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#### Key indicators

Single-crystal X-ray study  
T = 193 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$   
R factor = 0.041  
wR factor = 0.092  
Data-to-parameter ratio = 16.4

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

## 2-(3-Methyl-1,2-butadienyl)-2-oxo-1,3,2-oxazaphosphorinane

The structure of the title compound,  $\text{C}_8\text{H}_{14}\text{NO}_2\text{P}$ , the first of a compound containing a 1,2-butadienyl group bound to the phosphorus of a 1,3,2-oxazaphosphorinane ring, is reported.

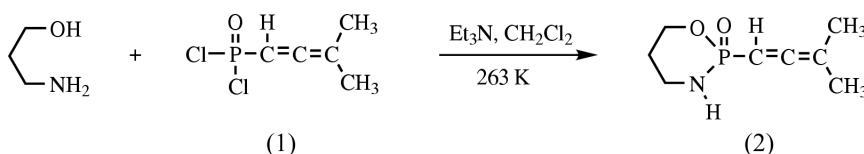
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### Comment

As early as the 1960's it was found that acetylene phosphines (obtained by the reaction of phosphorus trichloride with  $\alpha$ -acetylenic alcohols) spontaneously isomerized under mild conditions to afford allenephosphonates (Ignat'ev *et al.*, 1967; Mark, 1969). This simple strategy provides a synthetic route to numerous compounds with a variety of substituents on the phosphorus. These phosphorylated allenes are very reactive compounds and have great potential as intermediates for the preparation of many organophosphorus compounds with medical applications (Alabugin & Brel, 1997).

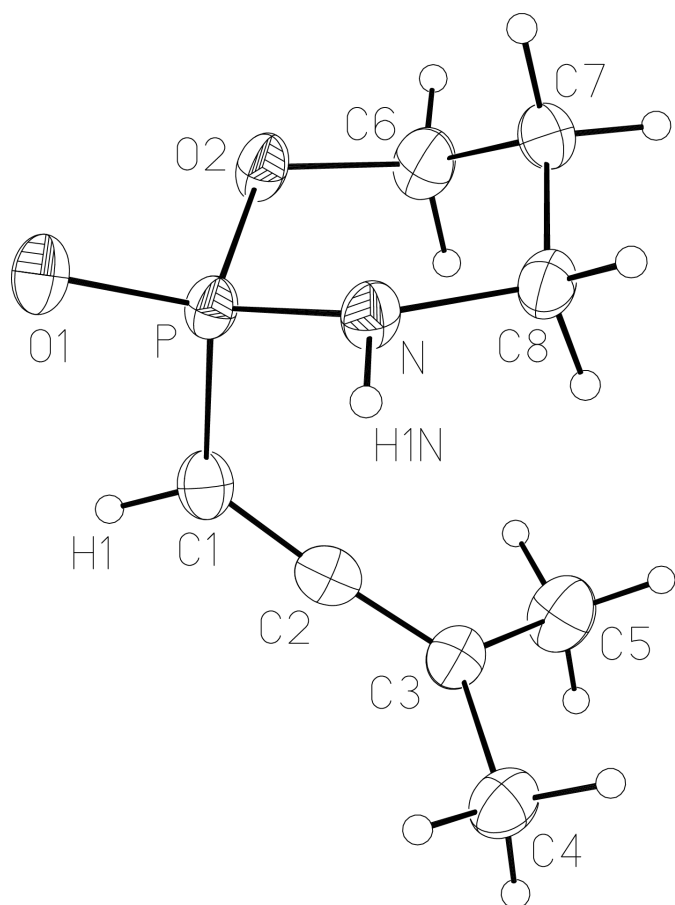


In this short communication, we describe a method to prepare 2-(3-methyl-1,2-butadienyl)-2-oxo-1,3,2-oxazaphosphorinane, (2), as a precursor for new synthetic anticancer agents. Compound (2) was prepared from (3-methyl-1,2-butadien-1-yl)phosphonic acid dichloride, (1), and 3-amino-propanol under basic conditions, as shown in the *Scheme* above.

The solid-state molecular structure of (2) is shown in Fig. 1. The 1,3,2-oxazaphosphorinane ring adopts a chair conformation, with atom C7 at the head and the P atom at the tail of the chair. The P—O2 and P—N distances and the O2—P—N, P—O2—C6 and P—N—C8 angles are consistent with those previously observed in compounds containing a 2-oxo-1,3,2-oxazaphosphorinane ring. The allenyl unit is essentially linear [ $\text{C}1-\text{C}2-\text{C}3 = 175.2(3)^\circ$ ], with the C1—C2 [1.296(4) Å] and C2—C3 [1.299(4) Å] distances almost identical, despite the dissimilar bonding environments of C1 (CHP) and C3 (CMe<sub>2</sub>). Interactions between adjacent molecules in the crystal lattice are illustrated in Fig. 2. The amino atom H1N forms a weak interaction with the O1 atom of the molecule generated by the *c*-glide plane [ $\text{H}1\text{N}\cdots\text{O}1(3/2-x, y, z-1/2) = 2.02 \text{ \AA}$ ].

### Experimental

Compound (2) was recrystallized from THF to give light-yellow crystals, with m.p. 383 K; <sup>31</sup>P NMR  $\delta(\text{CDCl}_3, \textit{versus ext. } 85\% \text{ H}_3\text{PO}_4)$  15.5.



**Figure 1**  
Perspective view of (2), showing the atom-labelling scheme. Non-H atoms are represented by Gaussian ellipsoids at the 50% probability level.

#### Crystal data

$C_8H_{14}NO_2P$   
 $M_r = 187.17$   
 Orthorhombic,  $Pca2_1$   
 $a = 9.6912$  (16) Å  
 $b = 13.389$  (2) Å  
 $c = 7.1699$  (12) Å  
 $V = 930.3$  (3) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.336$  Mg m<sup>-3</sup>

#### Data collection

Bruker PLATFORM/SMART 1000  
 CCD area-detector  
 diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.864$ ,  $T_{\max} = 0.980$   
 5403 measured reflections

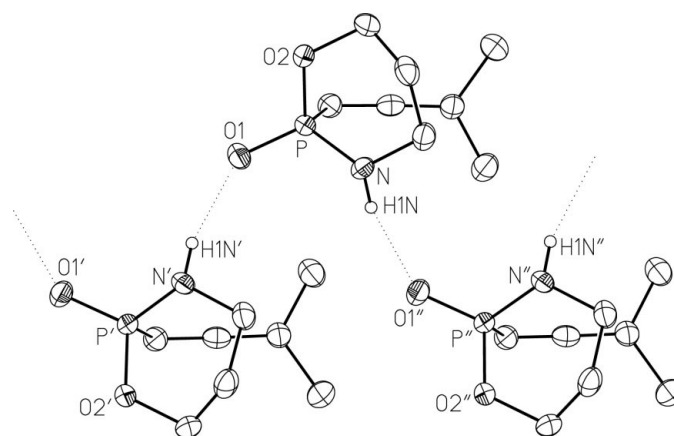
#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.092$   
 $S = 1.06$   
 1882 reflections  
 115 parameters  
 H atoms treated by a mixture of  
 independent and constrained  
 refinement

Mo  $K\alpha$  radiation  
 Cell parameters from 3122  
 reflections  
 $\theta = 2.6$ – $26.4^\circ$   
 $\mu = 0.26$  mm<sup>-1</sup>  
 $T = 193$  (2) K  
 Rod, colorless  
 $0.59 \times 0.09 \times 0.08$  mm

1882 independent reflections  
 1600 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.054$   
 $\theta_{\text{max}} = 26.4^\circ$   
 $h = -12 \rightarrow 12$   
 $k = -16 \rightarrow 16$   
 $l = -8 \rightarrow 8$

$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.49$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.23$  e Å<sup>-3</sup>  
 Absolute structure: Flack (1983),  
 844 Friedel pairs  
 Flack parameter = 0.00 (14)



**Figure 2**  
Illustration of interactions between neighbouring molecules of compound (2). Primed atoms are related to unprimed atoms via the symmetry operation  $(3/2-x, y, z-1/2)$  and double-primed atoms are related to unprimed atoms via the symmetry operation  $(1/2-x, y, z-1/2)$  (both of which are generated by the  $c$ -glide plane). All H atoms, except those bound to N atoms, have been omitted.

**Table 1**

Selected geometric parameters (Å, °).

P–O1	1.464 (2)	C1–C2	1.296 (4)
P–O2	1.5819 (16)	C2–C3	1.299 (4)
P–N	1.623 (2)	C3–C4	1.498 (4)
P–C1	1.787 (3)	C3–C5	1.498 (4)
O2–C6	1.445 (3)	C6–C7	1.507 (4)
N–C8	1.470 (4)	C7–C8	1.517 (4)
O1–P–O2	111.06 (12)	C8–N–H1N	111.6 (19)
O1–P–N	114.98 (12)	P–C1–C2	122.4 (2)
O1–P–C1	109.79 (12)	C1–C2–C3	175.2 (3)
O2–P–N	103.77 (11)	C2–C3–C4	121.9 (3)
O2–P–C1	105.35 (11)	C2–C3–C5	121.5 (3)
N–P–C1	111.33 (13)	C4–C3–C5	116.5 (3)
P–O2–C6	118.32 (17)	O2–C6–C7	109.6 (2)
P–N–C8	122.16 (18)	C6–C7–C8	110.9 (2)
P–N–H1N	114.7 (17)	N–C8–C7	110.4 (2)

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N–H1N <sup>i</sup> ⋯O1 <sup>i</sup>	0.88	2.02	2.872 (3)	161.7

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

H atoms attached to C atoms were refined with fixed C–H distances and with isotropic displacement parameters 20% greater than those for their attached atoms. The amino atom H1N was assigned a fixed N–H distance (0.88 Å) but its atomic coordinates and isotropic displacement parameter were otherwise allowed to refine freely.

Data collection: SMART (Bruker, 1997); cell refinement: SMART; data reduction: SHELXTL (Sheldrick, 1997a); program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997b); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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