

89937-18-8; 18 (isomer 1), 90024-43-4; 18 (isomer 2), 90024-44-5; 20 (isomer 1), 89937-21-3; 20 (isomer 2), 90024-45-6; 21, 89937-20-2; Cl(CN)C=C=O, 60010-89-1; cyclohexene, 110-83-8; 2-methyl-2-butene, 513-35-9; 2,3-dimethyl-2-butene, 563-79-1; (Z)-3-hexene, 7642-09-3; trans-3-hexene, 13269-52-8; styrene, 100-42-5; cyclo-

pentadiene, 542-92-7; 3,4-dihydro-2H-pyran, 110-87-2.

Supplementary Material Available: Spectral data and further experimental details (7 pages). Ordering information is given on any current masthead page.

Factors Conducive to the Cascade Rearrangement of Sterically Congested and Geometrically Restricted Three-Membered Rings. Facile Synthesis of a Topologically Nonplanar Heterocycle

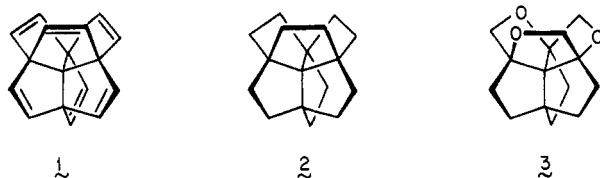
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The two triepoxide stereoisomers of 2,8,9-trimethylene[3.3.3]propellane rearrange under the influence of Lewis acids or heat to the topologically nonplanar trioxahexaquinane 3. The same involvement of the ensemble of three-membered rings in diepoxides 12 and monoepoxide 13 has not been realized. Whereas the former lead to a myriad of unidentified products under a variety of conditions, 13 was found to isomerize cleanly to aldehyde 14 in the presence of anhydrous zinc chloride. Although these developments signaled that cyclopropane rings are less prone to engage in the necessary cationic cascade, it proved possible to induce hydrative threefold cyclopropane ring cleavage in 9 by means of trifluoromethanesulfonic acid in dichloromethane solution with formation of an alcohol provisionally formulated as 15. Mechanistic considerations are presented where relevant.

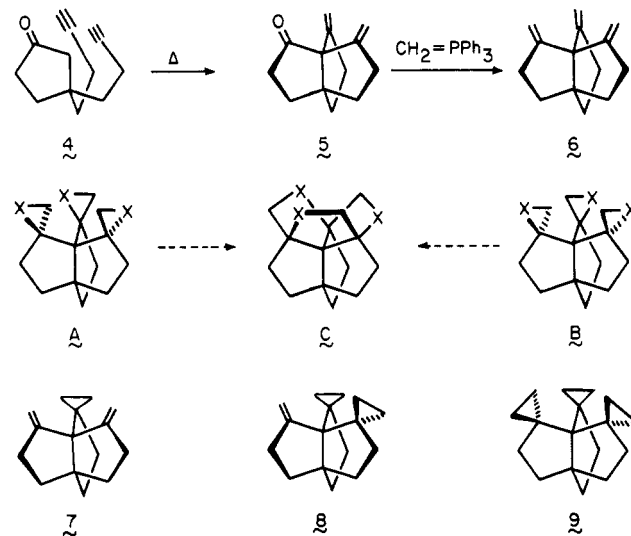
Some time ago, we called attention to the maximally unsaturated hydrocarbon 1 to which the name C_{17} -hexaquinacene was assigned.¹ This molecule compresses the



structural elements of [3.3.3]propellatriene and triquinacene into a highly compact spherical construction. Furthermore, the unusual connectivity in 1 causes it to be topologically nonplanar,^{2,3} an unusual three-dimensional property that brings into being a new kind of isomerism.⁴ Since the rare structural characteristic in question is molecularly based, the perhydrohexaquinane 2 and heterocyclic derivatives such as 3 also cannot be depicted in two dimensions without intercepting bonds. In addition, 3 is noninterconvertible with its mirror image. Since this group of molecules represents one of the simplest examples of topological nonplanarity,⁵ synthetic efforts were directed to their acquisition.

To this end, we were attracted to developments emanating from Conia's laboratory which showed that access

to ring-functionalized [3.3.3]propellanes (e.g., 5 and 6) could be gained by twofold ene cyclization of cyclopentanone 4 at elevated temperatures.⁶ Particularly in-



triguing to us was the possibility that homologation of these molecules as in A or B might subsequently set the stage for reorganization of the three-membered rings under proper reaction conditions to give C. At this point in time, Professor Conia informed us of his successful conversion of 6 to a mixture of 7-9 by means of the Simmons-Smith reaction.⁷ Importantly, the acquisition of 9 demonstrated that steric factors would not preclude access to highly strained hexacyclic molecules of this type.

(1) Paquette, L. A.; Snow, R. A.; Muthard, J. L.; Cynkowski, T. *J. Am. Chem. Soc.* 1978, 100, 1600.

(2) (a) Balaban, A. T. "Chemical Applications of Graph Theory"; Academic Press: New York, 1976; p 84. (b) Harary, F. "Graph Theory"; Addison-Wesley: Reading, MA, 1969.

(3) In the terms of graph theory, a graph is classified as *planar* if upon drawing it on a plane no two edges meet except at a vertex. If this is not possible, the graph is considered to be *nonplanar*.

(4) Fox, G. L. *Chem. Eng. News* 1982, 21.

(5) A second, more elaborate example is the Möbius strip molecule tris(THYME): Walba, D. M.; Richards, R. M.; Haltiwanger, R. C. *J. Am. Chem. Soc.* 1982, 104, 3219.

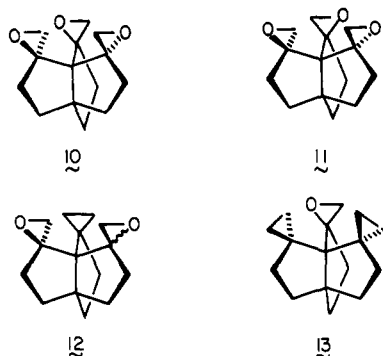
(6) Drouin, J.; Leyendecker, F.; Conia, J. M. *Tetrahedron Lett.* 1975, 4053; *Tetrahedron* 1980, 36, 1203.

(7) Drouin, J. Ph.D. Thesis, Université de Paris Sud, Centre d'Orsay, 1976.

Results

Synthetic Considerations. Tricyclopropyl derivative **9** was prepared according to Drouin.⁷ In an independent investigation, Maggio, Simmons, and Kouba noted that this C_3 -symmetric molecule is in fact fluxional in the temperature range -43 to 147 °C.⁸ By means of ^{13}C and ^1H NMR spectroscopy, activation parameters of $\Delta H^\ddagger = 11.3$ kcal/mol and $\Delta S^\ddagger = 6.1$ eu were determined. Furthermore, indications were obtained that this antipodal interconversion apparently does not proceed through an intermediate of higher (C_{3v}) symmetry.

By making recourse to peracid oxidation, it proved possible to arrive at **10–13** from **6–8**. In the case of **6**,



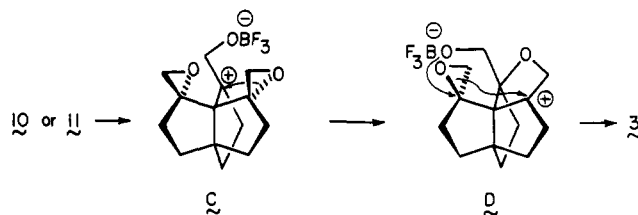
treatment with *m*-chloroperbenzoic acid in dichloromethane at 0 °C produced an ca. 1:1 mixture of stereoisomers **10** and **11**, which could be separated by MPLC on silica gel. The more highly symmetric **10**, which eluted more rapidly, exhibited a well-separated AB pattern (δ 2.65, $\Delta\nu_{\text{AB}} = 0.23$, $J = 3.5$ Hz) for its oxirane protons and a six-line ^{13}C NMR spectrum. In contrast, the $-\text{CH}_2\text{O}-$ absorptions of **11** appear as a series of multiplets in the region δ 2.9–2.42 and a full complement of 14 carbon signals is seen. Maggio et al. have determined that **10** adopts a conformation analogous to that of **9** but with the oxygens occupying the “inner” positions.⁸ Interestingly, the ^{13}C NMR spectrum of **10** remains invariant over the temperature range -80 to 140 °C, indicating the “oxygen-in” conformer to be significantly more stable than any “oxygen-out” form.

Diepoxide **12** was obtained as a mixture of isomers which were not separated. Monoepoxide **13** was available only in limited quantity and its temperature-dependent behavior was not examined.

Rearrangement Reactions. Exposure of cold (near 0 °C) benzene solutions of **10** to catalytic quantities of boron trifluoride etherate or aluminum chloride followed by the addition of water resulted in complete conversion to an isomeric crystalline substance.⁹ The same isomerization was encountered when samples of **10** were heated briefly at 230 – 240 °C; however, competing polymerization was noted under these circumstances. Simmons and Maggio have made similar observations.¹⁰ Since **11** reacted analogously, the rearrangement was clearly independent of epoxide stereochemistry. The ^1H NMR spectrum (in CDCl_3) of the new compound was extraordinarily simple, consisting only of a downfield AB quartet (δ 4.10, $\Delta\nu_{\text{AB}} = 0.46$, $J = 11$ Hz, 6 H) and a narrow upfield multiplet (2.2 – 1.65 , $w_{1/2} = 2.5$ Hz at 60 MHz) of area 12. The high symmetry of the substance was clearly apparent from its

six-line ^{13}C NMR spectrum. All indications pointed to the C_3 -trioxa- C_{17} -hexaquinane **3**, the topology of which is such that the endo and exo protons of the three identical $-\text{CH}_2\text{O}-$ groups are projected into quite different chemical environments while the methylene protons of the [3.3.3]propellane subunit are not.

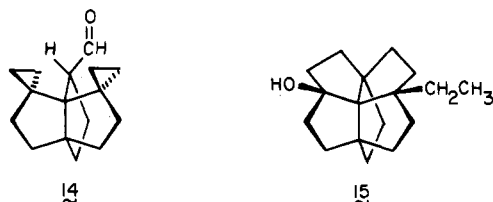
The unusual rearrangement of **10** and **11** to **3**, which materializes from a cascading rupture of an equivalent bond in three different epoxide rings, is undoubtedly made possible by the unique proximity factors contained within these systems. Two reasonable mechanistic pathways orbital symmetry theory, which is founded upon orbital symmetry theory, involves concerted cleavage of the lesser substituted C–O bonds in an unprecedented ($2\sigma + 2\sigma + 2\sigma$) to ($2\sigma + 2\sigma + 2\sigma$) electrocyclization. The second, which is illustrated in C and D, involves more conventional Lewis



acid promoted opening of one oxirane ring so as to induce cationic character at a more highly substituted cyclopentyl carbon. Neighboring-group participation by a second oxygen atom is thereby induced. This process continues intramolecularly until the trioxatriquinane part structure is fully knitted. Our preliminary suggestion⁹ that the latter reaction course is very likely favored has been elegantly confirmed by double-isotope labeling studies based on the perturbation of ^{13}C NMR chemical shifts by isotopes of oxygen.¹¹

The possibility of engaging a cyclopropane ring in the ionic cascade was investigated first with diether **12**. Exposure of this isomeric mixture to Lewis acids such as boron trifluoride etherate or anhydrous zinc chloride resulted in the formation of multicomponent mixtures. Conditions for channeling the isomerization selectively and for eliminating the coproduction of highly polar materials were not uncovered. Analogous complications surfaced when hydrogen chloride in dichloromethane (0 °C, 5 min) or methanesulfonic acid in the same solvent (0 °C, 10 min) were employed.

For these reasons, attention was turned to monoepoxide **13**. In this instance, various experimental settings likewise gave rise to a myriad of products. The lone exception proved to be anhydrous zinc chloride in chloroform which acted on **13** to deliver aldehyde **14** cleanly. On this basis, it seemed that cyclopropane rings are less responsive to involvement in that cationic rearrangement manifold which leads to evolution of the hexaquinane framework.



Accordingly, it was anticipated that **9** would be a relatively unreactive substance, a fact that was subsequently borne out in the laboratory. Despite its recalcitrancy to most reagents, however, **9** was consumed when treated with

(8) Maggio, J. E.; Simmons, H. E., III; Kouba, J. K. *J. Am. Chem. Soc.* 1981, 103, 1579.

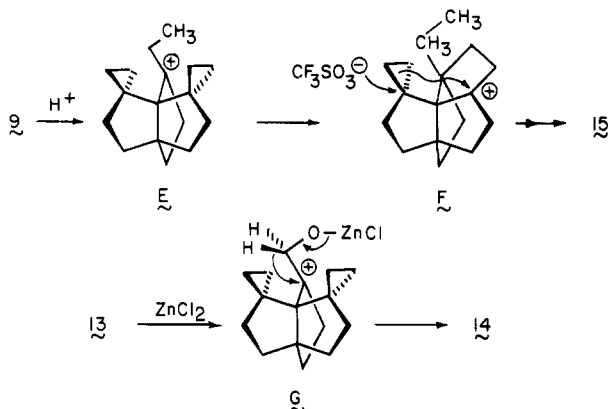
(9) Preliminary communication: Paquette, L. A.; Vazeux, M. *Tetrahedron Lett.* 1981, 22, 291.

(10) Simmons, H. E., III; Maggio, J. E. *Tetrahedron Lett.* 1981, 22, 287.

(11) Benner, S. A.; Maggio, J. E.; Simmons, H. E., III. *J. Am. Chem. Soc.* 1981, 103, 1581.

a small amount of trifluoromethanesulfonic acid in cold (0 °C) dichloromethane solution.¹² Following addition of water and silica gel chromatography, there was isolated a tertiary alcohol which is provisionally formulated as 15. That hydration of the hydrocarbon had occurred was evident from mass spectral (molecular ion at m/e 246) and infrared data (3620 cm^{-1}). The tertiary nature of the hydroxyl group was apparent from the ^1H NMR spectrum (in CDCl_3), which shows, in addition to a hydroxyl proton at δ 5.3, only a series of upfield multiplets in the region δ 2.1–1.0. The unmistakable presence of a methyl triplet (δ 1.05, $J = 6$ Hz) is consistent with the presence of an ethyl group. The ^{13}C NMR spectrum indicates the lack of a symmetry element in the product. These facts, coupled with suitable mechanistic considerations, suggest the alcohol to be 15, although more deep-seated structural rearrangement of its carbocation precursor has not been ruled out. Although 15 is a solid, crystals suitable for X-ray analysis could not be grown; also, its hydroxyl group proved to be too sterically hindered for functionalization.

The pathway to 15 involves the assumption that 9 is first transformed by $\text{CF}_3\text{SO}_3\text{H}$ to E, which subsequently en-



gages into bonding with one of the flanking cyclopropane rings. This process is repeated in F with ultimate nucleophilic capture. The underlying ability of cyclopropane rings to enter into neighboring-group participation has been confirmed in a host of studies¹³ and requires no further comment. The failure of 12 and 13 to undergo comparably smooth isomerization is perhaps less clear. The bottom line may be that initially formed intermediates such as G, have available to them reaction pathways not accessible to E. The lessening of effective Σ -participation capability which attends substitution of oxirane by cyclopropane (compare C and E) provides the opportunity for rechanneling of the operational reaction pathway. As concerns G, for example, conversion to aldehyde 14 by simple 1,2-hydride shift gains kinetic prominence.

The preceding study virtually ensures that the preparation of 2 (and ultimately 1) will not be realized by means of the strategy delineated herein. It becomes necessary to devise alternative schemes for interlacing the triquinane segment of these unusual molecules.

Experimental Section

Epoxidation of 6. To a cold (0 °C) stirred solution of 6⁷ (100 mg, 0.53 mmol) in dry dichloromethane (4 mL) was added during 30 min a solution of *m*-chloroperbenzoic acid (MCPBA) (550 mg

of 80% purity, 2.56 mmol) in the same solvent (5 mL). The reaction mixture was stirred at room temperature for 20 h before being washed with saturated aqueous solutions of sodium sulfite, sodium bicarbonate, and sodium chloride. After drying, the solvent was removed and the residual solid subjected to MPLC on silica gel (elution with 20% ethyl acetate in petroleum ether). There was isolated 60 mg (47%) of 10 and 60 mg (47%) of 11.

For 10: sublimed as white crystals, mp 230–240 °C dec; IR (cm^{-1} , KBr) 3050, 2925, 2860, 1450, 1430, 1378, 1148, 937, 908, 885, 780, 729; ^1H NMR (CDCl_3 , 90 MHz) δ 2.75 (d, $J = 3.5$ Hz, 2 H), 2.53 (d, $J = 3.5$ Hz, 2 H), 2.44–1.20 (series of m, 12 H); ^{13}C NMR (CDCl_3) ppm 65.74, 64.57, 61.71, 46.80, 35.97, 34.91; MS, m/e calcd (M^+) 234.1256, obsd 234.1252.

Anal. Calcd for $\text{C}_{14}\text{H}_{28}\text{O}_3$: C, 71.77; H, 7.74. Found: C, 71.63; H, 7.81.

For 11: colorless needles, mp 211–215 °C dec (from petroleum ether); IR (cm^{-1} , KBr) 3050, 2920, 2860, 1450, 1375, 1290, 1170, 1090, 939, 911, 868, 730; ^1H NMR (CDCl_3 , 90 MHz) δ 2.98–2.49 (m, 6 H), 2.39–1.24 (series of m, 12 H); ^{13}C NMR (CDCl_3) ppm 66.75, 65.98, 65.88, 65.78, 61.95, 49.42, 47.48, 47.19, 37.24, 36.31, 34.52, 34.42, 33.58, 32.72; MS, m/e calcd (M^+) 234.1256, obsd 234.1263.

Anal. Calcd for $\text{C}_{14}\text{H}_{18}\text{O}_3$: C, 71.77; H, 7.74. Found: C, 71.52; H, 7.61.

Epoxidation of 7. Reaction of 7 (79.7 mg, 0.399 mmol) and solid sodium bicarbonate in dichloromethane (5 mL) with MCPBA (210 mg of 85% purity, 2.6 equiv) in the prescribed manner afforded a crude product which was purified by column chromatography on silica gel (elution with 10% ethyl acetate in petroleum ether). The diepoxide mixture 12 (73 mg, 79%) was obtained as a white solid, which sublimed without melting: IR (CCl_4 , cm^{-1}) 3070, 3030, 2960, 1450, 1375, 1290; ^1H NMR (CDCl_3 , 90 MHz) δ 2.95–1.90 (series of m, 4 H), 1.88–0.90 (m, 12 H), 0.75–0.0 (m, 4 H); MS, m/e calcd (M^+) 232.1464, obsd 232.1504.

Epoxidation of 8. Oxidation of 8 (28.5 mg, 0.133 mmol) in dichloromethane (4 mL) with MCPBA (35 mg of 85% purity, 1.3 equiv) afforded a crude product, which was purified by column chromatography on silica gel (elution with 10% ethyl acetate in petroleum ether). Epoxide 13 was obtained as a white solid (24.7 mg, 81%), which sublimes on attempted melting. Attempted purification by VPC led to decomposition: IR (CCl_4 , cm^{-1}) 3080, 3005, 2970, 1450, 1380; ^1H NMR (CDCl_3 , 90 MHz) δ 2.75 (d, $J = 5$ Hz, 1 H), 2.54 (d, $J = 5$ Hz, 1 H), 1.80–1.05 (m, 12 H), 0.95 (m, 2 H), 0.7–0.1 (m, 6 H); MS, m/e calcd (M^+) 230.1670, obsd 230.1692.

Lewis Acid Catalyzed Rearrangement of 10 and 11. A cold (0 °C) solution of 10 and 11 (58.5 mg, 0.25 mmol) in dry ether (1 mL) was treated with a solution containing 0.75 mg of anhydrous aluminum chloride (1 equiv) in the same solvent (2 mL). After 2.3 h, water was added, the layers were separated, and the organic phase was dried and evaporated. The crude product (43 mg) was twice sublimed to give 3 (30 mg) as a colorless solid: mp 322–325 °C dec; ^1H NMR (CDCl_3 , 90 MHz) δ 4.10 (AB q, $\Delta\nu_{\text{AB}} = 0.46$, $J = 11$ Hz, 6 H), 2.2–1.65 (m, 12 H); ^{13}C NMR (CDCl_3) ppm 103.70 (s), 77.73 (t), 65.88 (s), 55.78 (s), 37.04 (t), 33.06 (t).

Anal. Calcd for $\text{C}_{14}\text{H}_{18}\text{O}_3$: C, 71.77; H, 7.74. Found: C, 71.44; H, 7.80.

The analogous result was encountered with boron trifluoride etherate at 0 °C for 1 h.

Lewis Acid Catalyzed Isomerization of 13. A CDCl_3 solution of 13 (8 mg, 0.035 mmol) contained in an NMR tube was treated with a few crystals of anhydrous zinc chloride, and the progress of reaction was monitored spectroscopically. After 28 h at room temperature, complete disappearance of starting material and clean conversion to 14 was noted. The solution was poured into saturated sodium bicarbonate solution and the product was taken up in dichloromethane. The organic phase was washed with water, dried, and carefully evaporated at 0–10 °C. The crude product was purified by column chromatography on silica gel (elution with 10% ethyl acetate in petroleum ether) to give 5 mg (62.5%) of 14 as a colorless solid: IR (CCl_4 , cm^{-1}) 3080, 3010, 2950, 1720, 1550, 1260; ^1H NMR (CDCl_3 , 90 MHz) δ 9.91 (d, $J = 1.1$ Hz, 1 H), 2.0–1.0 (series of m, 13 H), 0.80 (m, 2 H), 0.57–0.14 (m, 6 H); MS, m/e calcd (M^+) 230.1702, obsd 230.1733.

Acid-Catalyzed Hydration of 9. A cold (0 °C) stirred solution of 9 (10–20 mg) in dichloromethane (2 mL) was treated with 1

(12) (a) Takaishi, N.; Inamoto, Y.; Aigami, K.; Fujikura, Y.; Osawa, E.; Kawanisi, M.; Katsushima, T. *J. Org. Chem.* 1977, 42, 2041. (b) Inamoto, Y.; Aigami, K.; Takaishi, N.; Fujikura, Y.; Tsuchihashi, K.; Ikeda, H. *Ibid.* 1977, 42, 3833. (c) Paquette, L. A.; Balogh, D. W. *J. Am. Chem. Soc.* 1982, 104, 774.

(13) Ie Noble, W. J. "Highlights of Organic Chemistry"; Marcel Dekker, Inc.: New York, 1974; pp 751–768.

drop of trifluoromethanesulfonic acid. After 25 min at 0 °C, the reaction mixture was poured into saturated sodium bicarbonate solution and extracted with dichloromethane. The combined extracts were washed with saturated sodium bicarbonate solution and water prior to drying and careful solvent removal at 0-10 °C. Column chromatography of the residue on silica gel (elution with 5% ethyl acetate in petroleum ether) gave an alcohol presumed to be 15 (3-7 mg) as a white solid: mp (unrecrystallized) 120-124 °C; IR (CCl₄, cm⁻¹) 3620, 2930, 1470; ¹H NMR (CDCl₃, 300 MHz) δ 5.3 (br s, 1 H), 2.1-1.0 (series of m with clear triplet at 1.05 (J = 6 Hz)); ¹³C NMR (C₆D₆) ppm 84.02, 58.43, 56.42, 44.67, 36.98,

35.19, 31.17, 28.42, 28.09, 27.28, 26.85, 24.06, 23.64, 11.52 (2C not observed); MS, *m/e* calcd (M⁺) 246.1984, obsd 246.1999.

Acknowledgment. This investigation was made possible by the financial support of the National Science Foundation (Grant CHE 79-00333).

Registry No. 3, 77973-29-6; 6, 58461-87-3; 7, 90047-12-4; 8, 90047-14-6; 9, 77180-63-3; 10, 78037-97-5; 11, 78037-96-4; 12 (isomer 1), 90047-13-5; 12 (isomer 2), 90129-13-8; 13, 90047-15-7; 14, 90047-16-8; 15, 90047-17-9.

1-Nitrobenzotriazole-2-(Nitroimino)diazobenzene Isomerization: Formation of Triazenes by Azo Coupling with Cyclic Amines. Structure Determination and Dynamic NMR

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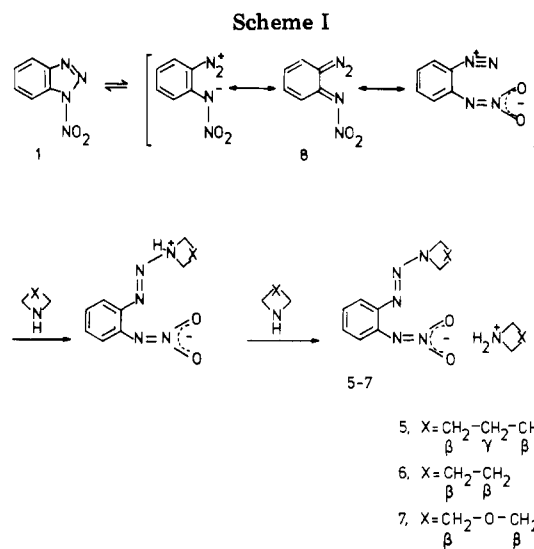
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The reaction of 1-nitrobenzotriazole (1) with the cyclic amines 2-4 affording the triazenes 5-7 revealed ring opening of the 1,2,3-triazole ring of benzotriazole to the isomeric diazo compound 8 responsible for the formation of these triazenes. Crystal analysis of 5 and IR and ¹H and ¹³C NMR spectra allowed the structure assignments for 5-7. From a dynamic NMR study of triazene 6 restricted rotation about the single N-N bond is inferred.

N-Nitroazoles are known to undergo a variety of unexpected and novel reactions, many of which are synthetically useful and mechanistically interesting reactions. Examples of such reactions are the thermal intramolecular rearrangement of the *N*-nitro group to a carbon atom in the azole ring of pyrazoles,¹ 1,2,4-triazoles,² imidazoles, and indazoles³ and the "cine" nucleophilic aromatic substitution of the *N*-nitro group by nucleophiles entering the azole ring at the adjacent carbon atom in pyrazoles,⁴ 1,2,4-triazoles,⁵ and indazoles.⁶ Intermolecular transfer reactions of the *N*-nitro group are also reported.⁸ This paper reports the very facile and novel reaction of *N*-nitrobenzotriazole (1) in acetonitrile solution with cyclic amines to give crystalline secondary alkyl ammonium salts, which are found to be triazenes. Also we present a study of the temperature-dependent ¹H and ¹³C NMR spectra of these salts.

Results and Discussion

Ring-Chain Isomerism. Addition of piperidine (2), pyrrolidine (3), or morpholine (4) at room temperature to a solution of 1-nitrobenzotriazole (1) in acetonitrile results in the precipitation of the crystalline compounds 5-7 in very high yields. Each of these products, according to elemental analysis, consisted of one molecule of 1 and two molecules of the respective cyclic amines 2-4. ¹H NMR spectral analysis revealed two signals in the region of the α -methylene protons. The low-field shift of one of the signals, a striking feature of the NMR spectra of these compounds, was initially interpreted only to indicate that one of the cyclic amines is in the protonated form (however see below). The signals of the aromatic protons, however,



were shifted approximately 0.5 ppm to higher external field as compared to those of 1-nitrobenzotriazole (1). A

* Presented in part at the 6th Lakeland Heterocyclic Chemistry Symposium, Grasmere, England, May, 1983.

(1) (a) Janssen, J. W. A. M.; Habraken, C. L. *J. Org. Chem.* 1971, 36, 3081-3084. (b) Janssen, J. W. A. M.; Koenen, H. J.; Kruse, C. G.; Habraken, C. L. *Ibid.* 1973, 38, 1777-1782. (c) Janssen, J. W. A. M.; Habraken, C. L.; Louw, R. *Ibid.* 1976, 41, 1758. Rufer, C. *Liebigs Ann. Chem.* 1975, 1465-1477. Schofield, K.; Grimmett, M. R.; Keene, B. R. T. "The Azoles"; Cambridge University Press: Cambridge, 1976; pp 234-235.

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(3) Cohen-Fernandes, P.; Habraken, C. L. *J. Org. Chem.* 1971, 36, 3084-3086.