07-Crystallography of Organometallic and Coordination Compounds

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ORGANIC INCLUSION COMPOUNDS: RELATING STRUCTURE TO THERMAL STABILITY.
By L. R. Nassimbeni,
Department of Chemistry, University of Cape Town, Rondebosch, 7700, South Africa.

WHEN a suitable host compound H is dissolved in a volatile guest G or is exposed to its vapour, it forms an inclusion compound H\(_2\)G (β-phase). The latter may decompose in one of several ways. It may lose the guest and the host may revert to its non-porous α-phase:

\[ \text{H}_2\text{G}(\beta) \rightarrow \text{H}(\alpha) + n\text{G} \uparrow \]  

Alternatively, the host may lose the guest but retain its structural integrity, displaying the empty cage, or β\(_0\) structure:

\[ \text{H}_2\text{G}(\beta) \rightarrow \text{H}(\beta_0) + n\text{G} \uparrow \]  

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Thirdly, it may lose only part of the guest and form a new γ phase:

\[ \text{H}_2\text{G}(\alpha) \rightarrow \text{H}_2\text{G}(\gamma) + (n-m)\text{G} \uparrow , n > m \]

We have synthesized a variety of inclusion compounds of crowded hydrox hosts which entrapped volatile guests, and have elucidated their crystal structures. Their thermal decompositions have been studied using Thermal Gravimetry (TG) and Differential Scanning Calorimetry (DSC). The vapour pressures of these compounds has been measured over a range of temperatures, yielding enthalpy values for the reactions. Phase changes during decomposition have been monitored by X-Ray powder diffraction. The kinetics of inclusion have been measured by subjecting the α-phase of the host to the vapour of the guest and recording the gain in mass under varying conditions of temperature and pressure.

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THE STRUCTURES OF CALIXARENES.

G. Ferguson & J.F. Gallagher, University of Guelph, Ontario, Canada. M. A. McKervey, Queen's University, Belfast, Northern Ireland and V. Böhm, Johannes Gutenberg Universität Mainz, Germany.

We are currently studying the molecular structures of calix[4]-calix[5]- and calix[6]-arenes and their metal complexes. Until recently, there have been few structural studies reported for calix[3]-arenes, mainly due to difficulties with synthesis and purification. Several structures will be described including the following:

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Calixarenes represent an interesting family of structures which exhibit characteristic features of cation receptors, carriers, etc. Treatment of (E)-2,4-dimethoxyxycinnamic acid isopropyl ester with BF\(_3\)-E\(_2\)O in CHCl\(_3\) at room temperature afforded various C-alkylcalix[4]-resorcinarenes (Botta et al., 1992), which differ for their conformation. Crystals of isomer C and D were obtained by slow evaporation of CHCl\(_3\)-C\(_2\)H\(_5\)OH mixtures at room temperature. Both isomers are nearly space group P1, with Z=2. Isomer C has