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(1*RS*,3a*SR*)-1-(chloromethyl)-1λ^5-phospha-2,6a-diazaperhydropentalene 1-oxide dihydrate

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The title compound, $C_{12}H_{16}ClN_2OP\cdot 2H_2O$, is a bicyclic phosphonamide, containing carbon and phosphorus asymmetric centers.

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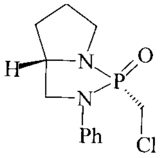
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.128
Data-to-parameter ratio = 15.7

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

Comment

Reaction of glutamic acid with aniline, followed by reduction with $LiAlH_4$, gave (*S*)-2-anilinomethylpyrrolidine, which then reacted with chloromethanephosphonyl dichloride to give the title compound, (I), along with another isomer (Yuan *et al.*, 2000). The title compound has been used to synthesize α-amino phosphonic acid derivatives in high enantiomeric purity (Yuan *et al.*, 2000) than by other methods (Hanessian & Bennani, 1994; Yuan & Cui, 1991).



(I)

In (I) (Fig. 1), the bicyclic chloromethylphosphonamide is not planar, with N—P—O angles of 115.88 (10) and 119.46 (10)°, N—P—C angles of 110.03 (11) and 111.37 (10)°, an O—P—C angle of 105.03 (10)°, P—N bond lengths of 1.669 (3) and 1.627 (4) Å, a P—C bond length of 1.812 (3) Å, and a P—O bond length of 1.4787 (16) Å. The molecular structure of (I) was also confirmed by physical and spectroscopic data (Yuan *et al.*, 2000).

Experimental

Compound (I) was synthesized and purified according to the method of Yuan *et al.* (2000). Single crystals suitable for X-ray data collection were obtained by slow evaporation of an ethyl acetate/petroleum

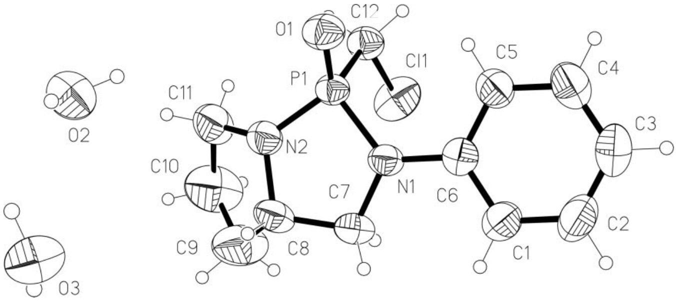


Figure 1
The structure of (I), showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level.

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ether (4:1) solution (m.p. 363–364 K). IR (KBr, cm^{-1}): ν 1240, 1080; ^{1}H NMR (CDCl$_3$): δ 7.26 (*m*, 5H), 6.87 (*m*, 2H), 4.16 (*m*, 1H), 3.87 (*d*, 2H, J = 6 Hz), 3.74 (*m*, 1H), 3.61 (*m*, 1H), 3.27 (*m*, 2H); MS (EI): 268 (M, 45), 219 (M–CH$_2$Cl, 44), 114 (M–CH$_2$Cl–PhNCH$_2$, 100%).

Crystal data

$C_{12}H_{16}ClN_2OP\cdot 2H_2O$
M_r = 306.72
Orthorhombic, *Pbca*
a = 10.8756 (4) Å
b = 8.9390 (3) Å
c = 31.227 (1) Å
V = 3035.79 (18) Å^3
Z = 8
D_x = 1.342 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 486 reflections
θ = 2.4–22.0°
μ = 0.36 mm^{-1}
T = 273 (2) K
Block, colorless
0.34 × 0.26 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min}$ = 0.887, $T_{\max}$ = 0.918
15 965 measured reflections

2976 independent reflections
2444 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.026
$\theta_{\max}$ = 26.0°
$h = -10 \rightarrow 13$
$k = -8 \rightarrow 11$
$l = -38 \rightarrow 35$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.128
S = 1.07
2976 reflections
189 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 1.4344P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.002
$\Delta\rho_{\max}$ = 0.33 e Å^{-3}
$\Delta\rho_{\min}$ = −0.28 e Å^{-3}

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
O3—H23$\cdots$O1$^{\rm i}$	0.851 (18)	1.98 (2)	2.819 (3)	168 (4)
O3—H24$\cdots$O2$^{\rm ii}$	0.828 (18)	1.95 (2)	2.757 (3)	166 (3)
O2—H22$\cdots$O3$^{\rm iii}$	0.842 (18)	1.91 (2)	2.740 (3)	171 (3)
O2—H21$\cdots$O1$^{\rm i}$	0.833 (18)	2.03 (2)	2.850 (3)	167 (4)

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

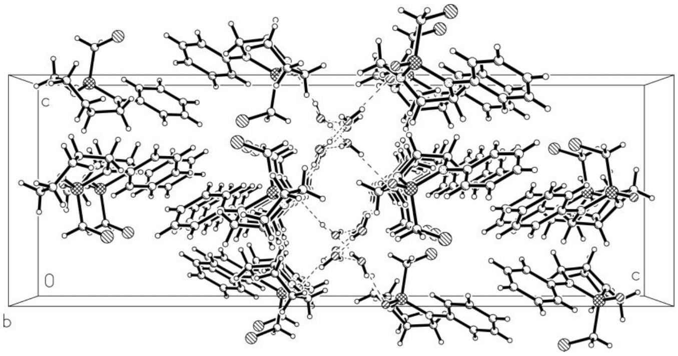

Figure 2
Packing diagram of (I), viewed down the *c* axis. Hydrogen bonds are shown as dashed lines.

All H atoms of the organic molecule were initially located in a difference Fourier map and later placed in idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å and $U_{\rm iso}$(H) = 1.2$_{\rm eq}$(C). The water H atoms were located in a difference Fourier map and refined without any constraints.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

Bruker (2000). *SMART*, *SAINT*, *SADABS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Hanessian, S. & Bennani, Y. L. (1994). *Synthesis*, **12**, 1272–1274.
Yuan, C. & Cui, S. (1991). *Phosphorus Sulfur Silicon*, **55**, 159–164.
Yuan, C., Li, S. & Wang, G. (2000). *Heteroatom. Chem.* **11**, 528–535.