

of *p*-chlorobenzoic acid are hydrogen-bonded in pairs across centres of symmetry, and the pairs are arranged in stacks. A detailed analysis of the crystal packing and of its relationship to the anisotropy of the solid-state reaction with gaseous ammonia has been given (Miller, Paul & Curtin, 1974).

The geometrical parameters obtained here for the O(2)—H(O)···O(1')* hydrogen bond are: distances O(2)···O(1') = 2.618 (2); O(2)—H(O) = 1.07 (4); H(O)···O(1') = 1.56 (4) Å; angle O(2)—H(O)···O(1') = 170 (4)°. Note that the O(2)···O(1') distance is the same as in *p*-fluorobenzoic acid (Colapietro, Domenicano & Pela Ceccarini, 1979).

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* The symmetry operation relating O(1') to O(1) is $-x, -y, -z$.

References

- BRUNO, G. & RANDACCIO, L. (1980). *Acta Cryst.* **B36**, 1711–1712.
- CAMERON, A. F. & DUNCANSON, F. D. (1976). *Acta Cryst.* **B32**, 1991–1994.
- COLAPIETRO, M. & DOMENICANO, A. (1977). *Acta Cryst.* **B33**, 2240–2243.
- COLAPIETRO, M. & DOMENICANO, A. (1978). *Acta Cryst.* **B34**, 3277–3280.
- COLAPIETRO, M., DOMENICANO, A., MARCIANTE, C. & PORTALONE, G. (1981). *Acta Cryst.* **B37**, 387–396.
- COLAPIETRO, M., DOMENICANO, A. & PELA CECCARINI, G. (1979). *Acta Cryst.* **B35**, 890–894.
- COLAPIETRO, M., DOMENICANO, A. & PORTALONE, G. (1980). *Acta Cryst.* **B36**, 354–358.
- DIEHL, P. & JOKISAARI, J. (1979). *J. Mol. Struct.* **53**, 55–60.
- DI RIENZO, F., DOMENICANO, A. & RIVA DI SANSEVERINO, L. (1980). *Acta Cryst.* **B36**, 586–591.
- DOMENICANO, A. & MURRAY-RUST, P. (1979). *Tetrahedron Lett.* pp. 2283–2286.
- DOMENICANO, A. & VACIAGO, A. (1979). *Acta Cryst.* **B35**, 1382–1388.
- DOMENICANO, A., VACIAGO, A. & COULSON, C. A. (1975). *Acta Cryst.* **B31**, 1630–1641.
- ESPIRITU, A. A. & WHITE, J. G. (1977). *Acta Cryst.* **B33**, 3899–3901.
- GOUGOUTAS, J. Z. & LESSINGER, L. (1975). *J. Solid State Chem.* **12**, 51–62.
- HØG, J. H. (1971). Thesis, Univ. of Copenhagen.
- HOPE, H. & VICTOR, D. (1969). *Acta Cryst.* **B25**, 1849–1853.
- KLYNE, W. & PRELOG, V. (1960). *Experientia*, **16**, 521–523.
- MICHEL, F., NERY, H., NOSBERGER, P. & ROUSSY, G. (1976). *J. Mol. Struct.* **30**, 409–415.
- MILLER, R. S., PAUL, I. C. & CURTIN, D. Y. (1974). *J. Am. Chem. Soc.* **96**, 6334–6339.
- OHKURA, K., KASHINO, S. & HAISA, M. (1972). *Bull. Chem. Soc. Jpn*, **45**, 2651–2652.
- PENIONZHKEVICH, N. P., SADOVA, N. I. & VILKOV, L. V. (1979). *Zh. Strukt. Khim.* **20**, 527–529.
- PÉREZ, S. & BRISSE, F. (1975). *Can. J. Chem.* **53**, 3551–3556.
- POLLOCK, J. MCC. & WOODWARD, I. (1954). *Acta Cryst.* **7**, 605.
- RABINOVICH, D. (1969). *J. Chem. Soc. (A)*, pp. 2361–2366.
- SCHULTZ, GY., HARGITTAI, I. & DOMENICANO, A. (1980). *J. Mol. Struct.* **68**, 281–292.
- SPIRLET, M. R., VAN DEN BOSSCHE, G., DIDEBERG, O. & DUPONT, L. (1979). *Acta Cryst.* **B35**, 203–205.
- STOUT, G. H. & JENSEN, L. H. (1968). *X-ray Structure Determination*, p. 411. London: Macmillan.
- WHEELER, G. L. & COLSON, S. D. (1975). *Acta Cryst.* **B31**, 911–913.
- WHEELER, G. L. & COLSON, S. D. (1976). *J. Chem. Phys.* **65**, 1227–1235.

Acta Cryst. (1982). **B38**, 1957–1962

Structures of (*RR*) and (*RS*)-Bis[1-(2,4,6-trimethylphenyl)ethyl] Ether: A Relative-Configuration Problem

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Abstract

$C_{22}H_{30}O$, $M_r = 310.481$; *RR* compound (*A*): monoclinic, $P2_1/c$, $a = 8.0827$ (2), $b = 31.6784$ (26), $c = 7.7624$ (2) Å, $\beta = 103.092$ (2)°, $V = 1935.9$ (2) Å³, $D_x = 1.065$ Mg m⁻³, $Z = 4$, $\mu(\text{Cu } K\alpha) = 0.4471$

mm⁻¹, final $R = 0.053$ for 2217 observed reflexions; *RS* compound (*B*): orthorhombic, $Pc2_1n$, $a = 11.8337$ (6), $b = 10.3267$ (4), $c = 15.5781$ (10) Å, $V = 1903.7$ (1) Å³, $D_x = 1.083$ Mg m⁻³, $Z = 4$, $\mu(\text{Cu } K\alpha) = 0.4546$ mm⁻¹, final $R = 0.067$ for 1193 observed reflexions. A description of the relative configuration of

the asymmetric C atoms is presented in terms of the torsion angles, as well as the twist along the chain. The first compound contains an approximate non-crystallographic twofold axis.



Introduction

This work deals with the solution of relative configuration to distinguish between two stereoisomeric compounds, *A* and *B*, a racemate and a *meso* form, the assignment of which to the actual product was not possible by IR or NMR spectroscopy.

Table 1. Additional crystal data and structure refinement details

	<i>A</i>	<i>B</i>
Unit-cell determination	Least-squares fit from: 60 57 reflexions with $\theta < 45^\circ$	
Crystal size (mm)	0.16 × 0.16 × 0.32	0.10 × 0.16 × 0.40
Stability	No significant variation	
Technique and geometry	Four-circle diffractometer. Philips PW 1100 with graphite-monochromated radiation. $\omega/2\theta$ scan mode	
Total measurements	3265	up to $\theta = 62^\circ$ 3088
No. of independent reflexions	3032	1593
Criterion of obs. reflexions	$I > 2\sigma(I)$	
No. of observed reflexions	2217	1193
Atomic scattering factors	International Tables for X-ray Crystallography (1974)	
Computer programs	XRAY 70 system	
Solution	MULTAN 78 system	
Refinement method	Block diagonal on observed F_o^2	
H atoms	From difference synthesis	
Parameters refined:		
non-H atoms	(Coordinates and anisotropic temperature factors)	
	207	206
H atoms	(Coordinates and isotropic temperature factors)	
	120	
Extinction correction	Not applied	
No. of reflexions per parameter	6.45	3.64
Weighting scheme (see Table 2)	Empirical as to give no trends in $\langle w\Delta^2 F_o \rangle$.	
R (obs.)	0.053	0.067
R_w (obs.)	0.056	0.080

Structure solution

Table 1 shows additional crystal data and structure refinement details. The structure was solved with MULTAN (Main, Hull, Lessinger, Declercq, Germain & Woolfson, 1978) and the XRAY system (Stewart, Kundell & Baldwin, 1970) used on a Univac 1100/80 computer with the least-squares e.s.d.'s as estimates of the precision.* Table 2 gives the weighting scheme employed and Table 3 gives the parameters of the full-normal δR_w plots (Abrahams & Keve, 1971) for

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36762 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. The δR_w full-normal probability plot parameters

	<i>A</i>	<i>B</i>
N	3032	1593
R_T	0.078	0.090
Points excluded	8	1
R	0.077	0.088
Zero intercept	0.010 (1)	0.022 (1)
Slope	0.851 (1)	0.821 (1)
ρ	0.998	0.999
χ^2	0.003	0.002

N : the total number of points. R : the usual crystallographic disagreement index. DP: the statistic: $|F_o - F_c|/\sqrt{w}$. ρ : the linear correlation coefficient. Points excluded if $|\text{DP}| < 3$.

Table 2. Weighting scheme

$$w = K/[f_1(F_o)]^2, f_2(\sin \theta/\lambda), f_1(F_o) = A_1 + B_1|F_o| + C_1|F_o|^2, f_2(\sin \theta/\lambda) = A_2 + B_2(\sin \theta/\lambda) + C_2(\sin \theta/\lambda)^2.$$

Compound	<i>A</i>			<i>B</i>		
K	0.5848			0.6922		
Coefficients	A_1	B_1	C_1	A_1	B_1	C_1
$ F_o \leq 2.7$	0.5361	0.2179	-0.0671	1.0174	-0.0622	-0.0023
$2.7 < F_o \leq 5.4$	1.4920	-0.5036	0.0644	0.7756	-0.0474	-0.0017
$5.4 < F_o \leq 6.0$	0.7048	-0.0165	-	0.3863	0.0256	-
$6.0 < F_o \leq 9.0$	0.5126	-0.0120	-	0.3182	0.0211	-
$9.0 < F_o \leq 20.0$	0.1847	0.0160	-	0.2559	0.0170	-
$20.0 < F_o $	0.1092	0.0129	-			
	A_2	B_2	C_2	A_2	B_2	C_2
$\sin \theta/\lambda \leq 0.40 \text{ \AA}^{-1}$	7.5943	-77.6018	90.3015	6.4467	-15.2881	7.7661
$0.40 < \sin \theta/\lambda$	2.5365	-3.4512	-	6.4229	-12.7305	5.5703

both compounds, showing the accuracy of the weighting scheme.

Results

The atomic numbering is in Fig. 1, where the structures are shown projected along the C(1)···C(3) line. The positional parameters and the bond distances and angles are presented in Tables 4, 5 and 6. In Fig. 2 (ORTEP, Johnson, 1971) the conformational and configurational differences are apparent, that is the interchange between Me and R at C(1), and this was taken into account for the half-normal probability comparison given below. In Table 7 are the main torsion angles describing the molecule.

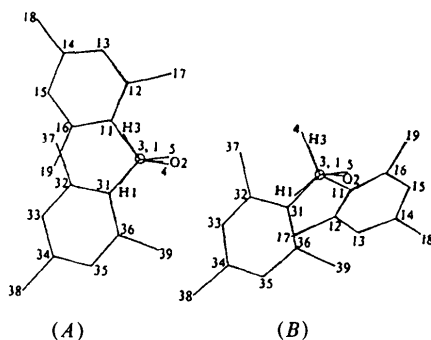


Fig. 1. Atomic numbering for both molecules showing the configuration along C(1)···C(3).

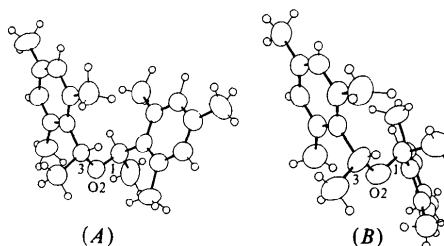


Fig. 2. Comparison of the relative configurations in both molecules.

Discussion

The interatomic distances up to 3.5 Å have been compared by a half-normal probability plot, see Fig. 3. Of the seven points excluded from the fit, the main differences are due to O(2)—C(3) and C(3)—C(5) being shorter and C(3)—C(31) longer in compound B than in A. Another marked difference is in the angles at C(1), which seem to be interchanged, as if the R substituent has no influence on the bond angle: O(2)—C(1)—C(4) = 106.4° in A vs 113.2° in B and O(2)—C(1)—C(11) = 113.9° in A vs 108.9° in B. These are the main features indicating that the C(5)—C(3)—O(2)—C(1)—C(4)/C(11) chain is slightly more open in compound B than in compound A.

For each asymmetric C atom, the relative configuration can be described in terms of the configurational angles (Foces-Foces, Cano & Garcia-Blanco, 1980): $\rho = \tau - \tau_0$, τ being the experimental

Table 4. Atomic coordinates and equivalent isotropic thermal parameters for the heavy atoms

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j \cos(a_i, a_j).$$

	Compound A				Compound B			
	x	y	z	U_{eq} (Å ² × 10 ⁴)	x	y	z	U_{eq} (Å ² × 10 ³)
C(1)	0.53956 (40)	0.11576 (9)	0.35178 (38)	729 (10)	0.6816 (6)	0.1606 (8)	0.1836 (4)	68 (2)
O(2)	0.61708 (23)	0.12982 (6)	0.52622 (24)	717 (7)	0.6793 (5)	0.2500	0.1131 (3)	85 (2)
C(3)	0.49879 (36)	0.13943 (9)	0.63179 (35)	661 (9)	0.7549 (8)	0.2317 (8)	0.0479 (5)	83 (3)
C(4)	0.68460 (64)	0.10500 (15)	0.26270 (62)	1049 (18)	0.7836 (7)	0.1799 (14)	0.2432 (7)	99 (4)
C(5)	0.60200 (53)	0.15761 (14)	0.80425 (48)	947 (14)	0.7445 (12)	0.3388 (9)	−0.0161 (7)	95 (4)
C(11)	0.41334 (34)	0.14702 (8)	0.24622 (33)	616 (9)	0.5708 (6)	0.1726 (8)	0.2324 (4)	64 (2)
C(12)	0.45177 (35)	0.18993 (8)	0.23370 (36)	666 (10)	0.5104 (7)	0.0590 (7)	0.2516 (4)	63 (2)
C(13)	0.32923 (38)	0.21673 (9)	0.13665 (41)	725 (11)	0.4110 (7)	0.0643 (8)	0.2968 (4)	65 (2)
C(14)	0.17113 (38)	0.20278 (9)	0.04991 (38)	717 (11)	0.3676 (6)	0.1812 (8)	0.3265 (4)	64 (2)
C(15)	0.13700 (42)	0.16044 (10)	0.06196 (39)	757 (11)	0.4298 (6)	0.2924 (8)	0.3102 (4)	64 (3)
C(16)	0.25389 (38)	0.13228 (9)	0.15685 (34)	690 (10)	0.5310 (6)	0.2920 (8)	0.2642 (4)	66 (2)
C(17)	0.62008 (50)	0.20926 (14)	0.32271 (68)	967 (16)	0.5512 (11)	−0.0748 (9)	0.2213 (7)	85 (4)
C(18)	0.04028 (48)	0.23298 (12)	−0.05296 (53)	1020 (15)	0.2585 (6)	0.1872 (9)	0.3731 (4)	77 (2)
C(19)	0.20110 (75)	0.08650 (13)	0.16021 (60)	1023 (18)	0.5885 (12)	0.4191 (13)	0.2488 (8)	82 (4)
C(31)	0.38820 (32)	0.10258 (8)	0.66140 (32)	576 (8)	0.7485 (7)	0.0953 (7)	0.0047 (4)	64 (2)
C(32)	0.21373 (33)	0.10914 (8)	0.64367 (33)	612 (9)	0.8502 (6)	0.0293 (8)	−0.0108 (4)	62 (2)
C(33)	0.11210 (37)	0.07605 (9)	0.67431 (39)	685 (10)	0.8474 (7)	−0.0882 (8)	−0.0498 (4)	63 (2)
C(34)	0.17550 (36)	0.03627 (8)	0.72163 (40)	698 (11)	0.7483 (7)	−0.1476 (7)	−0.0765 (4)	64 (2)
C(35)	0.34699 (37)	0.03024 (9)	0.73874 (42)	712 (11)	0.6496 (7)	−0.0815 (9)	−0.0638 (4)	68 (3)
C(36)	0.45472 (33)	0.06218 (9)	0.71013 (36)	646 (9)	0.6459 (7)	0.0402 (9)	−0.0239 (4)	73 (3)
C(37)	0.13019 (48)	0.15147 (11)	0.59286 (54)	786 (13)	0.9643 (8)	0.0865 (12)	0.0167 (7)	87 (4)
C(38)	0.06239 (62)	0.00089 (13)	0.75692 (85)	999 (19)	0.7504 (13)	−0.2780 (9)	−0.1172 (6)	89 (4)
C(39)	0.64029 (44)	0.05076 (15)	0.73247 (71)	953 (16)	0.5354 (11)	0.1062 (15)	−0.0138 (9)	100 (5)

Table 5. Final positional parameters and bond distances for the H atoms

	Compound A				Compound B			
	x	y	z	C-H (Å)	x	y	z	C-H (Å)
H(1)	0.469 (4)	0.089 (1)	0.368 (4)	1.03 (3)	0.683 (7)	0.069 (9)	0.161 (5)	1.01 (9)
H(3)	0.421 (3)	0.162 (1)	0.568 (3)	1.00 (3)	0.829 (11)	0.252 (14)	0.077 (8)	1.01 (13)
H(4a)	0.769 (6)	0.086 (1)	0.344 (6)	1.02 (4)	0.775 (7)	0.267 (12)	0.270 (6)	1.00 (12)
H(4b)	0.754 (5)	0.133 (1)	0.239 (5)	1.09 (4)	0.851 (8)	0.161 (10)	0.207 (5)	0.99 (9)
H(4c)	0.631 (5)	0.092 (1)	0.153 (6)	0.96 (4)	0.772 (6)	0.125 (7)	0.288 (5)	0.91 (8)
H(5a)	0.674 (5)	0.181 (1)	0.775 (5)	0.99 (4)	0.746 (9)	0.422 (13)	0.014 (7)	0.98 (13)
H(5b)	0.684 (5)	0.135 (1)	0.867 (6)	1.02 (4)	0.671 (7)	0.348 (8)	-0.037 (5)	0.93 (8)
H(5c)	0.526 (6)	0.166 (1)	0.881 (6)	0.98 (5)	0.799 (12)	0.328 (16)	-0.065 (10)	1.00 (15)
H(13)	0.362 (4)	0.246 (1)	0.125 (4)	0.98 (4)	0.359 (10)	-0.013 (13)	0.311 (8)	1.03 (13)
H(15)	0.027 (5)	0.150 (1)	-0.000 (5)	0.97 (3)	0.403 (4)	0.375 (5)	0.332 (3)	0.97 (6)
H(17a)	0.614 (7)	0.238 (2)	0.322 (7)	0.90 (6)	0.633 (6)	-0.067 (9)	0.234 (5)	0.99 (8)
H(17b)	0.694 (9)	0.212 (2)	0.246 (10)	0.94 (8)	0.534 (10)	-0.109 (14)	0.159 (9)	1.05 (14)
H(17c)	0.699 (7)	0.191 (2)	0.402 (7)	0.97 (5)	0.515 (6)	-0.156 (9)	0.241 (5)	0.99 (9)
H(18a)	0.085 (8)	0.261 (2)	-0.064 (8)	0.96 (6)	0.243 (9)	0.270 (11)	0.409 (8)	1.03 (12)
H(18b)	-0.044 (7)	0.237 (2)	0.011 (7)	0.93 (6)	0.271 (12)	0.132 (14)	0.403 (9)	0.75 (15)
H(18c)	-0.025 (5)	0.221 (1)	-0.144 (6)	0.87 (4)	0.188 (8)	0.184 (13)	0.331 (6)	1.07 (10)
H(19a)	0.206 (6)	0.077 (1)	0.279 (7)	0.96 (5)	0.603 (9)	0.428 (12)	0.184 (8)	1.03 (12)
H(19b)	0.279 (7)	0.070 (2)	0.108 (7)	0.97 (6)	0.657 (11)	0.420 (16)	0.293 (9)	1.06 (13)
H(19c)	0.078 (6)	0.083 (1)	0.090 (6)	1.03 (5)	0.544 (6)	0.481 (8)	0.255 (4)	0.83 (8)
H(33)	-0.008 (4)	0.082 (1)	0.662 (4)	0.97 (4)	0.902 (9)	-0.127 (11)	-0.058 (6)	0.77 (11)
H(35)	0.395 (4)	0.003 (1)	0.776 (4)	0.97 (3)	0.590 (8)	-0.118 (10)	-0.080 (6)	0.84 (10)
H(37a)	0.146 (4)	0.162 (1)	0.478 (5)	0.99 (4)	0.981 (15)	0.175 (22)	-0.021 (12)	1.10 (21)
H(37b)	0.183 (5)	0.172 (1)	0.670 (5)	0.93 (4)	0.993 (7)	0.110 (9)	0.082 (6)	1.09 (9)
H(37c)	0.005 (5)	0.150 (1)	0.597 (5)	1.02 (4)	1.001 (11)	0.032 (12)	-0.014 (7)	0.86 (12)
H(38a)	-0.013 (8)	0.008 (2)	0.830 (8)	0.95 (7)	0.799 (11)	-0.301 (15)	-0.160 (9)	0.91 (13)
H(38b)	0.121 (8)	-0.022 (2)	0.818 (8)	0.94 (6)	0.683 (7)	-0.290 (11)	-0.143 (6)	0.90 (9)
H(38c)	-0.021 (9)	-0.001 (2)	0.667 (9)	0.86 (6)	0.785 (9)	-0.345 (12)	-0.087 (7)	0.93 (12)
H(39a)	0.682 (9)	0.055 (2)	0.629 (10)	0.95 (8)	0.517 (13)	0.146 (16)	0.034 (11)	0.88 (17)
H(39b)	0.664 (6)	0.024 (2)	0.773 (7)	0.91 (5)	0.481 (9)	0.062 (9)	0.001 (5)	0.82 (10)
H(39c)	0.716 (7)	0.073 (2)	0.793 (8)	0.98 (5)	0.482 (13)	0.114 (14)	-0.063 (10)	1.00 (15)

Table 6. Bond distances (Å) and angles (°)

	A	B		A	B
C(1)—O(2)	1.428 (3)	1.434 (8)	C(14)—C(18)	1.513 (5)	1.483 (10)
C(1)—C(4)	1.528 (7)	1.536 (12)	C(15)—C(16)	1.384 (4)	1.395 (10)
C(1)—C(11)	1.521 (4)	1.521 (10)	C(16)—C(19)	1.514 (5)	1.498 (16)
O(2)—C(3)	1.426 (4)	1.366 (10)	C(31)—C(32)	1.401 (4)	1.404 (11)
C(3)—C(5)	1.521 (4)	1.496 (13)	C(31)—C(36)	1.406 (4)	1.413 (12)
C(3)—C(31)	1.519 (4)	1.563 (11)	C(32)—C(33)	1.385 (4)	1.356 (11)
C(11)—C(12)	1.403 (4)	1.406 (11)	C(32)—C(37)	1.513 (4)	1.534 (13)
C(11)—C(16)	1.398 (4)	1.410 (11)	C(33)—C(34)	1.379 (4)	1.388 (11)
C(12)—C(13)	1.390 (4)	1.371 (11)	C(34)—C(35)	1.376 (4)	1.367 (11)
C(12)—C(17)	1.509 (5)	1.538 (12)	C(34)—C(38)	1.510 (6)	1.489 (12)
C(13)—C(14)	1.376 (4)	1.391 (11)	C(35)—C(36)	1.385 (4)	1.404 (13)
C(14)—C(15)	1.377 (4)	1.387 (11)	C(36)—C(39)	1.514 (5)	1.483 (16)
	A	B		A	B
C(4)—C(1)—C(11)	113.0 (3)	111.4 (6)	C(11)—C(16)—C(15)	119.4 (3)	118.0 (8)
O(2)—C(1)—C(11)	113.9 (2)	108.9 (2)	C(15)—C(16)—C(19)	117.8 (3)	118.0 (8)
O(2)—C(1)—C(4)	106.4 (3)	113.2 (7)	C(11)—C(16)—C(19)	122.9 (3)	123.9 (8)
C(1)—O(2)—C(3)	113.8 (2)	117.9 (5)	C(3)—C(31)—C(36)	122.5 (2)	122.7 (8)
O(2)—C(3)—C(31)	114.7 (2)	114.5 (7)	C(3)—C(31)—C(32)	119.0 (2)	118.1 (7)
O(2)—C(3)—C(5)	106.3 (3)	109.8 (8)	C(32)—C(31)—C(36)	118.5 (2)	119.1 (7)
C(5)—C(3)—C(31)	112.5 (3)	112.0 (6)	C(31)—C(32)—C(37)	122.5 (3)	121.2 (8)
C(1)—C(11)—C(16)	118.7 (2)	118.5 (7)	C(31)—C(32)—C(33)	119.6 (2)	119.4 (7)
C(1)—C(11)—C(12)	122.4 (2)	122.4 (7)	C(33)—C(32)—C(37)	117.9 (3)	119.4 (8)
C(12)—C(11)—C(16)	118.9 (3)	119.0 (6)	C(32)—C(33)—C(34)	122.6 (3)	123.4 (7)
C(11)—C(12)—C(17)	123.5 (3)	121.6 (7)	C(33)—C(34)—C(38)	121.5 (3)	120.9 (9)
C(11)—C(12)—C(13)	119.2 (3)	120.8 (7)	C(33)—C(34)—C(35)	117.3 (3)	117.3 (7)
C(13)—C(12)—C(17)	117.3 (3)	117.6 (8)	C(35)—C(34)—C(38)	121.2 (3)	121.8 (9)
C(12)—C(13)—C(14)	122.6 (3)	121.4 (7)	C(34)—C(35)—C(36)	122.6 (3)	122.6 (8)
C(13)—C(14)—C(18)	121.1 (3)	121.1 (7)	C(31)—C(36)—C(35)	119.5 (3)	118.3 (7)
C(13)—C(14)—C(15)	117.3 (3)	117.5 (6)	C(35)—C(36)—C(39)	116.7 (3)	119.1 (9)
C(15)—C(14)—C(18)	121.7 (3)	121.4 (7)	C(31)—C(36)—C(39)	123.8 (3)	122.7 (9)
C(14)—C(15)—C(16)	122.7 (3)	123.2 (7)			

value for the torsion angle and τ_0 a reference internal rotation. Taking as reference (τ_0) that angle which makes the chain $R-C-O-C-R$ planar (staggering the substituents such that the $C-O-C-R$ torsion angles take the value π), we have the values given in Table 8 describing the configuration of the asymmetric C atoms C(1) and C(3). We can see that in compound *A* both C atoms have the same relative configuration, namely $\{R_\pi; H_+, Me_-\}$, while in molecule *B* both C atoms have a different configuration: $\{R_\pi; H(1)_-, Me(4)_+\}$ vs $\{R_\pi; H(3)_+, Me(5)_-\}$ (see Table 8), representing the

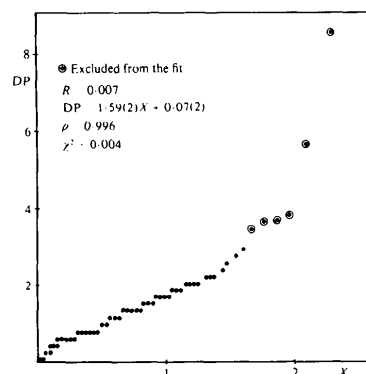


Fig. 3. Half-normal probability plot comparison of distances up to 3.5 Å between both isomers.

Table 7. Main torsional angles ($^\circ$)

	<i>A</i>	<i>B</i>
H(1)–C(1)–C(3)–C(31)	–2.6 (16)	5.7 (45)
H(1)–C(1)–C(3)–H(3)	–109.6 (22)	–109.2 (88)
C(11)–C(1)–C(3)–H(31)	103.3 (2)	–105.7 (7)
C(11)–C(1)–C(3)–H(3)	–3.7 (15)	139.4 (76)
C(11)–C(1)–C(3)–C(5)	–121.5 (4)	30.1 (14)
H(1)–C(1)–C(3)–C(5)	132.5 (17)	141.5 (46)
C(4)–C(1)–C(3)–C(5)	11.8 (5)	–110.2 (13)
C(4)–C(1)–C(3)–C(31)	–123.4 (3)	114.1 (7)
C(4)–C(1)–C(3)–H(3)	129.6 (16)	–0.9 (76)
H(3)–C(3)–O(2)–C(1)	–58.3 (15)	–68.2 (78)
C(5)–C(3)–O(2)–C(1)	–174.7 (2)	–176.3 (7)
C(31)–C(3)–O(2)–C(1)	60.3 (3)	56.7 (8)
C(4)–C(1)–O(2)–C(3)	–177.3 (3)	73.5 (7)
H(1)–C(1)–O(2)–C(3)	–58.6 (13)	–47.6 (48)
C(11)–C(1)–O(2)–C(3)	57.6 (3)	–162.5 (6)
O(2)–C(1)–C(11)–C(16)	–132.1 (3)	–54.2 (9)
O(2)–C(1)–C(11)–C(12)	48.7 (4)	130.4 (6)
O(2)–C(3)–C(31)–C(36)	43.3 (4)	49.6 (10)
O(2)–C(3)–C(31)–C(32)	–135.2 (2)	–134.3 (7)

interchange in the substituents, C(4) vs C(11), as it appears in Fig. 2. That is, compounds *A* and *B* have the relative configurations *RR* and *RS* respectively.

Considering the molecule as a whole and the pseudo-rotation angles around the $C(1)\cdots C(3)$ line, compound *A* would have a *threo* configuration (when analogous substituents at each end are staggered by π , the other two substituents are not similarly staggered) and compound *B* an *erythro* one (when staggering analogous substituents by π , the others are also staggered by π) (see Fig. 1). This is characterized by the pseudo-rotation and the pseudo-configurational angles when staggering the *R* groups (Table 8). The values for *A* are near those for the theoretical *threo* configuration ($0, +120, -120^\circ$) and for *B* are near those

Table 8. Configurational and conformational characteristics

All values in degrees (e.s.d.'s are 0.5 and 0.8° for *A* and *B* respectively).

Angle	Compound <i>A</i>	Compound <i>B</i>							
(a) Configurational angles									
$\rho[C(11)–C(1)^*–O(2)–C(3)]$	$57.6 + 122.4 = 180$	$–162.5 – 17.5 = –180$							
$\rho[H(1)–C(1)^*–O(2)–C(3)]$	$–58.6 + 122.4 = 63.8$	$–47.6 – 17.5 = –65.1$							
$\rho[C(4)–C(1)^*–O(2)–C(3)]$	$–177.3 + 122.4 = –54.9$	$73.5 – 17.5 = 56.0$							
$\rho[C(1)–O(2)–C(3)^*–C(31)]$	$60.3 + 119.7 = 180$	$56.7 + 123.3 = 180$							
$\rho[C(1)–O(2)–C(3)^*–H(3)]$	$–58.3 + 119.7 = 61.4$	$–68.2 + 123.3 = 55.1$							
$\rho[C(1)–O(2)–C(3)^*–C(5)]$	$–174.7 + 119.7 = –55.0$	$–176.3 + 123.3 = –53.0$							
(b) Pseudorotational angles around the $C(1)\cdots C(3)$ line									
$C(11)–C(1)\cdots C(3)–C(31)$	103.3	–105.7							
$H(1)–C(1)\cdots C(3)–H(3)$	–109.7	–109.2							
$C(4)–C(1)\cdots C(3)–C(5)$	11.8	–110.2							
(c) Pseudoconfigurational angles around the $C(1)\cdots C(3)$ line									
$\rho[C(11)–C(1)\cdots C(3)–C(31)]$	$103.3 – 103.3 = 0$	$–105.7 + 105.7 = 0$							
$\rho[H(1)–C(1)\cdots C(3)–H(3)]$	$–109.7 – 103.3 = –213.0$	$–109.2 + 105.7 = –3.6$							
$\rho[C(4)–C(1)\cdots C(3)–C(5)]$	$11.8 – 103.3 = –91.5$	$–110.2 + 105.7 = –4.5$							
(d) Deformations at the substituents of the asymmetric C atoms									
Compound	Atom	H \rightarrow Me	Me \rightarrow R	H \rightarrow R	Compound	Atom	H \rightarrow Me	Me \rightarrow R	H \rightarrow R
<i>A</i>	C(1)	–1.3	5.1	–3.8	<i>B</i>	C(1)	1.1	4.0	–5.1
<i>A</i>	C(3)	–3.6	5.0	–1.4	<i>B</i>	C(3)	–11.9	7.0	4.9

for the *erythro* (0,0,0°), in terms of the pseudo-configurational angles.

The torsion angles of Table 7 give a conformation for molecule *A* with both Me groups almost staggered, and for molecule *B* with *R* at C(1) staggered with the Me group at C(3), the other substituents in configurations as described. The deviations from this theoretical reference conformation (Cano, Foces-Foces & García-Blanco, 1979) are $\Delta\tau = 0.6^\circ$ at C(1)–C(2) and 2.4° at C(3)–O(2) for compound *A* and 14.5 and -2.6° respectively for compound *B*, to give overall twists of 3.0° in *A* and 11.9° in *B*. This means that from the reference conformation with the chain R–C–O–C–R planar, the torsion angles are -119.4 , -117.6 and 14.5 , -122.6° , respectively, as before, to give overall twists of 123.0° for compound *A* and -108.1° for compound *B* around the C(1)···C(3) line.

There are some deformations (Cano *et al.*, 1979) in the substituents of C(1) and C(3) from the ideal sp^3 geometry, which are given in Table 8.

The angles between rings are 30.3 and 40.3° respectively for each compound. These rings are about 48° on average from the O–C–C plane.

In compound *A* an approximate non-crystallographic twofold axis relates both halves of the molecule, passing, when adjusted by least squares through the mid-points of the related atoms, almost through O(2) and running almost parallel to the *x* axis.

Finally, no relevant intermolecular contacts have been detected in the packing.

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References

- ABRAHAM, S. C. & KEVE, E. T. (1971). *Acta Cryst.* **A27**, 157–165.
 CANO, F. H., FOCES-FOCES, C. & GARCÍA-BLANCO, S. (1979). *J. Cryst. Mol. Struct.* **9**, 107–115.
 FOCES-FOCES, C., CANO, F. H. & GARCÍA-BLANCO, S. (1980). *Acta Cryst.* **B36**, 377–384.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
 JOHNSON, C. K. (1971). *ORTEP II*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
 MAIN, P., HULL, S. E., LESSINGER, L., DECLERCQ, J. P., GERMAIN, G. & WOOLFSON, M. M. (1978). *MULTAN 78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
 STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Acta Cryst. (1982). **B38**, 1962–1965

Structure of 5-Acetyl-1-(3,5-*O*-isopropylidene- β -D-xylofuranosyl)uracil

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Abstract

The structure of 5-acetyl-1-(3,5-*O*-isopropylidene- β -D-xylofuranosyl)uracil (β -AXU), a nucleoside with a six-membered isopropylidene ring linked to a five-membered xylose ring, has been solved by direct methods and refined to a final residual *R* of 0.048 over 1684 reflections. $C_{14}H_{18}N_2O_7$, $M_r = 326$, is orthorhombic, $P2_12_12_1$, with $a = 6.135$ (1), $b = 9.227$ (2), $c = 26.11$ (1) Å, $Z = 4$, $V = 1478$ (1) Å³.

$D_m = 1.44$ (1), $D_x = 1.46$ Mg m⁻³, μ (Cu $K\alpha$) = 1.05 mm⁻¹. The slightly non-planar [maximum displacement of a ring atom is 0.03 (2) Å for the imido N(2)] uracil ring is β -linked to a 3'-endo, 4'-exo (³T₄ puckered) xylofuranose ring. Location of all 18 H atoms revealed a 2.715 (4) Å intermolecular hydrogen bond between OH(2') of the sugar and the acetyl O of the uracil.

Introduction

From its mode of preparation (Cusack, Robinson, Rugg, Shaw & Lofthouse, 1974), 5-acetyl-1-(3,5-

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