C(5)—C(6)	1.527 (5)	C(15)—C(16)	1.506 (5)
C(5)—C(13)	1.550 (5)	C(16)—N(17)	1.454 (5)
C(5)—C(21)	1.526 (5)	N(17)—C(22)	1.453 (5)
C(5)—O(2)	1.483 (4)	C(18)—C(19)	1.314 (5)
C(6)—C(7)	1.554 (5)	C(18)—O(3)	1.369 (4)
C(6)—C(18)	1.515 (5)	C(20)—O(1)	1.392 (6)
C(7)—C(8)	1.542 (5)	C(23)—O(3)	1.419 (5)
C(8)—C(14)	1.593 (5)	C(24)—O(4)	1.188 (5)
C(8)—C(24)	1.509 (5)	C(24)—O(5)	1.330 (5)
C(9)—C(10)	1.566 (5)	C(25)—C(26)	1.429 (8)
C(9)—C(14)	1.551 (5)	C(25)—O(5)	1.450 (6)

C(2)—C(1)—C(11)	120.5 (4)	C(5)—C(13)—C(12)	103.2 (3)
C(1)—C(2)—C(3)	122.4 (4)	C(5)—C(13)—C(14)	111.6 (3)
C(2)—C(3)—C(4)	116.4 (4)	C(5)—C(13)—C(15)	112.4 (3)
C(2)—C(3)—O(1)	127.3 (4)	C(12)—C(13)—C(14)	106.1 (3)
C(4)—C(3)—O(1)	116.3 (3)	C(12)—C(13)—C(15)	114.7 (3)
C(3)—C(4)—C(12)	120.7 (3)	C(14)—C(13)—C(15)	108.7 (3)
C(3)—C(4)—O(2)	125.3 (3)	C(8)—C(14)—C(9)	109.8 (3)
C(12)—C(4)—O(2)	113.7 (3)	C(8)—C(14)—C(13)	109.2 (3)
C(6)—C(5)—C(13)	107.7 (3)	C(8)—C(14)—C(19)	105.6 (3)
C(6)—C(5)—C(21)	110.4 (3)	C(9)—C(14)—C(13)	105.4 (3)
C(6)—C(5)—O(2)	111.5 (3)	C(9)—C(14)—C(19)	119.6 (3)
C(13)—C(5)—C(21)	117.4 (3)	C(13)—C(14)—C(19)	107.0 (3)
C(13)—C(5)—O(2)	105.3 (3)	C(13)—C(15)—C(16)	114.1 (3)
C(21)—C(5)—O(2)	104.4 (3)	C(15)—C(16)—N(17)	109.5 (3)
C(5)—C(6)—C(7)	110.4 (3)	C(9)—N(17)—C(16)	111.0 (3)
C(5)—C(6)—C(18)	105.9 (3)	C(9)—N(17)—C(22)	112.4 (3)
C(7)—C(6)—C(18)	106.8 (3)	C(16)—N(17)—C(22)	112.3 (3)
C(6)—C(7)—C(8)	109.8 (3)	C(6)—C(18)—C(19)	117.7 (3)
C(7)—C(8)—C(14)	108.9 (3)	C(6)—C(18)—O(3)	112.2 (3)
C(7)—C(8)—C(24)	111.0 (3)	C(19)—C(18)—O(3)	130.1 (3)
C(10)—C(9)—C(14)	111.4 (3)	C(14)—C(19)—C(18)	112.4 (3)
C(10)—C(9)—N(17)	113.8 (3)	C(8)—C(24)—O(4)	126.5 (4)
C(14)—C(9)—N(17)	107.9 (3)	C(8)—C(24)—O(5)	110.3 (3)
C(9)—C(10)—C(11)	117.3 (3)	O(4)—C(24)—O(5)	123.1 (5)
C(1)—C(11)—C(10)	125.8 (3)	C(26)—C(25)—O(5)	110.7 (5)
C(1)—C(11)—C(12)	115.6 (3)	C(3)—O(1)—C(20)	118.5 (3)
C(10)—C(11)—C(12)	117.4 (3)	C(4)—O(2)—C(5)	107.7 (3)
C(4)—C(12)—C(11)	123.7 (3)	C(18)—O(3)—C(23)	115.1 (3)
C(4)—C(12)—C(13)	109.4 (3)	C(24)—O(5)—C(25)	117.1 (4)
C(11)—C(12)—C(13)	125.0 (3)		

Data were corrected for Lorentz and polarization effects. The structure was solved using direct methods (*Xtal*3.2; Hall, Flack & Stewart, 1992) with full-matrix least-squares refinement of anisotropic heavy atoms using *CRYLSQ* in *Xtal*3.2. H atoms were included in idealized positions (sp^2, C—H = 0.95 Å; sp^3, C—H = 1.00 Å] with isotropic displacement parameters [U(H) = $1.1U_{\rm eq}$(C)] and were allowed to ride on the parent C atoms.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71710 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1065]

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2,2′,3,3′-Tetra-*O*-acetyl-6,6′-dichloro-4,4′,6,6′-tetradeoxy-α,α-trehalose†

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Abstract

The crystal structure of the title compound (2,3-di-*O*-acetyl-6-chloro-4,6-dideoxy-α-D-glucopyranosyl 2,3-di-*O*-acetyl-6-chloro-4,6-dideoxy-α-D-glucopyranoside, $C_{20}H_{28}Cl_2O_{11}$) has been determined by X-ray diffraction. The two symmetry-independent molecules have very similar molecular dimensions, except for minor variations in the orientations of the ring substituents. The hexopyranosyl residues have distorted chair $^4C_1$ conformations. As observed for α,α-trehalose [Taga, Senma & Osaki (1972). *Acta Cryst.* B**28**, 3258-3263; Brown, Rohrer, Berking, Beevers, Gould & Simpson (1972). *Acta Cryst.* B**28**, 3145-3158; Jeffrey & Nanni (1985). *Carbohydr. Res.* **137**, 21-30] and its derivatives [Lee & Koh (1994). *Carbohydr. Res.* In the press], the molecules have approximate C_2 symmetry, differing only in the orientation of the chloromethyl substituents, and in the torsion angles about the α 1 → 1 linkage.

Comment

The ubiquitous character of α,α-trehalose, also known as mycose or mushroom sugar, as a probable energy reserve in many organisms has stimulated much research on the sugar (Birch 1963; Lee, 1980). It has served as an ideal molecule for taste-structure studies (Lee, 1973; Birch & Lee, 1974) because the molecule contains two chemically equivalent glucopyranose residues in the most stable $^4C_1$

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† Crystal Structures of Trehalose Derivatives, Part 3. For Part 2, see Lee & Koh (1994).

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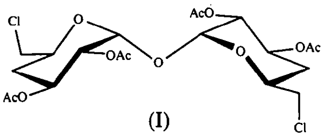
conformation, linked through their anomeric centre, and because the sugar and its derivatives are all extremely stable (Lee, 1980). The synthesis of the title compound, (I), has been reported by Birch, Lee & Richardson (1974) and the taste of its 2,2′,3,3′-tetrahydroxy analogue has been studied (Lee, 1973). We now report the X-ray crystal structure determination of the compound.



(I)

The atomic coordinates of the non-H atoms for the two symmetry-independent molecules are given in Table 1. A comparison of the atomic coordinates of the two molecules, by using the *MISSYM* routine (Le Page, 1987, 1988) of the program *PLATON* (Spek, 1990), confirmed that the molecules were unrelated by any crystallographic symmetry from a space group of higher symmetry. The two symmetry-independent molecules with their atomic numbering and the crystal packing are shown in Figs. 1 and 2, respectively. These figures also depict the correct absolute configuration of the molecule as established by the synthesis of the compound.

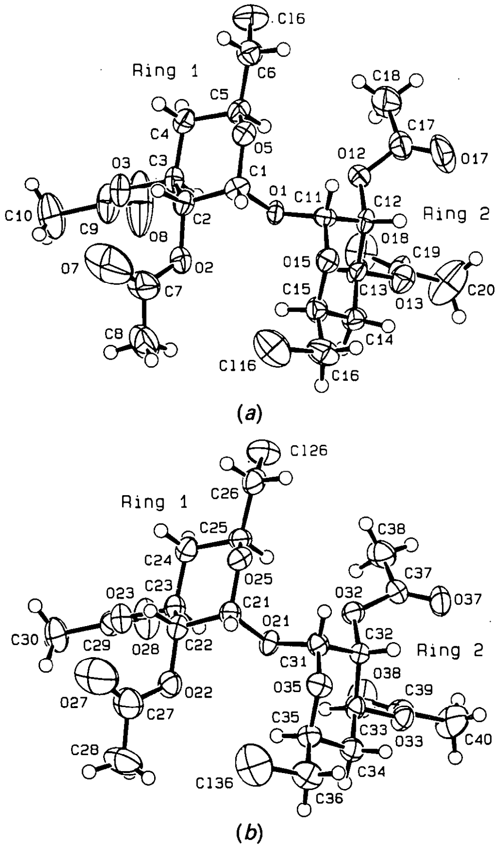


Fig. 1. Views of (*a*) molecule *A* and (*b*) molecule *B* showing the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by spheres of arbitrary radii.

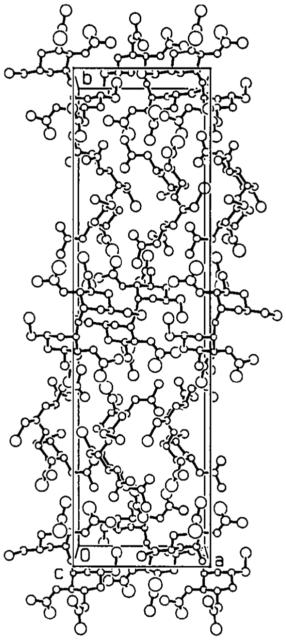


Fig. 2. The packing of the molecules in the unit cell viewed down *c*.

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

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

	x	y	z	$U_{\rm eq}$
Cl(6)	1.0003 (2)	0.99464 (5)	0.1261 (2)	0.0637 (7)
Cl(16)	0.1939 (2)	1.00082 (6)	−0.1884 (2)	0.0840 (9)
O(1)	0.5400 (3)	1.00889 (8)	0.0297 (3)	0.031 (1)
O(2)	0.4273 (4)	0.94962 (9)	0.0580 (4)	0.043 (2)
O(3)	0.6043 (4)	0.91487 (9)	0.1905 (4)	0.050 (2)
O(5)	0.7041 (4)	0.98483 (9)	−0.0643 (4)	0.038 (1)
O(7)	0.3985 (6)	0.9007 (2)	−0.0288 (7)	0.133 (4)
O(8)	0.6046 (8)	0.9340 (1)	0.3743 (5)	0.135 (4)
O(12)	0.6451 (4)	1.06540 (9)	0.1136 (4)	0.039 (1)
O(13)	0.4270 (4)	1.0973 (1)	0.2076 (4)	0.050 (2)
O(15)	0.4146 (4)	1.03853 (9)	−0.0966 (4)	0.037 (1)
O(17)	0.6959 (5)	1.1168 (1)	0.0598 (6)	0.100 (3)
O(18)	0.5107 (5)	1.0765 (1)	0.3707 (5)	0.079 (2)
C(1)	0.5810 (5)	0.9818 (1)	−0.0402 (6)	0.037 (2)
C(2)	0.5534 (6)	0.9499 (1)	0.0265 (6)	0.035 (2)
C(3)	0.6246 (6)	0.9477 (1)	0.1408 (6)	0.038 (2)
C(4)	0.7567 (6)	0.9525 (1)	0.1122 (6)	0.043 (2)
C(5)	0.7723 (5)	0.9852 (1)	0.0444 (6)	0.037 (2)
C(6)	0.9006 (6)	0.9899 (2)	0.0013 (6)	0.047 (2)
C(7)	0.3606 (7)	0.9250 (2)	0.0207 (8)	0.067 (3)
C(8)	0.2370 (8)	0.9274 (2)	0.0634 (9)	0.082 (4)
C(9)	0.5920 (8)	0.9115 (2)	0.3077 (6)	0.061 (3)
C(10)	0.5633 (9)	0.8766 (2)	0.3440 (8)	0.090 (4)
C(11)	0.5239 (5)	1.0384 (1)	−0.0364 (6)	0.031 (2)
C(12)	0.5314 (5)	1.0677 (1)	0.0515 (6)	0.033 (2)
C(13)	0.4311 (6)	1.0660 (1)	0.1428 (6)	0.037 (2)
C(14)	0.3108 (6)	1.0625 (1)	0.0781 (6)	0.041 (2)
C(15)	0.3133 (6)	1.0350 (1)	−0.0162 (6)	0.039 (2)
C(16)	0.2039 (6)	1.0368 (2)	−0.0937 (7)	0.058 (3)
C(17)	0.7189 (7)	1.0916 (2)	0.1109 (7)	0.055 (3)
C(18)	0.8291 (7)	1.0845 (2)	0.1810 (7)	0.066 (3)
C(19)	0.4658 (7)	1.0990 (2)	0.3123 (7)	0.046 (2)
C(20)	0.4498 (9)	1.1325 (2)	0.3700 (7)	0.094 (4)
Cl(26)	0.6527 (2)	0.75901 (5)	0.2682 (2)	0.0583 (7)
Cl(36)	0.9549 (2)	0.75741 (5)	1.0766 (2)	0.0888 (9)
O(21)	0.7475 (3)	0.74640 (8)	0.7338 (4)	0.032 (1)
O(22)	0.6954 (4)	0.80489 (9)	0.8515 (4)	0.042 (2)
O(23)	0.5456 (4)	0.83340 (9)	0.6764 (4)	0.041 (1)

O(25)	0.8302 (3)	0.7751 (1)	0.5698 (4)	0.037 (1)
O(27)	0.7685 (7)	0.8556 (2)	0.8770 (6)	0.126 (3)
O(28)	0.3735 (4)	0.8094 (1)	0.6239 (5)	0.066 (2)
O(32)	0.6793 (4)	0.69146 (9)	0.6165 (4)	0.035 (1)
O(33)	0.6132 (4)	0.64965 (9)	0.8272 (4)	0.041 (2)
O(35)	0.8860 (4)	0.7179 (1)	0.8498 (4)	0.039 (1)
O(37)	0.7057 (5)	0.6380 (1)	0.5655 (4)	0.063 (2)
O(38)	0.4407 (4)	0.6687 (1)	0.7504 (5)	0.066 (2)
C(21)	0.8056 (5)	0.7763 (1)	0.6950 (6)	0.035 (2)
C(22)	0.7241 (6)	0.8052 (1)	0.7242 (6)	0.036 (2)
C(23)	0.6114 (6)	0.8031 (1)	0.6513 (6)	0.036 (2)
C(24)	0.6427 (6)	0.8009 (1)	0.5183 (6)	0.041 (2)
C(25)	0.7266 (5)	0.7713 (1)	0.4972 (5)	0.036 (2)
C(26)	0.7718 (6)	0.7686 (2)	0.3698 (6)	0.045 (2)
C(27)	0.7223 (8)	0.8314 (2)	0.9171 (7)	0.064 (3)
C(28)	0.6830 (8)	0.8288 (2)	1.0440 (7)	0.079 (3)
C(29)	0.4267 (6)	0.8334 (2)	0.6596 (6)	0.042 (2)
C(30)	0.3729 (6)	0.8657 (2)	0.6928 (7)	0.054 (3)
C(31)	0.8230 (5)	0.7185 (1)	0.7400 (6)	0.033 (2)
C(32)	0.7470 (5)	0.6877 (1)	0.7265 (5)	0.032 (2)
C(33)	0.6645 (5)	0.6830 (1)	0.8309 (6)	0.034 (2)
C(34)	0.7336 (6)	0.6853 (1)	0.9492 (6)	0.041 (2)
C(35)	0.8070 (6)	0.7169 (1)	0.9521 (6)	0.038 (2)
C(36)	0.8827 (6)	0.7190 (2)	1.0632 (6)	0.052 (3)
C(37)	0.6613 (6)	0.6642 (1)	0.5475 (6)	0.041 (2)
C(38)	0.5814 (6)	0.6730 (2)	0.4448 (6)	0.054 (3)
C(39)	0.5006 (6)	0.6461 (2)	0.7828 (6)	0.044 (2)
C(40)	0.4665 (7)	0.6107 (2)	0.7792 (8)	0.071 (3)

Table 2. *Geometric parameters* (Å, °)

Molecule *A*		Molecule *B*	
Cl(6)—C(6)	1.789 (7)	Cl(26)—C(26)	1.789 (7)
Cl(16)—C(16)	1.792 (7)	Cl(36)—C(36)	1.747 (7)
O(1)—C(1)	1.414 (6)	O(21)—C(21)	1.433 (6)
O(1)—C(11)	1.406 (6)	O(21)—C(31)	1.406 (6)
O(2)—C(2)	1.455 (7)	O(22)—C(22)	1.448 (7)
O(2)—C(7)	1.305 (8)	O(22)—C(27)	1.326 (8)
O(3)—C(3)	1.448 (6)	O(23)—C(23)	1.449 (6)
O(3)—C(9)	1.315 (8)	O(23)—C(29)	1.345 (7)
O(5)—C(1)	1.411 (7)	O(25)—C(21)	1.417 (7)
O(5)—C(5)	1.428 (7)	O(25)—C(25)	1.421 (7)
O(7)—C(7)	1.198 (9)	O(27)—C(27)	1.188 (8)
O(8)—C(9)	1.174 (8)	O(28)—C(29)	1.198 (7)
O(12)—C(12)	1.451 (7)	O(32)—C(32)	1.445 (7)
O(12)—C(17)	1.339 (7)	O(32)—C(37)	1.350 (6)
O(13)—C(13)	1.447 (7)	O(33)—C(33)	1.460 (6)
O(13)—C(19)	1.243 (8)	O(33)—C(39)	1.361 (7)
O(15)—C(11)	1.395 (6)	O(35)—C(31)	1.409 (7)
O(15)—C(15)	1.451 (7)	O(35)—C(35)	1.439 (7)
O(17)—C(17)	1.188 (7)	O(37)—C(37)	1.181 (7)
O(18)—C(19)	1.221 (7)	O(38)—C(39)	1.183 (7)
C(1)—C(2)	1.511 (8)	C(21)—C(22)	1.513 (8)
C(2)—C(3)	1.501 (8)	C(22)—C(23)	1.502 (8)
C(3)—C(4)	1.525 (8)	C(23)—C(24)	1.521 (9)
C(4)—C(5)	1.523 (8)	C(24)—C(25)	1.532 (8)
C(5)—C(6)	1.526 (8)	C(25)—C(26)	1.506 (9)
C(7)—C(8)	1.47 (1)	C(27)—C(28)	1.48 (1)
C(9)—C(10)	1.495 (9)	C(29)—C(30)	1.478 (8)
C(11)—C(12)	1.532 (8)	C(31)—C(32)	1.511 (8)
C(12)—C(13)	1.514 (8)	C(32)—C(33)	1.494 (8)
C(13)—C(14)	1.533 (9)	C(33)—C(34)	1.526 (9)
C(14)—C(15)	1.521 (8)	C(34)—C(35)	1.515 (8)
C(15)—C(16)	1.499 (9)	C(35)—C(36)	1.499 (9)
C(17)—C(18)	1.487 (9)	C(37)—C(38)	1.492 (9)
C(19)—C(20)	1.499 (9)	C(39)—C(40)	1.474 (8)
C(1)—O(1)—C(11)	113.9 (4)	C(21)—O(21)—C(31)	114.2 (4)
C(2)—O(2)—C(7)	119.0 (5)	C(22)—O(22)—C(27)	118.5 (5)
C(3)—O(3)—C(9)	119.1 (5)	C(23)—O(23)—C(29)	118.4 (5)
C(1)—O(5)—C(5)	111.4 (5)	C(21)—O(25)—C(25)	113.7 (5)
C(12)—O(12)—C(17)	118.7 (5)	C(32)—O(32)—C(37)	118.1 (4)
C(13)—O(13)—C(19)	120.1 (5)	C(33)—O(33)—C(39)	118.0 (5)
C(11)—O(15)—C(15)	113.0 (4)	C(31)—O(35)—C(35)	112.0 (4)
O(1)—C(1)—O(5)	110.8 (5)	O(21)—C(21)—O(25)	110.8 (5)
O(1)—C(1)—C(2)	108.5 (5)	O(21)—C(21)—C(22)	107.8 (5)
O(5)—C(1)—C(2)	111.5 (5)	O(25)—C(21)—C(22)	110.7 (5)
O(2)—C(2)—C(1)	108.8 (5)	O(22)—C(22)—C(21)	109.6 (5)
O(2)—C(2)—C(3)	108.2 (5)	O(22)—C(22)—C(23)	109.8 (5)
C(1)—C(2)—C(3)	110.8 (5)	C(21)—C(22)—C(23)	110.4 (5)
O(3)—C(3)—C(2)	107.0 (5)	O(23)—C(23)—C(22)	106.1 (5)
O(3)—C(3)—C(4)	110.3 (5)	O(23)—C(23)—C(24)	110.7 (5)
C(2)—C(3)—C(4)	109.4 (5)	C(22)—C(23)—C(24)	109.4 (5)
C(3)—C(4)—C(5)	108.9 (5)	C(23)—C(24)—C(25)	109.6 (5)
O(5)—C(5)—C(4)	110.3 (5)	O(25)—C(25)—C(24)	109.3 (5)
O(5)—C(5)—C(6)	103.9 (5)	O(25)—C(25)—C(26)	105.4 (5)
C(4)—C(5)—C(6)	111.7 (5)	C(24)—C(25)—C(26)	114.0 (5)
Cl(6)—C(6)—C(5)	111.0 (5)	Cl(26)—C(26)—C(25)	110.9 (5)
O(2)—C(7)—O(7)	124.0 (8)	O(22)—C(27)—O(27)	123.4 (8)
O(2)—C(7)—C(8)	112.9 (7)	O(22)—C(27)—C(28)	113.4 (7)
O(7)—C(7)—C(8)	122.3 (8)	O(27)—C(27)—C(28)	123.1 (8)
O(3)—C(9)—O(8)	122.1 (7)	O(23)—C(29)—O(28)	122.6 (6)
O(3)—C(9)—C(10)	112.6 (7)	O(23)—C(29)—C(30)	111.6 (5)
O(8)—C(9)—C(10)	125.3 (7)	O(28)—C(29)—C(30)	125.8 (6)
O(1)—C(11)—O(15)	111.5 (4)	O(21)—C(31)—O(35)	110.9 (5)
O(1)—C(11)—C(12)	108.0 (5)	O(21)—C(31)—C(32)	108.1 (5)
O(15)—C(11)—C(12)	110.5 (5)	O(35)—C(31)—C(32)	110.8 (5)
O(12)—C(12)—C(11)	107.5 (4)	O(32)—C(32)—C(31)	107.0 (4)
O(12)—C(12)—C(13)	109.3 (5)	O(32)—C(32)—C(33)	110.1 (5)
C(11)—C(12)—C(13)	110.5 (5)	C(31)—C(32)—C(33)	111.9 (5)
O(13)—C(13)—C(12)	108.5 (5)	O(33)—C(33)—C(32)	109.6 (5)
O(13)—C(13)—C(14)	106.6 (5)	O(33)—C(33)—C(34)	106.1 (5)
C(12)—C(13)—C(14)	110.0 (5)	C(32)—C(33)—C(34)	110.3 (5)
C(13)—C(14)—C(15)	112.0 (5)	C(33)—C(34)—C(35)	110.0 (5)
O(15)—C(15)—C(14)	111.5 (5)	O(35)—C(35)—C(34)	109.9 (5)
O(15)—C(15)—C(16)	106.3 (5)	O(35)—C(35)—C(36)	107.4 (5)
C(14)—C(15)—C(16)	110.1 (6)	C(34)—C(35)—C(36)	111.9 (5)
Cl(16)—C(16)—C(15)	110.4 (5)	Cl(36)—C(36)—C(35)	112.5 (5)
O(12)—C(17)—O(17)	123.1 (7)	O(32)—C(37)—O(37)	124.3 (6)
O(12)—C(17)—C(18)	110.5 (6)	O(32)—C(37)—C(38)	109.3 (5)
O(17)—C(17)—C(18)	126.3 (7)	O(37)—C(37)—C(38)	126.4 (6)
O(13)—C(19)—O(18)	126.8 (7)	O(33)—C(39)—O(38)	123.8 (6)
O(13)—C(19)—C(20)	114.0 (6)	O(33)—C(39)—C(40)	110.5 (6)
O(18)—C(19)—C(20)	119.2 (7)	O(38)—C(39)—C(40)	125.7 (7)

The two symmetry-independent molecules have very similar molecular dimensions except for minor differences in the conformations of some of the acetyl groups. The two D-hexopyranosyl residues within each molecule have approximate C_2 symmetry. As observed with α,α-trehalose dihydrate (Taga, Senma & Osaki, 1972; Brown, Rohrer, Berking, Beevers, Gould & Simpson, 1972), the two glycosidic linkage torsion angles in each molecule show small differences [C(11)—O(1)—C(1)—O(5) = 77.2 (6) and C(1)—O(1)—C(11)—O(15) = 81.7 (6)° for molecule *A*; the corresponding torsion angles for molecule *B* are 73.5 (6) and 83.3 (6)°, respectively]. Therefore, the hexopyranosyl residues are not completely symmetrically oriented about the central glycosidic O atom. The largest conformational difference between the two hexopyranosyl residues within each molecule is shown by the chloromethyl substituent at C(5) of each ring. The torsion angles about C(5)—C(6) in one of the rings of each molecule [O(15)—C(15)—C(16)—Cl(16) = 68.4 (6) and C(14)—C(15)—C(16)—Cl(16) = −170.7 (4)° for molecule *A*; O(35)—C(35)—C(36)—Cl(36) = 66.1 (6) and C(34)—C(35)—C(36)—Cl(36) = −173.2 (4)° for molecule *B*] describe the *gauche-trans* arrangement. The corresponding torsion angles in the other ring [175.0 (4) and −66.2 (6)° for molecule *A*; 173.6 (4) and −66.5 (6)° for molecule *B*] describe the *trans-gauche* conformation. This conformation is generally forbidden for glucopyranosyl moieties (Fries,

Table 3. *Selected torsion angles* (°)

Within the pyranose rings			
Molecule *A*		Molecule *B*	
C(1)—C(2)—C(3)—C(4)	−53.5 (6)	C(21)—C(22)—C(23)—C(24)	−54.8 (7)
C(1)—O(5)—C(5)—C(4)	62.2 (6)	C(21)—O(25)—C(25)—C(24)	60.3 (6)
C(2)—C(1)—O(5)—C(5)	−60.3 (6)	C(22)—C(21)—O(25)—C(25)	−59.9 (6)
C(2)—C(3)—C(4)—C(5)	54.9 (7)	C(22)—C(23)—C(24)—C(25)	55.6 (6)
C(11)—C(12)—C(13)—C(14)	−52.3 (6)	C(31)—C(32)—C(33)—C(34)	−51.0 (6)
C(11)—O(15)—C(15)—C(14)	58.0 (6)	C(31)—O(35)—C(35)—C(34)	62.3 (6)
C(12)—C(11)—O(15)—C(15)	−61.6 (6)	C(32)—C(31)—O(35)—C(35)	−60.7 (6)
C(12)—C(13)—C(14)—C(15)	49.2 (6)	C(32)—C(33)—C(34)—C(35)	52.0 (6)
O(5)—C(1)—C(2)—C(3)	56.1 (7)	O(25)—C(21)—C(22)—C(23)	56.0 (7)
O(5)—C(5)—C(4)—C(3)	−59.1 (7)	O(25)—C(25)—C(24)—C(23)	−57.3 (6)
O(15)—C(11)—C(12)—C(13)	58.9 (6)	O(35)—C(31)—C(32)—C(33)	55.1 (6)
O(15)—C(15)—C(14)—C(13)	−50.9 (7)	O(35)—C(35)—C(34)—C(33)	−57.0 (6)
Outside the pyranose rings			
Molecule *A*		Molecule *B*	
C(1)—C(2)—O(2)—C(7)	122.6 (7)	C(21)—C(22)—O(22)—C(27)	122.0 (6)
C(2)—C(3)—O(3)—C(9)	−140.4 (7)	C(22)—C(23)—O(23)—C(29)	−156.6 (6)
C(2)—O(2)—C(7)—O(7)	7 (1)	C(22)—O(22)—C(27)—O(27)	0 (1)
C(3)—C(4)—C(5)—C(6)	−174.1 (6)	C(23)—C(24)—C(25)—C(26)	−175.0 (6)
C(3)—O(3)—C(9)—C(10)	176.0 (6)	C(23)—O(23)—C(29)—C(30)	178.2 (5)
C(4)—C(5)—C(6)—Cl(6)	−66.2 (6)	C(24)—C(25)—C(26)—Cl(26)	−66.5 (6)
C(11)—O(1)—C(1)—C(2)	−160.2 (5)	C(31)—O(21)—C(21)—C(22)	−165.2 (5)
C(11)—O(1)—C(1)—O(5)	77.2 (6)	C(31)—O(21)—C(21)—O(25)	73.5 (6)
C(11)—C(12)—O(12)—C(17)	125.0 (6)	C(31)—C(32)—O(32)—C(37)	142.2 (5)
C(12)—C(11)—O(1)—C(1)	−156.7 (5)	C(32)—C(31)—O(21)—C(21)	−155.1 (5)
C(12)—C(13)—O(13)—C(19)	−104.5 (7)	C(32)—C(33)—O(33)—C(39)	−100.2 (6)
C(12)—O(12)—C(17)—O(17)	0 (1)	C(32)—O(32)—C(37)—O(37)	−7 (1)
C(13)—C(14)—C(15)—C(16)	−168.7 (5)	C(33)—C(34)—C(35)—C(36)	−176.2 (5)
C(13)—O(13)—C(19)—O(18)	1 (1)	C(33)—O(33)—C(39)—O(38)	−2 (1)
C(14)—C(15)—C(16)—Cl(16)	−170.7 (4)	C(34)—C(35)—C(36)—Cl(36)	−173.2 (4)
O(1)—C(1)—C(2)—C(3)	−66.1 (6)	O(21)—C(21)—C(22)—C(23)	−65.3 (6)
O(1)—C(1)—O(5)—C(5)	60.6 (6)	O(21)—C(21)—O(25)—C(25)	59.7 (6)
C(1)—O(1)—C(11)—O(15)	81.7 (6)	C(21)—O(21)—C(31)—O(35)	83.3 (6)
O(1)—C(11)—C(12)—C(13)	−63.3 (6)	O(21)—C(31)—C(32)—C(33)	−66.5 (6)
O(1)—C(11)—O(15)—C(15)	58.6 (6)	O(21)—C(31)—O(35)—C(35)	59.3 (6)
O(5)—C(5)—C(6)—Cl(6)	175.0 (4)	O(25)—C(25)—C(26)—Cl(26)	173.6 (4)
O(15)—C(15)—C(16)—Cl(16)	68.4 (6)	O(35)—C(35)—C(36)—Cl(36)	66.1 (6)
O(2)—C(2)—C(3)—C(4)	−172.7 (4)	O(22)—C(22)—C(23)—C(24)	−175.7 (5)
O(12)—C(12)—C(13)—C(14)	−170.4 (4)	O(32)—C(32)—C(33)—C(34)	−169.9 (4)

Rao & Sundaralingam, 1971) because of interaction between the hydroxymethyl OH group at C(5) and the OH substituent at C(4) of the ring. The *trans-gauche* conformation is most probably adopted because of the absence of a bulky substituent at C(4). The torsion angles about the anomeric linkage (Table 3) describe the *A*1 conformer (de Hoog, Buys, Altona & Havinga, 1969; Eliel, 1969) for each of the two hexopyranosyl-O(1) residues.

The bond lengths and bond angles are within expected ranges. The C—O bond lengths associated with the anomeric C atoms and the ring O atoms show systematic trends similar to those observed in other α-pyranose sugars (Berman, Chu & Jeffrey, 1967).

All the pyranose rings have $^4C_1$ conformations with the torsion angles showing the normal variations commonly observed in other pyranose systems (Brown & Levy, 1963; Chu & Jeffrey, 1968; Jeffrey & Tagaki, 1977). The puckering parameters (Cremer & Pople, 1975) of the pyranose rings (Table 4) describe slightly distorted chair conformations ($Q \simeq q_3 \gg q_2$ and θ is very small). The φ_2 values indicate that the rings are distorted towards the half-chair conformation, with a flattening at the O atom apex allowing the ring C—O—C angle to increase to approximately 112° while the other ring angles remain close to tetrahedral values.

Table 4. *Puckering parameters* (Å, °) (*Cremer & Pople,* 1975) *for the pyranose rings of the two symmetry-independent molecules*

	Molecule *A*		Molecule *B*	
	Ring 1	Ring 2	Ring 1	Ring 2
Q	0.577	0.550	0.573	0.561
q_2	0.031	0.050	0.017	0.044
q_3	0.576	0.548	0.573	0.559
θ	3.11	5.19	1.67	4.50
φ_2	294.1	60.1	255.2	329.2

Experimental

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

Crystal data

$C_{20}H_{28}Cl_2O_{11}$
$M_r = 515.34$
Orthorhombic
$P2_12_12_1$
$a = 11.201$ (3) Å
$b = 40.167$ (2) Å
$c = 11.099$ (3) Å
$V = 4994$ (2) Å^3
$Z = 8$
$D_x = 1.371$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 22 reflections
$\theta = 15.5$-20°
$\mu = 0.310$ mm^{-1}
$T = 173$ (1) K
Prism
0.40 × 0.38 × 0.38 mm
Colourless

Data collection

Rigaku AFC-5*R* diffractometer
ω scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.77$, $T_{\max} = 1.05$
7235 measured reflections
7084 independent reflections
4201 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int} = 0.017$
$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 14$
$k = -1 \rightarrow 52$
$l = -1 \rightarrow 14$
3 standard reflections monitored every 150 reflections
intensity variation: insignificant

Refinement

Refinement on *F*
$R = 0.0516$
$wR = 0.0479$
$S = 2.125$
4201 reflections
595 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + (0.005F_o)^2]$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.40$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}

Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
Absolute configuration: assigned to agree with the known chirality at C(5) as established by the synthesis of the compound

During the refinement the behaviour of some of the acetyl groups indicated that they are slightly disordered; however, a disordered model could not be refined satisfactorily. An ordered model was employed for the final refinement and the enlarged thermal ellipsoids for some of the atoms of these groups reflect the 'smeared out' electron density resulting from the disorder. All of the H atoms were placed in geometrically calculated positions and were assigned fixed isotropic displacement parameters with values of $1.2 \times U_{\rm eq}$ of the parent C atom. 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: direct methods in *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 British Library Document Supply Centre as Supplementary Publication No. SUP 71833 (52 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1081]

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1-Methyl-2-methylthio-5-nitroimidazole, $C_5H_7N_3O_2S$

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Abstract

In the title molecule, the imidazole ring is almost coplanar with the neighbouring attached atoms.

Comment

Investigations of nitroimidazole derivatives have been performed in order to analyse the structure–activity relationships of these compounds, which are applied worldwide as drugs, either as radiosensitizers or antiprotozoic and antibiotic agents.

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