Supporting Information for:

Synthesis and characterization of layered metal sulfates containing $M^{II}_3(\mu_3\text{-}OH/F)_2$ (M = Mg, Co) diamond chains

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†Electronic Supplementary Information (ESI) available: PXRD and FTIR data as pictures, complete bond lengths and bond angles as tables (PDF) and X-ray crystallographic data in CIF format has been given as supporting information. CCDC contains the supplementary crystallographic data for this paper with a deposition number of CCDC**1406154** (**1**) and **1504134** (**2**). For ESI and crystallographic data in CIF or other electronic format see DOI: 10.1039/x0xx00000x

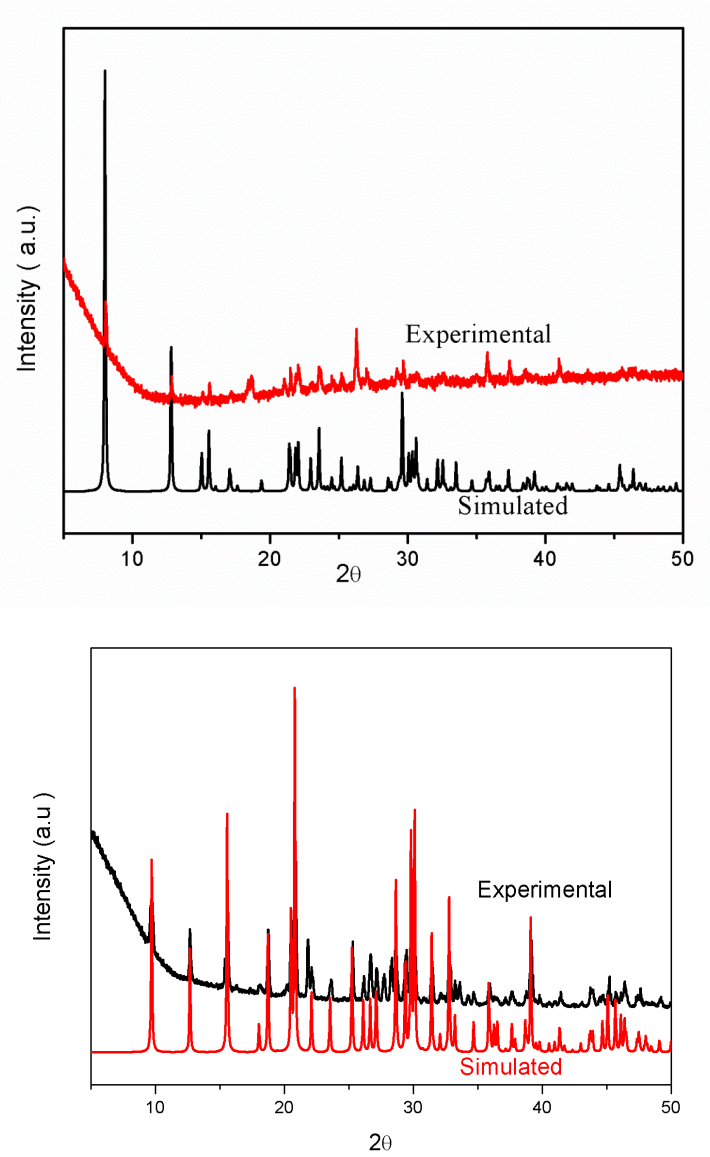

Fig S1 Experimental and simulated powder X-ray diffraction patterns of $[C_4N_2H_{12}]_2[Co_3F_2(SO_4)_3(H_2O)_2]$, **1** and $[NH_4]_2[Mg_3(SO_4)_3(OH)_2(H_2O)_2]$, **2**, indicating the phase purity of the as-synthesized product

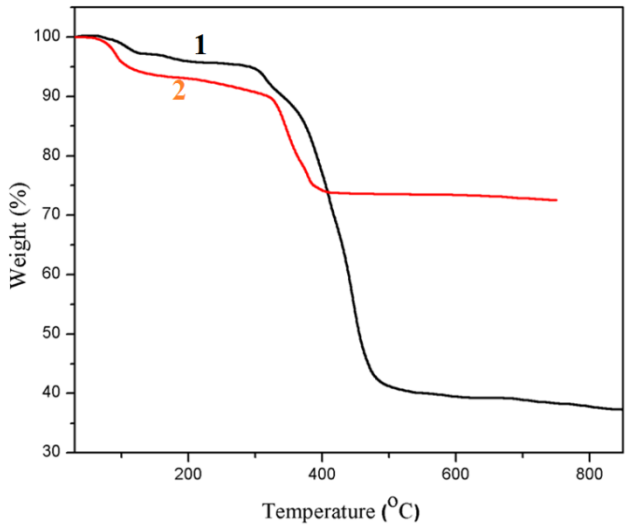

Fig S2 Thermogravimetric data for $[C_4N_2H_{12}]_2[Co_3F_2(SO_4)_3(H_2O)_2]$, **1** and $[NH_4]_2[Mg_3(SO_4)_3(OH)_2(H_2O)_2]$, **2**.

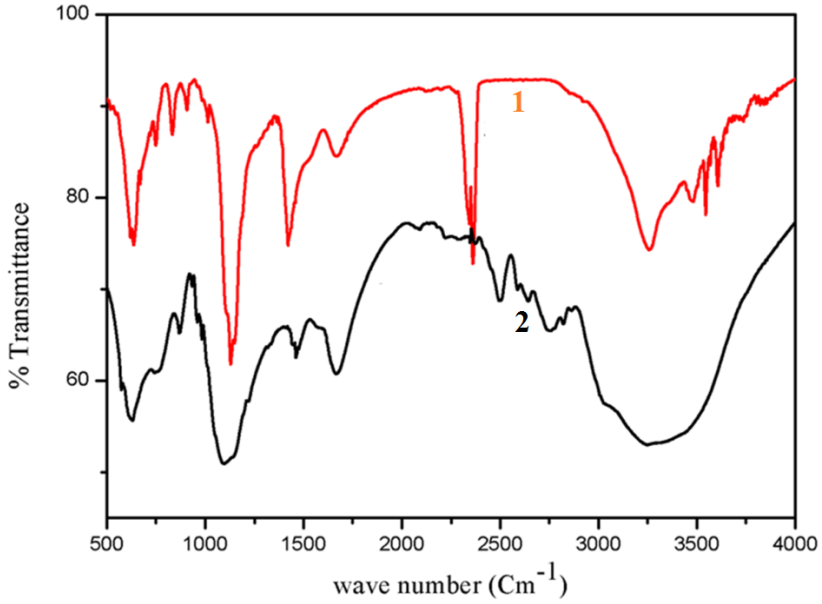

Fig S3 Infrared spectra of $[C_4N_2H_{12}]_2[Co_3F_2(SO_4)_3(H_2O)_2]$, **1** and $[NH_4]_2[Mg_3(SO_4)_3(OH)_2(H_2O)_2]$, **2.**

Table S1 Selected bond distances and angles for $[C_4N_2H_{12}]_2[Co_3F_2(SO_4)_3(H_2O)_2]$, **1.**

Moiety	Distance(A°)	Moiety	Angle(deg)
Co(1)-O(1)	2.0423(11)	O(5)-Co(2)-F(2)	97.34(3)
Co(1)-O(2)	2.0671(10)	O(5)#2-Co(2)-O(6)#3	82.83(3)
Co(1)-F(1)#1	2.0807(7)	O(5)#2-Co(2)-O(7)	90.50(3)
Co(1)-O(3)#1	2.0882(11)	O(6)#3-Co(2)-O(7)	81.25(6)
Co(1)-O(4)	2.1002(12)	O(8)-S(1)-O(5)	108.85(7)
Co(1)-F(2)	2.1159(7)	O(4)-Co(1)-F(2)	92.92(5)
Co(2)-O(5)#2	2.0150(12)	O(5)#2-Co(2)-O(5)	165.29(7)
Co(2)-O(5)	2.0150(12)	O(5)-Co(2)-F(1)	89.56(3)
Co(2)-F(1)	2.0518(11)	O(8)-S(1)-O(1)	109.52(7)
Co(2)-F(2)	2.0768(11)	O(5)-S(1)-O(1)	110.21(7)
Co(2)-O(6)#3	2.1021(14)	O(8)-S(1)-O(3)	110.87(7)
Co(2)-O(7)	2.1804(14)	O(5)-S(1)-O(3)	109.66(7)
S(1)-O(8)	1.4447(11)	O(1)-S(1)-O(3)	107.74(8)
S(1)-O(5)	1.4787(11)	O(6)-S(2)-O(2)#2	107.38(5)
S(1)-O(1)	1.4797(11)	O(2)-S(2)-O(2)#2	111.91(9)
S(1)-O(3)	1.4823(11)	O(6)-S(2)-O(7)	110.87(9)
S(2)-O(6)	1.4662(14)	O(2)-S(2)-O(7)	109.64(5)
S(2)-O(2)	1.4766(11)	F(1)#1-Co(1)-O(3)#1	100.60(4)
S(2)-O(7)	1.4796(14)	F(1)#1-Co(1)-O(4)	86.32(5)
F(1)-Co(1)#4	2.0807(7)	F(1)#1-Co(1)-F(2)	78.02(3)
F(2)-Co(1)#2	2.1159(7)	F(1)-Co(2)-F(2)	90.15(5)
O(3)-Co(1)#5	2.0882(11)	F(1)-Co(2)-O(6)#3	99.27(5)
O(6)-Co(2)#6	2.1022(14)	F(2)-Co(2)-O(6)#3	170.58(5)
N(1)-C(1)	1.493(2)	F(1)-Co(2)-O(7)	179.48(5)
N(1)-C(2)	1.501(2)	F(2)-Co(2)-O(7)	89.33(5)
C(2)-C(1)#7	1.505(2)	Co(2)-F(1)-Co(1)#4	127.67(3)
Moiety	Angle(deg)	Co(1)#4-F(1)-Co(1)#5	101.83(5)
O(1)-Co(1)-O(2)	98.10(5)	Co(2)-F(2)-Co(1)	119.77(4)
O(1)-Co(1)-F(1)#1	170.76(5)	Co(1)-F(2)-Co(1)#2	99.51(5)
O(2)-Co(1)-F(1)#1	88.25(5)	S(1)-O(1)-Co(1)	136.81(8)
O(1)-Co(1)-O(3)#1	86.63(5)	S(2)-O(2)-Co(1)	127.16(7)
O(2)-Co(1)-O(3)#1	85.77(5)	S(1)-O(3)-Co(1)#5	131.09(7)
O(1)-Co(1)-O(4)	87.41(5)	S(1)-O(5)-Co(2)	134.92(7)
O(2)-Co(1)-O(4)	174.48(5)	S(2)-O(6)-Co(2)#6	145.13(10)
O(3)#1-Co(1)-O(4)	94.25(6)	S(2)-O(7)-Co(2)	120.92(8)
O(1)-Co(1)-F(2)	95.53(4)	C(1)-N(1)-C(2)	111.00(13)
O(2)-Co(1)-F(2)	86.92(4)	N(1)-C(1)-C(2)#7	110.58(13)
O(3)#1-Co(1)-F(2)	172.60(5)	N(1)-C(2)-C(1)#7	110.33(13)

Symmetry transformations used to generate equivalent atoms: #1 x+1/2,y,-z+1/2 #2 x,-y+1/2,z #3 x-1/2,y,-z+3/2 #4 x-1/2, -y+1/2,-z+1/2 #5 x-1/2,y,-z+1/2 #6 x+1/2,y,-z+3/2 #7 -x+1,-y+1,-z

Table S2 Selected bond distances and angles for $[NH_4]_2[Mg_3(SO_4)_3(OH)_2(H_2O)_2]$, **2.**

Moiety	Distance(Aº)	Moiety	Angle(deg)
Mg(1)-O(1)	2.0822(17)	O(7)-S(1)-O(3)	109.11(7)
Mg(1)-O(2)	2.0863(16)	O(6)#3-Mg(2)-O(7)	86.36(5)
Mg(1)-O(3)#1	2.0899(11)	O(6)#3-Mg(2)-O(8)	83.67(6)
Mg(1)-O(3)	2.0899(11)	O(7)-Mg(2)-O(8)	88.80(6)
Mg(1)-O(4)#2	2.1207(17)	O(6)#3-Mg(2)-O(1)#4	95.70(6)
Mg(1)-O(5)	2.1491(18)	O(7)-Mg(2)-O(1)#4	172.22(6)
Mg(2)-O(6)#3	2.0444(13)	O(8)-Mg(2)-O(1)#4	98.87(6)
Mg(2)-O(7)	2.0761(13)	O(6)#3-Mg(2)-O(2)	169.74(6)
Mg(2)-O(8)	2.0920(12)	O(7)-Mg(2)-O(2)	96.07(5)
Mg(2)-O(1)#4	2.0949(11)	O(8)-Mg(2)-O(2)	86.41(6)
Mg(2)-O(2)	2.1093(11)	O(1)#4-Mg(2)-O(2)	83.22(5)
Mg(2)-O(9)	2.1304(13)	O(6)#3-Mg(2)-O(9)	92.33(6)
S(1)-O(10)	1.4626(12)	O(7)-Mg(2)-O(9)	83.29(6)
S(1)-O(6)	1.4694(12)	O(8)-Mg(2)-O(9)	171.36(6)
S(1)-O(7)	1.4756(13)	O(1)#4-Mg(2)-O(9)	89.13(6)
S(1)-O(3)	1.4811(11)	O(2)-Mg(2)-O(9)	97.84(6)
S(2)-O(5)	1.4694(16)	O(5)-S(2)-O(8)#1	110.63(7)
S(2)-O(8)#1	1.4739(12)	O(8)#1-S(2)-O(8)	108.74(10)
S(2)-O(4)	1.4756(16)	O(5)-S(2)-O(4)	110.11(9)
O(1)-H(6)	0.89(4)	O(8)-S(2)-O(4)	108.34(6)
O(2)-H(5)	0.73(4)	Mg(2)#5-O(1)-Mg(2)#6	93.99(6)
O(9)-H(10A)	0.84(3)	Mg(1)-O(1)-H(6)	90(3)
O(9)-H(10B)	0.63(4)	Mg(2)-O(2)-H(5)	110.2(18)
N(1)-H(2)	0.85(4)	Mg(2)#6-O(1)-H(6)	109.7(18)
N(1)-H(1)	0.71(5)	Mg(1)-O(1)-Mg(2)#5	126.23(5)
N(1)-H(4)	0.85(5)	Mg(1)-O(2)-Mg(2)	121.80(5)
N(1)-H(3)	0.87(5)	Mg(2)#1-O(2)-Mg(2)	93.15(6)
Moiety	Angle(deg)	Mg(1)-O(2)-H(5)	100(3)
O(1)-Mg(1)-O(2)	90.52(6)	Mg(2)-O(9)-H(10A)	115.2(18)
O(1)-Mg(1)-O(3)#1	92.26(4)	Mg(2)-O(9)-H(10B)	114(5)
O(2)-Mg(1)-O(3)#1	96.51(4)	S(1)-O(3)-Mg(1)	128.13(6)
O(3)#1-Mg(1)-O(3)	166.17(7)	S(2)-O(4)-Mg(1)#7	141.62(10)
O(1)-Mg(1)-O(4)#2	98.37(7)	S(2)-O(5)-Mg(1)	120.41(10)
O(2)-Mg(1)-O(4)#2	171.11(7)	S(1)-O(6)-Mg(2)#6	141.63(9)
O(3)#1-Mg(1)-O(4)#2	83.21(3)	S(1)-O(7)-Mg(2)	132.38(8)
O(1)-Mg(1)-O(5)	178.13(7)	S(2)-O(8)-Mg(2)	127.53(8)
O(2)-Mg(1)-O(5)	91.35(7)	H(10A)-O(9)-H(10B)	98(4)
O(3)#1-Mg(1)-O(5)	87.53(4)	H(2)-N(1)-H(1)	102(5)
O(4)#2-Mg(1)-O(5)	79.76(6)	H(2)-N(1)-H(4)	89(4)
O(10)-S(1)-O(6)	109.45(8)	H(1)-N(1)-H(4)	121(5)
O(10)-S(1)-O(7)	109.09(8)	H(2)-N(1)-H(3)	136(4)
O(6)-S(1)-O(7)	110.45(7)	H(1)-N(1)-H(3)	108(5)
O(10)-S(1)-O(3)	109.13(7)	H(4)-N(1)-H(3)	102(5)
O(6)-S(1)-O(3)	109.58(7)		

Symmetry transformations used to generate equivalent atoms: #1 -x+1,y,z #2 -x+1,-y+2,z+1/2 #3 x,-y+1,z-1/2 #4 -x+1,-y+1,z-1/2 #5 -x+1,-y+1,z+1/2 #6 x,-y+1,z+1/2 #7 -x+1,-y+2,z-1/2

Table S3 Atomic Coordinates [x104] and Equivalent Isotropic Displacement Parameters [Å2 x 103] for $[C_4N_2H_{12}]_2[Co_3F_2(SO_4)_3(H_2O)_2]$, **I** U(eq) is defined as one third of the trace of the orthogonal zed Uij tensor.

Atoms	X	Y	z	U(eq)
Co(1)	8236(1)	3232(1)	2123(1)	13(1)
Co(2)	6023(1)	2500	5381(1)	13(1)
S(1)	5873(1)	3928(1)	4413(1)	14(1)
S(2)	9204(1)	2500	5655(1)	14(1)
F(1)	4210(1)	2500	4079(2)	16(1)
F(2)	6980(1)	2500	2847(2)	16(1)
O(1)	7007(1)	3892(1)	3119(2)	30(1)
O(2)	9272(1)	3054(1)	4521(1)	21(1)
O(3)	4631(1)	3918(1)	3311(2)	27(1)
O(4)	7304(2)	3352(1)	-440(2)	28(1)
O(5)	5891(1)	3405(1)	5686(2)	24(1)
O(6)	10370(1)	2500	6869(2)	23(1)
O(7)	7958(1)	2500	6740(2)	19(1)
O(8)	5967(1)	4479(1)	5475(2)	23(1)
N(1)	4639(1)	5601(1)	580(2)	26(1)
C(1)	5932(2)	5325(1)	1146(2)	26(1)
C(2)	3601(2)	5123(1)	274(2)	28(1)

Table S4 Atomic coordinates (x 10^4) and Equivalent Isotropic Displacement Parameters (Å X 10^3) for $[NH_4]_2[Mg_3(SO_4)_3(OH)_2(H_2O)_2]$, **II** U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atoms	X	Y	Z	U(eq)
Mg(1)	5000	7995(1)	6102(1)	11(1)
Mg(2)	5840(1)	4650(1)	3894(1)	12(1)
S(1)	6712(1)	6942(1)	6189(1)	11(1)
S(2)	5000	8150(1)	2892(1)	10(1)
O(1)	5000	6877(2)	8037(2)	12(1)
O(2)	5000	5480(2)	5224(2)	12(1)
O(3)	6138(1)	8327(1)	6123(2)	17(1)
O(4)	5000	9300(2)	1679(2)	15(1)
O(5)	5000	9232(2)	4134(2)	18(1)
O(6)	6585(1)	5808(2)	7380(1)	19(1)
O(7)	6692(1)	5883(2)	4929(1)	18(1)
O(8)	5657(1)	7015(2)	2840(1)	19(1)
O(9)	6191(1)	2387(2)	5012(1)	20(1)
O(10)	7431(1)	7789(2)	6308(2)	21(1)
N(1)	8063(1)	5421(2)	8480(2)	26(1)

Table S5 The Hydrogen Bonding Interactions of the compounds $[C_4N_2H_{12}]_2[Co_3F_2(SO_4)_3(H_2O)_2]$, **1** and $[NH_4]_2[Mg_3(OH)_2(SO_4)_3(H_2O)_2]$, **2.**

D-H...A	D...A(A°)	H...A(A°)	D...H(A°)	D-H...A(A°)
		1		
N(1)-H(1)...O(4)	3.028(2)	2.53	0.90	116
N(1)-H(1)...O(8)	2.9347(18)	2.37	0.90	121
N(1)-H(2)...O(3)	3.1066(19)	2.24	0.90	162
O(4)-H(7)...O(7)	2.8566(17)	2.10(3)	0.86(3)	146(3)
O(4)-H(8)...O(6)	2.8961(18)	2.29(5)	0.72(5)	143(4)
O(4)-H(8)...O(2)	3.1873(19)	2.51(5)	0.72(5)	157(5)
C(2)-H(4)...O(8)	3.053(2)	2.60	0.97	109
C(1)-H(5)...O(8)	3.1833(19)	2.54	0.97	124
C(1)-H(6)...O(8)	3.1383(18)	2.56	0.97	118

D-H...A	D...A(A°)	H...A(A°)	D...H(A°)	D-H...A(A°)
		2		
N(1)-H(1)...0(9)	2.935(2)	2.32(5)	0.71(5)	146(5)
N(1)-H(2)...0(7)	3.041(2)	2.27(4)	0.86(4)	150(3)
N(1)-H(2)...0(6)	2.918(3)	2.59(4)	0.86(4)	104(3)
N(1)-H(3)...0(3)	3.163(2)	2.36(5)	0.88(5)	151(4)
N(1)-H(3)...0(10)	3.054(2)	2.39(5)	0.88(5)	133(4)
N(1)-H(4)...0(7)	3.165(2)	2.57(5)	0.85(5)	129(4)
N(1)-H(4)...0(10)	3.015(2)	2.46(5)	0.85(5)	124(4)
O(1)-H(6)...0(5)	3.130(2)	2.24(4)	0.89(4)	174(4)
O(9)-H(10A)...0(4)	3.0039(18)	2.17(3)	0.84(3)	174(3)
O(9)-H(10A)...0(8)	2.9808(19)	2.44(3)	0.84(3)	124(2)
O(9)-H(10B)...0(10)	2.8333(19)	2.24(4)	0.63(5)	156(5)