Transition-metal-free synthesis of aryl 1-thioglycosides with arynes at room temperature†

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A mild, convenient and transition-metal-free protocol for the synthesis of aryl 1-thioglycosides is presented via arynes generated in situ combined with glycosyl thiols in the presence of TBAF(BuOH). The methodology provides a general and efficient way to prepare a series of functionalized thioglycosides in good to excellent yields with a perfect control of the anomeric configuration at room temperature. In addition, the reaction conditions tolerate a variety of the pentoses and hexoses, and the reaction also performs smoothly on protected monosaccharides and disaccharides.

Introduction

Thioglycosides, as the analogue of the O-glycosides, are versatile intermediates in carbohydrate synthesis and have a wide range of potential applications,1 and are regularly used as glycosyl donors2 to construct various oligosaccharides and glycoconjugates. Additionally, they are more stable in both chemical and enzymatic degradations3 and have also been employed extensively as inhibitors in a great number of biochemical studies.4 Thioglycoside fragments are also widely used in various drugs, natural products and pharmaceutical active agents.5 Some examples of the thioglycosides derivatives include the cytotoxic Hsp90 inhibitor;6 MUS-CB,7 hSGLT1 inhibitor,8 lincomycin,9 clindamycin,10 as well as irreversible glycoside inhibitors11 (Fig. 1). On account of the great significance of S-glycosides, some elegant synthetic protocols have been developed.

Originally, the approaches to prepare the 1-thioglycosides were that glycosyl donors reacted with sulfurs (or thiophenols) in the presence of the stoichiometric Lewis acids12 or glycosyl halides reacted with thiolate anions under strong base conditions (Scheme 1a). However, the inferior stereo-selectivity or strong base of those traditional methods limited their application. With the efforts of Sticha, Xue, Messaoudi et al., the synthesis of 1-thioglycosides achieved great advances in recent years. As shown in Scheme 1b, the functionalization of glycosyl thiols had been catalysed by the Ni,13 Cu14, Pd15 transition metal via the Buchwald–Hartwig–Migita coupling reaction. These methods required an expensive catalyst or a high temperature (above 100 °C in some protocols of the Cu or Pd catalyst) and long reaction time, which limit the universality of the reaction to some extent. In 2019, Messaoudi and co-workers developed a protocol for the synthesis of aryl 1-thioglycosides via a Ni/photoredox dual catalyzed cross-coupling reaction16 (Scheme 1c). Recently, Messaoudi’s group described the first electrochemical method for coupling various anemic glycosyl thiols with aryl bromides17 (Scheme 1c). The reaction didn’t need to perform in a strong base environment, and demonstrated superiority to synthesize highly complex thioglycosides under mild conditions. However, some functional groups are not compatible with this procedure.

Arynes are highly reactive transient intermediates and are also useful synths in organic synthesis due to the unique triple bonds, and have been extensively used in the synthesis of natural products, drug molecules and functional materials.18 Arynes have made remarkable achievements in nucleophilic reactions,19 pericyclic reactions,20 transition metal catalyzed reactions21 and multicomponent reactions.22 In recent years, N-arylation of carbohydrate amines23 and O-arylation of carbohydrates24 have been reported via the aryne insertion reactions with glycosyl

Fig. 1 Examples of biologically active thioglycosides.
with arynes (Scheme 1c). The methodology of using the arynes generated in situ to realize the functionalized thioglycosides was not developed yet. On the basis of our previous research in carbohydrates, we report a novel protocol for the preparation of thioglycosides via a tandem intermolecular nucleophilic coupling of glycosyl thiols with arynes (Scheme 1c).

### Results and discussion

We started by optimizing the reaction conditions with tetra-O-acetylated 1-thio-β-D-glucopyranose 1a (1.0 equiv.), O-bearing glycosyl thiols. The methodology of using the aryne generated in situ to realize the functionalized thioglycosides was not developed yet. On the basis of our previous research in carbohydrates, we report a novel protocol for the preparation of thioglycosides via a tandem intermolecular nucleophilic coupling of glycosyl thiols with arynes (Scheme 1c).

| Entry | F⁻ sources | Solvent | Time/h | T°C | Yield (%) |
|-------|------------|---------|--------|-----|-----------|
| 1     | CsF        | CH₃CN   | 2.0    | 25  | 70(64)    |
| 2     | AgF        | CH₃CN   | 2.0    | 25  | 51        |
| 3     | KF         | CH₃CN   | 2.0    | 25  | 42        |
| 4     | ZnF₂       | CH₃CN   | 2.0    | 25  | 35        |
| 5     | TBAF-3H₂O  | CH₃CN   | 2.0    | 25  | 63        |
| 6     | TBAF(THF)  | CH₃CN   | 2.0    | 25  | 67        |
| 7     | TBAF-(BuOH)₄ | CH₃CN | 2.0    | 25  | 86        |
| 8     | TBAF-(BuOH)₄ | DCM    | 2.0    | 25  | 78        |
| 9     | TBAF-(BuOH)₄ | THF    | 2.0    | 25  | 40        |
| 10    | TBAF-(BuOH)₄ | Toluene | 2.0    | 25  | 33        |
| 11    | TBAF-(BuOH)₄ | MeOH   | 2.0    | 25  | 37        |
| 12    | TBAF-(BuOH)₄ | DMSO   | 2.0    | 25  | 26        |
| 13    | TBAF-(BuOH)₄ | DMF    | 2.0    | 25  | 30        |
| 14    | TBAF-(BuOH)₄ | CH₃CN  | 2.0    | 40  | 85        |
| 15    | TBAF-(BuOH)₄ | CH₃CN  | 2.0    | 60  | 60        |
| 16    | TBAF-(BuOH)₄ | CH₃CN  | 3.0    | 25  | 83        |
| 17    | TBAF-(BuOH)₄ | CH₃CN  | 4.0    | 25  | 82        |

* Standard conditions: 1a (0.1 mmol, 1.0 equiv.), 2a (0.11 mmol, 1.1 equiv.), TBAF-(BuOH)₄ (0.2 mmol, 2.0 equiv.), CH₃CN (1.5 mL) as solvent, r.t., 2 h. * * * 2a (0.1 mmol, 1.0 equiv.), TBAF-(BuOH)₄ (0.3 mmol, 3.0 equiv.), CH₃CN (1.5 mL) as solvent, r.t., 2 h.

**Scheme 2** Scopes of glycosyl thiols 1 reacted with arylene 2a. Standard conditions: 1 (0.1 mmol, 1.0 equiv.), 2a (0.11 mmol, 1.1 equiv.), TBAF-(BuOH)₄ (0.2 mmol, 2.0 equiv.), dry CH₃CN (1.5 mL).
successfully and obtained the β-disaccharide in good yields, indicated the electronic-effect of the protecting groups did not have significant influence for the reaction efficiency. Meanwhile, we also found the α-glycosyl thiols with OBen protected group can reacted with 2a when the temperature improved to 45 °C and the yield of corresponding product 3j was 78%. The sugar 1k with OPiv group provided 3k in 71% yield under optical condition. However, the reactivity of unprotected glycosyl thiol was decreased and the yield of product was only 30%.

Subsequently, we also investigated the electronic effects and regioselectivity of this reaction via different aryne precursors and glycosyl thiols 1. Regardless of the electron-donating or withdrawing properties of the symmetrical aryne precursors 2, the corresponding products were obtained in good yields. The difluoro substituted arynes 2b reacted with 1 gave the thio-glycosides derivatives 4a–4h in 71–82% yields. Aryne precursor 2c reacted with a series of glycosyl thiols under standard reaction condition to afford the corresponding products 4i–4l in good yields (72–84%). Also, the dimethyl substituted arynes precursors reacted with 1a under standard condition to give corresponding aryl 1-thioglucoside 4m with 78% yield.

Meanwhile, 3-methoxy non-symmetric aryne precursor 2d exhibited excellent regioselectivity due to the steric effect and electronic effects, and single target thio-glycosides 4n, 4o were obtained in good yields (80%, 78%). However, we found that 4-methylnapthylene precursor 2e reacted with 1a and 1f gave a near equimolar mixture of two inseparable regioisomers, providing corresponding products in 82% and 90% yield, respectively. 1,2-Naphthylene precursor 2f under the same conditions also provided a near equimolar mixture of two inseparable regioisomers (Scheme 3).

To demonstrate the synthetic utility of this transformation, we next performed a scale-up reaction of 3a. As shown in Scheme 4a, a gram scale reaction of 1-thio-β-D-glucopyranose 1a with aryne precursor 2a proceeded to give 3a in 85% yield. A plausible mechanism was proposed in Scheme 4b, based on the basis of the experimental results and the related report by Jin and co-workers.27

**Conclusions**

In conclusion, we have developed a convenient method for the preparation of aryl-thioglycosides under mild and metal-catalyst-free conditions via a tandem intermolecular nucleophilic coupling of glycosyl thiols with arynes. Meanwhile, the method is applicable for various monosaccharide and disaccharide substrates, which has wide practical value between biochemistry and medicinal chemistry. In addition, 1,2-trans-thioglycosides were stereoselectively formed by the reaction of the in situ generated arynes with glycosyl thiols in good to excellent yields. Importantly, we provided a new protocol for the synthesis of functionalized thioglycosides and the possibility for further derivatization of glycosyl donors.

**Experimental**

**General information**

All reactions were carried out in dried glassware. The solvent in the reaction were dried use activated 4 Å molecular sieve, commercial reagents were used without further purification unless otherwise stated. Purification of reaction products were carried out by flash chromatography on silica gel (200–300 mesh). NMR spectra were measured in CDCl₃ (with TMS as internal standard) on a Bruker AV400 (1H at 400 MHz, 13C at 100 MHz, 19F at 376 MHz) magnetic resonance spectrometer.
The general procedure for the reaction of glycosyl thiols 1 with aryl precursors 2

The glycosyl thiol 1 (50 mg, 0.14 mmol, 1.0 equiv.), aryl precursor 2 (45 mg, 0.150 mmol, 1.1 equiv.) and TBAF·(BuOH)4 (0.274 mmol, 2.0 equiv.) were sequentially added in a clean and dry Schlenk tube, and the tube was then evacuated and back-filled with nitrogen (this sequence was repeated three times). Under nitrogen atmosphere, MeCN (1.5 mL) was added to the mixture system, then the mixture was stirred at room temperature for 2.0 hours. Saturated NaCl solution was added to dilute the system and extracted with EtOAc (3 x 2 mL). The combined organic phase was dried over anhydrous Na2SO4, filtered, and evaporated in vacuo. Finally, the crude product was purified via flash column chromatography on silica gel to give the desired product.

Phenyl-2,3,5-tri-O-acetyl-1-thio-β-D-xylofuranoside (3g)

Purified by flash column chromatography Rf = 0.39 (petroleum ether/acetone = 3 : 1), white solid (29.8 mg, 81% yield); [α]D20 = −55.4 (c = 1.0, CHCl3); 1H NMR (400 MHz, CDCl3) δ 7.55-7.46 (m, 2H, ArH), 7.40-7.28 (m, 3H, ArH), 5.18 (t, J = 8.2 Hz, 1H), 5.06-4.87 (m, 2H), 4.80 (d, J = 8.4 Hz, 1H), 4.28 (dd, J = 11.8, 4.9 Hz, 1H), 3.42 (dd, J = 11.8, 8.7 Hz, 1H), 2.09 (s, 3H), 2.05 (s, 3H), 2.04 (s, 3H); 13C NMR (100 MHz, CDCl3) δ 130.3 (dd, J = 118.9, 169.1, 168.9, 168.3, 149.6 (dd, J = 250.8, 12.5 Hz), 148.9 (dd, J = 250.9, 13.0 Hz), 128.5 (dd, J = 6.2, 3.7 Hz), 127.1 (dd, J = 6.1, 4.2 Hz), 121.1 (d, J = 18.3 Hz), 116.4 (d, J = 17.7 Hz), 84.9, 73.6, 70.8, 66.2, 65.9, 60.8, 19.8, 19.6, 19.5, 19.5; 19F {1H} NMR (376 MHz, CDCl3) δ −135.92 (dt, J = 19.8, 9.3 Hz), −137.16 (m); 19F NMR (376 MHz, CDCl3) δ −135.92 (d, J = 21.0 Hz), −137.17 (d, J = 20.9 Hz). HRMS (ESI): [m/z] calcd for C26H32F2O9SNa+ (M + Na)+ 499.0845, found 499.0841.

3.4-Difluorophenyl-2,3,4,6-tetra-O-acetyl-1-thio-β-D-glucopyranoside (4d)

Purified by flash column chromatography Rf = 0.28 (petroleum ether/acetone = 1 : 2), white solid (33.6 mg, 71% yield); [α]D20 = −24.5 (c = 1.0, CHCl3); 1H NMR (400 MHz, CDCl3) δ 7.43 (dd, J = 10.0, 7.4, 2.1 Hz, 1H), 7.25-7.19 (m, 1H), 7.09 (dt, J = 10.1, 8.4 Hz, 1H), 5.76 (d, J = 9.1 Hz, 1H), 5.19 (t, J = 9.8 Hz, 1H), 5.02 (t, J = 9.7 Hz, 1H), 4.77 (d, J = 10.4 Hz, 1H), 4.21-4.13 (m, 2H), 4.00 (q, J = 9.8 Hz, 1H), 3.77-3.67 (m, 1H), 2.08 (s, 3H), 2.02 (s, 6H), 1.98 (s, 3H); 13C NMR (100 MHz, CDCl3) δ 171.0, 170.6, 170.1, 169.3, 150.6 (dd, J = 251.0, 12.6 Hz), 149.9 (dd, J = 250.9, 12.8 Hz), 129.6 (dd, J = 6.3, 3.6 Hz), 128.2 (dd, J = 6.3, 4.2 Hz), 122.1 (d, J = 18.2 Hz), 117.5 (d, J = 17.5 Hz), 86.4, 75.9, 73.5, 68.2, 62.3, 53.3, 23.6, 20.6, 20.5; 19F {1H} NMR (376 MHz, CDCl3) δ −135.93 (dt, J = 19.9, 9.3 Hz), −137.28 (m); 19F NMR (376 MHz, CDCl3) δ −135.93 (d, J = 21.4 Hz), −137.29 (d, J = 21.0 Hz). HRMS (ESI): [m/z] calcd for C26H32F2O9SNa+ (M + Na)+ 498.1005, found 498.1006.

3.4-Difluorophenyl-2,3,4,6-tetra-O-acetyl-1-thio-α-L-rhamnopyranoside (4e)

Purified by flash column chromatography Rf = 0.47 (petroleum ether/acetone = 3 : 1), white solid (31.8 mg, 76% yield); [α]D20 = −116.0 (c = 1.0, CHCl3); 1H NMR (400 MHz, CDCl3) δ 7.31 (dd, J = 10.0, 7.3, 2.2 Hz, 1H), 7.21-7.17 (m, 1H), 7.11 (dt, J = 10.0, 8.3 Hz, 1H), 5.45 (dd, J = 3.3, 1.7 Hz, 1H), 5.35 (d, J = 1.6 Hz, 1H), 5.21 (dd, J = 10.1, 3.2 Hz, 1H), 5.14 (t, J = 9.8 Hz, 1H), 4.34-4.26 (m, 2H), 2.14 (s, 3H), 2.07 (s, 3H), 2.01 (s, 3H), 1.25 (d, J = 6.2 Hz, 3H); 13C NMR (100 MHz, CDCl3) δ 170.1, 170.0, 170.4, 150.5 (dd, J = 250.5, 12.6 Hz), 150.3 (dd, J = 251.8, 13.1 Hz), 129.4 (dd, J = 6.1, 4.1 Hz), 128.6 (dd, J = 6.3, 3.7 Hz), 121.2 (d, J = 18.2 Hz), 118.0 (d, J = 17.7 Hz), 86.0, 71.1, 71.0, 69.4, 68.1, 21.0, 20.9, 20.8, 17.4; 19F {1H} NMR (376 MHz, CDCl3) δ −135.57 (dt, J = 20.9, 9.2 Hz), −137.48 (m); 19F NMR (376 MHz, CDCl3) δ −135.57 (d, J = 20.9 Hz), −137.50 (d, J = 21.0 Hz). HRMS (ESI): [m/z] calcd for C16H23F2O2SNa+ (M + Na)+ 441.0790, found 441.0793.

3.4-Difluorophenyl-2,3,5-tri-O-acetyl-1-thio-β-D-xylofuranoside (4g)

Purified by flash column chromatography Rf = 0.38 (petroleum ether/acetone = 3 : 1), white solid (29.9 mg, 74% yield); [α]D20 = −56.9 (c = 0.5, CHCl3); 1H NMR (400 MHz, CDCl3) δ 7.35 (dd, J = 10.0, 7.4, 2.2 Hz, 1H), 7.24-7.19 (m, 1H), 7.11 (dt, J = 10.1, 8.3 Hz, 1H), 5.17 (t, J = 8.1 Hz, 1H), 4.94-4.85 (m, 2H), 4.73 (d, J = 8.2 Hz, 1H), 4.27 (dd, J = 11.8, 4.9 Hz, 1H), 3.43 (dd, J = 11.8, 8.6 Hz, 1H), 2.10 (s, 3H), 2.04 (s, 6H); 13C NMR (100 MHz, CDCl3)
δ 170.0, 169.9, 169.5, 150.9 (dd, J = 250.9, 12.5 Hz), 150.1 (dd, J = 251.7, 12.9 Hz), 130.1 (dd, J = 5.9, 3.8 Hz), 128.0–127.7 (m), 122.6 (d, J = 18.0 Hz), 117.8 (d, J = 17.6 Hz), 85.9, 71.8, 69.7, 68.3, 65.3, 20.9, 20.9, 20.8; 19F {1H} NMR (376 MHz, CDCl3) δ = −135.81 (dt, J = 21.3, 9.2 Hz), −136.89 (m); 13C NMR (376 MHz, CDCl3) δ = −135.81 (d, J = 21.4 Hz), 67.3 (d, J = 8.0 Hz, 1H), 5.97 (s, 2H), 5.14 (dt, J = 14.9, 9.3 Hz, 2H), 5.05 (t, J = 9.6 Hz, 1H), 4.90 (dd, J = 9.2, 7.9 Hz, 1H), 4.82 (t, J = 9.7 Hz, 1H), 4.58 (dd, J = 11.9, 2.0 Hz, 1H), 4.50 (dd, J = 13.5, 9.0 Hz, 2H), 4.37 (dd, J = 12.5, 4.2 Hz, 1H), 4.07 (dd, J = 11.9, 5.3 Hz, 1H), 4.01 (dd, J = 12.5, 2.3 Hz, 1H), 3.69 (t, J = 9.5 Hz, 1H), 3.65–3.56 (m, 2H), 2.12 (s, 3H), 2.08 (s, 3H), 2.06 (s, 3H), 2.01 (s, 3H), 1.99 (s, 3H), 1.99 (s, 3H), 1.97 (s, 3H); 13C NMR (100 MHz, CDCl3) δ = 170.6, 170.4, 170.4, 169.9, 166.9, 169.4, 146.8, 147.9, 128.8, 122.8, 114.8, 108.8, 101.6, 100.9, 85.8, 76.4, 73.8, 73.0, 72.1, 71.7, 70.0, 67.8, 61.9, 61.6, 20.9, 20.8, 20.7, 20.6.

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Conflicts of interest

There are no conflicts to declare.
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