Tabelle 1. *Atomkoordinaten und isotrope äquivalente Verschiebungsparameter* (Å²) *nach Hamilton* (1959)

$$U_{\text{äq}} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\text{äq}}$
P	0,2573 (2)	0,6926 (2)	0,4297 (2)	0,044 (1)
S(1)	0,2568 (2)	0,5174 (2)	0,5715 (2)	0,058 (1)
S(2)	0,3037 (2)	1,0303 (2)	0,3282 (2)	0,067 (1)
O(1)	0,2124 (4)	0,7021 (4)	0,2930 (3)	0,051 (3)
O(2)	0,2019 (5)	1,0810 (5)	0,2266 (4)	0,089 (4)
O(3)	0,3033 (6)	0,9684 (5)	0,5994 (4)	0,074 (4)
O(4)	0,1554 (5)	0,7848 (6)	0,6742 (4)	0,080 (4)
O(5)	0,5312 (5)	1,0849 (6)	0,1313 (4)	0,079 (4)
O(6)	0,6184 (5)	0,8734 (5)	0,1923 (4)	0,068 (4)
N(1)	0,1408 (5)	0,8076 (5)	0,4373 (4)	0,045 (4)
N(2)	0,2016 (6)	0,9074 (5)	0,4585 (5)	0,055 (4)
N(3)	0,4197 (5)	0,7759 (5)	0,3966 (4)	0,043 (4)
N(4)	0,4132 (5)	0,9086 (5)	0,3031 (5)	0,051 (4)
C(1)	0,2623 (7)	0,6019 (6)	0,2562 (5)	0,042 (5)
C(2)	0,1694 (7)	0,4980 (7)	0,2800 (6)	0,052 (5)
C(3)	0,215 (1)	0,4007 (8)	0,2412 (7)	0,066 (6)
C(4)	0,358 (1)	0,4138 (9)	0,1762 (7)	0,070 (7)
C(5)	0,445 (1)	0,520 (1)	0,1504 (7)	0,076 (8)
C(6)	0,4023 (8)	0,6169 (8)	0,1898 (7)	0,061 (6)
C(7)	−0,0236 (9)	0,815 (1)	0,4220 (9)	0,070 (8)
C(8)	0,534 (1)	0,743 (1)	0,4929 (8)	0,060 (7)
C(9)	0,2294 (8)	0,8925 (8)	0,5832 (7)	0,057 (6)
C(10)	0,184 (1)	0,757 (1)	0,8100 (8)	0,161 (12)
C(11)	0,131 (2)	0,649 (2)	0,8953 (9)	0,218 (20)
C(12)	0,5275 (8)	0,9668 (9)	0,1989 (6)	0,059 (6)
C(13)	0,739 (1)	0,921 (1)	0,0854 (9)	0,099 (10)
C(14)	0,831 (1)	0,813 (1)	0,0968 (9)	0,147 (14)

Tabelle 2. *Geometrische Parameter* (Å, °)

P—S(1)	1,897 (2)	P—O(1)	1,615 (5)
P—N(1)	1,650 (6)	P—N(3)	1,665 (5)
S(2)—O(2)	1,440 (5)	S(2)—N(2)	1,713 (5)
S(2)—N(4)	1,758 (6)	O(1)—C(1)	1,407 (9)
N(1)—N(2)	1,377 (9)	N(3)—N(4)	1,385 (6)
N(1)—C(7)	1,49 (1)	N(3)—C(8)	1,49 (1)
N(2)—C(9)	1,41 (1)	N(4)—C(12)	1,420 (8)
C(9)—O(3)	1,18 (1)	C(12)—O(5)	1,19 (1)
C(9)—O(4)	1,329 (8)	C(12)—O(6)	1,32 (1)
O(4)—C(10)	1,49 (1)	O(6)—C(13)	1,47 (1)
Phenylring: C—C (Mittelwert)	1,37 (2)		
P—N(1)—N(2)	117,1 (4)	P—N(3)—N(4)	113,6 (4)
P—N(1)—C(7)	126,4 (6)	P—N(3)—C(8)	123,3 (4)
C(7)—N(1)—N(2)	116,6 (7)	C(8)—N(3)—N(4)	117,0 (6)
Winkelsumme um N(1)	360 (1)	Winkelsumme um N(3)	353,9 (8)
S(2)—N(2)—N(1)	116,6 (4)	S(2)—N(4)—N(3)	123,1 (4)
S(2)—N(2)—C(9)	116,0 (5)	S(2)—N(4)—C(12)	112,4 (5)
C(9)—N(2)—N(1)	123,8 (5)	C(12)—N(4)—N(3)	121,6 (6)
Winkelsumme um N(2)	356,4 (8)	Winkelsumme um N(4)	357,1 (9)
S(1)—P—O(1)	116,0 (2)	P—O(1)—C(1)	119,2 (3)
N(1)—P—O(1)	97,9 (3)	N(3)—P—O(1)	106,4 (2)
N(1)—P—S(1)	120,2 (2)	N(3)—P—S(1)	114,3 (2)
N(1)—P—N(3)	99,5 (3)	N(2)—S(2)—N(4)	90,9 (3)
N(2)—S(2)—O(2)	104,1 (3)	N(4)—S(2)—O(2)	105,9 (3)
Exocyclische Torsionswinkel um die N—N-Bindungen:			
C(7)—N(1)—N(2)—C(9)	98,3 (7)	C(8)—N(3)—N(4)—C(12)	71,9 (9)

Die Struktur wurde mit direkten Methoden gelöst (*MULTAN*; Main, Lessinger, Woolfson, Germain & Declercq, 1977). Alle anderen Rechnungen wurden mit dem Programmsystem *Xtal*2.6 (Hall & Stewart, 1989) durchgeführt. Die gegen Ende versuchte Verfeinerung der H-Atomlagen mit isotropen Verschiebungsparameter führte nicht in allen Fällen zu sinnvollen Atomlagen. Diese H-Atome [H(2), H(101), H(102), H(111), H(122), H(113), H(141) und H(142)] wurden geometrisch gesetzt. Ihnen wurden die isotropen Verschiebungsparameter der an sie gebundenen C-Atome zugewiesen.

Wir danken Herrn Kollegen D. Lentz für die Hilfe bei den Intensitätsmessungen. Dem Fonds der Chemischen Industrie gilt unser Dank für finanzielle Unterstützung.

Die Liste der Strukturfaktoren, anisotropen Verschiebungsparameter, H-Atom Koordinaten, und vollständigen geometrischen Daten sind bei dem IUCr (Aktenzeichen: SH1089) hinterlegt. Kopien sind erhältich durch: The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4,4′-Isopropylidenediphenyl Bis(chloroformate)

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Abstract

In the structure of $C_{17}H_{14}Cl_2O_4$, (1), each Cl—CO—O moeity is perfectly planar. The C=O carbonyl bonds appear normal with lengths of 1.177 (5) [C(1)—O(1)] and 1.162 (7) Å [C(17)—O(4)], as do the carbonyl-chlorine bonds at 1.762 (5) [C(1)—Cl(1)] and 1.742 (5) Å

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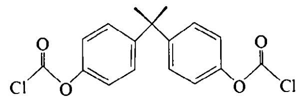
[C(17)—Cl(2)]. The planes defined by the Cl—CO—O groups are rotated through dihedral angles of 178.9 (3) [Cl(1)—C(1)—O(2)—C(2)] and 177.4 (3)° [C(14)—O(3)—C(17)—Cl(2)] relative to the adjacent phenyl rings. The two phenyl rings are canted relative to each other with a dihedral angle of 104.2 (10)°.

Comment

The crystal structure determination of (1) forms part of our continuing work on aryl carbonates (King & Bryant, 1990, 1992). There is a great deal of interest in the crystal structures of chloroformates (Exner & Fiedler, 1987; O'Gorman, Shand & Schomaker, 1950; Robinson, 1953; Wanner, Praschak & Nagel, 1990). Fig. 1 shows the molecule with the numbering scheme employed and Fig. 2 illustrates the projected packing of the molecules viewed down the *a* axis.



(1)

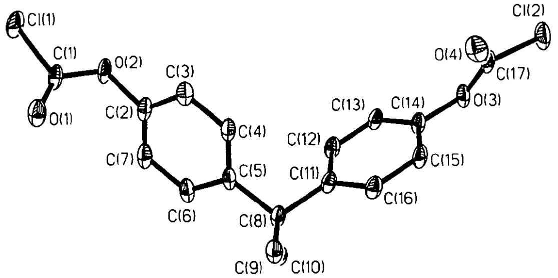


Fig. 1. Displacement-ellipsoid (50% probability) plot.

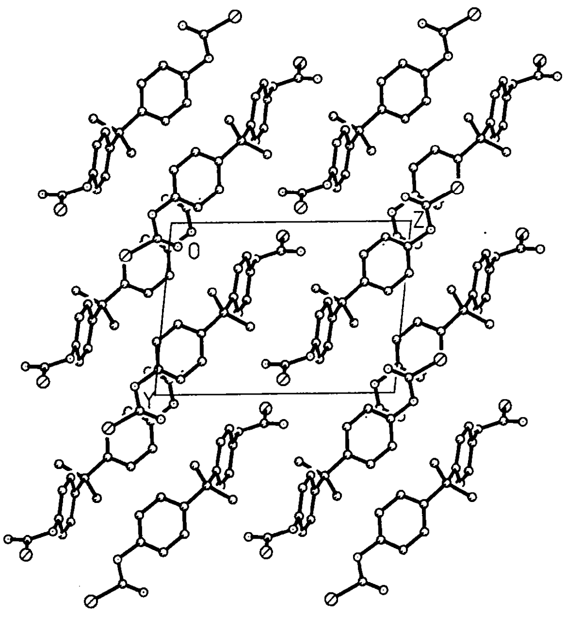


Fig. 2. Projected packing plot viewed down the *a* axis.

Experimental

Compound (1) was obtained by reaction of phosgene with bisphenol A in CH_2Cl_2 and was recrystallized from hexane at 253 K.

Crystal data

$C_{17}H_{14}Cl_2O_4$
$M_r = 353.2$
Triclinic
$P\bar{1}$
$a = 8.260\ (2)$ Å
$b = 8.394\ (2)$ Å
$c = 12.045\ (2)$ Å
$\alpha = 94.45\ (5)^\circ$
$\beta = 105.43\ (2)^\circ$
$\gamma = 96.17\ (2)^\circ$
$V = 795.3$ Å^3
$Z = 2$

$D_x = 1.475$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 23 reflections
$\theta = 15.3$–44.6°
$\mu = 3.89$ mm^{-1}
$T = 176$ K
Needles
0.36 × 0.32 × 0.18 mm
Colorless

Data collection

Siemens *R3m/V* upgrade of Nicolet *P3F* diffractometer
Wyckoff scans of variable speed
Absorption correction: none
2346 measured reflections
2166 independent reflections
2034 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.103$
$\theta_{\rm max} = 55^\circ$
$h = 0 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -13 \rightarrow 12$
2 standard reflections monitored every 48 reflections intensity variation: 7%

Refinement

Refinement on F
$R = 0.0588$
$wR = 0.0648$
$S = 5.91$
2034 reflections
208 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\rm max} = 0.003$
$\Delta\rho_{\rm max} = 0.41$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.76$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

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(1)	0.1724 (1)	0.0836 (2)	0.4690 (1)	0.032 (1)
C(1)	0.3771 (6)	0.1680 (5)	0.4687 (4)	0.020 (2)
O(1)	0.4955 (4)	0.1742 (4)	0.5498 (3)	0.030 (1)
O(2)	0.3658 (4)	0.2156 (4)	0.3663 (3)	0.028 (1)
C(2)	0.5202 (6)	0.2920 (6)	0.3475 (4)	0.025 (2)
C(3)	0.5346 (6)	0.4554 (6)	0.3446 (4)	0.024 (2)
C(4)	0.6764 (5)	0.5288 (6)	0.3146 (4)	0.022 (2)
C(5)	0.7954 (5)	0.4381 (5)	0.2894 (4)	0.018 (2)

C(6)	0.7754 (6)	0.2738 (6)	0.2962 (4)	0.023 (2)
C(7)	0.6358 (6)	0.1976 (6)	0.3262 (4)	0.026 (2)
C(8)	0.9466 (5)	0.5259 (5)	0.2559 (4)	0.019 (2)
C(9)	1.0380 (6)	0.4073 (6)	0.1970 (4)	0.026 (2)
C(10)	1.0739 (6)	0.6080 (6)	0.3698 (4)	0.027 (2)
C(11)	0.8821 (5)	0.6449 (5)	0.1696 (4)	0.019 (2)
C(12)	0.7515 (5)	0.5902 (5)	0.0690 (4)	0.021 (2)
C(13)	0.6925 (5)	0.6922 (5)	−0.0134 (4)	0.021 (2)
C(14)	0.7684 (6)	0.8509 (6)	0.0050 (4)	0.022 (2)
C(15)	0.8983 (6)	0.9095 (5)	0.1018 (4)	0.023 (2)
C(16)	0.9548 (5)	0.8067 (6)	0.1843 (4)	0.022 (2)
O(3)	0.7117 (4)	0.9488 (4)	−0.0853 (3)	0.024 (1)
O(4)	0.6515 (4)	1.1393 (4)	0.0364 (3)	0.032 (1)
C(17)	0.6589 (6)	1.0892 (6)	−0.0545 (4)	0.026 (2)
Cl(2)	0.6061 (2)	1.1890 (2)	−0.1781 (1)	0.035 (1)

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

Cl(1)—C(1)	1.762 (5)	C(15)—C(16)	1.387 (7)
C(1)—O(1)	1.177 (5)	O(3)—C(17)	1.359 (6)
C(1)—O(2)	1.310 (6)	O(4)—C(17)	1.162 (7)
O(2)—C(2)	1.445 (6)	C(17)—Cl(2)	1.742 (5)
C(14)—O(3)	1.424 (6)		
Cl(1)—C(1)—O(2)	107.3 (3)	C(14)—O(3)—C(17)	116.5 (4)
O(1)—C(1)—O(2)	130.3 (5)	O(3)—C(17)—O(4)	128.2 (5)
C(1)—O(2)—C(2)	116.7 (3)	O(3)—C(17)—Cl(2)	107.2 (4)
C(5)—C(8)—C(11)	109.2 (3)		

The crystal was sealed in a glass capillary under dry nitrogen because of its moisture sensitivity. Data were collected using Siemens *R3m/V* software. The structure was solved by direct methods and refined using the *SHELXTL-Plus* (Sheldrick, 1988) program package on a MicroVAX II computer. H atoms were placed in idealized positions and constrained to C—H = 0.96 Å with isotropic displacement parameters $U = 0.08$ Å^2. All non-H atoms were treated as anisotropic. *SHELXTL-Plus* was used throughout the study.

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 71735 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1085]

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

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Abstract

The low-temperature X-ray crystal structure of 2,2′,3,3′-tetra-*O*-acetyl-4,4′,6,6′-tetradeoxy-α,α-*xylo*-trehalose (I) (2,3-di-*O*-acetyl-4,6-dideoxy-α-D-*xylo*-hexopyranosyl 2,3-di-*O*-acetyl-4,6-dideoxy-α-D-*xylo*-hexopyranoside) is reported. The crystals were found to contain approximately 16% of 2,3-di-*O*-acetyl-6-chloro-4,6-dideoxy-α-D-*xylo*-hexopyranosyl 2,3-di-*O*-acetyl-4,6-dideoxy-α-D-*xylo*-hexopyranoside (II) as a result of incomplete hydrodechlorination of the intermediate 2,2′,3,3′-tetra-*O*-acetyl-4,4′,6,6′-tetrachloro-4,4′,6,6′-tetradeoxy-α,α-trehalose. Except for the chloro substituent, the two molecules are crystallographically identical and the structure may be refined using a disordered model with one H atom being replaced by a Cl atom in 16% of the molecules. The two hexopyranosyl residues in both molecules have $^4C_1$ conformations, with one of the residues adopting a nearly perfect chair conformation. The crystal packing places the Cl atom of (II) in a very crowded environment with a C—Cl bond length of 1.527 (7) Å and a short intermolecular Cl···C contact of 2.884 (9) Å.

Comment

α,α-Trehalose (α-D-glucopyranosyl α-D-glucopyranoside), a natural reserve sugar, is found in many bacteria and fungi, in plants, and in the blood of most insects, where it probably plays an important role in carbohydrate metabolism (Birch, 1963). A number of trehalose analogues, particularly the aminated ones, occur naturally as antibiotically active metabolites (Arcamone & Bizioli, 1957; Umezawa, Tasuta & Muto, 1967; Uramoto, Otaka & Yonehara, 1967; Naganawa, Usui, Takita, Hamada, Maeda & Umezawa, 1974; Dolak, Castle & Laborde, 1980). There is also considerable interest in the specificity and inhibitors of the trehalases, the specific hy-

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† Crystal Structures of Trehalose Derivatives, Part 5. For Part 4, see Lee & Linden (1994).

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