Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Chloro-1,3-dioxa-2$\sigma^3\lambda^3$-phosphaanthracen-4-one

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.083
Data-to-parameter ratio = 16.3

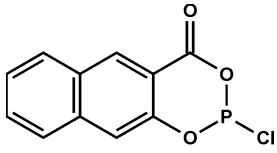
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 11 November 2002
Accepted 19 November 2002
Online 30 November 2002

The molecule of the title compound, $C_{11}H_6ClO_3P$, is essentially planar, except for the P, Cl and carbonyl O atoms. Bond lengths at phosphorus are P—O = 1.6110 (18) and 1.6290 (18) Å, and P—Cl = 2.0890 (9) Å. The molecules are linked to form ribbons parallel to the b axis by two C—H···O and one P···O interaction.

Comment

The title compound, (I), was obtained as a synthetic intermediate en route to phosphorus-substituted calix[4]arenes (Kunze, 2002; Kunze *et al.*, 2002).



(I)

The structure of (I) is shown in Fig. 1. Bond lengths and angles may be considered normal (Table 1). All non-H atoms, except P, Cl and O3, are coplanar (r.m.s. deviation 0.028 Å); these three atoms lie 0.491 (1), 2.525 (1) and −0.256 (2) Å, respectively, out of the mean plane (the minus sign indicating the opposite side of the plane).

The molecular packing involves three contacts, namely two 'weak' C—H···O hydrogen bonds (Table 2) and a P···O3 contact of 3.072 (2) Å (operator of O3: $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$). The overall effect of these is to link the molecules in ribbons parallel to the b axis (Fig. 2). The Cl···Cl contact between ribbons is, at 3.897 (1) Å (operator $x+\frac{1}{2}, -y+\frac{1}{2}, -z$), probably too long to be regarded as significant.

The structure of an isomeric material is presented in the following paper (Jones *et al.*, 2002).

Experimental

The title compound was prepared by the reaction of 3-hydroxynaphthalene-2-carboxylic acid with phosphorus trichloride in toluene (Hoechst, 1966) and recrystallized from dichloromethane/diethyl ether (2:1 *v*/*v*) (Kunze, 2002).

Crystal data

$C_{11}H_6ClO_3P$
M_r = 252.58
Orthorhombic, $P2_12_12_1$
a = 6.0031 (15) Å
b = 10.0697 (15) Å
c = 17.028 (3) Å
V = 1029.3 (4) Å^3
Z = 4
D_x = 1.630 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 56 reflections
θ = 10–11.5°
μ = 0.51 mm^{-1}
T = 173 (2) K
Prism, colourless
0.5 × 0.3 × 0.2 mm

Data collection

Stoe Stadi-4 diffractometer
ω/θ scans
Absorption correction: none
3936 measured reflections
2367 independent reflections
2114 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 27.5°$
$h = -7 \rightarrow 3$
$k = -13 \rightarrow 0$
$l = -22 \rightarrow 22$
3 standard reflections
frequency: 90 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.083$
$S = 1.06$
2367 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0289P)^2 + 0.1959P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}
Absolute structure: Flack (1983), 974 Friedel pairs
Flack parameter = −0.03 (10)

Table 1
Selected geometric parameters (Å, °).

P—O1	1.6110 (18)	P—Cl	2.0890 (9)
P—O2	1.6290 (18)		
O1—P—O2	100.95 (9)	C3—O1—P	123.43 (16)
O1—P—Cl	100.21 (7)	C11—O2—P	127.24 (15)
O2—P—Cl	98.33 (7)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O2^{i}	0.95	2.60	3.542 (3)	174
C8—H8···O3^{i}	0.95	2.56	3.484 (3)	164

Symmetry code: (i) $2 - x, \frac{1}{2} + y, \frac{1}{2} - z$.

The absolute structure was determined on the basis of 974 Friedel pairs. The bulk material is racemic, so that the concept of absolute configuration can only be applicable to the measured crystal. 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 the U_{eq} value of the parent atom.

Data collection: *DIF*4 (Stoe & Cie, 1992); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1992); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

Financial support from the Fonds der Chemischen Industrie is gratefully acknowledged. We thank Mr A. Weinkauf for technical assistance.

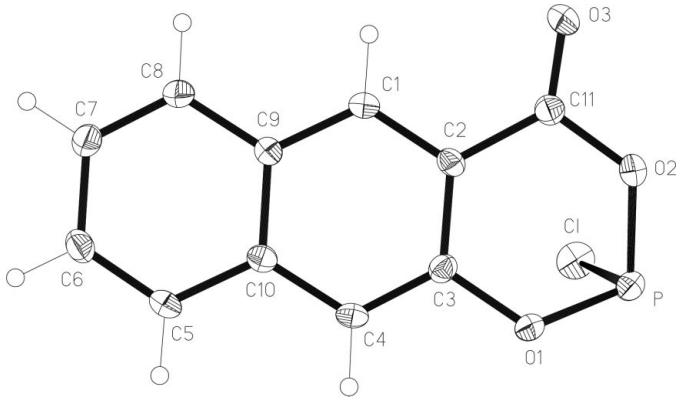


Figure 1
The molecule of the title compound in the crystal. Displacement ellipsoids are drawn at the 30% probability level. H-atom radii are arbitrary.

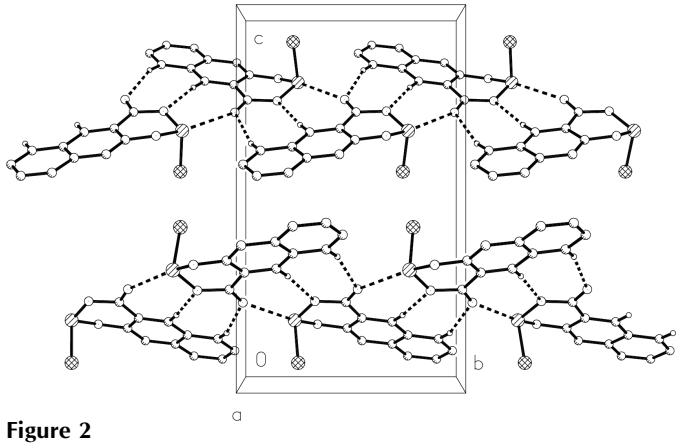


Figure 2
Packing diagram of the title compound, viewed parallel to the *a* axis. Secondary interactions are indicated by dashed bonds.

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