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SUPPLEMENTARY MATERIALS

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ASYMMETRIC CATALYSIS

High-turnover hypiodite catalysis for asymmetric synthesis of tocopherols

Muhammet Uyanik,¹ Hiroki Hayashi,¹ Kazuaki Ishihara^{1,2,3*}

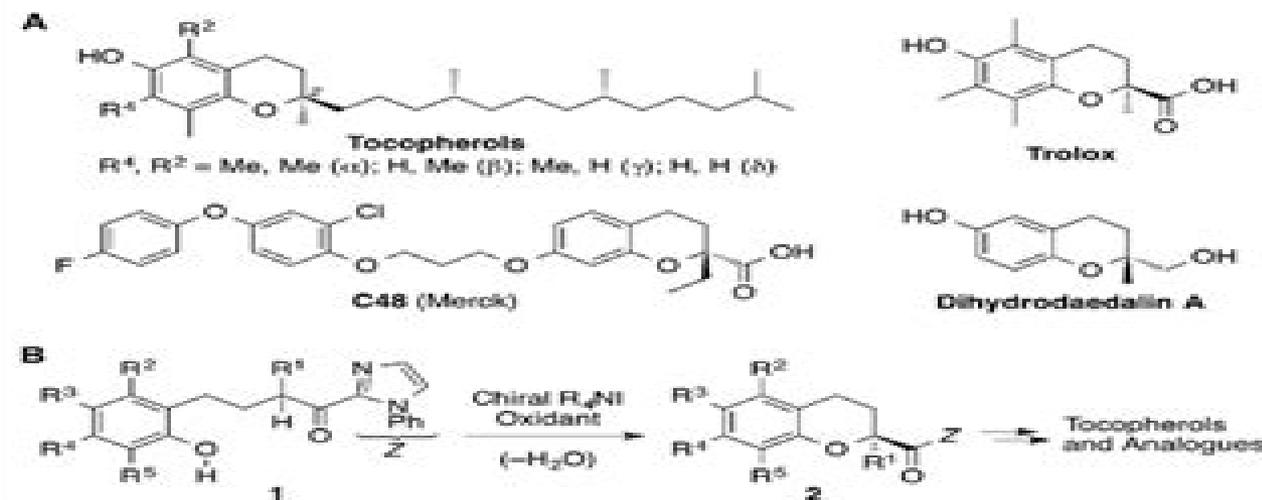
The diverse biological activities of tocopherols and their analogs have inspired considerable interest in the development of routes for their efficient asymmetric synthesis. Here, we report that chiral ammonium hypiodite salts catalyze highly chemo- and enantioselective oxidative cyclization of γ -(2-hydroxyphenyl)ketones to 2-acyl chromans bearing a quaternary stereocenter, which serve as productive synthetic intermediates for tocopherols. Raman spectroscopic analysis of a solution of tetrabutylammonium iodide and tert-butyl hydroperoxide revealed the in situ generation of the hypiodite salt as an unstable catalytic active species and triiodide salt as a stable inert species. A high-performance catalytic oxidation system (turnover number of ~200) has been achieved through reversible equilibration between hypiodite and triiodide in the presence of potassium carbonate base. We anticipate that these findings will open further prospects for the development of high-turnover redox organocatalysis.

Biologically active compounds containing a chiral chroman skeleton are abundant in nature (1). One of the most prominent chiral chromans is α -tocopherol, which is in the same family as vitamin E, together with β -, γ -, and δ -tocopherols and the corresponding α -, β -, γ -, and δ -tocotrienols (Fig. 1A) (2). α -Tocopherol acts as a lipid-soluble, chain-breaking antioxidant by capturing free radicals or singlet oxygen formed by oxidative metabolism in tissues (3). Furthermore, tocopherols show physiologically diverse properties, including anti-tumor, anti-inflammatory, anti-atherosclerosis, and cell-signaling activities (4–8). Various non-natural tocopherol analogs have also been developed because of their potency and distinct structural features. For example, trolox and its derivatives have been used as a chiral derivatizing reagent (9) and NO-releasing drug candidates (20). Dihydrodaedalin A, a synthetic intermediate for the natural product daedalin A (11), is a potent tyrosinase inhibitor and has been shown to suppress melanogenesis in human skin without

affecting cell viability (12). C48 developed by Merck research laboratories (Rahway, New Jersey) is a potent, selective peroxisome proliferator-

activated receptor- α/γ dual agonist and exhibits substantial antihyperglycemic and hypolipidemic activities (13).

Natural D - α -tocopherol is formally (2*R*,6*R*,8*R*)-2,5,7,8-tetramethyl-2-(4*R*,8',12'-trimethyltridecyl)-6-chromanol. The (2*R*)-configuration at the chroman ring among the three stereocenters of α -tocopherol is critical for its bioactivity; (2*S*)-stereoisomers are not accepted by the tocopherol transfer protein (5). Thus, asymmetric construction of the chroman ring has been an important challenge (1, 2). Enantioselective processes have been developed to address this need by using chiral transition metal complexes (Pd or Ru) (14–16) or organocatalysts (17, 18). However, the low catalytic activities and/or moderate enantioselectivities have limited their utility (2). Here, we report a transition metal-free (19) approach for the enantioselective synthesis of tocopherols and their analogs by using chiral hypiodite catalysts (20, 21) generated in situ from the corresponding quaternary ammonium iodide with alkyl hydroperoxides as environmentally benign co-oxidants (Fig. 1B). Chemoselective oxidative cyclization of hydroquinone-derived γ -(2-hydroxyphenyl)ketones



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