$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.003$
$\Delta\rho_{max} = 1.58$ e Å^{-3}
$\Delta\rho_{min} = -1.48$ e Å^{-3}

Absolute configuration:
Flack (1983)
Flack parameter = −0.01 (2)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Pt1	0.13252 (7)	0.82336 (10)	0.18220 (6)	0.0183 (2)
Cl1	0.2772 (5)	0.7982 (6)	0.1348 (5)	0.028 (2)
Cl2	−0.0101 (5)	0.8501 (8)	0.2322 (5)	0.029 (2)
Cl3	0.2481 (5)	0.9526 (6)	0.3362 (4)	0.0272 (15)
S1	0.0105 (5)	0.7124 (6)	0.0335 (4)	0.0203 (14)
O1	−0.0850 (13)	0.7944 (16)	−0.0497 (12)	0.038 (5)
C29	−0.046 (2)	0.572 (3)	0.073 (2)	0.041 (8)
C30	0.074 (2)	0.621 (3)	−0.040 (2)	0.033 (7)
C1	0.3010 (18)	0.777 (2)	0.7166 (18)	0.018 (5)
C2	0.2787 (17)	0.737 (2)	0.8061 (17)	0.017 (5)
C3	0.2375 (17)	0.601 (2)	0.7850 (17)	0.019 (5)
C4	0.2254 (18)	0.565 (2)	0.6796 (17)	0.024 (5)
C5	0.2670 (19)	0.672 (2)	0.6385 (19)	0.018 (6)
C21	0.2860 (18)	0.674 (2)	0.5346 (18)	0.025 (6)
C22	0.1979 (19)	0.587 (3)	0.4411 (17)	0.030 (6)
N1	0.4042 (14)	0.6264 (17)	0.5704 (14)	0.017 (4)
C23	0.423 (2)	0.482 (2)	0.593 (2)	0.034 (6)
C24	0.4404 (18)	0.664 (3)	0.4865 (19)	0.027 (6)
Fe1	0.1325 (3)	0.7315 (3)	0.6625 (2)	0.0185 (8)
C6	0.0463 (18)	0.906 (2)	0.6373 (16)	0.016 (5)
C7	0.0072 (16)	0.806 (3)	0.6903 (17)	0.025 (6)
C8	−0.0313 (19)	0.697 (3)	0.6252 (19)	0.031 (6)
C9	−0.0246 (19)	0.722 (3)	0.526 (2)	0.030 (6)
C10	0.0215 (16)	0.853 (3)	0.5321 (15)	0.020 (5)
C11	0.3488 (18)	0.906 (2)	0.7037 (17)	0.020 (5)
C12	0.2898 (19)	1.011 (2)	0.6272 (18)	0.016 (6)
C13	0.3735 (17)	1.104 (2)	0.6320 (17)	0.022 (5)
C14	0.482 (2)	1.062 (3)	0.7142 (18)	0.027 (6)
C15	0.4665 (16)	0.937 (2)	0.7539 (16)	0.014 (5)
C25	0.5592 (15)	0.846 (3)	0.8349 (14)	0.021 (5)
C26	0.6631 (18)	0.914 (2)	0.9215 (16)	0.027 (6)
N2	0.5902 (14)	0.7483 (18)	0.7647 (14)	0.018 (3)
C27	0.6446 (18)	0.626 (2)	0.8284 (17)	0.022 (4)
C28	0.6624 (16)	0.805 (3)	0.7206 (15)	0.020 (4)
Fe2	0.3810 (2)	1.0795 (3)	0.7879 (2)	0.0174 (7)
C16	0.3121 (18)	1.078 (2)	0.8968 (17)	0.026 (5)
C17	0.2774 (19)	1.194 (2)	0.8278 (18)	0.024 (6)
C18	0.3779 (19)	1.261 (3)	0.8495 (19)	0.032 (6)
C19	0.470 (2)	1.194 (2)	0.9216 (18)	0.025 (6)
C20	0.435 (2)	1.080 (3)	0.954 (2)	0.043 (7)

Table 2. *Selected geometric parameters (Å, °)*

Pt1—S1	2.202 (6)	S1—O1	1.49 (2)
Pt1—Cl1	2.305 (6)	S1—C29	1.78 (3)
Pt1—Cl3	2.316 (6)	S1—C30	1.82 (2)
S1—Pt1—Cl1	93.0 (2)	S1—Pt1—Cl3	175.0 (2)
S1—Pt1—Cl2	87.9 (2)	Cl1—Pt1—Cl3	90.8 (2)
Cl1—Pt1—Cl2	179.1 (2)	Cl2—Pt1—Cl3	88.3 (2)

H atoms were included in calculated positions using standard *SHELXL*93 *HFIX* (Sheldrick, 1993) procedures and their parameters were not refined.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *ORTEP* (Johnson, 1965).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1112). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Monosodium 4-Amino-1-hydroxy-1,1-butanediyldiphosphonate Trihydrate (Alendronate)

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Abstract

The cation in the title compound, $Na^+.C_4H_{12}NO_7P_2^-.3H_2O$, has the expected zwitterionic character, with a terminal protonated amine group on one side of the

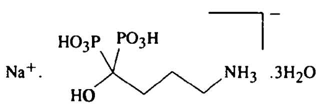
chain and a negatively charged diphosphonate group on the other, the latter interacting with the Na$^+$ ion. The structure is stabilized by a complex hydrogen-bonding scheme, which is found to be weaker than that observed in the free acid.

Comment

Over the last two and a half decades, many diphosphonate compounds have been the subject of intensive pharmacological studies (King, Francis & Michael, 1971; Francis & Centner, 1978) mainly due to their potential use in the treatment of bone diseases.

Among them, alendronate, the monosodium salt of 4-amino-1-hydroxy-1,1-butanediylbis(phosphonic acid), has been found to be most effective for clinical treatment. The drug has been developed as an inhibitor of bone resorption due to its ability to bind selectively to the mineral phase exposed at the endosteal bone surface, mainly at resorption lagunae, where it interacts with the osteoclasts, the macrophage cells that resorb bone tissue. In the presence of alendronate, osteoclasts lose their ruffled border, remaining inactive but intact (Sato *et al.*, 1991). To a lesser extent and mainly due to a feedback mechanism, alendronate also affects bone formation by turning the calcium balance positive. This property is the basis for the use of alendronate in the treatment of human osteoporosis and other bone diseases where the calcium balance is negative.

Due to the importance of the subject, some pioneering structural work on related compounds has been published over the last few years (Coiro & Lamba, 1989; Leroux, El Manouni, Safsaf, Neuman & Giller, 1991; Shkol'nikova, Sotman & Afonin, 1990), but to our knowledge none has been performed on the extensively used monosodium salt of 4-amino-1-hydroxy-1,1-butanediylbis(phosphonic acid), the single-crystal structure of which is reported here as its trihydrate, (I).



(I)

A view of the salt with the interactions to Na$^+$ indicated by dashed lines is shown in Fig. 1. The cation presents the usual zwitterionic form characteristic of this type of diphosphonate compound, with the protonated amine group bearing the positive charge. The fact that only one O atom is protonated in each of the phosphonate units suggests that the two extra electrons needed to account for the anionic character of the molecular group as a whole are located at each of these groups, evenly shared by the non-protonated O atoms. Inspection of the two different types of P—O distances [P—OH 1.579 (2) and 1.576 (2) Å, and P—O 1.506 (2), 1.504 (2), 1.498 (2) and 1.502 (2) Å] and O—P—O bond angles [O—P—OH 106.6 (1), 111.3 (1), 110.9 (1) and 106.0 (1)°, and O—P—O 116.8 (1) and 117.2 (1)°] clearly confirms the resonance with a partial double-bond character for the latter, as has also been reported in related structures (Coiro & Lamba, 1989).

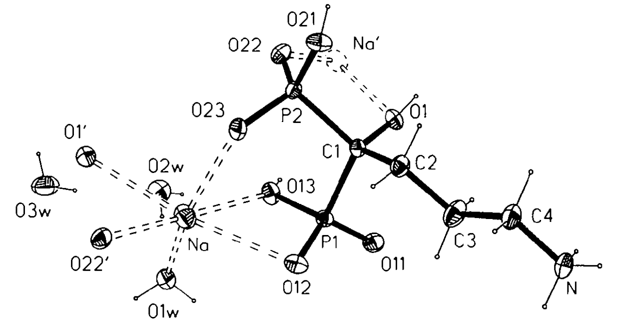


Fig. 1. View of the title salt showing the numbering scheme used and displacement ellipsoids drawn at the 50% probability level. Coordination to sodium is depicted by dashed open lines.

The torsion angles along the chain [P1—C1—C2—C3 57.3 (3), P2—C1—C2—C3 −179.5 (2), C1—C2—C3—C4 162.9 (2) and C2—C3—C4—N 178.8 (2)°] show that the C3 atom is *trans* with respect to the P2 atom and the chain length is stretched close to its maximum possible length. This is similar to what was found for the free acid by Leroux *et al.* (1991).

The same authors suggested an inverse correlation between the value of the P1—C1—P2 (φ) angle and a certain 'staggering' angle between the PO$_3$ groups when viewed along the P1· · ·P2 direction (ρ). As a measure of the latter, they used the average value of the highly scattered non-bonding torsion angles around the P1· · ·P2 direction. We found, however, that the addition of the two torsion angles O*x*—P1—C1—P2 and P1—C1—P2—O*y* proved more adequate for this kind of measurement, resulting in mean values with much lower dispersion. The trend of an inverse relationship between the two forementioned angles is confirmed, with alendronate at one of the extremes of the range. Our calculations of the φ/ρ pair show the following values (in °) for some related structures: 110.0 (1)/45 (8) (this work), 111.0/34 (2) and 112.1/32 (3) (Leroux *et al.*, 1991), 113.6/26 (7) (Coiro & Lamba, 1989) and 115.1/4 (4) (Uchtman & Gloss, 1972).

All 12 non-alkylic H atoms available, *i.e.* three from the amine, one from the hydroxyl, two from the phosphonic acid groups and six from the hydration water molecules, are involved in a very complex hydrogen-bonding scheme (Table 3 and Fig. 2). Even though these interactions have a strong character overall, as assessed by the rather short mean H· · ·O distance [1.82 (12) Å, for the 11 single hydrogen bonds], they rank in strength clearly below those present in the free acid, where six H···O distances are found to be under 1.52 Å, four of which are under 1.35 Å. This fact is probably the main reason for the very dissimilar solubility properties shown by the two compounds.

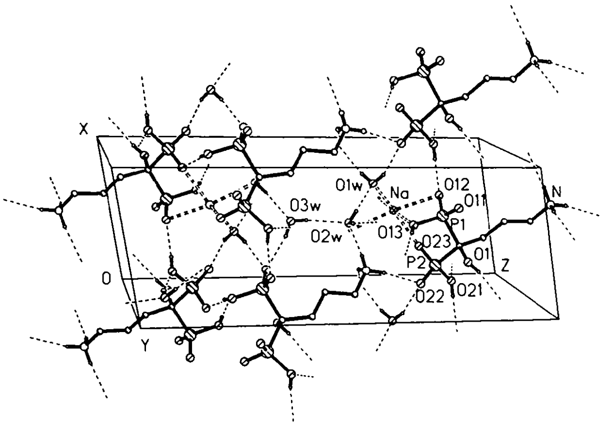


Fig. 2. A simplified packing diagram (alkylic H atoms omitted) showing the complex three-dimensional network. Hydrogen-bonding interactions are represented as simple dashed lines and the interactions to sodium as dashed open lines. Contacts to neighboring molecules are also shown, with the receptor atoms omitted.

Na···O contacts [range 2.288 (2)–2.580 (2) Å] are typical, but their lengths do not correlate with the individual charge ascribed to each of the O atoms involved, as would be expected for free pure electrostatic interactions. This suggests that the cations are accommodated where allowed by the considerations of stronger hydrogen bonding and steric interactions. This results in a distorted octahedral environment, with a rather planar equatorial array defined by the longer bonds [maximum deviation 0.011 (2) Å, with the cation lying 0.052 (2) Å out of the plane]. The two short apical contacts involving atoms O1*W* and O23 deviate significantly from normal values, as shown by the O1*W*—Na—O13 and O23—Na—O13 angles of 76.60 (7) and 77.39 (7)°, respectively.

The polar character of the molecule is also evidenced in the way in which packing is achieved (Fig. 2); while the negative part of the molecule is adjacent to the cation, the chain ending in the positive amino group stretches away as far as possible, interacting with a variety of philo-positive O atoms *via* hydrogen bonding.

Experimental

The title compound was obtained from Laboratorios Gador, Division Biofarma, Argentina, and crystals suitable for X-ray diffraction analysis were obtained through the insolubilization of a highly concentrated water solution by slow vapor diffusion of methanol.

Crystal data

$Na^+.C_4H_{12}NO_7P_2^-.3H_2O$
$M_r = 325.12$
Monoclinic
$P2_1/n$
$a = 7.275\ (1)$ Å
$b = 9.002\ (2)$ Å
$c = 19.503\ (4)$ Å
$\beta = 100.61\ (1)°$
$V = 1255.4\ (4)$ Å^3
$Z = 4$
$D_x = 1.720$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.5–12.5°$
$\mu = 0.426$ mm^{-1}
$T = 293\ (2)$ K
Prismatic
0.30 × 0.25 × 0.20 mm
Colorless

Data collection

Siemens $R3m$ diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4046 measured reflections
1643 independent reflections
1503 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0163$

$\theta_{\rm max} = 22.51°$
$h = -1 \rightarrow 7$
$k = -9 \rightarrow 9$
$l = -20 \rightarrow 20$
2 standard reflections monitored every 98 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0284$
$wR(F^2) = 0.0828$
$S = 1.114$
1641 reflections
164 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0619P)^2 + 7.3866P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = -0.025$
$\Delta\rho_{\rm max} = 0.333$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.353$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Na	0.4697 (2)	−0.02784 (12)	0.74476 (6)	0.0250 (3)
P1	0.49634 (9)	0.24792 (7)	0.84194 (3)	0.0137 (2)
P2	0.10487 (9)	0.11647 (8)	0.82045 (3)	0.0138 (2)
O11	0.5865 (2)	0.3918 (2)	0.86876 (10)	0.0200 (5)
O12	0.6103 (2)	0.1093 (2)	0.85755 (10)	0.0205 (5)
O13	0.4313 (3)	0.2518 (2)	0.76026 (9)	0.0198 (5)
O21	−0.0324 (2)	0.0534 (2)	0.86734 (9)	0.0206 (5)
O22	0.0014 (3)	0.2124 (2)	0.76285 (9)	0.0196 (4)
O23	0.2092 (2)	−0.0148 (2)	0.79883 (9)	0.0199 (4)
O1	0.2043 (2)	0.3831 (2)	0.87556 (9)	0.0171 (4)
C1	0.2775 (4)	0.2339 (3)	0.87803 (13)	0.0133 (6)
C2	0.3170 (4)	0.1744 (3)	0.95346 (13)	0.0170 (6)
C3	0.4575 (4)	0.2677 (3)	1.00353 (14)	0.0259 (7)
C4	0.4497 (4)	0.2351 (3)	1.07878 (14)	0.0205 (7)
N	0.5907 (3)	0.3252 (3)	1.12617 (11)	0.0200 (5)
O1*W*	0.6887 (3)	0.0728 (2)	0.68799 (10)	0.0256 (5)
O2*W*	0.4481 (3)	0.2423 (2)	0.59175 (11)	0.0349 (6)
O3*W*	0.4328 (3)	0.1189 (3)	0.45162 (12)	0.0423 (6)

Table 2. *Selected geometric parameters (Å, °)*

Na—O1*W*	2.288 (2)	P2—O22	1.504 (2)
Na—O23	2.335 (2)	P2—O23	1.506 (2)
Na—O22$^{\rm i}$	2.355 (2)	P2—O21	1.579 (2)
Na—O13	2.557 (2)	P2—C1	1.854 (3)

Na—O12	2.565 (2)	O1—C1	1.443 (3)
Na—O1^{i}	2.580 (2)	C1—C2	1.542 (4)
P1—O12	1.498 (2)	C2—C3	1.528 (4)
P1—O11	1.502 (2)	C3—C4	1.508 (4)
P1—O13	1.576 (2)	C4—N	1.489 (4)
P1—C1	1.860 (3)		
O12—P1—O11	117.2 (1)	O21—P2—C1	106.5 (1)
O12—P1—O13	106.0 (1)	O1—C1—C2	111.0 (2)
O11—P1—O13	111.3 (1)	O1—C1—P2	107.8 (2)
O12—P1—C1	110.3 (1)	C2—C1—P2	111.5 (2)
O11—P1—C1	106.0 (1)	O1—C1—P1	105.1 (2)
O13—P1—C1	105.4 (1)	C2—C1—P1	111.2 (2)
O22—P2—O23	116.8 (1)	P2—C1—P1	110.0 (1)
O22—P2—O21	110.9 (1)	C3—C2—C1	113.9 (2)
O23—P2—O21	106.6 (1)	C4—C3—C2	112.0 (2)
O22—P2—C1	108.4 (1)	N—C4—C3	110.7 (2)
O23—P2—C1	107.3 (1)		

Symmetry code: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z$.

Table 3. *Hydrogen-bonding geometry (Å, °)*

Normalized values are according to Jeffrey & Lewis (1978) and Taylor & Kennard (1983).

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1WA···O11^{i}	0.94	1.76	2.687 (3)	169
O1W—H1WB···O22^{ii}	0.94	1.87	2.767 (2)	159
O2W—H2WA···O1W	0.94	1.84	2.777 (3)	176
O2W—H2WB···O21^{iii}	0.94	2.04	2.945 (3)	163
O3W—H3WA···O11^{iv}	0.94	1.81	2.735 (3)	168
O3W—H3WB···O2W	0.94	2.00	2.932 (3)	172
N—HC···O11^{v}	1.03	1.96	2.866 (3)	145
N—HC···O1^{v}	1.03	2.33	3.023 (3)	124
N—HA···O2W^{vi}	1.03	1.84	2.868 (3)	172
N—HB···O22^{vi}	1.03	1.87	2.879 (3)	166
O1—H1···O3W^{vii}	0.94	1.74	2.677 (3)	173
O21—H21···O12^{viii}	0.94	1.70	2.619 (2)	164
O13—H13···O23^{iii}	0.94	1.59	2.520 (2)	170

Symmetry codes: (i) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$; (ii) $1+x, y, z$; (iii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (iv) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (v) $1 - x, 1 - y, 2 - z$; (vi) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; (vii) $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$; (viii) $x - 1, y, z$.

Data collection: *P3/P4-PC Diffractometer Program* (Siemens, 1991). Cell refinement: *P3/P4-PC Diffractometer Program.* Data reduction: *XDISK* in *SHELXTL/PC* (Sheldrick, 1991). Program(s) used to solve structure: *XS* in *SHELXTL/PC*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *XP* in *SHELXTL/PC*. Software used to prepare material for publication: *CIFTAB* in *SHELXL93*, *PARST* (Nardelli, 1983), *CSD* (Allen, Kennard & Taylor, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and indexed X-ray powder diffraction peaks have been deposited with the IUCr (Reference: FG1158). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(acetato-*O*)bis(pyridine-*N*)palladium(II) Monohydrate and Bis(acetato-*O*)bis(diethylamine-*N*)palladium(II)

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Abstract

The title complexes, $[Pd(C_2H_3O_2)_2(C_5H_5N)_2].H_2O$, (I), and $[Pd(C_2H_3O_2)_2(C_4H_{11}N)_2]$, (II), are typical of metal acetates with additional N-donor coordination. In both structures, the central Pd atom has a centrosymmetric square-planar environment. In the pyridine complex (I), carbonyl O atoms are involved in intermolecular O—H···O hydrogen bonds with water molecules and in the amine complex (II), they participate in intramolecular N—H···O hydrogen bonds. Owing to the latter, the Pd—N bond lengths are different with values of 2.010 (4) for complex (I) and 2.066 (2) Å for complex (II).

Comment

In the course of our work on the relationship between the geometry of molecular complexes and

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