phase angle of 46 (1)° (Altona, Geise & Romers, 1968). The molecule has an overall planar shape (Fig. 1*b*), with the acetylene group at C17 attached in an α (axial) position and the two methyl groups on the β face. The hydroxyl group is near-equatorial and the side chain attached at C33 is in an extended conformation.

In the crystal structure, the molecules are arranged in an end-to-end manner, with intermolecular hydrogen bonds between O21 and O31 [O···O distance is 2·871 (4) Å].

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# Structure of (R)-1-[(R)-1-Phenylethylamino]benzylphosphonic Acid Sesquihydrate

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**Abstract.** $C_{15}H_{18}NO_3P.1\cdot5H_2O$, $M_r = 318\cdot35$, orthorhombic, $P2_12_12_1$, $a = 6\cdot587$ (2), $b = 11\cdot769$ (3), $c = 20\cdot575$ (5) Å, $V = 1595\cdot0$ Å$^3$, $Z = 4$, $D_m = 1\cdot326$ (1), $D_x = 1\cdot325$ Mg m$^{-3}$, $T = 291$ K, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 0\cdot20$ mm$^{-1}$, $F(000) = 676$, $R = 0\cdot043$ for 1797 reflexions. The molecule exists as a zwitterion: the amino group is protonated and the phosphonic acid group is ionized. The conformation of the molecule is *trans–gauche*, angles $\chi^1$[P—C(1)—N—C(2)] and $\chi^2$[C(1)—N—C(2)—C(phenyl)] being 169·6 (3) and 54·5 (4)°, respectively. The crystal structure is stabilized by three intermolecular hydrogen bonds and four van der Waals contacts. The absolute configuration of the molecule was assigned as *R,R* and is consistent with the known *R* configuration of (+)-1-phenylethylamine.

**Introduction.** The determination of the molecular structure and absolute configuration of 1-aminophosphonic acids (1) is of considerable importance in view of the established inhibition of enzymes involved in the metabolism of amino acids by the phosphonic acid analogues of substrates (Neuzil & Cassaigne, 1980; Brand & Lowenstein, 1978; Lejczak, Starzemska & Mastalerz, 1981).

R—CH—PO$_3$H$_2$       NH—CH—C$_6$H$_5$ (with CH$_3$ above)
 NH$_2$           C$_6$H$_5$—CH—PO$_3$H$_2$
 (1)             (2)
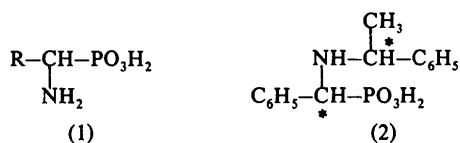
Recently, we reported the synthesis and absolute configuration of (S)-2-methyl-1-[(R)-1-phenylethylamino]-1-propanephosphonic acid monohydrate (Sawka-Dobrowolska, Głowiak, Kowalik & Mastalerz, 1985) and (R)-1-[(S)-1-phenylethylamino]benzylphosphonic acid monohydrate (Głowiak, Sawka-Dobrowolska, Kowalik, Mastalerz, Soroka & Zoń, 1977). The (R,S) diastereoisomer of (2) was prepared by addition of diethylphosphite to the aldimine prepared from benzaldehyde and (−)-(S)-1-phenylethylamine.

In continuation of our studies on the crystal structure and configuration of optically active 1-aminophosphonic acids we now report X-ray results for the title compound (2).

    

Table 1. *Positional parameters and equivalent isotropic temperature factors* ($\AA^2$) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\Sigma_i B_{if}$$

| | x | y | z | $B_{eq}$ |
|---|---|---|---|---|
| P | 0.1346 (2) | 0.3968 (1) | 0.5292 (1) | 1.9 (1) |
| O(1) | −0.0852 (4) | 0.3923 (2) | 0.5465 (1) | 2.5 (2) |
| O(2) | 0.2649 (5) | 0.3029 (2) | 0.5538 (1) | 2.9 (2) |
| O(3) | 0.2177 (5) | 0.5177 (2) | 0.5483 (1) | 2.8 (2) |
| O(4)_w | 0.6169 (5) | 0.5550 (3) | 0.5414 (2) | 5.3 (3) |
| O(5)_w* | 0.0318 (16) | 0.2315 (6) | 0.9693 (4) | 7.5 (9) |
| N | 0.1319 (5) | 0.2776 (3) | 0.4154 (1) | 1.9 (2) |
| C(1) | 0.1633 (5) | 0.3969 (3) | 0.4396 (2) | 1.8 (2) |
| C(11) | 0.0251 (7) | 0.4842 (3) | 0.4080 (2) | 2.2 (3) |
| C(12) | −0.1789 (7) | 0.4620 (4) | 0.3949 (2) | 2.7 (3) |
| C(13) | −0.2977 (8) | 0.5450 (4) | 0.3662 (2) | 3.6 (4) |
| C(14) | −0.2214 (10) | 0.6514 (5) | 0.3535 (3) | 4.2 (5) |
| C(15) | −0.0223 (10) | 0.6751 (4) | 0.3675 (3) | 4.2 (4) |
| C(16) | 0.1038 (7) | 0.5920 (4) | 0.3946 (2) | 3.2 (3) |
| C(2) | 0.1122 (7) | 0.2625 (3) | 0.3420 (2) | 2.2 (3) |
| C(3) | 0.0857 (8) | 0.1362 (4) | 0.3291 (2) | 3.2 (4) |
| C(21) | 0.2859 (7) | 0.3117 (3) | 0.3032 (2) | 2.3 (3) |
| C(22) | 0.4751 (7) | 0.2613 (4) | 0.3022 (2) | 2.9 (3) |
| C(23) | 0.6205 (8) | 0.2948 (5) | 0.2566 (2) | 3.8 (4) |
| C(24) | 0.5774 (9) | 0.3813 (5) | 0.2139 (2) | 4.2 (4) |
| C(25) | 0.3913 (10) | 0.4341 (4) | 0.2161 (2) | 4.3 (5) |
| C(26) | 0.2456 (8) | 0.3997 (4) | 0.2604 (2) | 3.3 (3) |

\* Occupancy 0.5.

**Experimental.** Synthesis by the reaction of triethyl-phosphite–aldimine mixture with benzenesulfonic acid, (Kowalik, 1985). Crystallization of crude (2) from water yielded pure (2), m.p. 461–465 K.

Colourless transparent crystals $0.4 \times 0.39 \times 0.25$ mm; $D_m$ by pycnometric method in benzene/1,1,2,2-tetrachloroethane; orthorhombic $P2_12_12_1$ from Weissenberg photographs; Syntex $P2_1$ computer-controlled four-circle diffractometer, scintillation counter, graphite monochromator; cell parameters by least squares from setting angles of 15 reflexions with $16 \le 2\theta(\text{Mo}) \le 25^\circ$ measured on diffractometer; 2129 independent reflexions; $2\theta_{max} = 55^\circ$; variable $\theta$–$2\theta$ scans, scan rate $2.0$–$29.3^\circ$ min$^{-1}$ depending on intensity; two standards (236, 322) measured every 50 reflexions, variation in intensities $\pm 2.5\%$; data corrected for Lorentz and polarization factor, not for absorption; 1797 with $I > 3.0\sigma(I)$ used for structure determination; index range $h$ 0 to 8, $k$ 0 to 15, $l$ 0 to 26; calculations performed with Syntex (1976) *XTL/XTLE* system; scattering factors for neutral atoms, corrected for anomalous dispersion, taken from *International Tables for X-ray Crystallography* (1974); structure solved by direct methods, Syntex (1976) version of *MULTAN* (Germain, Main & Woolfson, 1971); $E$ map and series of difference syntheses yielded the positions of the non-H atoms, except O(5)$_w$ of water molecule. The compound was identified as $C_{15}H_{18}NO_3P.H_2O$; structure refinement by block-diagonal least-squares techniques using $B_{iso}$ and then $B_{ij}$ anisotropic parameters (13 H atoms placed at computed positions) gave $R = 0.076$. A difference synthesis at this stage revealed relatively large peak ($\sim 1.6$ e $\AA^{-3}$) which

corresponded to the water molecule, O(5)$_w$. From measured density and refinement of thermal parameters it follows that the occupancy factor for O(5)$_w$ is approximately 0.5. Further difference synthesis located H atoms bonded to O(3), O(4)$_w$ O(5)$_w$, N and C(methyl group). Further refinement with anisotropic temperature factor for O(5)$_w$ and fixed parameters for H atoms ($B = 5.0 \AA^2$) gave a final $R = 0.043$, $wR = 0.047$, $S = 3.31$ (for the inverted structure $R = 0.044$ and $wR = 0.048$).

Max. $\Delta/\sigma$ in final LS cycle 0.01; max. electron density in final difference map $0.17$, max. negative $-0.16$ e $\AA^{-3}$; function minimized $\Sigma w(|F_o| - |F_c|)^2$, $w = 1/\sigma^2(F)$.

The absolute configuration of the molecule was assigned as $R,R$ and is consistent with the known $R$ configuration of (+)-1-phenylethylamine.

**Discussion.** Final atomic parameters are given in Table 1.\* The molecular structure and atom numbering are shown in Fig. 1.

The principal bond lengths and angles in the title molecule are given in Table 2. Most of the bond lengths are quite typical of those found in other aminophosphonic acids.

The title molecule occurs as a zwitterion. The phosphonic group is negatively charged, the charge being equally distributed between O(1) and O(2), P—O lengths being 1.492 (3) and 1.488 (3) $\AA$. The P—O(3) bond length of 1.574 (3)$\AA$ indicates a single bond. The P—C(1) distance, 1.853 (3) $\AA$, is slightly longer (by $3\sigma$) than the P—CH$_2$ distance in aminomethylphosphonic acid, $\beta$-AMPh [1.817 (3) $\AA$] (Darriet, Darriet, Cassaigne & Neuzil, 1975) and similar to those found in aminobenzylphosphonic acid [1.865 (7) $\AA$] (Sawka-

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\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51014 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
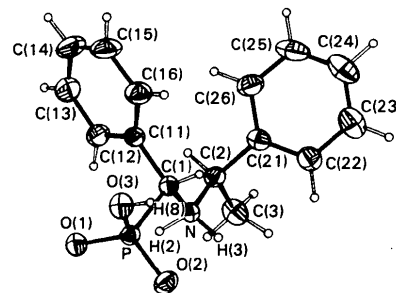


Fig. 1. *ORTEP* drawing (Johnson, 1976). The molecular structure of the title compound showing the atom labelling. Thermal ellipsoids are drawn at the 50% probability level.

**Table 2.** *Bond distances* (Å), *angles* (°) *and selected torsion angles* (°)

| | | | |
|---|---|---|---|
| P—O(1) | 1·492 (3) | C(14)—C(15) | 1·372 (9) |
| P—O(2) | 1·488 (3) | C(15)—C(16) | 1·399 (7) |
| P—O(3) | 1·574 (3) | C(16)—C(11) | 1·398 (6) |
| P—C(1) | 1·853 (3) | C(2)—C(21) | 1·511 (6) |
| C(1)—N | 1·504 (5) | C(21)—C(22) | 1·380 (6) |
| N—C(2) | 1·525 (4) | C(22)—C(23) | 1·397 (6) |
| C(2)—C(3) | 1·521 (6) | C(23)—C(24) | 1·376 (7) |
| C(1)—C(11) | 1·518 (5) | C(24)—C(25) | 1·375 (9) |
| C(11)—C(12) | 1·395 (6) | C(25)—C(26) | 1·384 (7) |
| C(12)—C(13) | 1·384 (7) | C(26)—C(21) | 1·386 (6) |
| C(13)—C(14) | 1·375 (7) | | |

| | | | |
|---|---|---|---|
| O(1)—P—O(2) | 116·9 (2) | C(14)—C(15)—C(16) | 120·6 (5) |
| O(1)—P—O(3) | 108·1 (2) | C(15)—C(16)—C(11) | 119·6 (4) |
| O(1)—P—C(1) | 109·7 (2) | C(1)—N—C(2) | 116·6 (3) |
| O(2)—P—O(3) | 112·6 (2) | N—C(2)—C(3) | 107·3 (3) |
| O(2)—P—C(1) | 106·3 (2) | N—C(2)—C(21) | 114·5 (3) |
| O(3)—P—C(1) | 102·2 (2) | C(3)—C(2)—C(21) | 111·6 (3) |
| P—C(1)—N | 108·3 (2) | C(2)—C(21)—C(22) | 121·9 (4) |
| P—C(1)—C(11) | 111·4 (3) | C(2)—C(21)—C(26) | 118·5 (4) |
| N—C(1)—C(11) | 114·1 (3) | C(22)—C(21)—C(26) | 118·9 (4) |
| C(1)—C(11)—C(12) | 122·2 (4) | C(21)—C(22)—C(23) | 120·6 (4) |
| C(1)—C(11)—C(16) | 118·5 (4) | C(22)—C(23)—C(24) | 119·8 (5) |
| C(11)—C(12)—C(13) | 119·7 (4) | C(23)—C(24)—C(25) | 119·8 (5) |
| C(12)—C(13)—C(14) | 121·2 (5) | C(24)—C(25)—C(26) | 120·5 (5) |
| C(13)—C(14)—C(15) | 119·7 (5) | C(25)—C(26)—C(21) | 120·3 (4) |

| | | |
|---|---|---|
| $\tau$ | O(3)—P—C(1)—N | 168·0 (3) |
| | O(2)—P—C(1)—N | 49·7 (3) |
| | O(1)—P—C(1)—N | −77·5 (3) |
| $\chi^1$ | P—C(1)—N—C(2) | 169·6 (3) |
| $\chi^2$ | C(1)—N—C(2)—C(21) | 54·5 (4) |
| | C(1)—N—C(2)—C(3) | 179·0 (4) |
| $\chi^{11}$ | N—C(1)—C(11)—C(12) | 40·3 (5) |
| $\chi^{12}$ | N—C(1)—C(11)—C(16) | −142·7 (4) |
| $\chi^{21}$ | N—C(2)—C(21)—C(22) | 72·7 (5) |
| $\chi^{22}$ | N—C(2)—C(21)—C(26) | −116·9 (5) |

Dobrowolska, 1980), α-(isopropylamino)salicylphosphonic acid hemihydrate [1·858 (3) Å] (Sawka-Dobrowolska, 1985) and in (S)-2-methyl-1-[(R)-1-phenylethylamino]-1-propanephosphonic acid monohydrate [1·856 (5) Å] (Sawka-Dobrowolska, Głowiak, Kowalik & Mastalerz, 1985).

The O—P—O and O—P—C angles in the title molecule range from 102·2 (2) to 116·9 (2)°. The smallest angle involves the protonated O(3) and C(1), while the largest angle involves the unsubstituted O(1) and O(2). The above values agree well with those of the corresponding angles in other aminophosphonic acids. The P—C(1)—N and C(1)—N—C(2) angles [108·3 (2) and 116·6 (3)°] are similar to the corresponding angles in (S)-2-methyl-1-[(R)-1-phenylethylamino]-1-propanephosphonic acid [108·0 (3) and 116·3 (3)°].

The phenyl moieties are nearly planar; the maximum deviations from the best planes through the ring atoms are 0·016 (5) and 0·014 (4) Å, respectively. The mean C—C bond length is 1·385 Å as in benzene and the C—C—C angles are close to 120°.

The conformation of the molecule is described by the torsion angles $\chi^1$, $\chi^2$, $\chi^{11}$, $\chi^{12}$, $\chi^{21}$, $\chi^{22}$ and $\tau$ listed in Table 2. The molecule assumes a *trans–gauche* conformation with $\chi^1$ and $\chi^2$, the torsion angles about C(1)—N and N—C(2), 169·6 (3) and 54·5 (4)° respectively.

**Table 3.** *Hydrogen-bond distances* (Å) *and angles* (°) *and short contact distances* (Å)

| D—H···A | | H···A | D—H···A | D—H |
|---|---|---|---|---|
| N—H(2)···O(2$^i$) | 2·672 (5) | 1·68 | 166 | 1·01 |
| N—H(3)···O(1$^{ii}$) | 2·843 (4) | 1·89 | 164 | 0·97 |
| O(3)—H(8)···O(4)$_w$ | 2·670 (5) | 1·85 | 168 | 0·83 |

| Short contacts | |
|---|---|
| O(5)$_w$···O(3$^{iii}$) | 3·028 (9) |
| O(4)$_w$···O(1$^{iv}$) | 2·744 (4) |
| O(5)···O(4$^v$)$_w$ | 3·117 (10) |
| O(5)···O(4$^{vi}$)$_w$ | 3·078 (9) |

Symmetry codes: (i) −0·5 + x, 0·5 − y, 1 − z; (ii) 0·5 + x, 0·5 − y, 1 − z; (iii) −x, −0·5 + y, 1·5 − z; (iv) 1 + x, y, z; (v) 1 − x, −0·5 + y, 1·5 − z; (vi) 0·5 − x, 1 − y, 0·5 + z.

An interesting feature of the present structure is the torsion angle $\tau$, 168·0 (3)°, which is greater than that in (S)-(1-amino-2-phenylethyl)phosphonic acid monohydrate [−59·0 (3)°] (Kowalik, Sawka-Dobrowolska & Głowiak, 1984), α-(isopropylamino)salicylphosphonic acid hemihydrate [−77·5 (2)°] (Sawka-Dobrowolska, 1985), 2-amino-3-phosphonopropionic acid [−74·1 (4)°] (Sawka-Dobrowolska, Głowiak, Siatecki & Soroka, 1985) and is similar to the $\tau$ values [−152·3 (3) and 135·7 (6)°] found in (S)-1-aminoethylphosphonic acid and (R)-1-aminobenzylphosphonic acid (Sawka-Dobrowolska, 1980).

As in other related aminophosphonic acids, the present structure also manifests the characteristic feature of forming a network of intermolecular hydrogen bonds in the crystal. Molecules related by the x screw axis are bound together by N···O(2) and N···O(1) hydrogen bonds (Table 3). Another hydrogen bond runs from O(3) to the water O(4) atom of the molecule at (x, y, z). O(4)$_w$ also forms a short contact with O(1) of the molecule at (x + 1, y, z) [O(4)$_w$··· O(1) = 2·744 (4) Å].

It is interesting to note that the H atoms of the O(4)$_w$ water molecule are not involved in hydrogen-bond formation. The site of O(5)$_w$ is only partially occupied and O(5)$_w$ is also not involved in hydrogen bonding. This atom is involved in intermolecular contacts with O(3) of 3·028 (9) Å, and O(4)$_w$ of 3·117 (10) and 3·078 (9) Å (Table 3).

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# Adamantylideneadamantane Epoxide

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**Abstract.** 2-(Tricyclo[3.3.1.1$^{3,7}$]dec-2-ylidene)tricyclo-[3.3.1.1$^{3,7}$]decane 2,2'-epoxide (2), $C_{20}H_{28}O$, $M_r = 284.45$, monoclinic, $P2_1/n$, $a = 6.539$ (1), $b = 21.002$ (2), $c = 11.195$ (1) Å, $\beta = 93.68$ (1)°, $V = 1534.3$ (3) Å$^3$, $Z = 4$, $D_x = 1.23$ g cm$^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.68$ cm$^{-1}$, $F(000) = 624$, $T = 293$ K, $R = 0.0427$ for 1548 reflections. The title compound has approximate *mm* symmetry. The bonds in the adamantane moieties average 1.524 (8) Å and the angles 109.4 (5)° indicating little bond or angle strain except in the three-membered epoxide ring.

**Introduction.** The reaction of singlet oxygen with olefins has been postulated to occur *via* a perepoxide intermediate with subsequent rearrangement into a dioxetane or to an allylic hydroperoxide *via* an ene mechanism. Compounds such as (1) are of interest because the flanking bridgehead hydrogen atoms cannot participate in the ene reaction and are classified as Bredt's rule protected olefins. Reaction of (1) with $^1O_2$ leads to the formation of a relatively stable dioxetane (3) which decomposes slowly at room temperature to adamantanone (Wieringa, Strating, Wijnberg & Adam, 1972). The reaction of (1) with singlet oxygen generated chemically or photogenetically leads to the formation of both (2) and (3) with the ratio of the two products dependent upon solvent and sensitizer (Jefford & Boschong, 1977). These authors postulate that the production of the epoxide occurs *via* loss of a hydroperoxy radical while the dioxetane is formed most likely *via* a radical cation reaction of (1) (Bartlett, 1978; Nelsen, Kapp, Akaba & Evans, 1986). A number of theoretical and experimental investigations of (1) and other Bredt's rule protected olefins have been reported (*e.g.* Clark, Teasley, Nelsen & Wynberg, 1987). Molecular-mechanics calculations have been reported for (1) and a number of its derivatives to evaluate strain energies and conformational changes due to short intramolecular contacts across the double bond (Lenoir & Frank, 1978; Cordt, Frank & Lenoir, 1979; Lenoir, Frank, Cordt, Gieren & Lamm, 1980). In order to provide additional structural data for this series of compounds, we report the structure of adamantylideneadamantane epoxide (2).



(1)          (2)

(3)
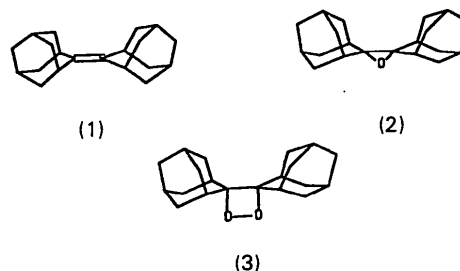
**Experimental.** Compound (2) prepared by peracid oxidation of (1); colorless, transparent crystal from CHCl$_3$ recrystallizations, 0.43 × 0.33 × 0×30 mm; Nicolet *R3M/μ* update of *P2$_1$* diffractometer; data collected in Wyckoff mode (2θ fixed, ω varied, 3° ≤ 2θ ≤ 45°), variable scan rate (4 to 29.3° min$^{-1}$), graphite-monochromated Mo *K*α radiation; lattice parameters from a least-squares refinement of 15

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