attempted synthesis of 4-thio-D-glucopyranose **5**. Reagents and conditions; i. BzCl, DCM, pyridine, −40 °C; ii. Tf_2O, DCM, 0 °C to RT; iii. KSAc, pyridine; iv. Ac_2O/AcOH/H_2SO_4 (35/15/1 *v/v/v*), RT; v. NaOMe, MeOH, RT; vi. DTT, H_2O.

Hardegger reported pyranose to furanose ring interconversion for 4-thioglucose during acidic hydrolysis or acetylation of 1,6-anhydro-4-thio-β-D-glucopyranose *via* observed complex product distributions [32].

Given these findings, and keen to avoid ring contraction to the furanose form, we approached the synthesis of **5** from fully protected glucopyranoside precursor **4**, as shown in Scheme 1.

Accordingly, regioselective benzoylation of galactose methyl glycoside **1** at −40 °C successfully delivered glycoside **2**, bearing a free hydroxyl at C4 [33]. A two-step conversion next installed the required C4 thioacetate. Treatment of **2** with Tf_2O at 0 °C furnished the triflate. Following aqueous workup, ^{19}F NMR analysis of a crude aliquot revealed a chemical shift at δ 74.2 ppm, confirming triflate formation. This crude material was treated with potassium thioacetate in pyridine to deliver thioacetate **3** in 89% yield, over two steps. Inversion of configuration at C4 was confirmed by ^3J coupling ($^3J_{H4\text{-}H5}$ = 11.4 Hz). Additionally, the SAc chemical shift (δ_H 2.21 ppm) further supported the formation of **3**.

Acetolysis of thioacetate **3** using a combination of Ac_2O/AcOH/H_2SO_4 (35/15/1, *v/v/v*) successfully converted the anomeric methyl glycoside to the corresponding α-acetate **4**. Gratifyingly, no ring contraction was observed during this reaction, supporting the observations of Hardegger and Bahl for fully protected D-glucose and D-mannose systems. [31,32] Finally, a global de-esterification of **4** using NaOMe in MeOH was completed. TLC analysis after 24 h revealed complete conversion of the starting material to a single spot [R_f = 0.54 (DCM/MeOH, 7/3]. The reaction mixture was neutralised with Amberlite IR120 (H^+ form) and the crude residue purified by column chromatography. ^1H NMR analysis of the isolated material (proposed to be **5**) revealed four distinct anomeric environments. Two of these matched with expected coupling constants and chemical shifts for a 4-thio pyranose [δ_H ppm, 5.19 (d, $^{\alpha}J_{H1\text{-}H2}$ = 3.5 Hz), 4.52 (d, $^{\beta}J_{H1\text{-}H2}$ = 8.0 Hz), δ_C 92.1 C1α, 95.5 C1β ppm, see supporting information S3]. Coupling constants and chemical shifts for the remaining anomeric environments didn't match those typically observed for pyranose systems [δ ppm, 5.64 (d, $^{\alpha}J_{H1\text{-}H2}$ = 4.1 Hz), 5.33 (app t, $^{\beta}J_{H1\text{-}H2}$ = <1.0 Hz), δ_C 80.1, 86.1 ppm, C1] [34,35]. We considered from these observations that ring interconversion to a thermodynamically favoured furanose form had occurred [36,37], and

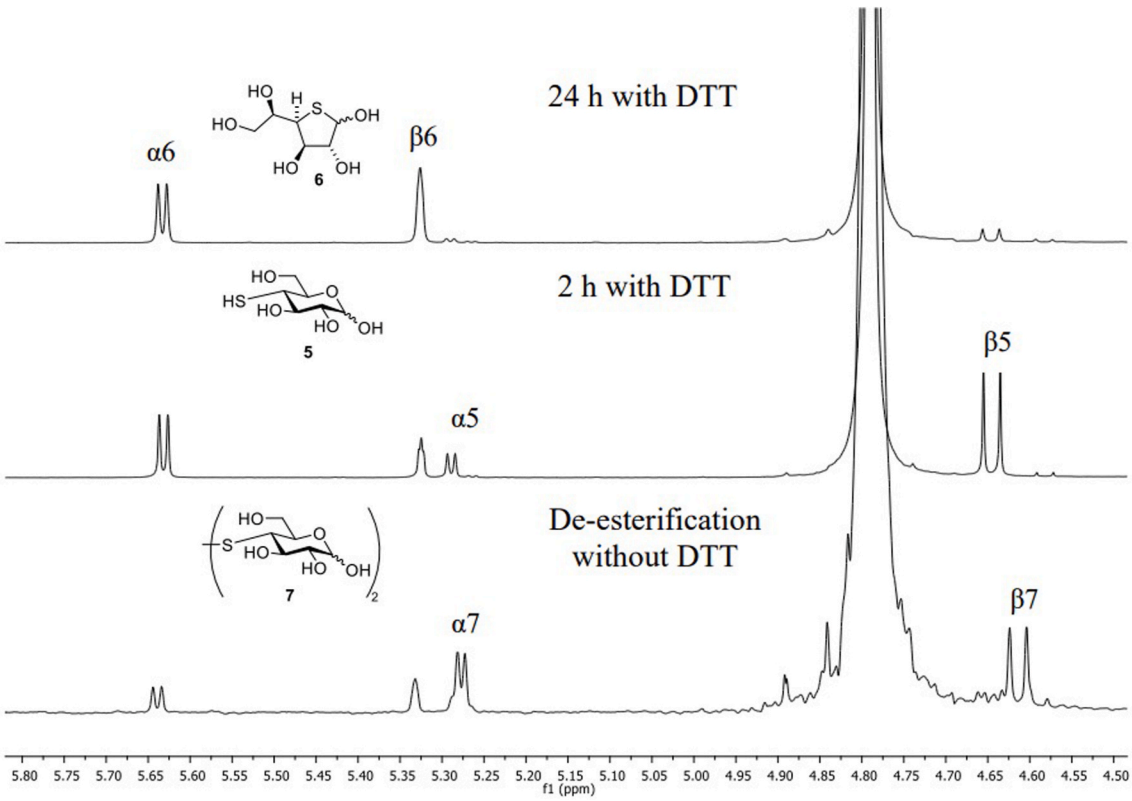


Fig. 3. Stacked ^{1}H NMR spectra (400 MHz, D_2O) comparing de-esterification of **4** with and without DTT. Top spectrum: Crude NMR analysis after stirring for 24 h with DTT. Middle spectrum: Crude NMR analysis of a reaction aliquot after 2 h with DTT. Bottom spectrum: Crude NMR analysis of furanose **6** and disulfide **7** mixture from first pass de-esterification without DTT.

thus assigned the product mixture as containing both 4-thiofuranose and pyranose, with an isolated yield of 71% and as an inseparable mixture (1/4, furanose/pyranose).

In a subsequent experiment, the inseparable mixture was treated with DTT (1.5 equiv.) and stirred for 24 h (Scheme 1). Analysis of the resulting ^{1}H NMR spectrum revealed the preferred tautomer, furanose **6**, to be the major product and indicated the prior assigned pyranose was likely the disulfide form **7**. In addition, a cyclic disulfide (due to DTT oxidation) was observed between δ 2.9–3.2 ppm (see supporting information S3), further supporting reduction of 4-thiopyranose disulfide **7** and generation of **6** [38]. The observed interconversion of **5** to **6** (from **7**) aligns with results observed by Bahl during their global de-esterification of 1,2,3,6-tetra-*O*-acetyl-4-*S*-benzoyl-4-thio-D-mannopyranose using NaOMe in MeOH [31].

To attempt suppression of 4-thiopyranose disulfide formation, a repeat experiment converting **4** but incorporating DTT (1.1 equiv. relative to **4**), was completed (see supporting information S4). After 2 h, TLC analysis revealed a complete conversion of **4** to a single spot and a reaction aliquot was taken for ^{1}H NMR analysis. This revealed four distinct anomeric environments that were assigned to the pyranose and furanose 4-thiosugars, **5** and **6**, with **5** distinguished from **6** again through larger vicinal ^{3}J coupling constants (e.g., $^{\beta-5}J_{H2\text{-}H3} = 9.2$ Hz *vs.* $^{\beta-6}J_{H2\text{-}H3} = 2.0$ Hz). Comparison of the anomeric chemical shifts for **5** (Fig. 2 middle spectrum), against **7** enabled their distinction (Fig. 3 bottom spectrum).

^{1}H NMR analysis after 2 h revealed the **5**/**6**/**7** ratio to be 61/37/2 (Fig. 3, middle spectrum). After 24 h a final aliquot was analysed by ^{1}H NMR and a ratio for **5**/**6**/**7** of 13/84/3 was observed (Fig. 3, top spectrum) indicating furanose **6** to be the thermodynamically favoured ring form for 4-thio-D-glucose. On scale-up and upon reaction completion the

Table 2
^{13}C NMR chemical shifts (δ, ppm) for **5** and **6** in D_2O at 101 MHz.[a]

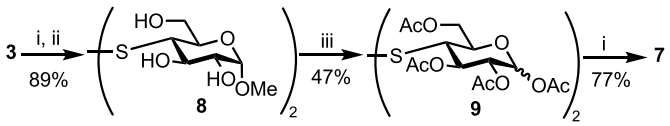
	C_1	C_2	C_3	C_4	C_5	C_6
α-5	92.2					
β-5	95.7	75.3	76.8	41.7	77.8	61.6
β-6	86.1	82.2	76.8	52.0	71.4	65.2
α-6	80.1	77.3	75.9	49.1	71.2	64.9

[a] **5** ($\alpha/\beta = 27/73$) could not be separated from **6** so ^{13}C NMR chemical shifts (δ, ppm) were extracted from the crude HSQC NMR of a mixture containing **5**/**6**/**7** = 61/37/2.

Table 1
^{1}H NMR chemical shifts (δ, ppm) and $^{3}J_{H/H}$ coupling constants (Hz) for **5** and **6** in D_2O at 400 MHz.[a]

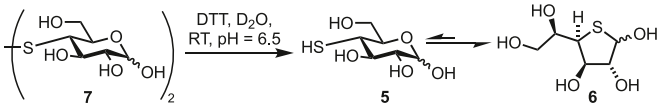
	H_1	H_2	H_3	H_4	H_5	H_6	$J_{1\text{-}2}$	$J_{2\text{-}3}$	$J_{3\text{-}4}$	$J_{4\text{-}5}$	$J_{5\text{-}6a}$	$J_{5\text{-}6b}$	$J_{6a\text{-}6b}$
α-5	5.29						3.6						
β-5	4.65	3.23	3.41	2.72	3.53	3.99	8.0	9.2	10.3		2.2		12.5
β-6	5.33	4.38	4.42	3.66	4.09	3.74/57	>1.0	2.0	4.2	9.6	2.8	6.0	12.1
α-6	5.64	4.21	4.45	3.79	3.86	3.69/54	4.1	4.0	4.3	9.2	2.7	5.9	12.2

[a] **5** ($\alpha/\beta = 27/73$) could not be separated from **6**.^{1}H NMR chemical shifts (δ, ppm) and ^{3}J coupling constants (Hz) are derived from crude ^{1}H, selective COSY and coupled HSQC NMR analysis of a mixture containing **5**/**6**/**7** (61/37/2). Where signals overlapped, ^{3}J coupling constants could not be determined and chemical shifts were extracted from selective COSY and coupled HSQC (see supporting information, S6).



Scheme 2. Alternative synthesis of bis(4-thio-D-glucopyranose)-4,4′-disulfide **7**. Reagents and conditions; i. NaOH, MeOH, H_2O, RT; ii, NEt_3, MeCN, sonication, RT; iii. Ac_2O, AcOH, H_2SO_4, RT.

Table 3
Ratio of pyranose/furanose components during reduction of disulfide **7** over 24 h.[a]



Entry	Time[b]	Ratio 7:5:6[c]
1	5 min	78:22:0
2	1 h	56:40:4
3	3 h	43:44:13
4	6 h	32:42:26
5	9 h	27:40:33
6	12 h	17:33:50
7	15 h	14:31:55
8	18 h	6:29:65
9	21 h	0:17:83
10	24 h	0:0:100

[a] NMR scale reaction for the reduction of disulfide **7**; reaction conditions: DTT (1.1 equiv.), D_2O (90 mM).
[b] Time allowed to pass until ^1H NMR experiment repeated.
[c] Ratio determined by combined anomeric integration values for each compound, see supporting information S5 for full details.

mixture was concentrated *in vacuo* and the residue was washed with diethyl ether. Lyophilisation of the resulting residue furnished **6** in 95% yield. Tabulated (see Tables 1 and 2) are the ^1H and ^{13}C NMR characterisation data for **6** (alongside key chemical shifts for **5** from reaction aliquot analysis).

As our original synthetic goal was to access **5**, we altered our approach, proposing instead to synthesise disulfide **9** prior to anomeric deprotection, thus preventing interconversion to the furanose form. Starting from methyl glycoside **3**, saponification followed by 4-sulfhydryl oxidation delivered **8** in 86% yield, over two steps (Scheme 2). The initial approach for thiol oxidation saw the reaction stirred for 24 h in air. A crude aliquot was then subjected to ^1H NMR analysis, but revealed only minimal conversion to **8** (monomer/dimer, 7/3), with the anomeric chemical shifts clearly distinguishable for each [δ_H ppm, 4.76 (d, $^{monomer}J_{H1\text{-}H2} = 3.6$ Hz), 4.74 (d, $^{dimer}J_{H1\text{-}H2} = 3.6$ Hz)]. Consulting the literature, we noted that solvation effects from MeCN in combination with NEt_3 had been shown to encourage disulfide formation by increasing the rate of the oxidation [39,40]. Gratifyingly, addition of NEt_3 in MeCN followed by sonicating for 1.5 h revealed full conversion to **8** with HRMS adding further confirmation of the disulfide form [41]. Acetolysis was next completed to give anomeric mixture **9** (α/β, 83/17) in a modest yield of 47%, noting the reaction failed to go to completion. A final saponification successfully delivered the target disulfide **7**, with observed coupling constants confirming the pyranose form [δ ppm 5.28 (d, $^{\alpha}J_{H1\text{-}H2} = 3.6$ Hz), 4.61 (d, $^{\beta}J_{H1\text{-}H2} = 8.0$ Hz)] [35].

With homogenous disulfide **7** in hand, we returned to the interconversion between **5** and **6**, performing an NMR scale reaction to monitor the interconversion over a 24-h period. The results from this are highlighted in Table 3 and Fig. 4. Formation of the free thiol **5** was observed immediately after DTT addition (Table 3, entry 1). After 1 h, trace amounts of the furanose **6** appeared (Table 3 entry 2). Compound **5** appeared to be the major species between 3 and 9 h (Table 3 entries 3–5), but from this point furanose **6** became dominant (Table 3 entry

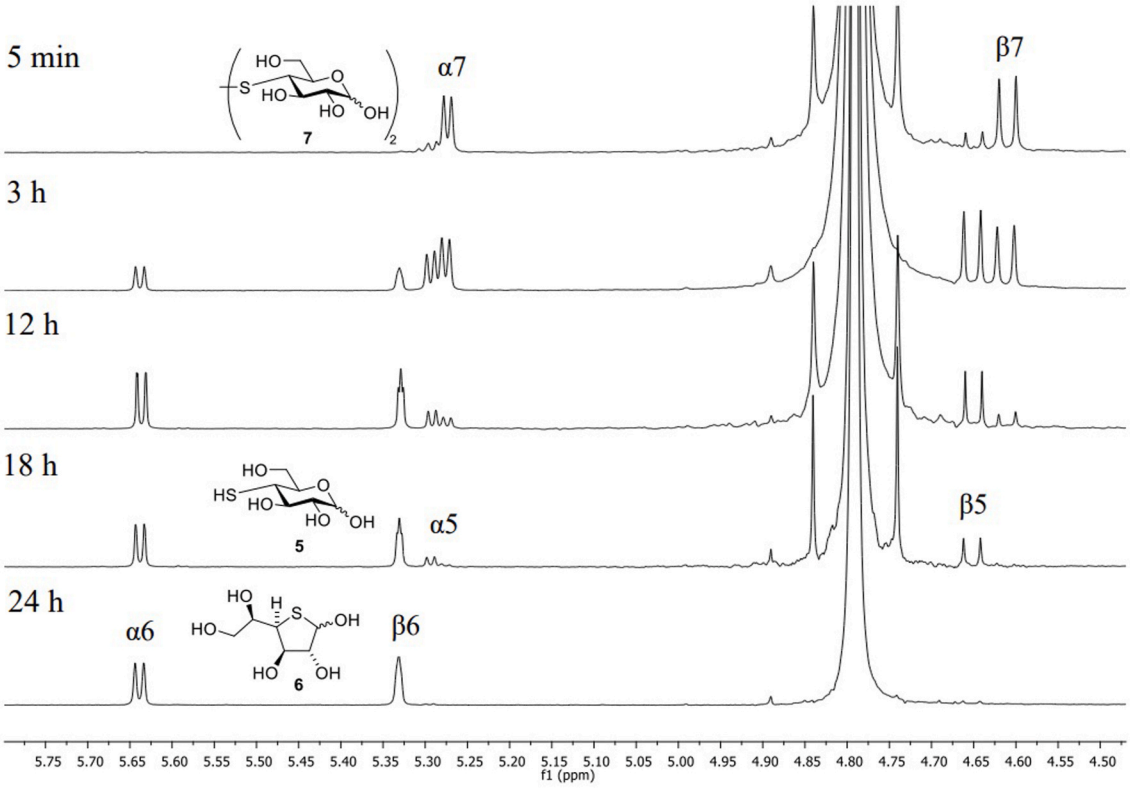


Fig. 4. Stacked ^1H NMR spectra (400 MHz, D_2O) illustrating the interconversion of 5 and 6 from 7, over 24 h. For the full stacked ^1H NMR spectra see supporting information section S5.

6–9), until being the only isomer visible after 24 h (Table 3 entry 10). Complete reduction of **7** required approximately 21 h (Table 3, entry 9).

These results indicate that a DTT mediated reduction of disulfide **7** could be harnessed as a reservoir to release **5,** opening the door to investigate enzymatic transformations therefrom e.g., 1-phosphate formation with a relevant kinase. These investigations are currently underway in our laboratory and will be reported in due course.

3. Conclusion

We have identified an improved method to synthesise both bis(4-thio-α-D-glucopyranoside)-4,4′-disulfide **7** and 4-thio-D-glucofuranose **6** from a single precursor. Isolation of homogenous **6** enabled its full characterisation. Whilst unable to access 4-thio-D-glucopyranose **5** directly, the disulfide form of this material can be used to release the free thiol form **5**, using DTT; however, a competing interconversion to the thermodynamically favoured 4-thiofuranose form is complete within 24 h. Access to these materials offers an intriguing prospect to further utilise them as building blocks for enzymatic (e.g., **6**/**7**) and chemical (e.g., **4**) synthesis of thiosugars, especially for the rarer 4-thioglucofuranose form.

Declaration of competing interest

Gavin Miller is guest editing the Early Career Researcher VSI this submission is included within. He had no involvement in the peer review of this article and has no access to information regarding its peer-review.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.carres.2023.108759.

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