mixture was filtered and the methanol evaporated under reduced pressure. Workup proceeded as in method B, giving quantitative yields of 13.

Acknowledgment. We gratefully thank the Research Corporation, the donors of the Petroleum Research Fund, administered by the American Chemical Society, the Camille and Henry Dreyfus Foundation, and Boston University Seed Grant Committee for support. We also thank Professors James Panek and Dale Boger for helpful discussions and advice and Dr. Jeff Fishpaugh for help in preparing this manuscript.

Registry No. 1, 120-72-9; 2, 603-76-9; 4, 2166-14-5; 6, 61429-06-9; 7, 576-15-8; 8, 1496-76-0; 13, 109906-82-3; 14, 109890-38-2; 15, 109890-39-3; 16, 109906-83-4; 17, 3787-10-8; 18, 109890-40-6.

Unexpected Regioselectivity in the Base-Promoted Cyclization of an ϵ-Epoxy Sulfone

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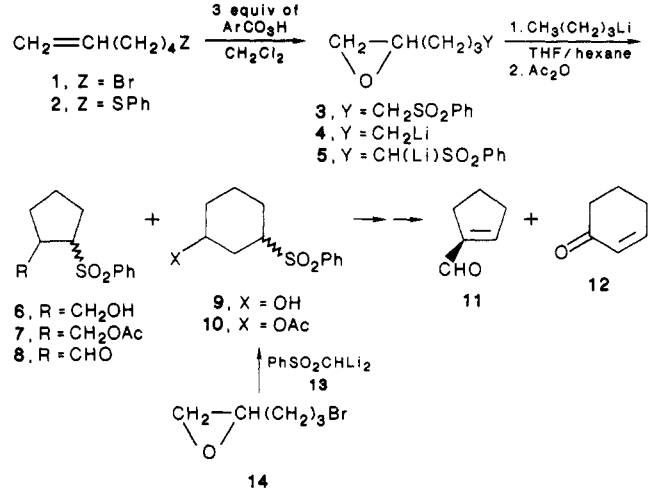
Received February 20, 1987

Recently, Eisch and co-workers published a communication[1] that described the use of [(phenylsulfonyl)methylene]dilithium (13) as a cyclizing reagent for electrophilic bifunctional organic substrates (e.g., α,ω-dihalides or halo epoxides). As an illustration of this methodology, they reported the conversion of 5-bromo-1,2-epoxypentane (14) to 3-(phenylsulfonyl)cyclohexanol (9) in 64% yield. Formation of the product of maximum ring size (i.e., 9 rather than 6), they claimed, argues for an initial attack of dilithio derivative 13 on the epoxide function, since the alternate pathway would have resulted in intermediate 5 and subsequent cyclization to afford the smaller cycloalkane (6), on the basis of previous studies[2] involving γ- and δ-epoxy sulfones. Indeed, further support for this argument (i.e., 5 → 6) can be found in a study of the regioselectivity in the base-promoted cyclization of epoxy nitriles by Stork and co-workers,[3] subsequently reinvestigated by Lallemand and Onanga,[4] as well as a recent report[5] that lithio epoxide 4 cyclized to afford predominantly cyclopentylmethanol.

In view of our interest[6,7] in intramolecular alkylations involving epoxides and the knowledge that, a priori, both modes of cyclization are feasible[8] for the lithio derivative 5 of epoxy sulfone 3, we decided to synthesize independently lithio derivative 5 and determine its cyclization products (i.e., 6 and/or 9).

ϵ-Epoxy sulfone 3 was obtained in 80% yield from the commerically available[9] 6-bromo-1-hexene (1) by reaction

Scheme I



of the latter with the anion derived from thiophenol, followed by subsequent oxidation of the corresponding thioether (2) with 3 equiv of 3-chloroperoxybenzoic acid (Scheme I). Contrary to expectations, treatment of sulfone 3 with 1 equiv of *n*-butyllithium afforded, in >85% yield, a cyclization product shown by ^{1}H NMR analysis to consist of both regioisomeric alcohols (6 and 9), the spectral properties of the major component being consistent with those reported[1] for 3-(phenylsulfonyl)cyclohexanol (9).

Since TLC analysis indicated that separation of this cyclization product (6 and 9, each an undetermined mixture of stereoisomers) would be problematical, further transformations were undertaken to confirm the structural identity and exact composition of its components. Oxidation of the cyclized product mixture with 4 molar equiv of pyridinium chlorochromate[10] was accompanied by substantial elimination of the phenylsulfonyl moiety to afford 2-cyclohexenone (12) as the major product, accompanied by minor amounts of aldehydic sulfone 8.[11] Subsequent treatment of this latter mixture of products in ether with 20% aqueous potassium hydroxide at 20 °C afforded 2-cyclohexenone (12) accompanied by a minor amount of 1-cyclopentenecarbaldehyde (11)[12] as the only volatile products, whose identities were further confirmed by ^{1}H NMR and VPC[13] analysis (co-injection with authentic samples).

Further evidence confirming the structure of the cyclization products derived from lithio derivative 5 was obtained after acetylation[14] of the mixture. Gratifyingly, ^{1}H NMR analysis[15] of the acetylated products (7 and 10)

(1) Eisch, J. J.; Dua, S. K.; Behrooz, M. *J. Org. Chem.* **1985**, *50*, 3674.
(2) Decesare, J. M.; Corbel, B.; Durst, T.; Blount, J. F. *Can. J. Chem.* **1981**, *59*, 1415.
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(5) Cooke, M. P., Jr.; Houpis, I. N. *Tetrahedron Lett.* **1985**, *26*, 3643 and references therein.
(6) Babler, J. H.; Tortorello, A. J. *J. Org. Chem.* **1976**, *41*, 885.
(7) Babler, J. H.; Bauta, W. E. *Tetrahedron Lett.* **1984**, *25*, 4323.
(8) Baldwin, J. E.; Thomas, R. C.; Kruse, L. I.; Silberman, L. *J. Org. Chem.* **1977**, *42*, 3846. Baldwin, J. E. *J. Chem. Soc., Chem. Commun.* **1976**, 734.
(9) Available from Wiley Organics, Inc., Columbus, OH.
(10) Corey, E. J.; Suggs, J. W. *Tetrahedron Lett.* **1975**, 2647.
(11) Aldehyde 8 was characterized by a ^{1}H NMR absorption band at δ 9.64 (CHO). The sluggishness toward elimination of the phenylsulfonyl moiety exhibited by aldehyde 8 indicates that hydroxy sulfone 6 probably has the trans configuration.
(12) An authentic sample of aldehyde 11 was prepared as described in the literature. See: Brown, J. B.; Henbest, H. B.; Jones, E. R. H. *J. Chem. Soc.* **1950**, 3634.
(13) A 6 ft × $^1/_8$ in. column packed with 5% OV-17 on 100–120-mesh Gas Chrom Q (oven temperature 88 °C, flow 15 mL/min) was used for this analysis. Retention times: 2.05 min (aldehyde 11) and 3.29 min (ketone 12).
(14) A procedure described by Hassner and co-workers was used to ensure quantitative acetylation of the cyclized product. See: Hassner, A.; Krepski, L. R.; Alexanian, V. *Tetrahedron* **1978**, *34*, 2069.
(15) The ^{1}H NMR spectrum of cyclopentanoid 7 exhibited a doublet (J = 6 Hz, CH_2OAc) at δ 3.99 and a singlet (O=CCH_3) at δ 1.98. The corresponding absorptions for acetate 10 occurred at δ 5.00–4.39 (CHOAc, cis stereoisomer), 5.43–5.11 (CHOAc, trans stereoisomer), and 2.03 (s, O=CCH_3).

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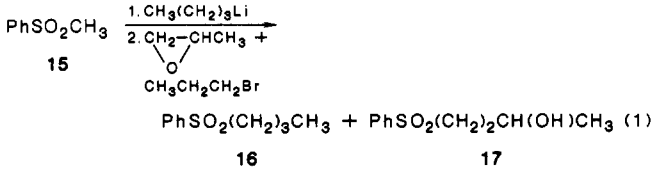
enabled an accurate determination of the ratio of regioisomers obtained in the cyclization process (>70% cyclohexanoid product **9**), as well as stereochemical information (i.e., sulfone ester **10** was obtained as a 2:1 cis/trans mixture). In addition, the identity of cyclohexanoid **10** was subjected to further confirmation by independent synthesis involving acetylation[14] of an authentic sample[16] of 3-(phenylsulfonyl)cyclohexanol (**9**; 5:1 mixture of cis/trans stereoisomers).

In view of the unexpected regioselectivity in the base-promoted cyclization of epoxy sulfone **3**, we decided to examine the chemoselectivity of the reaction between the dilithio derivative **13** of methyl phenyl sulfone (**15**)[17] and an equimolar mixture of propylene oxide and 1-bromopropane (eq 1). Eisch and co-workers[1] had speculated that dianion **13** attacks an epoxide moiety more rapidly than a carbon–halogen center, but based this on the assumption that lithio derivative **5** would cyclize to afford only cyclopentanoid **6**, in contrast to the results that we report herein.



As described in the Experimental Section, treatment of methyl phenyl sulfone (**15**) with 2 equiv of *n*-butyllithium, followed by the addition of a 1:1 mixture of propylene oxide/1-bromopropane, afforded hydroxy sulfone **17**[18] as the sole alkylation product, thereby confirming the hypothesis of Eisch and co-workers. Even alkylation of the corresponding monoanion (derived from **15** and less than 1 equiv of *n*-butyllithium[19]) as outlined in eq 1 afforded hydroxy sulfone **17** as the major product (2:1 ratio of **17**:**16**[20]). Since the latter product (**16**) was not observed in the experiment utilizing 2 equiv of *n*-butyllithium, the results give evidence for the existence of dianion **13**.

Experimental Section

General Methods. Unless specified otherwise, all organic reagents were purchased from Aldrich Chemical Co.; Florisil (60–100 mesh) was obtained from Fisher Scientific Co. Tetrahydrofuran was purified prior to use by distillation from lithium aluminum hydride. All reactions were carried out under a nitrogen atmosphere. Products were recovered from the organic extracts after drying over anhydrous magnesium sulfate and removal of the solvent with a rotary evaporator under reduced pressure. Evaporative distillation refers to bulb-to-bulb (Kugelrohr) short-path distillation. ^{1}H NMR spectra were obtained on a Varian EM-360A or Varian FT-80 spectrometer with $CDCl_3$ as the solvent and tetramethylsilane as an internal standard. Infrared spectra were recorded with a Beckman Acculab 1 spectrophotometer, and vapor-phase chromatography (VPC) was performed on a Hewlett-Packard 5750 chromatograph. Peak areas were determined by use of a Hewlett-Packard Model 3392A integrator and are uncorrected for response factors relative to an internal standard. TLC analyses were conducted on precoated silica gel sheets (E. M. Merck, catalog no. 5775). Microanalyses were performed by Micro-Tech Laboratories, Skokie, IL.

6-(Phenylsulfonyl)-1,2-epoxyhexane (3). A mixture of 1.00 mL (9.75 mmol) of thiophenol, 1.208 g (7.41 mmol) of 6-bromo-1-hexene (**1**),[9] and 1.402 g (10.14 mmol) of anhydrous potassium carbonate in 5.00 mL of absolute ethanol was heated at reflux for 14 h, after which it was cooled to room temperature and diluted with 50 mL of 1:1 (v/v) 1 M aqueous sodium hydroxide/saturated brine, and the product was isolated by extraction with ether. The organic layer was washed with 1:1 (v/v) 1 M aqueous sodium hydroxide/saturated brine (1 × 50 mL), 5% (w/v) aqueous sodium chloride (4 × 50 mL), and saturated brine (1 × 50 mL) in successive order. The product was then isolated from the organic extract in the usual manner and purified by evaporative distillation [bp 90–110 °C (bath temperature), 0.30 mm], affording 1.42 g (100%) of thioether **2**: ^{1}H NMR δ 7.30 (m, 5 aryl H), 6.16–5.48 (complex pattern, C*H*=CH$_2$), 5.18–4.78 (complex pattern, CH=C*H*$_2$), 2.89 (br t, J = 7 Hz, CH$_2$S), 2.06 (br q, J = 6 Hz, C*H*$_2$CH=CH$_2$). Since this latter compound (**2**) was susceptible to air oxidation, it was immediately oxidized by the following procedure: To a solution of unsaturated thioether **2** (1.244 g, 6.47 mmol) in 40 mL of dichloromethane cooled to 0 °C (ice/water bath) was added 5.322 g (26.2 mmol) of 85% *m*-chloroperoxybenzoic acid. This mixture was subsequently stirred at 0 °C for 15 min and at room temperature for an additional 20 h, after which it was diluted with 120 mL of ether and washed with 4:1 (v/v) 1 M aqueous sodium hydroxide/saturated brine (3 × 50 mL) and saturated brine (1 × 50 mL) in successive order. Isolation of the product from the organic extract in the usual manner afforded 1.24 g (80%) of epoxy sulfone **3** after evaporative distillation: bp 168–182 °C (bath temperature), 0.20 mm; IR ν_{max} (film) 1445, 1405, 1300, 1145, 1085, 835, 788, 745, 730, 690 cm^{-1}; ^{1}H NMR δ 8.08 (m, 2 aryl H), 7.73 (m, 3 aryl H), 3.16 (t, J = 7 Hz, CH$_2$S), 2.78 (m, CH$_2$O), 2.44 (m, OCH), 1.62 (br m, 6 H). Anal. Calcd for $C_{12}H_{16}SO_3$: C 59.97; H, 6.71; S, 13.34. Found: C, 59.99; H, 6.69; S, 13.15.

Base-Promoted Cyclization of Epoxy Sulfone 3. To a solution of 1.059 g (4.41 mmol) of epoxide **3** in 12.0 mL of 3:1 (v/v) anhydrous tetrahydrofuran/hexane cooled to −78 °C (dry ice/acetone bath) was added dropwise rapidly via syringe 4.00 mL of a 1.35 M solution of *n*-butyllithium in hexane.[21] The resulting solution was stirred at −78 °C for 10 min, at 0 °C for 90 min, and then for an additional 5 min at room temperature. The product was subsequently isolated by dilution of this mixture with 60 mL of 15% (w/v) aqueous sodium chloride and 5 mL of 2 M aqueous hydrochloric acid, followed by extraction with 3:1 (v/v) ether/dichloromethane. After the organic extract was washed with 15% (w/v) aqueous sodium chloride, the product was isolated in the usual manner and purified by evaporative distillation [bp 160–175 °C (bath temperature), 0.20 mm], affording 908 mg (86%) of a colorless oil, the spectral properties of which were consistent with those reported[1] for 3-(phenylsulfonyl)cyclohexanol (**9**). However, TLC analysis and subsequent reactions involving this cyclization product indicated the presence of cyclopentanoid **6** as a minor (<30%) component. Attempts to separate this mixture of **6** and **9** by flash chromatography[22] were unsuccessful due to their similar

(16) This compound was prepared by using the procedure described by Pinnick and Chang. See: Chang, Y.-H.; Pinnick, H. W. *J. Org. Chem.* **1978**, *43*, 373.

(17) Field, L.; Clark, R. D. *J. Org. Chem.* **1957**, *22*, 1129.

(18) The identity of this product was confirmed by independent synthesis, starting from methyl vinyl ketone as described previously[16] in the literature.

(19) This alkylation was conducted by addition of 1.00 mL of a 1.6 M solution of *n*-butyllithium in hexane to a solution of 270 mg (1.73 mmol) of methyl phenyl sulfone (**15**) in 8.00 mL of anhydrous THF at 0 °C, followed by stirring at 0 °C for 45 min and subsequent addition of 0.35 mL (3.8 mmol) of 1-bromopropane and 0.25 mL (3.6 mmol) of propylene oxide in 3.0 mL of hexane. After an additional 90 min at 0 °C, the reaction was quenched by rapid addition of 5 mL of saturated aqueous ammonium chloride, and the product mixture was isolated as described in the Experimental Section. The components (**16**, **15**, and **17**) could be separated by chromatography on Florisil (20 mL), butyl phenyl sulfone (**16**, elution with hexane/40% ether) being eluted first.

(20) Sulfone **16** exhibited the following ^{1}H NMR spectral properties: δ 8.01 (m, 2 aryl H), 7.70 (m, 3 aryl H), 3.16 (t, J = 6.5 Hz, CH$_2$S), 1.57 (br m, 4 H), 0.90 (t, J = 6 Hz, CH$_3$).

(21) In an experiment suggested by a reviewer, a solution of epoxide **3** in hexane/anhydrous tetrahydrofuran (THF) was added dropwise to 3.5 equiv of *n*-butyllithium in hexane/THF at −78 °C (conditions thought to favor formation of the dilithio derivative of **3**). The resulting solution was stirred at −78 °C for 45 min, at 0 °C for 90 min, and then for an additional 5 min at room temperature. Isolation of the product mixture in the usual manner, followed by acetylation by using the conditions described for the preparation of **10**, afforded a 3:1 mixture of **10**:**7**. Although no change in the regioselectivity in the cyclization was apparent with this modified procedure, cyclohexanoid **10** was shown by ^{1}H NMR analysis to be predominantly the trans stereoisomer.

(22) Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.

polarities and the presence of each as a stereoisomeric mixture.

3-(Phenylsulfonyl)cyclohexanol Acetate (10). A solution of 748 mg (3.11 mmol) of hydroxy sulfone **9**,[16,23] 2.00 mL (21.2 mmol) of acetic anhydride, and 71 mg (0.58 mmol) of 4-(*N*,*N*-dimethylamino)pyridine in 3.00 mL of triethylamine was stirred at room temperature for 18 h, after which it was poured into a mixture of 20 mL of 10% aqueous sodium hydroxide and 20 g of crushed ice to destroy excess acetic anhydride. Subsequent dilution of this mixture with 60 mL of 15% (w/v) aqueous sodium chloride, extraction with 3:1 (v/v) ether/dichloromethane, and washing of the latter extracts with 15% aqueous sodium chloride (1 × 60 mL), 1:1 (v/v) 2 M aqueous hydrochloric acid/saturated brine (2 × 60 mL), 1:1 (v/v) 1 M aqueous sodium hydroxide/saturated brine (1 × 60 mL), and saturated brine (1 × 60 mL) in successive order afforded 789 mg (90%) of sulfone ester **10** as a 5:1 mixture of cis/trans stereoisomers: bp 185–198 °C (bath temperature), 0.30 mm; ^{1}H NMR δ 7.97 (m, 2 aryl H), 7.71 (m, 3 aryl H), 5.43–5.11 (m, C*H*OAc, trans stereoisomer), 5.00–4.39 (m, C*H*OAc, cis stereoisomer), 3.10 (br m, CHS), 2.03 [s, OC(=O)CH_3]; IR ν_{max} (film) 1730 (C=O), 1305, 1240, 1145, 1030, 720, 690 cm^{-1}. Anal. Calcd for $C_{14}H_{18}SO_4$: C, 59.55; H, 6.42; S, 11.36. Found: C, 59.49; H, 6.35; S, 11.32.

Alkylation of [(Phenylsulfonyl)methylene]dilithium (13). To a solution of 312 mg (2.0 mmol) of methyl phenyl sulfone (**15**)[17] in 10.0 mL of anhydrous tetrahydrofuran (THF) cooled to 0 °C (ice/water bath) was added dropwise 3.00 mL of a 1.35 M solution of *n*-butyllithium in hexane. The resulting mixture was stirred at 0 °C for 60 min, after which a solution of 0.37 mL (4.07 mmol) of 1-bromopropane and 0.28 mL (4.00 mmol) of propylene oxide in 3.0 mL of hexane was added dropwise over 2 min. After an additional 20 min at 0 °C, the reaction was quenched by rapid addition of 5 mL of saturated aqueous ammonium chloride. Dilution of this mixture with 60 mL of 15% (w/v) aqueous sodium chloride and 5 mL of 2 M aqueous hydrochloric acid, followed by extraction with 3:1 (v/v) ether/dichloromethane and the usual isolation procedure, afforded 331 mg of crude product. Chromatography of the latter on Florisil (20 mL) gave 247 mg (79%) of recovered starting material (**15**, elution with 1:1 ether/hexane), accompanied by 40 mg of 4-(phenylsulfonyl)-2-butanol (**17**, elution with ether) as the only other identifiable component. The ^{1}H NMR spectral properties[24] of the latter (**17**) were identical with those exhibited by an authentic sample[16] of the same compound.

Acknowledgment. I thank Professor John J. Eisch of the State University of New York at Binghamton for a copy of his ^{1}H NMR spectrum of 3-(phenylsulfonyl)-cyclohexanol.

(23) The same procedure was used to acetylate in quantitative yield the cyclization product (i.e., a mixture of **6** and **9**), thereby verifying cyclohexanoid **9** as the major component as well as confirming its identity.

(24) Hydroxy sulfone **17** exhibited the following spectral properties: ^{1}H NMR δ 3.91 (m, C*H*OH), 3.30 (br t, J = 7 Hz, CH_2S), 1.17 (d, J = 6 Hz, CH_3); IR ν_{max} (film) 3490 (OH), 1295, 1148, 1090, 735, 690 cm^{-1}.

A Comment on the Structure and Proton NMR Spectrum of 2,8,17-Trithia[4^{5,12}][9]metacyclophane

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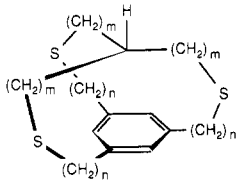
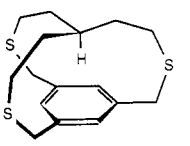
Received April 3, 1987

Ricci et al.[1] reported in 1976 the syntheses of several macrocyclic thioethers, which were assigned structures **1a–d**. The compounds were characterized by their melting points, elemental analyses, and 60-MHz ^{1}H NMR spectra. The complete assignments of the NMR spectra were not included in the Experimental Section of this paper, but the actual spectra were reproduced in the region from 0 to 8 ppm (δ). The spectrum of 2,8,17-trithia[4^{5,12}][9]-metacyclophane (**1b**) showed four resonances in this region, which were easily assigned to the aromatic ring protons and the three types of methylene groups; however, no methine resonance was visible. There are two possible stereoisomers of 2,8,17-trithia[4^{5,12}][9]metacyclophane, the "out" isomer **1b** and the "in" isomer **2**, but Ricci et al. apparently did not consider that the methine proton might lie *inside* the macrocycle as illustrated in structure **2**. Were this the case, the methine proton resonance would surely fall below 0 ppm due to the strong shielding effect of the benzene ring current.



1a n = 1, m = 3
1b n = 1, m = 2
1c n = 2, m = 3
1d n = 2, m = 2



2

We have been interested in compounds exhibiting short nonbonded distances between hydrogens and aromatic rings,[2,3] and the distance between the methine hydrogen and the aromatic ring plane in isomer **2** would be exceptionally short. Accordingly, we prepared 2,8,17-trithia-[4^{5,12}][9]metacyclophane using a minor modification of the method of Ricci et al.[1] The 250-MHz ^{1}H NMR spectrum of this material is essentially identical with that of Ricci et al. in the region from δ 0 to 8; however, an examination of the very high field region of the spectrum reveals the methine proton resonance as a septet at δ −1.68. This resonance is observed at a higher field than even the most highly shielded methylene protons of [9]- (δ 0.33), [8]- (0.19), [7]- (−0.3 to −0.9), [6] (−0.6),[4] and [5]paracyclophane (0.01).[5] This substance must therefore be the "in" isomer **2**. No material with spectral properties consistent with structure **1b** was isolated from the reaction mixture.[6]

Experimental Section

2,8,17-Trithia[4^{5,12}][9]metacyclophane. A solution of 1,3,5-tris(mercaptomethyl)benzene[8] (1.09 g, 5.0 mmol) and tris-(2-bromoethyl)methane[9] (1.69 g, 5.0 mmol) in benzene (100 mL) and a solution of KOH (1.03 g, 18 mmol) in ethanol (100 mL) were added simultaneously and dropwise over 3 h to rapidly stirred, refluxing ethanol (500 mL) under an argon atmosphere. After heating for 15 h, the reaction mixture was cooled and acidified with concentrated HCl (1 mL). The solvent was evaporated, and the solid residue was extracted twice with 50 mL portions of CCl_4. The extracts were combined, concentrated, and chromatographed

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(6) In this regard, we note that MM2(85)[7] calculations yield a steric energy for the "in" isomer **2** that is ca. 7 kcal/mol lower than that of the "out" isomer **1b**. In the calculated structure of compound **2**, the distance from the inside hydrogen to the mean plane of the aromatic ring is 2.21 Å.

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