

Matthias Scheibitz,
Hans-Wolfram Lerner and
Michael Bolte*

Institut für Anorganische Chemie, J. W. Goethe-
 Universität Frankfurt, Marie-Curie-Strasse 11,
 60439 Frankfurt/Main, Germany

Correspondence e-mail:
 bolte@chemie.uni-frankfurt.de

Key indicators

Single-crystal X-ray study
 $T = 173$ K
 Mean $\sigma(C-C) = 0.004$ Å
 R factor = 0.032
 wR factor = 0.066
 Data-to-parameter ratio = 17.4

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

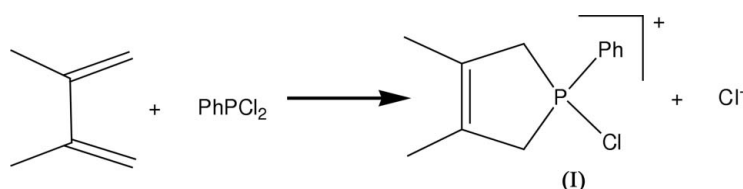
1-Chloro-3,4-dimethyl-1-phenyl-3-phos- pholenium chloride

Both ions of the title compound, $C_{12}H_{15}ClP^+ \cdot Cl^-$, are located on a crystallographic mirror plane. The phospholene ring adopts an envelope conformation. The crystal structure is stabilized by $C-H \cdots Cl$ hydrogen bonds.

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Comment

Today phosphole ligands play an important role in coordination chemistry. It has been suggested that phosphole derivatives can function as analogues for the common cyclopentadienyl (Cp) ligand. We report here the X-ray crystal structure analysis of an isolable intermediate in the synthesis of 3,4-dimethyl-1-phenylphosphole (Breque *et al.*, 1981). As indicated in the scheme below, the reaction of phenyl-dichlorophosphine with 2,3-dimethyl-1,3-butadiene produces the title compound, (I), in high yield. The subsequent reaction to obtain the desired phosphole is described in the following paper (Scheibitz *et al.*, 2006).



A perspective view of (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 5.27 plus one update; *MOGUL* Version 1.1; Allen, 2002). The phospholene heterocycle adopts an

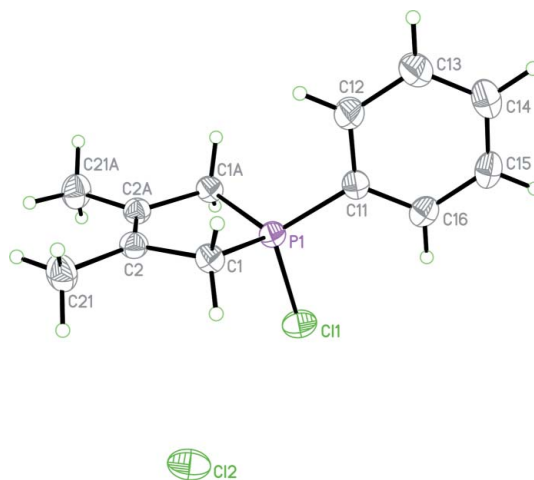


Figure 1
 Perspective view of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (A) $x, \frac{3}{2} - y, z$.]

envelope conformation. The four C atoms are exactly planar and the P atom deviates by 0.340 (4) Å from this plane. The methyl groups are exactly coplanar, since this ring lies across a crystallographic mirror plane. The phenyl ring lies in the mirror plane and is hence exactly perpendicular to both the C₄ plane and the PC₂ flap of the phospholene ring. The crystal structure is stabilized by C—H···Cl hydrogen bonds (Table 2).

Experimental

A mixture of phenyldichlorophosphine (5.84 g, 33 mmol) and 2,3-dimethyl-1,3-butadiene (5.89 g, 72 mmol) was stirred for 2 h at 273 K. After storing for 24 h at ambient temperature, colourless crystals of the title compound grew from this mixture (yield 90%).

Crystal data

C₁₂H₁₅ClP⁺·Cl⁻

M_r = 261.11

Orthorhombic, *Pnma*

a = 11.0359 (13) Å

b = 7.0120 (12) Å

c = 16.657 (2) Å

V = 1289.0 (3) Å³

Z = 4

D_x = 1.346 Mg m⁻³

Mo *K*α radiation

Cell parameters from 6837

reflections

θ = 3.6–26.5°

μ = 0.59 mm⁻¹

T = 173 (2) K

Needle, colourless

0.32 × 0.14 × 0.12 mm

Data collection

Stoe IPDS-II two-circle
diffractometer

ω scans

Absorption correction: multi-scan

(*MULABS*; *Spek*, 2003;

Blessing, 1995)

T_{min} = 0.833, *T_{max}* = 0.932

9136 measured reflections

1441 independent reflections

1081 reflections with *I* > 2σ(*I*)

R_{int} = 0.085

θ_{\max} = 26.5°

h = -12 → 13

k = -8 → 8

l = -20 → 20

Refinement

Refinement on *F*²

R [*F*² > 2σ(*F*²)] = 0.032

wR (*F*²) = 0.066

S = 0.91

1441 reflections

83 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0273P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

($\Delta\sigma$)_{max} < 0.001

$\Delta\rho_{\max} = 0.28 \text{ e } \text{Å}^{-3}$

$\Delta\rho_{\min} = -0.20 \text{ e } \text{Å}^{-3}$

Table 1

Selected bond lengths (Å).

Cl1—P1	1.9855 (10)	P1—C1	1.7914 (18)
P1—C11	1.786 (3)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C16—H16···Cl1	0.95	2.72	3.233 (3)	115
C1—H1A···Cl2	0.99	2.59	3.535 (2)	161
C1—H1B···Cl2 ⁱ	0.99	2.58	3.529 (2)	160

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

All H atoms were located in a difference Fourier synthesis, but were refined with fixed individual displacement parameters [*U*_{iso}(H) = 1.2*U*_{eq}(C) or 1.5*U*_{eq}(methyl C)] using a riding model, with aromatic C—H = 0.95 or methyl C—H = 0.98 Å. The methyl group was allowed to rotate but not to tip.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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