Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Peter G. Jones,* Christine Kunze, Matthias Freytag and Reinhard Schmutzler

Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany

Correspondence e-mail: jones@xray36.anchem.nat.tu-bs.de

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

Key indicators

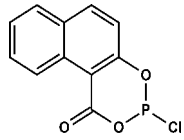
Single-crystal X-ray study
T = 143 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.088
Data-to-parameter ratio = 21.1

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

The molecule of the title compound, $C_{11}H_6ClO_3P$, is essentially planar, except for the P and Cl atoms. Bond lengths at phosphorus are P—O = 1.6141 (8) and 1.6143 (9), and P—Cl = 2.0958 (4) Å. The molecules are linked to form double ribbons parallel to the b axis by one C—H···O and one C—H···Cl 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 and Cl, are coplanar (r.m.s. deviation 0.031 Å); these atoms lie 0.451 (1) and 2.511 (1) Å, respectively, on the same side of the plane.

The molecular packing involves three contacts. Two 'weak' hydrogen bonds, one C—H···O and one C—H···Cl (Table 2), link the molecules in double ribbons parallel to the b axis (Fig. 2). A further Cl···Cl contact of 3.6114 (6) Å (operator $-x-1, -y, -z$), not shown in Fig. 2, links adjacent ribbons in the c direction.

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

Experimental

The title compound was prepared by the reaction of 2-hydroxynaphthalene-1-carboxylic acid with phosphorus trichloride in toluene and recrystallized from dichloromethane/diethyl ether (2:1 *v*/*v*) (Kunze, 2002). The bulk material had a greenish hue, although individual crystals are essentially colourless.

Crystal data

$C_{11}H_6ClO_3P$
M_r = 252.58
Triclinic, $P\overline{1}$
a = 7.1026 (6) Å
b = 8.3633 (8) Å
c = 10.1786 (10) Å
α = 103.794 (3)°
β = 102.520 (3)°
γ = 113.390 (3)°
V = 504.96 (8) Å^3
Z = 2
D_x = 1.661 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6669 reflections
θ = 2–30°
μ = 0.52 mm^{-1}
T = 143 (2) K
Tablet, colourless
0.40 × 0.36 × 0.14 mm

Data collection

Bruker SMART diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.812, T_{max} = 0.930
8958 measured reflections
3053 independent reflections
2780 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 30.5°
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.088
S = 1.04
3053 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.0767P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P—O2	1.6141 (8)	P—Cl	2.0958 (4)
P—O1	1.6143 (9)		
O2—P—O1	100.81 (4)	C2—O1—P	122.78 (6)
O2—P—Cl	99.62 (3)	C11—O2—P	127.24 (7)
O1—P—Cl	99.76 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O3^{i}	0.95	2.57	3.4512 (13)	155
C8—H8···Cl^{ii}	0.95	2.95	3.6622 (10)	133
C8—H8···O3	0.95	2.18	2.8402 (13)	126

Symmetry codes: (i) $x, y-1, z$; (ii) $-x, 1-y, 1-z$.

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 the U_{eq} value of the parent atom.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; 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.

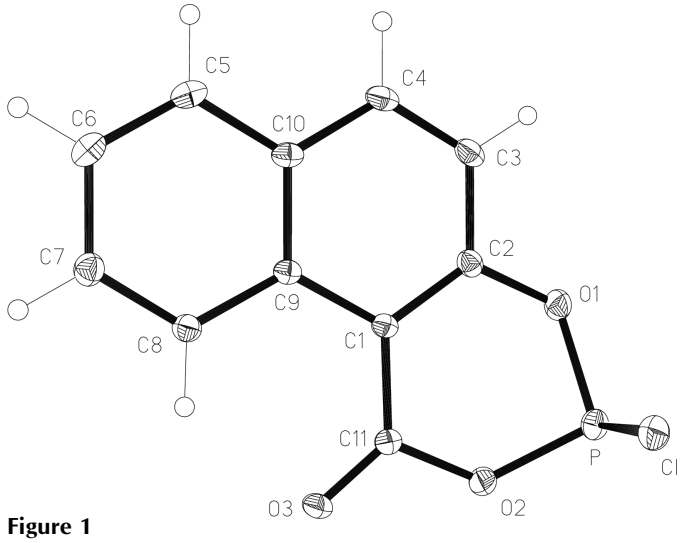


Figure 1
The molecule of the title compound in the crystal. Displacement ellipsoids are drawn at the 30% probability level. H-atom radii are arbitrary.

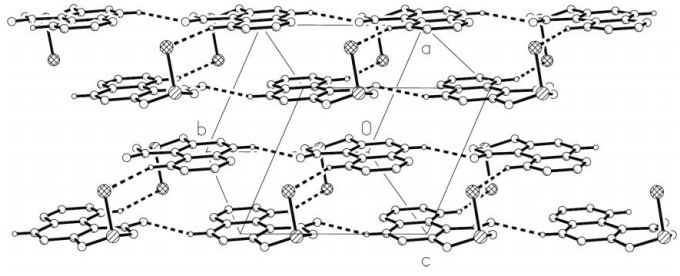


Figure 2
Packing diagram of the title compound, with the view direction approximately perpendicular to the *ab* plane. Secondary interactions are indicated by dashed bonds.

References

Bruker (1998). *SMART* (Version 5.0), *SAINT* (Version 4.0) and *SADABS* (Version 2.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Jones, P. G., Kunze, C., Freytag, M. & Schmutzler, R. (2002). *Acta Cryst.* E**58**, o1432–o1433.
Kunze, C. (2002). PhD Thesis, Technical University of Braunschweig, Germany. [ISBN 3-89720-612-9]
Kunze, C., Selent, D., Neda, I., Freytag, M., Jones, P. G., Schmutzler, R., Baumann, W. & Börner, A. (2002). *Z. Anorg. Allg. Chem.* **628**, 779–787.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1994). *XP*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.