

## Dimethyl (2-hydroxy-4-nitrobenzyl)phosphonate

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## Key indicators

Single-crystal X-ray study

T = 294 K

Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$ 

R factor = 0.040

wR factor = 0.152

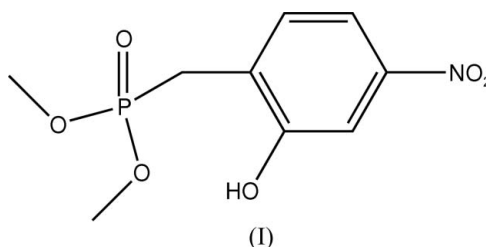
Data-to-parameter ratio = 15.3

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

In the title phosphonate derivative,  $\text{C}_9\text{H}_{12}\text{NO}_6\text{P}$ , intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds link the molecules in zigzag chains along the  $b$  axis.

## Comment

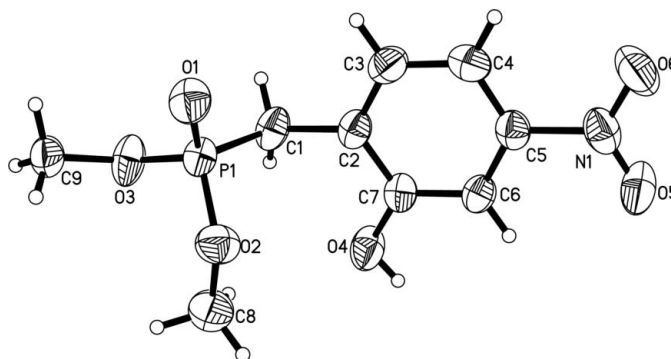
Phosphonate esters are important intermediates in the preparation of flame-retardant materials (Acher & Wakselman, 1982). As part of our work in this area, the title compound, (I), was prepared by the reaction of 1-bromo-2-hydroxy-4-nitrobenzene and trimethyl phosphite (Grawe *et al.*, 2002) and its structure is reported here (Fig. 1 and Table 1).



The benzene ring and its nitro substituent are essentially coplanar [interplanar angle =  $3.4(2)^\circ$ ], while the  $\text{O1}-\text{P1}-\text{O2}-\text{C8}$  and  $\text{C1}-\text{P1}-\text{O3}-\text{C9}$  torsion angles are  $164.7(2)$  and  $-159.2(2)^\circ$ , respectively, indicating that the atoms in these residues are also almost coplanar. In the crystal structure,  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds link the molecules into zigzag chains along the  $b$  axis (Fig. 2).

## Experimental

The title compound was prepared by the method of Grawe *et al.* (2002). Single crystals suitable for crystallographic analysis were



**Figure 1**  
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

obtained by slow evaporation of a tetrahydrofuran solution [m.p. 441 (2) K]. IR (KBr,  $\nu$ ,  $\text{cm}^{-1}$ ): 1502 (CN), 1195 (PO).  $^1\text{H}$  NMR (DMSO):  $\delta$  7.64–7.61 (*m*, 2H), 7.41–7.39 (*m*, 1H), 3.61 (*s*, 3H), 3.58 (*s*, 3H), 3.31 (*s*, 1H), 3.27 (*s*, 1H).

#### Crystal data

$\text{C}_9\text{H}_{12}\text{NO}_6\text{P}$	$D_x = 1.488 \text{ Mg m}^{-3}$
$M_r = 261.17$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2199 reflections
$a = 7.8154 (15) \text{ \AA}$	$\theta = 2.4\text{--}26.0^\circ$
$b = 12.963 (3) \text{ \AA}$	$\mu = 0.25 \text{ mm}^{-1}$
$c = 11.669 (2) \text{ \AA}$	$T = 294 (2) \text{ K}$
$\beta = 99.660 (3)^\circ$	Block, yellow
$V = 1165.4 (4) \text{ \AA}^3$	$0.30 \times 0.24 \times 0.12 \text{ mm}$
$Z = 4$	

#### Data collection

Bruker SMART CCD area-detector diffractometer	2408 independent reflections
$\varphi$ and $\omega$ scans	1671 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.036$
$T_{\text{min}} = 0.915$ , $T_{\text{max}} = 0.972$	$\theta_{\text{max}} = 26.6^\circ$
6476 measured reflections	$h = -9 \rightarrow 8$
	$k = -16 \rightarrow 15$
	$l = -12 \rightarrow 14$

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.1314P)^2 + 1.0878P]$
$R[F^2 > 2\sigma(F^2)] = 0.040$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.152$	$(\Delta/\sigma)_{\text{max}} = 0.002$
$S = 0.71$	$\Delta\rho_{\text{max}} = 0.19 \text{ e \AA}^{-3}$
2408 reflections	$\Delta\rho_{\text{min}} = -0.38 \text{ e \AA}^{-3}$
157 parameters	
H-atom parameters constrained	

**Table 1**

Selected geometric parameters ( $^\circ$ ).

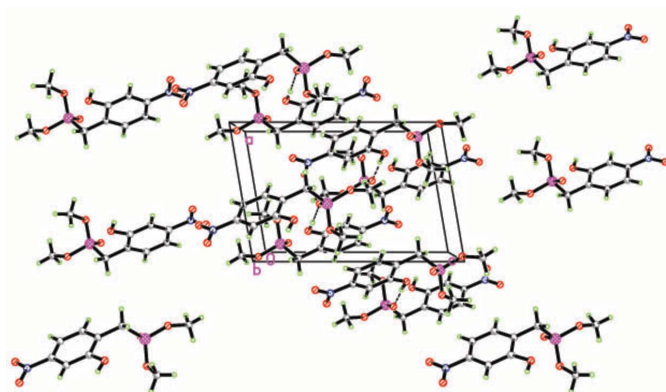
C8—O2—P1	123.1 (2)	C9—O3—P1	121.49 (18)
O1—P1—O2—C8	164.7 (2)	C1—P1—O3—C9	−159.2 (2)

**Table 2**

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O4—H4 $\cdots$ O1 $^i$	0.82	1.81	2.625 (3)	175

Symmetry code: (i)  $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$ .



**Figure 2**

Packing of (I), with hydrogen bonds drawn as dashed lines.

All H atoms were refined using a riding model, with C—H = 0.93  $\text{\AA}$  and  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$  for aromatic H atoms, C—H = 0.97  $\text{\AA}$  and  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$  for  $\text{CH}_2$  H atoms, C—H = 0.96  $\text{\AA}$  and  $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{C})$  for  $\text{CH}_3$  H atoms, and O—H = 0.82  $\text{\AA}$  and  $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{C})$  for OH H atoms.

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

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#### References

- Acher, F. & Wakselman, M. (1982). *Bull. Chem. Soc. Jpn.*, **55**, 3675–3676.  
 Bruker (1997). *SMART*, *SAINTE* and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.  
 Grawe, T., Schrader, T., Zadnarm, R. & Kraft, A. (2002). *J. Org. Chem.* **67**, 3755–3763.  
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.  
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.