

The combined weight of the clear and colorless distilled fractions was 29 g (77%). Separation of pure isomers was accomplished by VPC and their identities were confirmed by $^1\text{H NMR}$ (CCl_4): *Z* isomer, δ 6.65 (d, 1 H), 5.10 (d, 1 H), 4.02 (q, 2 H), 1.36 (t, 3 H); *E* isomer, δ 6.78 (d, 1 H), 5.38 (d, 1 H), 3.82 (q, 2 H), 1.31 (t, 3 H); bromoacetaldehyde diethyl acetal, δ 4.60 (t, 1 H), 3.60 (t, 4 H), 3.27 (d, 2 H), 1.19 (t, 6 H).

Acknowledgment. This work was supported in part by a grant from Sigma Xi, The Scientific Research Society. We would like to thank Steve Press for many of the VPC separations.

Registry No. 1, 2983-26-8; (*Z*)-2, 23521-49-5; (*E*)-2, 16339-88-1; 3, 2032-35-1; $\text{C}_2\text{H}_5\text{OC}\equiv\text{CH}$, 927-80-0.

Molecular Receptors. Synthesis and X-ray Crystal Structure of a Calix[4]arene Tetracarbonate-Acetonitrile (1:1) Clathrate

M. Anthony McKervey* and Eileen M. Seward

Department of Chemistry, University College, Cork, Ireland

George Ferguson* and Barbara L. Ruhl

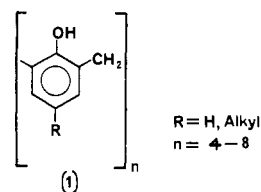
Department of Chemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1

Received January 22, 1986

Treatment of *p*-*tert*-butylcalix[4]arene with ethyl chloroformate and sodium hydride produces tetraethyl *p*-*tert*-butylcalix[4]arene tetracarbonate, which forms a crystalline inclusion complex with acetonitrile. The crystal and molecular structure have been determined by X-ray diffraction. The crystals are tetragonal, space group *P4/ncc* (no. 130) with four molecules in the unit cell of dimensions $a = b = 14.836$ (5) Å, $c = 26.720$ (8) Å. The macrocycle has fourfold crystallographic symmetry, and the benzene rings are inclined at 24.6° to the fourfold axis so as to increase intramolecular *tert*-butyl...*tert*-butyl separations; the acetonitrile molecule lies along the fourfold axis with the methyl group oriented toward the inside of the cavity. The structure was solved by direct methods and refined by full-matrix least-squares calculations to a final *R* value of 0.0754 for 843 reflections having $I > 3\sigma(I)$. Aspects of calixarene receptor topology are discussed.

In recent years there has been growing interest in inclusion phenomena with natural and synthetic unimolecular receptors with much emphasis on the macrocyclic effect.¹ In the natural series, the cyclodextrins²⁻⁷ have been a rich source of information and ideas on guest-host interactions in biomimetic chemistry whereas crown ethers, cryptands, and related cavitands have been the main focus of attention with synthetic receptors.⁸⁻¹⁴ Both solution and solid-state studies with unimolecular receptors have been used extensively to probe the guest-host interactions responsible for binding and transport of ionic and neutral molecules.

The calixarenes¹⁵ constitute an homology of synthetic metacyclophanes 1, produced by phenol-formaldehyde cyclocondensation, whose structures bear a cursory resemblance to the cyclodextrins in as much as each has a single recurring structural subunit with several hydroxyl groups peripherally arranged about a central cavity. Like



the cyclodextrins, the calixarenes have the ability to receive and retain neutral organic molecules, e.g., from solvent of crystallization, by imprisoning the guest inside the discrete central cavity, i.e., true clathrate behavior.¹⁶⁻¹⁹ They also exhibit *multimolecular* inclusion behavior in which the guest species is accommodated in continuous channels or layers within the crystal lattice.²⁰

(1) For a recent comprehensive account of many aspects of inclusion phenomena with natural and synthetic systems including macrocycles, see: *Inclusion Compounds*, Alwood, J. L., Davies, J. E. D., MacNicol, D. D., Eds.; Academic: London, 1984; Vol. 1, 2, 3.

(2) Tabushi, I. *Tetrahedron* 1984, 40, 296.

(3) Tabushi, I. *Acc. Chem. Res.* 1982, 15, 66.

(4) Bender, M. L.; Komiyama, M. *Cyclodextrin Chemistry*; Springer-Verlag: New York, 1978.

(5) Breslow, R.; Doherty, J. B.; Guillot, G.; Lipey, C. *J. Am. Chem. Soc.* 1978, 100, 3227.

(6) Breslow, R.; Bovy, P.; Hersh, C. L. *J. Am. Chem. Soc.* 1980, 102, 2115.

(7) Tabushi, I.; Kuroda, Y. *J. Am. Chem. Soc.* 1984, 106, 4580.

(8) Pedersen, C. J. *J. Am. Chem. Soc.* 1967, 89, 2495.

(9) Cram, D. J.; Cram, J. M. *Acc. Chem. Res.* 1978, 11, 8.

(10) Lehn, J.-M. *Struct. Bonding (Berlin)* 1973, 16, 1.

(11) *Progress in Macrocyclic Chemistry*; Izatt, R. M., Christensen, J. J., Eds.; Wiley: New York, 1979; Vol. 1, pp 1-276.

(12) Stoddart, J. F. *Chem. Soc. Rev.* 1979, 8, 85.

(13) Gokel, G. W. *Macrocyclic Polyether Synthesis*; Springer-Verlag: New York, 1982.

(14) Vögtle, F., Ed. *Top. Curr. Chem.* 1981, 98, 1; 1982, 101, 1; 1984, 121, 1.

(15) For a comprehensive recent review of calixarene chemistry including a critical appraisal of the older literature, see: Gutsche, C. D. *Top. Curr. Chem.* 1984, 123, 1.

(16) Andreetti, G. D.; Ungaro, R.; Pochini, A. *J. Chem. Soc., Chem. Commun.* 1979, 1005.

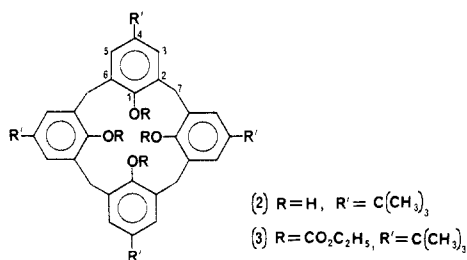
(17) Coruzzi, M.; Andreetti, G. D.; Bocchi, V.; Pochini, A.; Ungaro, R. *J. Chem. Soc., Perkin Trans. 2* 1982, 1133.

(18) Andreetti, G. D.; Pochini, A.; Ungaro, R. *J. Chem. Soc., Perkin Trans. 2* 1983, 1773.

(19) Rizzoli, C.; Andreetti, C. D.; Ungaro, R.; Pochini, A. *J. Mol. Struct.* 1982, 82, 133.

(20) MacNicol, D. D.; Kendrick, J. J.; Wilson, D. R. *Chem. Soc. Rev.* 1979, 7, 65.

Andreetti and his co-workers have conducted a systematic study of inclusion complexes of many of the parent calixarenes, though no simple trends leading to predictions of inclusion behavior have emerged.¹⁶⁻¹⁹ They did conclude, however, that intracavity (clathrate) inclusion only occurs when the calixarene is in the cone conformation, illustrated in **2** for the *p*-*tert*-butyl tetramer. Furthermore,



inclusion of aromatic guests was observed only when the para position of the calixarene carried a *tert*-butyl group, an arrangement believed to provide favorable CH₃... π interaction between the methyl groups and the π system of the guest molecule. The *tert*-butyl groups are unable to bend inwards, thus blocking the entrance to the cavity as appears to be the case with *p*-(1,1,3,3-tetramethylbutyl)-calix[4]arene.¹⁸

As part of a program of study of chemically modified calixarenes²¹ and related crown ether phenols²² and their potential as ion and molecule receptors we have converted the calix[4]-, calix[6]-, and calix[8]arenes into various ester derivatives and have found substantial size-related ion transport ability toward alkali metal cations.²¹ We have now converted the *p*-*tert*-butylcalix[4]arene **2** into tetracarboxylate **3** and report here the X-ray crystal and molecular structure of the 1:1 clathrate formed by **3** and acetonitrile. This is the first crystal structure reported of a clathrate of a nonaromatic guest and a *p*-*tert*-butylcalix[4]arene. It is also the first functionalized calixarene to bind a neutral guest in the true clathrate sense. The acetoxycalixarene derivative reported by Andreetti¹⁹ does form an inclusion complex with acetic acid, but it is of the *multimolecular* type with the guest molecules located in channels in the crystal lattice.

Results and Discussion

Treatment of *p*-*tert*-butylcalix[4]arene **2** with ethyl chloroformate in tetrahydrofuran in the presence of sodium hydride produced tetraethyl *p*-*tert*-butylcalix[4]arene tetracarboxylate **3** as a microcrystalline solid which was not suitable for X-ray analysis. Recrystallisation from aqueous ethanol-acetonitrile gave the 1:1 clathrate **3**·CH₃CN in crystalline form suitable for X-ray structure determination.

It has already been established¹⁶ by X-ray diffraction that the tetrameric phenol **2**, the precursor of the tetracarboxylate **3**, possesses a perfect cone conformation of C_{4v} symmetry; the molecule has a symmetrical cavity extending from the *tert*-butyl groups to the hydroxyl groups. The rigidity of this symmetrical arrangement is due to extensive intramolecular hydrogen bonding between the four hydroxyl groups. It is also known that functionalization of the hydroxyl groups of the tetramer can destroy the perfect cone conformation²³ and replace it with a less symmetrical arrangement, as for example in the tetra-

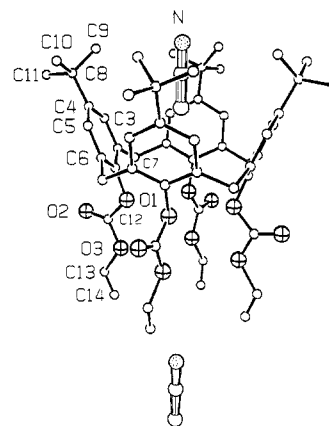


Figure 1. A review of **3**·CH₃CN showing the crystallographic numbering scheme. Two symmetry-related acetonitrile molecules shown lie on the crystallographic fourfold axis; the nitrogen atoms are stippled.

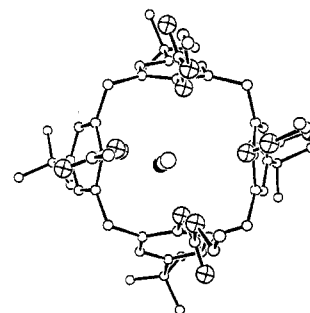


Figure 2. A view into the cavity of **3**·CH₃CN almost along the crystallographic fourfold axis. The CH₃CN is within the cavity, with the methyl carbon toward the viewer.

acetate.¹⁹ However, our X-ray analysis of **3**·CH₃CN shows that the tetracarboxylate derivative is the first to maintain the perfect cone conformation exhibited by the parent phenol.²⁴

The calixarene tetracarboxylate **3** has fourfold crystallographic symmetry and mean plane calculations show that the unique benzene ring is inclined at 24.6° to the fourfold axis; the tilting of the ring is such as to increase intramolecular *tert*-butyl...*tert*-butyl separations. Two views of the calixarene are shown in Figures 1 and 2.

The pendant OCOEt groups are maximally extended with torsion angles in the O(1)-C(12)-O(3)-C(13)-C(14) side chain close to 180°. The orientation of the side chain with respect to its benzene ring is defined by the C(12)-O(1)-C(1)-C(2) torsion angle of -75° and each carbonyl group is directed away from the calixarene cavity. The shortest intramolecular O...O contacts are between adjacent chains (O(1)...O(1) 3.15 Å, O(3)...O(3) 3.55 Å).

An unexpected result of our structure analysis was the location of the acetonitrile molecule within the calixarene cavity (Figures 1 and 2) with the nitrogen atom directed exo. The decision as to which terminal atom was nitrogen and which was carbon was made from electron density maxima and from bond lengths. Geometry calculations show that there is clearly room for such a linear molecule in the cavity. The shortest intracavity calixarene...acetonitrile distances are 3.80 Å (between aromatic carbon C(1) and acetonitrile methyl carbon) and 3.96 Å (between *tert*-butyl carbon C(9) and the nitrogen atom). Slightly

(21) McKervey, M. A.; Seward, E. M.; Ferguson, G.; Ruhl, B.; Harris, S. *J. Chem. Soc., Chem. Commun.* **1985**, 388.

(22) Browne, C. M.; Ferguson, G.; McKervey, M. A.; Mulholland, D. L.; O'Connor, T.; Parvez, M. *J. Am. Chem. Soc.* **1985**, *107*, 2703.

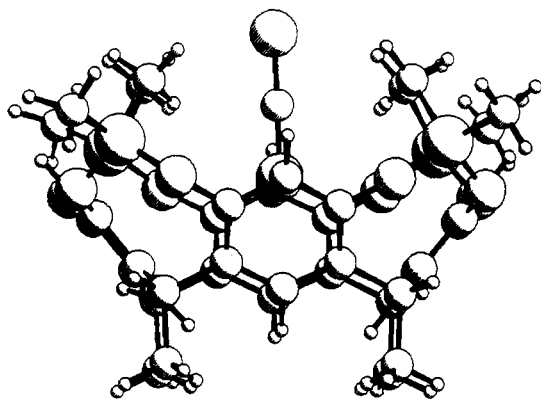
(23) Gutsche, C. D.; Dhawan, B.; Levine, J. A.; No, K. H.; Bauer, L. *J. Tetrahedron* **1983**, *39*, 409.

(24) It has recently been claimed on the basis of NMR data that *tert*-butylcalix[4]arene acetate also possesses a perfect cone conformation in solution; Arduini, A.; Pochini, A.; Reverberi, S.; Ungaro, R. *J. Chem. Soc., Chem. Commun.* **1984**, 981.

shorter C...N distances (3.86 Å) are found as *intercomplex* distances between nitrogen and the terminal carbonate methyl groups of a related calixarene half a unit cell distance along the fourfold axis (symmetry operation $1/2 - y, 1/2 - x, 1/2 - z$). All of these separations correspond to or are greater than normal van der Waals contacts.

The dimensions of the calixarene (e.g., $C_{sp^2}-C_{sp^2}$ 1.390–1.411 (8) Å, $C_{sp^2}-C_{sp^3}$ 1.502–1.587 (8) Å) are for the most part in accord with anticipated²⁵ values (Table 1, supplementary material) there is some disorder/large thermal motion associated with ester atoms C(13) and C(14).

The new calixarene–acetonitrile clathrate reported here shows remarkable similarity to the cavitand–carbon disulfide clathrate 4 reported recently by Cram and his co-workers,²⁶ despite the large differences in conformational mobility between the guest-free hosts. Whereas Cram's



(4)

compound is conformationally rigid by virtue of the four additional bridging silicon groups ortho to the four bridging methylene groups, our compound possesses singly bridging methylene groups only which permit some rotation about the short axis of each benzene ring. Thus in Cram's compound the guest CS₂ molecule is located within the rigidly preorganized host. Interestingly, Cram's compound also forms clathrates with oxygen and with methylacetylene. The latter clathrate has the methyl group located in the exo position with respect to the cavity whereas in our calixarene–acetonitrile clathrate the guest methyl group occupies the endo position.

These studies should facilitate the design and construction of host molecules with specific receptor activity toward selected guests.

Experimental Section

5,11,17,23-Tetra-*tert*-butyl-25,26,27,28-tetrakis(ethoxycarbonyloxy)calix[4]arene (3). *p-tert*-Butylcalix[4]arene 1 (0.648 g, 1 mmol), sodium hydride (0.696 g, 16 mmol), and ethyl chloroformate (3.1 mL, 32 mmol) were heated under reflux in dry tetrahydrofuran (50 mL) under nitrogen for 3 days. Excess sodium hydride was removed by the dropwise addition of water, and the solvent and excess ethyl formate were then removed at reduced pressure. The residue was taken up in chloroform, washed with water, and dried over magnesium sulfate. Removal of the solvent followed crystallization of the residue from dichloromethane–ethanol gave the tetracarboxylate 3 (60%) as a colorless microcrystalline powder: mp 298–300 °C; IR (KBr) 1740 cm⁻¹; ¹H NMR (400 MHz, CDCl₃, Me₄Si, 20 °C) δ 1.09 (s, 36 H), 1.46 (t, 12 H), 3.29 (d, 4 H, H_B, J_{AB} = 12 Hz), 3.92 (d, 4 H, H_A), 4.38 (q, 8 H),

6.93 (s, 8 H, Ar); ¹³C NMR (KMH₂, CDCl₃, Me₄Si) δ 14.49 (q), 30.80 (s), 31.25 (q), 34.05 (t), 64.06 (t), 125.33 (d), 133.00 (s), 144.11 (s), 147.68 (s), 155.35 (s). Anal. Calcd for C₅₆H₇₂O₁₂: C, 71.79; H, 7.69. Found: C, 72.01; H, 7.86. Slow evaporation of an acetonitrile–water–ethanol (1:1:1) solution of 3 gave the 1:1 clathrate 3·CH₃CN as colorless tetragonal crystals suitable for X-ray diffraction analysis. The *wet* crystal was coated with epoxy resin within 15 s of removal from the crystallization medium and mounted in the diffractometer. Crystals that had been allowed to dry were found to lose acetonitrile rapidly as revealed by NMR and infrared analysis.

Crystal Data: C₅₆H₇₂O₁₂·CH₃CN·1.6H₂O; M_r 1007.1, tetragonal, *a* = *b* = 14.836 (5) Å, *c* = 26.720 (8) Å, *V* = 5881 (6) Å³, *Z* = 4, *D_c* = 1.14 g/cm³, Mo Kα radiation, λ = 0.71069 Å, μ(Mo Kα) = 0.69 cm⁻¹; space group *P4/ncc* (no. 130) from systematic absences (*hk0* when *h* + *k* = 2*n* + 1; *0kl* when *l* = 2*n* + 1; *hhl* when *l* = 2*n* + 1) which together with the *4/mmm* Laue symmetry of the reciprocal lattice uniquely determine the space group.

All data were collected on an Enraf-Nonius Cad-4 diffractometer using graphite monochromatized Mo Kα radiation. Accurate cell parameters were obtained by a least-squares refinement of the setting angles of 25 reflections, with θ in the range 10–15°; the values obtained were consistent with a tetragonal system and a rapid scan through a 2–5° sphere of all reflections showed that the reciprocal lattice had *4/mmm* symmetry.

The space group has 16 general positions and with *Z* = 4 the molecule would have (in the absence of disorder) either $\bar{4}$, 4, or 222 crystallographically imposed symmetry. Our analysis determines that the calixarene has fourfold symmetry.

Intensity data were collected in the range 2 < θ < 20° by using the ω/2θ technique with a ω scan width of (0.60 + 0.35 tan θ). The intensities of the three standard reflections were monitored every 2 h of exposure time and showed no significant variation. Of the 1376 reflections measured, 843 were used in the structure solution and refinement. Data were corrected for Lorentz and polarization factors, but correction for absorption in this light atom structure was not considered necessary. The structure was solved by the direct phasing procedures of MULTAN 80²⁷ and by subsequent structure-factor and electron-density calculations which revealed that the unique benzene ring of the calixarene lay about a site with fourfold crystallographic symmetry. The electron-density maps also showed that additional unexpected electron density maxima were present in distinct and different parts of the unit cell. One set of maxima consisted of three well-resolved peaks lying on the same fourfold axis as the calixarene; after carefully contouring three-dimensional Fourier and difference-density maps we deduced (from the peak densities and bond lengths) that these represented an acetonitrile molecule of recrystallization lying within the calixarene cavity. We were unable to grow crystals of the carbonate unless acetonitrile was present in the recrystallization solvent mixture. The other set of diffuse peaks was clustered around a site of 4 symmetry and lay between calixarene molecules. Despite many attempts, we were unable to fit any chemically sensible molecule to this set of maxima which persisted in every electron density or difference Fourier map calculated after various rounds of isotropic full-matrix least-squares refinement. In the end we assigned these maxima to be disordered water of crystallization.

Difference maps also showed the presence of maxima consistent with the aromatic, methylene, and *tert*-butyl hydrogen atoms but not for the hydrogens of the terminal methyl groups of the carbonate function of the calixarene. The methyl hydrogen atoms of the acetonitrile molecule are disordered as this molecule has crystallographic fourfold symmetry. In subsequent refinement cycles the aromatic, methylene, and *tert*-butyl hydrogen atoms were positioned on geometrical grounds (C–H 1.10 Å) and included in structure factor calculations but not refined. For the final rounds of block diagonal refinement, the C, O, and N atoms of the calixarene and acetonitrile were allowed anisotropic motion, the water molecules were allowed isotropic vibration, and allowance was made for hydrogen as noted above. Refinement con-

(25) Winter, W.; Butlers, T.; Ricker, A.; Butsugan, Y. Z. *Naturforsch. B: Anorg. Chem., Org. Chem.* 1982, 37B, 855.

(26) Cram, D. J.; Stewart, K. D.; Goldberg, I.; Trueblood, K. N. *J. Am. Chem. Soc.* 1986, 107, 2574.

(27) Main, P.; Fiske, S. J.; Hull, S. E.; Lessinger, L.; Germain, G.; Declercq, J.-P.; Woolfson, M. M. MULTAN80, A system of computer programs for the automatic solution of crystal structures from X-ray diffraction data, Universities of York, England, and Louvain, Belgium, 1980.

verged with $R = 0.075$ and $R_w = 0.056$. A final difference map had no chemically significant features. In the refinement cycles weights were derived from counting statistics, and scattering factor data were taken from ref 28.

Acknowledgment. We thank the Department of Education (Ireland) for a postgraduate award to E.M.S. and

(28) *International Tables for X-ray Crystallography*; Kynoch: Birmingham, 1974; Vol. IV. (Present distributor: D. Reidel, Dordrecht.)

N.S.E.R.C. (Canada) for continuing financial support via operating grants.

Registry No. 1, 60705-62-6; 3, 103240-05-7; 3-CH₃CN, 103258-02-2.

Supplementary Material Available: Details of molecular geometry (Table 1), final atomic coordinates (Table 2), tables of calculated hydrogen coordinates (Table 3), and thermal parameters (Table 4) (5 pages). Ordering information is given on any current masthead page.

Trapping of the 6,6-Dimethylbicyclo[3.1.1]hept-2-yl Free Radical by S_H2 Reaction upon Peracid

Jacques Fossey, Daniel Lefort,* and Janine Sorba

GR12, CNRS, BP28, 94320 Thiais, France

Received March 17, 1986

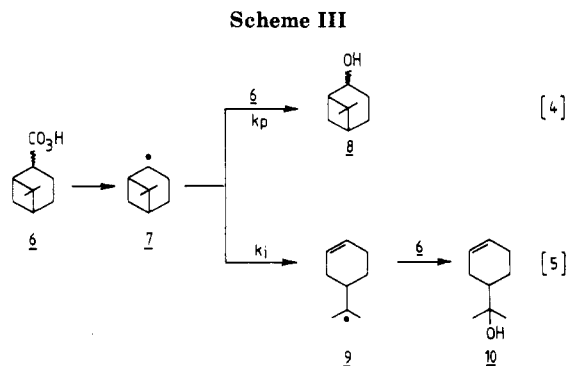
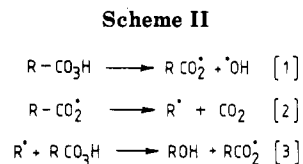
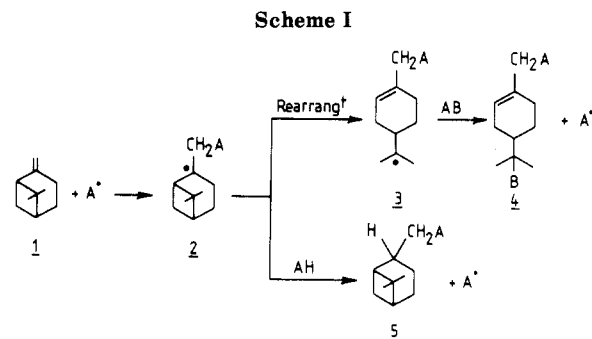
The free radical 6,6-dimethylbicyclo[3.1.1]hept-2-yl (**7**) has been obtained by decarboxylation of *cis*- or *trans*-6,6-dimethylbicyclo[3.1.1]heptane-2-peroxycarboxylic acids (**6a** and **6b**). **7** trapped by reaction with the initial peracid gave a stereochemical mixture of α - and β -nopinol (**8a** and **8b**). The ratio **8b**/**8a** is around 12, independent of the initial peracid **6a** or **6b** and its initial concentration. This value is mainly due to the steric effect of one of the methyl groups branched on C₇ in **7**. The structure of **7** is discussed. By a competitive reaction **7** undergoes ring opening to afford 2-(3-cyclohexenyl)-2-propyl free radical (**9**) which by reaction upon peracid leads to 2-(3-cyclohexenyl)-2-propanol (**10**). **7** was successfully trapped because its reaction with peracid is rapid enough. The ratio of the two alcohols **8**/**10** leads to an estimation of $1 \times 10^6 \text{ M}^{-1} \text{ s}^{-1}$ for the rate constant of the reaction of **7** with peracids.

In all examples given in the literature,¹ radicals of type **2** rearrange into radicals of type **3** by opening of the cyclobutane ring and give products of type **4** (see Scheme I).

Nevertheless, the unrearranged product **5** was obtained once² when **2** was generated in the presence of thiols: in these conditions H transfer from the thiols by **2** competes with the opening of the cyclobutane ring.

In preceding works³ we had shown that peracids, RCO₃H, in boiling hydrocarbon solution decarboxylate rapidly into the corresponding alcohols, ROH, through the chain mechanism given in Scheme II.

The rate-determining step of this chain mechanism is step 3. Its activation energy was estimated to be around 4 kcal/mol for the bicyclo[2.2.1]hept-1-yl³ radical. For cyclohexyl-type radicals the activation energy is still less than 4 kcal/mol.^{3b} One more example of the velocity of reaction 3 comes from the retention of configuration observed with the *cis*- and *trans*-decahydronaphthalen-9-yl radicals.⁴ The decompositions of peracids **6a** and **6b** have been undertaken in order to study the behavior of the radical **7** in these conditions. The different steps of the reaction are described on Scheme III.



(1) (a) Cazaux, M.; Maillard, B.; Lalande, R. *C. R. Seances Acad. Sci., Ser. C* 1972, 275, 1133. (b) Gaiffe, A.; Castanet, J. *C. R. Seances Acad. Sci., Ser. C* 1971, 272, 96. (c) Cazaux, M.; Lalande, R. *Bull. Soc. Chim. Fr.* 1971, 461. (d) Filliatre, C.; Villenave, J.; Prevot, J. *Bull. Soc. Chim. Fr.* 1979, 473. (e) Villenave, J.; Filliatre, C.; Prevot, J. *Bull. Soc. Chim. Fr.* 1979, 479. (f) Cazaux, M.; Lalande, R. *Bull. Soc. Chim. Fr.* 1972, 1887. (g) Paskoff, B.; Cazaux, M.; Lalande, R. *Bull. Soc. Chim. Fr.* 1970, 624. (h) Cazaux, M.; Lalande, R. *Bull. Soc. Chim. Fr.* 1972, 1984. (i) Lallemand, J. *Tetrahedron Lett.* 1975, 1217. (j) Kang, Y.; Kice, J. *J. Org. Chem.* 1984, 49, 1507. (k) Battisteux, D.; Haseldine, D. *Synth. Commun.* 1984, 14, 293. (l) Brace, N. *J. Org. Chem.* 1962, 27, 3027.

(2) Claisse, J.; Davies, D.; Parfitt, L. *J. Chem. Soc. C* 1970, 258.

(3) (a) Fossey, J.; Lefort, D. *Tetrahedron* 1980, 36, 1023. (b) Lefort, D.; Fossey, J.; Gruselle, M.; Nedelec, J. Y.; Sorba, J. *Tetrahedron* 1985, 41, 4237.

(4) Fossey, J.; Lefort, D.; Massoudi, H.; Nedelec, J. Y.; Sorba, J. *Can. J. Chem.* 1985, 63, 678.

This paper reports the first trapping of **7**, by peracid, before opening of the cyclobutane ring. From the ratio of the two alcohol epimers **8a** and **8b**, information concerning the structure of **7** has been obtained. From the relative