Synthesis of double winding vine-shaped biphenyl

(1) Department of Chemical Science and Engineering, Kobe University, 2 Research Center for Membrane and Film Technology, Kobe University) ○ Kohei Tabuchi,1 Aruto Maruka,2 Kentaro Okano,1 Atsunori Mori1,2

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During our ongoing efforts on the preparation of a novel class of compounds showing molecular asymmetry, we have recently shown that winding vine-shaped biphenyl 1 and 2 are synthesized and these molecules have exhibited molecular asymmetry.1 Our attention has thus focused on the synthesis of double winding vine-shaped molecules.

Biphenyl bearing four halomethyl groups at the 2,2',6,6'-positions 3 was prepared from pyrene as a starting material. The reaction of 3 (X=Br) with nosyl (Ns: 2-nitrobenzenesulfonyl) ethylenediamine2 in DMF underwent double cyclization to give winding vine-shaped product 4 in 97% yield. Biphenyl 3 (X=Cl) was subjected to allylation with allylmagnesium chloride to afford quadruply 3-butenylated prouct 5 in 55% yield. Following ring-closing metathesis3 provided the doubly cyclized product 6 in 62% yield.