Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl 3-(diphenylphosphinoyl)-3-phenylpropionate

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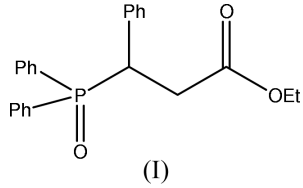
Received 30 July 2002
Accepted 9 August 2002
Online 16 August 2002

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.006 Å
R factor = 0.055
wR factor = 0.110
Data-to-parameter ratio = 13.8

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

The title compound, $Ph_2P(O)CH(Ph)CH_2COOEt$ or $C_{23}H_{23}O_3P$, (I), assembles into columns extending down the *a* axis, *via* C—H···O hydrogen bonds. Compound (I) was isolated as a by-product in studies related to the development of a novel cyclization reaction [Evans *et al.* (2002). *Tetrahedron Lett.* **43**, 299–301] and was synthesized independently to confirm its structure.



(I)

Experimental

The title compound was prepared in 70% yield by the reaction of ethyl propiolate with triphenylphosphine under reflux in a tetrahydrofuran–water mixture; this is similar in nature to the method reported previously by Richards & Tebby (1971).

Crystal data

$C_{23}H_{23}O_3P$
M_r = 378.38
Orthorhombic, $P2_12_12_1$
a = 5.788 (5) Å
b = 17.499 (5) Å
c = 19.463 (5) Å
V = 1971.3 (19) Å^3
Z = 4
D_x = 1.275 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3383 reflections
θ = 3.1–25.0°
μ = 0.16 mm^{-1}
T = 120 (2) K
Rod, colourless
0.15 × 0.04 × 0.03 mm

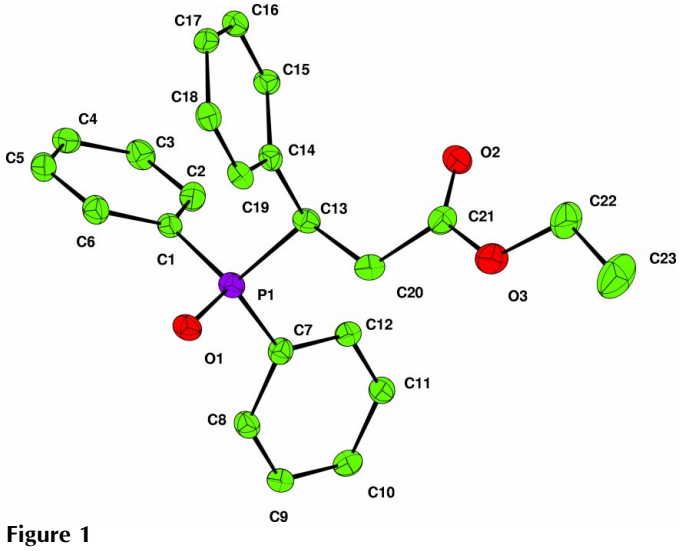


Figure 1
View of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level. H atoms have been omitted for clarity.

Data collection

Bruker–Nonius Kappa CCD area-detector diffractometer	$R_{int} = 0.096$
φ and ω scans	$\theta_{max} = 25.0^\circ$
10 345 measured reflections	$h = -6 \rightarrow 6$
3383 independent reflections	$k = -19 \rightarrow 20$
2169 reflections with $I > 2\sigma(I)$	$l = -22 \rightarrow 23$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.055$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
$wR(F^2) = 0.110$	$\Delta\rho_{min} = -0.22$ e Å^{-3}
$S = 0.98$	Extinction correction: *SHELXL*97
3383 reflections	Extinction coefficient: 0.0034 (9)
246 parameters	Absolute structure: Flack (1983)
H-atom parameters constrained	Flack parameter = 0.66 (17)
$w = 1/[\sigma^2(F_o^2) + (0.0301P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$	

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···O1^{i}	1.00	2.60	3.588 (5)	171
C12—H12···O1^{i}	0.95	2.49	3.366 (5)	154
C2—H2···O1^{i}	0.95	2.68	3.463 (5)	140
C19—H19···O2^{ii}	0.95	2.47	3.385 (6)	163

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

Despite the measurement of 67% of Friedel equivalents and the presence of phosphorus in the crystal structure the refined value of the Flack (1983) parameter did not allow the determination of the absolute configuration of the title compound in the crystal. 1387 Friedels were used in the refinement]

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*, *COLLECT* and *MAXUS* (Mackay *et al.*, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1998).

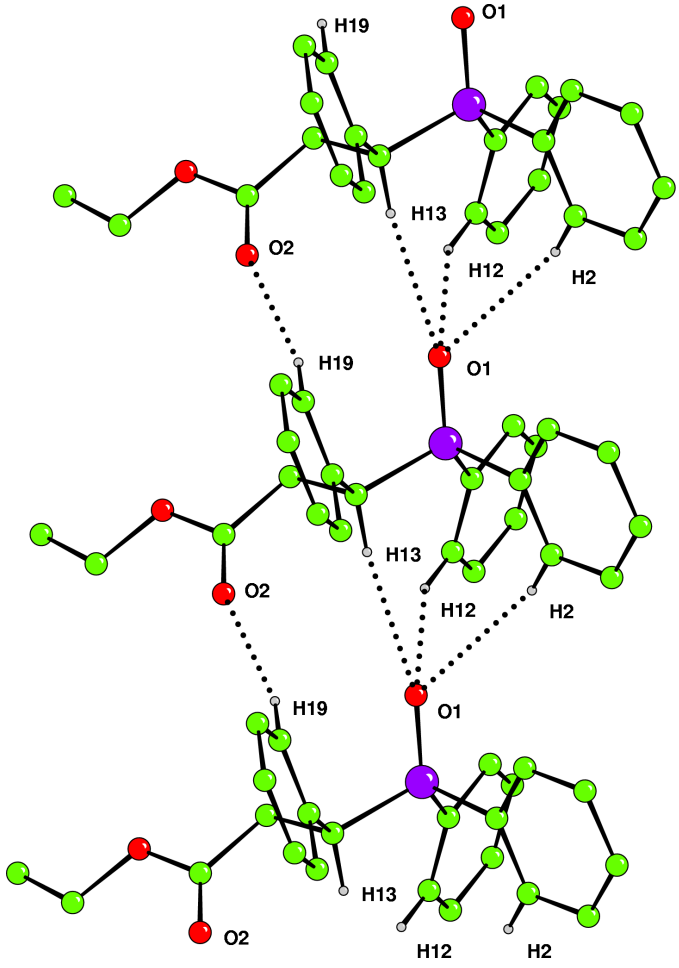


Figure 2
The C—H···O hydrogen-bonded column extending down the *a* axis. H atoms not involved in C—H···O bonds have been omitted.

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