Crystal structure of (1-hydroxy-1-phosphono-pentyl)-phosphonic acid dimethyl ammonium salt, $C_7H_{21}NO_7P_2$, and of (1,8-dihydroxy-1,8,8-tris-phosphono-octyl)-phosphonic acid bis-dimethylammonium salt tetrahydrate, $C_{12}H_{36}N_2O_{14}P_4 \cdot 4H_2O$, *evidence for trapped alcaline species by bisphosphonic and tetraphosphonic acids in the crystalline state*

A. Neuman, M. Coindet-Benramdane, H. Gillier, Y. Leroux, D. El Manouni and T. Prangé*

Université Paris-Nord, Chimie Structurale et Spectroscopie Biomoléculaire (UMR 7033 CNRS), UFR-SMBH, 74 rue M. Cachin, F-93017 Bobigny Cedex, France

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Abstract

$C_7H_{21}NO_7P_2$, triclinic, $P\bar{1}$ (No. 2), $a = 11.404(2)$ Å, $b = 10.426(2)$ Å, $c = 6.199(2)$ Å, $\alpha = 92.1(1)°$, $\beta = 95.9(1)°$, $\gamma = 110.5(1)°$, $V = 684.9$ Å^3, $Z = 2$, $R_{gt}(F) = 0.061$, $wR_{ref}(F^2) = 0.160$, $T = 291$ K.

$C_{12}H_{44}N_2O_{18}P_4$, triclinic, $P\bar{1}$ (No. 2), $a = 10.052(2)$ Å, $b = 9.336(2)$ Å, $c = 8.289(2)$ Å, $\alpha = 98.4(1)°$, $\beta = 104.9(1)°$, $\gamma = 106.6(1)°$, $V = 699.9$ Å^3, $Z = 1$, $R_{gt}(F) = 0.084$, $wR_{ref}(F^2) = 0.209$, $T = 291$ K.

Source of material

The syntheses of the acids $C_5H_{14}O_7P_2$ and $C_8H_{22}O_{14}P_4$ have already been reported [1,2]. Crystallizations were done by slow evaporation from methanolic solutions at room temperature: 0.1 g of $C_5H_{14}O_7P_2$ or $C_8H_{22}O_{14}P_4$ were dissolved in 25 ml of methanol:water (1:1). By slow evaporation, only oily products were obtained. If a separate solution of dimethylamine is set in the vicinity of the crystallization pot, the dimethylamonium salts ($C_7H_{21}NO_7P_2$, $C_{12}H_{36}N_2O_{14}P_4 \cdot 4H_2O$) shortly crystallize as large prisms.

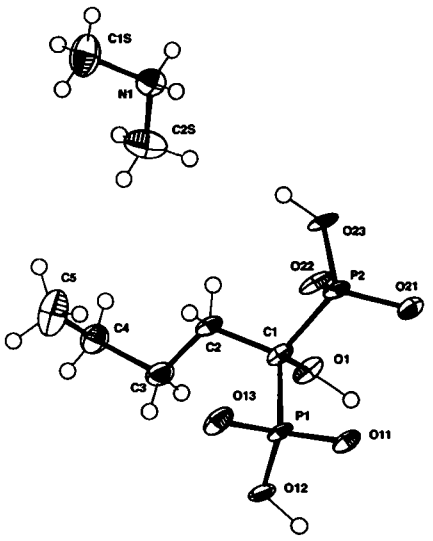
Experimental details

All H atoms were initially calculated at idealized positions and refined with C–H and O–H distances restraints. An isotropic displacement parameter was set for each hydrogen in HBPA and BHTP, riding 1.2 times of the value for the bonded atom.

Discussion

The hydroxy-bisphosphonic acid HBPA and the bishydroxy-tetraphosphonic acid BHTP are very efficient compounds for complexing metallic ions and for their transport properties in biological media [3-5]. The wide application of HBPA and BHTP ranges from their use in bone scintigraphy [6] to the extraction of trans actinides in nuclear industry [7]. In addition, several amino substituted hydroxy-bis phosphonic acids are under evaluation in medicinal treatment of bone deseases as they strongly interact and regulate the transport of calcium [3,8]. Most of these biological active bis-acids have an extended alkyl-amino chain linked to the central carbon. In this respect, the crystal structures of these uncomplexed amino phosphonic acids are zwitterionic species like amino-acids. HBPA and BHTP without amino function at C(1) show superacid properties [9,10]. These compounds as free acids have poor crystalline properties although a number of structures have been reported [10-13]. In this communication, it is given evidence that they can trap and stabilize cationic species derived from volatile alcaline compounds like dimethylamine to give particularly stable crystals. A possible catalytic intermolecular dehydration of methanol, the solvent used in crystallizations, cannot be ruled out because of the superacid properties of the title compounds. This will lead to a dimethyloxonium cation [14] with a similar density signature than dimethylamonium. To confirm the nature of the central atom of the cation (N or O) and for accurate hydrogen bon network location, a neutron study on HBPA has been undertaken. This study will be published elsewhere [15].

1. (1-Hydroxy-1-phosphono-pentyl)-phosphonic acid dimethyl ammonium salt, $C_7H_{21}NO_7P_2$



* Correspondence author (e-mail: prange@lure.u-psud.fr)

Table 1. Data collection and handling.

Crystal:	colourless, irregular, size 0.2 x 0.2 x 0.3 mm
Wavelength:	Cu K_α radiation (1.54180 Å)
μ:	31.26 cm^{-1}
Diffractometer, scan mode:	Philips PW1100, $\theta/2\theta$
$2\theta_{max}$:	128.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1790, 1790
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1776
$N(param)_{refined}$:	163
Programs:	SHELXS-86 [16], SHELXL-97 [17], Xtal-GX [18]

Table 2. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{iso}
H(12O)	2i	0.324(4)	-0.139(3)	0.082(7)	0.030
H(23O)	2i	0.490(4)	0.487(3)	-0.209(7)	0.029
H(1O)	2i	0.425(3)	0.030(4)	-0.277(7)	0.024
H(21)	2i	0.2832	0.2936	0.0032	0.025
H(22)	2i	0.2846	0.2838	-0.2487	0.025
H(31)	2i	0.1407	0.0507	-0.2570	0.037
H(32)	2i	0.1292	0.0800	-0.0118	0.037
H(41)	2i	0.0573	0.2214	-0.3511	0.045
H(42)	2i	-0.0390	0.1019	-0.2438	0.045
H(51)	2i	0.1022	0.3076	0.0595	0.077
H(52)	2i	-0.0451	0.2392	0.0191	0.077
H(53)	2i	0.0251	0.3596	-0.1184	0.077
H(1N1)	2i	0.6549	0.3360	0.3705	0.034
H(2N1)	2i	0.6845	0.3101	0.5947	0.034
H(1S1)	2i	0.7432	0.1731	0.3719	0.060
H(1S2)	2i	0.8621	0.2756	0.5171	0.060
H(1S3)	2i	0.8309	0.3044	0.2755	0.060
H(2S1)	2i	0.7348	0.5366	0.5724	0.066
H(2S2)	2i	0.8249	0.5315	0.4002	0.066
H(2S3)	2i	0.8566	0.5035	0.6421	0.066

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P(1)	2i	0.38297(8)	0.0808(1)	0.1529(1)	0.0287(5)	0.0162(7)	0.0107(5)	0.0147(4)	-0.0002(4)	0.0059(4)
P(2)	2i	0.53859(8)	0.3216(1)	-0.0715(1)	0.0285(5)	0.0121(7)	0.0159(5)	0.0124(4)	-0.0006(4)	0.0057(5)
O(12)	2i	0.2869(2)	-0.0661(3)	0.0891(4)	0.029(1)	0.014(2)	0.033(2)	0.011(1)	0.001(1)	0.009(1)
O(11)	2i	0.5106(2)	0.0840(3)	0.2379(4)	0.032(1)	0.020(2)	0.019(1)	0.017(1)	-0.004(1)	0.004(1)
O(13)	2i	0.3230(3)	0.1495(3)	0.3151(4)	0.047(2)	0.036(2)	0.015(1)	0.030(2)	0.004(1)	0.006(1)
O(21)	2i	0.6384(2)	0.2673(3)	-0.1274(4)	0.031(1)	0.018(2)	0.031(1)	0.017(1)	0.006(1)	0.010(1)
O(22)	2i	0.5616(2)	0.3980(3)	0.1491(4)	0.042(2)	0.018(2)	0.019(1)	0.020(1)	-0.007(1)	-0.000(1)
O(23)	2i	0.5233(3)	0.4147(3)	-0.2589(4)	0.043(2)	0.019(2)	0.019(1)	0.021(1)	0.004(1)	0.012(1)
O(1)	2i	0.3740(2)	0.0915(3)	-0.2838(4)	0.038(1)	0.018(2)	0.009(1)	0.020(1)	-0.005(1)	-0.002(1)
C(1)	2i	0.3851(3)	0.1787(4)	-0.0912(5)	0.031(2)	0.012(2)	0.011(2)	0.016(2)	-0.000(1)	0.001(2)
C(2)	2i	0.2759(3)	0.2313(4)	-0.1215(6)	0.030(2)	0.018(2)	0.019(2)	0.014(2)	-0.001(1)	0.006(2)
C(3)	2i	0.1444(4)	0.1219(5)	-0.1484(7)	0.030(2)	0.026(3)	0.039(2)	0.013(2)	-0.001(2)	0.011(2)
C(4)	2i	0.0409(4)	0.1779(5)	-0.2161(8)	0.029(2)	0.040(3)	0.046(3)	0.016(2)	-0.002(2)	0.013(2)
C(5)	2i	0.0298(5)	0.2804(7)	-0.049(1)	0.051(3)	0.083(5)	0.072(4)	0.043(3)	0.005(3)	0.000(4)
N(1)	2i	0.7185(3)	0.3460(4)	0.4759(5)	0.030(2)	0.033(2)	0.021(2)	0.011(2)	-0.003(1)	0.006(2)
C(1S)	2i	0.7954(4)	0.2680(6)	0.4038(9)	0.037(2)	0.062(4)	0.050(3)	0.023(3)	-0.008(2)	-0.011(3)
C(2S)	2i	0.7898(5)	0.4918(5)	0.527(1)	0.060(3)	0.033(3)	0.061(3)	0.003(3)	0.008(3)	0.010(3)

2. (1,8-Dihydroxy-1,8,8-tris-phosphono-octyl)-phosphonic acid bis-dimethylammonium salt tetrahydrate, $C_{12}H_{36}N_2O_{14}P_4 \cdot 4H_2O$

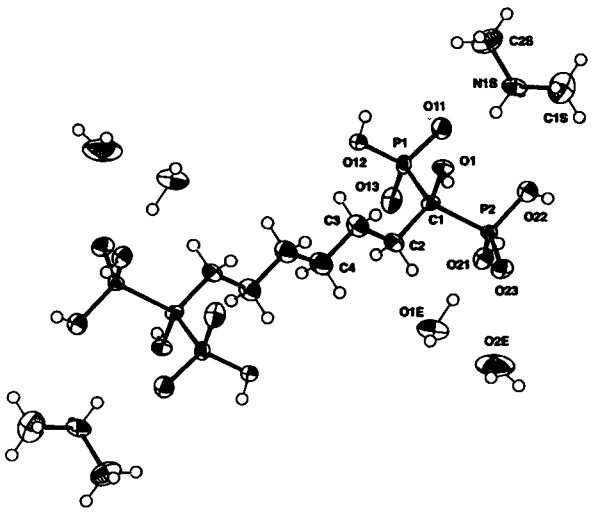


Table 4. Data collection and handling.

Crystal:	colourless, irregular, size 0.15 x 0.30 x 0.60 mm
Wavelength:	Cu K_α radiation (1.54180 Å)
μ:	32.07 cm^{-1}
Diffractometer, scan mode:	Philips PW1100, $\theta/2\theta$
$2\theta_{max}$:	123.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1538, 1538
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1520
$N(param)_{refined}$:	182
Programs:	SHELXS-86 [16], SHELXL-97 [17], Xtal-GX [18]

Table 5. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{iso}
H(12)	2*i*	0.2980	0.3702	0.2125	0.032
H(21)	2*i*	0.1214	0.4979	0.7642	0.043
H(22)	2*i*	-0.0936	0.3604	0.5749	0.037
H(1)	2*i*	0.0664	0.0737	0.3438	0.027
H(21C)	2*i*	0.3932	0.2914	0.6527	0.038
H(22C)	2*i*	0.2733	0.1443	0.6587	0.038
H(1C3)	2*i*	0.2737	0.0153	0.3953	0.038
H(2C3)	2*i*	0.3903	0.1630	0.3848	0.038
H(1C4)	2*i*	0.5610	0.1420	0.6143	0.044
H(2C4)	2*i*	0.4450	0.0006	0.6362	0.044
H(1NS)	2*i*	0.8657	0.3642	0.1982	0.036
H(2NS)	2*i*	0.9138	0.2315	0.1992	0.036
H(1S1)	2*i*	0.6301	0.2048	0.0495	0.068
H(2S1)	2*i*	0.6877	0.1782	0.2333	0.068
H(3S1)	2*i*	0.6859	0.0661	0.0717	0.068
H(1S2)	2*i*	0.8058	0.3187	-0.0911	0.060
H(2S2)	2*i*	0.8602	0.1772	-0.0886	0.060
H(3S2)	2*i*	0.9719	0.3445	-0.0062	0.060
H(1E)	2*i*	0.63(1)	0.598(3)	0.98(2)	0.142
H(2E)	2*i*	0.69(1)	0.48(1)	1.05(1)	0.142
H(3E)	2*i*	0.14(2)	-0.006(6)	-0.01(2)	0.157
H(4E)	2*i*	0.12(2)	0.14(1)	0.10(1)	0.157

Table 6. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P(1)	2*i*	0.2683(2)	0.4427(2)	0.4270(3)	0.021(1)	0.022(1)	0.030(2)	0.0108(9)	0.013(1)	0.010(1)
P(2)	2*i*	0.0752(2)	0.2905(2)	0.6310(3)	0.018(1)	0.019(1)	0.014(2)	0.0064(8)	0.004(1)	0.003(1)
O(13)	2*i*	0.3722(6)	0.5623(6)	0.5957(8)	0.032(3)	0.026(3)	0.037(5)	0.003(3)	0.013(4)	-0.006(3)
O(12)	2*i*	0.3531(5)	0.4056(6)	0.3116(7)	0.022(3)	0.038(3)	0.021(4)	0.010(2)	0.006(3)	0.011(3)
O(11)	2*i*	0.1473(5)	0.4981(5)	0.3460(7)	0.033(3)	0.028(3)	0.040(5)	0.016(2)	0.015(3)	0.013(3)
O(21)	2*i*	0.1579(6)	0.4307(5)	0.7783(7)	0.035(3)	0.024(3)	0.030(5)	0.014(2)	0.015(3)	0.001(3)
O(23)	2*i*	0.0379(5)	0.1421(5)	0.6954(7)	0.034(3)	0.021(3)	0.033(5)	0.003(2)	0.011(3)	0.014(3)
O(22)	2*i*	-0.0639(6)	0.3052(6)	0.5173(7)	0.037(3)	0.035(3)	0.025(5)	0.022(3)	0.012(4)	-0.002(3)
O(1)	2*i*	0.0886(6)	0.1624(5)	0.3324(8)	0.028(3)	0.017(2)	0.017(5)	0.006(2)	0.002(3)	0.001(3)
C(1)	2*i*	0.1873(8)	0.2638(8)	0.491(1)	0.028(5)	0.021(4)	0.011(7)	0.011(4)	0.007(6)	-0.001(5)
C(2)	2*i*	0.3087(9)	0.2037(9)	0.582(1)	0.031(5)	0.042(4)	0.032(7)	0.024(4)	0.010(5)	0.017(5)
C(3)	2*i*	0.3575(9)	0.1048(9)	0.463(1)	0.035(5)	0.027(4)	0.035(7)	0.016(4)	0.009(5)	0.004(4)
C(4)	2*i*	0.476(1)	0.052(1)	0.552(1)	0.040(5)	0.042(4)	0.035(8)	0.026(4)	0.010(6)	0.006(5)
N(1S)	2*i*	0.8490(7)	0.2688(7)	0.1385(9)	0.034(4)	0.021(3)	0.017(6)	0.000(3)	-0.006(4)	-0.004(3)
C(1S)	2*i*	0.700(1)	0.171(1)	0.122(2)	0.050(6)	0.044(5)	0.060(9)	-0.002(5)	0.019(7)	-0.002(6)
C(2S)	2*i*	0.874(1)	0.278(1)	-0.026(1)	0.076(7)	0.043(5)	0.036(8)	0.022(5)	0.025(7)	0.007(5)
O(1E)	2*i*	0.6139(9)	0.488(1)	0.967(1)	0.075(6)	0.18(1)	0.046(7)	0.060(7)	0.014(5)	0.055(7)
O(2E)	2*i*	0.147(1)	0.103(1)	0.003(1)	0.141(9)	0.18(1)	0.026(6)	0.114(9)	0.022(6)	0.022(7)

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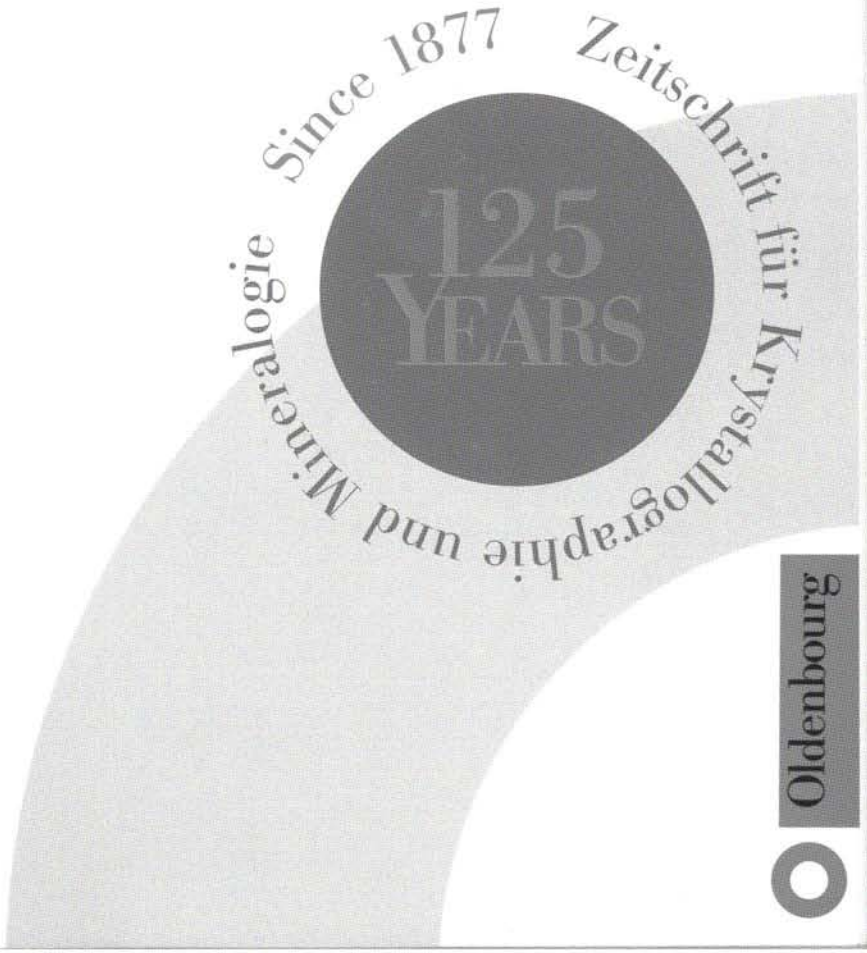

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Editor in chief:
Prof. W. Steurer

Managing Editor:
Dr. Yuri Grin.
Fax: +49-3 51-46 46-33 40
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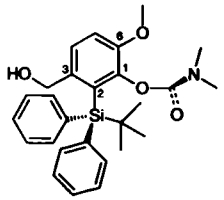

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Further details of the structure determination can be accessed at the relevant database:
Data with CSD-numbers can be obtained from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-76344 Eggenstein-Leopoldshafen, Germany (e-mail: docdel@fiz-karlsruhe.de).
Data with CCDC-numbers can be obtained from the Cambridge Crystallographic Data Centre (CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, (fax: +44-(0)1223-33 60 33 or e-mail: deposit@chemcrys.cam.ac.uk).