Supplementary Material (ESI) for Chemical Communications

Synthesis and crystal structure of $AlH_2P_3O_{10}\cdot 2H_2O$; a new structure-type for layered acid phosphates.

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SINGLE CRYSTAL DATA

Table 1. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for $AlH_2P_3O_{10}·2H_2O$. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Al(1)	-0.5	0	0.5	0.013(1)
P(1)	-0.2825(2)	0.5021(2)	0.5958(1)	0.014(1)
P(2)	-0.5	0.2374(4)	0.75	0.015(1)
O(1)	-0.3552(5)	0.3006(7)	0.5080(3)	0.018(1)
O(2)	-0.5694(5)	0.0815(8)	0.6468(3)	0.020(1)
O(3)	-0.3203(5)	0.7935(7)	0.5697(3)	0.020(1)
O(4)	-0.0919(5)	0.4650(8)	0.6232(4)	0.026(1)
O(5)	-0.3573(5)	0.4336(8)	0.7171(3)	0.024(1)
O(6)	0.0723(8)	-0.0740(20)	0.6346(7)	0.093(3)
H(1)	-0.042(6)	0.650(30)	0.621(12)	0.15(5)
H(2)	0.004(11)	0.070(13)	0.643(10)	0.10(5)
H(3)	0.174(7)	-0.017(19)	0.619(10)	0.15(6)

Table 2. Selected bond lengths (Å) and angles (º) for $AlH_2P_3O_{10}·2H_2O$.

	/Å		/º
O(1)-P(1)	1.497(4)	O(3)-P(1)-O(1)	116.2(2)
O(3)-P(1)	1.489(4)	O(3)-P(1)-O(4)	109.5(2)
O(4)-P(1)	1.531(4)	O(1)-P(1)-O(4)	111.7(2)
O(5)-P(1)	1.623(4)	O(3)-P(1)-O(5)	107.0(2)
O(2)-P(2)	1.484(4)	O(1)-P(1)-O(5)	107.8(2)
O(5)-P(2)	1.568(4)	O(4)-P(1)-O(5)	103.8(2)
Al-O(1)	1.871(4)	O(2)-P(2)-O(2)	117.8(3)
Al-O(2)	1.891(4)	O(2)-P(2)-O(5)	109.9(2)
Al-O(3)	1.871(4)	O(2)-P(2)-O(5)#1	107.2(2)
		O(5)-P(2)-O(5)	104.0(3)
		O(1)-Al-O(2)	91.34(16)
		O(1)-Al-O(3)	88.09(16)
		O(2)-Al-O(3)	90.29(16)

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POWDER DIFFRACTION DATA

Table 3. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for $AlH_2P_3O_{10}·2H_2O$.

	x	y	z	Ui/Ue *100
Al(1)	-0.5	0	0.5	0.12(20)
P(1)	-0.2830(5)	0.4981(9)	0.59703(25)	0.04(14)
P(2)	-0.5	0.2351(11)	0.75	1.26(20)
O(1)	-0.3550(11)	0.3049(17)	0.5053(6)	1.91(30)
O(2)	-0.5686(11)	0.0841(13)	0.6464(5)	0.55(27)
O(3)	-0.3268(11)	0.7980(17)	0.5675(5)	0.75(27)
O(4)	-0.0929(10)	0.4589(14)	0.6208(5)	0.40(26)
O(5)	-0.3512(10)	0.4312(14)	0.7162(6)	1.03(27)
O(6)	0.0760(10)	-0.0357(15)	0.6370(5)	6.49(38)
H(1)	-0.042	0.650	0.621	1.00
H(2)	0.004	0.070	0.643	1.00
H(3)	0.174	-0.017	0.619	1.00

Table 4. Selected bond lengths (Å) and angles (º) for $AlH_2P_3O_{10}·2H_2O$.

	/Å		/º
O(1)-P(1)	1.498(8)	O(3)-P(1)-O(1)	112.7(5)
O(3)-P(1)	1.546(7)	O(3)-P(1)-O(4)	111.0(5)
O(4)-P(1)	1.519(7)	O(1)-P(1)-O(4)	110.5(5)
O(5)-P(1)	1.572(8)	O(3)-P(1)-O(5)	107.8(4)
O(2)-P(2)	1.473(6)	O(1)-P(1)-O(5)	110.7(5)
O(5)-P(2)	1.604(7)	O(4)-P(1)-O(5)	103.7(4)
Al-O(1)	1.888(9)	O(2)-P(2)-O(2)	119.4(6)
Al-O(2)	1.884(7)	O(2)-P(2)-O(5)	108.87(31)
Al-O(3)	1.810(8)	O(2)-P(2)-O(5)#1	106.4(4)
		O(5)-P(2)-O(5)	106.1(6)
		O(1)-Al-O(2)	91.50(26)
		O(1)-Al-O(3)	89.30(34)
		O(2)-Al-O(3)	90.24(26)

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RIETVELD REFINEMENT OF X-RAY POWDER DATA

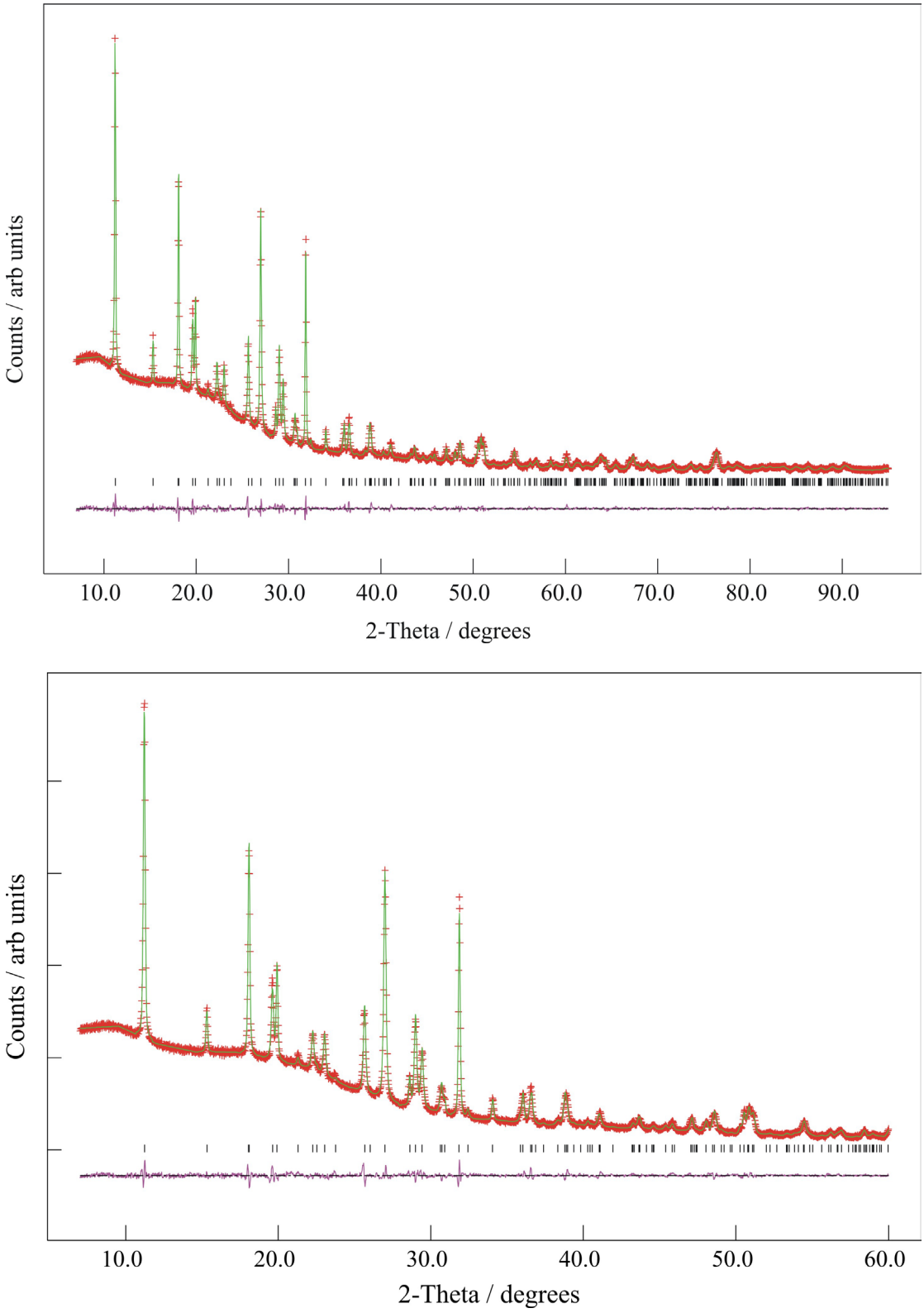

Fig. 4 Final observed (+), calculated (solid line) and difference (below) X-ray powder diffraction profile for the Rietveld refinement of $AlH_2P_3O_{10}\cdot 2H_2O$. Reflection positions are also marked.

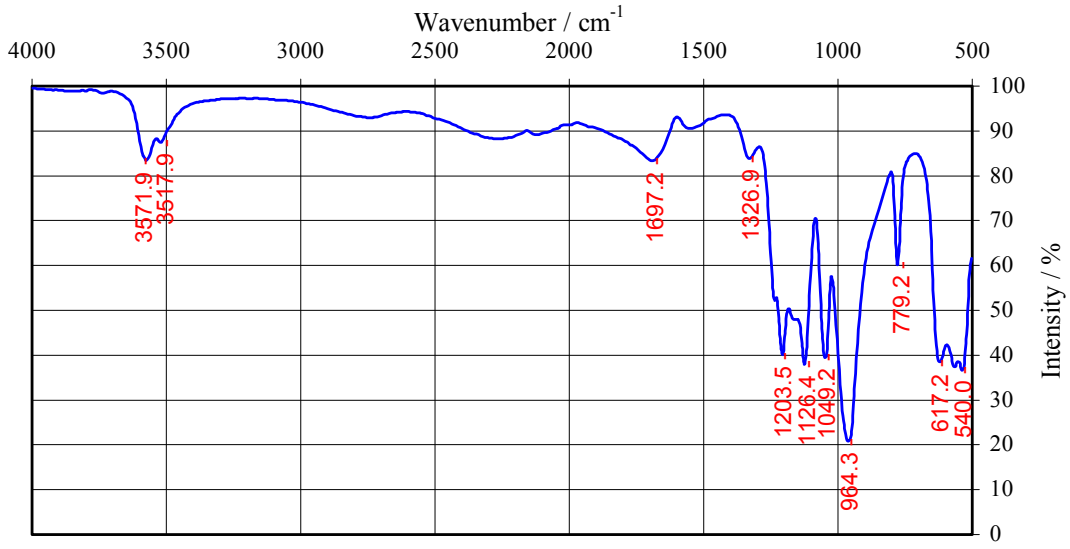

Fig 5. IR Spectrum of $AlH_2P_3O_{10}.2H_2O$

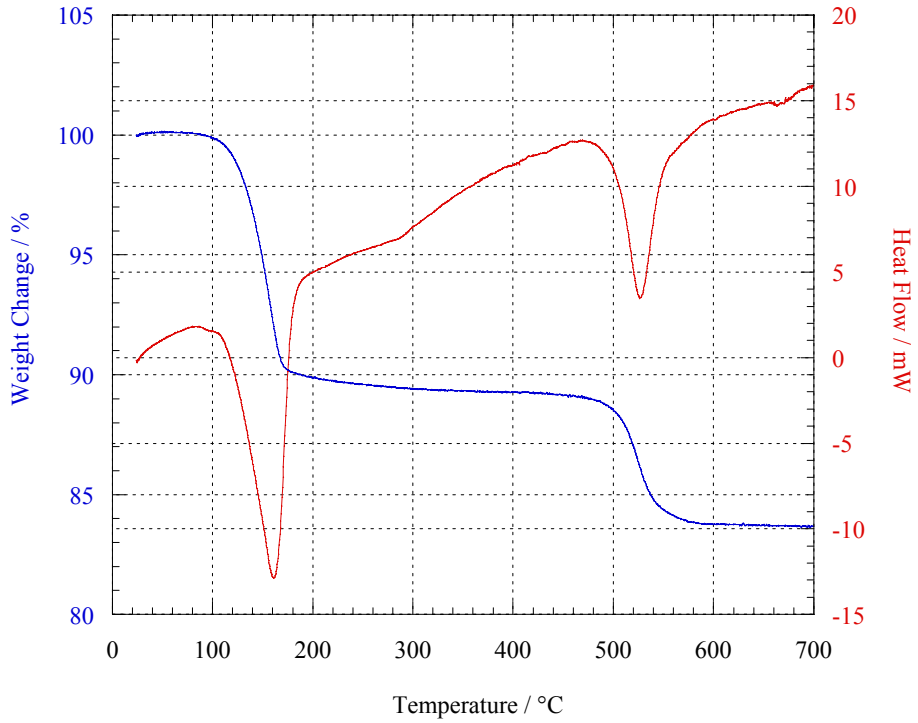

Fig 6. TGA of $AlH_2P_3O_{10}.2H_2O$

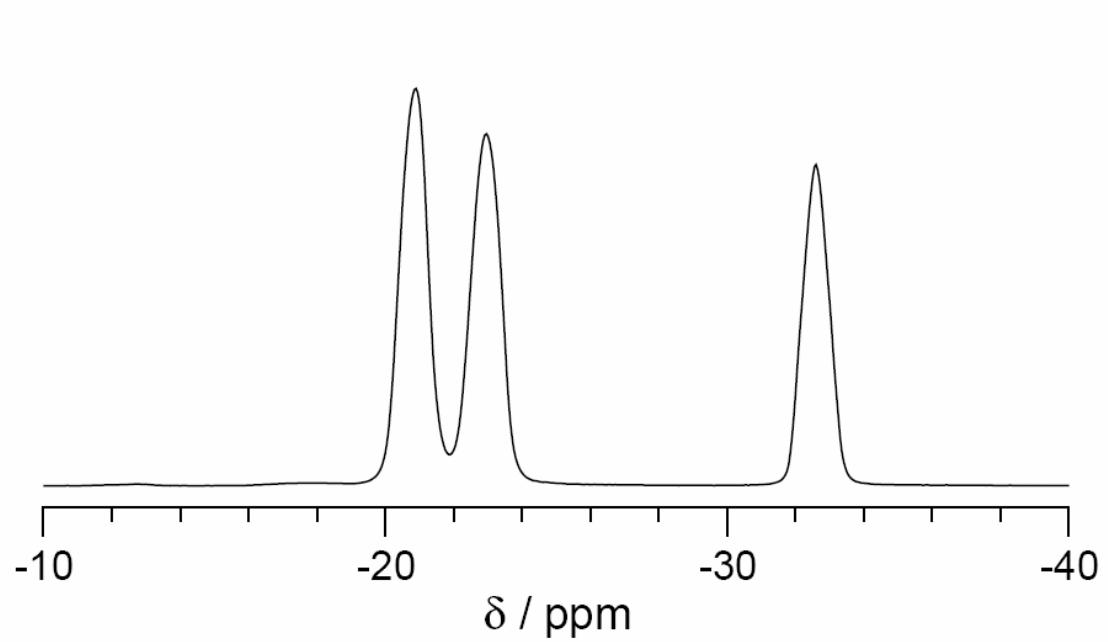

Fig 7. ^{31}P CP MAS NMR spectra of $AlH_2P_3O_{10}.2H_2O$, recorded with ^{1}H decoupling. Centre-band of the spectrum of $AlH_2P_3O_{10}.2H_2O$ recorded with an MAS spinning frequency of 7000 ± 3 Hz.