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## Preparation of High Purity Phosphatic Salts from Purified Phosphoric Acid

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**Abstract** Some phosphatic salts are prepared from the purified industrial phosphoric acid. Namely sodium ammonium phosphate, sodium aluminum phosphate and potassium magnesium phosphate. The structure of each product was confirmed by means of infrared spectroscopy (FT-IR), X-ray diffraction (XRD), EDAX and complete chemical analysis. The obtained results indicate that the prepared salts could be a suitable material. Chemical analyses of these products are in close agreement with their theoretical value.

**Keywords** phosphoric acid; phosphate salts; infrared spectroscopy; X-ray diffraction.

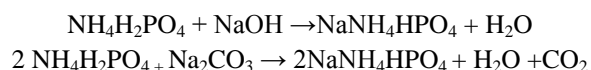
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### Introduction

Alkali metal phosphates are recovered from a water immiscible solvent solution of aqueous wet process phosphoric acid by neutralizing the acid with a basic alkali metal to phosphorus of 1 to 3.5 to form at least the mono alkali metal phosphate. The solvent is given a water wash to recover entrained alkali metal phosphate. The resulting aqueous solution and wash liquor containing the alkali metal salts are separated from the stripped solvent [1].

Production of pure alkali metal phosphate solution by neutralizing of wet processed phosphoric acid with an alkali liquor and/or carbonate to this end, wet processed phosphoric acid is neutralized to a pH from 4 to 9, The resulting neutralizing sludge consisting substantially of insoluble aluminum and iron phosphates are separated from the alkali metal phosphate solution and scrubbed with water, The sludge is mixed with alkali liquor, alkali metal phosphate solution and waterglass so as to establish in the resulting suspension a molar ratio of SiO<sub>2</sub> to Al<sub>2</sub>O<sub>3</sub> of at least 2:1 and a molar ratio of dissolved alkali metal oxide to dissolved P<sub>2</sub>O<sub>5</sub> ranging from 2.9:1 to 3.3:1, The whole is heated to temperature from 800 to 1000 °C to effect break up the sludge in suspension with the resultant formation of a solid matter residue together with a trialkali metal phosphate solution, water scrubbed and discard more particularly crude wet processed phosphoric acid containing more than 45 wt.% of P<sub>2</sub>O<sub>5</sub> is diluted with the a portion of the scrubbing water obtained during operation and its P<sub>2</sub>O<sub>5</sub> content is thereby reduced down to 30 % to 45 %, The acid so treated is neutralized with an alkali liquor and/or carbonate and with trialkali metal phosphate solution originating from the processed neutralization sludge [2].

Highly pure sodium ammonium hydrogen phosphate and ammonium chloride are prepared from wet process phosphoric acid using a mixture of phosphoric acid, ammonium and / or sodium chloride is added to circulating mother liquor, add ammonia until pH reaches 5.5 to 9 allow the phosphate to crystallize at 30 °C to 60 °C, Method for the preparation of sodium ammonium phosphate mole ratio of reaction NA/P is 2 [3]. As shows in the following equations:



The invention relates to a combined process for the manufacture of feed grade dicalcium phosphate and pure phosphoric acid. The process comprises the reaction of an excess of phosphate rocks with aqueous solutions of a mineral acid selected from hydrochloric acid, phosphoric acid and nitric acid, characterized by the fact that the



free acidity of the dissolution liquor does not exceed 60% and that in the clear solution feed grade dicalcium phosphate is precipitated with calcium hydroxide, calcium carbonate or a mixture thereof. The solids comprising the unattached phosphate are decomposed with hydrochloric acid solutions, gases containing hydrogen chloride or mixtures thereof and the acidulate containing the phosphoric acid is extracted with an organic solvent selected from butanols, pentanols or mixtures thereof, the phosphoric acid-solvent extract is separated and washed, thus obtaining the pure phosphoric acid [4].

A process for producing an alkalis metal polyphosphate comprising heating a solution comprising a solvent, alkalis metal ions, phosphate ions, and 3 to 50 % by weight organic matter, where in the solution conations, in total 5 to 65 % by weight of the alkalis metal ions and the phosphate ions, and the ratio M/P is 2 to 3 where in u is the number of moles of alkalis metal ions in the solution and P IS the number of mole of the phosphate ions in the solution at temperature of 800 to 1200 °C in the presence of excess air to produce the alkalis metal poly Phosphate [5].

A process for preparing mixed alkali metal salts of tripolyphosphate, a solution of mixed orthophosphate salts, such as sodium and potassium is dried by spraying the mixture on to a hot tumbling bed of mixture of phosphate salts, the dried salts are then calcined at a temperature in the range of from about 350 °C about 600 °C, to form the mixed alkali metal salt of tripolyphosphate [6].

Phosphate values can be recovered from an organic which is formed by contact of wet process phosphoric acid and a water immiscible solvent, in a process in which the extract is treated with an inorganic base or a dihydrogen phosphate salt to give an acid free solvent and an aqueous solution of phosphoric acid and phosphate salt with a cation to P usually in the 0.1- 0.5: 1 region. The aqueous solution represents a concentrated soluble phosphate source for subsequent neutralization and conversion to e.g. tri-poly phosphates [7].

A process for the preparation of amorphous aluminum phosphate or polyphosphate-based pigment by reacting aluminum phosphate and sodium aluminate is provided. The amorphous aluminum phosphate or polyphosphate is characterized by a skeletal density of less than 2.50 grams per cubic centimeter and phosphorus to aluminum mole ratio of greater than 0.8. In one embodiment, the composition is useful in paints as a substitute for titanium dioxide [8].

Recently, however, some alkali phosphate products such as ammonium hydrogen phosphate, calcium hydrogen phosphate, and sodium tri-poly phosphate are prepared from the purified industrial phosphoric acid. The structure of each product was confirmed by means of infrared spectroscopy (FT-IR), X-ray diffraction (XRD) and complete chemical analysis. Chemical analysis of these products are in close agreement with its theoretical value [9]. In the present study some phosphatic products such as sodium ammonium phosphate, sodium aluminum phosphate and potassium magnesium phosphate have been prepared from purified phosphoric acid to the food grade quality by a mixture of butanol and TBP [10].

## Experimental

### Materials

Purified phosphoric acid (43 %  $P_2O_5$ ) which is kindly supplied by polyserve for fertilizer company, Cairo, Egypt. Sodium hydroxide, potassium carbonate, ammonium hydroxide, aluminum hydroxide, sodium carbonate, magnesium chloride, potassium hydroxide were obtained from ADWIC Company, Egypt. All other chemicals were Prolabo products and were used as received. In all experiments, Double distilled water was used in all preparation.

### Procedure for Preparation of Salts

#### Preparation of Salts from Purified Phosphoric Acid ( $P_2O_5$ 43 %, Uranium (UDL)).

#### Preparation of Sodium aluminum phosphate

50 ml of purified phosphoric acid is added to 12.5 ml of aluminum hydroxide this mixed heated between at 60 °C to 80 °C until aluminum had substantially reacted, then after sodium hydroxide (55 %) was added and the product is dried at 80 °C in oven.



### Preparation of Sodium ammonium phosphate

50 ml of purified phosphoric acid is added to 75 ml of ammonia solution and the pH is adjusted at 3.5 to 6 and then 100 ml of sodium carbonate or sodium chloride is added the product is dried at 40 °C.

### Preparation of Potassium magnesium phosphate

50 ml of purified phosphoric acid is added to 150 ml of magnesium oxide then after 50ml of potassium hydroxide is added adjusted the pH at 7 to 8.the product produced is dried at 60 °C.

### Characterization of the Products

FT-IR measurements were performed in KBr discs using Nexeus-Nicolite-640-MSA FTIR. Energy dispersive X-ray measurements were measured using the philipsis X-ray diffraction unit PW-3710 with generator PW-1830 scintillation-counterPW-3020, target tube PW 2233, Ni filter at 40 Kv and 30 mA are used. Phosphate, sulfate, and uranium were determined spectrophotometrically using the colorimetric determination a shimadzu 160 A double beam UV spectrophotometer [11]. All measurements were carried out at laboratory temperature.

## Results and Discussion

### Chemical Analysis

After purification of phosphoric acid by mixture of TBP and butanol to the food grade quality as explained in our previous publication .The obtained purified phosphoric acid is the starting material for the present work. It is clear from table 1 that the elemental analysis of the prepared phosphatic salts are in good agreement of their theoretical values.

**Table 1:** Chemical analysis of the studied Phosphatic Salts

Salts prepared	Element analyses																	
	% C		%H		%N		%P		%O		%Na		% Mg		% Al		%K	
	Cal	F	Cal	F	Cal	F	Cal	F	Cal	F	Cal	F	Cal	F	Cal	F	Cal	F
Sodium amonium phosphate	-	-	-	-	9.7	8.6	4.3	4.8	2.1	2.4	5.9	6.6	-	-	-	-	-	-
Sodium aluminium phosphate	-	-	2.9	2.77	-	-	4.6	4.8	2.2	1.98	6.3	6.2	-	-	5.3	5.6	-	-
Potassium magnesium phosphate	-	-	5.2	6.8	-	-	8.5	7.9	1.68	1.99	-	-	11	11.8	-	-	6.8	6.9

### X-ray diffraction measurements:

Table 2 shows the X-ray diffraction pattern of sodium ammonium phosphate which is matches with ASTM card No. 24-1048.

**Table 2:** X-ray diffraction pattern of sodium ammonium phosphate

Sample		Identified Material	
		sodium ammonium phosphate	
		ASTM Card No. (24-1048)	
dA°	I/I <sub>o</sub>	dA°	I/I <sub>o</sub>
10.05	66	9.93	95
6.54	96	6.53	100
4.99	26	4.97	10
4.68	27	4.69	35
4.48	2	4.47	4
4.26	63	4.24	55
4.07	2	4.05	2
3.67	51	3.658	40
3.64	24	3.627	12
3.45	82	3.452	35
3.32	29	3.311	16



3.28	50	3.272	35
3.19	17	3.209	2
3.04	14	3.05	8
2.97	14	2.966	12
2.92	74	2.911	50
2.89	74	2.884	50
2.87	35	2.862	16
2.85	47	2.839	14
2.49	14	2.485	12
2.40	59	2.388	8
2.33	28	2.325	16
2.21	15	2.206	8
2.18	15	2.177	10

Table 3 shows the X-ray diffraction of sodium aluminum phosphate which matches well with ASTM card No. 84-2483.

**Table 3:** X-ray diffraction pattern of sodium aluminum phosphate

Sample		Identified Material	
		sodium aluminum phosphate	
		ASTM Card No(84-2483)	
dA°	I/I <sub>0</sub>	dA°	I/I <sub>0</sub>
5.73	3	5.75922	3.8
5.04	8	5.01853	30.4
4.50	14	4.4683	15.4
4.30	32	4.33125	5.3
3.82	8	3.855	7.5
3.74	20	3.77619	57.4
3.33	14	3.34527	27.8
3.01	100	3.06885	100
2.91	20	2.91068	16.3
2.88	26	2.8761	70
2.45	17	2.46385	12.4

Table 4 shows the X-ray diffraction of potassium magnesium phosphate which matches well with ASTM card No.50-0146.

**Table 4:** X-ray diffraction pattern of potassium magnesium phosphate

Sample		Identified Material	
		potassium magnesium phosphate	
		ASTM Card No (50-0146.)	
dA°	I/I <sub>0</sub>	dA°	I/I <sub>0</sub>
3.92	4	3.94626	4
3.80	6	3.85675	8
3.66	6	3.62013	3
3.59	8	3.57012	1
3.18	100	3.19099	65
3.07	11	3.11463	100
2.96	15	2.93926	16
2.87	5	2.88012	1
2.68	2	2.6857	30
2.55	3	2.54111	29
2.41	4	2.41314	6
2.36	6	2.36423	1



2.24	39	2.24051	7
2.15	4	2.138	13
2.03	3	2.04223	2
2.00	5	1.99553	2
1.94	2	1.94107	2
1.83	9	1.83716	2
1.58	7	1.57132	2

### 3.3. IR analysis

Figure 1 shows the infrared absorption spectrum of the produced sodium ammonium phosphate where as its absorption data is shown in **table (5)**, the band at  $3421.1 \text{ cm}^{-1}$  could be assigned to hydrogen bonding OH group stretching vibration. While the bands at  $2426.9$  and  $2360.4$  and  $2335.4 \text{ cm}^{-1}$  could be assigned to N-H. Ionic phosphate absorption bands at  $1070.3 \text{ cm}^{-1}$  and  $997.02 \text{ cm}^{-1}$  and its bending mode appears at  $693.29$ ,  $611.33$  and the peak at  $538.04$  may be due to metal oxygen band.

**Table 5:** Infrared absorption data of the prepared sodium ammonium phosphate

Peak number	Peak position wave number $\text{cm}^{-1}$	Intensity
1	3421.1	71.0178
2	2426.01	111.908
3	2360.44	104.625
4	2335.37	106.345
5	1966.07	114.852
6	1920.75	114.683
7	1694.16	110.914
8	1628.59	108.597
9	1458.89	100.465
10	1245.79	105.278
11	1070.3	77.3533
12	997.017	93.7978
13	882.274	106.243
14	817.67	112.44
15	693.284	106.306
16	611.324	100.049
17	538.04	95.4953

Figure 1 shows the infrared absorption spectrum of the prepared sodium aluminum phosphate where as its absorption data is shown in table 6, the bands at  $3440.39$ ,  $369.98$  and  $313.58 \text{ cm}^{-1}$  is assigned to hydrogen bonding OH group stretching vibration whereas its bending appears at  $1635.34 \text{ cm}^{-1}$ . The bands at  $1128.15$ ,  $1054.8$  and  $1005.7 \text{ cm}^{-1}$  is due to ionic  $\text{PO}_4$  phosphate vibration while its bending mode appears at  $618.07$  and  $547.68 \text{ cm}^{-1}$ .

**Table 6:** Infrared absorption data of the prepared sodium aluminum phosphate

Peak number	Peak position wave number $\text{cm}^{-1}$	Intensity
1	3693.98	94.3773
2	3440.39	73.6765
3	3138.58	86.5222
4	2460.72	108.821
5	2365.26	108.615
6	1635.34	106.755
7	1452.14	103.595
8	1389.46	107.655



9	1264.11	113.164
10	1128.15	102.341
11	1054.87	96.9564
12	1005.7	99.2234
13	864.917	112.738
14	618.074	109.151
15	547.685	103.818

Figure 1 shows the infrared absorption spectrum of the prepared potassium magnesium phosphate where as its absorption data is shown in table 7, the peak at  $3413.34\text{ cm}^{-1}$  is assigned to hydrogen bonding OH group stretching vibration whereas it's bending appears at  $1638.23\text{ cm}^{-1}$ . the bands at  $2272.7$  and  $2067.32\text{ cm}^{-1}$  could be assigned the structural OH group. The bands at  $1523.49$  and  $1072.23$  are assigned to  $\text{PO}_4^{3-}$  ions stretching vibration at different sites, its bending mode appears at  $600.717$ . The  $416.763\text{ cm}^{-1}$ .band could be assigned to metal oxygen band.

**Table 7:** Infrared absorption data of the prepared potassium magnesium phosphate

Peak number	Peak position wave number $\text{cm}^{-1}$	Intensity
1	3413.39	52.9344
2	2272.7	111.369
3	2067.32	112.977
4	1638.23	89.5865
5	152.49	110.598
6	1442.49	108.281
7	1072.23	93.2645
8	600.717	91.3699
9	410.763	92.1002

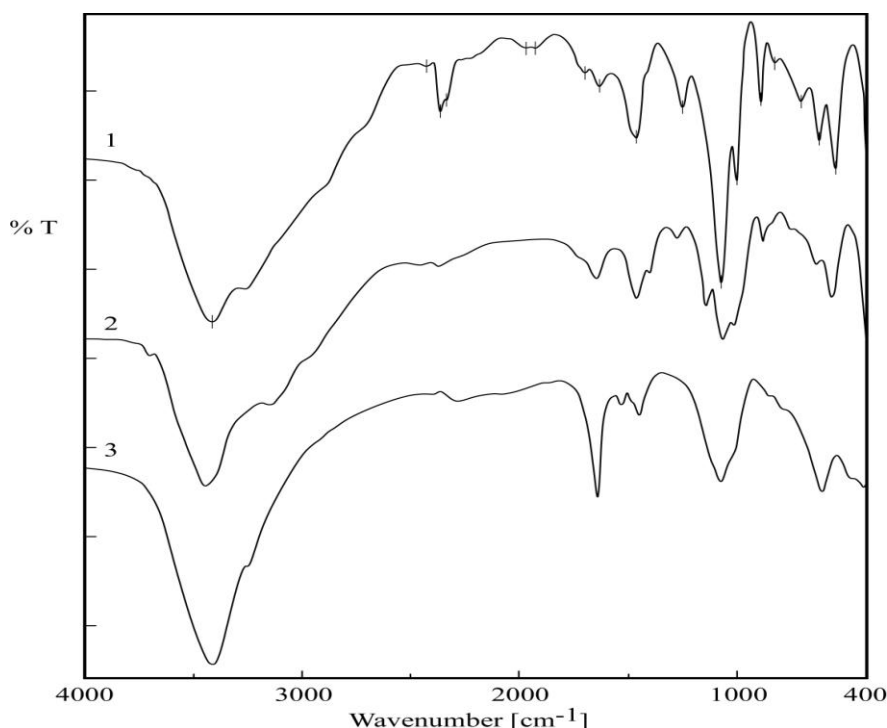


Figure 1: IR spectra of (1) sodium ammonium phosphate, (2) sodium aluminum phosphate, (3) potassium magnesium phosphate



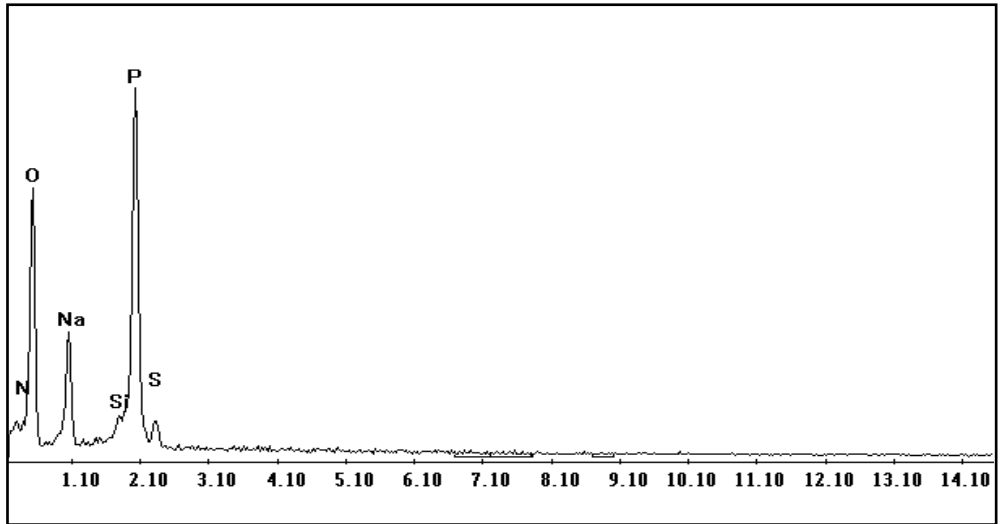


Figure 2: ESEM image and EDX analysis data of sodium ammonium phosphate

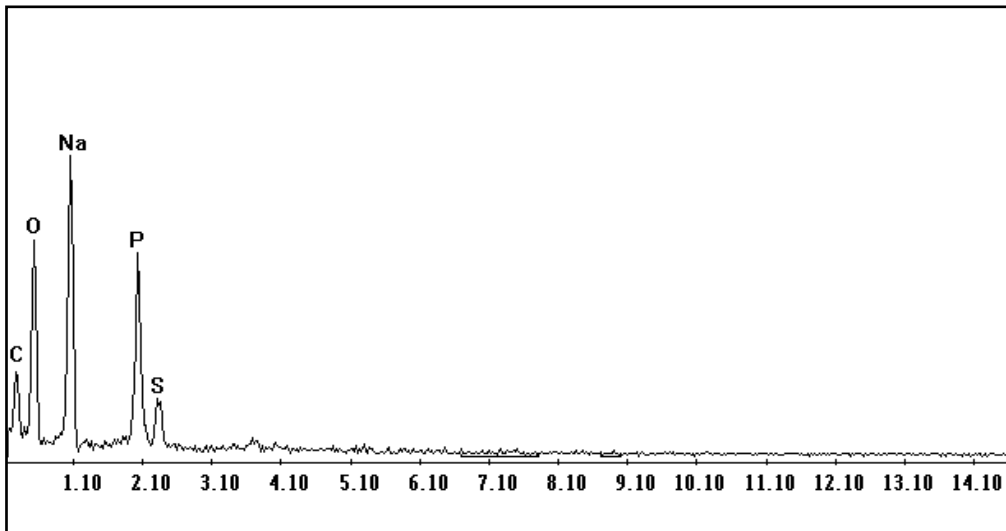


Figure 4.2: ESEM image and EDX analysis data of sodium aluminium phosphate

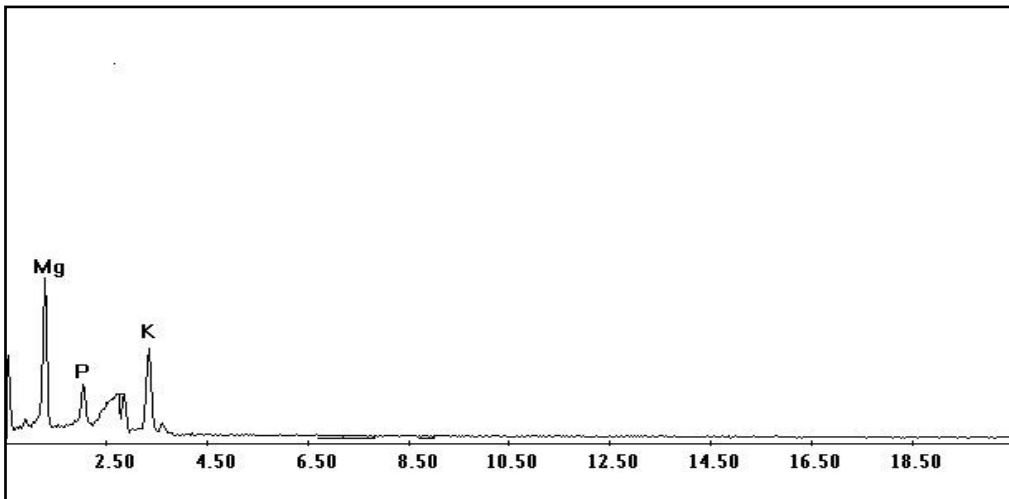


Figure 4: ESEM image and EDX analysis data of potassium magnesium phosphate

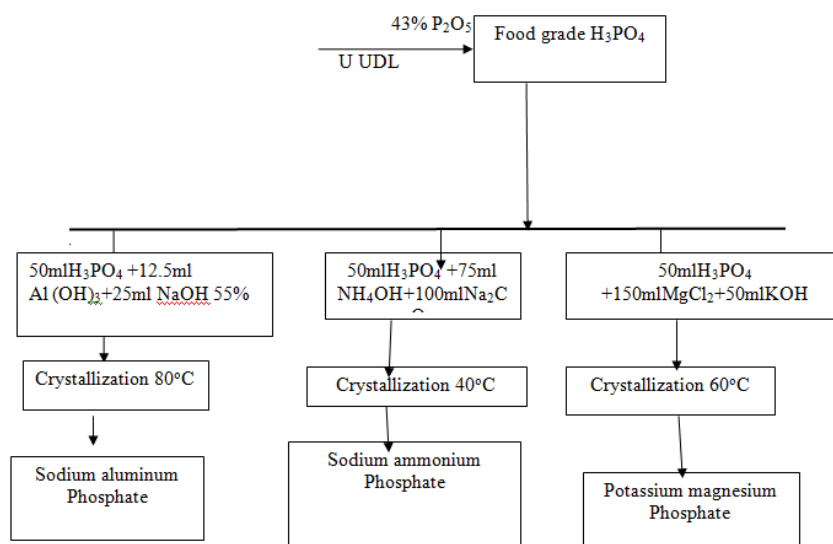


Figure 5: Technological flow sheet for the suggested process

### Conclusion

The present study on the preparation of the following phosphatic salts namely sodium ammonium phosphate, sodium aluminum phosphate, and potassium magnesium phosphate from the purified industrial phosphoric acid by TBP and butanol synergistic extractants are given. Finally on the light of the obtained results a technological flow sheet is elucidated in figure 5. The complete chemical analysis of these prepared salts are found to be in good agreement with their theoretical value. Both X-ray diffraction studied and IR spectroscopy show that our studied prepared phosphatic salts are in a very pure form.

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