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## Ethylidiphenylphosphine Oxide, (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>(C<sub>2</sub>H<sub>5</sub>)PO

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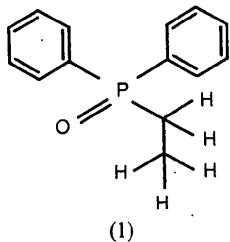
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### Abstract

The central P atom of the title compound, C<sub>14</sub>H<sub>15</sub>OP, has a distorted tetrahedral environment, with a P—O bond length of 1.484 (2) Å and P—C bond lengths in the range 1.796 (2)–1.800 (3) Å. The O—P—C angles vary from 111.6 (1) to 114.1 (1)° and C—P—C from 105.2 (1) to 107.5 (1)°.

### Comment

In an attempt to develop new synthetic routes to the tetrahydroindene ligand 3,4-bis(diphenylphosphino)-4,7-methano-3a,4,7,7a-tetrahydroindene (Orama, Hietala & Koskinen, 1990), we obtained colorless crystals of ethyldiphenylphosphine oxide (1).



The geometry around the central P atom is slightly distorted tetrahedral (Fig. 1). The O—P—C angles are similar to those found in tri-*p*-tolylphosphine oxide hemihydrate (Churchill, See, Randall & Atwood, 1993) and in bis(diphenylphosphonyl)acetylene (Orama, Karhu, Näsäkkälä, Sundberg & Ugglä, 1979), which have O—P—C angles in the range 111.6–114.5°. The C—P—C angles are slightly smaller than the C—P—O angles; the corresponding C—P—C angles in the above cited compounds are in the range 103.4–108.8°. The P(1)—O(1) bond length is also comparable, the corresponding bonds being 1.485 and 1.482 Å in the related compounds. The molecules pack so as to form chains with short contacts between an ethyl and

two phenyl groups of one molecule and the O atom of the neighboring molecule, with O···H distances in the range 2.458 (3)–2.551 (3) Å (Fig. 2).

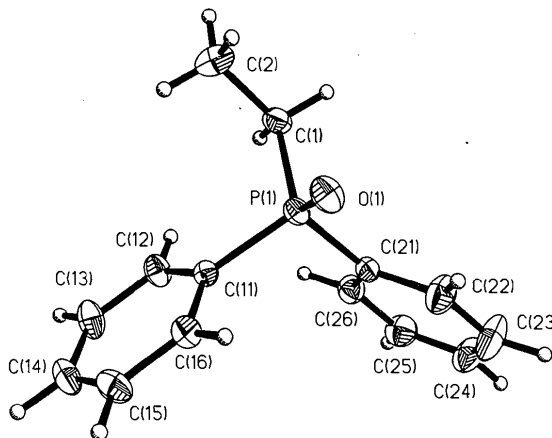


Fig. 1. A view of the title molecule (1) showing the labeling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels.

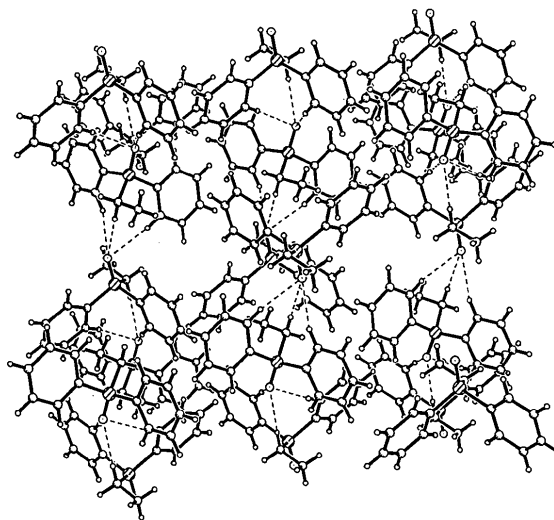


Fig. 2. Molecular packing with short C—H···O contacts indicated by dashed lines.

### Experimental

#### Crystal data

C<sub>14</sub>H<sub>15</sub>OP  
*M<sub>r</sub>* = 230.2  
 Monoclinic  
*P*2<sub>1</sub>/*n*  
*a* = 8.526 (3) Å  
*b* = 10.856 (4) Å  
*c* = 14.448 (4) Å  
 $\beta$  = 106.48 (5)°  
*V* = 1282 (1) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.193 Mg m<sup>-3</sup>

Mo *K*α radiation  
 $\lambda$  = 0.71069 Å  
 Cell parameters from 18 reflections  
 $\theta$  = 7–14°  
 $\mu$  = 0.191 mm<sup>-1</sup>  
*T* = 293 K  
 Plates  
 0.45 × 0.40 × 0.20 mm  
 Colorless

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**Data collection**

Nicolet P3F diffractometer	$\theta_{\max} = 24^\circ$
$\omega$ scans	$h = 0 \rightarrow 9$
Absorption correction:	$k = 0 \rightarrow 12$
none	$l = -16 \rightarrow 15$
2166 measured reflections	2 standard reflections
2017 independent reflections	monitored every 98
1600 observed reflections	reflections
[ $I > 4\sigma(I)$ ]	intensity variation: 2%
$R_{\text{int}} = 0.022$	

**Refinement**

Refinement on $F$	$w = 1/[\sigma^2(F) + 0.0002F^2]$
$R = 0.039$	$(\Delta/\sigma)_{\max} = 0.018$
$wR = 0.043$	$\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$
$S = 1.61$	$\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$
1600 reflections	Atomic scattering factors
145 parameters	from <i>International Tables</i>
Riding model, C—H 0.96 $\text{\AA}$ ,	for <i>X-ray Crystallography</i>
fixed isotropic $U = 0.08 \text{\AA}^2$	(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
P(1)	0.1715 (1)	0.1768 (1)	0.2207 (1)	0.037 (1)
O(1)	0.1395 (2)	0.0437 (1)	0.2298 (1)	0.054 (1)
C(1)	0.3326 (3)	0.2377 (2)	0.3187 (2)	0.048 (1)
C(2)	0.3015 (4)	0.2205 (3)	0.4164 (2)	0.076 (1)
C(11)	-0.0085 (3)	0.2680 (2)	0.2118 (2)	0.034 (1)
C(12)	-0.0032 (3)	0.3912 (2)	0.2362 (2)	0.047 (1)
C(13)	-0.1464 (3)	0.4558 (2)	0.2265 (2)	0.058 (1)
C(14)	-0.2959 (3)	0.3988 (3)	0.1923 (2)	0.056 (1)
C(15)	-0.3023 (3)	0.2775 (3)	0.1664 (2)	0.056 (1)
C(16)	-0.1594 (3)	0.2117 (2)	0.1764 (2)	0.045 (1)
C(21)	0.2283 (3)	0.2101 (2)	0.1125 (2)	0.040 (1)
C(22)	0.2385 (4)	0.1140 (3)	0.0514 (2)	0.069 (1)
C(23)	0.2809 (5)	0.1372 (3)	-0.0322 (2)	0.093 (2)
C(24)	0.3094 (4)	0.2543 (3)	-0.0563 (2)	0.076 (1)
C(25)	0.2991 (3)	0.3496 (3)	0.0027 (2)	0.062 (1)
C(26)	0.2594 (3)	0.3283 (2)	0.0873 (2)	0.049 (1)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

P(1)—O(1)	1.484 (2)	P(1)—C(1)	1.796 (2)
P(1)—C(11)	1.800 (2)	P(1)—C(21)	1.800 (3)
C(1)—C(2)	1.520 (4)	C(11)—C(16)	1.384 (3)
O(1)—P(1)—C(1)	114.1 (1)	O(1)—P(1)—C(11)	111.6 (1)
C(1)—P(1)—C(11)	107.5 (1)	O(1)—P(1)—C(21)	111.9 (1)
C(11)—P(1)—C(21)	105.2 (1)	C(1)—P(1)—C(21)	106.1 (1)
P(1)—C(1)—C(2)	113.0 (2)		

Data collection and cell refinement: Nicolet P3F diffractometer software. All other calculations and molecule graphics: *SHELXTL-Plus* (Sheldrick, 1990) program system. The strong reflections, 200, 020 and 002, were not used in the calculations due to an error in the intensity measurements.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, coordinates of equivalent atoms bonded to unique atoms and associated geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71627 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1090]

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## Diastereomeric Octahydronaphthoquinolizines

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**Abstract**

Crystal structures of three diastereoisomers of the 2,3,6,7,7a,8,12b,12c-octahydro-4*H*<sup>+</sup>,1*H*,5*H*-naphtho-[1,2,3-*ij*]quinolizinium cation,  $\text{C}_{16}\text{H}_{22}\text{N}^+$  (as the chloride dichloromethane solvate and as two perchlorate salts),  $\text{C}_{16}\text{H}_{22}\text{N}$ , are reported. The ring junctions *B/C*, *B/D* and *C/D* are *cis,cis,trans* (1), *trans,trans,trans* (2) and *cis,trans,cis* (3), respectively. In all three isomers, rings *C* and *D* of the quinolizine moiety adopt an almost perfect chair conformation while the fused ring *B* of the naphtho moiety adopts a half-chair conformation. These structural features are consistent with the presence of Bohlmann bands in the IR spectra of (1) and (2), but not (3).

**Comment**

In the course of preparation of new types of neuroreceptor ligands with high affinity to dopamine, serotonin and sigma receptor sites in the central nervous system (Largent, Wikström, Gundlach & Snyder, 1987), we have discovered a new tetracyclic ring system, the octahydronaphthoquinolizine compounds (OHNQ). These compounds have been synthesized in three steps from corresponding 2-tetralones (Cai, Pan, Dewan, Wink, Murphy & Schuster, 1993) and have been shown to have activity at selected neuroreceptor binding sites. Since