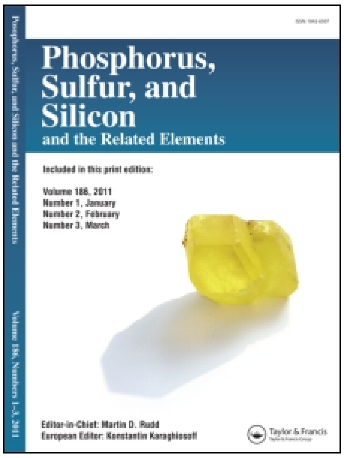

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Structural and Physicochemical Studies of [2,3-$(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$

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Structural and Physicochemical Studies of $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$

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Physicochemical properties of a new organic cation triphosphate, $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$, are discussed on the basis of X-ray crystal structure investigation. This compound crystallizes in a triclinic unit cell P1 with the parameters: $a = 11.963(6)$ Å, $b = 9.858(6)$ Å, $c = 16.205(7)$ Å, $\alpha = 95.50(4)^\circ$, $\beta = 95.21(4)^\circ$, $\gamma = 89.07(4)^\circ$, $V = 1894.4(16)$ Å^3, $Z = 2$, and $D_x = 1.365$ g.cm^{-3}. Crystal structure has been solved and refined to $R = 0.074$ using 3766 independent reflections. In this atomic arrangement, layers built by $HP_3O_{10}^{4-}$ groups are developed in (a, b) planes at $z = 1/2$. Between these layers the 2,3-dimethylphenylammonium entities are located, which form hydrogen bonds with some external atoms of the phosphoric groups. A characterization of this compound by solid-state ^{13}C and ^{31}P MAS NMR, IR spectroscopy and thermal analysis is also reported.

Keywords Crystal structure; hybrid compound; synthesis; triphosphate

INTRODUCTION

Since the preparation and the identification of $Na_5P_3O_{10}$,[1] this salt has been used as starting material to prepare other triphosphates. Depending on the cation associated with the phosphate anion, such compounds can have interesting structures and properties, potentially useful in various application fields (non-linear optics,[2] heterogeneous catalysis,[3] photochemical and photo physical processes,[4] molecular sieves,[5] and other areas that include electronic materials[6] and ceramic precursors[7]). In the present work, the chemical preparation, crystal structure and

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characterization by IR spectroscopy, NMR, and thermal analysis of a new organic cation triphosphate, $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$, are reported.

RESULTS AND DISCUSSION

Structure Description

The final atomic coordinates of all non hydrogen atoms of $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$ their B equivalent temperature factors are given in Table I. Those hydrogen atoms have also been determined but are not given to keep the table short. The asymmetric unit of this hybrid compound contains four independent 2,3-dimethylanilinium cations, one monohydrogentriphosphate anion and two water molecules (Figure 1). Its atomic arrangement is characterized by the existence of inorganic layers, built by $HP_3O_{10}^{4-}$ anions and the water molecules and parallel to the (a, b)plane at z = ½ (Figure 2). In such a layer, two $HP_3O_{10}^{-4}$ anions are held together by strong O—H···O hydrogen bonds ($d_{O\cdots O}$ = 2.554(6) Å) to form $H_2P_6O_{20}^{8-}$ cyclic units. Each one of these layers is interconnected by the water molecules to form an infinite layer parallel to the (a, b) plane (Figure 3).

In this triclinic unit-cell, the $HP_3O_{10}^{4-}$ group has no internal symmetry. It is built by three independent PO_4 tetrahedra, $P(1)O_4$, $P(2)O_4$, and $P(3)O_4$. Such geometry has been observed in other organic triphosphates, $[2\text{-}C_2H_5C_6H_4NH_3]_3H_2P_3O_{10}^{8}$ and $[3,5(CH_3O)_2C_6H_3NH_3]_3H_2P_3O_{10}$.[9] The main interatomic distances and angles in the $HP_3O_{10}^{4-}$ anion are given in Table II.

The four independent cations $[2,3\text{-}(CH_3)_2C_6H_3NH_3]^+$ establish hydrogen bonds involving the hydrogen atoms of NH_3 groups and some external oxygen atoms of $HP_3O_{10}^{4-}$ anions with distances $d_{N\cdots O}$ ranging from 2.674(6) to 3.056(6) Å (Table III). In these organic molecules, the carbon atoms of the aromatic rings, C_1, C_2, C_3, C_4,C_5, and C_6 (ring A); C_9, C_{10}, C_{11}, C_{12}, C_{13}, and C_{14} (ring B); C_{17}, C_{18}, C_{19}, C_{20}, C_{21}, and C_{22} (ring C) and C_{25}, C_{26}, C_{27}, C_{28},C_{29}, and C_{30} (ring D), are planar (r.m.s. deviation of fitted atoms equal to 0.0230 Å for ring A; 0.0076 Å for ring B; 0.0090 Å for ring C and 0.0123 Å for ring D). The dihedral angles (A,B); (A,C); (A,D); (B,C); (B,D) and (C,D) between these four rings are 43.23°; 71.28°; 84.01°; 114.33°; 127.24°, and 14.01°, respectively. The C···C interplanar distances range from 3.38 Å to 5.20 Å, indicating the existence of more or less Van der Waals interactions, between these organic molecules. These interactions participate with hydrogen and electrostatic bonds to the cohesion of the three-dimensional network.

TABLE I Final Atomic Coordinates and B_{eq} (Å^2) for the Nonhydrogen Atoms

Atoms	x(σ)	y(σ)	z(σ)	B_{eq}.
P(1)	0.11452(9)	0.80011(10)	0.47211(5)	1.70(2)
P(2)	0.26186(8)	0.59749(11)	0.54949(5)	1.65(2)
P(3)	0.25494(8)	0.31329(11)	0.47790(5)	1.64(2)
O(E11)	0.0419(3)	0.8259(3)	0.5474(2)	2.52(7)
O(E12)	0.1993(3)	0.9078(3)	0.4810(2)	2.56(7)
O(E13)	0.0439(2)	0.7748(3)	0.3919(1)	2.28(7)
O(E21)	0.3600(2)	0.6897(3)	0.5661(1)	2.34(6)
O(L12)	0.1746(3)	0.6548(3)	0.4818(1)	2.21(6)
O(E22)	0.2037(3)	0.5622(3)	0.6206(2)	2.68(7)
O(L23)	0.3052(2)	0.4676(3)	0.4969(1)	2.03(6)
O(E31)	0.3074(3)	0.2637(3)	0.3992(2)	2.59(7)
O(E32)	0.2988(3)	0.2378(3)	0.5509(2)	2.38(7)
O(E33)	0.1296(3)	0.3283(3)	0.4662(2)	2.66(7)
O(W1)	0.5348(3)	0.3054(4)	0.5874(2)	3.39(8)
O(W2)	0.5743(3)	0.0106(5)	0.4240(2)	4.59(10)
N(1)	0.1584(3)	0.0986(4)	0.6300(2)	2.00(8)
N(2)	0.3420(3)	−0.0063(4)	0.3713(2)	2.32(9)
N(3)	0.4633(3)	0.4179(4)	0.3422(2)	2.23(8)
N(4)	0.9882(3)	0.5158(4)	0.6457(2)	2.03(8)
C(1)	0.1954(5)	0.0653(10)	0.7104(4)	4.5(2)
C(2)	0.1901(6)	0.1330(9)	0.7776(5)	4.2(2)
C(3)	0.2331(6)	0.0956(7)	0.8567(3)	4.1(2)
C(4)	0.2804(7)	−0.0363(11)	0.8569(5)	6.2(2)
C(5)	0.2899(10)	−0.1067(10)	0.7838(6)	5.5(2)
C(6)	0.2593(6)	−0.0759(7)	0.7154(4)	3.7(2)
C(7)	0.1257(5)	0.2642(7)	0.7706(3)	4.4(2)
C(8)	0.2147(9)	0.1849(11)	0.9285(5)	7.2(3)
C(9)	0.3239(4)	−0.0536(5)	0.2829(2)	2.54(11)
C(10)	0.4135(6)	−0.1150(7)	0.2458(3)	3.8(2)
C(11)	0.3982(8)	−0.1598(8)	0.1618(3)	5.3(2)
C(12)	0.2981(7)	−0.1370(6)	0.1179(3)	4.6(2)
C(13)	0.2088(6)	−0.0758(6)	0.1542(3)	4.0(2)
C(14)	0.2211(5)	−0.0303(5)	0.2396(3)	2.92(12)
C(15)	0.1255(5)	0.0422(7)	0.2814(3)	4.0(1)
C(16)	0.1009(7)	−0.0497(10)	0.1028(3)	6.9(2)
C(17)	0.4648(4)	0.3776(5)	0.2526(2)	2.40(11)
C(18)	0.3979(5)	0.2708(6)	0.2184(3)	3.4(1)
C(19)	0.4037(7)	0.2236(7)	0.1374(3)	4.4(2)
C(20)	0.4760(5)	0.2880(6)	0.0900(3)	3.9(1)
C(21)	0.5415(5)	0.3930(6)	0.1224(3)	3.25(13)
C(22)	0.5369(4)	0.4461(5)	0.2067(3)	2.54(11)
C(23)	0.6047(5)	0.5624(6)	0.2445(3)	3.70(13)
C(24)	0.6198(6)	0.4571(8)	0.0697(3)	5.0(2)
C(25)	0.9799(4)	0.5611(5)	0.7346(2)	2.18(10)

TABLE I Final Atomic Coordinates and B_{eq} ($Å^2$) for the Nonhydrogen Atoms *(Continued)*

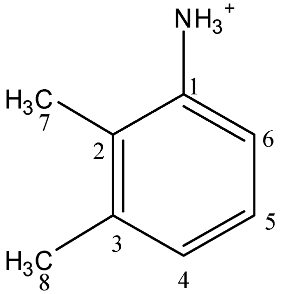
Atoms	x(σ)	y(σ)	z(σ)	B_{eq}.
C(26)	0.8945(4)	0.5146(5)	0.7756(2)	2.36(11)
C(27)	0.8966(5)	0.5528(6)	0.8611(3)	3.15(13)
C(28)	0.9798(5)	0.6356(7)	0.9001(3)	4.1(1)
C(29)	1.0614(6)	0.6863(8)	0.8563(3)	4.8(2)
C(30)	1.0628(5)	0.6460(6)	0.7730(3)	3.22(12)
C(31)	0.8045(4)	0.4220(6)	0.7320(3)	3.49(12)
C(32)	0.8077(6)	0.4972(7)	0.9114(3)	4.8(2)

Estimated standard deviations are given in parentheses.

NMR Results

Figure 4 shows the proton decoupled ^{31}P MAS-NMR spectrum of crystalline triphosphate $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$. This spectrum is in good agreement with the X-ray structure. Indeed, it exhibits three resonance peaks at −6.5, −9.2, and −20.5 ppm, with their corresponding satellite spinning side bands. These NMR components are related to the three crystallographically unequivalent sites which exist in the atomic arrangement. The chemical shift values are in full agreement with those of triphosphates.[8,10]

The ^{13}C CP MAS spectrum of the title compound is given on Fig. 5. To assign NMR components to different carbon atoms, we have calculated the isotropic chemical shift values δ_{iso} by means of the ChemDraw Ultra 10.0 software. The carbon atoms are labeled as depicted below:



The obtained result is: $\delta_{iso}(C7) = 14.2$ and $\delta_{iso}(C8) = 18.8$ ppm, while the aromatic carbon atoms chemical shift are gathered in Table IV. However, the spectrum exhibits three resonance regions. The first one, whose chemical shift peaks are 12.0, 13.8 and 14.8 ppm, is related to the methyl groups attached to the C2 phenyl ring carbon atom. Peaks

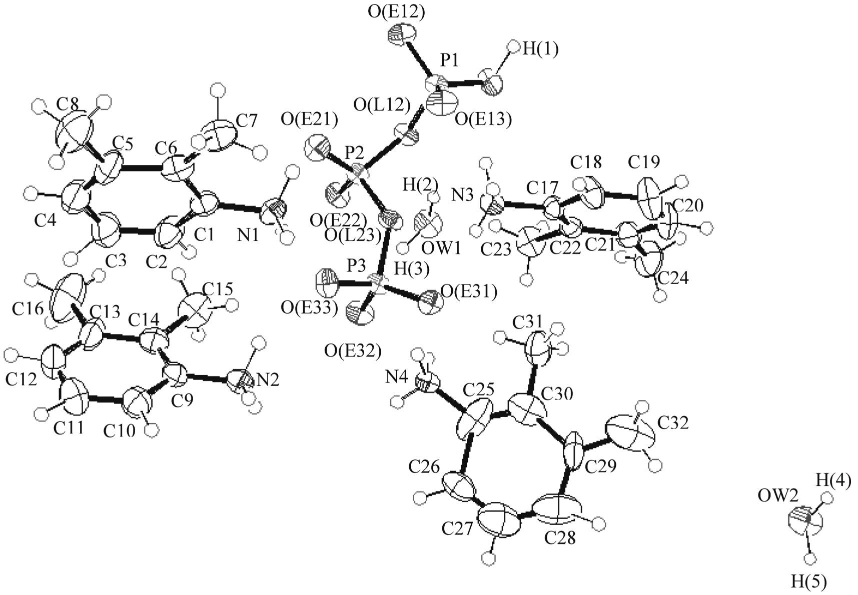


FIGURE 1 Representation ORTEP of the asymmetric unit of [2,3-$(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$. Thermal ellipsoids are given at 45% probability.

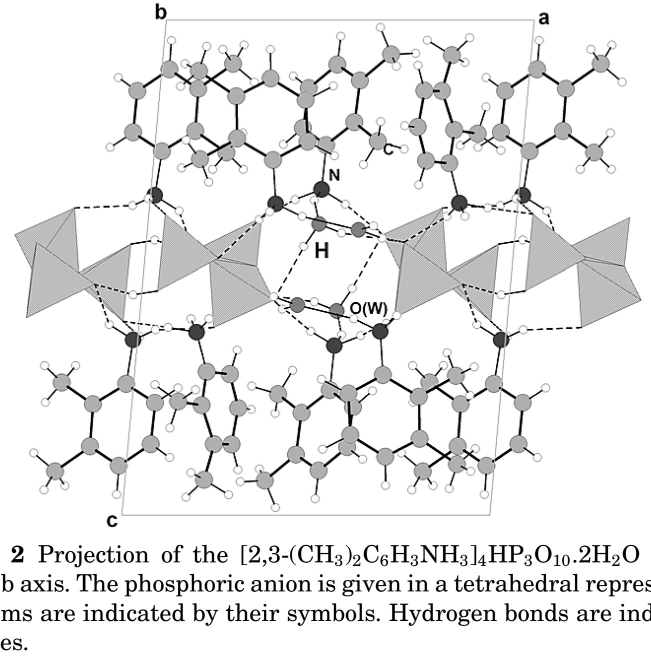


FIGURE 2 Projection of the [2,3-$(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$ structure along the b axis. The phosphoric anion is given in a tetrahedral representation. Other atoms are indicated by their symbols. Hydrogen bonds are indicated by dotted lines.

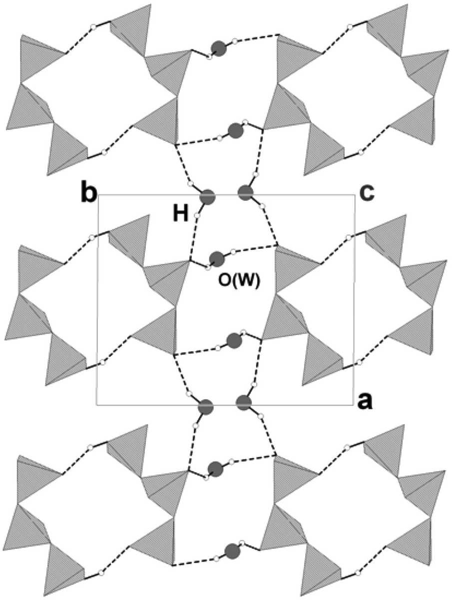


FIGURE 3 Representation of an inorganic layer in the $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$ structure. The phosphoric anion is given in polyhedral representation and labels are given for the other atoms. Hydrogen bonds are denoted by dotted line.

ranging from 20.3 to 22.9 ppm correspond to the carbon atom of the methyl groups in meta position of the amino groups of the four crystallographically independent organic cations. This spectral area is very significant. Indeed, the presence of three resonance peaks with a shoulder for the one at 20.3 ppm proves the existence of four organic cations in the unit cell of the compound which agree perfectly with the single crystal X-ray diffraction result. The most shifted NMR lines, whose chemical shifts ranging from 122.4 to 137.4 ppm, are attributed to the aromatic carbon atoms. The presence of this reduced number of NMR components in this spectral zone can be explained by the similar neighbouring of the four different organic groups in the unit cell. The attribution attempts for the aromatic carbon atoms of an organic entity are reported in Table IV.

Thermal Analysis

The two curves corresponding to DTA and TGA analysis in open air are given in (Fig. 6). The DTA curve shows a succession of endothermic

TABLE II Main Interatomic Distances (Å) and Bond Angles (°) in the $HP_3O_{10}^{4-}$ Anion

Tetrahedron $P(1)O_4$				
P(1)	O(E11)	O(E12)	O(E13)	O(L12)
O(E11)	1.560(4)	107.1(2)	111.8(2)	105.6(2)
O(E12)	2.437(5)	1.468(2)	119.1(2)	109.8(2)
O(E13)	2.523(5)	2.547(4)	1.486(4)	102.5(2)
O(L12)	2.523(6)	2.518(5)	2.412(5)	1.607(4)
Tetrahedron $P(2)O_4$				
P(2)	O(L12)	O(E21)	O(E22)	O(L23)
O(L12)	1.582(4)	109.6(2)	109.9(3)	99.8(2)
O(E21)	2.508(6)	1.486(4)	118.3(3)	105.3(2)
O(E22)	2.501(5)	2.539(6)	1.471(4)	112.3(2)
O(L23)	2.415(6)	2.434(6)	2.532(5)	1.576(4)
Tetrahedron $P(3)O_4$				
P(3)	O(L23)	O(E31)	O(E32)	O(E33)
O(L23)	1.636(4)	101.6(2)	105.9(2)	106.4(2)
O(E31)	2.438(6)	1.509(4)	112.5(2)	113.8(2)
O(E32)	2.510(6)	2.507(5)	1.507(4)	115.2(2)
O(E33)	2.514(6)	2.521(6)	2.539(6)	1.501(4)
P(1)-P(2)	2.938(3)	P(2)-P(3)	2.927(3)	
P(1)-O(L12)-P(2)	134.3(2)	P(2)-O(L23)-P(3)	131.4(2)	
P(1)-O(E11)-H(1)	131.4	P(1)-P(2)-P(3)	119.44(7)	

Estimated standard deviations are given in parentheses.

peaks. The first peak, at 120°C, corresponds to an elimination of the crystallization water molecules, well-confirmed by the weight loss observed on TGA curve (% water experimental 4.76, calculated 4.62). After this dehydration, the organic entity of the anhydrous compound begins to decompose in a wide temperature range by forming a viscous matter of polyphosphoric acids and a black residue of carbon.

IR Spectroscopy

The infrared absorption spectrum of crystalline triphosphate $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$ is shown in Fig. 7. To assign the IR peaks to vibrational modes, we examined the modes and frequencies observed in similar compounds.[8]

- Frequencies in the range 3600 and 2300 cm^{-1} are attributed to the stretching of the organic and hydroxyl groups ν (N—H), ν(C—H), and ν (O—H) of H_2O molecules and P—OH groups.

TABLE III Hydrogen Bond Scheme in $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$

D(N)–H···O	D–H(A°)	H...A(A°)	D–H···(A°)	D...A(A°)
O(E11)–H(1)···O(E33)	0.85	1.78	150.0	2.554(7)
O(W1)-H(2)···O(E32)	0.85	2.08	161.2	2.906(6)
O(W1)–H(3)···O(E21)	1.00	2.00	147.5	2.893(5)
O(W2)–H(4)···O(E32)	1.04	1.95	150.2	2.902(7)
O(W2)–H(5)···O(E21)	1.01	2.10	154.43	3.056(6)
N(1)-H(6)···O(E12)	0.87	2.33	131.5	2.987(2)
N(1)-H(7)···O(E13)	1.02	1.70	173.1	2.715(6)
N(1)-H(8)···O(E32)	0.87	1.84	158.2	2.674(6)
N(2)-H(9)···O(E12)	0.96	1.88	154.5	2.771(6)
N(2)-H(10)···O(W2)	0.96	1.89	167.7	2.835(8)
N(2)-H(11)···O(E31)	1.01	1.68	171.3	2.689(7)
N(3)-H(12)···O(E31)	0.97	1.83	148.4	2.705(6)
N(3)-H(13)···O(E21)	0.95	1.81	162.8	2.732(6)
N(3)-H(14)···O(W1)	0.97	1.92	161.2	2.854(7)
N(4)-H(15)···O(E13)	0.95	2.04	150.4	2.896(6)
N(4)-H(15)···O(E13)	0.94	2.02	133.3	2.763(6)
N(4)-H(15)···O(E13)	0.96	1.89	140.5	2.698(6)

Estimated standard deviations are given in parentheses.

- Bands in the 1600–1200 cm^{-1} region correspond to the bending vibrations of N-H and O-H groups and to the valence vibrations of C=C and C—N groups.
- The stretching vibrations of the PO_2 central groups are observed between 1100–1300 cm^{-1}. Those between 1260–975 cm^{-1} correspond to stretching vibrations of the PO_3 terminal groups, and those ranging from 960–675 cm^{-1} correspond to stretching POP modes.[11–13] The bending vibrations of PO_3 terminal groups are situated in the interval 600–400 cm^{-1}. Supplementary frequencies in $\nu(PO_3)$ domain are attributed to bending modes δg (C_{ary} – H) and δg (C_{ary}–C_{ary}).[14]

EXPERIMENTAL

Chemical Preparation

An aqueous solution of pentasodium triphosphate, $Na_5P_3O_{10}$ (1 g, 2.71 mmol), was passed through an ion exchange resin (Amberlite IR 120), to generate the triphosphoric acid. The obtained $H_5P_3O_{10}$ was immediately added to an alcoholic solution of the 2,3-dimethylaniline, $(CH_3)_2C_6H_3NH_3$ (1.32 mL, 10.86 mmol, ρg 0.993), until a final solution

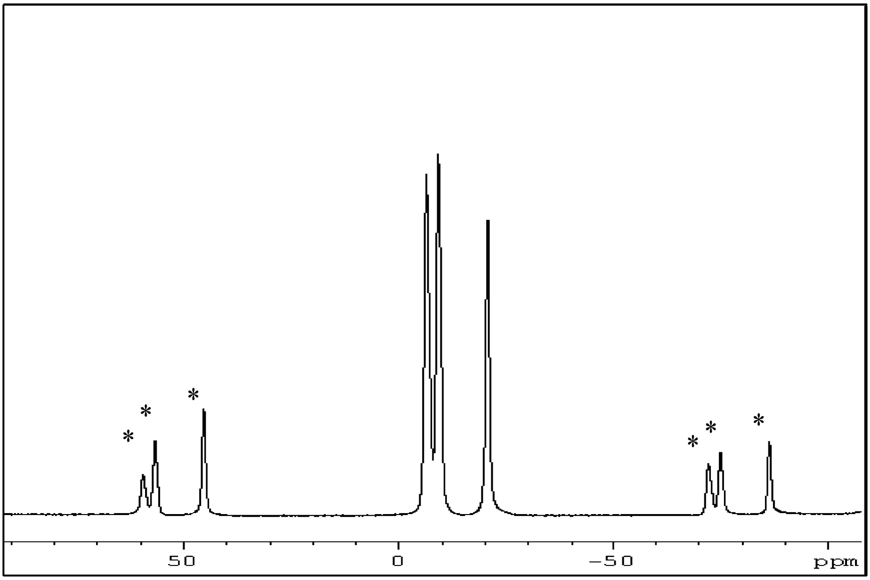


FIGURE 4 ^{31}P MAS-NMR spectrum of $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$ crystals. *Spinning side bands.

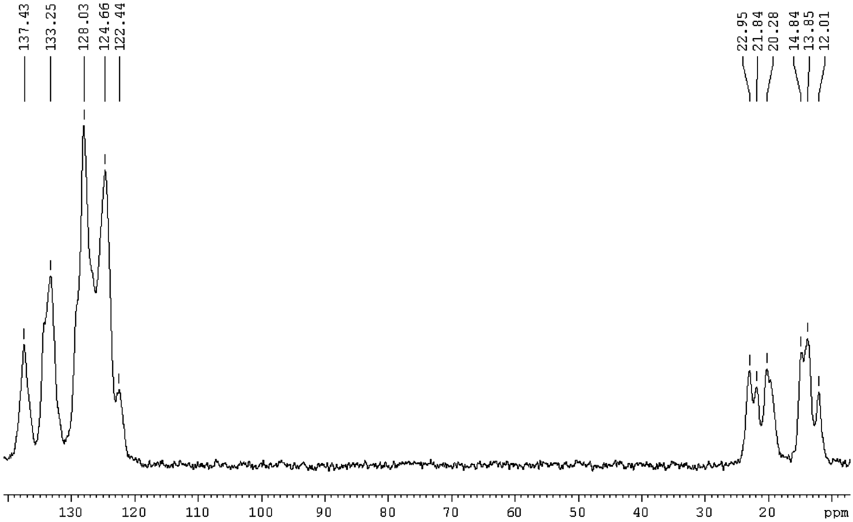


FIGURE 5 ^{13}C CP/MAS NMR spectrum of $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$ crystals.

TABLE IV Crystal Data and Experimental Parameters Used for the Intensity Data Collection

Empirical Formula	$C_{32}H_{53}N_4O_{12}P_3$
Formula weight	778.71
Crystal system	triclinic
Space group	$P\bar{1}$
a	11.963(6) (A)
b	9.858(6) (A)
c	16.205(7) (A)
α	95.50(4)°
β	95.21(4)°
γ	89.07(4)°
Z	2
V	1894.4(16) (Å$^{3)}$
$\rho_{cal.}$	1.367(g. cm$^{-3)}$
F(000)	828.0
μ(AgK$_\alpha$)	1.122(cm^{-1})
Crystal size [mm]	0.40 × 0.30 × 0.2
Index ranges: ±h, ±k, l	$h_{max.} = 17$, $k_{max.} = 14$, $l_{max.} = 14$
Independent reflections	3766
R_{int}	0.070
Refined parameters	460
R [I > 2 σ(I)]	0.074
R$(_w)$	0.078
Goodness-of-fit	1.86

pH = 2.

$$H_5P_3O_{10} + 4[2,3\text{-}(CH_3)_2C_6H_3(NH_2) + 2$$

$$H_2O \longrightarrow [2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O \qquad (1)$$

The resulting solution has a pH = 2. This solution was then slowly evaporated at room temperature for several days until the formation of single crystals of $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$ (m =1.41 g, yield = 67%). The obtained crystals are stable under normal conditions of temperature and humidity.

Investigation Techniques

X-Ray Diffraction

The intensity data collection was performed using a MACH3 Enraf Nonius diffractometer. The experimental conditions of data collection,

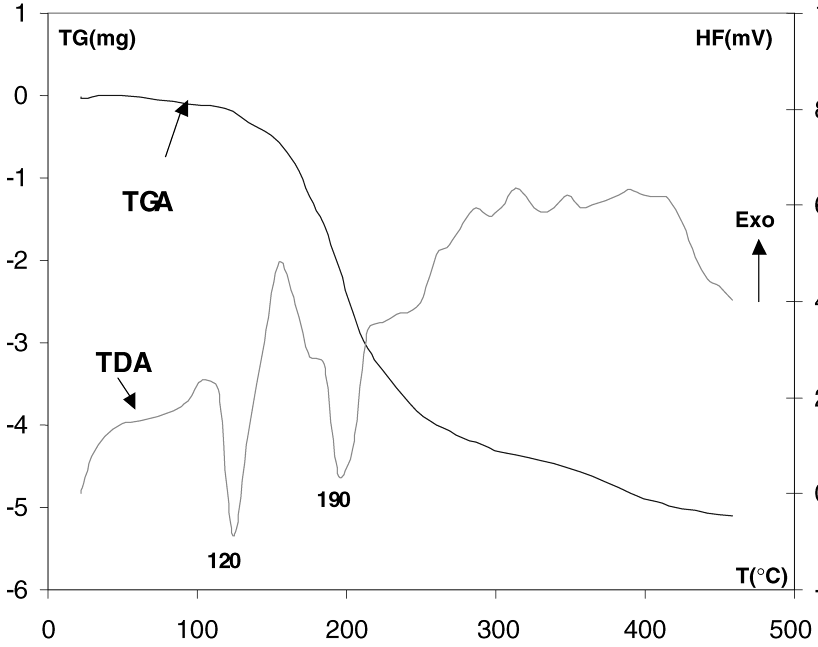


FIGURE 6 DTA and TGA curves of $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$ between room temperature and 500°C.

strategy followed for the structure determination, and its final results are given in Table V.

Crystallographic data (CIF) for the structure reported in this paper have been deposited in the Cambridge Crystallographic Data Center as supplementary publication No 640943 Copies of the data can be obtained, free of charge, on application to the CCDC, 12 Union Road, Cambridge CB12EZ, UK.

TABLE V Calculated (δ_{iso}) and Experimental (δ_{exp}) Chemical Shifts of the Organic Croup Carbon Atoms in $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$

Carbons	C1	C2	C3	C4	C5	C6
δ_{iso}(ppm)	140.1	138.0	136.8	130.3	125.6	120.6
δ_{exp}(ppm)	137.4	133.2	133.2	128.0	124.6	122.4

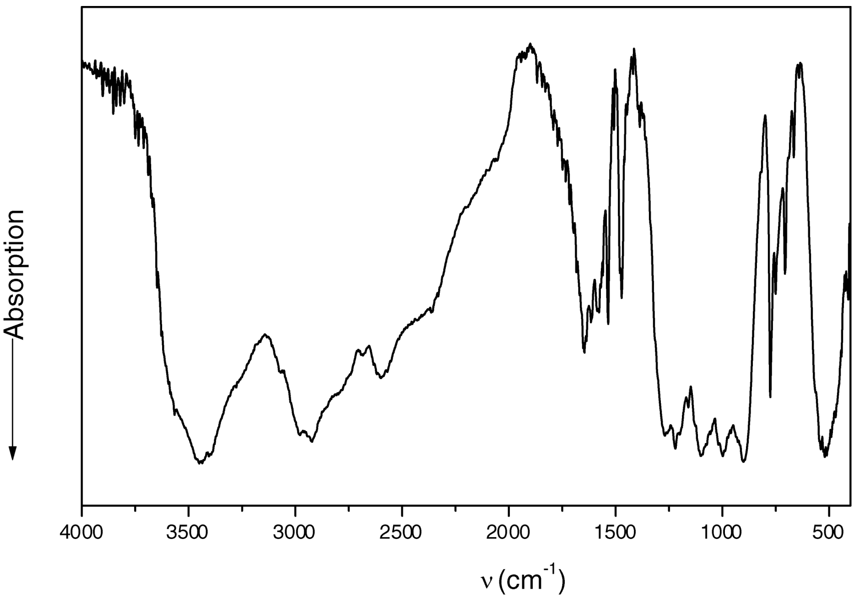


FIGURE 7 IR spectrum of $[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$ in KBr pellets.

NMR Spectroscopy

All NMR spectra were recorded on a Bruker DSX-300 spectrometer operating at 300 MHz for ^{1}H, 75.49 MHz for ^{13}C and 121.51 MHz for ^{31}P with a classical 4 mm probehead allowing spinning rates up to 10 KHz. ^{1}H and ^{13}C NMR chemical shifts are given relative to tetramethylsilane and ^{31}P ones relative to 85% H_3PO_4 (external references, precision 0.5 ppm). Phosphorous and proton spectra were recorded under classical MAS conditions while the carbon ones were recorded by use of cross-polarization from protons (contact time 5 ms). In all cases, it was checked that there was a sufficient delay between the scans allowing a full relaxation of the nuclei.

Thermal Behaviour

Thermal analysis was performed using the "multimodule 92 Setaram analyzer" operating from room temperature up to 500°C at an average heating rate of 5° min^{-1}.

Infrared Spectroscopy

IR Spectrum was recorded in the 4000–400 cm^{-1} range with a "Perkin-Elmer Spectrum 1000" spectrophotometer using a sample dispersed in spectroscopically-pure KBr pellet.

CONCLUSION

$[2,3\text{-}(CH_3)_2C_6H_3NH_3]_4HP_3O_{10}.2H_2O$ was prepared as single crystals at room temperature and characterized by various physicochemical methods. On the structural level, the atomic arrangement of the title compound can be described as built up by layers of $HP_3O_{10}^{4-}$ groups developing in (a, b) planes at z = 1/2. The interlayer spacing is occupied by the organic molecules. The number of solid-state ^{13}C and ^{31}P MAS NMR components is in full agreement with the ones of crystallographically independent sites. When heated, the compound loses the crystallization water molecules at 120°C. By heating further, the compound exhibits a degradation of the organic entity confirmed by the obtained carbon residue at the end of the experiment.

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