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2,2′,3,3′-Tetra-*O*-benzoyl-6,6′-dideoxy-4,4′-di-*O*-mesyl-6,6′-dithiocyanato-α,α-trehalose†

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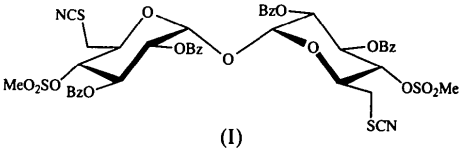
Abstract

The low-temperature X-ray crystal structure of 2,3-di-*O*-benzoyl-6-deoxy-4-*O*-mesyl-6-thiocyanato-α-D-glucopyranosyl 2,3-di-*O*-benzoyl-6-deoxy-4-*O*-mesyl-6-thiocyanato-α-D-glucopyranoside (the title compound), $C_{44}H_{40}N_2O_{17}S_4$, is reported. The absolute configuration has been determined. The molecule has only approximate C_2 symmetry but the differences in the orientation of the C(5) substituents and the torsion angles about the glycosidic linkage are very much less than in α,α-trehalose and its derivatives. Each of the hexopyranosyl residues has a nearly perfect $^4C_1$ conformation. The planes of the phenyl rings of the benzoyl groups are oriented such that they are approximately perpendicular to the plane of the parent pyranose ring.

† Crystal Structures of Trehalose Derivatives, Part 9. For Part 8, see Linden & Lee (1995).

Comment

α,α-Trehalose is widespread in nature, where it is found in bacteria, insect blood, fungi, algae lichens and some higher plants (Birch, 1963; Elbein, 1974; Lee, 1980). It is a non-reducing disaccharide, consisting of two α-D-glucopyranosyl residues, linked by a glycosidic O-atom bridge between their anomeric C atoms. We are interested in this disaccharide for two reasons. Firstly, the similarity of its structure to many food sugars and its unique molecular symmetry and conformational stability under relatively severe reaction conditions make it an ideal model in studies of the relationship between molecular structure and the organoleptic effect (Lee, 1987). Secondly, trehalases, which are distributed far wider in nature than their substrate, are highly specific glucosidases whose specificities, as well as those of related enzymes, for trehalose, and its analogues, are of interest (Elbein, 1974; Labat-Robert, 1982). Modified analogues of trehalose are important for such studies. The synthesis of the title compound, (I), has been described by Birch, Lee & Richardson (1974). We now report its X-ray crystal structure.



(I)

A view of (I) showing the displacement ellipsoids and the atomic numbering is given in Fig. 1. The figure depicts the correct absolute configuration of the molecule, which was assigned to agree with that of its known precursor (Birch, Lee & Richardson, 1974) and was further confirmed by the X-ray analysis.

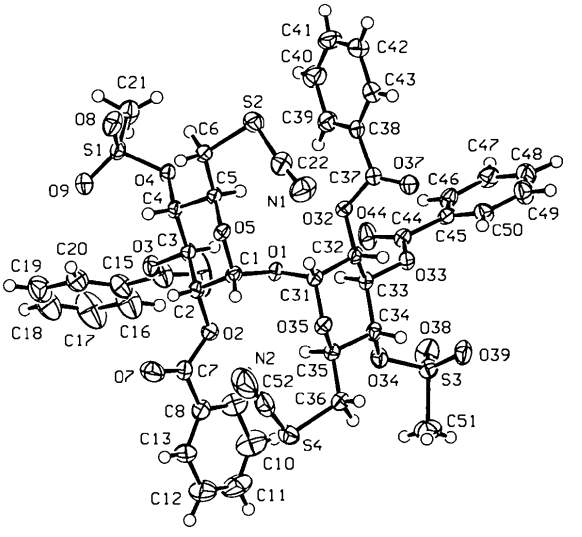


Fig. 1. View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by spheres of arbitrary size.

Bond lengths and bond angles exhibit normal values and generally correspond with those in other sugars (Berman, Chu & Jeffrey, 1967). The molecule has approximate C_2 symmetry with the major difference between the two pyranosyl residues being the relative orientations of the mesyl substituents at C(4) and C(34), with small differences in the orientations of the benzoyl substituents. The two glycosidic torsion angles, O(5)—C(1)—O(1)—C(31) and O(35)—C(31)—O(1)—C(1), show smaller differences (3.1°) than those observed in α,α-trehalose (approximately 13°) (Brown *et al.*, 1972; Taga, Senma & Osaki, 1972) and many of its analogues which do not possess crystallographic C_2 symmetry (range: 4.5–19°) (Lee & Koh, 1994; Lee, Koh, Xu & Linden, 1994; Lee & Linden, 1994; Linden & Lee, 1995). Similarly, the relative conformations of the substituents at C(5) and C(35) differ only very slightly. The torsion angles about the C(5)—C(6) and C(35)—C(36) bonds, which differ for the two pyranose rings by less than 2°, describe the *gauche–trans* arrangement in both rings. In this arrangement, the thiocyanate groups are at their farthest separation from the mesyl substituents. In α,α-trehalose and many of its analogues (Brown *et al.*, 1972; Taga, Senma & Osaki, 1972; Jeffrey & Nanni, 1985; Lee & Koh, 1994; Lee & Linden, 1994), the conformational differences between the C(5) substituents of the two glycosyl moieties are considerable. In all respects, therefore, the pyranosyl residues of (I) are more symmetrically oriented about the glycosidic O atom than in α,α-trehalose dihydrate (Brown *et al.*, 1972).

Both pyranose rings adopt slightly distorted $^4C_1$ conformations, with puckering parameters (Cremer & Pople, 1975) Q = 0.600 and 0.585 Å, θ = 5.97 and 2.78°, φ_2 = 216.5 and 211.6°, q_2 = 0.062 and 0.028 Å, and q_3 = 0.596 and 0.584 Å, for the rings defined by C(1)–O(5) and C(31)–O(35), respectively. The distortion is towards the $^4H_3$ conformation, as indicated by the φ_2 values (approximately 210°). The flattening of the O-atom apex allows the ring C—O—C angle in each of the pyranose rings to widen to 114.7 (2)°.

In each of the benzoyl substituents the plane of the phenyl ring is approximately parallel to that containing the carboxyl C atom, the two carboxyl O atoms and the attached pyranose ring C atom. These planes are, in turn, approximately perpendicular to the pyranose ring plane. This conformation ensures minimum steric interaction between the benzoyl groups and neighbouring substituents. There are no unusually short inter- or intramolecular contacts in the crystal.

Experimental

Synthesis was as reported by Birch, Lee & Richardson (1974). Suitable crystals were obtained from an ethanol solution.

Crystal data

$C_{44}H_{40}N_2O_{17}S_4$	Mo $K\alpha$ radiation
M_r = 997.04	λ = 0.71069 Å
Orthorhombic	Cell parameters from 24 reflections
$P2_12_12_1$	θ = 14–19.5°
a = 19.051 (3) Å	μ = 0.261 mm^{-1}
b = 26.274 (4) Å	T = 173 (1) K
c = 9.487 (3) Å	Prism
V = 4749 (2) Å^3	0.50 × 0.33 × 0.33 mm
Z = 4	Colourless
D_x = 1.394 Mg m^{-3}	

Data collection

Rigaku AFC-5*R* diffractometer	R_{int} = 0.025
ω scans	θ_{max} = 30°
Absorption correction: none	$h = -22 \rightarrow 26$
14 607 measured reflections	$k = -33 \rightarrow 36$
11 798 independent reflections	$l = -12 \rightarrow 13$
8854 observed reflections [$I > 3\sigma(I)$]	3 standard reflections monitored every 150 reflections
	intensity decay: insignificant

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = 0.0007
R = 0.0373	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
wR = 0.0381	$\Delta\rho_{min}$ = −0.25 e Å^{-3}
S = 1.376	Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
8854 reflections	
764 parameters	
All H-atom parameters refined	
$w = 1/[\sigma^2(F_o) + (0.01F_o)^2]$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S(1)	0.33688 (3)	0.73915 (2)	0.16411 (7)	0.0294 (2)
S(2)	0.54334 (4)	0.85604 (2)	0.33745 (8)	0.0373 (2)
S(3)	0.81158 (3)	0.54648 (2)	0.59680 (7)	0.0298 (2)
S(4)	0.59571 (4)	0.59573 (3)	0.97648 (8)	0.0423 (2)
O(1)	0.57668 (8)	0.69455 (6)	0.5567 (2)	0.0246 (5)
O(2)	0.47618 (8)	0.62476 (6)	0.6284 (2)	0.0306 (5)
O(3)	0.38741 (8)	0.63642 (6)	0.3916 (2)	0.0308 (5)
O(4)	0.41283 (8)	0.72325 (6)	0.2209 (2)	0.0267 (5)
O(5)	0.49451 (8)	0.76076 (6)	0.5547 (2)	0.0251 (5)
O(7)	0.3875 (1)	0.62365 (8)	0.7839 (3)	0.0595 (8)
O(8)	0.32818 (9)	0.79248 (7)	0.1866 (2)	0.0430 (6)
O(9)	0.28539 (9)	0.70647 (8)	0.2221 (2)	0.0461 (7)
O(14)	0.4642 (1)	0.58184 (9)	0.2936 (4)	0.088 (1)
O(32)	0.68245 (8)	0.73502 (6)	0.4005 (2)	0.0275 (5)
O(33)	0.77891 (8)	0.65215 (6)	0.4140 (2)	0.0269 (5)
O(34)	0.73608 (8)	0.57209 (6)	0.6119 (2)	0.0306 (5)
O(35)	0.64969 (8)	0.68590 (6)	0.7529 (2)	0.0264 (5)
O(37)	0.78190 (9)	0.78144 (8)	0.4140 (2)	0.0508 (7)
O(38)	0.8103 (1)	0.51902 (8)	0.4682 (2)	0.0518 (7)
O(39)	0.86459 (8)	0.58373 (7)	0.6216 (2)	0.0411 (6)
O(44)	0.71971 (9)	0.61776 (8)	0.2315 (2)	0.0453 (7)
N(1)	0.6181 (1)	0.8497 (1)	0.5940 (3)	0.061 (1)
N(2)	0.5276 (2)	0.6911 (1)	0.9958 (4)	0.076 (1)
C(1)	0.5123 (1)	0.71265 (9)	0.6120 (3)	0.0245 (7)
C(2)	0.4555 (1)	0.67415 (9)	0.5782 (3)	0.0266 (7)
C(3)	0.4436 (1)	0.67146 (9)	0.4203 (3)	0.0254 (7)
C(4)	0.4230 (1)	0.72440 (9)	0.3720 (3)	0.0243 (7)

C(5)	0.4832 (1)	0.76095 (9)	0.4050 (3)	0.0234 (6)
C(6)	0.4671 (1)	0.8157 (1)	0.3640 (3)	0.0292 (8)
C(7)	0.4365 (1)	0.60294 (9)	0.7300 (3)	0.0327 (8)
C(8)	0.4624 (1)	0.55164 (9)	0.7673 (3)	0.0333 (8)
C(9)	0.5138 (2)	0.5274 (1)	0.6890 (3)	0.046 (1)
C(10)	0.5403 (2)	0.4811 (1)	0.7365 (5)	0.065 (1)
C(11)	0.5170 (2)	0.4600 (1)	0.8597 (4)	0.062 (1)
C(12)	0.4649 (2)	0.4830 (1)	0.9340 (5)	0.059 (1)
C(13)	0.4380 (2)	0.5291 (1)	0.8899 (4)	0.047 (1)
C(14)	0.4049 (2)	0.5931 (1)	0.3232 (4)	0.0447 (9)
C(15)	0.3420 (2)	0.5615 (1)	0.2916 (3)	0.0427 (9)
C(16)	0.3542 (2)	0.5131 (1)	0.2391 (5)	0.069 (1)
C(17)	0.2968 (2)	0.4826 (2)	0.1986 (5)	0.081 (2)
C(18)	0.2305 (2)	0.5003 (2)	0.2158 (5)	0.069 (1)
C(19)	0.2177 (2)	0.5479 (1)	0.2700 (4)	0.058 (1)
C(20)	0.2743 (2)	0.5789 (1)	0.3088 (4)	0.044 (1)
C(21)	0.3530 (2)	0.7270 (2)	−0.0139 (3)	0.048 (1)
C(22)	0.5854 (1)	0.8514 (1)	0.4931 (3)	0.0409 (9)
C(31)	0.6366 (1)	0.71354 (9)	0.6285 (3)	0.0240 (7)
C(32)	0.6994 (1)	0.70961 (9)	0.5294 (3)	0.0245 (7)
C(33)	0.7158 (1)	0.65388 (9)	0.4983 (3)	0.0240 (7)
C(34)	0.7281 (1)	0.62647 (9)	0.6375 (3)	0.0248 (7)
C(35)	0.6631 (1)	0.63258 (9)	0.7320 (3)	0.0261 (7)
C(36)	0.6750 (1)	0.6107 (1)	0.8783 (3)	0.0334 (8)
C(37)	0.7279 (1)	0.77133 (9)	0.3549 (3)	0.0302 (7)
C(38)	0.7024 (1)	0.79564 (9)	0.2229 (3)	0.0289 (7)
C(39)	0.6581 (1)	0.7701 (1)	0.1307 (3)	0.0358 (8)
C(40)	0.6389 (2)	0.7933 (1)	0.0055 (3)	0.046 (1)
C(41)	0.6621 (2)	0.8416 (1)	−0.0261 (3)	0.049 (1)
C(42)	0.7050 (2)	0.8672 (1)	0.0664 (4)	0.048 (1)
C(43)	0.7259 (1)	0.8443 (1)	0.1915 (3)	0.0393 (9)
C(44)	0.7731 (1)	0.63459 (9)	0.2794 (3)	0.0280 (7)
C(45)	0.8405 (1)	0.63789 (9)	0.2009 (3)	0.0272 (7)
C(46)	0.8448 (1)	0.6138 (1)	0.0703 (3)	0.0314 (8)
C(47)	0.9056 (2)	0.6164 (1)	−0.0087 (3)	0.0383 (9)
C(48)	0.9630 (2)	0.6425 (1)	0.0433 (3)	0.0419 (9)
C(49)	0.9591 (2)	0.6661 (1)	0.1727 (3)	0.0428 (9)
C(50)	0.8977 (1)	0.6642 (1)	0.2512 (3)	0.0328 (8)
C(51)	0.8105 (2)	0.5031 (1)	0.7368 (5)	0.057 (1)
C(52)	0.5582 (2)	0.6530 (1)	0.9885 (3)	0.049 (1)

Table 2. *Selected geometric parameters* (Å, °)

S(1)—O(4)	1.599 (2)	S(3)—O(34)	1.594 (2)
S(1)—O(8)	1.427 (2)	S(3)—O(38)	1.417 (2)
S(1)—O(9)	1.415 (2)	S(3)—O(39)	1.426 (2)
S(1)—C(21)	1.746 (3)	S(3)—C(51)	1.750 (4)
S(2)—C(6)	1.817 (3)	S(4)—C(36)	1.819 (3)
S(2)—C(22)	1.685 (3)	S(4)—C(52)	1.671 (4)
O(1)—C(1)	1.416 (3)	O(1)—C(31)	1.420 (3)
O(2)—C(2)	1.438 (3)	O(32)—C(32)	1.431 (3)
O(2)—C(7)	1.352 (3)	O(32)—C(37)	1.359 (3)
O(3)—C(3)	1.438 (3)	O(33)—C(33)	1.445 (3)
O(3)—C(14)	1.351 (3)	O(33)—C(44)	1.363 (3)
O(4)—C(4)	1.447 (3)	O(34)—C(34)	1.457 (3)
O(5)—C(1)	1.417 (3)	O(35)—C(31)	1.409 (3)
O(5)—C(5)	1.437 (3)	O(35)—C(35)	1.438 (3)
O(7)—C(7)	1.197 (3)	O(37)—C(37)	1.201 (3)
O(14)—C(14)	1.202 (3)	O(44)—C(44)	1.198 (3)
N(1)—C(22)	1.143 (4)	N(2)—C(52)	1.160 (4)
C(1)—C(2)	1.516 (3)	C(31)—C(32)	1.526 (3)
C(2)—C(3)	1.517 (4)	C(32)—C(33)	1.526 (3)
C(3)—C(4)	1.516 (3)	C(33)—C(34)	1.523 (3)
C(4)—C(5)	1.529 (3)	C(34)—C(35)	1.538 (3)
C(5)—C(6)	1.521 (3)	C(35)—C(36)	1.519 (3)
C(7)—C(8)	1.478 (3)	C(37)—C(38)	1.487 (3)
C(14)—C(15)	1.488 (4)	C(44)—C(45)	1.487 (3)
O(4)—S(1)—O(8)	108.1 (1)	O(34)—S(3)—O(38)	106.1 (1)
O(4)—S(1)—O(9)	109.7 (1)	O(34)—S(3)—O(39)	109.5 (1)
O(4)—S(1)—C(21)	96.8 (1)	O(34)—S(3)—C(51)	101.3 (2)
O(8)—S(1)—O(9)	117.2 (1)	O(38)—S(3)—O(39)	120.2 (1)
O(8)—S(1)—C(21)	110.2 (2)	O(38)—S(3)—C(51)	108.8 (2)
O(9)—S(1)—C(21)	112.8 (2)	O(39)—S(3)—C(51)	109.2 (2)
C(6)—S(2)—C(22)	102.5 (1)	C(36)—S(4)—C(52)	101.3 (1)
C(2)—O(2)—C(7)	117.8 (2)	C(32)—O(32)—C(37)	117.1 (2)
C(3)—O(3)—C(14)	116.5 (2)	C(33)—O(33)—C(44)	117.5 (2)
S(1)—O(4)—C(4)	116.7 (1)	S(3)—O(34)—C(34)	121.5 (1)
C(1)—O(5)—C(5)	114.7 (2)	C(31)—O(35)—C(35)	114.7 (2)
O(1)—C(1)—O(5)	111.4 (2)	O(1)—C(31)—O(35)	111.3 (2)
O(1)—C(1)—C(2)	108.4 (2)	O(1)—C(31)—C(32)	108.2 (2)
O(5)—C(1)—C(2)	110.1 (2)	O(35)—C(31)—C(32)	110.0 (2)
O(2)—C(2)—C(1)	109.7 (2)	O(32)—C(32)—C(31)	108.5 (2)
O(2)—C(2)—C(3)	109.0 (2)	O(32)—C(32)—C(33)	109.2 (2)
C(1)—C(2)—C(3)	110.3 (2)	C(31)—C(32)—C(33)	110.1 (2)
O(3)—C(3)—C(2)	109.1 (2)	O(33)—C(33)—C(32)	107.9 (2)
O(3)—C(3)—C(4)	109.7 (2)	O(33)—C(33)—C(34)	109.7 (2)
C(2)—C(3)—C(4)	107.1 (2)	C(32)—C(33)—C(34)	108.5 (2)
O(4)—C(4)—C(3)	108.3 (2)	O(34)—C(34)—C(33)	109.6 (2)
O(4)—C(4)—C(5)	108.4 (2)	O(34)—C(34)—C(35)	106.5 (2)
C(3)—C(4)—C(5)	108.6 (2)	C(33)—C(34)—C(35)	109.4 (2)
O(5)—C(5)—C(4)	108.2 (2)	O(35)—C(35)—C(34)	109.0 (2)
O(5)—C(5)—C(6)	106.6 (2)	O(35)—C(35)—C(36)	105.6 (2)
C(4)—C(5)—C(6)	112.9 (2)	C(34)—C(35)—C(36)	111.9 (2)
S(2)—C(6)—C(5)	115.2 (2)	S(4)—C(36)—C(35)	115.2 (2)
O(2)—C(7)—O(7)	123.3 (2)	O(32)—C(37)—O(37)	123.5 (2)
O(2)—C(7)—C(8)	111.8 (2)	O(32)—C(37)—C(38)	111.2 (2)
O(7)—C(7)—C(8)	124.9 (2)	O(37)—C(37)—C(38)	125.3 (2)
O(3)—C(14)—O(14)	123.5 (3)	O(33)—C(44)—O(44)	123.3 (2)
O(3)—C(14)—C(15)	111.6 (2)	O(33)—C(44)—C(45)	112.3 (2)
O(14)—C(14)—C(15)	124.9 (3)	O(44)—C(44)—C(45)	124.4 (2)
S(2)—C(22)—N(1)	175.0 (3)	S(4)—C(52)—N(2)	175.2 (3)
C(1)—O(1)—C(31)	113.6 (2)		

O(5)—C(1)—C(2)—C(3)	56.1 (3)
O(5)—C(5)—C(4)—C(3)	−60.7 (2)
C(1)—O(5)—C(5)—C(4)	59.9 (2)
C(1)—C(2)—C(3)—C(4)	−58.8 (2)
C(2)—C(1)—O(5)—C(5)	−57.6 (2)
C(2)—C(3)—C(4)—C(5)	61.1 (2)
O(1)—C(1)—O(5)—C(5)	62.8 (2)
O(1)—C(1)—C(2) O(2)	54.1 (2)
O(1)—C(1)—C(2)—C(3)	−66.0 (2)
O(2)—C(2)—C(3)—O(3)	62.1 (2)
O(2)—C(2)—C(3)—C(4)	−179.2 (2)
O(2)—C(7)—C(8)—C(9)	−9.6 (4)
O(3)—C(3)—C(4)—O(4)	−63.0 (2)
O(3)—C(3)—C(4)—C(5)	179.4 (2)
O(3)—C(14)—C(15)—C(16)	−171.4 (3)
O(4)—C(4)—C(5)—O(5)	−178.2 (2)
O(4)—C(4)—C(5)—C(6)	63.9 (3)
O(5)—C(1)—O(1)—C(31)	84.4 (2)
C(2)—C(1)—O(1)—C(31)	−154.3 (2)
C(1)—O(5)—C(5)—C(6)	−178.4 (2)
C(1)—C(2)—O(2)—C(7)	116.7 (2)
C(2)—O(2)—C(7)—C(8)	177.8 (2)
C(2)—C(3)—O(3)—C(14)	−113.1 (2)
C(3)—O(3)—C(14)—C(15)	−176.5 (2)
C(4)—O(4)—S(1)—C(21)	−176.3 (2)
S(1)—O(4)—C(4)—C(3)	120.9 (2)
S(1)—O(4)—C(4)—C(5)	−121.4 (2)
S(2)—C(6)—C(5)—O(5)	83.2 (2)
S(2)—C(6)—C(5)—C(4)	−158.1 (2)
O(35)—C(31)—C(32)—C(33)	56.5 (2)
O(35)—C(35)—C(34)—C(33)	−57.7 (2)
C(31)—O(35)—C(35)—C(34)	60.1 (2)
C(31)—C(32)—C(33)—C(34)	−56.6 (2)
C(32)—C(31)—O(35)—C(35)	−59.5 (2)
C(32)—C(33)—C(34)—C(35)	57.3 (2)
O(1)—C(31)—O(35)—C(35)	60.4 (2)
O(1)—C(31)—C(32)—O(32)	54.2 (2)
O(1)—C(31)—C(32)—C(33)	−65.2 (2)
O(32)—C(32)—C(33)—O(33)	65.6 (2)
O(32)—C(32)—C(33)—C(34)	−175.6 (2)
O(32)—C(37)—C(38)—C(39)	−26.1 (3)
O(33)—C(33)—C(34)—O(34)	−68.7 (2)
O(33)—C(33)—C(34)—C(35)	175.0 (2)
O(33)—C(44)—C(45)—C(46)	169.9 (2)
O(34)—C(34)—C(35)—O(35)	−176.0 (2)
O(34)—C(34)—C(35)—C(36)	67.6 (3)
O(35)—C(31)—O(1)—C(1)	81.3 (2)
C(32)—C(31)—O(1)—C(1)	−157.7 (2)
C(31)—O(35)—C(35)—C(36)	−179.6 (2)
C(31)—C(32)—O(32)—C(37)	126.7 (2)
C(32)—O(32)—C(37)—C(38)	−178.1 (2)
C(32)—C(33)—O(33)—C(44)	−114.3 (2)
C(33)—O(33)—C(44)—C(45)	176.2 (2)
C(34)—O(34)—S(3)—C(51)	119.2 (2)

S(3)—O(34)—C(34)—C(33)	96.2 (2)
S(3)—O(34)—C(34)—C(35)	−145.7 (2)
S(4)—C(36)—C(35)—O(35)	82.0 (2)
S(4)—C(36)—C(35)—C(34)	−159.6 (2)

The data collection was extended to include the measurement of the intensities of the Friedel opposites of all reflections in the unique octant with $2\theta < 50°$. Friedel pairs were not averaged during the data reduction so that the effects of anomalous dispersion could be used for the determination of the absolute configuration.

The displacement ellipsoids for one of the benzoyl groups indicate the presence of slight disorder or thermal motion, however, a disordered model could not be refined satisfactorily.

The absolute configuration was determined by using the *CRYSTALS* program system (Watkin, Carruthers & Betteridge, 1985) to refine the final atomic coordinates together with the enantiopole parameter (Flack, 1983). The refined value of the enantiopole parameter was 0.04 (5), thus confirming that the atomic coordinates represented the correct enantiomorph.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991). Cell refinement: *MSC/AFC Diffractometer Control Software.* Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *TEXSAN LS.* Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN FINISH.*

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1147). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Dimeric Uracil Derivative

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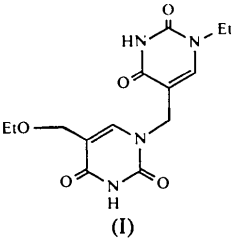
Abstract

The structure of a dimeric uracil derivative, 5-ethoxymethyl-1′-ethyl-1,5′-methylenedi-2,4(1*H*,3*H*)-pyrimidinedione, $C_{14}H_{18}N_4O_5$, was determined. The two uracil groups present in this molecule are almost identical. The torsion angles involving the bonds connecting the two uracil moieties, C6—N1—C13—C14 and C19—C14—C13—N1, are 77.5 (3) and 75.9 (3)°, respectively.

Comment

Since the discovery of the new anti-HIV-1 (human immunodeficiency virus type 1) lead compound HEPT, 1-[(2-hydoxyethoxy)methyl]-6-(phenylthio)thymine (Miyasaka *et al.*, 1989), extensive synthetic studies of HEPT analogues have been carried out (for a recent report see Tanaka *et al.*, 1992). The title compound, (I), was obtained as a by-product during the preparation of 5-ethoxymethyl-1-ethyluracil, which was then converted to the required regioisomeric analogue of HEPT by a procedure based on lithiation chemistry (Tanaka *et al.*, 1994).



(I)