Electronic Supplementary Material (ESI) for CrystEngComm

Amine-templated polymeric Mg formates: crystalline scaffolds exhibiting extensive hydrogen bonding

Andrea Rossin,[a,*] *Michele R. Chierotti,*[b,c] *Giuliano Giambastiani,*[a] *Roberto Gobetto,*[b] *Maurizio Peruzzini,*[a,*]

[a] Consiglio Nazionale delle Ricerche, Istituto di Chimica dei Composti Organometallici (ICCOM-CNR), Via Madonna del Piano 10, 50019 Sesto Fiorentino (Firenze), Italy.

[b] Dipartimento di Chimica IFM, Università di Torino (Torino), Italy.

[c] NIS Centre of Excellence, Via Quarello 11, 10135 Torino, Italy.

Supporting Information

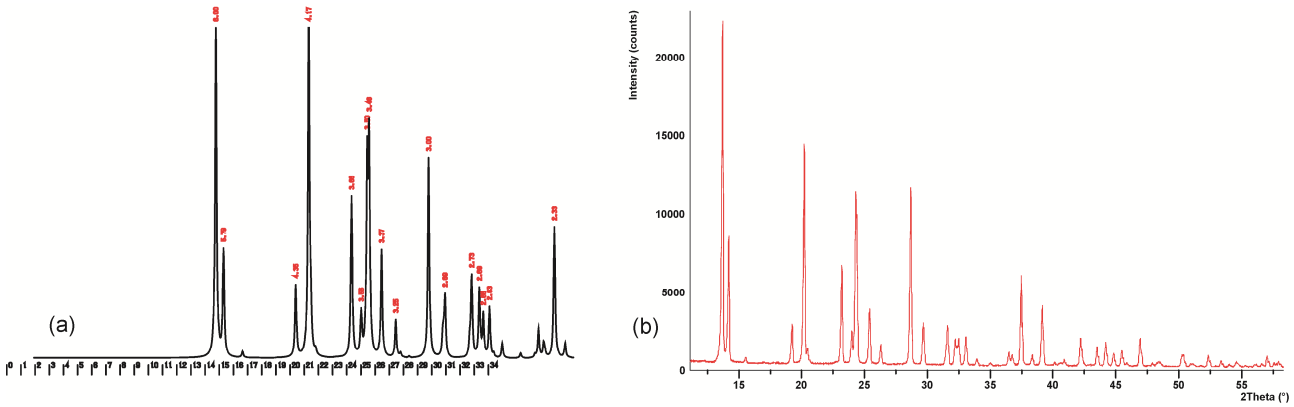

Figure S1. (a) Simulated and (b) experimental powder X-ray diffractogram of **1**. The red numbers on the simulated pattern indicate the corresponding *d*-spacing.

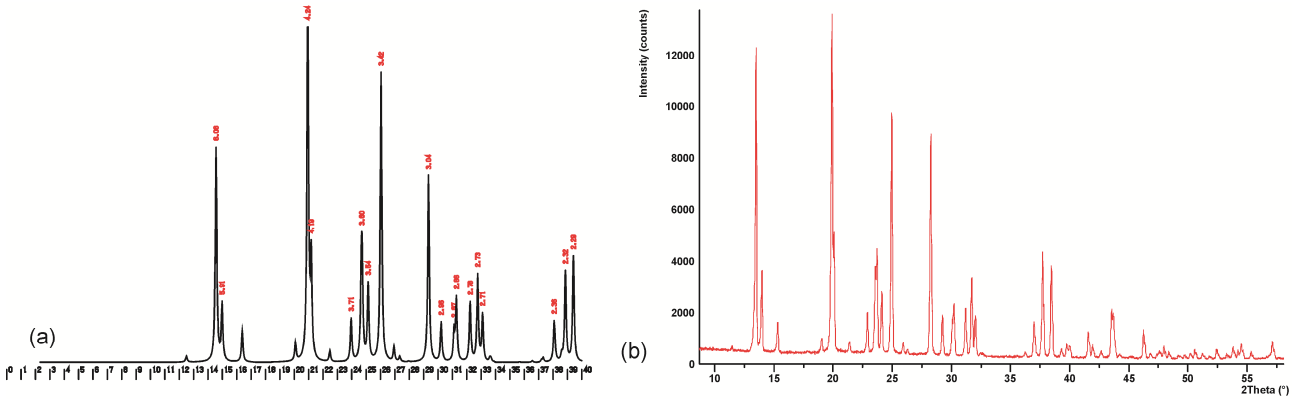

Figure S2. (a) Simulated and (b) experimental powder X-ray diffractogram of **2**. The red numbers on the simulated pattern indicate the corresponding *d*-spacing.

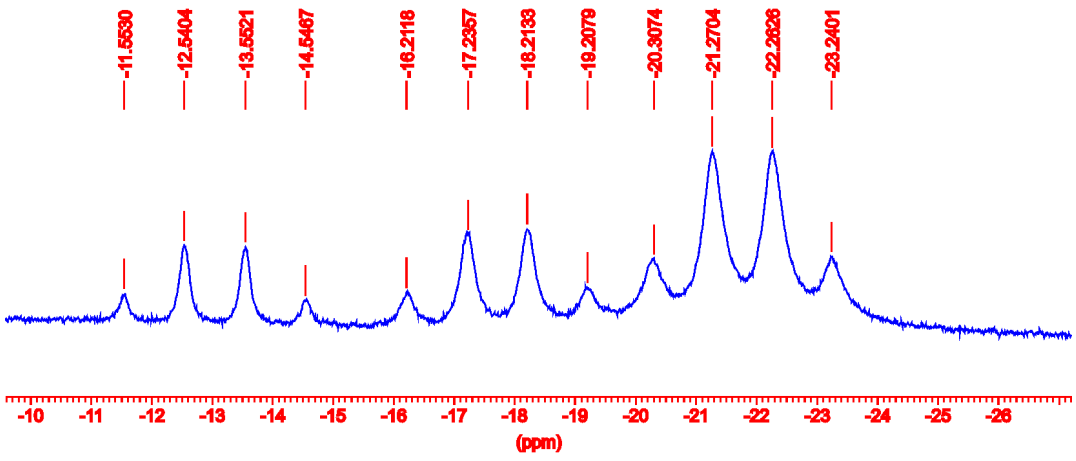

Figure S3. ^{11}B NMR spectrum of the **1**/Ca(BH$_4$)$_2$ THF mixture after reflux for 18 h.

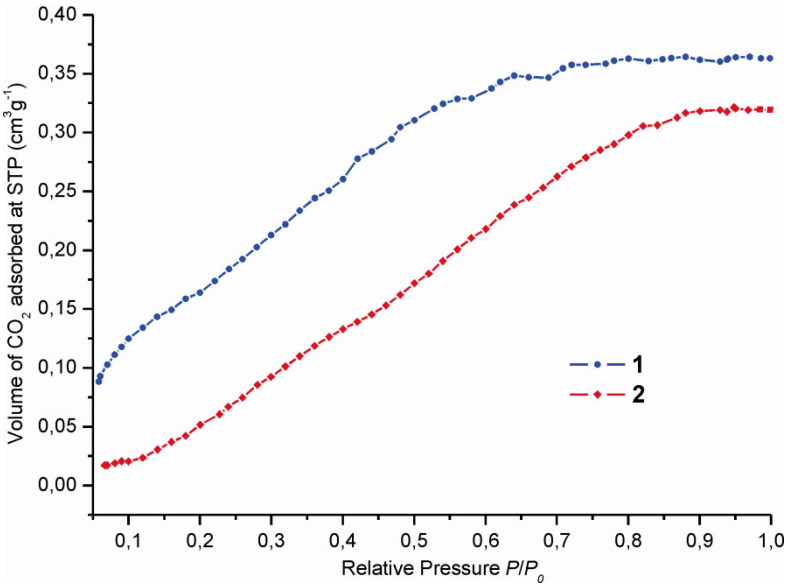

Figure S4. CO_2 adsorption isotherm (at 195 K) of **1** and **2**.

Table S1. Crystal data and structure refinement for **1**.

Empirical formula	$C_4 H_8 Mg N_2 O_6$	
Formula weight	204.43	
Temperature	120(2) K	
Wavelength	0.71069 Å	
Crystal system	Orthorhombic	
Space group	*P n n a*	
Unit cell dimensions	a = 8.709(4) Å	α= 90°.
	b = 11.587(5) Å	β= 90°.
	c = 8.280(3) Å	γ = 90°.
Volume	835.5(9) $Å^3$	
Z	4	
Density (calculated)	1.625 Mg/m^3	
Absorption coefficient	0.216 mm^{-1}	
F(000)	424	
Crystal size	0.1 x 0.4 x 0.4 mm^3	
Theta range for data collection	4.68 to 28.95°.	
Index ranges	$-5 \le h \le 11$, $-14 \le k \le 10$, $-10 \le l \le 10$	
Reflections collected	3247	
Independent reflections	991 [R(int) = 0.0195]	
Completeness to theta = 28.95°	88.8 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	991 / 0 / 68	
Goodness-of-fit on F^2	1.109	
Final R indices [I>2sigma(I)]	R1 = 0.0293, wR2 = 0.0791	
R indices (all data)	R1 = 0.0331, wR2 = 0.0819	
Largest diff. peak and hole	0.190 and – 0.305 e.$Å^{-3}$	

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **1**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2500	5000	1884(2)	14(1)
C(2)	-297(1)	2197(1)	257(1)	15(1)
C(3)	7500	0	2059(2)	19(1)
N(1)	6504(1)	651(1)	2794(1)	21(1)
O(1)	2406(1)	4040(1)	1187(1)	14(1)
O(2)	875(1)	1798(1)	908(1)	17(1)
O(3)	4159(1)	1858(1)	1064(1)	16(1)
Mg(1)	2410(1)	2500	2500	10(1)

Table S3. (Selected) bond lengths [Å] and angles [°] for **1**.

C(1)-O(1)	1.2553(12)
C(2)-O(2)	1.2431(14)
C(2)-O(3)#2	1.2550(14)
C(3)-N(1)	1.3009(14)
C(3)-N(1)#3	1.3009(14)
C(3)-H(3)	0.97(2)
N(1)-H(4)	0.878(16)
N(1)-H(5)	0.842(16)
O(1)-Mg(1)	2.0897(11)
O(2)-Mg(1)	2.0464(11)
O(3)-C(2)#4	1.2550(14)
O(3)-Mg(1)	2.0706(11)
Mg(1)-O(2)#5	2.0464(11)
Mg(1)-O(3)#5	2.0706(11)
Mg(1)-O(1)#5	2.0897(11)
O(1)-C(1)-O(1)#1	125.29(15)
O(2)-C(2)-O(3)#2	124.82(11)
N(1)-C(3)-N(1)#3	124.22(17)
N(1)-C(3)-H(3)	117.89(9)
N(1)#3-C(3)-H(3)	117.89(9)

C(3)-N(1)-H(4)	117.6(10)
C(3)-N(1)-H(5)	120.1(10)
H(4)-N(1)-H(5)	122.2(14)
C(1)-O(1)-Mg(1)	121.16(9)
C(2)-O(2)-Mg(1)	131.91(8)
C(2)#4-O(3)-Mg(1)	131.72(7)
O(2)-Mg(1)-O(2)#5	98.38(7)
O(2)-Mg(1)-O(3)#5	173.45(3)
O(2)#5-Mg(1)-O(3)#5	88.16(5)
O(2)-Mg(1)-O(3)	88.16(5)
O(2)#5-Mg(1)-O(3)	173.45(4)
O(3)#5-Mg(1)-O(3)	85.29(6)
O(2)-Mg(1)-O(1)#5	89.69(4)
O(2)#5-Mg(1)-O(1)#5	90.18(4)
O(3)#5-Mg(1)-O(1)#5	90.55(4)
O(3)-Mg(1)-O(1)#5	89.61(4)
O(2)-Mg(1)-O(1)	90.18(4)
O(2)#5-Mg(1)-O(1)	89.69(4)
O(3)#5-Mg(1)-O(1)	89.61(4)
O(3)-Mg(1)-O(1)	90.55(4)
O(1)#5-Mg(1)-O(1)	179.79(5)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1/2,-y+1,z #2 x-1/2,y,-z #3 -x+3/2,-y,z #4 x+1/2,y,-z #5 x,-y+1/2,-z+1/2

Table S4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for **1**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	17(1)	14(1)	11(1)	0	0	0(1)
C(2)	16(1)	15(1)	14(1)	-1(1)	-2(1)	1(1)
C(3)	25(1)	21(1)	12(1)	0	0	-2(1)
N(1)	23(1)	25(1)	16(1)	2(1)	-4(1)	7(1)
O(1)	18(1)	11(1)	12(1)	0(1)	0(1)	0(1)
O(2)	18(1)	16(1)	19(1)	-1(1)	-8(1)	1(1)
O(3)	15(1)	18(1)	14(1)	3(1)	4(1)	3(1)
Mg(1)	10(1)	11(1)	9(1)	0(1)	0	0

Table S5. Crystal data and structure refinement for **2**.

Empirical formula	$C_4 H_9 Mg N_3 O_6$	
Formula weight	219.45	
Temperature	298(2) K	
Wavelength	0.71069 Å	
Crystal system	Orthorhombic	
Space group	*P n n a*	
Unit cell dimensions	a = 8.391(2) Å	α= 90°.
	b = 11.831(2) Å	β= 90°.
	c = 8.842(1) Å	γ = 90°.
Volume	877.8(3) $Å^3$	
Z	4	
Density (calculated)	1.661 Mg/m^3	
Absorption coefficient	0.214 mm^{-1}	
F(000)	456	
Crystal size	0.05 x 0.05 x 0.08 mm^3	
Theta range for data collection	4.61 to 27.00°.	
Index ranges	$-10 \le h \le 10, -14 \le k \le 14, -10 \le l \le 11$	
Reflections collected	9952	
Independent reflections	873 [R(int) = 0.2563]	
Completeness to theta = 27.00°	90.1 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	873 / 0 / 75	
Goodness-of-fit on F^2	1.124	
Final R indices [I>2sigma(I)]	R1 = 0.1031, wR2 = 0.2709	
R indices (all data)	R1 = 0.1345, wR2 = 0.3236	
Largest diff. peak and hole	1.169 and – 0.779 $e.Å^{-3}$	

Table S6. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **2**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	7500	0	8000(6)	42(2)
C(2)	5181(5)	2770(4)	10190(4)	37(1)
C(3)	7500	5000	2436(6)	35(2)
N(1)	8372(5)	4255(3)	3166(4)	56(2)
N(2)	7500	5000	941(5)	47(2)
O(1)	7516(3)	943(3)	8602(3)	35(1)
O(2)	5608(4)	3048(2)	8894(3)	37(1)
O(3)	9024(4)	3124(2)	9117(3)	41(1)
Mg(1)	7474(2)	2500	7500	28(1)

Table S7. (Selected) bond lengths [Å] and angles [°] for **2**.

C(1)-O(1)	1.236(4)
C(1)-H(1)	0.9300
C(2)-O(2)	1.245(4)
C(2)-H(2)	0.9300
C(3)-N(1)	1.315(4)
C(3)-N(2)	1.322(7)
N(1)-H(4)	0.84(6)
N(1)-H(5)	1.00(5)
N(2)-H(3)	0.75(5)
O(1)-Mg(1)	2.084(3)
O(2)-Mg(1)	2.095(3)
O(3)-C(2)#4	1.222(5)
O(3)-Mg(1)	2.069(3)
O(1)-C(1)-H(1)	115.5
O(3)#2-C(2)-H(2)	116.6
O(2)-C(2)-H(2)	116.6
N(1)-C(3)-N(1)#3	121.2(5)
N(1)-C(3)-N(2)	119.4(2)
N(1)#3-C(3)-N(2)	119.4(2)
C(3)-N(1)-H(4)	114(4)
C(3)-N(1)-H(5)	122(3)

H(4)-N(1)-H(5)	124(5)
C(3)-N(2)-H(3)	119(5)
C(1)-O(1)-Mg(1)	126.6(3)
C(2)-O(2)-Mg(1)	132.5(3)
C(2)#4-O(3)-Mg(1)	136.3(3)
O(3)-Mg(1)-O(3)#5	102.12(19)
O(3)-Mg(1)-O(1)#5	89.83(11)
O(3)#5-Mg(1)-O(1)#5	88.96(11)
O(3)-Mg(1)-O(1)	88.96(11)
O(3)#5-Mg(1)-O(1)	89.83(10)
O(3)#5-Mg(1)-O(2)#5	87.30(12)
O(1)#5-Mg(1)-O(2)#5	90.63(10)
O(1)-Mg(1)-O(2)#5	90.80(11)
O(3)-Mg(1)-O(2)	87.30(12)
O(3)#5-Mg(1)-O(2)	170.58(13)
O(1)#5-Mg(1)-O(2)	90.80(11)
O(1)-Mg(1)-O(2)	90.63(10)
O(2)#5-Mg(1)-O(2)	83.29(17)

Symmetry transformations used to generate equivalent atoms:

#1 -x+3/2,-y,z #2 x-1/2,y,-z+2 #3 -x+3/2,-y+1,z #4 x+1/2,y,-z+2 #5 x,-y+1/2,-z+3/2

Table S8. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for **2**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	88(6)	24(3)	14(3)	0	0	-2(2)
C(2)	52(3)	38(3)	23(2)	5(2)	5(2)	4(2)
C(3)	55(5)	28(4)	20(3)	0	0	0(2)
N(1)	83(4)	59(3)	25(2)	5(2)	5(2)	21(2)
N(2)	84(5)	41(4)	17(3)	0	0	16(3)
O(1)	60(3)	29(2)	16(2)	1(1)	0(1)	3(1)
O(2)	49(2)	43(2)	18(2)	-1(1)	7(1)	4(1)
O(3)	55(2)	41(2)	26(2)	-2(1)	-16(1)	0(1)
Mg(1)	47(2)	26(2)	12(1)	0(1)	0	0