

Matthias Scheibitz,  
Hans-Wolfram Lerner and  
Michael Bolte\*Institut für Anorganische Chemie, J. W. Goethe-  
Universität Frankfurt, Marie-Curie-Strasse 11,  
60439 Frankfurt/Main, GermanyCorrespondence e-mail:  
bolte@chemie.uni-frankfurt.de

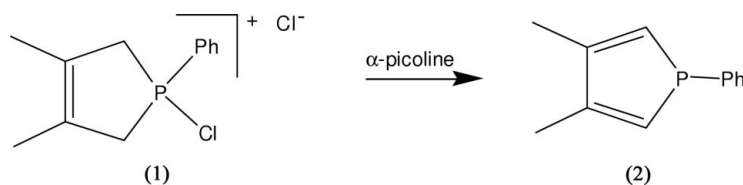
## Key indicators

Single-crystal X-ray study  
 $T = 173$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.052  
 $wR$  factor = 0.118  
Data-to-parameter ratio = 19.4For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

## 3,4-Dimethyl-1-phenylphosphole

The heterocycle of the title compound,  $\text{C}_{12}\text{H}_{13}\text{P}$ , is planar and forms a dihedral angle of  $78.45(8)^\circ$  with the phenyl ring.Received 3 March 2006  
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## Comment

In the preceding paper (Scheibitz *et al.*, 2006), we have described the synthesis of 1-chloro-3,4-dimethyl-1-phenyl-3-phospholene, (1). 3,4-Dimethyl-1-phenylphosphole represents the reaction product of the adduct (1) and  $\alpha$ -picoline (Scheibitz *et al.*, 2006; Breque *et al.*, 1981). The treatment of (1) with  $\alpha$ -picoline gives the phosphole (2), as shown in the scheme below.

A perspective view of (2) 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). Whereas the cyclic C—P—C angle is close to  $90^\circ$ , the other two angles at P adopt almost tetrahedral values (Table 1). The phosphole heterocycle is planar (r.m.s. deviation for the five ring atoms =  $0.047$  Å). Both methyl groups are almost coplanar with the heterocycle; the deviation from the ring plane is  $0.050(4)$  Å for C21 and  $0.185(4)$  Å for C31. The dihedral angle between the phosphole ring and the phenyl ring is  $78.45(8)^\circ$ .

## Experimental

A solution of  $\alpha$ -picoline (6.9 ml, 70 mmol) in dichloromethane (10 ml) was added dropwise to a mixture of (1) (30 mmol) in hexane and  $\text{CH}_2\text{Cl}_2$  (2:1, 30 ml) at ambient temperature. After addition of aqueous HCl ( $3 \text{ mol l}^{-1}$ , 6 ml), two layers separated. On removal of the solvent from the organic layer (313 K, 40 mbar), phosphole (2) remained as a colourless liquid. Single crystals of (2) were obtained by cooling the product to 233 K for 48 h (yield 4.02 g, 71%).

## Crystal data

$\text{C}_{12}\text{H}_{13}\text{P}$   
 $M_r = 188.19$   
Orthorhombic,  $P2_12_12_1$   
 $a = 5.9662(7)$  Å  
 $b = 7.9654(9)$  Å  
 $c = 22.227(2)$  Å  
 $V = 1056.3(2)$  Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.183 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation  
Cell parameters from 9609  
reflections  
 $\theta = 3.6\text{--}27.3^\circ$   
 $\mu = 0.21 \text{ mm}^{-1}$   
 $T = 173(2)$  K  
Block, colourless  
 $0.30 \times 0.25 \times 0.25 \text{ mm}$

**Data collection**

Stoe IPDS-II two-circle diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)  
 $T_{\min} = 0.937$ ,  $T_{\max} = 0.946$   
 11664 measured reflections

2331 independent reflections  
 1934 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.082$   
 $\theta_{\text{max}} = 27.3^\circ$   
 $h = -7 \rightarrow 7$   
 $k = -10 \rightarrow 10$   
 $l = -28 \rightarrow 28$

**Refinement**

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.052$   
 $wR(F^2) = 0.118$   
 $S = 1.01$   
 2331 reflections  
 120 parameters  
 H-atom parameters constrained

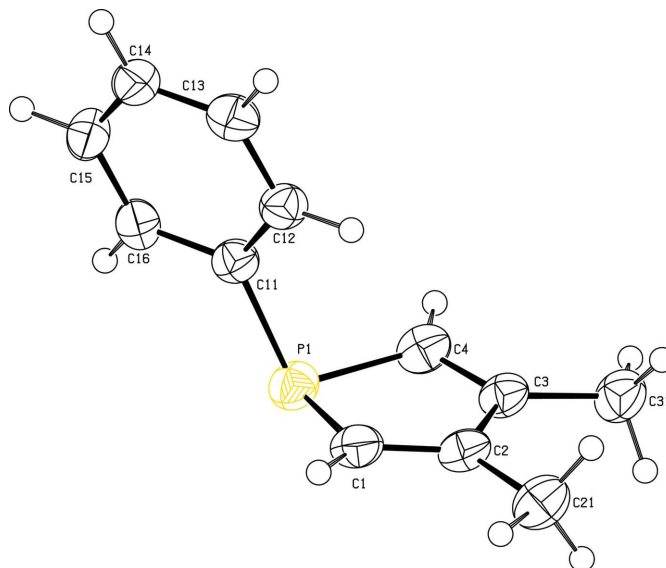
$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.18 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.27 \text{ e } \text{\AA}^{-3}$   
 Absolute structure: Flack (1983),  
 952 Friedel pairs  
 Flack parameter:  $-0.04$  (15)

**Table 1**Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

P1–C1	1.794 (3)	C1–C2	1.344 (4)
P1–C4	1.793 (3)	C2–C3	1.480 (3)
P1–C11	1.843 (2)	C3–C4	1.355 (4)
C1–P1–C4	90.02 (12)	C4–P1–C11	105.95 (11)
C1–P1–C11	103.08 (11)		

All H atoms were located in a difference Fourier synthesis, but were refined with fixed individual displacement parameters [ $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{methyl C})$ ] using a riding model, with aromatic C–H =  $0.95 \text{ \AA}$  or methyl C–H =  $0.98 \text{ \AA}$ . The methyl groups were 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:

**Figure 1**

Perspective view of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level.

*PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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