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A Vicarious One-Pot Transformation of a Fluorocyclitol to a Polycyclitol: Observation of a Formal 4-Fold Axial−Equatorial Epimerization on a Conformationally Locked Scaffold

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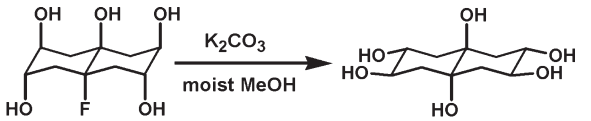
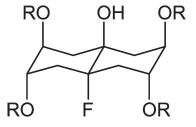


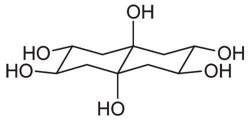
A conformationally locked fluoropentol undergoes an interesting transformation to (*trans*,*anti*,*trans*,*anti*,*trans*)-perhydro-2,3,4a,6,7,8a-naphthalenehexol essentially under conditions of base-induced transesterification. The proposed rationale for the observed metamorphosis involves a nucleophilic displacement of fluoride, and subsequent stereo- and regioselective anti-Fürst−Plattner-type ring-opening of the epoxide thus formed.

Organofluorine compounds have been known to display intriguing structural and physicochemical characteristics.[1] Indeed, the moniker “flustrates”, as coined by Seebach for fluorine-containing substrates, was largely inspired by the often noted unanticipated physicochemical responses of fluorine derivatives in an otherwise straightforward chemical transformation.[2] In this context, the present report deals with a novel observation, which adds to the existing repertoire of abnormal albeit interesting characteristics displayed by many organofluorine compounds. We document herein an intriguing metamorphosis of a conformationally locked fluoropentol **1** to the (*trans*,*anti*,*trans*,*anti*,*trans*)-perhydro-2,3,4a,6,7,8a-naphthalenehexol **2**, mediated by an unexpectedly facile expulsion of fluorine in a bridgehead fluorohydrin to an epoxide intermediate under mild basic conditions. It needs to be highlighted that acquisition of a 1,2-diol moiety in a *trans*-diequatorial configuration on a conformationally locked *trans*-decalin scaffold would entail circuitous synthetic maneuvers that are, by no means, trivial.

Formation of the hexol **2** as a rather insoluble microcrystalline solid was first observed rather serendipitously during an attempted transesterification (K_2CO_3/MeOH milieu) of the tetra-acetate derivative **3** of the fluoropentol **1**.[3] Though seemingly bizarre at the outset, the extrusion of the fluoro moiety and the apparent axial−equatorial epimerization of all the four secondary hydroxy groups in a conformationally locked scaffold could eventually be reconciled (Scheme 1).



1: R = H, **3**: R = Ac



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Though alternate mechanistic viewpoints and likely intermediates can be considered to account for the unusual transformation of **1** to **2**, our putative rationale is that the *trans*-fluorohydrin moiety in **1**, formed from **3** during transesterification, undergoes base-induced fluorine displacement to furnish the epoxide moiety in the intermediate **4**.[4] As indicated by DFT-based geometry optimization performed at the B3LYP/6-31+G(d) level,[5] the epoxytetrol **4** has a folded structure that is stabilized by an intramolecular O−H···O hydrogen bond between the two hydroxy groups, *anti* to the epoxide (Figure 1). The folded molecular topology of **4** renders position “a” of the epoxide functionality significantly more sterically encumbered to nucleophilic attack and facilitates, in consequence, an otherwise less favorable anti-Fürst−Plattner-type epoxide ring-opening in **4** by OH^- at position “b”.[6,7] This engenders the hexol **2a**, bearing the

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(4) Formation of an epoxide from a bridgehead fluorohydrin remains intriguing from a mechanistic point of view, and one can only speculate in terms of a S_N1 or even less likely S_N2 process. However, there is precedence, although somewhat occasional, for the formation of an epoxide from a fluorohydrin moiety under various reaction regimes. See: (a) Yamaguchi, H.; Schuerch, C. *Carbohydr. Res.* **1980**, *81*, 192. (b) Bridge, A. W.; Morrison, G. A. *J. Chem. Soc., Perkin Trans. 1* **1983**, 2933. (c) Alvernhe, G.; Laurent, A.; Haufe, G. *J. Fluorine Chem.* **1986**, *34*, 147. (d) Corey, E. J.; Yi, K. Y.; Matsuda, S. P. T. *Tetrahedron Lett.* **1992**, *33*, 2319. (e) Corey, E. J.; Lee, J. *J. Am. Chem. Soc.* **1993**, *115*, 8873. (f) Chambers, R. D.; Gilani, A. H. S.; Gilbert, A. F.; Hutchinson, J.; Powell, R. L. *J. Fluorine Chem.* **2000**, *106*, 53. (g) van Steenis, J. H.; van der Gen, A. *Eur. J. Org. Chem.* **2001**, 897. (h) Sundby, E.; Holt, J.; Vik, A.; Anthonsen, T. *Eur. J. Org. Chem.* **2004**, 1239.

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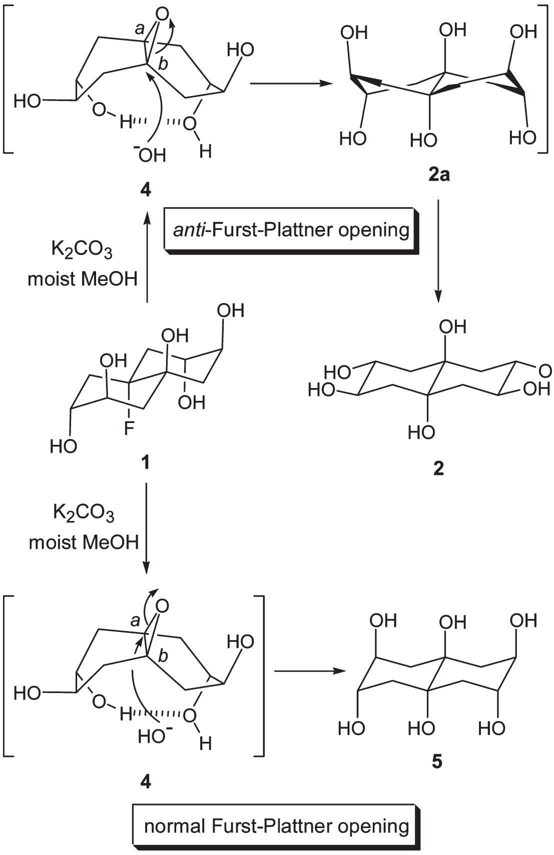
SCHEME 1. Mechanism Proposed to Account for the Formation of the Hexol 2 from the Fluorocyclitol 1



1,2-transdiaxial secondary hydroxy substituents in a high energy twist-boat conformation. Relaxation of the carbocyclic skeleton in **2a** to a chair conformation in **2** brings the secondary OH functionalities to the observed tetraequatorial orientation. It is worth noting at this point that a normal epoxide opening in **4** via OH^- attack at position "a" would lead to the formation of the all-*axial* naphthalenehexol **5**, whose synthesis and in-depth characterization has already been reported by us (Scheme 1).[8]

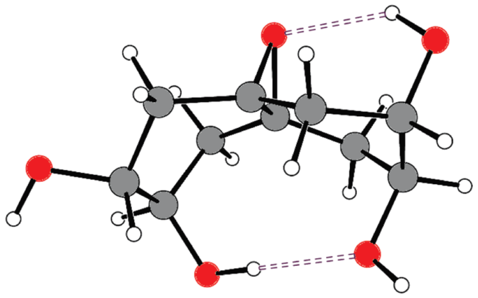


FIGURE 1. Energy optimized geometry of the epoxytetrol **4**, as computed at the B3LYP/6-31+G(d) level. The purple dotted lines indicate intramolecular O−H···O hydrogen bonds.

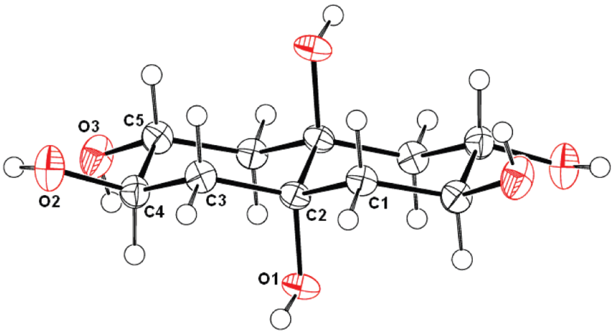


FIGURE 2. ORTEP diagram of the hexol **2** with atom numbering for the asymmetric unit. Displacement ellipsoids have been drawn at 50% probability level, and H atoms are shown as small spheres of arbitrary radii.

In order to test the feasibility of our proposition, a pure recrystallized sample of the fluoropentol **1** was treated with K_2CO_3 in methanol under ambient conditions. To our delight, the reaction proceeded to completion (as indicated through TLC analysis) in just over 1 h, and the hexol **2** could easily be isolated from the reaction mixture in almost quantitative yield. Although the *trans-anti-trans-anti-trans* relationship of the hydroxy groups in the C_{2h}-symmetric hexol **2** could be discerned from its NMR spectral data, it appeared prudent, given the unconventional nature of its formation, to confirm the stereostructure of **2** through single crystal X-ray diffraction studies. Implicit in this endeavor was also an opportunity to study the solid-state self-assembly of **2** and glean a possible rationale for its unusually high melting point (> 300 °C) and near insolubility in most organic solvents as compared to its known diastereomers.[8,9]

Single crystals of the hexol **2** were obtained as small plates by slow evaporation of its solution in deionized water under ambient temperature and pressure. The crystal structure of **2** was solved in the centrosymmetric monoclinic space group $P2_1/c$ ($Z = 2$), with the C_{2h} symmetric hexol molecules occupying the inversion centers at (1/2, 0, 0) and (1/2, 1/2, 1/2) (Figure 2). Employing the well-known $R^2_2(10)$ O−H···O hydrogen bonding motif,[10] the equatorial hydroxy groups linked the hexol molecules into H-bonded chains, growing along the [1 0 1] direction. These molecular chains, translated along the *a* axis, were connected *via* intermolecular O−H···O hydrogen bonds to form sheets, which were, in turn, H-bonded along the *b* axis to generate the solid-state self-assembly of the hexol **2** (Figure 3, Table 1).

Hence, the molecular packing in crystalline **2** was effected entirely *via* the agency of strong intermolecular O−H···O

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(6) The Fürst−Plattner rule (the trans-diaxial effect) is a direct consequence of the inherent preference of a cyclohexane ring for a chair over a twist-boat conformation. Initially proposed to account for the diastereoselectivity observed for ring-opening reactions in monosubstituted epoxycyclohexanes, the Fürst−Plattner rule has subsequently been applied even to polycyclic systems such as steroids. See: (a) Fürst, A.; Plattner, P. A. *Helv. Chim. Acta* **1949**, *32*, 275. (b) Fürst, A.; Plattner, P. A. *12th International. Congress on Pure and Applied Chemistry*, New York, 1951; Book of Abstracts, p 409.

(7) Formation of the hexol **2** has been proposed to follow an anti-Fürst−Plattner pathway because ring-opening in the epoxytetrol **4** generates, in this case, a twist-boat intermediate **2a**. For other examples of anti-Fürst−Plattner opening of epoxides, see: (a) Barton, D. H. R.; Lewis, D. A.; McGhie, J. F. *J. Chem. Soc.* **1957**, 2907. (b) Leffingwell, J. C.; Royals, E. E. *Tetrahedron Lett.* **1965**, *6*, 3829. (c) Glotter, E.; Greenfield, S.; Lavie, D. *Tetrahedron Lett.* **1967**, *8*, 5261. (d) Lunnon, M. W.; MacMillan, J. *J. Chem. Soc., Perkin Trans. 1* **1977**, 2317. (e) Hashimoto, H.; Araki, K.; Yoshimura, J. *Bull. Chem. Soc. Jpn.* **1981**, *54*, 3015. (f) Sudha, R.; Narasimhan, K. M.; Saraswathy, V. G.; Sankararaman, S. *J. Org. Chem.* **1996**, *61*, 1877. (g) Mehta, G.; Sen, S. *Tetrahedron* **2009**, *65*, 9713.

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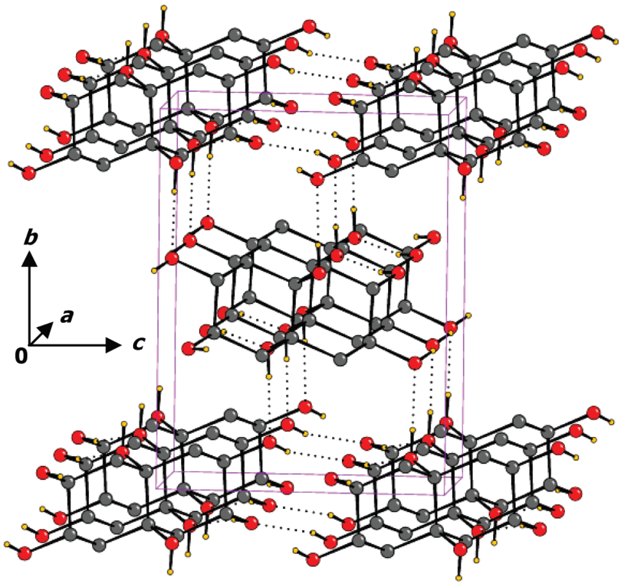

FIGURE 3. Molecular packing in the hexol **2**. Dotted lines indicate the intermolecular O−H···O hydrogen bonds.

hydrogen bonds, with each hexol molecule engaging itself in 12 H-bonds and being linked to 8 nearest neighbors. In fact, the large number of intermolecular O−H···O hydrogen bonds (the highest per molecule in the crystal structure of any of the perhydro-2,3,4a,6,7,8a-naphthalenehexols studied so far) and an efficient close packing (packing index = 72.4%) in the solid-state self-assembly of **2** would probably explain the unique physical properties (melting point and solubility) of the hexol, already alluded to.[8,9]

In conclusion, as an offshoot of our ongoing interest in the synthesis of carbasugars and cyclitols,[11] we have encountered a novel, one-pot, base-induced transformation of a fluorocyclitol[12] to a polycyclitol,[13] involving a nucleophilic displacement of fluoride and a subsequent anti-Fürst−Plattner-type ring-opening of the epoxide thus formed. Though decidedly serendipitous, this rather facile and simple chemical reaction afforded us a quantitative access to the most elusive of the perhydro-2,3,4a,6,7,8a-naphthalenehexols that can be constructed on a conformationally locked *trans*-decalin scaffold.

TABLE 1. Hydrogen Bond Geometry in the Hexol 2

D−H···A[a]	D−H (Å)	H···A (Å)	D···A (Å)	D−H···A (deg)
O1−H1O···O2[(i)]	0.820	1.91	2.733(2)	177
O2−H2O···O3[(ii)]	0.820	1.98	2.745(2)	155
O3−H3O···O1[(iii)]	0.820	2.00	2.805(2)	167

[a] Symmetry code: [(i)] $x, -y + 1/2, z - 1/2$; [(ii)] $-x + 2, -y, -z + 1$; [(iii)] $-x + 2, -y, -z$.

Experimental Section

Synthesis of (2*S,3*S**,4aα,6*R**,7*R**,8a*β*)-Perhydro-2,3,4a,6,7,8a-naphthalenehexol (2). a. From the Tetra-acetate Derivative (3) of 8a-Fluoroperhydro-2,3,4a,6,7-naphthalenepentol 1.** The tetra-acetate derivative **3** was prepared by acetylating the fluoropentol **1** (0.030 g, 0.127 mmol) at ambient temperature in presence of acetic anhydride (1.5 mL) and 4-dimethylaminopyridine (0.065 g, 0.534 mmol). The reaction takes 18 h to complete as indicated by TLC analysis. At the end of this period, the reaction was quenched with water, and the product was extracted with dichloromethane. The combined extract was washed with saturated sodium bicarbonate solution and brine and dried over anhydrous sodium sulfate. Removal of the solvent and subsequent purification by column chromatography with 50% ethyl acetate/hexane afforded the tetra-acetate **3** (0.046 g, 89%) as a colorless solid: mp 226.4−226.7 °C; IR (KBr) 3578, 3496, 2963, 1728, 1373, 1248, 1082 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.09 (brs, 2H), 4.98 (brs, 2H), 2.32−2.01 (series of m, 6H), 2.10 (s, 6H), 2.07 (s, 6H), 1.87 (d, J = 16 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0 (2C), 169.1 (2C), 93.6 (d, J = 177 Hz), 69.9 (d, J = 28 Hz), 69.8 (2C), 68.4 (2C), 33.1 (2C), 31.3 (d, J = 20 Hz, 2C), 21.2 (4C); ^{19}F NMR (376 MHz, $CDCl_3$) δ −158.1; HRMS (ES) m/z calc for $C_{18}H_{25}FO_9Na$ $(M + Na)^+$ 427.1380, found 427.1369.

Solid potassium carbonate (0.041 g, 0.297 mmol) was added to a solution of the tetra-acetate **3** (0.030 g, 0.074 mmol) in methanol (2 mL), and the resultant reaction mixture was stirred at ambient temperature for 2 h. The solvent was then removed under vacuum, and the product was isolated from the residue, following the workup procedure described below. The amount of the pure hexol obtained was 0.016 g (92%).

b. From 8a-Fluoroperhydro-2,3,4a,6,7-naphthalenepentol 1. A solution of potassium carbonate (0.058 g, 0.424 mmol) and the fluoropentol **1** (0.025 g, 0.106 mmol) in methanol (1.3 mL) was stirred vigorously for 75 min under ambient conditions. During this interval, the reaction mixture, initially clear, quickly turned turbid and soon formed a heavy white precipitate. After the completion of the reaction (as indicated by TLC), the solvent was removed completely under vacuum, and the residue was dissolved in deionized water. The solution was passed through a short column of pretreated DOWEX50W ion-exchange resin (8−200 mesh, acidic cation) and washed thoroughly with deionized water. The aqueous solution of the product thus obtained was concentrated under vacuum to obtain a white microcrystalline solid. The latter, when washed twice with methanol and dried under vacuum, afforded the pure hexol **2** (0.021 g, 96%): mp >300 °C; IR (KBr disk) 3330, 2944, 1058 cm^{-1}; ^{1}HNMR (300 MHz, DMSO-d_6) δ 4.20 (d, J = 11 Hz, 4H), 3.48 (brs, 2H, OH), 1.57−1.41 (m, 8H); ^{13}CNMR (75 MHz, DMSO-d_6) δ 73.2 (2C), 71.0 (4C), 40.8 (4C); HRMS (ES) m/z calc for $C_{10}H_{18}O_6Na$ $(M + Na)^+$ 257.1001, found 257.1003.

X-ray Crystallography. Single crystal X-ray diffraction data was collected at 291 K on a Bruker AXS SMART APEX CCD diffractometer using graphite monochromated Mo Kα radiation (λ = 0.71073 Å). The X-ray generator was operated at 50 KV and 40 mA. The data was collected with a ω scan width of 0.3°. A total of 606 frames per set were collected using *SMART*[14] in three different settings of φ (0°, 90°, and 180°). During data collection, the sample to detector distance and the 2θ value was

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kept fixed at 6.062 cm and −25° respectively. The data were reduced by *SAINTPLUS*;[14] an empirical absorption correction was applied using the package *SADABS*,[15] and *XPREP*[14] was used to determine the space group. The crystal structures were solved by direct methods using *SIR92*[16] and refined by full-matrix least-squares methods on F^2 using *SHELXL97*.[17] Molecular and packing diagrams were generated using *ORTEP-3*[18] and *CAMERON*,[19] respectively. The geometric calculations were done by *PARST*[20] and *PLATON*.[21] All hydrogen atoms, including those of hydroxy groups, were placed in geometrically idealized positions and constrained to ride on their parent atoms with C−H distances in the range 0.97−0.98 Å and $U_{\mathrm{iso}}(\mathrm{H}) = 1.2U_{\mathrm{eq}}(\mathrm{C})$ and O−H distances fixed at 0.82 Å and $U_{\mathrm{iso}}(\mathrm{H}) = 1.5U_{\mathrm{eq}}(\mathrm{O})$.

Crystal Data for Hexol 2. $C_{10}H_{18}O_6$, $M = 234.24$, monoclinic, $P2_1/c$, $a = 5.8081(6)$, $b = 10.9082(10)$, $c = 8.2899(8)$ Å, $\beta = 91.556(6)°$, $V = 525.02(9)$ Å^3, $Z = 2$, $\rho_{\mathrm{calcd}} = 1.482$ g/cm^3, 3571 reflections measured, 1026 unique ($R_{\mathrm{int}} = 0.0354$), $R1 = 0.0410$ and $wR2 = 0.1006$ for 733 observed reflections. The CIF file for the crystal data of the hexol **2** is available from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif under CCDC ref no. 789910.

Acknowledgment. We thank the Department of Science and Technology (DST), India for the CCD facility at the Indian Institute of Science (IISc), Bangalore. We sincerely acknowledge the useful experimental input by Ms. Meenu Thangam during the present study. G.M. thanks the Government of India for the award of National Research Professorship and the Council for Scientific and Industrial Research (CSIR), India for research support and the award of the Bhatnagar Fellowship.

Supporting Information Available: Scanned copies of ^{1}H and ^{13}CNMR spectra of the hexol **2** and the tetra-acetate **3**, and the final results (optimized coordinates, bond distances, bond angles, torsion angles and total energy) of the DFT based geometry optimization of the epoxytetrol **4**. This material is available free of charge via the Internet at http://pubs.acs.org.

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