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Structure of Diphenyl Carbonate

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Abstract. Phenyl phenoxyformate, $C_{13}H_{10}O_3$, $M_r = 214.2$, orthorhombic, $P2_12_12_1$, $a = 6.062\,(2)$, $b = 7.242\,(1)$, $c = 23.375\,(4)$ Å, $V = 1026.2$ Å^3, $Z = 4$, $D_x = 1.387$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0.71069$ Å, $\mu = 0.92$ cm^{-1}, $F(000) = 448$, $T = 164$ K, $R = 0.0384$ for 1375 unique reflections with $I > 2\sigma(I)$. Diphenyl carbonate constitutes the simplest congener in the aromatic polycarbonate family. The two O—C$_{carbonyl}$ bond lengths are 1.345 (2) and 1.337 (2) Å. The O—C bond length of the carbonyl moiety is 1.191 (3) Å. The two C$_{aryl}$—O—C$_{carbonyl}$ angles are 118.4 (2) and 118.8 (2)°, while the two O—C$_{carbonyl}$—O$_{carbonyl}$ angles are 127.5 (2) and 127.8 (2)°. The two benzene rings are canted relative to the plane defined by the carbonate group. The dihedral angles between each benezene ring and the carbonate plane are 52.7 (2) and 57.7 (2)°.

Experimental. Compound (1), obtained by reaction of phosgene with phenol and triethylamine in methylene chloride, recrystallized from anhydrous ethanol at 298 K. A crystal was sealed in a glass

(1)
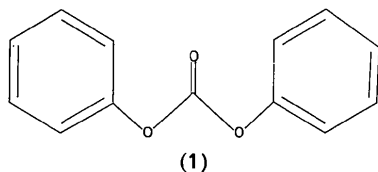
capillary for low-temperature data collection on a Siemens *R*3*m*/*V* upgrade of a Nicolet *P*3*F* automated diffractometer, using Wyckoff scans of variable scan speed. The structure was solved by direct methods and refined on *F* using the *SHELXTL-Plus* (Micro-

Table 1. *Experimental details*

Crystal	
Habit	Hexagonal rod
Size (mm)	0.52 × 0.24 × 0.24
Lattice-parameters determination	
No. of reflections	22
2θ range (°)	14.8–26.7
Reflection range	
h	−7 to 7
k	−9 to 2
l	−7 to 30
Maximum sinθ/λ (Å^{-1})	0.650
Check reflections	$0\bar{1}6$, $1\bar{1}3$
Variation (%)	6, 5
Reflections	
Collected	1495
Unique observed	1375
R_{int}	0.0161
Observed criterion	$I > 2\sigma(I)$
No. of parameters	146
R	0.0384
wR	0.0418
S	1.75
Secondary-extinction parameters, χ, in $F^* = F[1 + 0.002\chi F^2 / \sin(2\theta)]^{-1/4}$	0.0032 (5)
Weighting factor, g, in $w^{-1} = \sigma^2(F) + gF^2$	0.0002
Difference Fourier peaks	
Minimum (e Å^{-3})	−0.20
Maximum (e Å^{-3})	0.28
Maximum (Δ/σ)	0.005

VAX II) program package (Sheldrick, 1988). H atoms were placed in idealized positions and constrained to have C—H = 0.96 Å and isotropic thermal parameters, $U = 0.08$ Å^2. All non-H atoms were treated as anisotropic. No absorption correction was applied. A correction for secondary extinction of the type described by Zachariasen (1967) refined in the later stages with a maximum correction of 25% for the 041 reflection. There were no apparent groups of

Table 2. *Atom coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\mathrm{\AA}^2 \times 10^3$)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	151 (3)	8859 (3)	3651 (1)	18 (1)
O(1)	−1381 (2)	9822 (2)	3763 (1)	22 (1)
O(2)	1966 (2)	8564 (2)	3968 (1)	21 (1)
C(2)	2078 (3)	9374 (3)	4517 (1)	18 (1)
C(3)	4029 (3)	10273 (3)	4645 (1)	20 (1)
C(4)	4322 (4)	10945 (3)	5198 (1)	22 (1)
C(5)	2707 (4)	10688 (3)	5608 (1)	23 (1)
C(6)	762 (4)	9774 (3)	5468 (1)	22 (1)
C(7)	434 (3)	9103 (3)	4914 (1)	20 (1)
O(3)	457 (2)	7841 (2)	3180 (1)	25 (1)
C(8)	−1266 (3)	7732 (3)	2773 (1)	18 (1)
C(9)	−3322 (4)	7066 (3)	2920 (1)	22 (1)
C(10)	−4859 (4)	6763 (3)	2492 (1)	24 (1)
C(11)	−4319 (3)	7131 (3)	1925 (1)	24 (1)
C(12)	−2261 (4)	7827 (3)	1788 (1)	25 (1)
C(13)	−699 (3)	8133 (3)	2214 (1)	21 (1)

hkl affected by the correction, and the correction exceeded 15% for only 20 reflections. Details of the data collection are given in Table 1. Scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic coordinates are contained in Table 2.* Fig. 1 illustrates the molecule with the numbering scheme employed. Fig. 2 illustrates the projected packing of the molecules viewed down the *a* axis.

Related literature. For additional information on related structures and chemistry see King & Bryant (1990), King & Codella (1990), Bicerano & Clark (1988), Perez & Scaringe (1987), Hutnik, Argon &

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55582 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1002]
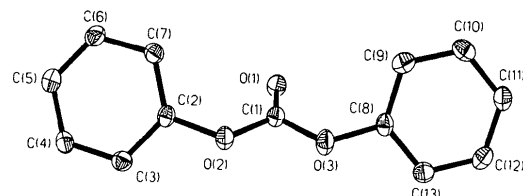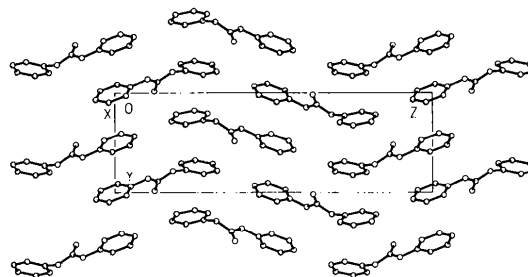

Fig. 1. Thermal ellipsoid (50% probability) plot.

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

Suter (1991), and Schmidt, Dybal, Turska & Kalczycki (1991).

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Structures of Two Polymorphic Acylpyridinium Salts

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Abstract. *N*-Methoxycarbonyl-3-methylpyridinium tetraphenylborate (I), $C_8H_{10}NO_2^+.C_{24}H_{20}B^-$, $M_r = 471.4$, monoclinic, $P2_1/n$, $a = 13.723 (4)$, $b = 9.827 (3)$, $c = 19.328 (7)$ Å, $\beta = 96.57 (3)°$, $V = 2589.3$ Å^3, $Z = 4$, $D_x = 1.209$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.69$ cm^{-1}, $F(000) = 1000$, $T = 173$ K, $R = 0.0543$ for 3472 unique reflections with $I > 4\sigma(I)$. *N*-Methoxycarbonyl-3-methylpyridinium

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