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Two Axles Threaded Using a Single Template Site: Active Metal Template Macrobicyclic [3]Rotaxanes
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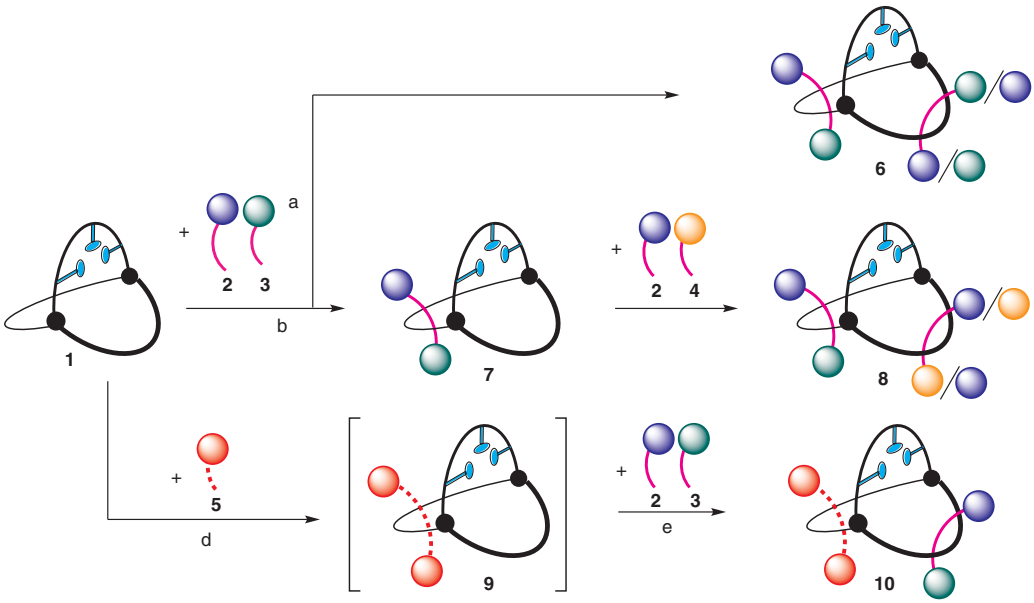
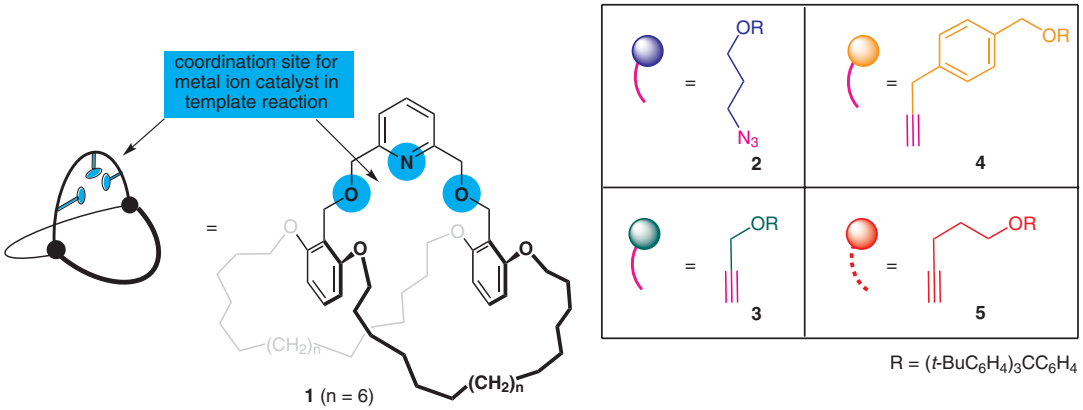
Templated Threading to [3]Rotaxanes





Reaction conditions: a) (i) **2**/**3** (10 equiv), $[Cu(MeCN)_4]PF_6$, DCE, 70 °C; (ii) EDTA, NH_3, 86% yield **6** (+ 14% yield **7**)
b) (i) **2**/**3** (5 equiv), $[Cu(MeCN)_4]PF_6$, DCE, 70 °C; (ii) EDTA, NH_3, 57% yield **7** (+ 40% yield **6**)
c) (i) $[Cu(MeCN)_4]PF_6$, DCE, 70 °C; (ii) EDTA, NH_3, 43% yield
d) (i) **5** (30 equiv), $PdCl_2(MeCN)$, *i*-Pr_2NH, CuI, I_2, C_6H_6, r.t., ~10% yield **9**
e) (i) **2**/**3** (5 equiv), $[Cu(MeCN)_4]PF_6$, DCE, 70 °C; (ii) EDTA, NH_3, ~40% yield **10**

Significance: A synthesis of [3]rotaxanes is described, where two molecules are threaded through the rings of macrocycle **1**. An active template methodology is used, in which a metal ion acts as both template and catalyst for the interlocking processes.

Comment: Two threads of the same kind (**6**) or two different molecules (**8**, **10**) are employed. A copper(I)-catalyzed click reaction and a palladium(II)-catalyzed alkyne homocoupling are used; the metal coordinates to the upper hoop of the macrocycle.

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Category

Synthesis of Materials and Unnatural Products

Key words

rotaxanes

template reaction

macrocycles