

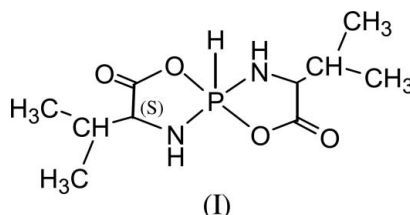
Shu-Xia Cao,<sup>a\*</sup> Jin-Ming Liu,<sup>a</sup>  
Ya-Li Xie,<sup>a</sup> Xin-Cheng Liao<sup>a</sup> and  
Yu-Fen Zhao<sup>a,b</sup><sup>a</sup>Key Laboratory of Chemical Biology,  
Department of Chemistry, Zhengzhou  
University, Henan 450052, People's Republic of  
China, and <sup>b</sup>Key Laboratory for Bioorganic  
Phosphorus Chemistry and Chemical Biology,  
Ministry of Education, Department of Chemistry,  
School of Life Science and Engineering,  
Tsinghua University, Beijing 100084, People's  
Republic of China

Correspondence e-mail: csx@zzu.edu.cn

## Key indicators

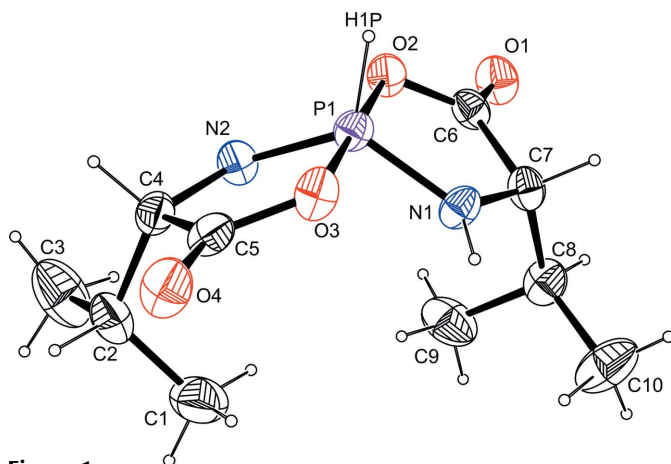
Single-crystal X-ray study  
 $T = 291$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å  
 $R$  factor = 0.053  
 $wR$  factor = 0.136  
Data-to-parameter ratio = 15.3For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.(3*S*,8*S*)-3,8-Diisopropyl-1,6-dioxo-4,9-diaza-  
5 $\lambda^5$ -phosphaspiro[4.4]nonane-2,7-dioneIn the title compound,  $\text{C}_{10}\text{H}_{19}\text{N}_2\text{O}_4\text{P}$ , the molecule adopts a slightly distorted trigonal-bipyramidal structure. The molecules are linked by intermolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds, building a chain along the  $b$  axis parallel to the (001) plane.Received 4 September 2006  
Accepted 16 September 2006

## Comment

The title compound, (I), was synthesized for studies of its spectroscopic characteristics and its biochemical activity. According to experimental evidence, this compound shows a 45% rate inhibition to tyrosinase (Yu, Liu, Fang, Zeng & Zhao, 2005), an important enzyme in the formation of melanin which is widely distributed in microorganisms, animals and plants (Seo *et al.*, 2003). This type of compound and its analogues may afford potent and efficient new inhibitors of tyrosinase.The molecule adopts a slightly distorted trigonal-bipyramidal structure, with the two O atoms in axial positions and the N and H atoms in equatorial positions, as shown in Fig. 1. Major distortion from the ideal angle of  $120^\circ$  occurs for  $\text{N1}-\text{P1}-\text{N2}$ , which is  $124.34(14)^\circ$ . This type of distortion is even more pronounced in the related compounds 6-dioxo-4,9-diaza-5-phosphaspiro[4.4]nonane (Meunier *et al.*, 1978), where the  $\text{N}-\text{P}-\text{N}$  angle is  $130.8^\circ$ , and (3*S*,8*S*)-3,8-dimethyl-1,6-dioxo-4,9-diaza-5 $\lambda^5$ -phosphaspiro[4.4]nonane-2,7-dione (Yu, Liu, Fang & Zhao, 2005), where the  $\text{N}-\text{P}-\text{N}$  angle is  $127.6^\circ$ .The crystal structure of (I) involves intermolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds with an  $R_2^2(12)$  graph-set pattern (Etter *et al.*, 1990), leading to the formation of infinite chains along the  $b$  axis extending parallel to the (001) plane (Fig. 2 and Table 2).

## Experimental

Following a general procedure (Garrigues *et al.*, 1977), triethylamine (25.5 mmol) in tetrahydrofuran (5 ml) was added to a suspension of L-valine (1.00 g, 8.5 mmol) and phosphorus trichloride (1.2 g, 8.5 mmol) in tetrahydrofuran (20 ml) at 343 K over a period of 30 min. After stirring at 343 K for 2 h, the reaction mixture was



**Figure 1**

The molecular structure of compound (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as spheres of arbitrary radii.

neutralized with  $\text{NaHCO}_3$  to pH 7–8 and then extracted with ethyl acetate ( $4 \times 15$  ml). The ethyl acetate layers were combined and dried with magnesium sulfate. The filtrate was concentrated at reduced pressure to give a white solid. The solid was dissolved in methanol and the solution was chromatographed (silica gel,  $3.0 \times 25$  cm, chloroform–methanol–17% aqueous ammonia, 4:4:0.5 v/v/v). Recrystallization from acetone gave the desired compound, (I).

#### Crystal data

$\text{C}_{10}\text{H}_{19}\text{N}_2\text{O}_4\text{P}$	$Z = 2$
$M_r = 262.24$	$D_x = 1.273 \text{ Mg m}^{-3}$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
$a = 10.3925 (17) \text{ \AA}$	$\mu = 0.21 \text{ mm}^{-1}$
$b = 6.0362 (10) \text{ \AA}$	$T = 291 (2) \text{ K}$
$c = 11.2115 (19) \text{ \AA}$	Block, colourless
$\beta = 103.413 (2)^\circ$	$0.24 \times 0.11 \times 0.09 \text{ mm}$
$V = 684.1 (2) \text{ \AA}^3$	

#### Data collection

Bruker APEX2 CCD area-detector diffractometer	4127 measured reflections
$\varphi$ and $\omega$ scans	2471 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 2004)	2053 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.953$ , $T_{\max} = 0.981$	$R_{\text{int}} = 0.024$
	$\theta_{\text{max}} = 25.5^\circ$

#### Refinement

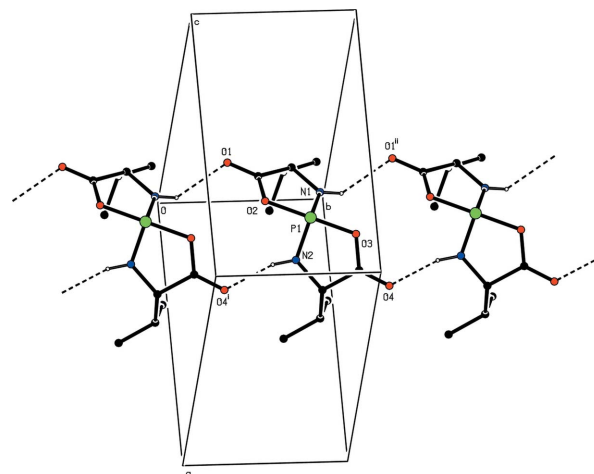
Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0824P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.053$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.136$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.77 \text{ e \AA}^{-3}$
2471 reflections	$\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$
161 parameters	Absolute structure: Flack (1983),
H atoms treated by a mixture of independent and constrained refinement	1069 Friedel pairs
	Flack parameter: $-0.05 (16)$

**Table 1**

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{O4}^{\text{i}}$	0.86	2.27	2.989 (4)	141
$\text{N1}-\text{H1}\cdots\text{O1}^{\text{ii}}$	0.86	2.27	2.981 (4)	141

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



**Figure 2**

A partial packing diagram, showing the  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds (dashed lines) and the formation of infinite chains along the  $b$  axis extending parallel to the (001) plane. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i)  $x, y - 1, z$ ; (ii)  $x, y + 1, z$ ].

H atoms attached to C and N atoms were positioned geometrically and treated as riding on their parent atoms, with  $\text{C}-\text{H} = 0.98$  (methine) or  $0.96 \text{ \AA}$  (methyl), and  $\text{N}-\text{H} = 0.86 \text{ \AA}$ , and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$  for methine H and NH, or  $1.5U_{\text{eq}}(\text{C})$  for methyl H. The position of the H atom attached to the P atom was refined freely, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{P})$ . The absolute configuration of the compound was assigned by reference to the unchanging chiral centres C4 (*S*) and C7 (*S*) in the synthetic procedure and further confirmed by the refinement of the Flack (1983) parameter.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINTE* (Bruker, 2004); data reduction: *SAINTE* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

The authors are grateful for financial support from the Education Department of Henan Province (grant No. 200510459015).

#### References

- Bruker (2004). *SMART, SAINTE and SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
- Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Garrigues, B., Munoz, A., Koenig, M. & Sanchez, M. (1977). *Tetrahedron*, **33**, 635–643.
- Meunier, P. F., Day, R. O. & Devillers, J. R. (1978). *Inorg. Chem.* **17**, 3270–3276.
- Seo, S. Y., Sharma, V. K. & Sharma, N. (2003). *J. Agric. Food Chem.* **51**, 2837–2853.
- Sheldrick, G. M. (1997). *SHELXS97 and SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Yu, L., Liu, Z., Fang, H., Zeng, Q. L. & Zhao, Y. F. (2005). *Amino Acids*, **28**, 369–372.
- Yu, L., Liu, Z., Fang, H. & Zhao, Y.-F. (2005). *Acta Cryst.* **E61**, o261–o262.