magnetism. Preparative scale solutions of either 2 or 3 purposely decolorized with CO_2-free oxygen produced copious amounts of carbon dioxide upon acidification. An approximate gravimetric evaluation indicated 30 mol % CO_2 yield with respect to original dimethiodide. Furthermore, carbon monoxide was detected with the I_2O_5 test for both 2 and 3. The liberated iodine in the case of 3 was determined both colorimetrically and by titration to be about 12 mol %. These observations were useful in the formulation of a reaction mechanism. When the base concentration was lower than ∼0.1 *M*, no blue color developed. On the other hand, when the base concentration was very high (∼10 *M*), the methiodides were no longer soluble and only a very weak blue color developed.

Unlike those of its methiodides, sodium hydroxide solutions of the parent dipyridyl ketone remained colorless and diamagnetic under similar conditions.[12]

A plausible mechanism for viologen radical formation from 3 is shown in Scheme I. Although the hydroxide ion could abstract protons from the methyl groups activated by the pyridinium nitrogens, it seems to attack preferentially the carbonyl group, followed by a 1,2 "push–pull" migration of a 4-pyridinium group to generate cation 5. One can easily visualize proton abstraction from 5 and subsequent facile decarboxylation of zwitterion 6 with formation of the powerful reducing agent[10,13,14] *N,N'*-dimethyldihydro-4,4'-bipyridyl (7), which is rapidly oxidized by dimethiodide 3 to viologen radical 1. It is known that such molecules as 7, which have also been called "alkali-metal analogs," are capable of generating viologen radicals in reaction even with common solvents.[13,14] We found that radical cation 8 formed by metal reduction of 3 has a long lifetime in degassed acetonitrile.[15] Apparently, however, in aqueous hydroxide it undergoes rapid decarbonylation via 9 and 10, as shown in Scheme I, with ultimate formation of another viologen radical. It is conceivable that radical 8 leads reversibly to 9 with slow leakage over an energy barrier to 10. Presence of both carbon monoxide and carbonate among products together with viologen-radical yields of roughly 50 mol %[16] tends to confirm the postulated mechanism. A reaction path for viologen-radical formation from monomethiodide 2 requires decarboxylation, decarbonylation, and either fission at the carbonyl site with subsequent coupling of

N-methylpyridine moieties or acquisition of a second methyl group at the nonmethylated nitrogen. This latter alternative can be visualized as shown in eq 1. However, this possibility should be considered only tentative, since further analytical work may be necessary to substantiate such a mechanism.

A Stereoselective Synthesis of trans-Isobornylcyclohexanol

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The long-recognized value of sandalwood oil as a perfume essential to the formulation of a variety of fine soap fragrances has prompted us to consider syntheses for other materials reputed to possess sandalwood-like odors. The curious discovery that a mixture of terpene cyclohexanols, formed by catalytic reduction of the condensation products of camphene and guiacol or camphene and phenol, provides a sandalwood odor first appeared about 20 years ago.[1] Although there has been some use of this material in perfumery, it was some years before Demole[2] isolated and synthesized the one material responsible for the sandalwood-like odor, *trans*-3-(*exo*-5-isocamphyl)cyclohexanol. Meanwhile, Russian workers reported[3] that the somewhat simpler system, *trans*-3-isobornylcyclohexanol (7), also possessed a "strong sandalwood" odor.

The reported odor properties for trans isomer 7 seemed to mark it as a unique member in a family of similar compounds. Each of the 2- and 4-isobornylcyclohexanols, prepared by others,[4] and 4-bornylcyclohexanol, prepared in these laboratories,[5] were characterized as almost odorless. Furthermore, the *cis*-3-isobornyl-cyclohexanol (8) was claimed to possess a cedar note. Because of what appeared to be very unique and strict structural requirements for the sandalwood odor, we wished to validate this observation through independent synthesis.
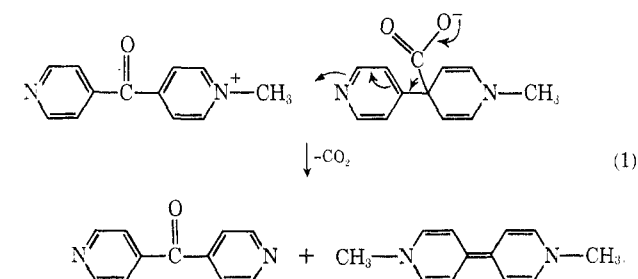

$$\downarrow -CO_2 \qquad (1)$$

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(12) F. L. Minn, C. L. Trichilo, C. R. Hurt, and N. Filipescu, *J. Amer. Chem. Soc.*, **92**, 3600 (1970). Preparation and identification of the methiodides 2 and 3 were also given in this article.

(13) E. Müller and K. A. Bruhn, *Ber.*, **86**, 1122 (1953).

(14) E. Weitz and A. Nelken, *Justus Liebigs Ann. Chem.*, **425**, 187 (1921).

(15) N. Filipescu, F. E. Geiger, C. L. Trichilo, and F. L. Minn, *J. Phys. Chem.*, in press.

(16) The conversion of the methiodides to viologen radical was evaluated spectroscopically in degassed samples of known concentrations from maximum absorbance at 395 and 602 nm.

(1) J. R. Byers, Jr., *Amer. Perfumer*, **49**, 483 (1947); German Patent 834,593 (1952); *Chem. Abstr.*, **47**, 5446*i* (1953).

(2) E. Demole, *Helv. Chim. Acta*, **47**, 319, 1766 (1964).

(3) (a) L. A. Kheifits and I. S. Aul'chenko, "Synthese der Reichstoffeaus Terpenpbenolen," Preprint from conference held at Geneva, Nov 17, 1966. (b) L. A. Kheifits and I. S. Aul'chenko, *Biol. Aktiv. Soedin*, 166 (1968); *Chem. Abstr*, **71**, 102026 (1969). (c) Note Added in Proof.—I. S. Aul'chenko and L. A. Kheifits. *Amer. Perfum., Cosmet.*, **85**, 37 (July 1970).

(4) L. A. Kheifits, G. I. Moldovanskaya, A. V. Kokhmanskii, and V. N. Belov, *Zh. Obshch. Khim.*, **33**, 1676 (1963); *Chem. Abstr.*, **59**, 12845*e* (1963); G. I. Moldovanskaya, L. A. Kheifits, A. V. Kokhmanskii, and V. N. Belov, *Zh. Obshch. Khim.*, **33**, 3392 (1963); *Chem. Abstr.*, **60**, 5555*h* (1964).

(5) W. F. Erman, unpublished observations.

Synthetic schemes directed toward terpene cyclohexanols have been several in number.[2-9] As those which allow the preparation of pure materials are particularly limited, it was our intent to develop a scheme (Scheme I) which possessed stereoselectivity. There are theoretically 16 diastereomers of the bicyclo[2.2.1]-heptylcyclohexanol I. By starting with optically pure


I
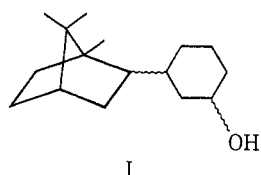
d-camphor, it is possible by this procedure to isolate stereoselectively the two diastereomeric cyclohexanols **7a** and **7b**. However, in our work we began with *dl*-camphor and obtained principally the four diastereomeric alcohols **7a-d**.

d
7a

d
7b

l
7c

l
7d
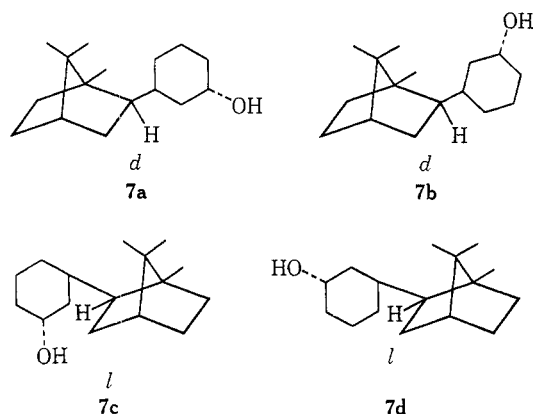
The required carbon framework was created through the interaction of camphor (**1**) and the Grignard of bromo ether **2**. Alcohol **3**, obtained in modest yield, was contaminated with several by-products including substantial amounts of benzyl phenyl ether. As the oily alcohol **3** resisted distillation, rigorous purification was not possible. Spectral data, however, corroborated assignment of structure **3** to the major product from the Grignard reaction.

Prior dehydration studies on model systems had revealed that while boron trifluoride etherate was a satisfactory catalyst for dehydrating 2 and 4 isomers,[6,7] it was not suitable for structure type **3**. The 3-substituted isomers were, in fact, prone to rearrangement under these conditions and afforded substantial amount of 1-arylcamphene **9**. Thionyl chloride proved to be a useful substitute for boron trifluoride, and olefin **4** could be smoothly generated using this reagent. This mate-
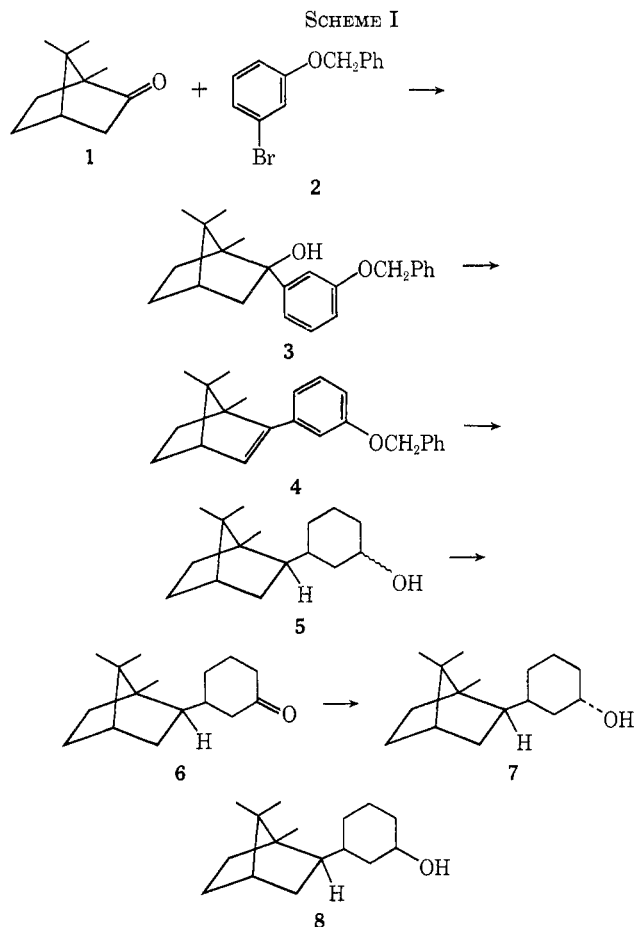

9
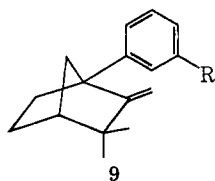
(6) W. F. Erman and T. J. Flautt, *J. Org. Chem.*, **27**, 1526 (1962).
(7) W. F. Erman, *J. Amer. Chem. Soc.*, **86**, 2887 (1964).
(8) I. S. Aul'chenko and L. A. Kheifits, *J. Org. Chem. USSR*, **2**, 2015 (1966).
(9) L. A. Kheifits, I. S. Aul'chenko, and G. M. Shchegoleva, *ibid.*, **2**, 2019 (1966).

SCHEME I

rial could subsequently be fully saturated using fresh W-6 Raney nickel.[10]

The product mixture obtained on reduction was usually complex and could be only partially separated by careful column chromatography. In addition to 60–80% of the expected alcohol mixture **5**, several hydrocarbons, aromatics, and carbonyl-containing materials were also isolated. The ratio of cis alcohol **8** to trans alcohol **7** varied between *ca.* 6:4 and 7:3. While the various by-products described above could be minimized through slight experimental modifications, other alcoholic impurities, believed to be bornyl mixture **10** (10–15%), were carried through the remainder of the sequence.

10
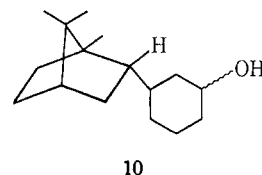
Since chromatographic efforts to isolate the pure trans isomer from this mixture proved inefficient, we chose to examine an alternate, more selective synthetic route. The use of iridium compounds as reduction transfer catalysts has been reported to be a very effective method for generating high purity axial alcohols. Following published reports,[11] we found a strong pref-

(10) A. A. Pavlic and H. Adkins, *J. Amer. Chem. Soc.*, **68**, 1471 (1946).
(11) (a) Y. M. Y. Haddad, H. B. Henbest, J. Husbands, and T. R. B. Mitchell, *Proc. Chem. Soc.*, 361 (1964). (b) We thank Professor Eliel for communicating this procedure to us prior to its publication.

erence for the formation of trans isomer **7** on reduction of ketone **6**. Gas chromatographic examination demonstrated the presence of trans isomer **7** (80–90%) mainly contaminated with a material tentatively identified as *trans*-bornyl isomer **10** (axial OH). Further purification of alcohol **7** was possible through careful glpc. Less than 5% cis isomer **8** was detected in the reduction mixture.

The purest samples of *cis*- or *trans*-isobornylcyclohexanol (**7, 8**) or of mixture **5** exhibited rather weak odors. Although trans isomer **7** possessed a sandalwood note, it did not compare well in intensity with *trans*-3-(*exo*-5-isocamphyl)cyclohexanol.[12]

Experimental Section[13]

3-Benzyloxyphenylborneol (3).—The procedure of Erman and Flautt[6] was employed. A solution of 17.6 g (0.07 mol) of 3-benzyloxy-1-bromobenzene (**2**)[14] in 16 ml of tetrahydrofuran (Fisher Certified) was added dropwise over 0.5 hr to 1.87 g (0.08 g-atom) of magnesium (20 mesh).[13b] The resulting mixture was refluxed for 2 hr, cooled slightly, and treated over 1 hr with a solution of 10.3 g (0.07 mol) of camphor in 16 ml of tetrahydrofuran. The reaction was refluxed for 2 hr, cooled to 0–5°, and decomposed cautiously with 17 ml of a saturated aqueous solution of sodium sulfate over 15 min. The resulting ether layer was decanted and the residual salts were washed well with several portions of ether. The combined organic layers were washed once with saturated aqueous sodium sulfate solution and dried (MgSO$_4$). Solvent removal afforded 31 g of oil which was subjected to distillation. Starting materials, predominantly camphor, were removed first, bp 90–110° (bath) (0.35 mm), followed by 5.7 g of benzyl phenyl ether, bp 85–95° (0.35 mm), mp 35.5–37.5°. Recrystallization (CH$_3$OH) gave pure ether: mp 39–40°; ir (CCl$_4$) 6.31, 8.12, 9.75 μ (lit.[15] mp 39–40°). Distillation up to 165° (0.5 mm) gave only a small amount of additional crude ether, mp 33–35°.

The residual 10 g (44%) of material could not be induced to crystallize and was used directly for the next reaction. The material exhibited spectral properties consistent with the expected product **3**: ir (film) 2.81, 2.88, 3.26, 3.29, 6.21, 6.30, 7.91, 10.3, 11.6, 12.7, 13.5, 14.3 μ; nmr (CCl$_4$) τ 2.50–3.40 (complex, 2, aromatic), 5.02 (s, 2, OCH$_2$), 8.80 (s, 3, C$_1$ CH$_3$), 9.15, 9.18 (2 s, 6, C$_7$ CH$_3$'s).

3-Benzyloxyphenylbornylene (4).—A solution of 4.6 g (0.014 mol) of crude borneol **3** in 50 ml of anhydrous pyridine cooled to −5 to −10° was treated over 5 min with 28 ml of a 1:1 mixture of thionyl chloride and anhydrous pyridine.[13b] The reaction was stirred at −5° for an additional 10 min and was poured into *ca.* 100 ml of cold pentane. The pentane solution was treated cautiously with ice chips and the resulting layers separated. Several

pentane extracts were combined and washed with two portions of 3% aqueous hydrochloric acid, followed by brine until neutral. The pentane extract was dried (MgSO$_4$) and the solvent removed. As distillation could not be effected [up to 170° (bath) (0.4 mm)], the crude was purified by passage through 150 ml of Florisil. Monitoring by infrared analysis indicated that 2.6 g (59%) of essentially pure bornylene **4** was eluted with hexane and 2% ether in hexane. Material purified by glpc (SE-30, 225°) exhibited the following properties: $n^{25.5}$D 1.5745; ir (film) 3.26, 3.30, 6.18, 6.24, 6.33, 8.30, 9.52, 9.75, 12.8, 13.6, 14.3 μ; nmr (CCl$_4$) τ 2.65–3.60 (complex, 9, aromatic), 4.19 (d, 1, J = 4 Hz, C=CH), 5.11 (s, 2, OCH$_2$), 7.75 (t, 1, J = 3 Hz, C$_4$ H), 9.00 (s, 3, C$_1$ CH$_3$), 9.18, 9.25 (2 s, 6, C$_7$ CH$_3$'s).

Anal. Calcd for C$_{23}$H$_{26}$O: C, 86.74; H, 8.23. Found: C, 86.75; H, 8.15.

Further elution (5% ether in hexane through 100% ether) afforded 1.1 g of additional, semicrystalline material. Repeated recrystallization from ethanol afforded 3,3'-dibenzyloxybiphenyl: mp 120–121°; ir (CHCl$_3$) 3.26, 3.32, 6.23, 6.33, 9.71, 11.6, 14.3 μ; nmr (CDCl$_3$) τ 2.2–3.17 (complex, 18, aromatic), 4.91 (s, 4, OCH$_2$); mass spectrum *m/e* 366.

Anal. Calcd for C$_{26}$H$_{22}$O$_2$: C, 85.21; H, 6.05. Found: C, 85.25; H, 6.0.

cis-**3-Isobornylcyclohexanol (8).**—A solution of 3.5 g (0.01 mol) of bornylene **4** in 30 ml of absolute ethanol was hydrogenated for 20 hr at 112° and 2400 psi of hydrogen over *ca.* 3 g (wet with ethanol) of freshly prepared Raney nickel catalyst.[10] The catalyst was removed by filtration and the solvent was removed at reduced pressure. A 1.51-g portion of the residual 1.95 g (83%) of oil was chromatographed on 150 g of Woelm neutral alumina (activity grade I). Fraction monitoring by infrared analysis indicated several hydrocarbons, aromatics, and carbonyl-containing materials eluted prior to the desired cyclohexanols. Several 2–5% ethyl acetate in ether fractions which contained predominantly trans isomer **7** and the cis isomer **8** were collected. Although the first fractions were enriched in **7** and the latter in isomer **8**, the majority of the fractions were poorly resolved. Further elution afforded mixtures of cyclohexanols and incompletely reduced phenolic products.

In latter experiments we found it possible to obtain satisfactory purity by carrying out the reduction at 130° for 36 hr. The resultant cyclohexanol mixture **5** was obtained in 70–80% yield by direct distillation at 115–130° (0.03 mm). The mixture usually contained 60–70% isomer **8** and 30–40% isomer **7** by gas chromatographic analysis. Further purification of the cis isomer **8** by gas chromatography (FFAP, 200°) gave material exhibiting the following properties: n^{22}D 1.5086 (lit.[8] n^{20}D 1.5088); ir (film) 3.00, 7.19, 9.52, 10.4 μ; nmr (CDCl$_3$) τ 6.55 (m, 1, CHOH), 7.15 (s, 1, OH), 9.00–9.30 (complex, CH$_3$'s). The 3,5-dinitrobenzoate derivative exhibited mp 152° (lit.[8] mp 157–159°).

3-Isobornylcyclohexanone (6).—A solution of 4.24 g (0.02 mol) of cyclohexanol mixture **5** in 40 ml of acetone, cooled to 5°, was treated with 5 ml of Jones reagent[16] over 15 min. The reaction was stirred an additional 5 min at 5°, and the excess oxidizing agent was decomposed by the dropwise addition of isopropyl alcohol. The mixture was poured into brine and the organic material was isolated with ether. The combined extracts were washed with saturated aqueous sodium bicarbonate and brine until neutral. Removal of the dried (MgSO$_4$) solvent afforded 4.47 g of crude product which was distilled to afford 3.86 g (29%) of light yellow product, bp 103–115° (0.05 mm). Further purification by redistillation, bp 105° (0.05 mm), and gas chromatography (SE-30, 200°) gave ketone **6**: $n^{25.5}$D 1.5033; ir (film) 5.81, 7.19, 7.59, 8.11 μ; nmr (CDCl$_3$) τ 9.20 (CH$_3$).

Anal. Calcd for C$_{16}$H$_{26}$O: C, 81.99; H, 11.18. Found: C, 82.1; H, 11.2.

The semicarbazone was recrystallized from ethanol as a microcrystalline powder, mp 219–220°.

Anal. Calcd for C$_{17}$H$_{29}$N$_3$O: C, 70.06; H, 10.03; N, 14.42. Found: C, 70.2; H, 10.2; N, 14.35.

trans-**3-Isobornylcyclohexanol (7).**—The procedure of Henbest *et al.*,[11a] was employed. A solution of 0.52 g (2.2 mmol) of isobornylcyclohexanone **6**, 40 mg (0.12 mmol) of chloroiridic acid (Alpha), 0.55 ml of trimethyl phosphite, and 1.0 ml of water in 7 ml of isopropyl alcohol was refluxed for 100 hr.[13b] Isolation (ether)[13f] afforded 0.7 g of crude product which was distilled to

(12) We want to thank Dr. E. Demole for a sample of *trans*-3-(*exo*-5-isocamphyl)cyclohexanol.

(13) (a) The prefix *dl* is omitted from the names of racemic substances. (b) The apparatus described by W. S. Johnson and W. P. Schneider [*Org. Syn.*, **30**, 18 (1950)] was used to maintain a nitrogen atmosphere. (c) Infrared spectra were determined on a Perkin-Elmer Model 237 spectrophotometer; nmr spectra were determined with a Varian Model HA-100 spectrometer by R. Reavill and associates of these laboratories [chemical shifts measured relative to tetramethylsilane (τ 10)]; gas–liquid partition chromatography was accomplished with an Aerograph Model 202B using a flow rate of 100 cc/min on 5 ft × 0.25 in. columns packed with 20% FFAP on 60–80 Chromosorb P or 20% SE-30 on 60–80 Chromosorb W at the temperature indicated unless otherwise specified. (d) Microanalyses were performed by Spang Microanalytical Laboratory, Ann Arbor, Mich. (e) Anhydrous pyridine was obtained by distillation from barium oxide. (f) The isolation procedure consisted of thorough extraction with the specified solvent, washing the combined extracts with brine solution, and drying over anhydrous magnesium sulfate. The solvent was removed from the filtered extracts under reduced pressure on a hot water bath. (g) Melting points are corrected.

(14) Benzyloxy-1-bromobenzene (**2**), mp 55–57°, was prepared according to Y. Wu, W. A. Gould, W. G. Lobeck, Jr., H. R. Roth, and R. F. Feldkamp, *J. Med. Pharm. Chem.*, **5**, 752 (1962), in quantitative yield. Pure material, mp 58–59°, could be obtained in 90% yield by recrystallization from methanol.

(15) E. Vowinkel, *Chem. Ber.*, **99**, 1479 (1966); *Chem. Abstr.*, **65**, 2159d (1966).

(16) K. Bowden, I. M. Heilbron, E. R. H. Jones, and B. C. L. Weedon, *J. Chem. Soc.*, 39 (1946).

give 0.5 g (95%) of colorless, viscous oil, bp 120–130° (bath) (0.1 mm). Gas chromatographic examination utilizing a 20 ft × $^1/_8$ in. column packed with 10% FFAP on Aeropak 30, 80–100, indicated less than 5% of the cis isomer 8. The major product impurity (~10–15%) has been tentatively identified as bornyl isomer 10 (trans). The trans isomer 7 on further purification (FFAP, 200°) exhibited the following properties: n^{20}D 1.5115 (lit.[3] n^{20}D 1.5112); ir (film) 2.98, 7.19, 10.3 μ; nmr (CDCl₃) τ 6.05 (m, 1, CHOH), 7.33 (s, 1, OH), 9.10–9.40 (complex, CH₂'s).

Anal. Calcd for $C_{16}H_{28}O$: C, 81.29; H, 11.94. Found: C, 81.4; H, 12.0.

The 3,5-dinitrobenzoate derivative after several crystallizations exhibited mp 101–103° (lit.[3] 107–108°). Iridium tetrachloride[11b] could be used in place of chloroiridic acid with comparable results.

Registry No.—4, 26988-38-5; 3,3'-dibenzyloxybiphenyl, 26988-39-6; 6, 26988-40-9; 6 semicarbazone, 24739-52-4; 7, 24739-41-1.

Acknowledgments.—The technical assistance of Mr. Kerry M. Fitzpatrick on part of this work is gratefully acknowledged.


5,6-Dibromoacenaphth[5,6-cd]-1,2-oxathiole 2,2-Dioxide. A Potential Sulfene Precursor

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Chemistry Department, Trinity University, San Antonio, Texas 78212

The evidence for a sulfene intermediate generated through photochemical processes is limited.[1–4] We report the synthesis of 5,6-dibromoacenaphth[5,6-cd]-1,2-oxathiole 2,2-dioxide (1) and adduce evidence that implicates the ketosulfene intermediate (2) in photochemical reactions.
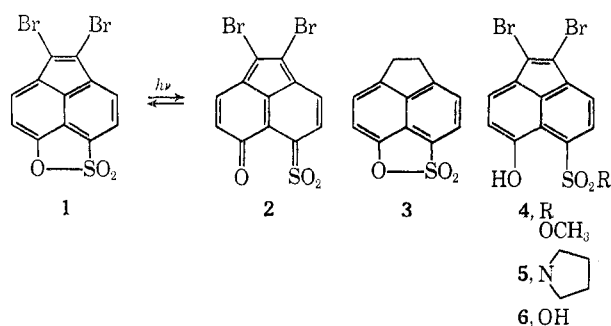
Acenaphthene sultone (3),[5,6] was treated with 4 equiv of NBS, and the product was isolated in the usual manner.[7] After recrystallization from acetonitrile, a

62% yield of bright red needles of 1, mp >300°, was obtained. An ethereal solution of 1 when treated with excess pyrrolidine instantly produced an intense purple solution. Evaporation of the ether gave a purple solid that upon heating to 85° (1 mm) in an Abderhalden changed to a deep red solid. Preparative tlc of the substance yielded compound 5, mp 180–181°. The rapid reaction of 1 with pyrrolidine compared to the slow reaction of 3 with pyrrolidine[8] suggests the existence of ring strain in the ground state of 1. The purple color is undoubtedly due to the formation of the anion of 5 because the color can be reversibly generated by repeated acid and base treatment of 5.[9]

A 200-mg sample of 1 was irradiated in 500 ml of absolute methanol using the standard Hanovia 450-W immersion apparatus, Pyrex filter, and continuous nitrogen purge, and maintaining a temperature of 18–20°. A portion of the original reaction mixture was set aside in an opaque container. Periodic analysis of the irradiation reaction by tlc showed that compound 1 disappeared within 30 min. The control mixture maintained in the dark showed no change during the same interval. Removal of the methanol from the irradiated mixture by vacuum rotary evaporation produced 170 mg of deep red 4 that after crystallizing from hexane had mp 107–108°.

A methanolic solution of 1 was allowed to stand undisturbed for 2 weeks in an opaque container. Vacuum rotary evaporation of this solution left a brown residue whose aqueous solution was acidic to Hydrion paper[10] and formed a precipitate upon treatment with $BaCl_2$ solution. Although the brown residue was not further characterized, its properties reflect those to be associated with 6.

Reaction of 1 with methanolic sodium methoxide followed by neutralization with acid produced a deep red compound whose physical and chemical properties were identical with those of compound 4. The SN2 ring-opening process of sultones is well documented.[11,12] Thus, this ground-state reaction verifies the structure of 4.

The differences between ground-state and excited-state solvolysis reactions of 1 are obvious. Although a variety of reactive species in the excited state can be envisioned, we currently favor the ketosulfene 2 as a reactive intermediate derived from an excited singlet-state process. Intermediate 2 is structurally analogous to the proposed diketene intermediates derived from the excited-state chemistry of pyracyloquinone.[13,14]

Additional corroborative evidence for our proposal is a comparison of the irradiation reaction of 1 with acrylonitrile to the irradiation of 1,2-dibromoacenaphthylene in acrylonitrile. No acrylonitrile polymer was found when 20 mg of 1 was irradiated in 5 ml of acrylonitrile through Pyrex during an 8-hr interval. Under similar conditions 1,2-dibromoacenaphthylene causes

(1) J. F. King, P. de Mayo, E. Morkved, A. B. M. A. Sattar, and A. Stoessel, *Can. J. Chem.*, **41**, 100 (1963).

(2) J. F. King and T. Durst, *ibid.*, **44**, 1859 (1966).

(3) R. J. Mulder, A. M. van Leusen, and J. Strating, *Tetrahedron Lett.*, 3057 (1967).

(4) J. L. Charlton and P. de Mayo, *Can. J. Chem.*, **46**, 55 (1968).

(5) The rigorous *Chemical Abstracts* name for **3** is 5,6-dihydroacenaphth-[5,6-cd]-1,2-oxathiole 2,2-dioxide.

(6) M. T. Bogert and R. B. Concklin, *Collect. Czech. Chem. Commun.*, **5**, 187 (1933).

(7) This procedure is similar to that for the synthesis of 1,2-dibromoacenaphthylene: B. M. Trost and D. R. Britelli, *J. Org. Chem.*, **32**, 2620 (1967).

(8) Compound **3** was quantitatively recovered after standing for 1 hr admixed with an ethereal solution of excess pyrrolidine.

(9) This result is in accord with the observations[1] made by de Mayo and coworkers upon structurally similar compounds.

(10) Under similar conditions de Mayo and coworkers[1] found "acidic" products resulting from reaction of their sultones.

(11) A. Mustafa, *Chem. Rev.*, **54**, 195 (1954).

(12) O. R. Zaborsky and E. T. Kaiser, *J. Amer. Chem. Soc.*, **92**, 860 (1970).

(13) B. Trost, *ibid.*, **91**, 918 (1969).

(14) F. M. Beringer, R. E. K. Winter, and J. A. Castellano, *Tetrahedron Lett.*, 6183 (1968).