**Synthesis of C-Alkyl Glycoamino Acids via Palladium-Catalyzed C(sp³)–H Glycosylation**

**Significance:** The authors report a highly efficient, regio- and diastereoselective palladium-catalyzed glycosylation of inert β-C(sp³)–H bonds of N-phthaloyl amino acids under mild conditions. The method utilizes a combination of silver carbonate and trifluoroacetic acid in a polar solvent (THF), which enabled the activation cycle of this C–H bond. The reaction has a high tolerance towards functional groups and a broad scope, providing over 30 β-substituted C-alkyl glycoamino acids with up to 88% yield.

**Comment:** This method allows for the first time to activate C(sp³)–H bonds for glycosylation reactions, thus advancing the state-of-the-art techniques in carbohydrate chemistry. Liu and co-workers propose a mechanistic cycle for the C–H activation, based on experimental studies. Therein, the formation of a palladacycle via cyclometallation plays a key role in the activation and the resulting diastereoselectivity.