Acta Cryst. (1995). C51, 2377-2381

1,2;3,4-Bis{8',11'-dioxa[4.3.3]propella-(3',4')}benzene: a Bis(propellene) from Prehnitene

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(Received 2 February 1995; accepted 6 June 1995)

Abstract

The asymetric unit of the title compound, 1,3,3a,4,-7,7a,8,10,10a,11,12,12a-dodecahydro-3a,12a:7a,10abis(methanoxymethano)phenanthro[2,3-c:6,7-c']difuran, $C_{22}H_{26}O_4$, contains two U-shaped molecules related by a pseudo-twofold screw axis. Each of the molecules displays its own pseudo-mirror plane. All the tetrahydrofuran rings have envelope forms and the cyclohexane rings adopt boat conformations. There are only weakly dispersive intermolecular interactions.

Comment

Cyclization of a 1.1,2.2-tetrasubstituted ring leads, in general, to the formation of a 1.2-dispiran or the isomeric propellane (Ginsburg, 1987). Both, the complexity of the transannular effects and strain in the ring systems makes *a priori* prediction of the final product difficult (Jamrozik, 1985). The present work was undertaken to verify the molecular structure of the title bis(propellane), (I), the synthesis of which has been reported recently (Jamrozik & Żesławski, 1994).



According to the observed systematic absences, two possible space groups were considered: Pcam (No. 57, D_{2h}^{11}) and $Pca2_1$ (No. 29, C_{2v}^5). Although the unit cell contains eight molecules, no reasonable solution was found in the centrosymmetric space group, mainly because Pcam implies the of the presence of a mirror plane perpendicular to [001]. Eventually, the structure was successfully solved in space group $Pca2_1$ with two symmetry-independent molecules in the unit cell.

Each of the molecules A and B displays a pseudomirror plane intersecting the benzene ring bonds C1— C6 and C3—C4 of molecule A and C31—C36 and



Fig. 1. Views of (a) molecule A and (b) molecule B, with the atomnumbering scheme. Displacement ellipsoids are drawn at the 30% probability level (*ORTEPII*; Johnson, 1976). One O atom of molecule A is disordered between two positions, O41 and O42, with site-occupancy factors of 0.76 (3) and 0.24 (3), respectively.

C33—C34 of molecule B (cf. Fig. 1). The mirror planes, described by the equations 2.6421x + 9.7739y-11.7861z = -5.9918 and -2.7567x + 9.5604y +12.6420z = 4.8772, for A and B, respectively, make angles of about 60.5° and are oblique with respect to the crystallographic axes. The two symmetry-independent molecules have similar dimensions except for minor differences in bond lengths and valence angles (Table 2). The benzene rings of both molecules are planar with maximum deviations from planarity of 0.017(6)and 0.018 (7) Å for A and B, respectively, and with an average C-C distance of 1.392(18) for A and 1.389 (5) Å for B. The bond lengths of the cyclohexene rings [average 1.505 (49) for A and 1.511 (55) Å for B] show a gradual increase in length from the value characteristic for aromatic rings towards that expected for the conjoining bond.* Mean values of the remaining C-C bonds of the five-membered rings are 1.531 (13) and 1.528 (10) Å for A and B, respectively, whereas average C—O distances are 1.425 (28) and 1.423 (11) Å. The mean value of the endocyclic angles in the aromatic ring is $120.0(15)^{\circ}$ and deviations from the mean value exceed 3σ only for C2–C3–C4 in A and C33– C34-C35 in B. The endocyclic angles fall into two groups characteristic of the cyclohexene ring: those at

^{*} The term conjoining is widely used for the $(C_{sp^3})_3$ —C—C— $(C_{sp^3})_3$ bond type, common to three propellane rings (Tobe, 1992). The length of such a bond is significantly longer (1.588 Å) than that of a typical single C—C bond, which is estimated as 1.530 Å (Allen *et al.*, 1987).

the bond common to benzene [average 116.2 (1) in A and 116.9 (15)° in B], and the remaining [average 112.3 (4) in A and 112.5 (11)° in B]. The average values of the endocyclic angles of the five-membered rings are 104.7 (15) for A and 104.4 (17)° for B.

Ring-puckering parameters and asymmetry parameters are given in Table 3. The tetrahydrofuran rings have envelope conformations (C_s) and the cyclohexene rings adopt boat conformations (C_s) , unlike in cyclohexene in the vapour phase where a half-chair (C_2) form was determined by electron diffraction as the most stable conformation for the isolated molecule (Chiang & Bauer, 1969). In the crystal structure of 8,11-dithia[4.3.3]propella-3ene (Herbstein et al., 1986) both the five-membered and the cyclohexene rings were found to have C_2 symmetry (twist and screw-boat conformations, respectively), while in its $PdCl_2$ complex C_s symmetry was observed for all the rings (i.e. envelope and boat conformations) due to specific interactions with Pd²⁺. In the title compound, the boat form of the cyclohexene moiety may be stabilized by the influence of the two tetrahydrofuran rings at the conjoining bond. All the rings are of the same symmetry, in agreement with the non-mixing rule observed for this class of propellanes (Herbstein et al., 1986). Al-



Fig. 2. Packing diagram of the structure projected along [010]. Only non-H atoms are shown; O atoms are shown as open circles bigger than those for C atoms. Single molecules A and B are marked at positions (x + 1, y, z) and (x - 1/2, y, -z), respectively. The symmetry elements of space group $Pca2_1$ are introduced together with those of the pseudo space group $P2_1/a$ [2(0, 1/2, 0) 1/8, y, 1/4 + 0.031, and $\overline{1}$ (3/8, 1/4, 1/4 + 0.031)].

though rapid fluctuation of the six-membered ring conformations was observed in solution by ¹H NMR spectroscopy (Jamrozik & Żesławski, 1994), only one of the possible conformations is present in the crystal structure and both symmetry-independent molecules are U-shaped.

The shortest intramolecular contacts were found between the benzyl H atoms, which are coplanar with the aromatic ring [2.02 (6) in A, 2.12 (8) Å in B]. Distances between H atoms at C11 and C19 of A and C41 and C49 of B are equal to the sum of the van der Waals radii within the limit of error. The packing projected along [010] is depicted in Fig. 2. Intermolecular interactions are purely weak dispersive. There are no $H \cdots H$ intermolecular distances less than 2.19 (10) Å.

Finally, we note that closer inspection of the atomic coordinates of the two independent molecules using the program PARST (Nardelli, 1983) revealed that although the molecules A and B could be considered as related by a pseudo-twofold screw axis: 2(0, 0.495, 0) 0.125, y, 0.281, and one can find a pseudo centre of symmetry at (0.376,0.245, 0.282) which brings the molecule A at (x + 1/2,-y, z) close to B (Fig. 2), the probability for such a symmetry element appearing is low, as verified by the χ^2 test. For the twofold screw axis the sums $\sum_i (\Delta_i / \sigma_i)^2$ for the x, y and z coordinates are 45.66, 30.62 and 5.16, respectively, and for the centre of symmetry, 45.68, 30.54 and 5.16, respectively. The theoretical value is $\chi^2 = 38.90$ at 95% for 26 degrees of freedom (only non-H atoms were taken into account). It can be seen in Fig. 2 that the pseudo-symmetry elements together with the true glide plane a, perpendicular to [010], form a pseudo space group $P2_1/a$ with the same lattice parameters a and b as in $Pca2_1$, but with a β angle of 103°. The new unit cell also contains two symmetry-independent molecules, but in this description glide plane c (which is evident in the observed systematic absences) is lost. The existence of a pseudo centre of symmetry makes any simple physical test for non-centrosymmetricity (e.g. of pyroelectricity) non-relevant in distinguishing between space groups *Pca2*₁ and *Pcam* of this particular structure.

Experimental

(I) was obtained from prehnitene in four steps, with the final step being the cyclization by reaction of the suitable octol with KHSO₄ at 423 K. Crystals suitable for X-ray diffraction were obtained from an acetic acid solution by slow evaporation at room temperature. D_m was measured by flotation in aqueous KI solution.

Crystal data

$C_{22}H_{26}O_4$	Mo $K\alpha$ radiation
$M_r = 354.43$	$\lambda = 0.7107 \text{ Å}$
Orthorhombic	Cell parameters from 35
Pca2 ₁	reflections
a = 12.380(2) Å	$\theta = 2.8 - 12.2^{\circ}$
<i>b</i> = 11.194 (2) Å	$\mu = 0.086 \text{ mm}^{-1}$
c = 26.892 (5) Å	T = 293 (2) K

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V = 372	26.7 (11) Å ³	F	Plate		O33	0.3589 (4)	0.5830 (4) 0.2670 ((2) 0.0695 (13)
Z = 8 0.3 × 0.3 × 0.1 mm			034	0.0678 (4)	0.6834 (4) 0.2646	(3) 0.091 (2)		
$D_{\rm r} = 1.263 \mathrm{Mg}\mathrm{m}^{-3}$ Colourless			C31	0.1963 (4)	0.4382	5) 0.1521 ((3) 0.0385 (15)		
$D_{1} = 1$	263 Mg m ⁻	- 3	50.00.000		C32	0.2302(5)	0.5460 0	(5) 0.13210	(3) 0.0440(14)
$D_m = 1$.	.20 (2) Mg III				C33	0.2943(5)	0.3480 ((0) 0.0897 ((4) 0.0697 ((3) $0.050(2)$
Data co	llection				C35	0.3243(0) 0.2888(4)	0.4414	(0) 0.0082 ((5) 0.0865 ((3) 0.034(2) (2) 0.0439(14)
Duiu CO	necnon				C36	0.2000(4) 0.2237(4)	0.3314 (5) 0.1291 ((2) 0.0437(14) (3) 0.0382(13)
Kuma K	KM-4 diffracto	meter <i>b</i>	$P_{\rm max} = 25.0^{\circ}$		C37	0.1884(5)	0.2092	5) 0.1469	$\begin{array}{c} (3) \\ (3) \\ (3) \\ (3) \\ (3) \\ (10) \\ $
$\omega/2\theta \ {\rm sc}$	ans	k	$i = 0 \rightarrow 14$		C38	0.2808 (4)	0.1174 (5) 0.1471	(3) 0.0408 (13)
Absorpt	tion correction	: k	$z = 0 \rightarrow 13$		C39	0.3499 (4)	0.1180 (5) 0.0984 ((2) 0.0430 (14)
none		1	$= -32 \rightarrow 0$		C40	0.3150 (5)	0.2143 (5) 0.0616	(3) 0.0480 (14)
3003 m	essured reflect	tions 7	standard reflecti	ions	C41	0.3645 (5)	0.1445 (7) 0.1879	(3) 0.059 (2)
2271 :	domondont roff	aotiona -	manitara davan	- 50	C42	0.4644 (5)	0.1386	7) 0.1181 ((3) 0.057 (2)
1755	dependent ten	ections		y 50	C43	0.2368 (7)	-0.0103	(5) 0.1489 ((4) 0.055 (2)
1/55 of	oserved reflect	ions	reflections		C44	0.3312(6)	-0.0081	(6) 0.0780 ((5) 0.059(2)
[l > 1]	$2\sigma(I)$]		intensity variati	ion: $<1.5\%$	C45	0.1312(5) 0.1854(5)	0.44/80	5) 0.2002 (5) 0.2371	$\begin{array}{ccc} (3) & 0.0422 (14) \\ (3) & 0.0406 (14) \\ \end{array}$
$R_{\rm int}=0.$.0203				C40	0.1834(3) 0.2217(4)	0.53221	(3) 0.2371 (3) 0.2371 (3) 0.2138 (3) 0.213	(3) 0.0400(14)
					C48	0.2217(4) 0.1961(6)	0.6544	(f) 0.21580	(3) 0.0413(13)
Refinem	ient				C49	0.2919(6)	0.4837	6) 0.2574 ((3) 0.055(2) (4) 0.054(2)
D C		Ŧ			C50	0.3415 (5)	0.6604	6) 0.2250	(3) 0.059(2)
Refinen	nent on F	1	extinction correct	tion:	C51	0.1093 (7)	0.5698	6) 0.2779	(3) 0.063 (2)
$R[F^2 >$	$2\sigma(F^2)] = 0.0$)317	SHELXL93 (Sh	neldrick,	C52	0.1563 (5)	0.7455	5) 0.2439	(3) 0.059 (2)
$wR(F^2)$	= 0.0709		1993)						
S = 1.14	46	E	Extinction coeffic	ient:			† Occupancy	y = 0.76(3).	
2270 ге	flections		0.0043(7)				‡ Occupanc	y = 0.24(3).	
670 par	ameters	,	Atomic scattering	factors					0
U stom	ameters	-	from Internatio	, lacions	Tat	ole 2. Sele	cted geom	etric parame	ters (Å, °)
	s renned isotro	opi-	for Constalla	mai Tables	Molecule A	4		Molecule B	
cally;	; riding model	IOF	for Crystallogr	apny (1992,	01-C11	-	1.421 (7)	O31C42	1.424 (8)
H521	and H522 on	1.7	Vol. C. Tables	4.2.6.8 and					
		liy		inglicite and	O1C12		1.437 (7)	O31C41	1.435 (7)
w = 1/[a]	$\sigma^2(F_o^2) + (0.02)$	$(552P)^2$	6.1.1.4)	in little und	O1C12 O2C14		1.437 (7) 1.424 (9)	O31C41 O32C43	1.435 (7) 1.411 (10)
w = 1/[a]	$\sigma^2(F_o^2) + (0.05)$ e $P = (F_o^2 + 1)$	$(552P)^2$] $(2F_c^2)/3$	6.1.1.4) Absolute configu	ration:	O1C12 O2C14 O2C13		1.437 (7) 1.424 (9) 1.430 (9)	O31C41 O32C43 O32C44	1.435 (7) 1.411 (10) 1.425 (9)
w = 1/[a] where (Δ/σ)	$\sigma^{2}(F_{o}^{2}) + (0.05)$ e $P = (F_{o}^{2} + 2)$ = -0.037	$552P)^{2}]$ $2F_{c}^{2})/3$	6.1.1.4) Absolute configur Flack (1983) p	ration: arameter	O1C12 O2C14 O2C13 O3C20		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8)	O31C41 O32C43 O32C44 O33C49	1.435 (7) 1.411 (10) 1.425 (9) 1.411 (8)
$w = 1/[\alpha]$ where $(\Delta/\sigma)_{\rm m}$	$\sigma^{2}(F_{o}^{2}) + (0.05)$ e $P = (F_{o}^{2} + 2)$ hax = -0.037 = 0.175 e Å^{-3}	$552P)^2]$ $2F_c^2)/3$	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80)	ration: arameter	O1C12 O2C14 O2C13 O3C20 O3C19		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.461 (9)	O31C41 O32C43 O32C44 O33C49 O33C50	1.435 (7) 1.411 (10) 1.425 (9) 1.411 (8) 1.441 (8)
$w = 1/[\alpha]$ where $(\Delta/\sigma)_{\rm m}$ $\Delta\rho_{\rm max} = 0$	$\sigma^{2}(F_{o}^{2}) + (0.02)$ e $P = (F_{o}^{2} + 2)$ hax $= -0.037$ = 0.175 e Å ⁻³	$\frac{1}{552P}^{2}$ $2F_{c}^{2}$ /3	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80)	ration: arameter	01C12 02C14 02C13 03C20 03C19 041C21		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.461 (9) 1.411 (10)	O31C41 O32C43 O32C44 O33C49 O33C50 O34C52	1.455 (7) 1.411 (10) 1.425 (9) 1.411 (8) 1.441 (8) 1.412 (7)
$w = 1/[\sigma]$ where $(\Delta/\sigma)_{max} = \Delta\rho_{max} = \Delta\rho_{min} = 0$	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + T_{o}^{2})$ = 0.175 e Å ⁻³ = -0.119 e Å ⁻³	$\frac{1}{552P}^{2}]$ $2F_{c}^{2})/3$	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80)	ration: arameter	01C12 02C14 02C13 03C20 03C19 041C21 041C22		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.461 (9) 1.411 (10) 1.451 (10)	O31C41 O32C43 O32C44 O33C49 O33C50 O34C52 O34C51	1.435 (7) 1.411 (10) 1.425 (9) 1.411 (8) 1.441 (8) 1.441 (8) 1.412 (7) 1.416 (8)
$w = 1/[\sigma]$ where $(\Delta/\sigma)_{m}$ $\Delta\rho_{max} = \Delta\rho_{min} =$	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + 2)$ = -0.037 = 0.175 e Å ⁻³ = -0.119 e Å ⁻³	$\frac{1}{552P}^{2}$ $\frac{552P}{c}^{2}/3$	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80)	ration: arameter	01C12 02C14 02C13 03C20 03C19 041C21 041C22 042C22		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.461 (9) 1.411 (10) 1.451 (10) 1.36 (2) 1.45 (2)	O31C41 O32C43 O32C44 O33C50 O34C52 O34C51	1.435 (7) 1.411 (10) 1.425 (9) 1.411 (8) 1.441 (8) 1.442 (7) 1.416 (8)
$w = 1/[\alpha]$ where $(\Delta/\sigma)_{m}$ $\Delta\rho_{max} = \Delta\rho_{min} = Table 1$	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + 2)$ = -0.037 = 0.175 e Å ⁻³ = -0.119 e Å ⁻¹	19 552P) ²] 2F ² _c)/3 -3 ! atomic co	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) poordinates and	ration: arameter equivalent	01C12 02C14 02C13 03C20 03C19 041C21 041C22 042C22 042C21 C1C2		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.461 (9) 1.411 (10) 1.451 (10) 1.36 (2) 1.45 (2) 1.409 (9)	O31—C41 O32—C43 O32—C44 O33—C49 O33—C50 O34—C52 O34—C51	1.435 (7) 1.411 (10) 1.425 (9) 1.411 (8) 1.441 (8) 1.412 (7) 1.416 (8)
$w = 1/[\alpha]$ where $(\Delta/\sigma)_{m}$ $\Delta\rho_{max} = \Delta\rho_{min} = Table 1$	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + 1)$ = 0.037 = 0.175 e Å ⁻³ = -0.119 e Å ⁻³ 1. Fractional isotropic di	s52P) ²] 2F ² _c)/3 -3 atomic co splacemen.	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) pordinates and t parameters (Å	ration: arameter <i>equivalent</i> ²)	01C12 02C14 02C13 03C20 03C19 041C21 041C22 042C22 042C22 042C22 C1C2 C1C2		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.461 (9) 1.411 (10) 1.451 (10) 1.36 (2) 1.45 (2) 1.409 (9) 1.418 (8)	O31—C41 O32—C43 O32—C44 O33—C49 O33—C50 O34—C52 O34—C51 C31—C32 C31—C32 C31—C36	1.455 (7) 1.411 (10) 1.425 (9) 1.411 (8) 1.412 (7) 1.416 (8) 1.386 (8) 1.389 (7)
$w = 1/[\alpha]$ where $(\Delta/\sigma)_{m}$ $\Delta\rho_{max} = \Delta\rho_{min} = Table 1$	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + 2)$ = 0.037 = 0.175 e Å ⁻³ = -0.119 e Å ⁻³ 1. Fractional isotropic di	s52P) ²] 2F ² /3 - ³ atomic co splacement	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) pordinates and t parameters (Å	ration: arameter equivalent ²)	01C12 02C13 03C20 03C19 041C21 041C22 042C22 042C21 C1C2 C1C6 C1C15		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.401 (8) 1.461 (9) 1.411 (10) 1.451 (10) 1.36 (2) 1.45 (2) 1.459 (9) 1.418 (8) 1.494 (8)	O31C41 O32C43 O32C44 O33C49 O33C50 O34C52 O34C51 C31C32 C31C36 C31C36	1.455 (7) 1.411 (10) 1.425 (9) 1.411 (8) 1.411 (8) 1.412 (7) 1.416 (8) 1.386 (8) 1.389 (7) 1.528 (8)
w = 1/[4] where $(\Delta/\sigma)_{m}$ $\Delta\rho_{max} = 2$ $\Delta\rho_{min} = 1$ Table 1	$\sigma^{2}(F_{o}^{2}) + (0.0);$ $e P = (F_{o}^{2} + 2);$ = -0.037; $= -0.175 \text{ e } \text{Å}^{-3};$ $= -0.119 \text{ e } \text{Å}^{-3};$ 1. Fractional isotropic di $U_{eq} = 0.013;$	$[symmetry] = (1/3)\sum_{i}\sum_{j} \sum_{i} \sum_{j} \sum_{j} \sum_{i} \sum_{j} \sum_{j} \sum_{j} \sum_{j} \sum_{j} \sum_{j}$	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) bordinates and t parameters (Å	equivalent	01C12 02C13 03C20 03C19 041C21 041C22 042C22 042C22 C1C6 C1C15 C2C3		$\begin{array}{c} 1.437(7)\\ 1.424(9)\\ 1.430(9)\\ 1.404(8)\\ 1.461(9)\\ 1.411(10)\\ 1.451(10)\\ 1.451(10)\\ 1.45(2)\\ 1.45(2)\\ 1.418(8)\\ 1.494(8)\\ 1.370(9) \end{array}$	O31C41 O32C43 O32C44 O33C49 O33C50 O34C52 O34C51 C31C32 C31C35 C31C45 C32C33	1.455 (7) 1.411 (10) 1.425 (9) 1.411 (8) 1.441 (8) 1.412 (7) 1.416 (8) 1.386 (8) 1.389 (7) 1.528 (8) 1.391 (9)
$w = 1/[\alpha where (\Delta/\sigma)_m (\Delta/\sigma)_m = \Delta\rho_{min} = \Delta\rho_{min} = Table 1$	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + 2)$ = 0.037 = 0.175 e Å ⁻³ = -0.119 e Å ⁻³ 1. Fractional isotropic di U_{eq} =	$[symmetry] = (1/3)\Sigma_i \Sigma_j c$	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) bordinates and t parameters (Å $U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$	ration: arameter equivalent ²)	01C12 02C13 03C20 03C19 041C21 041C22 042C22 042C22 042C22 C1C2 C1C2 C1C15 C2C3 C2C18		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.461 (9) 1.411 (10) 1.451 (10) 1.45 (2) 1.409 (9) 1.418 (8) 1.494 (8) 1.370 (9) 1.494 (9)	O31—C41 O32—C43 O32—C44 O33—C49 O33—C50 O34—C52 O34—C51 C31—C32 C31—C32 C31—C35 C31—C45 C32—C33 C32—C48	1.435 (7) 1.411 (10) 1.425 (9) 1.411 (8) 1.441 (8) 1.412 (7) 1.416 (8) 1.386 (8) 1.389 (7) 1.528 (8) 1.391 (9) 1.520 (8)
w = 1/[4] where $(\Delta/\sigma)_{max} = \Delta\rho_{max} = \Delta\rho_{min} = Table 1$	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + 2)$ = 0.037 = 0.175 e Å ⁻³ = -0.119 e Å ⁻¹ 1. Fractional isotropic di $U_{eq} = x$	$[1] = (1/3)\Sigma_i \Sigma_j v$	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) boordinates and t parameters (Å $U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$ z	ration: arameter equivalent ²) U _{eq}	01C12 02C13 03C20 03C19 041C21 041C22 042C22 042C22 042C22 C1C6 C1C15 C2C3 C2C18 C3C4		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.461 (9) 1.411 (10) 1.451 (10) 1.45 (2) 1.409 (9) 1.418 (8) 1.494 (8) 1.370 (9) 1.494 (9) 1.378 (9)	O31—C41 O32—C43 O32—C44 O33—C49 O33—C50 O34—C52 O34—C51 C31—C32 C31—C32 C31—C36 C31—C45 C32—C48 C33—C34	1.435 (7) 1.411 (10) 1.425 (9) 1.411 (8) 1.411 (8) 1.412 (7) 1.416 (8) 1.386 (8) 1.389 (7) 1.528 (8) 1.391 (9) 1.520 (8) 1.383 (9)
$w = 1/[where (\Delta/\sigma)_m (\Delta/\sigma)_m + \Delta\rho_{max} = \Delta\rho_{min} = Table 1$	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + 2)$ = -0.037 = 0.175 e Å ⁻³ = -0.119 e Å ⁻¹ 1. Fractional isotropic di $U_{eq} = x$ e A	$[1] = \frac{1}{2} \sum_{k=1}^{2} \sum_{j=1}^{2} \sum_$	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) pordinates and t parameters (Å $U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$ z	equivalent ²) U _{eq}	01C12 02C13 03C20 03C19 041C21 041C22 042C22 C1C2 C1C6 C1C15 C2C3 C2C18 C3C4 C4C5		1.437 (7) 1.424 (9) 1.430 (9) 1.430 (9) 1.404 (8) 1.461 (9) 1.411 (10) 1.451 (10) 1.45 (2) 1.409 (9) 1.418 (8) 1.494 (8) 1.494 (8) 1.370 (9) 1.494 (9) 1.371 (8)	O31—C41 O32—C43 O32—C44 O33—C49 O33—C50 O34—C52 O34—C51 C31—C32 C31—C32 C31—C36 C31—C45 C32—C48 C33—C34 C34—C35	$\begin{array}{c} 1.435 \ (\prime) \\ 1.411 \ (10) \\ 1.425 \ (9) \\ 1.411 \ (8) \\ 1.412 \ (7) \\ 1.416 \ (8) \\ \end{array}$
$w = 1/[where (\Delta/\sigma)_m (\Delta/\sigma)_m + \Delta\rho_{max} = \Delta\rho_{min} = Table 1$	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + 1)$ = 0.037 = 0.175 e Å ⁻³ = -0.119 e Å ⁻³ 1. Fractional isotropic di $U_{eq} = x$ = $A^{-0.2010}(3)$	$\begin{bmatrix} 1 \\ 552P \\ 552P \\ 2F_c^2 \end{bmatrix} = \begin{bmatrix} 2F_c^2 \\ 3 \end{bmatrix} = \begin{bmatrix} 2F_c^2 \\ 3 \end{bmatrix} = \begin{bmatrix} 2F_c^2 \\ 3 \end{bmatrix} = \begin{bmatrix} 2F_c \\ 3 \end{bmatrix}$	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) pordinates and t parameters (Å $U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$ z 0.3971 (2) 0.4637	ration: arameter equivalent U_{eq} 0.0572 (10)0.0590 (11)	01C12 02C13 03C20 03C19 041C21 041C22 042C22 042C22 042C22 C1C6 C1C15 C2C3 C2C18 C3C4 C4C5 C5C6 C5C7 C5C6 C5C7 C5C6 C5C7 C5C6 C5C7 C5C6 C5C7 C5C6 C5C7 C5C6 C5C7 C5C6 C5C7 C5C7 C5C7 C5C7 C5C7 C5C7 C5C7		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.401 (8) 1.401 (10) 1.451 (10) 1.36 (2) 1.45 (2) 1.409 (9) 1.418 (8) 1.494 (8) 1.370 (9) 1.371 (8) 1.401 (7)	O31—C41 O32—C43 O32—C44 O33—C49 O33—C50 O34—C52 O34—C52 O34—C51 C31—C32 C31—C36 C31—C45 C32—C38 C32—C48 C33—C34 C34—C35 C35—C36	1.455 (7) 1.411 (10) 1.425 (9) 1.411 (8) 1.441 (8) 1.412 (7) 1.416 (8) 1.386 (8) 1.389 (7) 1.528 (8) 1.391 (9) 1.520 (8) 1.383 (9) 1.383 (9) 1.387 (8) 1.400 (7)
$w = 1/[u]$ where $(\Delta/\sigma)_{m}$ $\Delta\rho_{max} = \Delta\rho_{min} = Table 1$ Molecula 01 02 03	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + 2)$ = -0.037 = 0.175 e Å ⁻³ = -0.119 e Å ⁻³ 1. Fractional isotropic di U_{eq} = x e A -0.2010 (3) 0.0201 (4) -0.1045 (3)	$[1] = \frac{1}{2} \int_{c}^{2} $	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) pordinates and t parameters (Å $U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$ z 0.3971 (2) 0.4637 0.3014 (2)	ration: arameter equivalent 2^{2} U_{eq} 0.0572 (10) $0.0590 (11)$	01C12 02C13 03C20 03C19 041C21 041C22 042C22 042C22 042C22 042C2 C1C6 C1C15 C2C18 C3C4 C4C5 C5C6 C5C10 C6-C7		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.461 (9) 1.411 (10) 1.451 (10) 1.45 (2) 1.45 (2) 1.45 (2) 1.490 (9) 1.418 (8) 1.370 (9) 1.494 (8) 1.371 (8) 1.371 (8) 1.401 (7) 1.504 (8)	O31C41 O32C43 O32C44 O33C49 O33C50 O34C52 O34C51 C31C36 C31C36 C31C36 C31C45 C32C33 C32C48 C33C48 C33C35 C35C36 C35C36 C35C40 C35C40	1.455 (7) 1.411 (10) 1.425 (9) 1.411 (8) 1.412 (7) 1.416 (8) 1.386 (8) 1.389 (7) 1.528 (8) 1.391 (9) 1.520 (8) 1.383 (9) 1.387 (8) 1.400 (7) 1.519 (8) 1.514 (7)
$w = 1/[u]$ where $(\Delta/\sigma)_{m}$ $\Delta\rho_{max} = \Delta\rho_{min} = Table D$ Molecule 01 02 03 041t	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + 2)$ = -0.037 = -0.175 e Å ⁻³ = -0.119 e Å ⁻³ 1. Fractional isotropic di U_{eq} = e A -0.2010 (3) 0.0201 (4) -0.1045 (3) 0.1900 (7)	$[1] = \frac{1}{2} \int_{c}^{2} $	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) pordinates and t parameters (Å $U_{ij}a_i^*a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$. z 0.3971 (2) 0.4637 0.3014 (2) 0.304 (4)	ration: arameter equivalent U_{eq} 0.0572 (10) 0.0590 (11) 0.0667 (13) 0.060 (3)	$\begin{array}{c} 01C12 \\ 02C14 \\ 03C20 \\ 03C21 \\ 041C22 \\ 042C22 \\ 042C22 \\ 042C22 \\ 042C22 \\ 042C22 \\ 042C21 \\ C1C6 \\ C1C15 \\ C2C3 \\ C2C18 \\ C3 - C4 \\ C4C5 \\ C5C6 \\ C5C10 \\ C6 - C7 \\ C7 - C8 \end{array}$		1.437 (7) 1.424 (9) 1.404 (9) 1.404 (8) 1.401 (9) 1.411 (10) 1.451 (10) 1.451 (10) 1.45 (2) 1.45 (2) 1.49 (9) 1.418 (8) 1.370 (9) 1.494 (8) 1.370 (9) 1.494 (9) 1.371 (8) 1.401 (7) 1.504 (8) 1.507 (8)	O31C41 O32C43 O33C49 O33C49 O33C50 O34C52 O34C51 C31C32 C31C36 C31C45 C32C33 C32C48 C33C45 C32C36 C35C36 C35C36 C35C40 C36C37 C37C38	1.455 (7) 1.411 (10) 1.425 (9) 1.411 (8) 1.412 (7) 1.416 (8) 1.386 (8) 1.389 (7) 1.528 (8) 1.389 (7) 1.520 (8) 1.381 (9) 1.520 (8) 1.387 (8) 1.400 (7) 1.519 (8) 1.519 (8) 1.514 (7) 1.527 (7)
$w = 1/[u]$ where $(\Delta/\sigma)_{m}$ $\Delta\rho_{max} = \Delta\rho_{min} = Table II$ Molecula O1 O2 O3 O41† O42t	$\sigma^{2}(F_{o}^{2}) + (0.0)$ $e P = (F_{o}^{2} + 2)$ $= -0.037$ $= 0.175 e Å^{-3}$ $= -0.119 e Å^{-3}$ $= -0.119 e Å^{-3}$ $I. Fractional$ $isotropic di$ $U_{eq} = x$ $e A$ $-0.2010 (3)$ $0.0201 (4)$ $-0.1045 (3)$ $0.1900 (7)$ $0.122 (4)$	[1] = [1]	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) bordinates and t parameters (Å $U_{ij}a_i^*a_j^*a_i.a_j.$ 2 0.3971 (2) 0.4637 0.3014 (2) 0.3004 (4) 0.2768 (10)	<i>equivalent</i> 2) <i>U</i> _{eq} 0.0572 (10) 0.0590 (11) 0.0667 (13) 0.079 (13)	$\begin{array}{c} 01C12 \\ 02C13 \\ 03C20 \\ 03C19 \\ 041C22 \\ 042C22 \\ 042C2 \\ 042C$		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.401 (8) 1.401 (8) 1.411 (10) 1.451 (10) 1.451 (2) 1.45 (2) 1.499 (9) 1.418 (8) 1.494 (8) 1.370 (9) 1.494 (8) 1.378 (9) 1.371 (8) 1.494 (8) 1.371 (8) 1.494 (8) 1.371 (8) 1.494 (8) 1.371 (8) 1.494 (8) 1.371 (8) 1.494 (8) 1.371 (8) 1.494 (8) 1.376 (8) 1.507 (8) 1.507 (8) 1.536 (8) 1.517 (8)	$\begin{array}{c} 031 - C41 \\ 032 - C43 \\ 033 - C49 \\ 033 - C50 \\ 034 - C52 \\ 034 - C51 \\ \hline \\ C31 - C32 \\ C31 - C36 \\ C31 - C45 \\ C32 - C43 \\ C32 - C48 \\ C33 - C34 \\ C34 - C35 \\ C35 - C36 \\ C35 - C40 \\ C36 - C37 \\ C37 - C38 \\ C38 - C43 \\ \hline \\ \end{array}$	$\begin{array}{c} 1.435 \ (7) \\ 1.411 \ (10) \\ 1.425 \ (9) \\ 1.411 \ (8) \\ 1.441 \ (8) \\ 1.412 \ (7) \\ 1.416 \ (8) \\ \end{array}$ $\begin{array}{c} 1.386 \ (8) \\ 1.389 \ (7) \\ 1.528 \ (8) \\ 1.391 \ (9) \\ 1.520 \ (8) \\ 1.387 \ (8) \\ 1.400 \ (7) \\ 1.519 \ (8) \\ 1.514 \ (7) \\ 1.537 \ (7) \\ 1.537 \ (7) \\ 1.537 \ (7) \\ 1.530 \ (8) \\ \end{array}$
w = 1/[4] where $(\Delta/\sigma)_{m}$ $\Delta\rho_{max} = 2$ $\Delta\rho_{min} = 1$ Table 1 Molecula O1 O2 O3 O41† O42‡ C1	$\sigma^{2}(F_{o}^{2}) + (0.0)$ $e P = (F_{o}^{2} + 2)$ = -0.037 $= 0.175 e Å^{-3}$ $= -0.119 e Å^{-3}$ 1. Fractional isotropic di $U_{eq} = x$ e A -0.2010 (3) 0.0201 (4) -0.1045 (3) 0.1900 (7) 0.122 (4) 0.0689 (4)	[1] = [1]	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) bordinates and t parameters (Å $U_{ij}a_i^*a_j^*a_i.a_j.$ 2 0.3971 (2) 0.4637 0.3014 (2) 0.3004 (4) 0.2768 (10) 0.4130 (2)	<i>equivalent</i> <i>u</i> _{eq} <i>U</i> _{eq} 0.0572 (10) 0.0590 (11) 0.0607 (13) 0.079 (13) 0.037 (13)	$\begin{array}{c} 01C12 \\ 02C13 \\ 03C20 \\ 03C19 \\ 041C21 \\ 041C22 \\ 042C22 \\ 042C2 \\ 042C$		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.401 (8) 1.401 (10) 1.451 (10) 1.451 (10) 1.45 (2) 1.409 (9) 1.418 (8) 1.494 (8) 1.370 (9) 1.494 (8) 1.370 (9) 1.371 (8) 1.371 (8) 1.507 (8) 1.536 (8) 1.517 (8) 1.532 (8)	O31C41 O32C43 O33C49 O33C50 O34C52 O34C51 C31C32 C31C35 C31C45 C32C33 C32C48 C33C34 C33C36 C35C36 C35C36 C35C40 C36C37 C37C38 C38C41	$\begin{array}{c} 1.435 \ (7) \\ 1.411 \ (10) \\ 1.425 \ (9) \\ 1.411 \ (8) \\ 1.441 \ (8) \\ 1.412 \ (7) \\ 1.416 \ (8) \\ \end{array}$ $\begin{array}{c} 1.386 \ (8) \\ 1.389 \ (7) \\ 1.528 \ (8) \\ 1.391 \ (9) \\ 1.520 \ (8) \\ 1.383 \ (9) \\ 1.387 \ (8) \\ 1.400 \ (7) \\ 1.519 \ (8) \\ 1.514 \ (7) \\ 1.537 \ (7) \\ 1.530 \ (8) \\ 1.540 \ (8) \\ \end{array}$
$w = 1/[u \text{ where} (\Delta/\sigma)_m (\Delta/\sigma)_m + \Delta\rho_{min} = \Delta\rho_{min} = Table 1$ Molecule 01 02 03 041† 042‡ C1 C2	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + 2)$ = 0.175 e Å ⁻³ = -0.119 e Å ⁻³ = -0.119 e Å ⁻³ 1. Fractional isotropic di $U_{eq} =$ x e A -0.2010 (3) 0.0201 (4) -0.1045 (3) 0.1900 (7) 0.122 (4) 0.0332 (4)	[1] = [1]	6.1.1.4) Absolute configur Flack (1983) p. = 0.46 (1.80) bordinates and t parameters (Å $U_{ij}a_i^*a_j^*a_i.a_j.$ 2 0.3971 (2) 0.4637 0.3014 (2) 0.3004 (4) 0.2768 (10) 0.4130 (2) 0.4348 (3)	<i>equivalent</i> <i>u</i> _{eq} <i>U</i> _{eq} 0.0572 (10) 0.0590 (11) 0.0667 (13) 0.079 (13) 0.037 (13) 0.044 (2)	$\begin{array}{c} 01C12 \\ 02C14 \\ 03C20 \\ 03C19 \\ 041C21 \\ 041C22 \\ 042C22 \\ 042C22 \\ 042C22 \\ 042C22 \\ 042C21 \\ C1C2 \\ C1C2 \\ C1C2 \\ C1C2 \\ C1C2 \\ C2C18 \\ C3C4 \\ C4C5 \\ C5C10 \\ C5C10 \\ C6 - C7 \\ C7C8 \\ C8C11 \\ C8C13 \\ C8 - C9 \end{array}$		1.437 (7) 1.424 (9) 1.430 (9) 1.404 (8) 1.461 (9) 1.411 (10) 1.451 (10) 1.45 (2) 1.409 (9) 1.418 (8) 1.494 (8) 1.370 (9) 1.494 (8) 1.370 (9) 1.371 (8) 1.371 (8) 1.507 (8) 1.507 (8) 1.532 (8) 1.532 (8) 1.563 (7)	O31-C41 O32-C43 O33-C49 O33-C49 O33-C50 O34-C52 O34-C51 C31-C32 C31-C32 C31-C36 C31-C45 C32-C33 C32-C48 C33-C34 C34-C35 C35-C36 C35-C36 C35-C40 C36-C37 C37-C38 C38-C41 C38-C39	$\begin{array}{c} 1.435 \ (7) \\ 1.411 \ (10) \\ 1.425 \ (9) \\ 1.411 \ (8) \\ 1.412 \ (7) \\ 1.416 \ (8) \\ \end{array}$
$w = 1/[u \text{ where} (\Delta/\sigma)_m (\Delta/\sigma)_m + \Delta\rho_{min} = \Delta\rho_{min} = Table 1$ Table 1 Molecule 01 02 03 041† 042‡ C1 C2 C3	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + 1)$ = 0.75 e Å ⁻³ = -0.119 e Å ⁻³ = -0.119 e Å ⁻³ 1. Fractional isotropic di $U_{eq} =$ x e A -0.2010 (3) 0.0201 (4) -0.1045 (3) 0.1900 (7) 0.122 (4) 0.0332 (4) -0.0274 (5)	[1] = [1]	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) boordinates and t parameters (Å $U_{ij}a_i^*a_j^*a_i.a_j.$ z 0.3971 (2) 0.4637 0.3014 (2) 0.3004 (4) 0.2768 (10) 0.4130 (2) 0.4348 (3) 0.4773 (3)	ration: arameter equivalent $^{2})$ U_{eq} 0.0572 (10) 0.0590 (11) 0.0667 (13) 0.060 (3) 0.079 (13) 0.0387 (13) 0.044 (2) 0.0483 (15)	$\begin{array}{c} 01C12 \\ 02C14 \\ 02C13 \\ 03C20 \\ 03C19 \\ 041C21 \\ 041C22 \\ 042C22 \\ 042$		$\begin{array}{c} 1.437(7)\\ 1.424(9)\\ 1.430(9)\\ 1.430(9)\\ 1.404(8)\\ 1.461(9)\\ 1.411(10)\\ 1.36(2)\\ 1.45(2)\\ 1.409(9)\\ 1.418(8)\\ 1.494(8)\\ 1.494(8)\\ 1.370(9)\\ 1.474(8)\\ 1.370(9)\\ 1.371(8)\\ 1.371(8)\\ 1.507(8)\\ 1.507(8)\\ 1.536(8)\\ 1.517(8)\\ 1.532(8)\\ 1.532(8)\\ 1.533(7)\\ 1.530(8)\\ \end{array}$	O31—C41 O32—C43 O33—C49 O33—C49 O33—C50 O34—C52 O34—C51 C31—C32 C31—C32 C31—C36 C31—C45 C32—C33 C32—C48 C33—C44 C34—C35 C35—C40 C36—C37 C37—C38 C38—C41 C38—C41 C38—C40	$\begin{array}{c} 1.435 \ (7) \\ 1.411 \ (10) \\ 1.425 \ (9) \\ 1.411 \ (8) \\ 1.412 \ (7) \\ 1.416 \ (8) \\ \end{array}$
$w = 1/[u \text{ where} (\Delta/\sigma)_m (\Delta/\sigma)_m + \Delta\rho_{max} = \Delta\rho_{min} = Table 1$ Table 1 Molecule 01 02 03 041† 042‡ C1 C2 C3 C4	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + (0.0))$ e $P = (F_{o}^{2} + (0.0))$ e $P = (F_{o}^{2} + (0.0))$ = -0.037 = -0.037 = -0.037 = -0.037 = -0.037 = -0.037 is $e^{A^{-3}}$ = -0.019 e Å ⁻³ is $otropic$ di U_{eq} = x e A -0.02010 (3) 0.0201 (4) -0.045 (3) 0.0201 (4) -0.045 (3) 0.032 (4) -0.032 (4) -0.0274 (5) -0.0520 (5)	$\sum_{i=1}^{3} \sum_{j=1}^{3} \sum_{j$	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) bordinates and t parameters (Å $U_{ij}a_i^*a_j^*a_i.a_j.$ z 0.3971 (2) 0.4637 0.3014 (2) 0.3004 (4) 0.2768 (10) 0.4130 (2) 0.4348 (3) 0.4773 (3) 0.5012 (3)	ration: arameter equivalent 2^{2} U_{eq} 0.0572 (10) 0.0590 (11) 0.0667 (13) 0.060 (3) 0.079 (13) 0.0387 (13) 0.0387 (13) 0.0483 (15) 0.048 (2)	$\begin{array}{c} 01 - C12 \\ 02 - C13 \\ 03 - C20 \\ 03 - C19 \\ 041 - C21 \\ 041 - C22 \\ 042 - C22 \\ 042 - C22 \\ 042 - C22 \\ 042 - C21 \\ C1 - C2 \\ C1 - C6 \\ C1 - C15 \\ C2 - C3 \\ C2 - C18 \\ C3 - C4 \\ C4 - C5 \\ C5 - C6 \\ C5 - C10 \\ C6 - C7 \\ C7 - C8 \\ C8 - C11 \\ C8 - C13 \\ C8 - C1 \\ C9 - C10 \\ C9 - C10 \\ C9 - C12 \\ \end{array}$		$\begin{array}{l} 1.437(7)\\ 1.424(9)\\ 1.430(9)\\ 1.430(9)\\ 1.404(8)\\ 1.461(9)\\ 1.411(10)\\ 1.36(2)\\ 1.451(10)\\ 1.36(2)\\ 1.494(8)\\ 1.370(9)\\ 1.418(8)\\ 1.370(9)\\ 1.494(8)\\ 1.370(9)\\ 1.371(8)\\ 1.537(8)\\ 1.507(8)\\ 1.536(8)\\ 1.517(8)\\ 1.532(8)\\ 1.563(7)\\ 1.530(8)\\ 1.538(8)\\ \end{array}$	O31C41 O32C43 O33C49 O33C49 O33C50 O34C52 O34C51 C31C32 C31C36 C31C45 C32C33 C32C48 C33C48 C33C48 C33C40 C36C37 C37C38 C38C41 C38C49 C39C42	$\begin{array}{c} 1.435 \ (7) \\ 1.411 \ (10) \\ 1.425 \ (9) \\ 1.411 \ (8) \\ 1.441 \ (8) \\ 1.412 \ (7) \\ 1.416 \ (8) \\ \end{array}$ $\begin{array}{c} 1.386 \ (8) \\ 1.389 \ (7) \\ 1.528 \ (8) \\ 1.391 \ (9) \\ 1.520 \ (8) \\ 1.383 \ (9) \\ 1.583 \ (9) \\ 1.583 \ (9) \\ 1.514 \ (7) \\ 1.519 \ (8) \\ 1.514 \ (7) \\ 1.519 \ (8) \\ 1.537 \ (7) \\ 1.530 \ (8) \\ 1.540 \ (8) \\ 1.540 \ (8) \\ 1.527 \ (8) \\ 1.527 \ (8) \\ 1.527 \ (8) \\ 1.527 \ (8) \\ 1.527 \ (8) \\ 1.530 \ (8) \\ 1.527 \ (8) \\ 1.530 \ (8) \\ 1.527 \ (8) \\ 1.530 \ (8) \ (8) \ (8) \ (8) \ (8) \ (8) \ (8) \ (8) \ (8) \ (8) \ ($
$w = 1/[u \text{ where} (\Delta/\sigma)_m + \Delta\rho_{max} = \Delta\rho_{min} = \Delta\rho_{min} = Table 1$ Molecula O1 O2 O3 O41† O42‡ C1 C2 C3 C4 C5 C4 C5	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + (0.0))$ e $P = (F_{o}^{2} + (0.0))$ e $P = (F_{o}^{2} + (0.0))$ = $(0.175 \text{ e } \text{Å}^{-3})$ = $(0.175 \text{ e } \text{Å}^{-3})$ = $(0.119 \text{ e } \text{Å}^{-3})$ = $(0.0110 \text{ (} 3))$ = $(0.0201 \text{ (} 4))$ = $(0.0201 \text{ (} 4))$ = $(0.0201 \text{ (} 4))$ = $(0.0151 \text{ (} 4))$ = $(0.0151 \text{ (} 4))$	$[1] y = (1/3)\sum_{i}\sum_{j} (1/3)\sum_{j} (1/3)\sum_{j} (1/3)\sum_{i}\sum_{j} (1/3)\sum_{i}\sum_{j$	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) pordinates and t parameters (Å $U_{ij}a_i^*a_j^*a_i.a_j.$ z 0.3971 (2) 0.4637 0.3004 (4) 0.2768 (10) 0.4130 (2) 0.4348 (3) 0.4773 (3) 0.5012 (3) 0.4814 (2)	ration: arameter equivalent 2^2 U_{eq} 0.0572 (10) 0.0590 (11) 0.0667 (13) 0.0387 (13) 0.044 (2) 0.048 (2) 0.0396 (13)	$\begin{array}{c} 01 &C12 \\ 02 &C14 \\ 03 &C20 \\ 03 &C19 \\ 041 &C22 \\ 042 &C21 \\ 041 &C22 \\ 042 &C22 \\ 042 &C21 \\ C1 &C2 \\ C1 &C6 \\ C1 &C15 \\ C2 &C3 \\ C2 &C18 \\ C3 &C4 \\ C4 &C5 \\ C5 &C6 \\ C5 &C10 \\ C6 & -C7 \\ C7 &C8 \\ C8 &C11 \\ C8 &C13 \\ C8 &C9 \\ C9 &C10 \\ C9 &C12 \\ C9 &C14 \\ \end{array}$		$\begin{array}{l} 1.437(7)\\ 1.424(9)\\ 1.430(9)\\ 1.440(8)\\ 1.461(9)\\ 1.411(10)\\ 1.451(10)\\ 1.451(10)\\ 1.451(2)\\ 1.45(2)\\ 1.45(2)\\ 1.494(8)\\ 1.370(9)\\ 1.494(8)\\ 1.370(9)\\ 1.494(8)\\ 1.370(9)\\ 1.494(8)\\ 1.370(9)\\ 1.540(8)\\ 1.507(8)\\ 1.532(8)\\ 1.532(8)\\ 1.533(8)\\ 1.533(8)\\ 1.533(8)\\ 1.533(8)\\ 1.533(8)\\ 1.534(9)\\ \end{array}$	$\begin{array}{c} \text{O31C41} \\ \text{O32C43} \\ \text{O33C49} \\ \text{O33C49} \\ \text{O33C50} \\ \text{O34C52} \\ \text{O34C51} \\ \hline \\ \text{C31C36} \\ \text{C31C36} \\ \text{C31C36} \\ \text{C31C45} \\ \text{C32C33} \\ \text{C32C48} \\ \text{C33C44} \\ \text{C34C35} \\ \text{C35C36} \\ \text{C35C36} \\ \text{C35C40} \\ \text{C36C37} \\ \text{C37C38} \\ \text{C38C41} \\ \text{C38C43} \\ \text{C38C41} \\ \text{C38C42} \\ \text{C39C42} \\ \text{C39C44} \\ \hline \end{array}$	$\begin{array}{c} 1.455 \ (\prime) \\ 1.411 \ (10) \\ 1.425 \ (9) \\ 1.411 \ (8) \\ 1.412 \ (7) \\ 1.416 \ (8) \\ \end{array}$
$w = 1/[u \text{ where} (\Delta/\sigma)_m (\Delta/\sigma)_m = \Delta\rho_{max} = \Delta\rho_{min} = Table 1$ Molecula 01 02 03 041† 042‡ 01 02 03 041† 042‡ 02 03 041† 042‡ 03 041† 042‡ 03 041† 042‡ 03 041† 042‡ 03 041† 042‡ 03 041† 042‡ 03 041† 042‡ 04 05 04 05 04 05 05 05 05 05 05 05 05 05 05 05 05 05	$\sigma^{2}(F_{o}^{2}) + (0.0)$ e $P = (F_{o}^{2} + 2)$ = -0.037 = -0.175 e Å ⁻³ = -0.119 e Å ⁻³ = -0.119 e Å ⁻³ 1. Fractional isotropic di U_{eq} = x e A -0.2010 (3) 0.0201 (4) -0.1045 (3) 0.1900 (7) 0.122 (4) 0.0332 (4) -0.0274 (5) -0.0520 (5) -0.0151 (4) 0.0450 (4)	$[1] y = (1/3) \sum_{i} \sum_{j} \sum_$	6.1.1.4) Absolute configur Flack (1983) p = 0.46 (1.80) pordinates and t parameters (Å $U_{ij}a_i^*a_j^*a_i.a_j.$ z 0.3971 (2) 0.4637 0.3014 (2) 0.3004 (4) 0.2768 (10) 0.4130 (2) 0.4348 (3) 0.4773 (3) 0.4814 (2) 0.4373 (3) 0.4190 (2)	ration: arameter equivalent U_{eq} 0.0572 (10) 0.0590 (11) 0.0667 (13) 0.0044 (2) 0.0483 (15) 0.0382 (13) 0.0382 (13) 0.0382 (13)	01C12 02C13 03C20 03C19 041C21 041C22 042C22 042C22 042C22 042C22 C1C6 C1C15 C2C3 C2C18 C3C4 C4C5 C5C10 C6-C7 C7C8 C8C11 C8C13 C8C13 C8C9 C9C12 C9C14 C15C16		$\begin{array}{l} 1.437(7)\\ 1.424(9)\\ 1.430(9)\\ 1.404(8)\\ 1.461(9)\\ 1.401(10)\\ 1.451(10)\\ 1.451(10)\\ 1.451(2)\\ 1.45(2)\\ 1.45(2)\\ 1.494(9)\\ 1.370(9)\\ 1.494(8)\\ 1.370(9)\\ 1.494(8)\\ 1.370(9)\\ 1.494(8)\\ 1.370(9)\\ 1.540(8)\\ 1.530(8)\\ 1.532(8)\\ 1.530(8)\\ 1.538(8)\\ 1.538(8)\\ 1.537(9)\\ 1.5$	$\begin{array}{c} \text{O31-C41} \\ \text{O32-C43} \\ \text{O33-C49} \\ \text{O33-C49} \\ \text{O33-C50} \\ \text{O34-C52} \\ \text{O34-C51} \\ \end{array}$	$\begin{array}{c} 1.435 \ (\prime)\\ 1.411 \ (10)\\ 1.425 \ (9)\\ 1.411 \ (8)\\ 1.412 \ (7)\\ 1.411 \ (8)\\ 1.412 \ (7)\\ 1.416 \ (8)\\ \end{array}$

01	0.1045 (3)	0.0004 (4)	0.0014 (0)	0.0447.4.2	÷		
03	-0.1045(3)	0.0836 (4)	0.3014 (2)	0.0667 (13)	C6C7	1.507 (8)	C36—C37
O41†	0.1900 (7)	0.1827 (6)	0.3004 (4)	0.060 (3)	C7C8	1.536 (8)	C37C38
O42‡	0.122 (4)	0.1971 (16)	0.2768 (10)	0.079 (13)	C8-C11	1.517 (8)	C38C43
Cl	0.0689 (4)	-0.0613 (5)	0.4130 (2)	0.0387 (13)	C8C13	1.532 (8)	C38C41
C2	0.0332 (4)	0.0463 (6)	0.4348 (3)	0.044 (2)	C8C9	1.563 (7)	C38C39
C3	-0.0274 (5)	0.0404 (6)	0.4773 (3)	0.0483 (15)	C9C10	1.530 (8)	C39C40
C4	-0.0520 (5)	-0.0650 (6)	0.5012 (3)	0.048 (2)	C9C12	1.538 (8)	C39—C42
C5	-0.0151 (4)	-0.1702 (5)	0.4814 (2)	0.0396 (13)	C9C14	1.541 (9)	C39C44
C6	0.0450 (4)	-0.1706 (5)	0.4373 (3)	0.0382 (13)	C15C16	1.537 (9)	C45C46
C7	0.0789 (4)	-0.2913 (5)	0.4180 (3)	0.0444 (14)	C16C19	1.501 (10)	C46-C51
C8	-0.0156 (4)	-0.3802 (5)	0.4168 (3)	0.0405 (13)	C16-C21	1.542 (9)	C46—C49
C9	-0.0828 (4)	-0.3785 (5)	0.4660 (2)	0.0437 (14)	C16-C17	1.553 (8)	C46—C47
C10	-0.0380 (5)	-0.2902 (5)	0.5042 (3)	0.0439 (14)	C17C18	1.527 (9)	C47C50
C11	-0.0989 (5)	-0.3519 (6)	0.3772 (3)	0.0493 (15)	C17C20	1.538 (8)	C47C52
C12	-0.1966 (4)	-0.3448 (6)	0.4479 (3)	0.053 (2)	C17—C22	1.538 (8)	C47—C48
C13	0.0240 (6)	-0.5097 (6)	0.4140 (4)	0.051 (2)		104.2 (4)	C42 021 C41
C14	-0.0778 (7)	-0.5091 (6)	0.4840 (4)	0.056 (2)	C11 = 01 = C12	104.2 (4)	C42 = 031 = C41
C15	0.1287 (5)	-0.0497 (6)	0.3650 (3)	0.0447 (14)	C14 - 02 - C13	105.1 (5)	C43-032C44
C16	0.0698 (5)	0.0335 (5)	0.3285 (3)	0.0427 (14)	$C_{20} = 0.03 = 0.019$	104.2 (5)	C49 = 033 = C30
C17	0.0361 (4)	0.1539 (4)	0.3526 (3)	0.0443 (14)	$C_{21} = 041 = C_{22}$	105.5 (6)	$C_{32} = C_{34} = C_{31}$
C18	0.0629 (5)	0.1585 (5)	0.4080 (3)	0.051 (2)	$C_{22} = 042 = C_{21}$	106.4 (17)	$C_{32} - C_{31} - C_{43}$
C19	-0.0342 (7)	-0.0192 (7)	0.3102 (4)	0.067 (3)	(2	116.1 (5)	$C_{30} - C_{31} - C_{43}$
C20	-0.0862 (5)	0.1581 (6)	0.3425 (3)	0.055 (2)	C_{0}	125.1(5)	$C_{31} - C_{32} - C_{48}$
C21	0.1451 (7)	0.0723 (6)	0.2858 (3)	0.067 (2)	$C_3 - C_2 - C_{18}$	125.2(6)	C33C32C48
C22	0.0990 (6)	0.2463 (6)	0.3218 (3)	0.058 (2)	CIC2C18	116.2 (6)	C34C35C40
Malaa	-1- D				C4C5C10	123.0(5)	C36-C35-C40
woleci		0.0000.00		0.0000 (1.0)	C6C5C10	116.3 (5)	C31—C36—C37
031	0.4643 (3)	0.0989 (4)	0.1684 (2)	0.0698 (12)	C5—C6—C7	116.2 (5)	C35—C36—C37

0.0600 (12)

C1-C6-C7

123.9 (5)

C43-C38-C37

0.0989 (2)

O32

0.2320 (3)

-0.0488 (4)

1.527 (9)

1.570 (8)

1.514 (8) 1.533 (7)

1.535 (8)

103.7 (5)

104.0 (6) 104.0 (6)

105.1 (5)

115.2 (5) 124.6 (5) 118.3 (5)

121.1 (6)

122.8 (5)

118.4 (5) 124.5 (5)

115.7 (5)

111.1 (5)

C22H26O4

Molecule A, five-membered rings

C11-C8-C13	112.4 (5)	C43-C38-C41	113.7 (6)
C11-C8-C7	113.5 (5)	C37-C38-C41	111.8 (5)
C13—C8—C7	111.7 (5)	C43—C38—C39	103.0 (5)
C10-C9-C12	112.7 (5)	C37—C38—C39	113.7 (5)
C10-C9-C14	112.8 (6)	C41-C38-C39	103.1 (4)
C12—C9—C14	111.7 (6)	C40—C39—C42	112.3 (5)
01C11C8	106.6 (5)	C40-C39-C44	112.0 (6)
01—C12—C9	105.0 (4)	C42—C39—C44	113.8 (6)
02C13C8	106.1 (6)	O31C41C38	104.4 (5)
02—C14—C9	104.8 (6)	O31—C42—C39	106.3 (5)
	111.7 (5)	032 - 043 - 038	105.7 (6)
	112.3 (3)	032 - 044 - 039	100.4 (6)
C19 - C16 - C21	112.0(7)	$C_{40} - C_{45} - C_{51}$	111.2 (5)
C13 - C16 - C21	103.2(5)	C_{31} $-C_{40}$ $-C_{43}$ C_{51} C_{46} C_{40}	111.8 (3)
$C_{21} = C_{10} = C_{17}$	103.2 (5)	C45 C46 C49	112.2(7)
$C_{18} - C_{17} - C_{20}$	112.0(5)	C_{4}	103.0 (5)
C_{20} C_{17} C_{22}	112.2(5)	$C_{50}^{}C_{47}^{}C_{57}^{}C_{57}^{}C_{57}^{}C_{57}^{$	1125(5)
03-019-016	104.7 (5)	C50-C47-C48	113.2 (5)
03C20C17	106.2 (5)	C52C47C48	112.1 (5)
041-C21-C16	106.1 (6)	O33C49C46	107.0 (5)
O42—C21—C16	106.3 (11)	O33-C50-C47	106.0 (5)
O42-C22-C17	108.4 (11)	O34-C51-C46	107.1 (6)
O41-C22-C17	106.1 (6)	O34—C52—C47	106.9 (5)
C15-C1-C2-C3	176 3 (5)	C45_C31_C32_C48	-25(7)
	179.2 (5)	$C_{45} = C_{51} = C_{36} = C_{47}$	-2.5(7)
$C_{15} - C_{1} - C_{6} - C_{7}$	11(8)	C40-C35-C36-C37	18(7)
C5-C6-C7-C8	48.5 (7)	C35 - C36 - C37 - C38	44.7 (7)
C6-C7-C8-C9	-46.3(7)	C36-C37-C38-C39	-46.1(7)
C11-C8-C9-C10	-123.4(5)	C43-C38-C39-C40	123.2 (6)
C13-C8-C9-C10	119.5 (5)	C37—C38—C39—C40	2.9 (6)
C7-C8-C9-C10	-0.8 (6)	C41-C38-C39-C40	-118.3 (5)
C11—C8—C9—C12	-1.9 (6)	C37-C38-C39-C42	124.1 (5)
C7—C8—C9—C12	120.8 (5)	C41—C38—C39—C42	2.9 (6)
C13-C8-C9-C14	-2.6 (6)	C43C38C39C44	2.6 (6)
C7C8C9C14	-122.8 (5)	C37C38C39C44	-117.7 (6)
C6-C5-C10-C9	-49.3 (7)	C36-C35-C40-C39	-46.3 (7)
$C_8 - C_9 - C_{10} - C_5$	4/.6(6)	C38—C39—C40—C35	41.5 (7)
$C_{12} = 0 = 0 = 0 = 0$	41.3 (6)	C42 = 031 = C41 = C38	44.3 (6)
$C_{9} = C_{8} = C_{11} = O_{1}$	-23.4(6)	$C_{39} - C_{38} - C_{41} - O_{31}$	-28.2 (6)
$C_{1} = 0 = 0 = 0 = 0$	-42.1 (0)	C41 - O31 - C42 - C39	-42.9(7)
$C_{0} - C_{1} - C_{12} - C_{12} - C_{13}$	20.2 (0)	$C_{44} = C_{39} = C_{42} = C_{31}$	80.1 (7)
$C_{1} = 0_{2} = 0_{1} = 0_{3}$	-222(6)	$C_{44} = 0.32 = C_{43} = 0.32$	43.1(7)
$C_{1}^{2} = 0^{2} = C_{1}^{2} = 0^{2}$	-419(7)	$C_{43} = C_{30} = C_{43} = C_{30} = C_{43} = C_{30} = C_{43} = C_{30} = C_{43} = C_{30} = C$	-27.4(0) -414(8)
C8 - C9 - C14 - O2	26.5 (7)	C_{38} C_{39} C_{44} C_{39}	22.8 (8)
C2C1C15C16	-47.5(6)	C32-C31-C45-C46	-47.1(7)
C1-C15-C16-C17	49.0 (6)	C31-C45-C46-C47	47.6 (6)
C19C16C17C18	118.5 (6)	C45-C46-C47-C50	-123.5 (5)
C15C16C17C18	- 3.7 (6)	C49C46C47C50	-1.3 (7)
C21—C16—C17—C18	-123.7 (6)	C51—C46—C47—C52	- 2.2 (6)
C19—C16—C17—C20	-2.7 (7)	C45C46C47C52	119.1 (5)
C15—C16—C17—C20	-124.8 (5)	C51—C46—C47—C48	-122.5 (6)
C15—C16—C17—C22	118.3 (5)	C45—C46—C47—C48	-1.1 (6)
$C_{1} = C_{10} = C_{17} = C_{22}$	-1.7(6)	C49 - C46 - C47 - C48	121.1 (6)
$C_1 = C_2 = C_{18} = C_{17}$	49.5 (7)	$C_{31} = C_{32} = C_{48} = C_{47}$	50.6(7)
$C_{10} = C_{17} = C_{18} = C_{26}$	-44.4(0)	$C_{40} - C_{47} - C_{48} - C_{52}$	-46.0 (6)
C_{17} $-C_{16}$ $-C_{19}$ $-C_{10}$	-42.3(8)	C_{47} C_{46} C_{49} C_{40}	-41.5 (8)
C19 - 03 - C20 - C17	40.6 (7)	C49 - O33 - C50 - C47	40.1 (6)
C16-C17-C20-O3	-23.1(6)	C46-C47-C50-O33	-22.9(6)
C22-041-C21-C16	-42.4 (10)	C52-034-C51-C46	- 39.6 (7)
C22	35 (3)	C47-C46-C51-034	24.9 (7)
C17-C16-C21-O41	27.0 (9)	C51O34C52C47	37.7 (7)
C17—C16—C21—O42	-19(2)	C46—C47—C52—O34	-21.0 (6)
C21-042-C22-C17	-36(3)		
C21O41C22C17	41.2 (10)		
C16-C17-C22-O42	23 (2)		
$U_{10} - U_{1} - U_{22} - 041$	-23.2 (8)		

Table 3. Puckering and asymmetry parameters

Puckering parameters (Cremer & Pople, 1975):

Molecule A	SIX-0	nembered	rings
in could i i	1. 517. 1	noniocica	111123

molecule A, six-memocicu rings								
	$q_{2}(Å)$	q_3 (Å)	$\varphi_2(^\circ)$	$Q_T(\text{\AA})$	θ_2 (°)			
C5-C10	0.664 (6)	-0.004 (6)	119.8 (5)	0.664 (6)	90.4 (5)			
C1C2C18C15	0.656 (6)	-0.10 (6)	117.0 (5)	0.656 (6)	90.9(5)			

01-C11-C8-C9-C	12	0.389 (5)	176.9 (9)		
02-C13-C8-C9-C	14	0.384 (6)	176.0 (9)		
O3C20C17C16	-C19	0.388 (8)	176.5 (11)		
041-C22-C17-C16	-C21	0.393 (9)	177.4 (10)		
042-C21-C16-C17	C22	0.32 (3)	176.8 (12)		
Molecule B, six-mem	bered rir	igs			
	q_2 (Å)	\tilde{q}_3 (Å)	$\varphi_2(^{\circ})$	$Q_T(Å)$	θ_2 (°)
C35-C40	0.618 (6)	0.010 (6)	122.4 (6)	0.618 (6)	89.1 (6)
C31—C32—C48–C45	0.673 (6)	0.005 (6)	118.9 (5)	0.673 (6)	89.6 (5)
Molecule <i>B</i> , five-men	nbered ri	ngs			
		q_2 (A)	$\varphi_2(^\circ)$		
O31C41C38C39	C42	0.409 (6)	175.7 (8)	
O32—C43—C38—C39	C44	0.392 (7)	-176.1 (10	0)	
O33C50C47C46	C49	0.379 (7)	177.7 (1	1)	
O34—C52—C47—C46	C51	0.354 (7)	176.6 (1	2)	
Asymmetry paramete	rs (Nard	elli, 1983)	:		
Molecule 4					
Molecule		\boldsymbol{N}		\mathcal{N}	
C5-C10		$\frac{1}{100}$, $\frac{1}{100}$)6 (3)	$\frac{1}{100}$	0.003(2)
C1-C2-C18-C15		C18 = 0.02	25 (3)	$C^{2}-C^{1}=1$	0.002(2)
		01 - 0.01			010-1 (2)
0^{-1}	212	01 = 0.01	10(3)		
0^{2} 0^{2} 0^{2} 0^{2} 0^{2} 0^{2} 0^{2} 0^{2}	C10	$O_2 = 0.02$	21 (3)		
03 - 020 - 017 - 010	-019	03 = 0.01	19 (4)		
041 - 022 - 017 - 010	$-C_{21}$	041 = 0.01	13 (4)		
042-021-010-017	C22	042 = 0.01	10 (9)		
Molecule <i>B</i>					
Molecule D		Λ		Δc	
C35-C40		$\frac{1}{C37} = 0.02$	20 (3)		-0.017(2)
$C_{31} - C_{32} - C_{48} - C_{45}$		C48 = 0.01	3 (3)	C_{32} C_{31}	= 0.017(2)
		C +0 = 0.01		CJ2CJ1	- 0.010 (2)
U31-C41-C38-C39	C42	0.031 = 0.02	24 (3)		
U32-C43-C38-C39	-C44	0.032 = 0.02	21 (4)		

 $i q_2 (Å) \varphi_2 (°)$

Data collection: Kuma KM-4 diffractometer software. Cell refinement: Kuma KM-4 diffractometer software. Data reduction: Kuma KM-4 diffractometer software. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPII (Johnson, 1976). Software used to prepare material for publication: SHELXL93.

O33 = 0.012(4)

O34 = 0.017 (4)

Lists of structure factors, anisotropic displacement parameters, Hatom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1266). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

O33-C50-C47-C46-C49

O34-C52-C47-C46-C51

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). J. Chem. Soc. Perkin Trans. 2, pp. S1-S19.
- Chiang, J. F. & Bauer, S. H. (1969). J. Am. Chem. Soc. 91, 1898-1901. Cremer, D. & Pople, J. A. (1975). J. Am. Chem. Soc. 97, 1354-1358.
- Flack, H. D. (1983). Acta Cryst. A39, 876-881.
- Ginsburg, D. (1987). Top. Curr. Chem. 137, 1-17.
- Herbstein, F. H., Ashkenazi P., Kaftory, M., Kapon, M., Reisner, G. M. & Ginsburg, D. (1986). Acta Cryst. B42, 575-601.
- Jamrozik, J. (1985). Monatsh. Chem. 116, 229-235.
- Jamrozik, J. & Żesławski, W. (1994). Chem. Ber. 127, 2471-2474.
- Johnson, C. K. (1976). ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Nardelli, M. (1983). Comput. Chem. 7, 95-98.
- Sheldrick, G. M. (1990). Acta Cryst. A46, 467-473.

Sheldrick, G. M. (1993). SHELXL93. Program for the Refinement of Crystal Structures. Univ. of Göttingen, Germany.

Tobe, Y. (1992). Propellanes in Carboxylic Cage Compounds: Chemistry and Applications, edited by E. Osawa & O. Youemitsu, pp. 125-153, and references therein. Weinheim: VCH.

Acta Cryst. (1995). C51, 2381-2383

4-Sulfonyl-1,6-dioxaspiro[4.5]decanes

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(Received 9 March 1995; accepted 10 May 1995)

Abstract

The relative configurations of 4-(phenylsulfonyl)-1,6dioxaspiro[4.5]decan-10-one ($C_{14}H_{16}O_5S$) and its 2,7dimethyl derivative, 2,7-dimethyl-4-(phenylsulfonyl)-1,6-dioxaspiro[4.5]decan-10-one ($C_{16}H_{20}O_5S$), are confirmed.

Comment

Since the spiroketal unit is found in a wide variety of natural products (Mishima *et al.*, 1975; Kato & Scheuer, 1974), considerable attention has been focused on the development of methods for the synthesis of this moiety (Perron & Albizati, 1989). Most of these methods are based on the thermodynamically controlled acid-catalysed spiroketalization of dihydroxyketones. However, recently, some one-step convergent syntheses from two fragments have been described (Gosselin, Bonfand, Hayes & Maignan, 1994; Ann & Cohen, 1994; Boons, Downham, Kim, Ley & Woods, 1994; Carretero, Díaz & Rojo, 1994).



The structures, with the atomic numbering schemes, of the title compounds (1) and (2) are shown in Figs. 1 and 2. The non-aromatic six-membered ring has a distorted-chair conformation which is slightly flattened around the carbonyl group. This effect is greater for compound (1) [torsion angle C8–C9–C10–C5 35.5 (6) in (1) and 46.3 (3)° in (2)]. The five-membered ring conformation is slightly different in these two compounds. In (1), the ring has an envelope conformation

with C4 (-0.53 Å) out of the plane defined by O1, C2, C3 and C5, whereas in (2) the ring has a half-envelope conformation with C3 (-0.35 Å) and C4 (0.28 Å) out of the plane defined by the other three atoms. The shortening of the C5—O1 and C5—O6 bond distances and the geometrical disposition of these groups (both in axial positions) are similar in the two compounds, probably to maximize the thermodynamic anomeric effect (Deslongchamps, Rowan, Pothier, Sauvé & Saunders, 1981). The sulfone group is in a pseudo-equatorial position in both compounds. In (2), the methyl groups C21 and C71 are in pseudo-axial and equatorial positions, respectively. The most significant difference is the conformation around the C4—S1 bond [torsion angle C5—C4—S1—C11 -42.4 (4) in (1) and 70.5 (2)° in (2)].

Each molecule is involved in one hydrogen bond. In (1), C14—H14···O12ⁱ = 2.490 (6), C14···O12ⁱ = 3.408 (6) Å and C14—H14···O12ⁱ = 169.3 (1)° [symmetry code: (i) x, y + 1, z]; in (2), C4—H4···O10ⁱⁱ = 2.550 (3), C4···O10ⁱⁱ = 3.401 (3) Å and C4—H4···O10ⁱⁱ = 145.1 (1)° [symmetry code: (ii) 1 - x, 1 - y, 2 - z].



Fig. 1. Molecular structure of (1) showing 50% probability displacement ellipsoids.



Fig. 2. Molecular structure of (2) showing 50% probability displacement ellipsoids.