Acta Crystallographica Section E
**Structure Reports
Online**

ISSN 1600-5368

A monoclinic polymorph of *α*-diphenylphosphoryl-*o*-cresol

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Received 13 June 2005
Accepted 7 July 2005
Online 16 July 2005

Key indicators

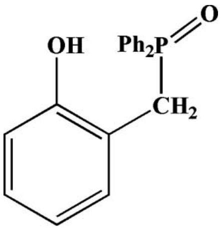
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.119
Data-to-parameter ratio = 16.9

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

When attempting the synthesis of the copper complex of the title compound {systematic name: 2-[(diphenylphosphoryl)-methyl]phenol}, $C_{19}H_{17}O_2P$, a new monoclinic polymorphic modification of the ligand was obtained instead of the complex. The variations of molecular geometry and supramolecular organization in the crystal structures of the two polymorphs are discussed.

Comment

α-Diphenylphosphoryl-*o*-cresol, (I), is a useful substrate to obtain phosphorylated podands (*i.e.* a ligand usually of chelate type, containing a number of ether bonds) (Evreinov *et al.*, 1989). We suggested that this compound potentially may act as a bidentate ligand, which should be able to form chelate complexes. However, it did not form a complex with copper(II) acetate in ethanol solution when using a 2:1 substrate–metal ratio. The starting substrate precipitated from the reaction mixture by slow evaporation to give a new monoclinic polymorphic modification, (I), characterized by different crystal packing according to our X-ray investigation, and a lower melting point than that reported for the known orthorhombic phase (Tkachev & Atovmyan, 1993).



(I)

A comparison of the molecular geometries reveals that in the two polymorphs the bond lengths are approximately equal. The main differences are observed for the orientations of aromatic rings. Thus, in the orthorhombic modification, the mutual arrangement of the phenyl rings with respect to the P═O bond is the same, with C—C—P═O torsion angles of 154.3 (5) and 160.2 (4)°. In the present monoclinic polymorph (Fig. 1), the relative orientations of one of the phenyl rings and the P═O group can be considered as eclipsed, as characterized by the torsion angle C9—C8—P1═O1 of 74.65 (14)°. The arrangement of the *o*-cresol moiety in both polymorphs is also different. The corresponding C—C—P═O torsion angles are 47.5 (4) and 17.83 (13)° for the orthorhombic and monoclinic modifications, respectively.

Consequently, the different conformations for these polymorphs determine their supramolecular organization. In the orthorhombic modification, molecules are assembled into

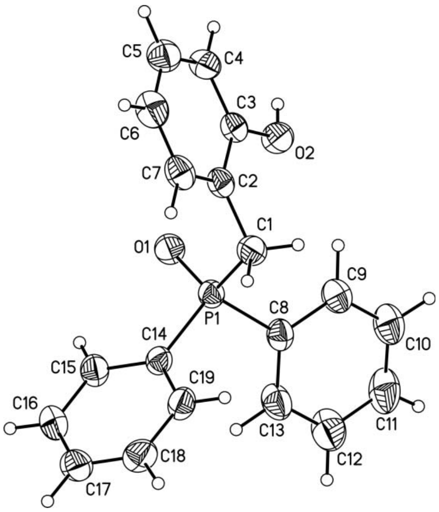


Figure 1
A view of the title compound showing the atom labeling and displacement ellipsoids at the 50% probability level.

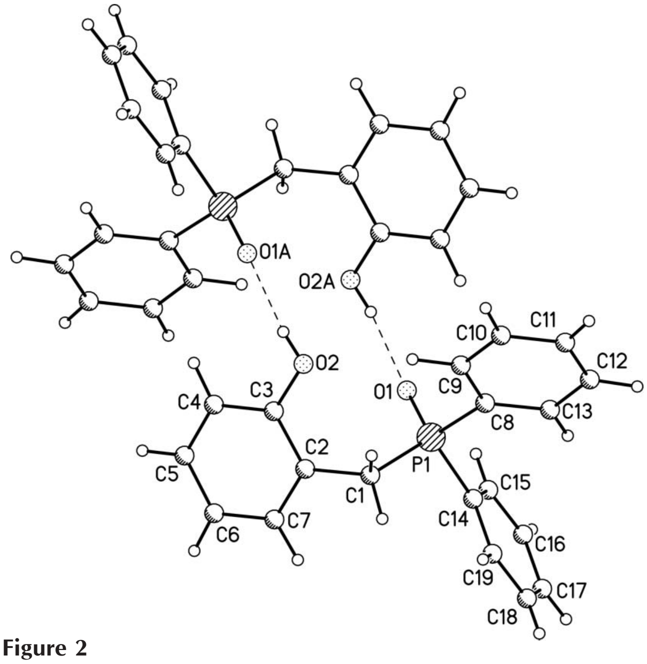


Figure 2
A view of the centrosymmetric dimer in the crystal structure of (I). Dashed lines indicate hydrogen bonding interactions. [Symmetry code: (A) $1 - x, 1 - y, -z$.]

infinite hydrogen-bonded chains, while in (I), centrosymmetric dimers are observed (Fig. 2 and Table 2). Surprisingly, the lengths of intermolecular O2—H2O···O1 separations are very close in both phases, *viz*. O···O = 2.634 (3) Å in the orthorhombic form and 2.6513 (16) Å in (I). Thus, the character of the O2—H2O···O1═P1 system is determined by the nature of the hydrogen-bonded O—H···O═P system rather than by crystal packing.

Experimental

α-Diphenylphosphoryl-*o*-cresol was obtained by the Arbusov rearrangement from 2-(hydroxymethyl)phenol and ethyl diphenylphosphinite, according to a known procedure (Bondarenko *et al.*, 1979) (m.p. 451–452 K). To a solution of α-diphenylphosphoryl-*o*-cresol in ethanol, copper(II) acetate monohydrate was added in one portion and the mixture was stirred overnight. A pale-blue solution was then decanted from the precipitated salt and allowed to evaporate slowly to afford crystals of (I) in the new monoclinic form (m.p. 429 K).

Crystal data

$C_{19}H_{17}O_2P$
M_r = 308.30
Monoclinic, $P2_1/n$
a = 10.092 (2) Å
b = 10.521 (2) Å
c = 14.699 (3) Å
β = 90.43 (3)°
V = 1560.7 (5) Å^3
Z = 4
D_x = 1.312 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 48 reflections
θ = 4.2–12.2°
μ = 0.18 mm^{-1}
T = 298 (2) K
Prism, colorless
0.35 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4817 measured reflections
4519 independent reflections
3352 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.033
$\theta_{\max}$ = 30.0°
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 14$
$l = -20 \rightarrow 20$
2 standard reflections every 98 reflections intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.119
S = 1.00
4519 reflections
267 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2 + 0.2609P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.30 e Å^{-3}
$\Delta\rho_{\min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P1—O1	1.4925 (11)	P1—C8	1.8035 (15)
P1—C14	1.8031 (14)	P1—C1	1.8298 (16)
O1—P1—C14	109.99 (6)	O1—P1—C1	114.68 (7)
O1—P1—C8	112.60 (7)		
O1—P1—C1—C2	17.83 (13)	O1—P1—C8—C9	74.65 (14)
O1—P1—C14—C15	12.19 (14)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2O···O1$^{\rm i}$	0.81 (2)	1.84 (3)	2.6513 (16)	172 (2)

Symmetry codes: (i) $-x + 1, -y + 1, -z$.

All H atoms were located in difference Fourier maps and were refined freely [C—H = 0.89 (3)–1.04 (3) Å].

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *PROFIT* (Strel'tsov & Zavodnik, 1989); program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1994); program(s) used to refine structure: *SHELXTL-Plus*; molecular graphics: *SHELXTL-Plus*; software used to prepare material for publication: *SHELXTL-Plus*.

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This work was supported by the Russian Foundation for Basic Research (project Nos. 03–03-32716 and 05–03-32692).

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