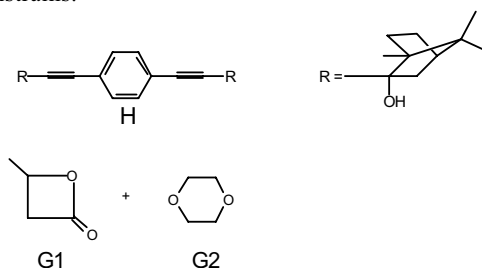


[s9.m3.p11] Enantioselectivity in Chlathrates. A Ternary Crystal of a Wheel-and-Axle Host with two Guests: All Three Molecules Disordered. P. Bombicz^{1,2}, I. Csöreg², M. Czugler¹, A. Kálmán¹, E. Weber³ 1. Institute of Chemistry, Chem. Res. Center, Hung. Acad. of Sciences, Budapest POB 17. H-1525 Hungary 2. Structural Chemistry, Arrhenius Laboratory, Stockholm University, Stockholm S106 91, Sweden. 3. Institut für Org. Chem., TU Bergakademie Freiberg, Leipziger Strasse 29, D-09596 Freiberg, Germany.

Keywords: enantioselectivity, disorder, wheel-and-axle host.

Bulky hosts composed of natural chiral compounds may be used for enantioselective crystallisation experiments. Hence, series of optically active terpenoid hosts containing camphor head groups and different more or less rigid central units, have been synthesized and tested for enantioselective inclusion of chiral compounds¹. The efficiency of the *e.g.* wheel-and-axle type hosts in the recognition of chiral guests is originated from the fine harmony of intermolecular interactions and spatial constraints.



Crystallisation of the optically active diol host (H) from a dioxane solution of the racemic methyl-β-propiolactone (G1) exhibits selectivity and form a ternary inclusion crystal. The dioxane solvent (G2) is included in the crystal with 1:1:1 H:G1:G2 stoichiometric ratio. The guest molecules have special positions: the ring carbon and oxygen atoms of the lactone are placed on the twofold axis at $z=0.5$, while the dioxane ring is located around the twofold axis at $z=0$. Both guests are disordered, each occupies two disorder sites with equal probability. The camphor head groups of the host are held tight in the crystal, whereas the central phenyl ring occur in two disorder positions, which form the dihedral angle of 28.5° .

In the crystal O-H...O bonds link the host molecules into infinite chains. The methyl-β-propiolactone guest is included in a hydrophobic cage surrounded by camphor head groups. The cage is maintained by repulsive interactions thus, emphasising the importance of spatial requirements in stereoselective inclusion formation.

[1] Korkas P.P.; Weber E.; Czugler, M.; Náray-Szabó G. "New Terpenoid Hosts for Chiral Recognition: Crystal Structure and Molecular Modelling Study of an Inclusion Complex with (S)-(+)-Phenylloxirane." *J. Chem. Soc., Chem. Commun.*, (1995), 2229-2230.

[s9.m3.p12] Structure of a Calix[4]arene by X-ray diffraction. L.A. González-Ramírez¹, F. Santoyo-González², J.D. Martín-Ramos³, J. López-Jaramillo¹, J.M. García-Ruiz¹. 1. Laboratorio de Estudios Cristalográficos, IACT-CSIC-Univ. Granada, Facultad de Ciencias (Campus Fuentenueva), 18002 Granada, Spain. 2. Instituto Biotecnología. Univ. Granada, Fac. Ciencias. 18002 Granada, Spain. 3. Dept. Mineralogía y Petrología. Univ. Granada. Fac. Ciencias. 18002 Granada, Spain. Keywords: supramolecular compounds, crystallization, X-ray diffraction.

The development of supramolecular chemistry has led to a growing interest in the design and synthesis of macrocyclic molecules containing intramolecular cavities¹.

Like crown ethers and cyclodextrins, calixarenes are cavity shaped oligomeric macrocyclic molecules made of phenol units. They offer the advantage of their easy synthesis on a large scale in a simple one-pot procedure from inexpensive starting material^{2,3}.

They are able to form inclusion compounds with a wide variety of organic guest species, such as toluene, ammonium cations and fullerenes³.

In this work the crystallization and structural determination by X-ray diffraction of the 1,10-{5,11,17,23-tetra-*tert*-butyl-calix[4]arene-26,28-dioxy}-4,6-decadiyne was carried out. This compound was obtained from 5,11,17,23-tetra-*tert*-butyl-26,28-dihydroxy-25,27-(4-pentyn-1-yloxy)calix[4]arene by Glaser reaction. The crystallization was carried out by the evaporation method.

The structure has been determined from the diffraction of a single crystal at room temperature in rotating anode. The space group is C2/c with unit cell dimensions $a = 22.987(2)\text{Å}$, $b = 19.206(1)\text{Å}$, $c = 12.201(1)\text{Å}$, and $\beta = 112.400(6)^\circ$. $R = 0.05$ and $Z=4$.

[1] Lehn J.M., Atwood J.L., Davies J.E.D., McNicol D.D. "Comprehensive Supramolecular Chemistry". Vogtle F., Eds.; Pergamon Elsevier Science: Oxford, (1996).

[2] Vicens J. "Calixarenes. A Versatile Class of Macrocyclic Compounds". Böhmer, V., Eds.; Kluwer Academic Publishers: Dordrecht, The Netherlands, (1991).

[3] Gutsche C.D. "Calixarenes." Stoddart J.F., Eds.; Royal Society of Chemistry: Cambridge, (1989).