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Switchable synthesis of benzimidazole/quinoxaline *C*-glycosides with *o*-phenylenediamines and sulfoxonium ylide glyco-reagents†

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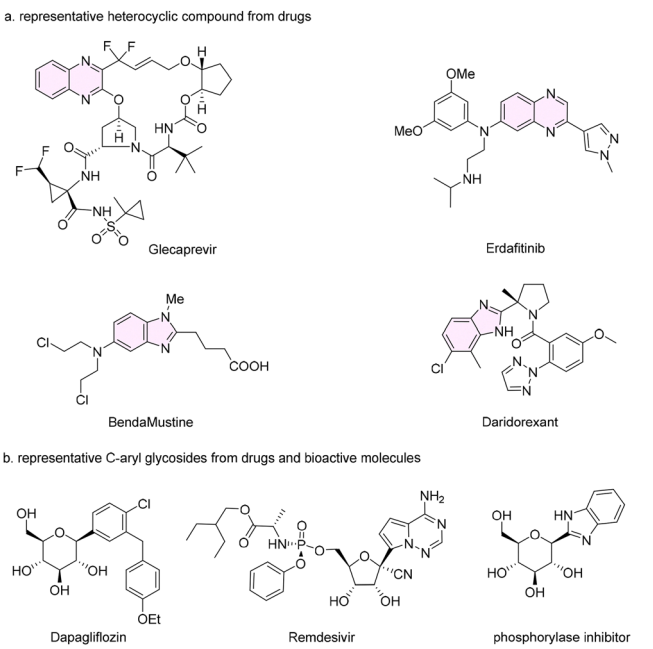
A novel switchable approach for the synthesis of benzimidazole/quinoxaline *C*-glycosides is introduced. This versatile catalytic system facilitates the reaction of *o*-phenylenediamines with carbonyl sulfoxonium ylide glyco-reagents under mild conditions to selectively form 2-glycoside quinoxalines and 2-glycoside benzimidazoles, through $[OsCl_2(p\text{-cymene})]_2$ and $AgSbF_6$ catalysts, respectively. The process is adaptable as it can generate a variety of heteroarene *C*-glycosides by accommodating a wide range of sugar donors. It is also ideal for the modification of compounds like varenicline due to its excellent functional group compatibility, mild reaction conditions, and wide substrate range.

Carbohydrates are fundamental biomolecules that function as a primary energy source for living organisms and also play a vital role in various biological processes. In addition to their biological importance, carbohydrates have broad industrial applications in healthcare, energy, materials science, and agriculture.[1,2] Meanwhile, the synthesis of aromatic heterocyclic compounds has become increasingly significant in the chemical industry due to their diverse applications. These compounds act as key precursors in the development of advanced materials, including pharmaceuticals, polymers, and pesticides.[3] The quinoxaline scaffold is an important heterocyclic structure with a variety of biological functions. It is a significant component of different medicines and bioactive substances, such as antioxidants,[4] adenosine receptor antagonists, anti-inflammatory agents, antidepressants,[5] and anticancer and antibacterial drugs.[6] Glecaprevir (Scheme 1a), for instance, is used to treat hepatitis C because of its intricate stereochemical quinoxaline structure. Erdafitinib (Scheme 1a) is also prescribed for advanced or metastatic bladder cancer, with a particular emphasis on patients with specific genetic mutations. Benzimidazole is an aromatic heterocyclic structure that serves as a crucial scaffold in numerous bioactive substances. Its significance in pharmaceutical research and drug discovery positions it as a primary focus in the development of novel therapeutic agents.[7] It was shown that 5,6-dimethyl-1-(α-D-ribofuranosyl)benzimidazole is an important component in the chemical structure of vitamin B12. Since the 1950s, benzimidazole has garnered significant attention in agriculture, biochemical medicine, and polymer technology owing to its unique electron-rich aromatic structure.[8] Bendamustine (Scheme 1a) acts as a bifunctional alkylating agent, displaying potent anti-tumor and cytotoxic effects. Furthermore, daridorexant (Scheme 1a) is formulated to address insomnia by extending sleep duration while ensuring



Scheme 1 Selected examples of heterocyclic compounds and *C*-aryl glycosides.

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safety for prolonged use. Therefore, benzimidazole and quinoxaline have become significant building blocks for the development of several novel compounds with potential therapeutic applications.

Under chemical or enzymatic hydrolysis, *C*-glycosides are well known to show more stability than *O*- and *N*-glycosides.[9] This improved stability makes *C*-glycoside molecules quite valuable in fields including medicinal chemistry and biomedicine.[10] For instance, dapagliflozin, a widely used diabetic mellitus drug, remdesivir, a prospective COVID-19 treatment with broad-spectrum antiviral action, and a phosphorylase inhibitor (Scheme 1b). Given their significance in fields like industrial synthesis and medicinal chemistry, the development of heterocyclic compounds, particularly those incorporating quinoxaline and benzimidazole *C*-glycosides, holds considerable potential for advancement in organic synthesis.[11]

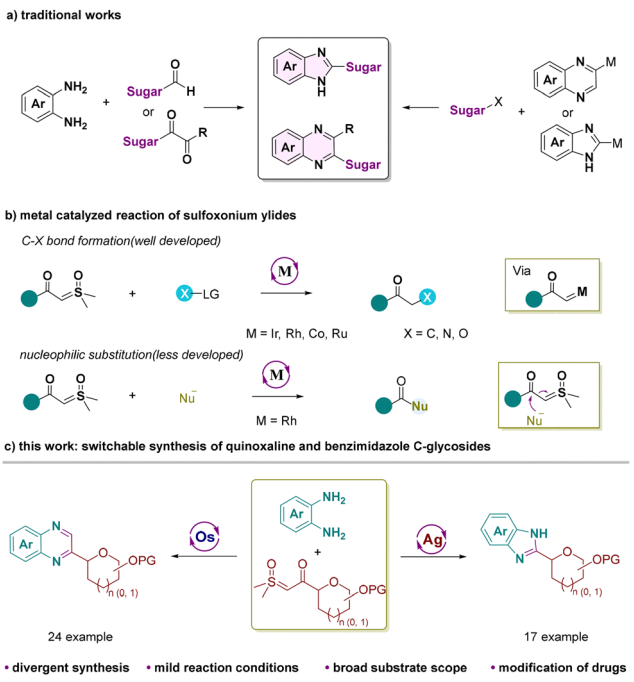
In recent years, the synthesis of 2-glycoside benzimidazole and 2-glycoside quinoxaline has garnered considerable attention.[12–15] Traditionally, two main strategies are employed to synthesize these compounds (Scheme 2a). The first method involves the condensation of glycosyl aldehydes or glycosyl diketones with *o*-phenyl enediamine to produce benzimidazole/quinoxaline *C*-glycosides.[14] However, this approach requires long reaction steps and the glycosyl reagents are often unstable. The second method forms C–C bonds between glycosyl reagents and active benzimidazole/quinoxaline reagents through nucleophilic reactions.[15] These syntheses, however, are often unsatisfactory due to harsh conditions and limited compatibility with various functional groups. The applicability of these methods to the synthesis of complex *C*-glycosides is somewhat limited. Therefore, a switchable synthetic approach to benzimidazole/quinoxaline *C*-glycoside would be highly valuable.

Sulfoxonium ylides have garnered significant attention in organic synthesis, especially in transition metal-catalyzed reactions such as C–H functionalization, insertion reactions, and cyclopropanation.[16] These processes predominantly rely on the generation of metal carbene intermediates, leveraging the unique reactivity profiles of sulfoxonium ylides (Scheme 2b). Despite their well-established utility in these contexts, the exploitation of sulfoxonium ylides as acyl precursors remains a relatively underexplored area, with only a handful of reports documented in the current literature[17] (Scheme 2b).

Recently, Liu's group has developed a carbonyl sulfoxonium ylide glyco-reagent for constructing heteroaryl *C*-glycosides.[18] We propose that the regulation of the reaction site may facilitate the synthesis of diverse heteroarene *C*-glycosides. In this context, herein, a switchable approach is presented for synthesizing quinoxaline/benzimidazole *C*-glycosides. Under different catalytic conditions, *o*-phenylenedi amines react with carbonyl sulfoxonium ylide glyco-reagents to selectively form 2-glycoside benzimidazoles and 2-glycoside quinoxalines (Scheme 2c). This approach facilitates the synthesis of *C*-glycosides with a variety of heteroaromatic structures, greatly enhancing the available strategies for producing heteroarene *C*-glycosides.

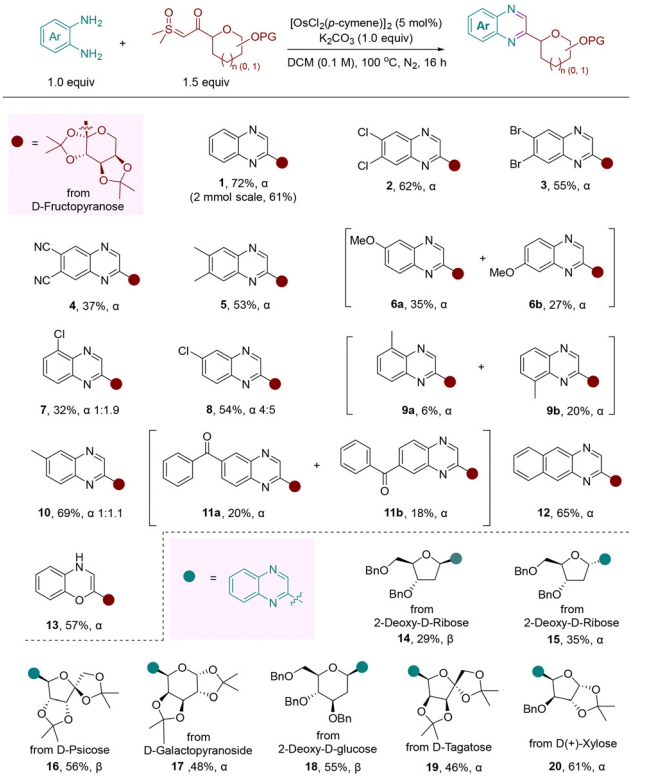
First, we initially conducted our investigation by selecting *o*-phenylenediamine and carbonyl sulfoxonium ylide glyco-reagents from D-fructopyranose as the model partners. Pleasingly, the expected product **1** was obtained in 72% yield in the presence of $[OsCl_2(p\text{-cymene})]_2$ (5 mol%), K_2CO_3 (1.0 equiv.), and DCM (0.1 M) under nitrogen at 100 °C for 16 h through condition optimization. To evaluate the feasibility of this strategy, we conducted scale-up experiments at a scale of 1 millimolar and synthesized compound **1** in a yield of 61%. Subsequently, a variety of 2-glycosylation quinoxalines were synthesized from *o*-phenylenediamines and carbonyl sulfoxonium ylide glyco-reagents using the optimal conditions (Table 1). The reaction demonstrated good functional group compatibility, accommodating a diverse array of *o*-phenylenediamine substrates containing electron-withdrawing (**2**, **3**, **4**, **7**, **8**, **11**), and electron-donating groups (**5**, **6**, **9**, **10**). The substrates with strong electron-withdrawing groups exhibited unsatisfication yields (**4**, **11**). Additionally, the yield of 5-chloro-2-glycosyl-quinoxaline (**7**) was 32%, with an isomerism ratio of 1 : 1.9. The yield of 6-chloro-2-glycosyl-quinoxaline (**8**) was 54% and its isomerism ratio was 4 : 5. Notably, 6-methyl-2-glycosyl-quinoxaline (**10**) achieved a satisfactory yield of 69% and a positional isomer ratio of 1 : 1.1 in substrates with neutral group substitutions. Furthermore, this strategy was well-tolerated by 2-glycosyl-benzo[*g*]quinoxaline (**12**), giving 65% yield. Interestingly, when *o*-aminophenol reacts with carbonyl sulfoxonium ylide glyco-reagents derived from D-fructopyranose, 2-glycosyl-4*H*-benzo[*b*][1,4]oxazine (**13**) was obtained with a yield of 57%. It is worth mentioning that this strategy demonstrated excellent tolerance towards halogens (**2**, **3**, **7**, **8**), a cyano-group (**4**) and a carbonyl group (**11**), providing promising prospects for late-stage modification. Next, substrate expansion on the carbonyl sulfoxonium ylide glyco-reagents could also be derived smoothly from 2-deoxy-D-ribose (**14**, **15**), D-psicose (**16**), D-galactopyranoside (**17**), 2-deoxy-D-glucose (**18**) and D-tagatose (**19**), as well as D(+)-xylose (**20**), all of which exhibited moderate yields. It is noted that this reaction proceeds *via* a cascade annulation/oxidation process, with DMSO serving as the oxidant.

Subsequently, we developed a method for synthesizing 2-glycosyl benzimidazole derivatives using *o*-phenylenediamine and carbonyl



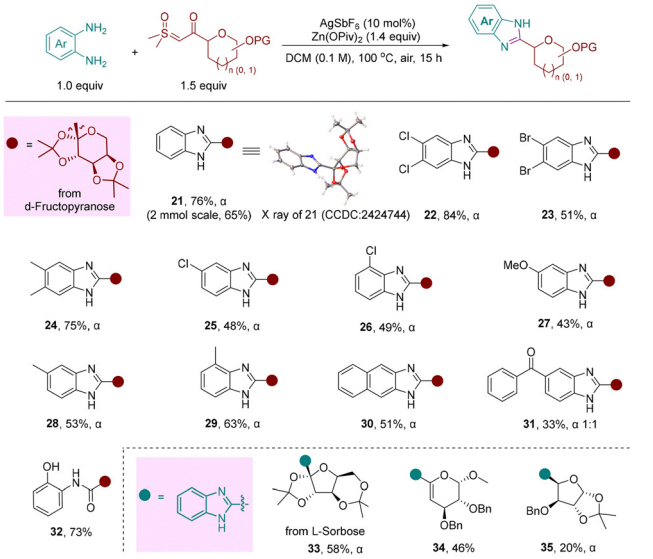
Scheme 2 General methods for the synthesis of *C*-aryl glycosides.

Table 1 Synthesis of 2-glycoside quinoxaline



Reaction conditions: *o*-phenylenediamine (0.2 mmol), carbonyl sulfoxonium ylides glycogen anomeric (0.3 mmol), $[OsCl_2(p\text{-cymene})]_2$ (5 mol%), K_2CO_3 (1.0 equiv.), in DCM (0.1 M), 100 °C, N_2, 16 h.

Table 2 Synthesis of 2-glycosyl benzimidazole



Reaction conditions: *o*-phenylenediamine (0.2 mmol), carbonyl sulfoxonium ylides glycogen anomeric (0.3 mmol), $AgSbF_6$(10 mol%), $Zn(OPiv)_2$ (1.4 equiv.) in DCM (0.1 M), 100 °C, air, 15 h.

sulfoxonium ylide glycol-reagents. As shown in Table 2, 2-glycosyl-1*H*-benzo[*d*]imidazole (**21**) was obtained in 76% yield in the presence of $AgSbF_6$ (10 mol%) and $Zn(OPiv)_2$ (1.4 equiv.) in DCM (0.1 M) under air for 15 h at 100 °C. The absolute configuration of the anticipated product **21** was determined by nuclear magnetic resonance (NMR) and X-ray crystallography analysis (CCDC 2424744). Under the optimal conditions, we performed scale-up experiments at the 1 millimolar level and successfully synthesized compound **21** with a yield of 65%. Subsequently, we explored the substrate scope of 2-glycosyl benzimidazoles derived from the reaction between *o*-phenylenediamine and carbonyl sulfoxonium ylide glyco-reagents. This method successfully accommodated a wide range of *o*-phenylenediamine substrates, including those with electron-donating (**24**, **27**, **28**, **29**), and electron-withdrawing (**22**, **23**, **25**, **26**, **31**) groups, yielding the desired products in moderate to good yields. We further extended the applicability of carbonyl sulfoxonium ylide glyco-reagents, achieving yields of 58% for compound (**33**) and 46% for compound (**34**). Of note, this strategy demonstrates good compatibility with halogenated (**22**, **23**, **25**, **26**), carbonyl-containing (**31**), naphthalene-2,3-diamine (**30**), providing a solid foundation for later stage modifications. To our surprise, the *o*-aminophenol substrate had produced an acyclic product (**32**).

Varenicline is a medication used for treating nicotine addiction. To demonstrate the practicality of our strategy, we designed and synthesized novel derivatives of varenicline (Table 3). To our surprise, under $[OsCl_2(p\text{-cymene})]_2$ (5 mol%), K_2CO_3 (1.0 equiv.), and DCM (0.1 M), the *o*-phenylenediamine derivative (**36**) and carbonyl sulfoxonium ylide glyco-reagents were reacted under nitrogen at 100 °C for 16 hours to yield 2-glycosides quinoxaline (**37–40**). Moreover, in the presence of $AgSbF_6$ (10 mol%), and $Zn(OPiv)_2$ (1.4 equiv.) in DCM (0.1 M), the *o*-diamine intermediate (**36**) was reacted with carbonyl sulfoxonium ylide glyco-reagents under 100 °C for 15 hours, resulting in the formation of 2-glycosides benzimidazole (**41**, **42**), and both of them had satisfactory yields. Meanwhile, these results further demonstrated the universality and usefulness of the switchable method in the synthesis of aromatic heterocyclic *C*-glycosides.

In conclusion, we developed a switchable catalytic platform that enables the selective synthesis of 2-glycosylquinoxaline and 2-glycosylbenzimidazole derivatives. This methodology utilizes *o*-phenylenediamine derivatives in conjunction with carbonyl sulfoxonium ylide glycogen reagents as fundamental building blocks, enabling efficient construction of complex molecular architectures through a cascade annulation mechanism. Additionally, the methodology exhibits remarkable versatility by accommodating a diverse array of sugar donors, thereby generating various glycosyl-substituted heteroaryl *C*-glycosides. Moreover, the reaction features excellent functional group tolerance and broad substrate scope, making it suitable for the synthesis of *C*-glycoside derivatives of varenicline.

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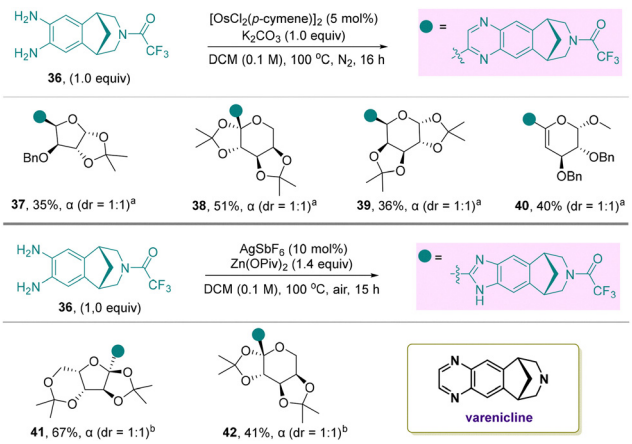
Conflicts of interest

There are no conflicts to declare.

Table 3 Synthetic application



Reaction conditions. [a] *o*-Phenylenediamine (0.1 mmol), carbonyl sulfoxonium ylides glycogen anomeric (0.15 mmol), $[OsCl_2(p\text{-cymene})]_2$ (5 mol%), K_2CO_3 (1.0 equiv.), in DCM (0.1 M), 100 °C, N_2, 16 h. [b] *o*-Phenylenediamine (0.1 mmol), carbonyl sulfoxonium ylides glycogen anomeric (0.15 mmol), $AgSbF_6$(10 mol%), $Zn(OPiv)_2$ (1.4 equiv.) in DCM (0.1 M), 100 °C, air, 15 h.

Data availability

The data supporting this article have been included in the ESI.†

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