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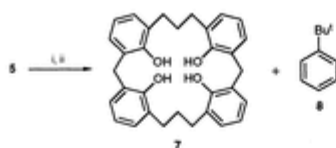
[Journal of the Chemical Society, Chemical Communications](#)

Additions and corrections

[Raphael C. Mordi](#), [Roy Fields](#) and [John Dwyer](#)

Abstract

The first page of this article is displayed as the abstract.



Scheme 3 Reagents: i, $\text{AlCl}_3\text{-MeNO}_2$, benzene (80%)

of calix[4]arene (65.7 kJ mol^{-1}).¹⁰ This is attributed to the increase of ring size by the introduction of the two propane-bridges into the two methylene bridges of calix[4]arene.

It was also found that below -40°C , the singlet signal of the phenolic-OH at δ 9.40 splits into two singlets at δ 9.15 and 10.12. This phenomenon seems to be attributable to the formation of two sets of non-equivalent phenolic-OHs these are formed because the conformational fluctuation of the cyclophane ring is frozen below this temperature by the intramolecular hydrogen bonding between the two OH groups substituted on the benzene rings opposite to each other as shown in Fig. 1. The estimated free energy for fluctuation is 43.9 kJ mol^{-1} ($T_c = -40^\circ\text{C}$, $\Delta\nu = 261.72 \text{ Hz}$).

From the dynamic $^1\text{H NMR}$ studies and considering the Corey-Pauling-Koltun (CPK) model of macrocycle 5, it is concluded that below 0°C the conformation of macrocycle 5 would be expected to be the 'flattened 1,3-alternate form' owing to intramolecular hydrogen bonding between the OH groups below 0°C .

On the other hand, in the spectra of tetramethoxy derivative 6,§ which was prepared by methylation of 5 with MeI (Scheme

2), protons of *tert*-butyl and methoxy groups and methylene bridges appeared each as singlets even below -60°C . This indicates a much more flexible structure for 6 than macrocycle 5. It is concluded that the calix[4]arene-like intramolecular hydrogen bonds can fix the conformation of tetrahydro[3.1.3.1]metacyclophane 5.

It was also found that the $\text{AlCl}_3\text{-MeNO}_2$ catalysed *trans-tert*-butylation of 5 in benzene gave the desired tetrahydro[3.1.3.1]metacyclophane 7[†] in 80% yield along with *tert*-butylbenzene 8. However, using toluene as an acceptor for the *tert*-butyl groups failed. In this case ring cleavage reactions due to the transbenzylation occurred rather than *trans-tert*-butylation.

In conclusion, the propane-bridged metacyclophanes 5 and 7 could be useful sources of new types of host compounds.

Received, 17th February 1992; Com. 2000818A

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§ 6: Colourless prisms (benzene) m.p. $>300^\circ\text{C}$; $^1\text{H NMR}$ (CDCl_3) δ 1.214 (36 H, s), 1.72-1.83 (4 H, m), 2.53 (8 H, t, J 7.3 Hz), 3.14 (12 H, s), 3.82 (4 H, s), 6.96 (4 H, d, J 2.4 Hz) and 7.01 (4 H, d, J 2.4 Hz); m/z : 761 (M^+).

† 7: Colourless prisms (hexane); m.p. $170\text{-}175^\circ\text{C}$; IR (KBr) ν_{max} cm^{-1} 3529 and 3485; $^1\text{H NMR}$ (CDCl_3) δ 1.86 (4 H, br s), 2.90 (8 H, br s), 4.07 (4 H, s), 6.80 (4 H, t, J 7.3 Hz), 6.96 (4 H, dd, J 1.5 and 7.3 Hz), 7.15 (4 H, dd, J 1.5 and 7.3 Hz) and 9.50 (4 H, s).

Additions and Corrections

Gasoline Range Chemicals from Zeolite-catalysed Thermal Degradation of Polypropylene

Raphael C. Mordl, Roy Fields and John Dwyer

J. Chem. Soc., Chem. Commun., 1992, 374.

This communication included the comment 'for a given zeolite, as the Si:Al ratio increases the relative catalytic activity increases even though the activity per acid site remains constant at higher Si:Al. Therefore, for our degradation reactions H-ZSM-5 (Si:Al = 17) would be expected to have higher activity than H-mordenite (Si:Al = 8)'.

Whereas it is clear that for aluminium-rich zeolites activity per site increases as Si:Al increases this is not the case for zeolite frameworks with Si:Al > 7 . Consequently, the differences in observed activity for H-ZSM-5 and H-mordenite probably represent differences in diffusive restrictions, and in rates of catalyst reactivation, for the two catalysts rather than differences in acid-site activity.

Earlier work at UMIST in this area should have been referred to: see G. N. Folefoc, MSc Diss., UMIST, 1981; H. Akpan, MSc Diss., UMIST, 1982; H. A. Masheye, MSc Diss., UMIST, 1983.

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Citation: *J. Chem. Soc., Chem. Commun.*, 1992,0, 862-862

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