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STRUCTURE, CONFORMATION AND STEREOELECTRONICS OF 9,10-ANNELATED-1,4,5,8-TETRAOXADECALINS: A MULTIDISCIPLINARY PROBE[1]

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Abstract. The structure and conformation of the 9,10-annelated-1,4,5,8-tetraoxadecalins (**3**, **4** and **5**) were investigated in solution using NMR spectroscopy, in the crystal using X-ray diffraction analysis and by computation, using the MM2 force field, suitably parameterized to include structural manifestations of the *anomeric effect*. The study focused on the geometry, viz., the structural parameters of the anomeric moieties and the ring conformation and degree of puckering, as affected by changes in the dihedral angle of the 9,10-annelating system. Very good agreement between the three methods was obtained, showing increased chair deformation, as the 9,10-substituents approach coplanarity.

INTRODUCTION

We have reported some time ago studies of cis-1,4,5,8-tetraoxadecalin (TOD)[2] (1) and some of its derivatives[2-7] within our quest for new polyoxacyclic systems with coordination ability. These had revealed at the time, considerable lacunae in the knowledge and understanding of the behaviour and the structural features of such systems and led to more fundamental, recently reported studies of 1,4-dioxanes bearing polar substituents[8-11] and on various aspects of the *anomeric effect*[12] in O-C-O containing systems. In the present study we focus our attention in two directions: (i) the conformational behaviour and the structural implications of substituting positions 9 and 10 in TOD and going, by judicious changes in the substitution pattern, from largely *gauche* to eclipsed dihedral angles around the 9,10 bond, (ii) how do the structural manifestations of the *anomeric effect* affect these systems. In fact, we wanted to "see" how the cis-decalin type cavity behaves under such constraints. To this end we chose for examination three 9,10-annelated-TOD, i.e., 2,5,7,10-tetraoxa[n.4.4]propellane (2) derivatives from among those we had reported earlier.[6]

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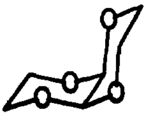

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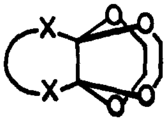

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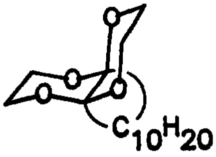

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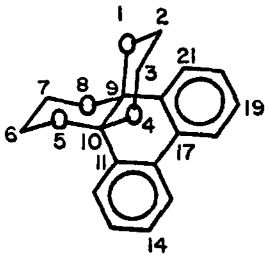

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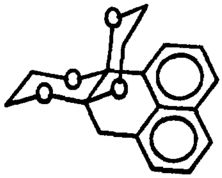

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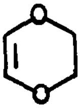

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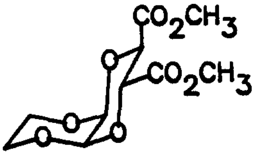

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Specifically, we sought to analyze reliably the detailed structural and conformational features of the TOD system as we change gradually the 9,10-annelating moiety from 1,10-decylidene (**2**, X-X = CH_2–$(CH_2)_8$–CH_2), through 2,2'–biphenylene (**2**, X-X = o–C_6H_4–C_6H_4–o') to 1,8-naphthylene (**2**, X-X = 1,8-$C_{10}H_6$) These compounds (**3**, **4** and **5** respectively)[6] were, hence, probed by two experimental techniques in solution, using NMR spectroscopy and in the solid, using X-ray diffraction analysis, as well as by suitable empirical force field calculations

Methodology and Results

The characteristic ^{1}H-NMR AA'BB' patterns of the -OCH_2-CH_2O- fragment in 3, 4 and 5 at ca 4 ppm, were analyzed in detail, to extract the coupling constants and, hence, the dihedral angles around the central bond within that fragment It turned out that attempts to freeze out the ring inversion processes failed, since very low temperatures were needed to decrease the rate of inversion in particular of 4 and 5 (*vide infra*) and the low solubility of these compounds hampered the low temperature measurements We could, though, use the data of the inverting system for our purpose, by making use of the known **R** value,[13] viz , $R = J_{trans}/J_{cis} = 0\ 5(J_{ax,ax} + J_{eq,eq})/0\ 5(J_{ax,eq} + J_{eq,ax}) \approx 2$ for chair conformations, R>2 for highly puckered chairs and R ≈ 1.5 for twist-boat conformations, of six-membered rings One may recall that this approach has also been quantified,[14] for evaluation of the corresponding dihedral angle (D_R), viz , $\cos D_R = [3/(4R+2)]^{1/2}$ The results achieved by this approach are assembled in Table 1 The trend obtained was evident and somewhat surprising at the time, namely that starting with the normal six-membered TOD chair in 3 towards increasing coplanarity around the 9,10 bond, first in 4 and reaching a maximum in 5, increased puckering of the -OCH_2-CH_2O- part of the chair conformations was observed throughout

Table 1. Data from ^{1}H-NMR measurements of the -OCH_2-CH_2O- moiety in compounds 3-5 and calculated R values[13] and dihedral angles (D_R,deg)[14] thereof

Comp	3		4		5	
Solvent	$CDCl_3$		$CDCl_3$		$CDCl_3+CS_2$ (1 1)	
T(K)	333	253	333	253	297	313
J_{gem}	-11 47	-11 47	-11 45	-11 86	-11 77	-11 76
J_{trans}	6 56	6 58	6 55	6 26	6 28	6 26
J_{cis}	3 45	3 59	2 86	2 42	2 52	2 48
R	1 9	1 9	2 3	2 6	2 5	2 5
D_R	56	56	59	61	60	60
D_{MM2-AB}	54 4		57 6		66 9	

We decided to verify our above, largely qualitative results, by other and more quantitative means, along with probing the structural manifestation of the O-C-O *anomeric effect* in these systems Crystallography was the method of choice and, hence, single crystals of **3**, **4** and **5** were grown and subjected to X-ray diffraction analysis For enhanced reliability and accuracy, compound **3** was analyzed in a low temperature X-ray diffraction experiment. In addition to that, these three compounds, as well as the parent cis-TOD (**1**) system were calculated, using MM2[15,16] with suitable parameterization for the *anomeric effect*[9,17,18] The combined crystallographic and computational results for the most relevant structural parameters in the TOD rings are given in Table 2, while the general crystallographic data are given in Tables 3-9 and Figure 1

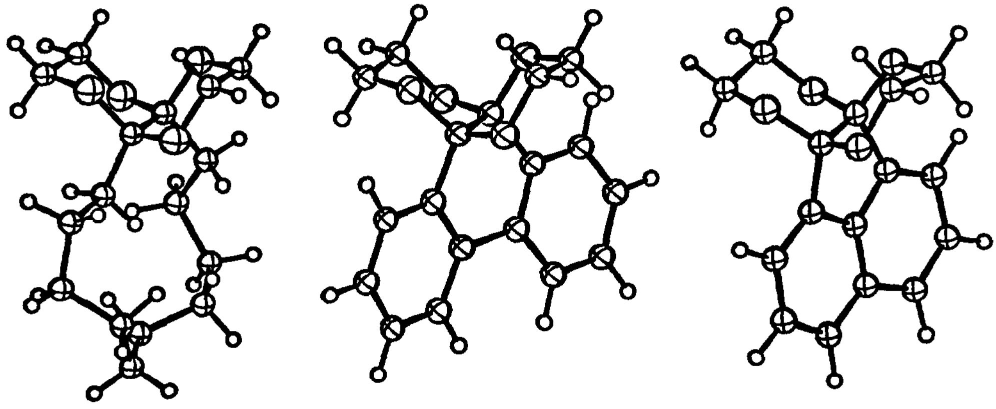
Table 2. Observed[a] (calculated[b]) C-O bond lengths (*L*, Å), C-O-C bond angles (*A*,deg) anddihedral angles (*D*,deg) within the C-O-C-O-C moieties of compounds **3** - **5** and **1**

	Compound Parameter	**3**[c]	**4**	**5**[d]	**1**[e]
	O1-C2	1.435 (1.433)	1.441 (1.432)	1.437, 1.430 (1.427)	1.439 (1.432)
	O1-C9	1.436 (1.433)	1.424 (1.432)	1.416, 1.411 (1.426)	1.420 (1.425)
	O8-C9	1.406 (1.408)	1.408 (1.409)	1.407, 1.411 (1.406)	1.410 (1.400)
	C7-O8	1.440 (1.441)	1.442 (1.440)	1.434, 1.430 (1.437)	1.432 (1.443)
L	C3-O4	1.444 (1.441)	1.439 (1.440)	1.434, 1.430 (1.437)	1.441 (1.443)
	O4-C10	1.409 (1.408)	1.415 (1.409)	1.410, 1.411 (1.406)	1.393 (1.400)
	O5-C10	1.434 (1.433)	1.429 (1.432)	1.401, 1.411 (1.426)	1.420 (1.425)
	O5-C6	1.437 (1.433)	1.430 (1.432)	1.438, 1.437 (1.427)	1.430 (1.432)
	C2-O1-C9	113.1 (112.6)	113.4 (112.2)	113.0, 113.4 (112.4)	109.1 (110.0)
	C3-O4-C10	113.7 (113.7)	114.0 (113.7)	113.7, 113.1 (113.2)	113.3 (112.3)
A	C6-O5-C10	113.0 (112.6)	113.4 (112.2)	113.5, 113.5 (112.4)	109.5 (110.0)
	C7-O8-C9	113.6 (113.7)	113.7 (113.7)	113.6, 113.1 (113.2)	113.2 (112.3)
	O1-C2-C3-O4	56.1 (54.4)	57.3 (57.6)	62.8, 62.6 (66.9)	58.4 (59.2)
	O1-C9-C10-O4	-53.3 (-54.9)	-49.5 (-52.4)	-41.0, -41.8 (-35.1)	-53.7 (-52.4)
D	O5-C6-C7-C8	55.6 (54.3)	57.0 (57.0)	51.7, 62.6 (66.9)	57.9 (59.2)
	O8-C9-C10-O5	52.7 (-54.8)	-51.4 (-52.5)	-41.2, -41.8 (-35.9)	-51.8 (-52.4)
	X-C9-C10-X	-48.1 (-44.3)	-40.6 (-44.3)	-24.7, -26.4 (-22.1)	-54.8 (-50.3)
D_R		56	59	60	58
$D_{MM2\text{-}AE}$		54.4	57.6	66.9	59.2

a) X-ray diffraction analysis, results are uncorrected for libration
b) Molecular mechanics MM2-AE (MMP2-AE for the aromatic derivatives **3** & **4**)[18]
c) Low temperature (128K) X-ray diffraction measurement
d) Two crystallographic values are given, for the two different molecules in the unit cell (see text)
e) Literature data for the parent cis-1,4,5,8-tetraoxadecalin (**1**) are given for comparison X-ray data from ref 2 and NMR data from ref 22

Table 3. Summary of Crystal Data and Experimental Parameters

Compound	3	4	5
formula	$C_{16}H_{28}O_4$	$C_{18}H_{16}O_4$	$C_{16}H_{14}O_4$
mol wt	284 40	296.32	270 28
space group	C2/c	$P2_12_12_1$	C2/c
Z	8	4	20
a, A	36 278(3)	6 992(7)	35 472(16)
b, A	7 346(1)	8 883(2)	8 738(3)
c, A	11 107(1)	22 334(6)	20 126(5)
β, deg	96 40(1)	90 0	98 88(3)
V, A^3	2941 6	1387 2	6163 4
d_c, gcm^{-1}	1 284	1 419	1 456
μ, cm^{-1}	0 84	0.93	0 98
2θ limits, deg	50	54	46
scan rate, deg /min	3	4	3
no of unique data>0	2229	1666	3816
data with I>3σ(I)	1928	1165	2298
F(000), e	1248	624	2840
R	0 037	0 042	0 042
wR	0 042	0 042	0 040
$\lvert\Delta\rho\rvert_{max}$	0 21	0 23	0 23
"goodness of fit", e	1 33	1 07	1 20



3 **4** **5**

Figure 1. ORTEP drawings of the molecular structures of 3, 4, and 5

DISCUSSION

We start by stating that we are gratified by the excellent agreement of the results achieved from the three - NMR, X-ray and computational - probes.

The above results should be viewed in several contexts We start with the initial purpose of this work, namely, the analysis of the shape of the internal decalin-type cavity as a function of 9,10-bridge in TOD We had expected a gradual puckering in the $-OCH_2-CH_2O-$ part of the rings, as the $X-C_9-C_{10}-X$ dihedral angle is decreased, culminating with a ring flip to a twist-boat conformation when (near) coplanarity is reached in 5 The latter, however, does not happen, the increased puckering in the ring periphery being the only observed effect, indicating strained-chair conformations[†] A search for precedence led to some interesting findings on related systems with similar structural constraints Thus, dihydro-1,4-dioxin (6) (in which the O-CH=CH-O part of the ring is largely coplanar) and some of its derivatives were found[20] to exhibit NMR constants in the AA'BB' ($-OCH_2-CH_2O-$) system which indicated a *gauche* dihedral angle of 60° Similar calculations on the data from an earlier study,[21] gave 61 ° In the latter, the barrier to ring inversion was also measured and found (by line-shape analysis) to be 7 6 kcal/mol[21] Such a low barrier demands very low temperature measurements, down to -150°C, which could indeed not be applied to our low solubility compounds

These results can be instructively compared to the few available ^1H-NMR and X-ray diffraction data for the general cis-1,4,5,8-tetraoxadecalin case The parent compound (1) itself exhibits an R value of 2 17 (as extracted from an early ^1H-NMR analysis[22]) with a corresponding D_R of 58°, in excellent agreement with our own X-ray diffraction results[2] (Table 2). To the best of our knowledge, only one former X-ray diffraction analysis of a 1,4,5,8-TOD derivative is available, namely that of trans-2,3-carboethoxy-cis-1,4,5,8-tetraoxadecalin (7)[23] For the sake of completeness, some of its relevant structural parameters are given here (MM2-AE calculated values in parentheses), the bond lengths in the C-O-C-O-C moiety and the $O-CH_2-CH_2-O$ dihedral angle, to be compared with those in Table 2 O1-C9 1 42 (1 42), O8-C9 1 40 (1 40), C7-O8 1 47 (1 43) Å, O5-C6-C7-O8 57 1 (59 0) deg

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[†] From the structural data one can readily obtain a quantitative estimate of ring-puckering[19] in terms of the total puckering amplitude Q and the extent of distortion of an "ideal chair" structure, *tan* θ (cf reference 19) Q values quoted in this paper[19] Q = 0 63 Å for an ideal cyclohexane chair with all C-C bond lengths at 1 54 Å and Q = 0 56 Å for a pyranoid ring representing a puckering at the C-O-C and a slight flattening at opposite part of the chair structure Similarly, the puckering parameters of the individual asymmetrically constrained 6-membered rings of the tetraoxadecalins 1 and 3 describe a distorted chair in which the increased puckering on one side of the ring is counterbalanced by the flattening of its other part, the Q and *tan* θ values ranging within 0 54-0.55 Å and 0 03-0 11, respectively The distortion is somewhat higher for compound 4, Q ≈ 0 53-0 54 Å, and largest for compound 5, 0 51-0 53 Å, (*tan* θ = 0 28)

This brings us to the next issue, namely, the stereoelectronic behaviour of the systems under scrutiny All of them exhibit the well established[11,12] structural features implicit to the *anomeric effect* in C-O-C-O-C fragments, viz., long axial C-O bonds and short equatorial ones, along with relatively wide C-O-C bond angles. This leads to an anticipated widening effect of the TOD cavity, having two oxygens of increased electron density facing each another across it The full potential of this arrangement (in terms of reactivity, coordinating power, etc) is still to be explored.

Finally, some crystallographic features While the three tetraoxadecalins (3, 4 and 5) are optically inactive in solution because they racemize spontaneously due to rapid enantiomer interconversion by ring-inversion, they are of course intrinsically chiral However, only 4 crystallizes in an enantiomorphous space group ($P2_12_12_1$), whereas 3 and 5 (as well as 1[2]) occur in racemic crystals (C2/c), 5 having a peculiar packing mode of three crystallographically independent molecules in the asymmetric unit with eight of the latter in the unit cell We feel compelled also to mention here the extensive probes of propellanes, carried out by the Haifa school[23] However, there is very little to relate to our present study, since they have usually a neopentylic ring-conjoining bond in their systems, with no heteroatom on it

EXPERIMENTAL SECTION

Compounds 3, 4 and 5 were prepared according to the previously reported procedures,[6] and recrystallized several times from toluene NMR spectra were taken on Brucker WH-90 and AM-360 WB spectrometers, chemical shifts are given in ppm downfield from TMS and coupling constants in Hz

3 ^{1}H-NMR (CDCl$_3$, 300 K) δ 4 009, 3.997, 3 991, 3 981, 3 971, 3.961 3 948, 3 937, 3 932, 3 918, 3 648, 3 636, 3 630, 3 618, 3 605, 3 596, 3 586, 3 577 (AA'BB' system, J_{gem}-11 47, J_{trans} 6 58, J_{cis} 3 50, 8H), 1 7-2 0 (m, 4H), 1 25-1 5 (m, 16H) ^{13}C-NMR (CDCl$_3$) δ 133 52, 133 28, 95.67, 60.79, 32 05, 26.36, 25 09, 23 24, 19 79

4 ^{1}H-NMR (CDCl$_3$, 295 K) δ 8 37-8 27, 7 96-7 71, 7 55-7 25 (3xm, 8H), 4 325, 4 253 4.238, 4 199, 4 181, 4 130, 4 072, 3 804, 3 746, 3 695, 3 676, 3 638, 3 623, 3 550 (AA'BB' system, J_{gem} -11 45, J_{trans} 6 55, J_{cis} 2 86, 8H) ^{13}C-NMR (CDCl$_3$) δ 133 50, 129 33, 128 17 126 90, 126.02, 123 33, 92 24, 60 99

5 ^{1}H-NMR (CDCl$_3$, 297 K) δ 7 79-7 74, 7 57-7.47, 7 29-7 22 (3xm, 6H), 4 153, 4 147, 4 139, 4 128, 4 113, 4 101, 4 097, 4 087, 3.682, 3 673, 3 668, 3 656, 3 641, 3 630, 3.623, 3 616 (AA'BB' system, J_{gem} -11 77, J_{trans} 6 28, J_{cis} 2 52, 6H) ^{13}C-NMR (CDCl$_3$) δ 137 50, 129 06, 128 14, 127 15, 119 12, 119 08, 99 22, 61 75

Crystal Structure Analyses.

X-ray diffraction data for compounds **3**, **4** and **5** were measured at ca. 20°C on a CAD4 diffractometer equipped with a graphite monochromator, using MoKα (λ=0 7107 Å) radiation and the ω scan technique with scan range of 0 9 + tan θ° The data for compound 3 were collected on an upgraded Picker diffractometer at 128K, using MoKα radiation and the ω-2θ scans from 1.3° below Kα_1 to 1 6° above Kα_2 [25] Possible deterioration of the analyzed crystals was tested by detecting frequently the intensities of three standard reflections of different zones of the reciprocal space, and was found negligible during the measurements The data sets were not corrected for absorption or secondary extinction effects Several very strong reflections which appeared to suffer from extinction, viz, 2,2,2; -6,0,2, 4,0,2, -1,1,2; -2,0,4, -5,1,5 and -8,0,6 in **4**, were excluded from the final calculations The cell constants and pertinent details of the experimental conditions are summarized in Table 3 and ORTEP plots of **3**, **4** and **5** are given in Figure 1 The three structures were solved by direct methods (SHELXS-86) [26] Their refinements were carried out by large-block least-squares (SHELX-76),[27] including the positional and anisotropic thermal parameters of all the nonhydrogen atoms. The hydrogens were included in the structure factor computations in calculated positions, and were assigned a fixed isotropic temperature factor of U=0 05 Å^2 (4 & 5) or U=0 04 Å^2 (3) The final refinements were based only on those observations that satisfied the condition $F_o^2 > 3\sigma(F^2)$, using experimental weights [$w=\sigma^{-2}(F_o)$] and minimizing $w(\Delta F)^2$ They converged smoothly at relatively low discrepancy factors of R=0 04

Final atomic coordinates of **3**, **4** and **5** are listed in Tables 4-6 and the resulting bond lengths, bond angles and torsion angles in Tables 7-9 The crystallographic atom-labeling scheme used is identical with the molecular one shown in **1** Three crystallographically independent molecules contribute to the asymmetric unit of **5** two lie in general positions, while the third is located on a twofold axis of rotation at (0 5,y,0 75) which coincides with the C(15)-C(20) bond The corresponding atoms in the three different species are marked by unprimed, primed and doubly primed labels, respectively

Acknowledgments. The calculations were performed on the IBM 4381 and CYBER 180/990 computers at the Computational Center of Tel-Aviv University, the staff of which provided valuable assistance We thank Mrs Sarah Weinman for skillful technical assistance This work was started during the tenure of one of us (B F) of the Francqui Visiting Chair at the State University of Gent The Francqui Foundation is gratefully acknowledged for this nomination This research has been supported by the Basic Research Fund of the Israel National Academy of Sciences

Table 4. Atomic Positional and Isotropic Thermal Parameters of 3

atom	x/a	y/b	z/c	U_{eq}
O(1)	0 2087(1)	-0 0707(1)	0 1170(1)	0.0204(4)
C(2)	0 2110(1)	0.0024(2)	-0 0017(1)	0 0220(6)
C(3)	0 2060(1)	-0 1476(2)	-0.0942(1)	0.0218(6)
O(4)	0.1713(1)	-0 2404(1)	-0 0856(1)	0 0202(4)
O(5)	0.1966(1)	-0 4478(1)	0 0513(1)	0 0201(4)
C(6)	0 1996(1)	-0 5215(2)	0 1718(1)	0.0230(6)
C(7)	0 2064(1)	-0 3721(2)	0 2642(1)	0.0218(6)
O(8)	0.1777(1)	-0.2364(1)	0 2451(1)	0 0198(4)
C(9)	0 1741(1)	-0 1607(2)	0 1281(1)	0 0184(6)
C(10)	0.1680(1)	-0 3131(2)	0 0301(1)	0.0184(6)
C(11)	0.1295(1)	-0 3959(2)	0.0238(1)	0.0199(5)
C(12)	0 1199(1)	-0 5424(2)	-0 0723(1)	0 0243(6)
C(13)	0 0777(1)	-0 5673(2)	-0 0921(1)	0 0248(6)
C(14)	0 0574(1)	-0 4013(2)	-0 1542(1)	0.0269(6)
C(15)	0.0258(1)	-0 3244(2)	-0 0893(1)	0 0291(6)
C(16)	0.0380(1)	-0 2374(2)	0 0344(1)	0.0258(6)
C(17)	0.0557(1)	-0 0499(2)	0 0242(1)	0.0261(6)
C(18)	0 0739(1)	0 0279(2)	0 1447(1)	0.0264(6)
C(19)	0 1100(1)	-0 0646(2)	0 1961(1)	0.0228(6)
C(20)	0 1423(1)	-0 0209(2)	0 1220(1)	0 0202(6)

U_{eq} is one third of the trace of the orthogonalized U_{ij} tensor

Table 5. Atomic Positional and Isotropic Thermal Parameters of 4.

atom	x/a	y/b	z/c	U_{eq}
O(1)	0.4846(4)	0 0943(3)	0 1523(1)	0 0359(7)
C(2)	0 6628(6)	0 0516(5)	0 1247(2)	0 0444(9)
C(3)	0 7317(5)	-0 0942(4)	0 1497(2)	0 0418(9)
O(4)	0 5866(4)	-0 2067(3)	0.1406(1)	0 0355(7)
O(5)	0 4412(3)	-0 1679(3)	0.2294(1)	0 0345(7)
C(6)	0 2804(6)	-0 1165(4)	0 2633(2)	0 0432(10)
C(7)	0 2233(6)	0 0376(4)	0 2439(2)	0 0396(8)
O(8)	0 1831(3)	0 0345(3)	0 1806(1)	0 0342(6)
C(9)	0 3380(5)	-0 0152(4)	0 1455(2)	0 0278(8)
C(10)	0 4076(5)	-0.1701(4)	0 1663(1)	0 0285(8)
C(11)	0 2793(5)	-0 2983(4)	0 1478(2)	0 0278(8)
C(12)	0 2837(5)	-0 4318(4)	0 1802(2)	0 0349(7)
C(13)	0 1826(6)	-0 5561(4)	0 1611(2)	0 0444(9)
C(14)	0 0737(6)	-0 5469(4)	0 1102(2)	0 0466(9)
C(15)	0 0686(6)	-0 4147(4)	0 0778(2)	0 0412(9)
C(16)	0 1733(5)	-0 2879(4)	0 0949(2)	0 0310(8)
C(17)	0 1752(5)	-0 1479(4)	0 0593(2)	0 0311(8)
C(18)	0 0927(6)	-0 1401(5)	0 0025(2)	0 0424(9)
C(19)	0 0885(6)	-0 0078(5)	-0 0297(2)	0 0511(9)
C(20)	0 1691(7)	0 1203(5)	-0 0059(2)	0 0507(10)
C(21)	0 2557(6)	0 1142(4)	0 0495(2)	0 0403(9)
C(22)	0 2591(5)	-0 0187(4)	0 0819(2)	0 0299(7)

U_{eq} is one third of the trace of the orthogonalized U_{ij} tensor

Table 6. Atomic Positional and Isotropic Thermal Parameters of **5**

atom	x/a	y/b	z/c	U eq
O(1)	0 2706(1)	0 1760(3)	0 0949(1)	0.0306(9)
C(2)	0 2959(1)	0 1929(4)	0.0461(2)	0 0354(13)
C(3)	0 3351(1)	0.2274(4)	0 0807(2)	0 0350(13)
O(4)	0.3485(1)	0 1002(3)	0 1228(1)	0 0326(9)
O(5)	0 3359(1)	0.1886(3)	0 2222(1)	0 0335(10)
C(6)	0 3108(1)	0 1949(5)	0 2718(2)	0 0410(16)
C(7)	0 2707(1)	0 2156(4)	0 2377(2)	0.0368(14)
O(8)	0 2597(1)	0 0872(3)	0 1947(1)	0 0307(9)
C(9)	0 2830(1)	0 0653(4)	0 1449(2)	0 0269(13)
C(10)	0 3266(1)	0 0727(4)	0 1744(2)	0 0273(13)
C(11)	0 3353(1)	-0 0896(4)	0 1960(2)	0.0283(13)
C(12)	0 3656(1)	-0 1548(5)	0 2362(2)	0.0388(15)
C(13)	0 3673(1)	-0 3170(5)	0 2409(2)	0 0431(16)
C(14)	0 3401(1)	-0 4080(5)	0.2055(2)	0 0431(16)
C(15)	0 3087(1)	-0 3442(4)	0 1630(2)	0 0323(14)
C(16)	0 2784(1)	-0 4217(4)	0 1231(2)	0 0395(15)
C(17)	0 2495(1)	-0 3387(5)	0 0863(2)	0 0410(16)
C(18)	0.2481(1)	-0 1767(4)	0 0865(2)	0 0338(14)
C(19)	0.2771(1)	-0 1015(4)	0 1245(2)	0 0271(12)
C(20)	0.3073(1)	-0 1854(4)	0.1609(2)	0 0263(12)
O(1')	0 1471(1)	0 2005(3)	0 5597(1)	0 0332(9)
C(2')	0 1512(1)	0 2065(5)	0 4897(2)	0 0426(15)
C(3')	0 1132(1)	0 2307(4)	0 4478(2)	0 0361(13)
O(4')	0 0888(1)	0 1049(3)	0 4573(1)	0 0323(9)
O(5')	0 0560(1)	0 1891(3)	0 5376(1)	0 0319(8)
C(6')	0 0518(1)	0 1984(4)	0 6072(2)	0 0366(14)
C(7')	0 0892(1)	0 2359(4)	0 6487(2)	0 0364(14)
O(8')	0 1153(1)	0 1140(3)	0.6411(1)	0 0318(9)
C(9')	0 1214(1)	0 0862(4)	0 5752(2)	0 0271(11)
C(10')	0 0831(1)	0 0791(4)	0 5241(2)	0 0250(13)
C(11')	0 0720(1)	-0 0869(4)	0 5254(2)	0 0262(13)
C(12')	0 0394(1)	-0 1631(4)	0 4989(2)	0 0335(15)
C(13')	0 0398(1)	-0 3242(5)	0.5006(2)	0 0373(15)
C(14')	0 0713(1)	-0 4056(4)	0 5259(2)	0 0374(16)
C(15')	0 1056(1)	-0 3307(4)	0 5534(2)	0 0314(13)
C(16')	0 1411(1)	-0 3962(5)	0 5813(2)	0 0395(15)
C(17')	0 1716(1)	-0 3046(5)	0 6045(2)	0 0381(16)
C(18')	0 1694(1)	-0 1426(4)	0 6032(2)	0 0327(15)
C(19')	0 1357(1)	-0 0772(4)	0 5764(2)	0 0293(13)
C(20')	0 1044(1)	-0 1709(4)	0 5517(2)	0 0260(12)
O(1")	0 4662(1)	0 1781(3)	0.6883(1)	0 0358(8)
C(2")	0 4901(1)	0 1908(5)	0 6375(2)	0 0386(14)
C(7")	0 4696(1)	0 2176(4)	0 8302(2)	0 0395(14)
O(8")	0 4569(1)	0 0881(3)	0 7894(1)	0 0341(9)
C(9")	0 4782(1)	0 0641(4)	0 7366(2)	0 0288(14)
C(15")	0.5000	-0 3455(6)	0 7500	0 0391(23)
C(16")	0 4690(1)	-0 4185(5)	0 7089(2)	0 0468(16)
C(17")	0 4412(1)	-0 3313(5)	0 6729(2)	0 0485(18)
C(18")	0 4408(1)	-0 1691(5)	0 6754(2)	0 0391(14)
C(19")	0 4707(1)	-0 0997(4)	0 7150(2)	0 0312(14)
C(20")	0 5000	-0 1870(6)	0 7500	0 0296(18)

U_{eq} is one third of the trace of the orthogonalized U_{ij} tensor

Table 7. Bond Distances (Å), Bond Angles and Torsion Angles (deg) of **3**.

(a) Bond distances

O1 - C2	1.435(2)	C9 - C20	1.540(2)
O1 - C9	1.436(2)	C10 - C11	1.518(2)
C2 - C3	1.504(2)	C11 - C12	1.528(2)
C3 - O4	1.444(2)	C12 - C13	1.533(2)
O4 - C10	1.409(1)	C13 - C14	1.547(2)
O5 - C6	1.437(2)	C14 - C15	1.529(2)
O5 - C10	1.434(2)	C15 - C16	1.535(2)
C6 - C7	1.504(2)	C16 - C17	1.529(2)
C7 - O8	1.440(2)	C17 - C18	1.535(2)
O8 - C9	1.406(1)	C18 - C19	1.528(2)
C9 - C10	1.560(2)	C19 - C20	1.539(2)

(b) Bond angles

C2 - O1 - C9	113.1(1)	O4 - C10 - C9	110.2(1)
O1 - C2 - C3	109.8(1)	O4 - C10 - O5	106.1(1)
C2 - C3 - O4	109.9(1)	C9 - C10 - C11	112.2(1)
C3 - O4 - C10	113.7(1)	O5 - C10 - C11	112.1(1)
C6 - O5 - C10	113.0(1)	O4 - C10 - C11	106.3(1)
O5 - C6 - C7	110.5(1)	C10 - C11 - C12	116.8(1)
C6 - C7 - O8	110.1(1)	C11 - C12 - C13	109.3(1)
C7 - O8 - C9	113.6(1)	C12 - C13 - C14	112.8(1)
O1 - C9 - O8	105.6(1)	C13 - C14 - C15	115.2(1)
O8 - C9 - C20	107.2(1)	C14 - C15 - C16	114.8(1)
O8 - C9 - C10	110.6(1)	C15 - C16 - C17	113.0(1)
O1 - C9 - C20	110.3(1)	C16 - C17 - C18	114.5(1)
O1 - C9 - C10	109.1(1)	C17 - C18 - C19	115.3(1)
C10 - C9 - C20	113.6(1)	C18 - C19 - C20	112.3(1)
O5 - C10 - C9	109.7(1)	C9 - C20 - C19	116.7(1)

(c) Torsion angles

C2 -O1 -C9 -C10	56.2(2)	O8 - C9 - C10 -O4	-169.1(1)
C2 -O1 -C9 -C20	-69.2(2)	O8 - C9 - C20 -C19	-33.5(2)
C2 -O1 -C9 -O8	175.2(1)	O1 - C9 - C20 -C19	-148.0(1)
C9 -O1 -C2 -C3	-58.6(2)	O8 - C9 - C10 -C11	72.6(2)
O1 -C2 -C3 -O4	56.1(2)	O1 - C9 - C10 -C11	-171.6(1)
C2 -C3 -O4 -C10	-57.2(2)	C20 -C9 - C10 -O5	-173.3(1)
C3 -O4 -C10 -C11	177.2(1)	C20 -C9 - C10 -O4	70.2(2)
C3 -O4 -C10 -O5	-63.3(2)	C10 -C9 - C20 -C19	89.1(2)
C3 -O4 -C10 -C9	55.4(2)	C20 -C9 - C10 -C11	-48.1(2)
C6 -O5 -C10 -O4	173.6(1)	C9 -C10 - C11 -C12	177.9(1)
C6 -O5 -C10 -C11	-70.7(2)	O5 -C10 - C11 -C12	-58.1(2)
C6 -O5 -C10 -C9	54.6(2)	O4 -C10 - C11 -C12	57.4(2)
C10 -O5 -C6 -C7	-57.1(2)	C10 -C11 -C12 -C13	-162.7(1)
O5 -C6 -C7 -O8	55.6(2)	C11 -C12 -C13 -C14	69.0(2)
C6 -C7 -O8 -C9	-56.8(2)	C12 -C13 -C14 -C15	-127.9(2)
C7 -O8 -C9 -O1	-62.9(2)	C13 -C14 -C15 -C16	66.7(2)
C7 -O8 -C9 -C10	55.0(2)	C14 -C15 -C16 -C17	75.5(2)
C7 -O8 -C9 -C20	179.4(1)	C15 -C16 -C17 -C18	-170.6(1)
O1 -C9 -C10 -O5	63.1(2)	C16 -C17 -C18 -C19	71.9(2)
O1 -C9 -C10 -O4	-53.3(2)	C17 -C18 -C19 -C20	71.1(2)
O8 -C9 -C10 -O5	-52.7(2)	C18 -C19 -C20 -C9	-154.9(1)

Table 8. Bond Dιstances (Å), Bond Angles and Torsιon Angles (deg) of 4

(a) Bond dιstances

C9 - O1	1 421(4)	C22 - C21	1 385(5)
C9 - C10	1 532(5)	C22 - C17	1 384(5)
C9 - O8	1 408(4)	C21 - C20	1 379(6)
C9 - C22	1 524(6)	C20 - C19	1 377(6)
O1 - C2	1 441(5)	C19 - C18	1.378(6)
C2 - C3	1 490(5)	C18 - C17	1 395(6)
C3 - O4	1 438(4)	C17 - C16	1 476(5)
O4 - C10	1 415(4)	C16 - C15	1 397(5)
C10 - O5	1 429(3)	C16 - C11	1 398(6)
C10 - C11	1 507(5)	C15 - C14	1 380(5)
O8 - C7	1 442(5)	C14 - C13	1 371(6)
C7 - C6	1 490(5)	C13 - C12	1 379(5)
C6 - O5	1.430(4)	C12 - C11	1 390(5)

(b) Bond angles.

O8 - C9 - C22	104.3(3)	C9 - C22 - C17	120 7(4)
C10 - C9 - C22	112 3(3)	C9 - C22 - C21	118 4(3)
C10 - C9 - O8	110 9(3)	C21 - C22 - C17	120 6(4)
O1 - C9 - C22	112 0(3)	C22 - C21 - C20	120 7(4)
O1 - C9 - O8	106 3(3)	C21 - C20 - C19	119 6(4)
O1 - C9 - C10	110 7(3)	C20 - C19 - C18	119.6(4)
C9 - O1 - C2	113 4(3)	C19 - C18 - C17	121 7(4)
O1 - C2 - C3	110 4(3)	C22 - C17 - C18	117 7(4)
C2 - C3 - O4	108 8(3)	C18 - C17 - C16	121 9(4)
C3 - O4 - C10	114 0(3)	C22 - C17 - C16	120 4(4)
C9 - C10 - O4	111.4(3)	C17 - C16 - C11	120 4(3)
O4 - C10 - C11	104 0(3)	C17 - C16 - C15	122 5(4)
O4 - C10 - O5	104 9(3)	C15 - C16 - C11	117 1(3)
C9 - C10 - C11	114.0(3)	C16 - C15 - C14	122 0(4)
C9 - C10 - O5	109 8(3)	C15 - C14 - C13	120 0(4)
O5 - C10 - C11	112 2(2)	C14 - C13 - C12	119 6(4)
C9 - O8 - C7	113.7(3)	C13 - C12 - C11	120 7(4)
O8 - C7 - C6	108.7(3)	C16 - C11 - C12	120 6(4)
C7 - C6 - O5	110 5(3)	C10 - C11 - C12	119 3(3)
C10 - O5 - C6	113 4(3)	C10 - C11 - C16	119 8(3)

(c) Torsιon angles

O8 -C9 -C22 -C21	82 8(4)	C22 -C9 -O8 -C7	176 5(3)
C10 -C9 -C22 -C21	-157 0(4)	C9 -O1 -C2 -C3	-57 7(4)
O1 -C9 -C22 -C21	-31 8(5)	O1 -C2 -C3 -O4	57 3(4)
O8 -C9 -C22 -C17	-90.8(4)	C2 -C3 -O4 -C10	-57 5(4)
C10 -C9 -C22 -C17	29 4(5)	C3 -O4 -C10 -C9	53 5(4)
O1 -C9 -C22 -C17	154 7(4)	C3 -O4 -C10 -O5	-65 2(3)
C10 -C9 -O8 -C7	55 4(4)	C3 -O4 -C10 -C11	176 7(3)
O1 -C9 -O8 -C7	-65 0(4)	O4 -C10 -C11 -C16	-91 4(4)
O1 -C9 -C10 -O5	66 3(4)	C9 -C10 -C11 -C16	30 0(5)

Table 8. Torsion Angles (deg.) of **4** (cont)

O1 -C9 -C10 -C11	-166 7(3)	O4 -C10 -C11 -C12	81 7(4)
C22 -C9 -C10 -O4	76 5(4)	C9 -C10 -C11 -C12	-156.9(3)
O8 -C9 -C10 -O4	-167 2(3)	O4 -C10 -O5 -C6	173 5(3)
O1 -C9 -C10 -O4	-49 5(4)	C9 -C10 -O5 -C6	53 7(4)
C10 -C9 -O1 -C2	52.5(4)	O5 -C10 -C11 -C16	155 7(3)
O8 -C9 -O1 -C2	173 1(3)	O5 -C10 -C11 -C12	-31.2(5)
C22 -C9 -O1 -C2	-73 6(4)	C11 -C10 -O5 -C6	-74 2(4)
O8 -C9 -C10 -C11	75 5(4)	C9 -O8 -C7 -C6	-57 9(4)
O8 -C9 -C10 -O5	-51.4(4)	O8 -C7 -C6 -O5	57 0(4)
C22 -C9 -C10 -C11	-40 7(4)	C7 -C6 -O5 -C10	-57 9(4)
C22 -C9 -C10 -O5	-167 7(3)		

Table 9 Bond Distances (Å), Bond Angles and Torsion Angles (deg) of **5**

(a) Bond distances

bond	molecule 1 (unprimed)	molecule 2 (primed)	molecule 3 (d. primed)
C9 - O1	1 416(4)	1 419(5)	1 411(4)
C9 - C10	1 570(4)	1 574(5)	1 557(5)
C9 - O8	1 407(5)	1 398(5)	1 411(5)
C9 - C19	1 520(5)	1 514(5)	1 508(5)
O1 - C2	1 437(5)	1 439(5)	1 430(5)
C2 - C3	1 487(4)	1 491(5)	1 495(5)
C3 - O4	1 433(4)	1 430(5)	
O4 - C10	1 410(5)	1 408(5)	
C10 - O5	1 401(4)	1 415(5)	
C10 - C11	1 501(5)	1 504(5)	
O8 - C7	1 434(4)	1 435(5)	1 429(4)
C7 - C6	1 492(4)	1 491(5)	
C6 - O5	1 438(5)	1 434(5)	
C19 - C18	1 354(4)	1 359(4)	1 366(5)
C19 - C20	1 407(4)	1 407(4)	1 390(4)
C18 - C17	1 416(5)	1 418(5)	1 418(6)
C17 - C16	1 375(5)	1 369(5)	1 363(5)
C16 - C15	1 411(4)	1 418(5)	1 421(4)
C15 - C14	1 410(5)	1 417(4)	
C15 - C20	1 389(5)	1 397(5)	1 385(7)
C14 - C13	1 363(5)	1 356(5)	
C13 - C12	1 421(6)	1 408(5)	
C12 - C11	1 366(5)	1 369(4)	
C11 - C20	1 403(4)	1 397(4)	

Table 9 Bond Distances (Å), Bond Angles and Torsion Angles (deg) of 5 (cont)

(b) Bond Angles

angle	molecule 1 (unprimed)	molecule 2 (primed)	molecule 3 (d primed)
O8 - C9 - C19	104.6(3)	104 4(3)	105 5(3)
C10 - C9 - C19	103 2(3)	103 1(3)	102 8(3)
C10 - C9 - O8	112 2(3)	112 5(4)	111 0(3)
O1 - C9 - C19	116 6(3)	116 2(4)	116 8(3)
O1 - C9 - O8	105 1(3)	106 6(3)	105 9(3)
O1 - C9 - C10	114 9(3)	113.8(3)	114 6(3)
C9 - O1 - C2	113 4(3)	114 0(3)	113 5(3)
O1 - C2 - C3	109 8(3)	109.9(4)	109.5(3)
C2 - C3 - O4	108 7(3)	109 2(3)	
C3 - O4 - C10	113 4(3)	115 4(3)	
C9 - C10 - O4	110 5(3)	112 4(4)	
O4 - C10 - C11	105 2(3)	104 3(3)	
O4 - C10 - O5	106 5(3)	106 1(3)	
C9 - C10 - C11	102 5(3)	103 2(3)	
C9 - C10 - O5	113 8(4)	113 0(3)	
O5 - C10 - C11	117 9(3)	117 7(4)	
C9 - O8 - C7	113 7(3)	115 3(3)	113 1(3)
O8 - C7 - C6	109 5(3)	108 1(3)	108 7(3)
C7 - C6 - O5	109 5(3)	110 0(4)	
C10 - O5 - C6	113 5(3)	113 7(3)	
C9 - C19 - C20	107 3(3)	107 4(3)	106 9(3)
C9 - C19 - C18	132 8(4)	132 4(4)	132 5(4)
C18 - C19 - C20	119 5(3)	119 6(3)	120 3(4)
C19 - C18 - C17	117 6(4)	118 0(4)	116 9(4)
C18 - C17 - C16	123 3(4)	122 6(4)	123 4(4)
C17 - C16 - C15	119 5(4)	120 4(4)	119 3(4)
C16 - C15 - C20	116 2(4)	115 8(4)	116 7(2)
C16 - C15 - C14	128 0(4)	128 7(4)	126 7(3)
C14 - C15 - C20	115 8(4)	115 5(4)	
C15 - C14 - C13	121 0(4)	120 8(4)	
C14 - C13 - C12	121 8(4)	122 6(4)	
C13 - C12 - C11	118 5(4)	118 2(4)	
C10 - C11 - C12	132 3(4)	132 0(4)	
C12 - C11 - C20	118 7(4)	119 2(3)	
C10 - C11 - C20	108 7(3)	108 1(3)	
C15 - C20 - C11	124 2(4)	123 7(4)	
C19 - C20 - C11	112 0(3)	112 7(3)	113 4(2)
C19 - C20 - C15	123 9(4)	123 6(4)	123 3(2)

Table 9. Bond Distances (Å), Bond Angles and Torsion Angles (deg) of 5 (cont)

(c) Torsion angles

angle	molecule 1 (unprimed)	molecule 2 (primed)	molecule 3 (d. primed)
O8 -C9 -C19 -C18	75 2(6)	-72 4(6)	79 2(6)
C10 -C9 -C19 -C18	-167.3(5)	169.9(5)	
O1 -C9 -C19 -C18	-40.3(7)	44 7(6)	-38.1(7)
O8 -C9 -C19 -C20	-96.8(4)	98 3(4)	-94 2(4)
C10 -C9 -C19 -C20	20 7(4)	-19.5(4)	
O1 -C9 -C19 -C20	147.6(4)	-144 7(4)	148 5(4)
C10 -C9 -O8 -C7	47 6(5)	-46.9(5)	
O1 -C9 -O8 -C7	-77.9(4)	78.4(4)	-74 5(4)
O1 -C9 -C10 -O5	78.8(4)	-82.1(4)	78 1(4)
O1 -C9 -C10 -C11	-152.7(3)	149.7(3)	-154 1(3)
C19 -C9 -C10 -O4	87.0(4)	-88 9(4)	86 0(4)
O8 -C9 -C10 -O4	-161 0(3)	159.2(3)	-161 7(3)
O1 -C9 -C10 -O4	-41 0(5)	37.9(5)	-41 8(4)
C10 -C9 -O1 -C2	44 3(5)	-44.7(5)	45 4(4)
O8 -C9 -O1 -C2	168 1(3)	-169 2(3)	168 1(3)
C19 -C9 -O1 -C2	-76 6(4)	74 9(4)	-74 8(5)
O8 -C9 -C10 -C11	87 3(4)	-89 0(4)	
O8 -C9 -C10 -O5	-41 2(5)	39 2(5)	
C19 -C9 -C10 -C11	-24.7(4)	22 9(4)	-26 4(4)
C19 -C9 -C10 -O5	-153 1(3)	151.1(3)	
C19 -C9 -O8 -C7	158 7(3)	-158.0(3)	161 0(3)
C9 -O1 -C2 -C3	-55 1(4)	56 9(4)	-55 6(4)
O1 -C2 -C3 -O4	62 8(4)	-61 4(4)	62 6(4)
C2 -C3 -O4 -C10	-62 7(4)	57 9(4)	
C3 -O4 -C10 -C9	50 3(4)	-45 4(5)	
C3 -O4 -C10 -O5	-73 7(4)	78 6(4)	
C3 -O4 -C10 -C11	160 3(3)	-156 6(3)	
O4 -C10 -C11 -C20	-94 7(4)	98 6(4)	
C9 -C10 -C11 -C20	21 0(4)	-19 0(4)	
O4 -C10 -C11 -C12	78 5(6)	-72 0(6)	
C9 -C10 -C11 -C12	-165 8(5)	170 4(4)	
O4 -C10 -O5 -C6	168.7(3)	-169 2(3)	
C9 -C10 -O5 -C6	46 6(5)	-45.7(5)	
O5 -C10 -C11 -C20	146 8(4)	-144 2(4)	
O5 -C10 -C11 -C12	-40 0(7)	45 2(6)	
C11 -C10 -O5 -C6	-73 5(5)	74 6(5)	
C9 -O8 -C7 -C6	-58 9(4)	58 6(4)	-62 0(4)
O8 -C7 -C6 -O5	61 7(4)	-62 1(4)	
C7 -C6 -O5 -C10	-57.2(5)	58 2(4)	

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