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Key indicators

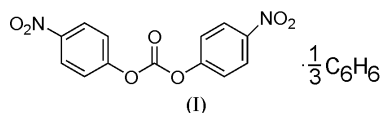
Single-crystal X-ray study
 $T = 133\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.034
 wR factor = 0.079
Data-to-parameter ratio = 13.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis(*p*-nitrophenyl) carbonate–benzene (3/1)The asymmetric unit of the title compound, $\text{C}_{13}\text{H}_8\text{N}_2\text{O}_7 \cdot \frac{1}{3}\text{C}_6\text{H}_6$, contains three molecules of the carbonate and one of benzene. The conformation of all carbonates is *s-cis-s-cis*, with some slight deviations from linearity. The aromatic rings subtend interplanar angles of 45–68° with the carbonate plane.

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Comment

In the previous paper (Simon *et al.*, 2003) we presented introductory material concerning diaryl carbonates, together with the structure of bis(*o*-nitrophenyl) carbonate. Here we present the structure of the isomeric bis(*p*-nitrophenyl) carbonate, which crystallizes from benzene as a solvate in which the asymmetric unit contains three molecules of the carbonate and one of benzene (Fig. 1). Atoms of the second and third independent molecules are distinguished by primes and double primes respectively.The carbonate moieties in the title compound, (I), all display the *s-cis-s-cis* conformation (*cf.* torsion angles in Table 1); the largest deviation of an *ipso* carbon from the carbonate plane (defined by the four atoms C1, O1; O2, O3) is 0.289 (2) Å for C21'. The aromatic rings are inclined to the carbonate plane at angles varying from 45 to 68° (for details see supplementary material).

Bond lengths and angles of the carbonate moieties (Table 1) may be considered normal, although the angle O2–C1–O3 is among the narrowest recorded for a diaryl carbonate.

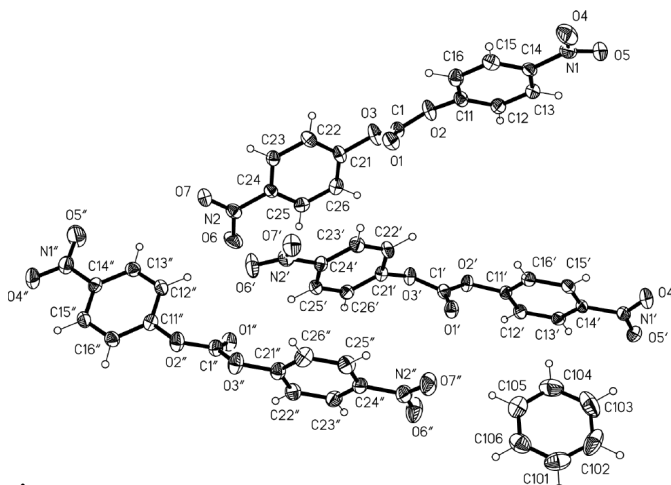


Figure 1

The asymmetric unit of the title compound in the crystal. Ellipsoids are drawn at the 50% probability level and H-atom radii are arbitrary.

There are eight short intramolecular contacts $X \cdots O$ ($X = C, N, O$) (see supplementary material). The molecular packing also involves a series of $C-H \cdots O$ hydrogen bonds (Table 2). However, perhaps the most striking feature of the packing is the $C104-H104 \cdots \pi$ contact involving the benzene ring and the centroid of $C11'-C16'$, with normalized $H \cdots \pi$ 2.73 Å, angle 172°. This can be recognized in Fig. 1.

Experimental

Triphosgene [bis(trichloromethyl) carbonate] was treated with six equivalents of *p*-nitrophenol and six equivalents of triethylamine in dichloromethane and the product was recrystallized from benzene (Simon *et al.*, 2001).

Crystal data

$C_{13}H_8N_2O_7 \cdot \frac{1}{3}C_6H_6$
 $M_r = 330.25$
 Monoclinic, $P2_1/c$
 $a = 21.074$ (2) Å
 $b = 8.4949$ (8) Å
 $c = 25.828$ (2) Å
 $\beta = 111.618$ (5)°
 $V = 4298.6$ (7) Å³
 $Z = 12$

$D_x = 1.531$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 6133 reflections
 $\theta = 2.5-26.4$ °
 $\mu = 0.12$ mm⁻¹
 $T = 133$ (2) K
 Prism, colourless
 0.40 × 0.25 × 0.15 mm

Data collection

Bruker SMART 1000 CCD diffractometer
 ω scans
 Absorption correction: none
 26129 measured reflections
 8781 independent reflections

5569 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.048$
 $\theta_{max} = 26.4$ °
 $h = -26 \rightarrow 18$
 $k = -10 \rightarrow 10$
 $l = -31 \rightarrow 32$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.079$
 $S = 0.92$
 8781 reflections
 649 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0333P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.24$ e Å⁻³
 $\Delta\rho_{min} = -0.21$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O1—C1	1.1809 (19)	O3'—C1'	1.3514 (19)
O2—C1	1.3494 (19)	O3'—C21'	1.4087 (18)
O2—C11	1.3981 (18)	O1''—C1''	1.1839 (19)
O3—C1	1.3466 (19)	O2''—C1''	1.3458 (19)
O3—C21	1.4019 (18)	O2''—C11''	1.4070 (19)
O1'—C1'	1.1872 (19)	O3''—C1''	1.355 (2)
O2'—C1'	1.3448 (19)	O3''—C21''	1.4024 (19)
O2'—C11'	1.4061 (18)		
O1—C1—O3	127.89 (15)	O2'—C1'—O3'	105.20 (13)
O1—C1—O2	128.36 (15)	O1''—C1''—O2''	127.82 (16)
O3—C1—O2	103.74 (14)	O1''—C1''—O3''	127.52 (16)
O1'—C1'—O2'	127.94 (15)	O2''—C1''—O3''	104.66 (14)
O1'—C1'—O3'	126.85 (15)		
C21—O3—C1—O1	0.1 (3)	C21'—O3'—C1'—O1'	14.2 (2)
C11—O2—C1—O1	-1.4 (3)	C11''—O2''—C1''—O1''	-6.6 (2)
C11'—O2'—C1'—O1'	-6.9 (2)	C21''—O3''—C1''—O1''	3.1 (2)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C23—H23 ⁱ ···O2 ⁱ	0.95	2.60	3.249 (2)	126
C16'—H16' ⁱⁱ ···O4 ⁱⁱ	0.95	2.56	3.476 (2)	161
C22'—H22' ⁱⁱⁱ ···O4 ⁱⁱⁱ	0.95	2.55	3.495 (2)	174
C22'—H22' ⁱⁱⁱ ···O5 ⁱⁱⁱ	0.95	2.64	3.3478 (19)	132
C15—H15 ^{iv} ···O6 ^{iv}	0.95	2.59	3.356 (2)	138
C13—H13 ^v ···O7 ^v	0.95	2.47	3.210 (2)	134
C16''—H16'' ^{vi} ···O1 ^{vi}	0.95	2.48	3.203 (2)	133
C26'—H26' ^{vii} ···O4 ^{vii}	0.95	2.62	3.2461 (19)	124
C22''—H22'' ^{viii} ···O5 ^{viii}	0.95	2.67	3.418 (2)	136
C23''—H23'' ^{ix} ···O5 ^{ix}	0.95	2.54	3.256 (2)	132
C12''—H12'' ^x ···O6 ^x	0.95	2.60	3.439 (2)	147
C22—H22 ^{xi} ···O7 ^{xi}	0.95	2.56	3.3540 (19)	141
C25'—H25' ^{xii} ···O1 ^{xii}	0.95	2.58	3.4736 (19)	157
C12'—H12' ^{xiii} ···O7 ^{xiii}	0.95	2.56	3.357 (2)	142

Symmetry codes: (i) $-x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $-x, 1 - y, -z$; (iii) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (iv) $x, \frac{3}{2} - y, \frac{1}{2} + z$; (v) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (vi) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (vii) $-x, y - \frac{1}{2}, \frac{1}{2} - z$; (viii) $x, y - 1, z$.

H atoms were included using a riding model, with fixed C—H bond lengths of 0.95 Å; $U_{iso}(H)$ values were fixed at 1.2 times U_{eq} of the parent atom.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP (Siemens, 1994); software used to prepare material for publication: SHELXL97.

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