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Bis(dimethylphosphorylmethyl)durene

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The structure of the title compound [systematic name: tetramethyl (2,3,5,6-tetramethyl-*p*-phenylenedimethylene)bisphosphonate], $C_{16}H_{28}O_6P_2$, was determined by single-crystal X-ray diffraction at 100 K. The conformer present in the crystal structure lies on an inversion center.

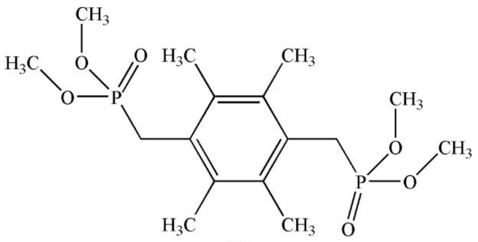
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.110
Data-to-parameter ratio = 20.0

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

Comment

Compounds with functional groups such as a phosphorylmethylene unit are versatile starting materials not only for the synthesis of a wide range of phosphines, but they may also be converted into phosphaalkenes or alkynes (Grandin *et al.*, 1992; Guillemin *et al.*, 2001). For this purpose, bis(dimethylphosphorylmethylene)durene, (I), was synthesized by the reaction of trimethyl phosphite with bis(chloromethyl)durene using a variation of the Michaelis–Arbuzov reaction.



(I)

The compound crystallizes in the space group $P2_1/c$ with Z = 2. The molecule is located on an inversion center (Fig. 1). All bond distances and angles within the molecule itself are in the range expected for this type of compound (Allen *et al.*, 1995).

A number of weak non-classical C—H$\cdots$O hydrogen bonds are observed between the methylene and the methoxy CH_3 groups on the one side and O atoms of neighboring molecules on the other (Table 1), but all of these distances are greater than the sum of the van der Waals radii for oxygen and carbon of 3.22 Å (Vainshtein *et al.* 1982). Of these, the shortest intermolecular contact is between atom O1 of the P=O double bond and one of the O—CH_3 H atoms, with a C7$\cdots$O1^{iii} distance of 3.272 (2) Å and O1$\cdots$H7A^{i} = 2.31Å [symmetry code: (iii) 1 + x, y, z].

Experimental

Bis(chloromethyl)durene (1 g, 4.31 mmol) was dissolved in trimethyl phosphite (100 ml) and the mixture was heated to reflux for 24 h. The excess trimethyl phosphite was removed in an oil pump vacuum and the white residue was recrystallized from acetone to yield (I) (1.54 g, 4.07 mmol, 95%) as colorless crystals, some of which were suitable for single crstal X-ray investigation. ^{1}H NMR (399.886 MHz, $\mathrm{CDC}L_3$): δ

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3.6345 [*d*, $^3J(^1H,^{31}P)$ = 10.8 Hz, 12H, OCH_3], 3.366 [*d*, $^2J(^1H,^{31}P)$ = 20.8 Hz, 4H, CH_2], 2.325 (*s*, 12H, CH_3).

Crystal data

$C_{16}H_{28}O_6P_2$
M_r = 378.32
Monoclinic, $P2_1/c$
a = 5.8561 (4) Å
b = 19.6347 (14) Å
c = 8.2554 (6) Å
β = 105.792 (1)°
V = 913.40 (11) Å^3

Z = 2
D_x = 1.376 Mg m^{-3}
Mo *Kα* radiation
μ = 0.27 mm^{-1}
T = 100 (2) K
Plate, colorless
0.42 × 0.26 × 0.05 mm

Data collection

Bruker SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 2003)
$T_{\min}$ = 0.81, $T_{\max}$ = 0.99

8742 measured reflections
2258 independent reflections
1910 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.031
$\theta_{\max}$ = 28.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.110
S = 1.07
2258 reflections
113 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.5709P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.53 e Å^{-3}
$\Delta\rho_{\min}$ = −0.31 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C7—H7*C*⋯O3	0.98	2.59	2.964 (2)	103
C7—H7*B*⋯O1$^{\rm i}$	0.98	2.54	3.386 (2)	145
C6—H6*B*⋯O2$^{\rm ii}$	0.99	2.53	3.436 (2)	153
C6—H6*A*⋯O1$^{\rm iii}$	0.99	2.60	3.566 (2)	167
C7—H7*A*⋯O1$^{\rm iii}$	0.98	2.31	3.272 (2)	167

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

All H atoms were positioned geometrically, with C—H = 0.98 Å for methyl and 0.99 Å for methylene H atoms. They were refined with an isotropic displacement parameter of 1.2 (methylene) or 1.5 times (methyl) that of the parent C atom. The methyl groups were allowed to rotate to best fit the experimental electron density. The s.u. values of the cell parameters are taken from the software, recognizing that the values are unreasonably small (Herbstein, 2000).

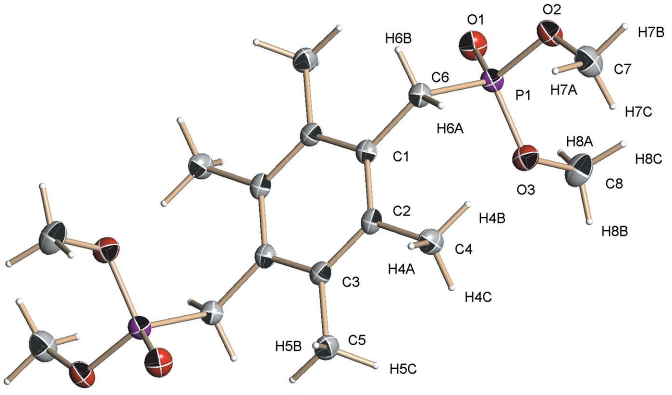

Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids. Unlabelled atoms are related to labeled atoms by $1 - x, 1 - y, 1 - z$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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