

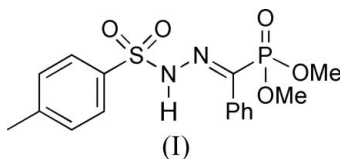
Dimethoxyphosphinoyl phenyl ketone
p-tolylsulfonylhydrazoneDustin Wayne Demoin, Michael
Pluth, Han Sen Soo and Yue Xu*Department of Chemistry, University of Cali-
fornia, Berkeley, CA 94720, USA

Correspondence e-mail: yuexu@berkeley.edu

Key indicators

Single-crystal X-ray study
 $T = 166$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.040
 wR factor = 0.094
Data-to-parameter ratio = 12.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title compound, $\text{C}_{16}\text{H}_{19}\text{N}_2\text{O}_5\text{PS}$, is the first phosphonate sulfonylhydrazone to be structurally characterized. The structure exhibits intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonding.Received 21 June 2006
Accepted 2 July 2006

Comment

The title compound, (I), was synthesized as a carbene precursor for the preparation of an allyl enamine (Gilbert *et al.*, 1984). Interestingly, no other crystal structures of phosphonate sulfonylhydrazones have been archived in the Cambridge Structural Database (Allen, 2002).The X-ray diffraction study of (I) shows that the *E* isomer crystallizes from methanol (Fig. 1). The bond lengths and angles of (I) are unexceptional and the molecule does not display intramolecular hydrogen bonding. The crystal packing of (I) (Fig. 2) exhibits intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds between N1 of a one molecule and O5 of a neighboring molecule related by an *n*-glide (Table 1).

Experimental

Compound (I) was prepared by condensation of *p*-tolylsulfonylhydrazine with one equivalent of dimethyl benzoylphosphonate in the presence of 0.5 equivalents of HCl, according to the procedure described by Seyferth *et al.* (1971). Single crystals were obtained by recrystallization from methanol.

Crystal data

$\text{C}_{16}\text{H}_{19}\text{N}_2\text{O}_5\text{PS}$	$Z = 2$
$M_r = 382.36$	$D_x = 1.422$ Mg m ⁻³
Monoclinic, <i>Pn</i>	Mo $K\alpha$ radiation
$a = 8.4692$ (19) Å	$\mu = 0.30$ mm ⁻¹
$b = 10.889$ (3) Å	$T = 166$ (1) K
$c = 9.690$ (2) Å	Plate, colorless
$\beta = 92.514$ (4)°	$0.16 \times 0.15 \times 0.05$ mm
$V = 892.8$ (4) Å ³	

Data collection

Bruker APEX CCD diffractometer	4987 measured reflections
ω scans	2788 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	2383 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.753$, $T_{\max} = 0.985$	$R_{\text{int}} = 0.034$
	$\theta_{\text{max}} = 26.4^\circ$

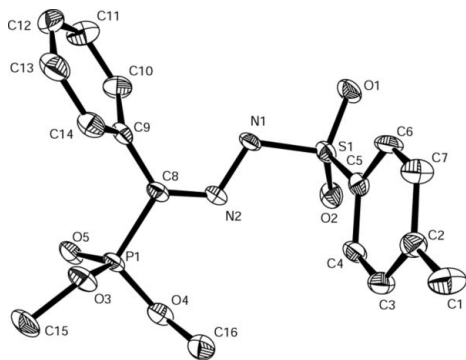


Figure 1
The molecular structure of (I), showing the atom-labeling scheme and displacement ellipsoids drawn at the 50% probability level. H atoms are omitted.

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.094$
 $S = 1.04$
 2788 reflections
 232 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.014$
 $\Delta\rho_{\max} = 0.36 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.29 \text{ e } \text{Å}^{-3}$
 Absolute structure: Flack (1983),
 971 Friedel pairs
 Flack parameter: $-0.05 (9)$

Table 1

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N1-H1 \cdots O5^i$	0.71 (4)	2.11 (4)	2.797 (4)	162 (4)

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

Atom H1 of the NH group was located in a difference Fourier map and refined isotropically. H atoms bound to C atoms were placed in idealized positions and allowed to ride during subsequent refinement, with $C-H = 0.93 \text{ Å}$ and $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$ for Csp^2 , and $C-H = 0.96 \text{ Å}$ and $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(C)$ for the methyl groups.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve

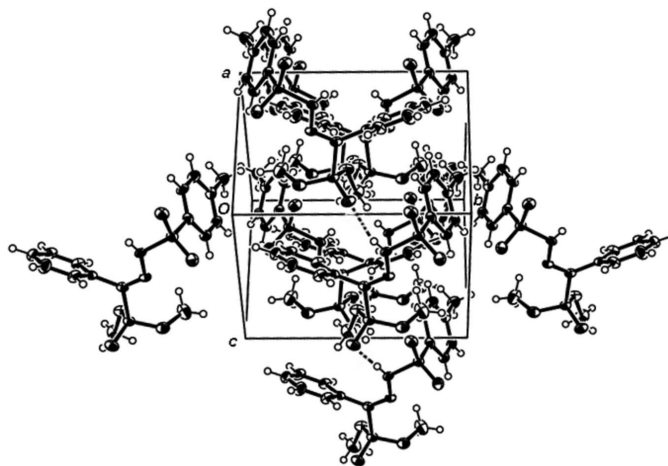


Figure 2
Packing diagram of (I), with the intermolecular $N-H \cdots O$ hydrogen bonds shown as dashed lines.

structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

We thank Drs Fred Hollander and Allen Oliver for guidance and expertise throughout the course Chem 208 and the structure solution and refinement described here. Further, we thank Professor Kenneth N. Raymond for advice and helpful discussions as the instructor for Chem 208.

References

Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
 Bruker (2001). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2003). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
 Gilbert, J. C. & Senaratne, K. P. A. (1984). *Tetrahedron Lett.* **25**, 2303–2306.
 Seyferth, D., Marmor, R. S. & Hilbert, P. (1971). *J. Org. Chem.* **36**, 1379–1386.
 Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.