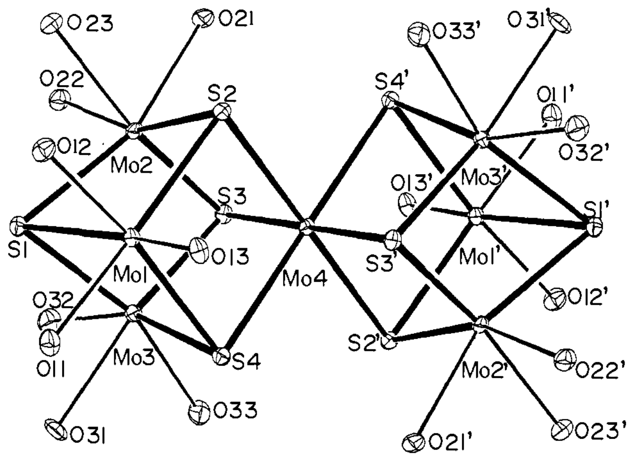

**Figure 1.** Perspective view of $[(H_2O)_9Mo_3S_4MoS_4Mo_3(H_2O)_9]^{8+}$ aqua ion. Bond distances (Å): Mo1–Mo2, 2.773 (2); Mo1–Mo3, 2.766 (2); Mo2–Mo3, 2.770 (2); Mo1–Mo4, 3.026 (1); Mo2–Mo4, 3.051 (1); Mo3–Mo4, 3.060 (1); Mo1–S1, 2.351 (3); Mo1–S2, 2.327 (3); Mo1–S4, 2.331 (3); Mo2–S1, 2.354 (3); Mo2–S2, 2.321 (3); Mo2–S3, 2.325 (3); Mo3–S1, 2.346 (3); Mo3–S3, 2.325 (3); Mo3–S4, 2.323 (3); Mo4–S2, 2.446 (3); Mo4–S3, 2.462 (3); Mo4–S4, 2.447 (3); Mo–O($H_2O$), 2.18 [2]. (The primed atoms are related to the unprimed ones by a center of symmetry.)

X-ray structure analysis[5] of the crystal revealed the existence of a double-cubane-type cluster aqua ion $[(H_2O)_9Mo_3S_4MoS_4Mo_3(H_2O)_9]^{8+}$ (**2**) (Figure 1). Two incomplete cubane-type cores, $Mo_3S_4$'s, are bridged by a molybdenum atom (Mo4) which lies on a center of symmetry. The Mo4–Mo*i* (*i* = 1, 2, 3) bond distances are distinctly longer than Mo1–Mo2, Mo1–Mo3, and Mo2–Mo3. Although many cubane-type cluster compounds with $Mo_4S_4$ cores are known,[6] no double-cubane-type cluster with an $Mo_3S_4MoS_4Mo_3$ core has been reported. It seems that the triangular core of **1** is broken by the reaction with magnesium which is a stronger reducing agent than iron and that the novel double-cubane-type core is constructed by way of some intermediate(s). It should be noted that different double-cubane-type molybdenum–iron–sulfur clusters (e.g., $[Mo_2Fe_6S_8(SR)_9]^{3-}$[7] and $[Mo_2Fe_7S_8(SR)_{12}]^{3-}$[8]) have been prepared, and their properties (electronic spectra, electrochemical behavior, and catalytic activity[9]) had been investigated in detail.

The electronic spectrum of **2**, which is depicted in Figure 2, together with that of **1**, shows $\lambda_{max}$'s at 950 nm ($\epsilon$ 606 ($M^{-1}$ $cm^{-1}$)/Mo), 635 (1266), 518 (1469), 480 (sh, 1295), 416 (1027), and 360 (770). These $\epsilon$ values in the visible and near-infrared region are very large compared to those of **1**. In **2**, as described above, are present two, structurally different types of Mo's whose mean oxidation number is 3.43,[10] whereas in **1** there are three equal Mo atoms, each in the oxidation state of 4.00. On the other hand, the $Mo_4S_4$ cubane-type compounds composed of structurally equivalent Mo atoms do not have such a large $\epsilon$ value, even though some of them have fractional oxidation numbers.[11] Thus, a charge-transfer transition might be responsible for the strong absorption of **2**.

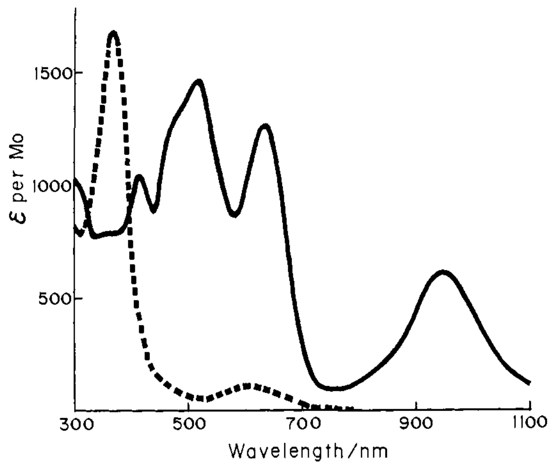

**Figure 2.** Electronic spectra in 1 M HPTS: (—) $[(H_2O)_9Mo_3S_4MoS_4Mo_3(H_2O)_9{}^{8+}$; (---) $Mo_3S_4{}^{4+}$

Compound **2** is fairly resistant toward air oxidation, not only in the solid state but also in solution. The absorbance of a solution of **2** changes by less than few percent in a day.

**Acknowledgment.** This work was supported by a Grant-in-Aid for Scientific Research (No. 59470039) from the Ministry of Education, Science and Culture.

**Supplementary Material Available:** Tables of atomic coordinates, thermal parameters, and bond distances and angles (3 pages). Ordering information is given on any current masthead page.

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(5) Crystal data: triclinic system, space group $p\bar{1}$, $a$ = 15.816 (4) Å, $b$ = 16.589 (4) Å, $c$ = 11.722 (3) Å, $\alpha$ = 97.29 (2)°, $\beta$ = 108.91 (2)°, $\gamma$ = 72.47 (2)°, $V$ = 2772.6 (12) $Å^3$, $Z$ = 1. The structure was solved by direct method (MULTAN) and refined by least squares to a current $R$ value of 0.058 for 5131 reflections ($F_o > 5\sigma(F_o)$).

(6) References cited in ref 1 and; (a) Vahrenkamp, H. *Angew. Chem., Int. Ed. Engl.* **1975**, *14*, 322–329. (b) Vandenberg, J. M.; Brasen, D. *J. Solid State Chem.* **1975**, *14*, 203–208. (c) Muller, A.; Eltzner, W.; Clegg, W.; Sheldrick, G. M. *Angew. Chem.* **1982**, *94*, 555–556. (d) Brunner, H.; Kauermann, H.; Wacher, J. *J. Organomet. Chem.* **1984**, *265*, 189–198. (e) Muller, A.; Jostes, R.; Eltzner, W.; Nie, C.-S.; Diemann, E.; Bogge, H.; Zimmermann, M.; Dartmann, M.; Reinsch-Vogel, U.; Che, S.; Cyvin, S. J. *Inorg. Chem.* **1985**, *24*, 2872–2884. (f) Cotton, F. A.; Diebold, M. P.; Dori, Z.; Llusar, R.; Schwotzer, W. *J. Am. Chem. Soc.* **1985**, *107*, 6735–6736. (g) Shibahara, T.; Kuroya, H.; Matsumoto, K.; Ooi, S. *Inorg. Chim. Acta* **1986**, *116*, L25–L27. (h) Shibahara, T.; Kawano, E.; Okano, M.; Nishi, M.; Kuroya, H. *Chem. Lett.* **1986**, 827–828.

(7) For example: Wolff, T. E.; Berg, J. M.; Hodgson, K. O.; Frankel, R. B.; Holm, R. H. *J. Am. Chem. Soc.* **1979**, *101*, 4140–4150. Simulation of the EXAFS of the unsynthesized double-cubane structure $Fe_3S_4MoS_4Fe_3$ is included in this paper.

(8) For example: Palermo, R. E.; Power, P. P.; Holm, R. H. *Inorg. Chem.* **1982**, *21*, 173–181.

(9) For example: Tanaka, K.; Nakamoto, M.; Tashiro, Y.; Tanaka, T. *Bull. Chem. Soc. Jpn.* **1985**, *58*, 316–321.

(10) Compound **2** is diamagnetic at room temperature: we have no conclusive explanation of this at present.

(11) For example, ref 6g.

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# 2,3-Methano-2,4-didehydroadamantane: A [4.1.1]Propellane Possessing Two Unequivalent Inverted Carbons

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*Received December 22, 1986*

We report the first synthesis and reactivity studies of 2,3-methano-2,4-didehydroadamantane (**2**), a [4.1.1]propellane derivative containing the *trans*-bicyclo[4.1.0]heptane unit.[1] This is the smallest carbocyclic propellane system possessing two

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(1) Certain cyclosteroids also contain this unit.[2a] Recently, a few other derivatives of *trans*-bicyclo[4.1.0]heptane have been prepared.[2b] Its lower homologue, *trans*-bicyclo[3.1.0]hexane, still remains an illusive target.

(2) (a) Cf.: Dauben, W. G.; Wipke, W. T. *Pure Appl. Chem.* **1964**, *9*, 539–553. Dauben, W. G.; Willey, F. G. *Tetrahedron Lett.* **1962**, 893–899. Just, G.; DiTullio, V. *Can. J. Chem.* **1964**, *42*, 2153–2160. Gassman, P. G.; Hymans, W. E. *J. Chem. Soc., Chem. Commun.* **1967**, 795–796. Gassman, P. G. *Ibid.* **1967**, 793–795. (b) Gassman, P. G.; Bonser, S. M. *J. Am. Chem. Soc.* **1983**, *105*, 667–669. Gassman, P. G.; Mlinarič-Majerski, K. *J. Org. Chem.* **1986**, *51*, 2397–2398.

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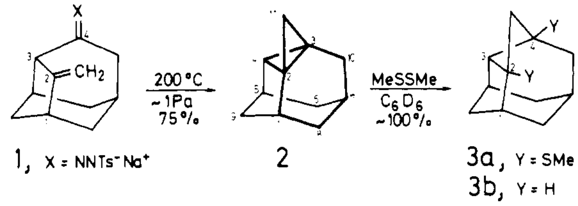
**Scheme I**

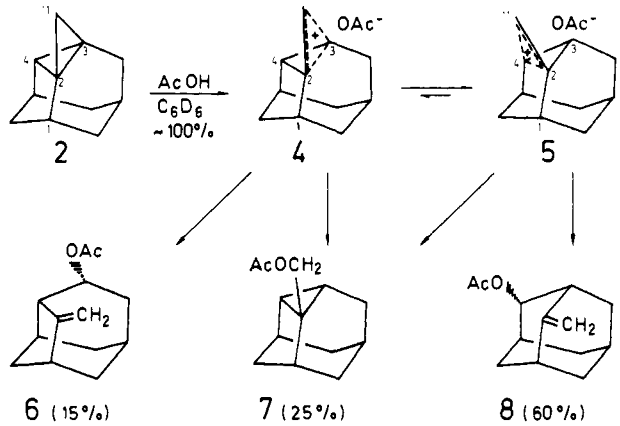


*unequivalent* inverted carbon atoms which has been prepared. The results clearly show that reactivities of such two carbons can be very different.

Propellanes containing six or less bridge carbons have been of considerable recent interest.[3,4-8] They possess two inverted carbon atoms and are highly reactive. Besides being attractive synthetic targets and reactivity study subjects,[4] these propellanes have also been subjects of numerous theoretical calculations,[5] X-ray[6a] and electron-diffraction[6b] analyses, and vibrational[7] and photoelectron[8] spectroscopic studies. The results indicate an increase in electron density outside inverted carbon atoms (not necessarily along the line passing through them) and, consequently, a decrease in electron density between these carbons. According to this qualitative picture, electron densities outside unequivalent inverted carbons in an unsymmetrical propellane should not be equal and, hence, such two carbons will behave differently. To test this hypothesis and provide further insight into the nature of the bond between inverted carbons, we prepared **2**, a prototype propellane with two unequivalent inverted carbons, and studied its chemical behavior.

Propellane **2** was prepared in 75% yield by the intramolecular cycloaddition of 2-methylene-4-protoadamantylidene to the olefinic bond (Scheme I). The carbene was generated by pyrolysis of dry tosylhydrazone sodium salt of 2-methylene-4-protoadamantanone[9] (**1**) at 200 °C in vacuo. The propellane was trapped in a liquid-nitrogen cooled trap. It was more than 95% pure by $^{13}C$ NMR.

**Scheme II**



The structure of **2** was established by spectral means and confirmed by its conversion into a known compound (vide infra). In particular, the mass spectrum showed a molecular ion peak at $m/z$ 146 (55%), the IR spectrum exhibited the characteristic cyclopropane C–H vibrational band at 3010 $cm^{-1}$, and the $^1H$ NMR spectrum displayed two multiplets at $\delta$ 2.7–2.1 (4 H) and 1.9–0.8 (10 H). The $^{13}C$ NMR spectrum unambiguously established the structure of **2**: $\delta$ ($C_6D_6$) 58.7 (d, $J$ = 174 Hz, $C_4$), 52.3 (t, $J$ = 131 Hz, $C_9$), 39.0 (dd, $J$ = 149, 170 Hz, $C_{11}$), 31.6 (t, $J$ = 128 Hz, $C_6$ or $C_8$), 31.4 (d, $J$ = 137 Hz, $C_1$ or $C_5$), 30.7 (t, $J$ = 127 Hz, $C_6$ or $C_8$), 30.5 (t, $J$ = 127 Hz, $C_{10}$), 30.2 (d, $J$ = 138 Hz, $C_1$ or $C_5$), 26.9 (d, $J$ = 136 Hz, $C_7$), 18.5 (s, $C_2$), 10.6 (s, $C_3$). The C–H coupling constants of the dd signal at $\delta$ 39.0 are typical of a methylene carbon in the bicyclobutane system.[11a] The other $^{13}C$ NMR signals were assigned by using their characteristic chemical shifts,[11b] the splitting patterns in the proton off-resonance decoupled spectra, the C–H coupling constants, and the $^1\Delta$ and $^2\Delta$ deuterium isotope effects on carbon-13 chemical shifts[11c] of 2,3-methano-2,4-didehydroadamantane-*10,10-$d_2$* [$\delta$ 30.5 (−870 ppb, $^1\Delta$), 26.9 (−200 ppb, $^2\Delta$), 10.6 (−170 ppb, $^2\Delta$)].

Propellane **2** is thermally rather stable. No decomposition was detected (by $^{13}C$ NMR) after 24 h at 80 °C in a dry toluene-$d_8$ solution under a nitrogen atmosphere. At room temperature, in a benzene-$d_6$ solution, **2** was entirely inert toward nucleophiles, such as diethylamine, but highly reactive toward free radicals and electrophiles.

With dimethyl disulfide, **2** reacted instantaneously with an apparent addition across the bond between inverted carbons (Scheme I). The only product was 2,4-bis(methylthio)-2,4-methanoprotoadamantane (**3a**).[9c] No rearranged product was detected (by $^{13}C$ NMR), which indicates that this was a free radical process.[12] Disulfide **3a** was readily (68%) desulfurated

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(9) (a) The synthesis of 2-methylene-4-protoadamantanone (**1**, X = O) originated with 2-protoadamantenone.[10] It was converted (78%) into 2-*endo*-acetoxy-2-*exo*-methylprotoadamantene by treatment with methyl Grignard reagent followed by acetylation. Hydroboration of this olefin with siamylborane followed by oxidations with $H_2O_2/OH^-$ and Jones reagent furnished 78% of a 9:1 mixture of 2-*endo*-acetoxy-2-*exo*-methyl-4-protoadamantanone and -5-protoadamantanone. Pyrolysis of this mixture at 600–650 °C in vacuo yielded 90% of a 9:1 mixture of **1**, X = O, and its isomer with the carbonyl group at position 5. Omitting the Jones oxidation in this sequence resulted in a 9:1 mixture (75%) of 2-methylene-4-*exo*-protoadamantanol and -5-*exo*-protoadamantanol. (b) Acetates **6** and **7** were prepared by acetylation of the corresponding alcohols.[4h,9a,c] (c) Satisfactory elemental analyses and adequate $^{13}C$ NMR, $^1H$ NMR, IR, and/or mass spectra were obtained for all new compounds.

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(12) Huyser, E. S. *Free Radical Chain Reactions*; Wiley: New York, 1970; pp 148–151.

with Raney nickel to 2,4-methanoprotoadamantane (**3b**), which was identified by comparison of its $^{13}$C NMR, $^{1}$H NMR, IR, and mass spectra with spectral data reported for this hydrocarbon.[13] This strongly confirms the structure established for **2**.

The central bond in **2** was readily cleaved by electron-transfer hydrogenation with lithium in an ethylamine–hexane mixture at −30 °C.[14] The only product was 2,4-methanoprotoadamantane (**3b**) (80%). Catalytic hydrogenolysis with Pd/C, however, gave a mixture of products. With oxygen, **2** reacted slowly producing a polymeric material, which was scarcely soluble in toluene-$d_8$.

Glacial acetic acid reacted with **2** instantaneously and exothermally, yielding almost quantitatively (by $^{13}$C NMR) a mixture of 4-*exo*-acetoxy-2-methyleneprotoadamantane (**6**), 2-(acetoxymethyl)-2,4-didehydroadamantane (**7**), and 2-*anti*-acetoxy-4-methyleneadamantane (**8**) (Scheme II). Products **6–8** were identified by comparison of their $^{13}$C NMR and $^{1}$H NMR spectra with those of authentic samples.[4h,9b] The major product[4h] (**8**) was previously isolated by GC, while **6** and **7** were identified in product mixtures enriched with them. The mechanism presumably involves a proton attack on carbon 3 of the propellane central bond leading to bicyclobutonium protoadamantyl cation **4**. (If the proton attacked carbon 2, rather than 3, other products would be formed instead of **6–8**.) The exclusive formation of the exo epimer of homoallyl acetate **6**, along with the formation of cyclopropylcarbinyl acetate **7**, strongly suggests that this cation is bridged. The adamantyl acetate **8**, however, cannot be formed directly from **4**. It is probably formed from bicyclobutonium adamantyl cation **5** by the attack of an acetoxy anion on carbon 4 from the side unhindered by bridging. Cations **4** and **5** are in a fast equilibrium. Owing to its adamantane structure, the latter should be more stable. Cyclopropylcarbinyl acetate **7** can arise from either of the two bridged cations (**4** and/or **5**) by attack on carbon 11.

In conclusion, it should be noted that the proton attacks exclusively one (carbon 3) of the two unequivalent inverted carbon atoms in the starting propellane **2**. Neither of these two carbons appears to be substantially more sterically hindered than the other, and the resulting carbocation **4** should be more distorted from planarity (higher in energy) than a carbocation that would arise by the attack on carbon 2.[15] This is consistent with our starting hypothesis of different electron densities near unequivalent inverted carbons in unsymmetrical propellanes such as **2**.[16] The electron density outside carbon 3 should be higher than that outside carbon 2, while between these two carbons it is most probably lower than outside either of them.

**Acknowledgment.** This investigation was supported by a grant from the Research Council of the Republic of Croatia (SIZ II) and US–Yugoslav Cooperative Program PN-531 (NSF). We thank Dr. K. Mlinarić-Majerski for an authentic sample of 2-(hydroxymethyl)-2,4-didehydroadamantane.

**Registry No. 1**, 107616-71-7; **2**, 107616-72-8; **3a**, 107616-73-9; **3b**, 55638-02-3; **6**, 107616-74-0; **7**, 87433-57-6; **8**, 87433-51-0; MeSSMe, 624-92-0.

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(15) This was estimated by careful examinations of molecular models of **2** and the relevant carbocations.

(16) Electron densities near unequivalent inverted carbons in some other unsymmetrical propellane may, naturally, be less different or even equal.

# *Book Reviews*[*]

**Crystallographic Computing. Volume 3. Data Collection, Structure Determination, Proteins, and Databases.** Edited by G. M. Sheldrick (University of Göttingen) and C. Krüger and R. Goddard (Max-Planck-Institute). Oxford University Press: New York. 1985. ix + 314 pp. $35.00. ISBN 0-19-855211-4.

This book containing 33 papers from an international summer school in Germany in 1984 is aimed at specialists. The descriptions of crystallographic databases and some suggestions for speeding up electronic computations may also have interest for some who are not crystallographers. A discussion of least-squares methods by D. Watkin clarifies important points relevant to any type of experiment which involves a large data set and a complicated model. Most of the rest concerns highly specialized practical details of experiment and analysis in crystal structure research or descriptions of particular software systems and computer hardware. It is rich in useful hints for better technique and warnings of errors and pitfalls. The authors are experts. This volume is highly recommended to others who are, and to those who aspire to become, experts in structure analysis.

**David H. Templeton**, *University of California, Berkeley*

## Volumes of Proceedings

**Development of Drugs and Modern Medicines: A Conference To Honour Professor Arnold H. Beckett.** Edited by J. W. Gorrod, G. G. Gibson, and J. Mitchard. VCH Publishers: New York. 1986. x + 669 pp. $163.00. ISBN 0-89573-557-1

This "Festschrift" contains 63 papers delivered at a 1985 conference held in Chelsea, U.K. The subjects range from design to evaluation, metabolism, toxicity, delivery, and pharmacokinetics. Indexed.

**Stereochemistry of Organic and Bioorganic Transformations.** Edited by Wilhelm Bartmann and K. Barry Sharpless. VCH Publishers: New York. 1987. xii + 330 pp. $54.00. ISBN 0-89573-607-1

The Editor's preface begins with these statements: "The gathering, distribution, and cataloging of the ever increasing scientific information can be solved with the instruments of the computer age. However, the selection and classification of those results which determine the frontiers of scientific progress must remain the privilege of a few." These thoughts were presumably the guiding spirit in the selection of an outstanding group of chemists, who have contributed 18 papers, drawn from a conference held in Schloss Reisenburg in 1986. They are reproduced from typescript and are accompanied by a good index.

**Surface and Interface Analysis. Volume 9.** John Wiley & Sons: New York. 1986. iv + 522 pp. $160.00. ISBN 0471-91160-7

Proceedings of a European Conference on applications of the title subject, held in The Netherlands in 1985, consisting of a large number of papers and short abstracts, under the general headings Techniques, Quantification, Metallurgy, Catalysis, Electronics, Semiconductors and Thin Films, Corrosion, Polymers, Adhesion, Ceramics, and Tribology and Environmental. The papers are nicely set in type, and there are many photographic illustrations. There is an index only of contributors.

**Organic Pollutants in Water: Sampling, Analysis, and Toxicity Testing.** Edited by I. H. (Mel) Suffet and Murugan Malaiyandi. American Chemical Society: Washington, DC. 1987. xvi + 797 pp. $94.95. ISBN 0-8412-0951-0

The symposium from which this book was developed took place in Philadelphia in 1984. It contains 36 papers and the transcript of a panel discussion, all of which have gone through more than the usual editorial attention, and are, appropriately, set in type. The subject headings are the following: Protocols; Reverse Osmosis to Isolate Organic Pollutants from Water; Synthetic Polymers for Concentrating Organic Chemicals from Water; Novel Methods to Isolate and Fractionate Organic Chemicals in Water Samples; Comparison of Isolation Methods; Case Histories: Biological Testing; and Toxicological Testing–Analysis Interface. The overall picture drawn of what we drink is unsettling but fascinating. The index is uncommonly thorough.

**Bioactive Analytes, Including CNS Drugs, Peptides, and Enantiomers.** Edited by Eric Reid, Bryan Scales, and Ian D. Wilson. Plenum Press: New York. 1986. xiv + 422 pp. $69.50. ISBN 0-306-42400-2

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[*] Unsigned book reviews are by the Book Review Editor.