

A New Synthesis of Tetrakis(C-methyl)octakis(hydroxyethyl)calix[4]-resorcinarene *via* an Ethoxy-Tethered Trimethylsiloxy Precursor

Ion Neda, Tjark Siedentop, Alexander Vollbrecht, Holger Thönnessen, Peter G. Jones, Reinhard Schmutzler*

Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, D-38023 Braunschweig, Germany

Dedicated to Professor Achim Müller on the occasion of his 60th birthday

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Calix[4]resorcinarenes, Conformational Isomers, Spacer Groups, X-Ray Data

The octakis(trimethylsiloxy)calix[4]resorcinarenes, **3** and **4**, were synthesized by reaction of **1** and its tetrabromo derivative **2** with hexamethyldisilazane, and were found to exhibit dynamic behaviour in solution. Temperature-dependent NMR investigations confirmed the presence of at least two conformational isomers of **3** in solution. The conformation of **3** in the solid state was determined by an X-ray crystal structure analysis; the calixarene displays a boat conformation.

The introduction of the ethoxy group as a spacer into the molecule of **1** was effected via its reaction with ethyl bromoacetate and subsequent reduction with lithium aluminium hydride forming the ethoxy-tethered C-methylcalix[4]resorcinarene **6** in an impure state. Reaction of crude **6** with hexamethyldisilazane furnished the ethoxy-tethered octakis(trimethylsiloxy)calix[4]resorcinarene **7**; its hydrolysis led to pure **6**.

* Reprint requests to R. Schmutzler.