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Synthesis and structures of 1,3,1′,3′-tetrabenzyl-2,2′-biimidazolidinylidenes (electron-rich alkenes), their aminal intermediates and their degradation products

(Note: The full text of this document is currently only available in the PDF Version)

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Abstract

Benzyl (R) substituted enetetramines 9 and 3 have been studied. From $HNR(CH_2)_2NRH$ and $CH(NMe_2)_2OBu^t$ or $CH(OMe)_2NMe_2$, two new intermediates along the pathway to 9, namely the orthoamide 11 and the bis(orthoamide) 12 were isolated. Each of 11 and 12 was converted into 9 by refluxing in toluene. Photolysis of 9 yielded the isomer 10, while thermolysis of 9 gave the di(debenzylated) product 1,1′-dibenzyl-2,2′-biimidazoline 13. A route to 3 (R = R′ = CH_2Ph) similar to those used for 9, involving the condensation of 1,2-$C_6H_4[N(R)H]_2$ with $CH(OMe)_2NMe_2$, or the reaction between 1,3-dibenzylbenzimidazolium chloride 8 (X = Cl) and NaH, did not give the expected enetetramine 3 (the dibenzo-analogue of 9), the bis(debenzylated) product 15 being obtained instead. Heating the orthoamide 1,2-R[illegible]$NC_6H_4N(R)C$[illegible](H)NMe_2, prepared from $CH(NMe_2)_2OBu^t$ and 1,2-$C_6H_4[N(H)R]_2$, also gave 15. The reactions of S_8, PhNCS or KOH with a mixture of 8 (X = Cl) and NaH gave 17, 18 or 19, respectively, consistent with the transient formation in each reaction of the tetrabenzylenetetramine 3 (R = R′ = CH_2Ph). The molecular structure of each of the crystalline compounds 10, 11, 12 and 13 was established by X-ray diffraction.