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4,5-Bis[bis(diethylamino)phosphino]-9,9-dimethylxanthene

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.139
Data-to-parameter ratio = 16.1

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

The structure of the title compound, $C_{31}H_{52}N_4OP_2$, reveals a near-planar xanthene skeleton.

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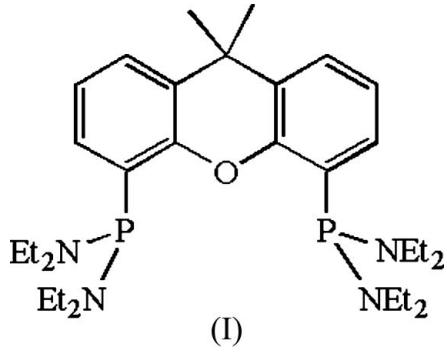
Comment

Xantphos, and related bidentate ligands with the xanthene backbone, have been shown to be remarkable ligands for the rhodium-catalysed hydroformylation of long-chain alkenes, giving exceptionally high selectivity to the industrially useful linear aldehyde (linear/branched ratio = 50:1) (Kranenburg *et al.*, 1995). The title compound, (I), was prepared according to the literature procedure of Goertz *et al.* (2001) as an intermediate in work directed towards the synthesis of perfluoroalkylated derivatives of Xantphos (Adams *et al.*, 2004). Crystals suitable for X-ray analysis were grown from hexane solution. The bond lengths and angles within the structure are unremarkable. The molecule has a pseudo-*C*2 axis of symmetry through O1 and C6, such that the lone pairs on phosphorus point either in front or behind an approximately planar xanthene skeleton.



(I)

Experimental

The title compound was prepared according to the literature procedure of Goertz *et al.* (2001), Crystals suitable for X-ray analysis were grown from a hexane solution.

Crystal data

$C_{31}H_{52}N_4OP_2$
M_r = 558.71
Monoclinic, $P2_1/c$
a = 18.799 (1) Å
b = 11.4968 (6) Å
c = 15.2179 (8) Å
β = 100.880 (1)°
V = 3229.9 (3) Å^3
Z = 4
D_x = 1.149 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6949 reflections
θ = 2.2–26.9°
μ = 0.16 mm^{-1}
T = 150 (2) K
Block, colourless
0.34 × 0.21 × 0.16 mm

Data collection

Bruker APEX CCD area-detector diffractometer	5685 independent reflections
φ and ω scans	4653 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.034$
$T_{min} = 0.93$, $T_{max} = 0.96$	$\theta_{max} = 25.0^\circ$
22958 measured reflections	$h = -22 \rightarrow 22$
	$k = -13 \rightarrow 13$
	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0817P)^2 + 0.3182P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.050$	$(\Delta/\sigma)_{max} = 0.002$
$wR(F^2) = 0.139$	$\Delta\rho_{max} = 0.53$ e Å^{-3}
$S = 1.06$	$\Delta\rho_{min} = -0.35$ e Å^{-3}
5685 reflections	
353 parameters	
H-atom parameters constrained	

All H atoms were included in calculated positions, riding on the bonded atom [C—H = 0.95 (CH), 0.98 (CH_3) and 0.99 Å (CH_2)], and with U_{iso}(H) values set at $1.5U_{eq}$ of the bonded C atom for methyl H atoms and at $1.2U_{eq}$ for all other H atoms.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

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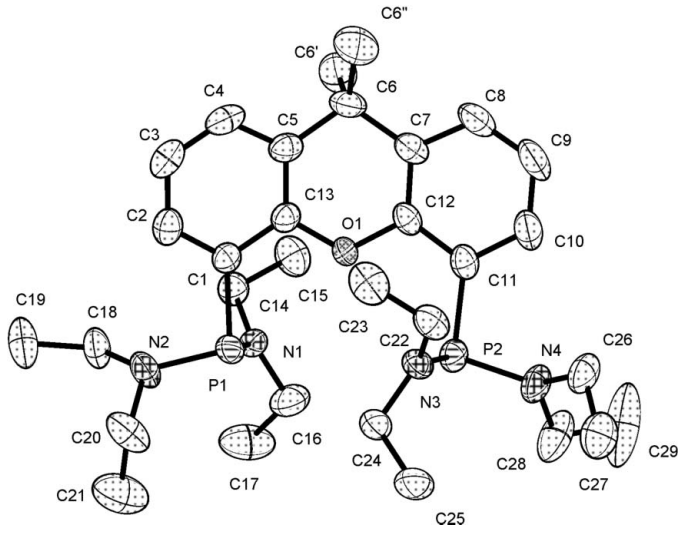

Figure 1
The molecular structure of (I), showing the atom-labelling scheme and 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

References

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