

The thermal formation of cyclic peroxides from *cis,cis* dienes and triplet oxygen has precedence in the literature. Barton et al.¹⁰ have suggested that the spin barrier is overcome by reversible formation of a tetroxide from the primarily produced triplet diradical and decomposition of the tetroxide to two molecules of singlet diradical which cyclize to the endoperoxide.

Experimental Section

General Methods. ¹H and ¹³C NMR spectra were obtained on Varian HA-100 and JEOL FX 60 spectrometers, respectively, using CDCl₃ or C₆D₆ as solvents and Me₄Si as an internal standard. Mass spectral measurements were recorded on an AEI MS 902 spectrometer and the GC/MS results were obtained with a Carlo Erba gas chromatograph combined with a Micromass 7070 F mass spectrometer.

Melting points are uncorrected. TLC analyses were carried out on silica gel plates, mostly with petroleum ether/ethyl acetate as eluent. Authentic samples of 4 and 5 were purchased from Ventron and Aldrich, respectively.

2,2-Dimethyl-1,3-diphenylisoindene (1). The reaction vessel consisted of a two-necked 25-mL reaction flask fitted with two rotatable side arms, one connected to a two-necked 10-mL addition flask (from which solid 2 could be added to the reaction flask) and the other connected to a two-necked 10-mL conical receiver flask via a glass filter. An N₂ inlet and an outlet to a vacuum pump completed the reaction vessel.

The reaction flask was loaded under N₂ with Hg (19 g), a piece of Li (0.5–1 g) and a Teflon-coated stirring bar. After 50 min of stirring, the excess Li was removed with a pair of tweezers and 2.5 mL of solvent (C₆H₆ or C₆D₆) was added to the lithium amalgam. The system was degassed four times and crystalline 2¹ (400 mg) was transferred from the addition flask to the reaction flask by rotation of the side arm. After a few minutes the solution turned orange-red. The suspension was stirred for 5 h and then filtered through the glass filter into the receiving flask. For the NMR measurements the solution was filtered directly into the NMR tube (which was connected to the side arm) and then sealed off with a gas burner. When CDCl₃ was used as NMR solvent, this solvent was added under N₂ after evaporation of the benzene: ¹H NMR (C₆D₆) δ 1.14 (s, 6 H), 5.97–6.15 (q, 2 H), 6.65–6.82 (q, 2 H), 7.0–7.3 (m, 10 H); ¹H NMR (CDCl₃) δ 1.25 (s, 6 H), 6.28–6.39 (q, 2 H), 6.74–6.85 (q, 2 H), 7.0–7.3 (m, 10 H); ¹³C NMR (C₆D₆) δ 20.5 (CH₃, J_{CH} = 129 Hz), 56.5 (C₂), 124.2–128.6 (C₄, C₅, C₆, C₇, *o*-, *m*-, and *p*-phenyl), 137.0 and 137.5 (C₈, C₉, and ipso-phenyl), 149.7 (C₁, C₃).

When solutions of 1 were treated with *N*-phenylmaleimide, the orange-red color instantly disappeared and a white solid precipitated. After crystallization from ethanol the Diels–Alder adduct with mp 276–279 °C (lit.⁵ mp 265–266 °C) was obtained; mass spectrum, *m/e* 469.2047 (30%) calcd. for C₃₃H₂₇NO₂ 469.2042), 296.1563 (100%, calcd for C₂₃H₂₀ 296.1565). The base peak is obviously due to a retro-Diels–Alder reaction. A similar treatment with maleic anhydride resulted in the corresponding adduct, mp ca. 350 °C dec (lit.¹ mp 344 °C).

Treatment of the dibromide 2 as described above, but without rigorous drying of the solvent and glass apparatus, resulted in appreciable amounts of the ring-opened ketone 3, identified by NMR, IR and mass spectra. This ketone was isolated by column chromatography (silica gel and petroleum ether/ethyl acetate). The ¹H NMR values were found to be in complete agreement with published values⁶ (both in CDCl₃ and C₆D₆ solutions). The ¹³C NMR (CDCl₃) gave signals for the methyl carbons at δ 21.96 and 23.07; mass spectrum, *m/e* 312 (33%), 297 (100%).

An especially fast conversion of 2 to 3 was found to take place on TLC plates with silica gel.

Treatment of 1 with Air. When air was bubbled through a solution of 1 in C₆D₆ as prepared above, the orange-red solution first turned colorless and then after a few hours yellow with a green-blue fluorescence. The colorless solution gave the following: ¹H NMR (CDCl₃) δ 1.20 (br s, 6 H), 7–8 (m, 14 H); ¹³C NMR

(CDCl₃) δ 21.2 (CH₃), 22.3 (CH₃). The other ¹³C signals were less well resolved but not incompatible with the peroxide structure 6. A peroxide test with the iodide/starch reagent was positive. The TLC analysis of this compound is reported below.

The yellow, fluorescent solution was first analyzed by combined gas chromatography and mass spectrometry which showed the presence of acetone. TLC revealed two further components, identified as 1,3-diphenylisobenzofuran (4) and 1,2-dibenzoylbenzene (5) by comparison with authentic samples. NMR analysis of the crude reaction mixture confirmed the presence of these three products. After more than 24 h of contact with air, ost of 4 had been oxidized to 5.

When solutions containing the colorless compound believed to be the peroxide 6 were analyzed by TLC a band with an *R_f* value between the *R_f* values for 4 and 5 was found. When this band on the TLC plate was exposed to air after evaporation of the solvent, an interesting rapid change of color took place: colorless → bright yellow → colorless. Isolation of the band revealed an almost quantitative conversion to 5. Obviously the same reaction takes place on the TLC plates as in solution.

Registry No. 1, 64836-60-8; 2, 42003-48-5; 3, 18949-20-7; 4, 5471-63-6; 5, 1159-86-0; 6, 78020-04-9; 3a,4,9,9a-tetrahydro-10,10-dimethyl-2,4,9-triphenyl-4,9-methano-1*H*-benz[*f*]isoindole-1,3(2*H*)-dione, 78087-14-6; 3a,4,9,9a-tetrahydro-10,10-dimethyl-4,9-diphenyl-4,9-methanonaphtho[2,3-*c*]furan-1,3-dione, 78020-05-0; *N*-phenyl maleimide, 941-69-5; maleic anhydride, 108-31-6.

Strained Benzene Rings: Preparation and Crystal Structure of a Dithiahexahydro[3.3]paracyclophane, S₂C₁₆H₂₂

P. N. Swepston, S.-T. Lin, A. Hawkins, S. Humphrey, S. Siegel,* and A. W. Cordes*

Department of Chemistry, University of Arkansas, Fayetteville, Arkansas 72701

Received April 17, 1981

Introduction

The most stable conformations of strained molecules such as paracyclophanes may be computed, in principle, by quantum mechanical procedures, but semiempirical molecular mechanics calculations generally are used for such molecules of interest to organic chemists.^{1,2} The required force field parameters are often obtained by reference to observed structures. Accordingly, the structure of 2,11-dithia-4,5,6,7,8,9-hexahydro[3.3]paracyclophane (3), which is clearly strained because it includes a bent benzene ring,³ is of interest because of the variety of chemical linkages it presents. It is the first structurally characterized molecule of the paracyclophane class in which one unit is a cyclohexane ring. One might have supposed that the intramolecular strain would have been relieved through distortions of the saturated cycle; however, only part of the strain is relieved in this way; the cyclohexane moiety has bond distances and angles close to those expected for a typical *cis*-1,4-dialkylcyclohexane.

Compound 3 was prepared in 31% yield from the reaction of α,α' -dichloro-*p*-xylene (1) and *cis*-1,4-bis(mercaptomethyl)cyclohexane (2) in an alcoholic NaOH solution, using the high dilution technique described by Davis.⁴ The *cis*-1,4-bis(mercaptomethyl)cyclohexane was prepared either by hydrolysis of the diisothiuronium salt which was

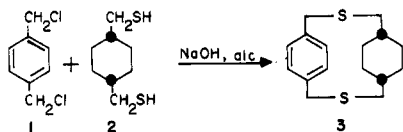
(1) Allinger, N. L.; Tribble, M. T.; Miller, M. A.; Wertz, D. H. *J. Am. Chem. Soc.* 1971, 93, 1637–1648.

(2) Engler, E. M.; Andose, J. D.; von R. Schleyer, P. *J. Am. Chem. Soc.* 1973, 95, 8005–8025.

(3) Cram, D. J.; Cram, J. M. *Acc. Chem. Res.* 1971, 4, 204–213.

(4) Davis, A. C. *Chem. Ind.* 1977, 203–204.

(10) D. H. R. Barton, R. K. Haynes, G. Leclerc, P. D. Magnus, and I. D. Menzies, *J. Chem. Soc., Perkin Trans. 1*, 2055 (1975).



obtained by heating *cis*-bis(bromomethyl)cyclohexane with thiourea or somewhat more directly from the reaction of the ditosylate of *cis*-1,4-bis(hydroxymethyl)cyclohexane with Na_2S and H_2SO_4 in DMF. The later method is the more economical in that the *cis* ditosylate may be prepared easily from the commercially available *cis*,*trans* mixture of 1,4-bis(hydroxymethyl)cyclohexane.

Complete structural proof is provided by a single-crystal X-ray structure determination. Figure 1a is an ORTEP drawing⁵ of the refined model; it shows that the benzene ring is directly above and tipped 25° with respect to the cyclohexane ring. While there are no highly unusual bond distances or angles in the molecule, a number of parameters reflected the internal strain. As a result of the intramolecular crowding, the axial hydrogen atom H(C7) and the methylene hydrogen atom H(C2) are only 2.59–2.64 (6) Å from the benzene carbon atoms; these distances are slightly shorter than the sum of the van der Waal's radii.⁶ Also as a result of this strain, two of the methylene C–C–S bond angles [114.0 (3°) at C2 and 117.6 (3°) at C9] are larger than typical paraffinic angles,⁷ the bond angles at the sulfur atoms of 102.7 (2°) are 3° larger than that in dimethyl sulfide,⁸ and the cyclohexane ring is somewhat "flattened" from the idealized chair (CCCC torsion angles range from 48.7 to 57.3°). More significantly, the benzene ring and the para groups on the ring are nonplanar, to the extent shown in Figure 1b.

Experimental Section

Proton NMR spectra were measured by using a Varian EM-360 60-MHz spectrometer. Chemical shifts are reported in δ units relative to internal tetramethylsilane. Mass spectra were obtained on a Hitachi RMU-7 double-focusing mass spectrometer at an ionization potential of 60 eV. IR spectra were recorded with a Perkin-Elmer 283. Elemental analyses were performed by Guelph Chemical Laboratories.

Benzene and pyridine were distilled from sodium ketyl and potassium hydroxide, respectively. Dimethylformamide (DMF) was purified by distillation. All solvents were stored over 3A molecular sieves. *cis*-1,4-Bis(hydroxymethyl)cyclohexane, the mixture of 1,4-bis(hydroxymethyl)cyclohexane isomers, α,α' -dichloro-*p*-xylene, and *p*-toluenesulfonyl chloride were obtained from Aldrich Chemical Co. and were used as received.

***cis*-1,4-Bis(bromomethyl)cyclohexane (4).**⁹ *cis*-1,4-Bis(hydroxymethyl)cyclohexane (20 g, 0.14 mol) was added dropwise over 1 h to a stirred solution of PBr_3 (37.7 g, 0.14 mol) in 30 mL of benzene at 15°C . The solution was then refluxed for 75 min, cooled, and poured slowly onto 200 g of crushed ice. The aqueous layer was washed with methylene chloride (3×40 mL), and the methylene chloride extracts were combined with the organic layer and washed first with a saturated solution of NaHCO_3 (3×50 mL) and then with distilled water (3×50 mL). The organic layer was dried over MgSO_4 , filtered, and evaporated to an oil which distilled at 121°C (0.1 mm) to give 23.5 g of dibromide 4 (62%).

***cis*-1,4-Bis(amidinithio)methylcyclohexane Dibromide (5).** A mixture of 4 (6.0 g, 0.022 mol), thiourea (3.55 g, 0.044 mol), and 20 mL of 95% ethanol was refluxed for 5 h. Half of the solvent

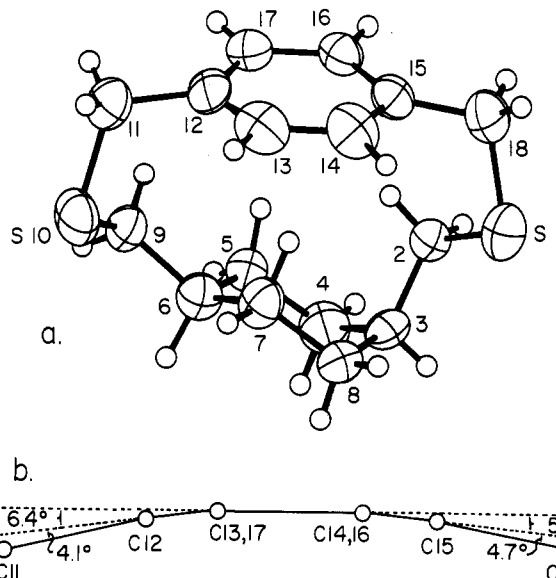


Figure 1. (a) ORTEP drawing of dithiahexahydro[3.3]paracyclophane. The thermal ellipsoids for hydrogen are reduced for clarity. (b) the angles of the bent benzene: 5.6 (6°) and 6.4 (6°) are the angles formed by the C13–C14–C16–C17 plane with the C14–C15–C16 plane and the C12–C13–C17 plane, and the 4.7 (5°) and 4.1 (6°) angles are formed between the para C–C bonds and the latter planes.

was removed by evacuation, the remaining solution was chilled in ice water, and the solid that separated was filtered and washed with 20 mL of cold 95% ethanol. The product obtained was 7.42 g (79%) of a white solid, 5: $^1\text{H NMR}$ (CF_3COOH) δ 7.4 (br, 8 H), 3.2 (d, $J = 20$ Hz, 4 H), and 1.7 (br, 10 H); ν_{max} (KBr) 3260, 1645, 1625, 1440, 690 and 150 cm^{-1} .

***cis*-1,4-Bis(mercaptomethyl)cyclohexane (2). Method 1.** A mixture of 5 (5.09 g, 1.45 mmol), 10 g (15 mmol) of 85% aqueous KOH, and 40 mL of H_2O was refluxed for 12 h. The solution was cooled, poured into 50 mL of ice water, neutralized with 10% H_2SO_4 , and extracted with diethyl ether (4×30 mL). The extracts were combined, washed with water (2×50 mL), dried over MgSO_4 , filtered, and purified by vacuum distillation at 97°C (0.1 mm) to give 1.34 g (52%) of thiol 2 as a colorless liquid: $^1\text{H NMR}$ (CDCl_3) δ 2.5 (br d, 4 H), 1.52 (br, 10 H), and 1.30 (t, $J = 10$ Hz, 2 H); ν_{max} (film) 2550, 1450, 1425, and 710 cm^{-1} , mass spectrum, m/e 176 (M^+).

Anal. Calcd for $\text{C}_8\text{H}_{16}\text{S}_2$: C, 54.49; H, 9.15; S, 36.36. Found: C, 53.95; H, 9.35; S, 36.68.

Method 2. *p*-Toluenesulfonyl chloride (162 g, 0.84 mol) was added to 1,4-bis(hydroxymethyl)cyclohexane (*cis/trans* = 1/1) (55.3 g, 0.38 mol) in 300 mL of pyridine at 0 – 5°C . The mixture was refrigerated for 18 h and poured into 1000 mL of ice water. The solution was neutralized with HCl to pH 7 and filtered to give a white solid. The solid was extracted with methanol (3×300 mL) and dried by vacuum evaporation to give 55.4 g (31%) of the ditosylate 6 in the form of long, white needles, mp 93 – 95°C (lit.¹⁰ mp 96 – 96.5°C).

A solution of 6 (27.6 g, 0.061 mol) in 150 mL of DMF was added over 1 h to a mixture of 80.8 g (0.34 mol) of $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$, 9.2 mL of H_2SO_4 , and 200 mL of DMF at 70°C . After the mixture was stirred for 3 h at 70°C , it was poured over 500 mL of ice, extracted with pentane (3×50 mL), dried over MgSO_4 , filtered, and vacuum distilled (100 – 102°C (0.1 mm)) to give 6.24 g (58%) of thiol 2.

2,11-Dithia-4,5,6,7,8,9-hexahydro[3.3]paracyclophane (3). A solution of 2 (1.24 g, 0.7 mmol) and α,α' -dichloro-*p*-xylene (1) was added over a period of 70 h to a solution of NaOH (0.6 g, 1.5 mmol) in 95% ethanol (200 mL), using the Herschberg funnel-high dilution technique.⁴ The solution was refluxed for an additional 2 h and then vacuum evaporated to give a viscous residue. The residue was extracted with CCl_4 (3×20 mL), dried over MgSO_4 , filtered, and vacuum evaporated to give a waxy residue. The residue was separated chromatographically on silica gel

(5) Johnson, C. K. ORTEP Report ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, TN, 1965.

(6) Pauling, L. "The Nature of the Chemical Bond", 3rd ed.; Cornell University Press: Ithaca, NY, 1960; p 260.

(7) Bonham, R. A.; Bartell, L. S. *J. Am. Chem. Soc.*, 1959, 81, 3491–3496.

(8) Iijima, T.; Tsuchiya, S.; Kimura, M. *Bull. Chem. Soc. Jpn.* 1977, 50, 2564–2567.

(9) Retrowich, J. P.; Anderson, J. D.; Baizer, M. M. *J. Org. Chem.* 1966, 31, 3897–3903.

(10) Haggis, G. A.; Owen, L. N. *J. Chem. Soc.* 1953, 404–407.

(benzene elution) to yield 0.61 g (31%) of a solid. The solid was recrystallized from benzene-hexanes to give 0.57 g (29%) of cyclophane 3: mp 140-141.5 °C; ¹H NMR (CDCl₃) δ 6.94 (s, 4 H), 3.58 (s, 4 H), 2.33 (d, *J* = 20 Hz, 4 H), 1.65-0.95 (br, 8 H), and 0.45 (br t, 2 H); *v*_{max} (KBr) 3200, 1500, 1460, 1445, 1410, 840, 830, and 715 cm⁻¹; mass spectrum, *m/e* 278 (M⁺).

Anal. Calcd for C₁₆H₂₂S₂: C, 69.01; H, 7.96; S, 23.03. Found: C, 68.81; H, 8.14; S, 22.82.

X-ray Structural Analysis. C₁₂H₂₂S₂, *M_r* = 278.5, monoclinic space group *P*2₁/*c*, *a* = 11.749 (3), *b* = 7.959 (2), *c* = 16.097 (4) Å, β = 100.72 (1)°, *V* = 1479 Å³, *Z* = 4, *d*_{calcd} = 1.25 g cm⁻³. The unit cell was obtained by a least-squares treatment of 14 reflections with 2θ values above 66°, using λ for Cu Kα₁ of 1.5404 Å. A θ-2θ scan using Ni-filtered Cu Kα (λ = 1.5418 Å) X-rays, 2° (1 min) scans, 10-s stationary backgrounds, and manual data collection procedures with a GE XRD-5 diffractometer gave 1628 observed reflections out of 1857 scanned to the limit of 110° in 2θ. The absorption correction (μ = 29.66 cm⁻¹) ranged from 0.66 to 0.91. The structure was solved by symbolic addition and Fourier methods. The final full-matrix least-squares refinement included

positional and anisotropic thermal parameters for the C and S atoms and positional parameters for the H atoms; the standard deviation of an observation of unit weight was 0.73, the largest peak on the final difference map was 0.26 e/Å³, the weighting scheme was without bias, and the final *R* was 0.055. Additional derived parameters are contained in the supplementary material.

Acknowledgment. The mass spectrometer was obtained with the aid of a grant from the National Science Foundation.

Registry No. 1, 623-25-6; *cis*-2, 78307-98-9; *cis*-3, 78307-99-0; *cis*-4, 15898-77-8; *cis*-5, 78328-73-1; *cis*-1,4-bis(hydroxymethyl)cyclohexane, 3236-47-3; *trans*-1,4-bis(hydroxymethyl)cyclohexane, 3236-48-4; 6, 35541-78-7.

Supplementary Material Available: Tables I-VI listing crystal data, fractional coordinates, temperature factors, bond distances, bond angles, best planes data, and torsion angles for C₁₆H₂₂S₂ (6 pages). Ordering information is given on any current masthead page.

Communications

Acylations and Alkylations of an Ester Enolate in High Yield at Room Temperature on Polystyrene Supports¹

Summary: Polymer-bound esters of 3-phenylpropanoic acid are converted to enolates at room temperature and acylated and alkylated in 73-87% isolated yields with little or no self-condensation of ester.

Sir: Insoluble polymeric supports enable separations of synthetic reaction mixtures by simple filtration.² They permit use of large excesses of reagents in peptide syntheses to drive the coupling reactions to completion³ and provide easy separation of troublesome byproducts from reaction mixtures. Another advantage of polymeric supports sometimes cited is "site isolation" by which supposedly rigid polymer chains prevent polymer-bound species from reacting with one another, thus favoring either intramolecular reactions or reactions with reagents in solution rather than interchain reactions within the polymer matrix.⁴ Numerous experiments have demonstrated that polymer chains in 1-2% cross-linked polystyrene are highly

flexible, not rigid.⁵⁻¹⁰ For synthetic purposes, however, one can achieve site isolation if polymer chain motion is slow enough to retard interchain reactions more than it retards the desired reaction. Kraus and Patchornik¹¹ avoided self-condensation of polymer-bound ester enolates and achieved acylation and alkylations of the enolates in low yields at 0-25 °C. Crowley and Rapoport^{4d} did not obtain nine-membered rings by Dieckmann cyclization with diesters bound to 2% cross-linked polystyrene even when as little as 0.1 mmol/g of polymer-bound diester was used. The lifetimes for dimerization of polymer-bound benzyne¹² and for interchain reactions of polymer-bound amines and active esters¹³ are on the order of minutes with use of 2% or 4% cross-linked polystyrene.

Chain mobility in cross-linked polystyrenes decreases as the degree of cross-linking increases and as the swelling of the polymer decreases.⁵⁻¹⁰ Most attempts at site-isolation syntheses have employed conditions of high polymer chain mobility, lightly cross-linked (1-4% divinylbenzene) polystyrenes and good swelling solvents.^{4-8,11-13} We describe here the generation, acylation, and alkylation at room temperature of an unhindered ester enolate supported on 10% and 20% cross-linked polystyrenes. Normally the generation of enolates from unhindered esters in solution is carried out at -78 °C because self-condensation of the ester predominates at higher temperature.^{11,14}

The syntheses are outlined in Scheme I. By the same method Kraus and Patchornik¹¹ generated enolate 3 and

(1) This research was supported by the U.S. Army Research Office.

(2) For reviews, see (a) Hodge, P. In "Polymer-supported Reactions in Organic Synthesis"; Hodge, P., Sherrington, D. C., Eds.; Wiley: New York, 1980; pp 83-155. (b) Mathur, N. K.; Narang, C. K.; Williams, R. E. "Polymers as Aids in Organic Chemistry"; Academic Press: New York, 1980. (c) Manecke, G.; Reuter, P. *Pure Appl. Chem.* 1979, 51, 2313-2330. (d) Leznoff, C. C. *Acc. Chem. Res.* 1978, 11, 327-333. (e) Frechet, J. M. J.; Farrall, M. J. In "Chemistry and Properties of Crosslinked Polymers", Labana, S. S., Ed.; Academic Press: New York, 1977; pp 59-83. (f) Heitz, W. *Adv. Polym. Sci.* 1977, 23, 1-23. (g) Patchornik, A.; Kraus, M. A. *Encycl. Polym. Sci. Technol.* 1976, Suppl. No. 1, 468. (h) Marnatt, L. J.; Neckers, D. C.; Schaap, A. P. In "Applications of Biochemical Systems in Organic Chemistry", Part 2; Jones, J. B., Sih, C. J., Perlman, D., Eds.; Wiley-Interscience: New York, 1976; pp 995-1044.

(3) Erickson, B. W.; Merrifield, R. B. In "The Proteins", 3rd ed.; Neurath, H., Hill, R. L., Boeder, C. L., Eds.; Academic Press: New York, 1976; Vol. 2, pp 255-527.

(4) For critical reviews of this concept, see (a) Warshawsky, A. *Isr. J. Chem.* 1979, 18, 318-324. (b) Daly, W. H. *Makromol. Chem., Suppl.* 2 1979, 3-25. (c) Kraus, M. A.; Patchornik, A. *Isr. J. Chem.* 1978, 17, 298-303. (d) Crowley, J. I.; Rapoport, H. *Acc. Chem. Res.* 1976, 9, 135-144. (e) Crowley, J. I.; Rapoport, H. *J. Org. Chem.* 1980, 45, 3215-3227.

(5) (a) Crowley, J. I.; Harvey, T. B. III; Rapoport, H. *J. Macromol. Sci. Chem.* 1973, A7(5), 1117-1126. (b) Scott, L. T.; Rebek, J.; Ovsyanko, L.; Sims, C. L. *J. Am. Chem. Soc.* 1977, 99, 625-626.

(6) Regen, S. L.; Lee, D. P. *Macromolecules* 1977, 10, 1418-1419.

(7) (a) Wulff, G.; Schulze, I. *Angew. Chem., Int. Ed. Engl.* 1978, 17, 537. (b) *Isr. J. Chem.* 1978, 17, 291-297.

(8) Crosby, G. A.; Kato, M. *J. Am. Chem. Soc.* 1977, 99, 278-280.

(9) Regen, S. L. *Macromolecules* 1975, 8, 689-690.

(10) Ford, W. T.; Balakrishnan, T. *Macromolecules* 1981, 14, 284-288.

(11) Kraus, M. A.; Patchornik, A. *J. Polym. Sci., Polym. Symp.* 1974, 47, 11-18.

(12) Mazur, S.; Jayalekshmy, P. *J. Am. Chem. Soc.* 1979, 101, 677-683.

(13) Rebek, J., Jr.; Trend, J. E. *J. Am. Chem. Soc.* 1979, 101, 737.

(14) Rathke, M. W.; Lindert, A. *J. Am. Chem. Soc.* 1971, 93, 2318-2320.