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# 2,2,2-Tris(cyclohexyloxy)-4,5-(2′,2″-biphenylo)-1,3,2-dioxaphospholene

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**Key indicators**

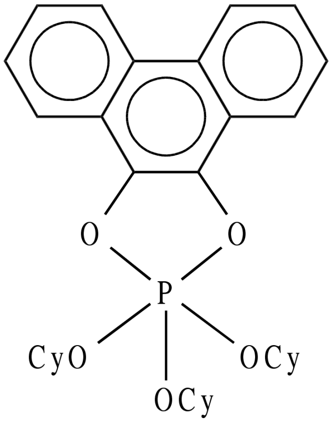
Single-crystal X-ray study
$T$ = 143 K
Mean $\sigma$(C–C) = 0.004 Å
Disorder in main residue
$R$ factor = 0.048
$wR$ factor = 0.119
Data-to-parameter ratio = 13.7

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

The title compound (2,2,2-tris(cyclohexyloxy)phenanthro-[9,10-d][1,3,2]dioxaphosphole), $C_{32}H_{41}O_5P$, displays trigonal–bipyramidal geometry at the central P atom. The P—O bond lengths (Å) are: in the five-membered ring 1.652 (2) Å equatorial and 1.744 (2) Å axial, and to the cyclohexyloxy groups 1.581 (2) and 1.589 (2) Å equatorial, and 1.624 (2) Å axial.

## Comment

We are interested in the correlation between structure and reactivity (see *e.g.* Amos *et al.*, 1992). The structure of the title compound, (I), the adduct of tricyclohexyl phosphite and phenanthraquinone, was determined as part of a combined crystallographic and theoretical investigation of pKa values of pentaoxyphosphoranes (Davies *et al.*, 2002).



(I)

The structure is shown in Fig. 1; a minor (11%) disorder component of the ring C11–16 is not shown. Of the three cyclohexyloxy groups, one is axial (O5, C31 *etc.*) and the other two are equatorial. This is reflected in the normal bond-length pattern for trigonal–bipyramidal geometry (axial bonds longer than equatorial), as was also observed in pentakis(cyclohexyloxy)phosphorane (Davies *et al.*, 2001; average values in four independent molecules 1.662 Å for axial and 1.606 Å for equatorial P—O bonds). The triisopropyloxy analogue of the title compound was subjected to structure analysis in 1967. There were two modifications, of which the more precisely determined modification (Spratley *et al.*, 1967) displayed a similar P—O bond-length pattern to the title compound: ring apical 1.751 (7) Å, equatorial 1.633 (7) Å; $^i$Pr apical 1.638 (6) Å, and equatorial 1.588 (7) and 1.574 (7) Å.

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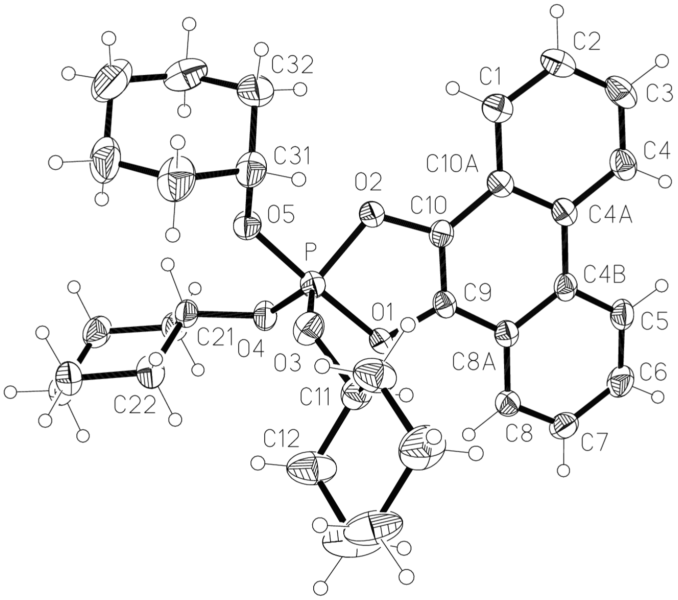

**Figure 1**
The molecule of the title compound in the crystal. The minor disorder component of the ring C11–C16 has been omitted. Ellipsoids represent 50% probability levels.

## Experimental

Compound (I) was prepared by adding triisopropyl phosphite to one equivalent of phenanthraquinone at room temperature, according to the method of Hamilton *et al.* (1967).

*Crystal data*

$C_{32}H_{41}O_5P$
$M_r = 536.62$
Monoclinic, $P2_1/n$
$a = 9.665$ (2) Å
$b = 15.394$ (3) Å
$c = 19.200$ (4) Å
$\beta = 93.92$ (2)°
$V = 2850.1$ (10) Å$^3$
$Z = 4$

$D_x = 1.251$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 50 reflections
$\theta = 10$–11.5°
$\mu = 0.14$ mm$^{-1}$
$T = 143$ (2) K
Prism, colourless
0.65 × 0.30 × 0.30 mm

*Data collection*

Stoe Stadi-4 diffractometer
$\omega/\theta$ scans
Absorption correction: none
5588 measured reflections
5037 independent reflections
3708 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$

$\theta_{\rm max} = 25.0°$
$h = -11 \rightarrow 0$
$k = -18 \rightarrow 1$
$l = -22 \rightarrow 22$
3 standard reflections
frequency: 60 min
intensity decay: none

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.119$
$S = 1.04$
5037 reflections
368 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 1.4014P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.21$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.31$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| P—O3 | 1.581 (2) | P—O2 | 1.652 (2) |
| P—O4 | 1.589 (2) | P—O1 | 1.744 (2) |
| P—O5 | 1.624 (2) | | |
| O3—P—O4 | 117.35 (9) | O5—P—O2 | 88.52 (8) |
| O3—P—O5 | 92.73 (9) | O3—P—O1 | 94.57 (8) |
| O4—P—O5 | 91.98 (8) | O4—P—O1 | 84.38 (8) |
| O3—P—O2 | 115.31 (9) | O5—P—O1 | 172.69 (9) |
| O4—P—O2 | 127.24 (9) | O2—P—O1 | 88.66 (8) |

H atoms were positioned geometrically and refined using a riding model. Cyclohexyl ring C11–16 is disordered over two positions with a relative occupancy of *ca* 9:1. The minor component was refined isotropically with an appropriate system of restraints.

Data collection: *DIF*4 (Stoe & Cie, 1992); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1992); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

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