ORIGINAL ARTICLE

CHEMICAL PREPARATION AND CRYSTALLOGRAPHIC CHARACTERIZATION OF $BaCsP_3O_9.2H_2O$, $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ AND ITS CORRESPONDING ANHYDROUS $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$

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|Received | 18 May 2017| |Accepted | 23 May 2017| |Published 04 June 2017 |

ABSTRACT

Objectives: Report the Methods of chemical preparation and X-ray diffraction XRD data for three condensed phosphates $BaCsP_3O_9.2H_2O$, $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ and its corresponding anhydrous $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$. $BaCsP_3O_9.2H_2O$. **Methods**: the three condensed phosphates were prepared by the method of ion resin exchange, $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ by the reaction between triphosphoric acid $H_5P_3O_{10}$ and 4-aminobenzoic acid, and $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$ by total dehydration of $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ at 130°C. The structures of $BaCsP_3O_9.2H_2O$ and $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ were determined and solved on the basis of their single crystals. The unit-cell parameters of $(C_7H_8NO_2)H_2P_3O_{10}$ were calculated by means of the computer program Dicvol. **Conclusions:** $BaCsP_3O_9.2H_2O$ is monoclinic, space group $P2_1/n$, Z = 4. $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ crystallizes in the triclinic system with centric space group P-1. The anhydrous form $(C_7H_8NO_2)H_2P_3O_{10}$ was obtained by total thermal dehydration of $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ at 130°C. $(C_7H_8NO_2)H_2P_3O_{10}$ crystallizes in the triclinic system space group P-1 and its figures of merit are M(10)= 91.9 and F (10) = 55.6(0.0032, 56).

Keywords: *$(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$, $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$, $BaCsP_3O_9.2H_2O$, Chemical preparation, crystallographic characterization.*

1. INTRODUCTION

In a systematic study of the physico-chemical properties of alkaline earth cation cyclotriphosphates, M^{II} = Ca^{2+}, Sr^{2+} and Ba^{2+}, and M^{I} = Rb^{+}, K^{+}, Cs^{+} and NH^{4+} alkaline cations, and organic cations carried out at the laboratory, we obtained three condensed phosphates $BaCsP_3O_9.2H_2O$, $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ and $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$. The synthesis, optionally, of the mixed barium cyclotriphosphate associated with the cesium ion, $BaCsP_3O_9.nH_2O$, $BaCsP_3O_9.2H_2O$ was prepared for the first time using the Boullé's process [1]. Moreover the oligophosphates are the simplest term for condensed phosphates. Their anions correspond to the general formula $[P_nO_{3n+1}]^{(n+2)-}$. The triphosphates (n = 3) were mainly studied with mineral cations. However, several organic compounds with acidic triphosphoric anions; e.g.$[HP_3O_{10}^{4-}]$, $[H_2P_3O_{10}^{3-}]$, $[H_3P_3O_{10}^{2-}]$ and $[H_4P_3O_{10}^{-}]$, have been reported. These acidic anions reveal the flexibility of their aggregation, via H-bonds, with respect to the organic cations inducing various geometries: chains, ribbons, layers and three dimensional networks. Many combinations between organic and inorganic components may be achieved to create materials in several areas; e.g. sorbents, catalysts and biotechnological materials [2,3], because of the nature (molecular, ionic, hydrogen bonding) [4] of the interaction between them. As a contribution to the study of organic triphosphate compounds and cyclotriphosphates with alkaline earth and alkaline cations, we report here the preparation and crystallographic characterization of the dihydrogenotriphosphate trihydrate of para carboxyphenylammonium, $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$, its anhydrous form $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$ and $BaCsP_3O_9.2H_2O$.

2. MATERIALS AND METHOD EXPERIMENTAL

2.1 Synthesis:

2.1.1 $BaCsP_3O_9.2H_2O$: The precursor $Na_3P_3O_9$ is first prepared by heat treatment of sodium dihydrogenomonophosphate, NaH_2PO_4, at 530°C for 5 hours, according to the following reaction:

$$3NaH_2PO_4 \longrightarrow Na_3P_3O_9 + 3H_2O \qquad (1)$$

In the second step, cyclophosphoric acid, $H_3P_3O_9$, is prepared. $H_3P_3O_9$ is prepared from a concentrated solution of sodium cyclotriphosphate $Na_3P_3O_9$, flown through a column of ion exchange resin Na^+ by H^+, type Amberlite IR 120 [5]. The cyclotriphosphoric acid, $H_3P_3O_9$, formed is immediately neutralized by a mixture of cesium carbonate and barium carbonate in stoichiometric ratios. After 24 hours of mechanical stirring, the excess carbonates is removed by filtration. The resulting liquor is left at room temperature. The spontaneous evaporation of the solution leads, after about one month, to prismatic single crystals of sufficient size for structural study. This study showed that it is $BaCsP_3O_9.2H_2O$. The overall reaction scheme is as follows:

$$H_3P_3O_9(l) + BaCO_3(s) + 1/2\ Cs_2CO_3(s) \longrightarrow BaCsP_3O_9.2H_2O(aq) + 3/2CO_2 \quad (2)$$

$$BaCsP_3O_9.2H_2O(aq) \longrightarrow BaCsP_3O_9.2H_2O(s) \quad (3)$$

2.1.2. $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$

Single crystals of $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ were prepared by slowly adding dilute triphosphoric acid, $H_5P_3O_{10}$, to an aqueous solution of para carboxyphenylammonium, $NH_2C_6H_4COOH$ according to the following chemical reaction :

$$H_5P_3O_{10} + 3\ NH_2C_6H_4COOH + 3H_2O \longrightarrow (NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O \quad (4)$$

The so-obtained solution was then slowly evaporated at room temperature until large prisms of $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ were obtained. The triphosphoric acid, $H_5P_3O_{10}$, used in this reaction was prepared from an aqueous solution of $Na_5P_3O_{10}$ passed through an ion-exchange resin 'Amberlite IR120' [6]. $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ is stable in the normal conditions of temperature and hygrometry until 90°C.

2.1.3. $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$: The product resulting from the total thermal dehydration of $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$, at 130°C, is the anhydrous new triphosphate of para carboxyphenylammonium $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$. The reaction is the following :

$$(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O \xrightarrow{130°C} (NH_3C_6H_4COOH)_3H_2P_3O_{10} + 3H_2O \quad (5)$$

$(NH_3C_6H_4COOH)_3H_2P_3O_{10}$ is stable in the normal conditions of temperature and hygrometry. With further increase in temperature, $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$ is decomposed by evolving NH_3 followed by CO_2

2.3 Patients Data collection and reduction:

2.1.1. $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ and $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$: Powder diffraction patterns for the two title compounds were collected with a SIEMENS D 5000 diffractometer using CuK = 1.5406 Å. The experimental 2θ range was λKα1 radiation (from 5 to 60° (2θ) with a step size of 0.01° and a counting time of 30s per step. The program of graphic tool for powder diffraction named Win PLOTR was used to determine the observed diffraction peak positions for $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ and $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$. The unit-cell parameters of $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$ were calculated by means of the computer program Dicvol [7].

2.1.2. $BaCsP_3O_9.2H_2O$: The diffraction data were measured by using an Oxford Xcalibur S diffractometer at room temperature. It is a 4-circle diffractometer (50 kV, 50 mA) equipped with a Mo Kα radiation source (λ = 0.71073 Å). The reduction of data, the determination of the space group and the refinement of the unit-cell parameters were carried out using the CrysAlisPro software [8]. An analytical absorption correction was applied using a multi-faceted crystal model based on expressions derived by R.C. Clark and J. S. Reid [9].

The X-ray diffractograms of $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ and its anhydrous form $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$ are shown in Figure 1. The X-ray diffractogram of $BaCsP_3O_9.2H_2O$ is shown in Figure 2. The parameters concerning the data collections of $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ and $BaCsP_3O_9.2H_2O$ are shown respectively in Tables 1 and 2.

Table 1: The table presents the Crystallographic data and refinement of the structure of single crystals of $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$

Temperature	300 K
Crystal system	Triclinic
Space group	P-1
Unit-cell parameters	a = 9.844(1) Å, b =13.086(4) Å, c = 13.320(2)Å α = 107.12(2)°, β = 106.08(1)°, γ = 97.87(2)° V = 1530(1) $Å^3$, Z = 2
Density (calculated)	1.57 g/cm^3
Size/color	0 .40x0.26x0.20mm^3, brownish prism
Wavelength	Mo($Kα_1$) = 0.71073 Å
Absorption coefficient	0.283
Absorption correction	Psi scan type, min = 0.9616, max = 1.0000
Measuring range	5.0 ≤ 2θ≤52.58
indice limit	-12 ≤ h ≤12, 0≤ k ≤ 16, -16 ≤ 1 ≤ 15
Number of measured reflections	6501
Number of independent reflections	6218
Method of refinements	Method of least squares on F
Data / deductions / parameters	4075 / 0 / 544
Goodness-of-fit on F^2	1.581
Final index R for all data	R1 = 0.082 wR2 = 0.085
Final index for I> 3	R1 = 0.036 wR2 = 0.063

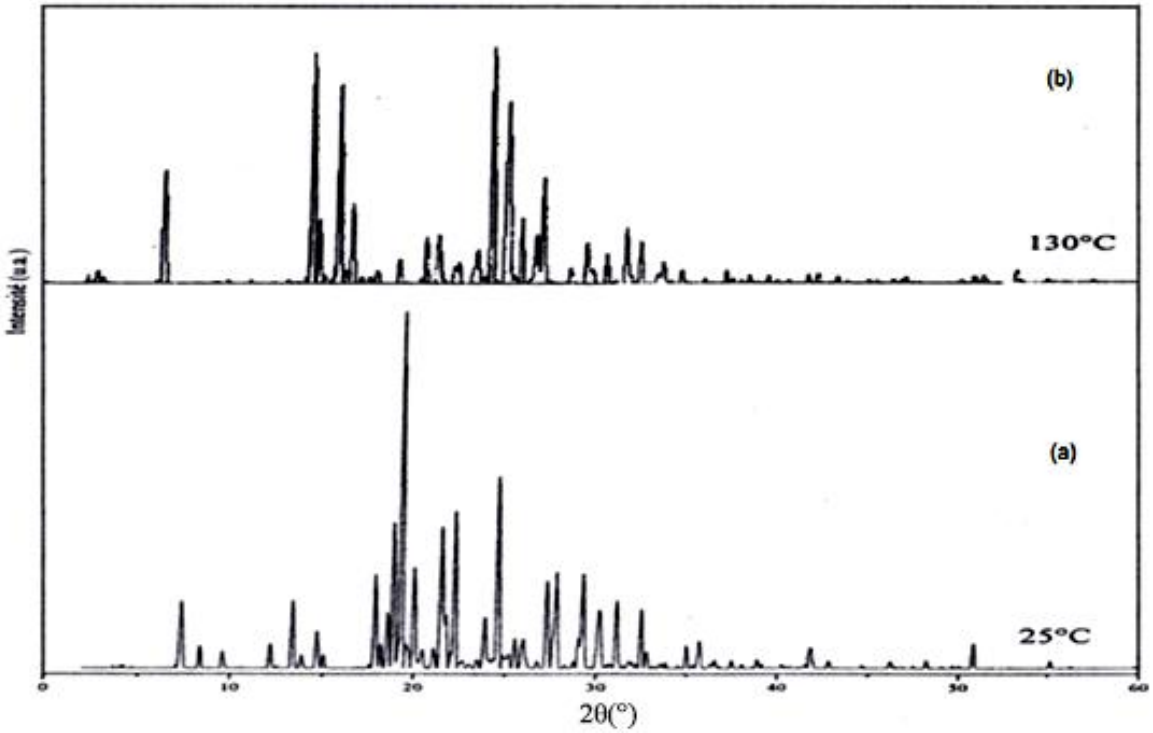

Figure 1: The figure presents X-ray powder diffractograms of the phosphates :(a) $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ and (b) $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$

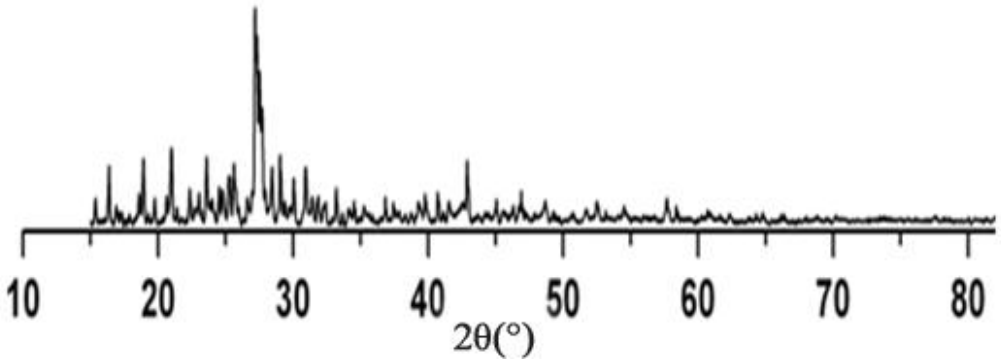

Figure 2: The figure presents X-ray powder diffractogram of $BaCsP_3O_9.2H_2O$

Table 2: The table presents the Crystallographic data and refinement of the structure of $BaCsP_3O_9.2H_2O$

I. Crystal data	
Formula/formula weight	$BaCsH_4O_{11}P_3$ /543.20g.mol^{-1}
Crystal system	Monoclinic
Space group/Z	P $2_1/n$ / 4
parameters	a= 7.6992(2) Å b= 12.3237(3) Å c= 11.8023(3) Å, β= 101.18(3)°
Volume	V= 1098.57(5)Å^3
Calculated density (g/cm^3)	3.284
Size/color	(0.3296 × 0.1602 ×0.0957mm^3)/colorless
Absorption coefficient μ (mm^{-1})	7.362
F(0 0 0)	992.0
II. Intensity measurement	
Diffractometer	Oxford Xcalibur S
Monochromator	Graphite
Wavelength (Mo Kα)	λ= 0.71073 Å
Temperature	293(2)
Theta range	3.50°/ 27.59°
h, k, l range	-3
Reflections collected	9176
Number of independent reflections	2448
III. Structure determination	
Unique reflections included (I > 2σI)	
Programs used	SHELXL-13
Number of refined parameters	147
Goodness-of-fit on F2	1.113
R (anisotropic)/Rw (anisotropic)	0.0401/ 0.0378
Extinction coefficient	0.170(15)
Final R indexes [I>=2σ (I)]	R_1 = 0.0285, wR_2 = 0.0611
Final R indexes [all data]	R_1 = 0.0329, wR_2 = 0.0638
Largest diff. peak/hole / e Å^{-3}	0.78/-1.40

3. RESULTS

$BaCsP_3O_9.2H_2O$ crystallizes in the monoclinic system $P2_1/n$ with the following unit-cell parameters : a = 7.6992 (2) Å, b = 12.3237 (3) Å, c = 11.8023 (3) Å, β = 101.18(3)° and Z = 4. The powder diffraction pattern of $BaCsP_3O_9.2H_2O$ is plotted in Figure 2 and the powder diffraction data for the compound are reported in Table 3.

Table 3: The table presents the Powder Diffraction Data Of $BaCsP_3O_9.2H_2O$

$2\theta_{obs}$ (°)	$(100I/I_0)$	hkl	d_{obs}(Å)	$2\theta_{cal}$ (°)	d_{cal}(Å)	Δ 2θ(°)
15.36	18	1 0 1	5.78	15.3	5.78	-0.06
18.90	40	1 2 0	4.7	18.9	4.69	0
19.26	5	1 1 -2	4.61	19.2	4.61	-0.06
21.37	9	0 2 -2	4.16	21.3	4.16	-0.07
22.33	20	1 1 2	3.95	22.5	3.96	0.17
22.71	7	1 1 2	3.95	22.5	3.957	-0.21
23.00	18	0 0 -3	3.816	23.3	3.82	0.3
23.57	41	0 3 1	3.79	23.4	3.806	-0.17
23.97	14	2 0 0	3.71	24	3.712	0.03
24.49	22	0 1 3	3.64	24.4	3.64	-0.09

25.24	27	1 1 -3	3.52	25.3	3.56	0.06
27.18	100	2 1 -1	3.27	27.2	3.28	0.02
28.38	35	1 3 -2	3.14	28.4	3.14	0.02
29.33	13	0 4 0	3.02	29.5	3.03	0.17
30.00	28	2 2 -2	2.97	30.1	2.97	0.1
34.46	14	2 3 -2	2.59	34.4	2.6	-0.06
36.75	15	3 1 -1	2.46	36.5	2.46	-0.25
38.66	6	3 2 -1	2.32	38.8	2.32	0.14
42.79	38	3 1 2	2.105	43	2.104	0.21
46.76	19	1 2 5	1.97	46.6	1.97	-0.16

Tri(4-carboxyphenylammonium) dihydrogenotriphosphate trihydrate $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ crystallizes in the triclinic system with centric space group P-1. Its unit-cell dimensions are a = 9.844(1) Å, b = 13.086(4) Å, c = 13.320(2) Å, α = 107.12 (2)°, β = 106.08 (1)°, γ = 97.87(2)°, Z = 2 and V = 1530 $Å^3$. The powder diffraction pattern of $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ is plotted in Figure 1(a), and the powder diffraction data for the compound are reported in Table 4.

Table 4: The table presents the Powder diffraction data of $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$

$2\theta_{obs}$ (°)	$(100I/I_0)$	hkl	d_{obs}(Å)	$2\theta_{cal}$ (°)	d_{cal}(Å)	Δ 2θ(°)
7.33	19	0 1 0	12.05	7.27	12.15	-0.06
9.61	5	1 0 0	9.20	9.62	9.19	0.01
12.15	7	1 1 -1	7.28	12.18	7.26	0.03
13.38	19	1 1 0	6.61	13.38	6.61	-
13.87	4	0 2 -1	6.38	13.85	6.39	-0.02
14.7	10	1 0 -2	6.02	14.68	6.03	-0.02
15.06	4	1 1 -2	5.88	15.06	5.88	-
17.91	26	1 1 1	4.95	17.91	4.95	-
18.2	7	2 0 -1	4.87	18.2	4.87	-
18.59	16	1 2 -2	4.77	18.59	4.77	-
19.32	100	2 0 0	4.59	19.32	4.59	-
20.03	28	2 0 -2	4.43	20.03	4.43	-
20.49	5	0 3 -1	4.33	20.54	4.32	0.05
21.14	6	2 1 -2	4.20	21.14	4.20	-
21.55	40	1 -3 1	4.12	21.5	4.13	-0.05
21.77	15	0 3 -2	4.08	21.77	4.08	-
22.25	44	2 1 0	3.993	22.23	3.996	-0.02
25.58	8	1 -2 3	3.479	25.56	3.482	-0.02
26.05	8	1 3 0	3.418	26.06	3.417	0.01
26.81	2	1 1 -4	3.323	26.82	3.322	0.01
27.33	24	-3 1 1	3.261	27.34	3.260	0.01
27.87	27	1 -3 3	3.199	27.87	3.199	-
28.82	2	-1 3 2	3.095	28.83	3.094	0.01
29.12	8	-3 2 1	3.064	29.11	3.065	-0.01
29.35	26	2 3 -1	3.041	29.35	3.041	-
30.19	16	2 3 -1	2.958	30.2	2.957	0.01
31.1	19	3 -1 1	2.873	31.09	2.874	-0.01
32.48	17	3 0 1	2.754	32.48	2.754	-
32.79	5	2 3 0	2.729	32.75	2.732	-0.04
33.84	2	1 2 -5	2.647	33.86	2.645	0.03
34.98	6	1 -5 2	2.563	34.95	2.565	-0.03
32.79	5	2 3 0	2.729	32.75	2.732	-0.04
33.84	2	1 2 -5	2.647	33.86	2.645	0.03
34.98	6	1 -5 2	2.563	34.95	2.565	-0.03
35.71	8	2 4 -3	2.512	35.71	2.512	-
36.56	2	2 4 -1	2.456	36.52	2.458	-0.03
37.52	2	2 -5 0	2.395	37.5	2.396	-0.02
38.07	1	4 -1 0	2.362	38.06	2.362	-
41.85	6	2 3 2	2.157	41.84	2.157	-
42.85	2	2 3 6	2.109	42.86	2.108	0.02

$(C_7H_8NO_2)H_2P_3O_{10}$ has a powder diffraction pattern plotted in Figure 1(b). $(C_7H_8NO_2)H_2P_3O_{10}$ crystallizes in the triclinic system space group P-1. Its unit-cell dimensions are: a = 14.566(6) Å, b = 6.866 (1)Å, c = 6.445 (6)Å, α = 112.36 (3)°, β = 94.51 (1)°, γ = 95.94(7)°, Z = 2 and V = 587.98 (1)Å3. The figures of merit are M(10)= 91.9 and F (10) = 55.6(0.0032, 56). Its powder diffraction data is reported in Table 5.

Table 5: The table presents the Powder diffraction data of $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$

$2\theta_{obs}$ (°)	$(100I/I_0)$	hkl	d_{obs}(Å)	$2\theta_{cal}$ (°)	d_{cal}(Å)	Δ 2θ(°)
6.14	48	-1 0 0	14.39	6.15	14.37	0.01
14.11	98	0 1 0	6.27	14.09	6.28	- 0.02
14.56	28	-1 1 0	6.08	14.56	6.08	-
15.45	85	-1 0 1	5.73	15.48	5.72	0.03
16.19	34	1 1 0	5.47	16.19	5.47	-
16.71	4	-1 0 -1	5.3	16.91	5.24	0.20
17.17	4	-1 1 -1	5.16	17.17	5.16	-
17.51	6	-2 1 0	5.06	17.37	5.1	- 0.14
18.87	11	-3 0 0	4.7	18.51	4.79	- 0.36
20.31	20	-2 1 -1	4.37	20.21	4.39	- 0.10
20.93	21	2 0 1	4.24	20.64	4.3	- 0.29
22.04	9	-3 1 0	4.03	21.66	4.1	- 0.38
23.05	15	3 0 -1	3.856	22.35	3.975	- 0.70
24.77	100	-4 0 0	3.591	24.76	3.593	- 0.01
25.48	77	-2 1 1	3.493	25.5	3.49	0.02
26.38	28	0 2 -1	3.376	26.38	3.376	-
26.6	45	-1 2 -1	3.348	26.6	3.348	-
28.07	7	1 1 -2	3.176	28.15	3.168	0.08
28.94	18	-1 1 -2	3.083	28.93	3.084	- 0.01
29.98	13	-1 0 2	2.978	30.05	2.971	0.07
31.29	24	-2 1 -2	2.856	31.26	2.859	- 0.03
31.88	18	-4 1 1	2.805	31.98	2.796	0.10
31.69		0 2 -2	2.821	32.42	2.759	0.73
33.08	10	1 2 -2	2.706	33.09	2.705	0.01
34.12	6	-2 0 -2	2.626	34.18	2.621	0.06
36.57	7	-5 1 1	2.455	36.51	2.459	- 0.06

5. CONCLUSION

Two triphosphates associated to para carboxyphenylammonium have been prepared by two different methods. The hydrate triphosphate $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ was prepared by the method of ion exchange resin and its anhydrous new form, $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$, was obtained by total thermal dehydration of $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ at 130°C. $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ crystallizes in the triclinic system with centric space group P-1. Its unit-cell dimensions are a = 9.844(1) Å, b = 13.086(4) Å, c = 13.320(2) Å, α = 107.12 (2)°, β = 106.08 (1)°, γ = 97.87(2)°, Z = 2 and V = 1530 $Å^3$. $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$, was characterized by X-ray diffraction and its unit-cell parameters were calculated by means of the computer program Dicvol. $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$ crystallizes in the triclinic system space group P-1. Its unit-cell dimensions are: a = 14.566(6) Å, b = 6.866 (1) Å, c = 6.445 (6)Å, α = 112.36 (3)°, β = 94.51 (1)°, γ = 95.94(7)°, Z = 2 and V = 587.98 (1)Å3. The figures of merit are M (10)= 91.9 and F (10) = 55.6(0.0032, 56). It is worth noticing that $(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ and $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$ can be used as binary organic fertilizers NP second generation and corrosion inhibitors.

The barium and cesium cyclotriphosphate dihydrate, $BaCsP_3O_9.2H_2O$, was obtained as single crystals by the resin exchange method. It crystallizes in the monoclinic system, space group $P2_1/n$, Z = 4 with the following unit-cell parameters a= 7.6992 (2) Å, b = 12.3237 (3) Å, c = 11.8023 (3) Å, β = 100.92 (5), V = 1098.57(5) $Å^3$. $BaCsP_3O_9.2H_2O$, $BaNH_4P_3O_9.2H_2O$ and $BaTlP_3O_9.2H_2O$ are isotypic compounds.

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Cite this article: Mustafa Belhabra, Soufiane Zerraf, Aziz Kheireddine, Khadija El Kababi, Azzeddine atibi, Malika tridane, Mohamed Radid and Said Belaaouad. CHEMICAL PREPARATION AND CRYSTALLOGRAPHIC CHARACTERIZATION OF $BaCsP_3O_9.2H_2O$,$(NH_3C_6H_4COOH)_3H_2P_3O_{10}.3H_2O$ AND ITS CORRESPONDING ANHYDROUS $(NH_3C_6H_4COOH)_3H_2P_3O_{10}$. *Am. J. innov. res. appl. sci.* 2017; 4(6): 223-229.