

Abstract:

The synthesis of large, shape-persistent, trimeric macrocycles comprising helical units was investigated. Since the previous synthetic approaches studied in our group failed (mainly alkene and alkyne ring closing metathesis), a more robust route toward the desired macrocycle was sought. A sequential construction of an acyclic trimer employing cross-coupling reactions was expected to overcome the unwanted oligomerization encountered in the case of the earlier metathesis approaches. The closure of the trimer would be accomplished *via* the intramolecular McMurry reaction or alkene metathesis. An optimized synthesis of the solubilized dibenzo[5]helicene and its derivatives was developed. These compounds served as proposed building blocks of the acyclic trimer. Both Heck and Suzuki-Miyaura couplings used for the preparation of the acyclic trimers provided mixtures containing the desired trimers but the Suzuki-Miyaura reaction proved to be much more efficient. However, the solubility of the trimers turned out to be very low, making the separation of the pure products from the mixture impossible and seriously limiting the further synthetic progress. Only alkene metathesis of the corresponding divinyltrimer was accomplished and despite a low solubility of the starting material, the MS analysis of the resulting mixture confirmed a preferential formation of the desired trimeric macrocycle. Despite the encountered problems, the achieved results proved the synthetic concept to be correct and left a room for further development in the project.

Key words:

Shape persistent macrocycles, dibenzohelicenes, Heck reaction, Suzuki-Miyaura reaction, alkene metathesis, McMurry reaction.