O(1)—C(1)—C(2)	114.9 (3)	C(1)—C(6)—C(5)	116.6 (3)
O(1)—C(1)—C(6)	122.9 (3)	C(1)—C(6)—C(7)	121.3 (3)
C(2)—C(1)—C(6)	122.1 (4)	C(5)—C(6)—C(7)	122.1 (3)
C(1)—C(2)—C(3)	119.4 (3)	C(4)—N(1)—N(2)	119.2 (3)
C(1)—C(2)—C(8)	119.1 (3)	N(1)—N(2)—C(9)	121.9 (3)
C(3)—C(2)—C(8)	121.5 (3)	N(2)—C(9)—C(10)	117.4 (3)
C(2)—C(3)—C(4)	118.9 (3)	N(2)—C(9)—C(14)	120.9 (3)
C(3)—C(4)—C(5)	120.7 (3)	C(10)—C(9)—C(14)	121.7 (4)
C(3)—C(4)—N(1)	125.0 (3)	C(9)—C(10)—C(11)	119.6 (3)
C(5)—C(4)—N(1)	114.2 (3)	C(10)—C(11)—C(12)	119.6 (3)
C(4)—C(5)—C(6)	122.2 (3)	C(11)—C(12)—C(13)	120.0 (4)
C(11)—C(12)—S	119.6 (3)	C(9)—C(14)—C(13)	118.1 (3)
C(13)—C(12)—S	120.4 (3)	C(12)—C(13)—C(14)	121.0 (3)
C(12)—S—O(2)	105.7 (2)	C(12)—S—O(3)	106.1 (2)
C(12)—S—O(4)	105.5 (2)	O(2)—S—O(3)	112.8 (2)
O(2)—S—O(4)	113.1 (2)	O(3)—S—O(4)	112.9 (2)

C(5)—C(4)—N(1)—N(2)	177.1 (3)
C(4)—N(1)—N(2)—C(9)	−177.9 (3)
N(1)—N(2)—C(9)—C(10)	170.8 (3)

D—H···A	D···A	D—H···A
O(1)—H(1)···O(5)	2.541 (4)	151 (3)
N(2)—H(20)···O(2^{i})	2.889 (4)	124 (3)
N(2)—H(20)···O(3^{ii})	2.877 (4)	124 (3)
O(5)—H(50)···O(2^{iii})	2.809 (4)	173 (4)
O(5)—H(51)···O(4^{iv})	2.778 (4)	161 (4)

Symmetry codes: (i) $\frac{1}{2}+x, \frac{1}{2}-y, z-\frac{1}{2}$; (ii) $x, y, z-1$; (iii) $\frac{1}{2}-x, \frac{1}{2}+y, -\frac{1}{2}-z$; (iv) $-x, -y, -1-z$.

Table 4. *Selected geometric parameters* (Å, °) *for* (II)

O(1)—C(1)	1.313 (3)	C(1)—C(2)	1.424 (3)
C(2)—C(3)	1.365 (3)	C(3)—C(4)	1.413 (2)
C(4)—C(5)	1.411 (3)	C(5)—C(6)	1.366 (3)
C(6)—C(7)	1.501 (3)	C(1)—C(6)	1.426 (3)
C(2)—C(8)	1.499 (3)	C(4)—N(1)	1.359 (3)
N(1)—N(2)	1.276 (2)	N(2)—C(9)	1.417 (3)
C(9)—C(10)	1.380 (3)	C(10)—C(11)	1.386 (3)
C(11)—C(12)	1.386 (2)	C(12)—C(13)	1.388 (3)
C(13)—C(14)	1.382 (3)	C(9)—C(14)	1.386 (3)
C(12)—S	1.773 (2)	S—O(2)	1.454 (1)
S—O(3)	1.447 (1)	S—O(4)	1.453 (2)
O(1)—C(1)—C(2)	123.3 (2)	C(1)—C(6)—C(5)	117.6 (2)
O(1)—C(1)—C(6)	115.0 (2)	C(1)—C(6)—C(7)	119.6 (2)
C(2)—C(1)—C(6)	121.7 (2)	C(5)—C(6)—C(7)	122.8 (2)
C(1)—C(2)—C(3)	118.4 (2)	C(4)—N(1)—N(2)	120.4 (2)
C(1)—C(2)—C(8)	120.8 (2)	N(1)—N(2)—C(9)	119.4 (2)
C(3)—C(2)—C(8)	120.8 (2)	N(2)—C(9)—C(10)	117.8 (2)
C(2)—C(3)—C(4)	121.1 (2)	N(2)—C(9)—C(14)	120.6 (2)
C(3)—C(4)—C(5)	119.2 (2)	C(10)—C(9)—C(14)	121.6 (2)
C(3)—C(4)—N(1)	126.9 (2)	C(9)—C(10)—C(11)	119.3 (2)
C(5)—C(4)—N(1)	113.9 (2)	C(10)—C(11)—C(12)	119.9 (2)
C(4)—C(5)—C(6)	121.8 (2)	C(11)—C(12)—C(13)	119.9 (2)
C(9)—C(14)—C(13)	118.4 (2)	C(11)—C(12)—S	120.8 (1)
C(13)—C(12)—S	119.3 (1)	C(12)—C(13)—C(14)	120.8 (2)
C(12)—S—O(2)	106.33 (8)	C(12)—S—O(3)	106.05 (9)
C(12)—S—O(4)	105.7 (1)	O(2)—S—O(3)	112.76 (9)
O(2)—S—O(4)	112.56 (9)	O(3)—S—O(4)	112.76 (9)

C(5)—C(4)—N(1)—N(2)	177.7 (3)
C(4)—N(1)—N(2)—C(9)	−177.9 (3)
N(1)—N(2)—C(9)—C10)	−170.0 (3)

D—H···A	D···A	D—H···A
O(1)—H(1)···O(5)	2.555 (4)	163 (3)
N(2)—H(20)···O(2^{i})	2.779 (3)	167 (3)
O(5)—H(50)···O(4^{ii})	2.787 (5)	168 (3)
O(5)—H(51)···O(3^{iii})	2.768 (2)	168 (3)

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

In both structures, H atoms bonded to C atoms were placed in calculated positions. H atoms bonded to N atoms and O atoms were located in difference maps.

Data reduction: *SDP* (B. A. Frenz & Associates Inc., 1982). Program(s) used to solve structures: *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993). Program used for geometric calculations: *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: PA1174). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

B. A. Frenz & Associates Inc. (1982). *SDP Structure Determination Package*. College Station, Texas, USA.

Ehlinger, N., Lecocq, S., Perrin, R. & Perrin, M. (1993). *Supramol. Chem.* **2**, 77–82.

Hanson, A. W. (1973). *Acta Cryst.* B**29**, 454–460.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Nomura, E., Taniguchi, H. & Otsuji, Y. (1993). *Bull. Chem. Soc. Jpn*, **66**, 3797–3801.

Nomura, E., Taniguchi, H. & Tamura, S. (1989). *Chem. Lett.* pp. 1125–1126.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

Shinkai, S., Araki, K., Shibata, J., Tsugawa, D. & Manabe, O. (1989). *Chem. Lett.* pp. 931–934.

Xu, B. & Swagen, T. M. (1993). *J. Am. Chem. Soc.* **115**, 1159–1160.

Yeh, M., Tang, F., Chen, S., Liu, W. & Lin, L. (1994). *J. Org. Chem.* **59**, 754–757.

Acta Cryst. (1995). C**51**, 1849–1851

(2-Methoxyphenyl)(phenyl)vinylphosphine Oxide

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(*Received* 17 *January* 1995; *accepted* 21 *February* 1995)

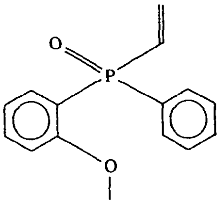
Abstract

In the molecule of the title compound, $C_{15}H_{15}O_2P$, the C=C—P=O fragment has an *S-cis* conformation. The methoxyphenyl ring assumes an almost coplanar arrangement with the P=O bond and is almost perpendicular to the phenyl ring.

Acta Crystallographica Section C
ISSN 0108-2701 ©1995

Comment

X=P—C=C (where X = O, S, Se) vinylphosphine derivatives are reactive in the *S-cis* conformation; this is particularly important with respect to the optically active forms of vinylphosphine oxides, where a transfer of chirality occurs from the P to the C atom.



(I)

In this paper we present the structure of (2-methoxyphenyl)(phenyl)vinylphosphine oxide, (I). The molecular structure with labelling of the non-H atoms is shown in Fig. 1. As expected, the O1—P—C1—C2 torsion angle of −5.3 (3)° indicates that the relative arrangement of the P=O and C=C groups in the molecule is *S-cis*. Similar conformations have been found in the molecules of other vinylphosphine oxides (Pietrusiewicz, Zabłocka, Kuźnikowski, Wieczorek, Maniukiewicz & Rospenk, 1991; Pietrusiewicz, Zabłocka, Wieczorek & Brandi, 1991; Pietrusiewicz & Wieczorek, 1993), vinylphosphine sulfides (Pietrusiewicz, Kuźnikowski, Wieczorek & Brandi, 1992; Pietrusiewicz, Wieczorek, Cicchi & Brandi, 1991) and a vinylphosphine selenide (Pietrusiewicz, Kuźnikowski, Wieczorek & Brandi, 1992).

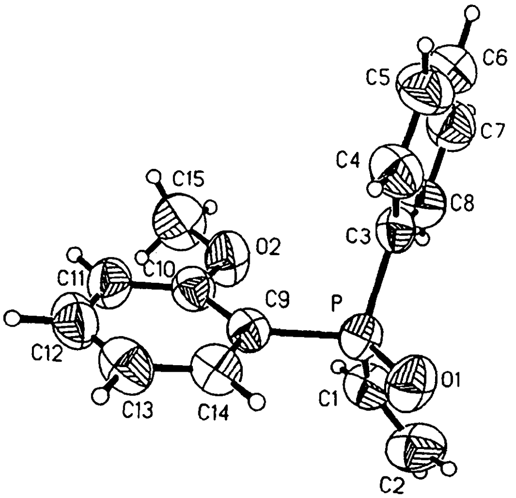


Fig. 1. Molecular structure of the title compound showing 50% probability displacement ellipsoids for the non-H atoms.

The tetrahedral geometry about the P atom is deformed in the usual way, showing increased O=P—C and decreased C—P—C angles, with values in the ranges 110.6 (1)–112.3 (1) and 106.4 (1)–107.6 (1)°, respectively. This deformation is smaller than in the molecules cited above.

The methoxyphenyl ring assumes an almost coplanar arrangement with the P=O bond, contrary to the arrangement of P=O and the phenyl ring. The angles between the P=O bond and these rings are 1.7 (1) and 52.0 (1)°, respectively, while in the molecule of (S_P)-L-menthyl(2-methoxyphenyl)phenylphosphinylacetate (Pietrusiewicz & Wieczorek, 1993) the corresponding angles ares 10.0 (1) and 12.1 (1)°, respectively.

The dihedral angle between the planes of the two aryl rings is 88.2 (1)°. The methoxy group lies practically in the plane of the aryl ring [C15—O2—C10—C11 = −0.7 (3)°].

Experimental

The title compound was prepared by the method of Maffei & Buono (1988). The crystal density D_m was measured by flotation in $CH_3I + C_6H_6$.

Crystal data

$C_{15}H_{15}O_2P$
$M_r = 258.26$
Monoclinic
$P2_1/n$
$a = 8.892$ (1) Å
$b = 8.614$ (1) Å
$c = 18.094$ (1) Å
$\beta = 102.62$ (1)°
$V = 1352.5$ (2) Å^3
$Z = 4$
$D_x = 1.268$ Mg m^{-3}
$D_m = 1.26$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 25 reflections
$\theta = 16.0$–25.0°
$\mu = 1.72$ mm^{-1}
$T = 293$ K
Prism
0.50 × 0.36 × 0.25 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3181 measured reflections
2566 independent reflections
2323 observed reflections [$F > 4\sigma(F)$]

$R_{\rm int} = 0.013$
$\theta_{\rm max} = 74.89°$
$h = -11 \rightarrow 0$
$k = -10 \rightarrow 0$
$l = -22 \rightarrow 22$
3 standard reflections frequency: 60 min intensity decay: 1.3%

Refinement

Refinement on F
$R = 0.040$
$wR = 0.046$
$S = 2.41$
2323 reflections
182 parameters
H atoms treated as riding atoms
$w = 1/[\sigma^2(F) + 0.00022F^2]$
$(\Delta/\sigma)_{\rm max} = 0.04$

$\Delta\rho_{\rm max} = 0.36$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.30$ e Å^{-3}
Extinction correction: $F^* = F_c[1.0 + (0.002\chi \times F_c^2/\sin\theta)]^{-1/4}$
Extinction coefficient: $\chi = 0.011$ (1)
Atomic scattering factors from *SHELXTL/PC* (Sheldrick, 1990)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
P	0.1668 (1)	0.1901 (1)	0.8535 (1)	0.049 (1)
O1	0.1001 (2)	0.0315 (2)	0.8493 (1)	0.068 (1)
O2	0.2808 (2)	0.5033 (2)	0.9112 (1)	0.071 (1)
C1	0.0662 (2)	0.3135 (3)	0.7799 (1)	0.063 (1)
C2	−0.0438 (3)	0.2553 (4)	0.7273 (2)	0.086 (1)
C3	0.3663 (2)	0.1883 (2)	0.8477 (1)	0.050 (1)
C4	0.4663 (3)	0.0983 (3)	0.9003 (1)	0.067 (1)
C5	0.6196 (3)	0.0875 (3)	0.8981 (2)	0.080 (1)
C6	0.6756 (3)	0.1648 (3)	0.8434 (2)	0.078 (1)
C7	0.5791 (3)	0.2531 (3)	0.7912 (2)	0.075 (1)
C8	0.4241 (3)	0.2669 (3)	0.7931 (1)	0.061 (1)
C9	0.1597 (2)	0.2803 (2)	0.9423 (1)	0.048 (1)
C10	0.2200 (2)	0.4275 (2)	0.9640 (1)	0.053 (1)
C11	0.2153 (3)	0.4863 (3)	1.0347 (1)	0.065 (1)
C12	0.1471 (3)	0.3995 (3)	1.0829 (1)	0.071 (1)
C13	0.0852 (3)	0.2558 (3)	1.0629 (1)	0.068 (1)
C14	0.0923 (2)	0.1969 (3)	0.9924 (1)	0.057 (1)
C15	0.3433 (4)	0.6549 (3)	0.9284 (2)	0.092 (1)

Table 2. *Selected geometric parameters* (Å, °)

P—O1	1.484 (2)	O2—C10	1.363 (3)
P—C1	1.783 (2)	O2—C15	1.427 (3)
P—C3	1.800 (2)	C1—C2	1.307 (3)
P—C9	1.799 (2)		
O1—P—C1	112.3 (1)	C1—P—C9	107.4 (1)
O1—P—C3	112.2 (1)	C3—P—C9	106.4 (1)
O1—P—C9	110.6 (1)	C10—O2—C15	118.7 (2)
C1—P—C3	107.6 (1)	P—C1—C2	119.3 (2)

Data collection, reduction and cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989). Structure solution, refinement, molecular graphics and preparation of material for publication: *SHELXTL/PC* (Sheldrick, 1990).

This research was supported by the Polish Scientific Research Council (KBN) under grant 2 0575 91 01. The author thanks M. Koprowski for the crystals and Professors M. Bukowska-Strzyżewska and K. M. Pietrusiewicz for helpful discussions.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: NA1158). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Maffei, M. & Buono, G. (1988). *New J. Chem.* **12**, 923–927.

Pietrusiewicz, K. M., Kuźnikowski, M., Wieczorek, W. & Brandi, A. (1992). *Heteroatom Chem.* **3**, 37–40.

Pietrusiewicz, K. M. & Wieczorek, W. (1993). *Phosphorus Sulfur Silicon*, **82**, 99–107.

Pietrusiewicz, K. M., Wieczorek, W., Cicchi, S. & Brandi, A. (1991). *Heteroatom Chem.* **2**, 661–664.

Pietrusiewicz, K. M., Zabłocka, M., Kuźnikowski, M., Wieczorek, W., Maniukiewicz, W. & Rospenk, M. (1991). *Heteroatom Chem.* **2**, 111–122.

Pietrusiewicz, K. M., Zabłocka, M., Wieczorek, W. & Brandi, A. (1991). *Tetrahedron Asymmetry*, **2**, 419–428.

Sheldrick, G. M. (1990). *SHELXTL/PC User's Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1995). C**51**, 1851–1853

Diphenyl(2-oxocyclohexyl)phosphine Sulfide

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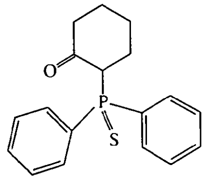
(*Received* 1 *November* 1994; *accepted* 1 *February* 1995)

Abstract

The structure of $(C_6H_5)_2(C_6H_9O)PS$ is reported. The phenyl groups are not magnetically equivalent and P═S and C═O are not coplanar.

Comment

We have reported recently a general route leading to the formation of the title compound, (I) (Barkallah, Ben Akacha, Boukraa, Zantour & Baccar, 1993).



(I)

^{13}C and ^1H NMR data show that the phenyl groups linked to the P atom are not magnetically equivalent. X-ray diffraction data confirms this. Indeed, the torsion angles C2′—C1′—P—S and C2″—C1″—P—S are dif-

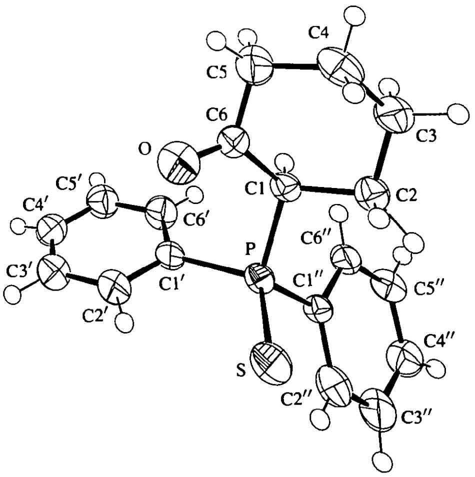


Fig. 1. Molecular structure of the title compound showing 50% probability displacement ellipsoids.