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2-Hydroxy-2-phenyl-4,7-dihydroisophosphindolinium chloride

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The title compound, $C_{14}H_{16}OP^{+}\cdot Cl^{-}$ (systematic name: 2-hydroxy-2-phenyl-1,3,4,7-tetrahydroisophosphindenium chloride), displays a nearly planar isophosphindolinium skeleton, where a significant deviation from planarity is observed only for the P atom. Strong O—H···Cl bonds result in ion pairs, but an extended hydrogen-bonding network is not present.

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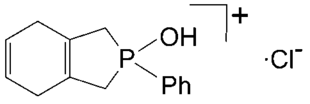
Key indicators

Single-crystal X-ray study
T = 198 K
Mean σ(C–C) = 0.003 Å
R factor = 0.025
wR factor = 0.066
Data-to-parameter ratio = 10.3

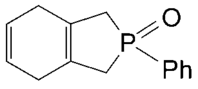
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The title compound, (I), is an intermediate in one of the avenues pursued in the synthesis and characterization of tricarbonyl(η^5-isophosphindolyl)manganese, the first complex bearing the isophosphindolyl skeleton (Decken *et al.*, 2004).



(I)



(II)

The isophosphindolinium ring system in (I) is not completely planar; the eight C atoms of the bicyclic unit fit a least-squares plane to within 0.037 (2) Å, but the P atom deviates by 0.378 (2) Å from this plane. This results in a folding angle of 17.6 (1)° with the C1/P/C3 plane. However, the distortion from planarity is not as pronounced as in the closely related compound 2-oxo-2-phenyl-1,3,4,7-tetrahydroisophosphindole, (II), (Decken *et al.*, 2004), where the deviation of the P atom is 0.484 (2) Å, leading to a folding angle of 22.58 (6)°. In addition, the envelope is folded toward the hydroxy group and away from the phenyl group in (I), opposite to the conformation adopted in (II). As a result, torsion angles are 99.6 (1) (C4a—C3—P—O), 102.8 (1) (C7a—C1—P—O), 145.7 (1) (C4a—C3—P—C8) and 145.2 (1)° (C7a—C1—P—8) in compound (I), while the corresponding angles are 135.3 (1), 134.6 (1), 88.9 (1) and 87.7 (1)° in (II). The title compound is ionic, with Cl^{-} anions located between sheets of the organic cations and held by strong O—H···Cl hydrogen bonds [1.87 (3) Å]. However, a much shorter hydrogen bond is observed in Ph_3P—O—H···Cl, (III) [1.63 (4) Å; Haupt *et al.*, 1977], the only hydroxyphosphonium chloride previously characterized by X-ray crystallography. The P—O bond length [1.5516 (14) Å] is comparable to the corresponding distances in (III) [1.517 (2) Å], Ph_3P—O—H···Br [1.550 (6) Å; Lane *et al.*, 1992] and $^{i}Pr_3P$—O—H···I [1.573 (2) Å; Ruthe *et al.*, 2000]. The P atom in (I) is in a tetrahedral environment and the phenyl group is almost orthogonal to the isophosphindolinium fragment [87.56 (4)°], similar to the geometry observed in (II) [87.06 (6)°].

Experimental

The title compound is an intermediate in the published synthesis of 2-phenyl-1,3,4,7-tetrahydroisophosphindole 2-oxide (Middlemas & Quin, 1979). ^{31}P {^{1}H} NMR (121.356 MHz, CDCl$_3$): δ 62.0. ^{1}H NMR (399.945 MHz, CDCl$_3$): δ 7.75 (*dd*, J = 7.2, 7.2 Hz, 2H), 7.58 (*d*, J = 7.2 Hz, 1H), 7.51 (*dd*, J = 7.2, 7.2 Hz, 2H), 5.80 (*s*, 2H), 2.98 (*t*, J = 16.8 Hz, 2H), 2.75 (*m*, 6H).

Crystal data

$C_{14}H_{16}OP^+\cdot Cl^-$
$M_r = 266.69$
Orthorhombic, $Pna2_1$
$a = 14.9111\ (15)$ Å
$b = 8.7445\ (9)$ Å
$c = 10.4465\ (10)$ Å
$V = 1362.1\ (2)$ Å^3
$Z = 4$
$D_x = 1.300$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 4853 reflections
$\theta = 2.7$–$25.5°$
$\mu = 0.38$ mm^{-1}
$T = 198\ (2)$ K
Irregular fragment, colourless
$0.50 \times 0.28 \times 0.25$ mm

Data collection

Bruker AXS SMART 1000/*P*4 diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.816$, $T_{\max} = 0.910$
6519 measured reflections

2254 independent reflections
2180 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\max} = 25.0°$
$h = -17 \rightarrow 16$
$k = -9 \rightarrow 10$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.066$
$S = 1.06$
2254 reflections
218 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2 + 0.02P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.11$ e Å^{-3}
Absolute structure: Flack (1983), 997 Friedel pairs
Flack parameter = 0.04 (6)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O—H14$\cdots$Cl$^{\rm i}$	0.98 (3)	1.87 (3)	2.8449 (16)	172 (2)

Symmetry code: (i) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} + z$.

H atoms were located in Fourier difference maps and refined with isotropic displacement parameters; C—H distances are in the range 0.90 (2)–0.99 (2) Å.

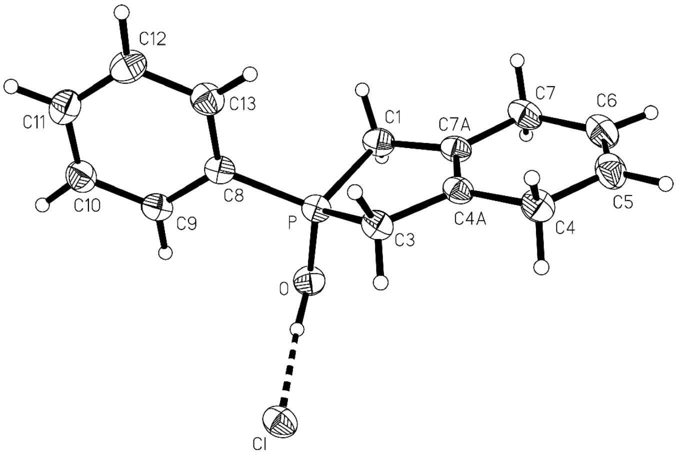


Figure 1
A view of the title compound, with displacement ellipsoids drawn at the 30% probability level. The dashed line indicates a hydrogen bond.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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