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The synthesis, structure and magnetism studies of two manganese sulfates with a 3D zeolite GIS framework and 1D chain structure

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Crystal structure determinations

Suitable single crystals of **1** (0.22 × 0.17 × 0.32 mm3) and **2** (0.12 × 0.12 × 0.50 mm^3) were selected for single-crystal X-ray diffraction analyses. The measurements were carried out on a Bruker SMART APEX CCD diffractometer operating at room temperature. Intensities were collected with graphite monochromatized Mo-Kα radiation (λ = 0.71073 Å) operating at 50 kV and 30 mA. Data reductions and absorption corrections were performed using the SAINT and SADABS programs [1], respectively. The structures were solved by direct methods using the SHELXS-97 program and refined with full-matrix least squares on F^2 using the SHELXL-97 program [2]. Anisotropic thermal parameters were refined for all non-hydrogen atoms. Details of the crystal parameters, data collection and refinement results are summarized in the Table S1. Selected interatomic bond lengths and angles are listed in Table S2.

Table S1 Crystallographic and structural data for **1** and **2**

Compound reference	**1**	**2**
Chemical formula	$C_2H_6MnO_6S$	$C_8H_{28}N_4Mn_2S_4O_{18}$
Formula Mass	213.07	706.46
Crystal system	Orthohombic	Monoclinic
a/Å	12.4527(18)	13.0864(10)
b/Å	13.616(2)	11.9903(9)
c/Å	13.984(2)	14.3224(11)
α/°	90.00	90.00
β/°	90.00	90.897(2)
γ/°	90.00	90.00
Unit cell volume/$Å^3$	2371.2(6)	2247.1(3)
Temperature/K	291(2)	296(2)
Space group	*Fddd*	*P*21/*c*
No. of formula units per unit cell, *Z*	16	4
Absorption coefficient, μ/mm^{-1}	2.546	1.592
No. of reflections measured	2983	17692
No. of independent reflections	590	5628
R_{int}	0.0401	0.0480
Final R_1 values ($I > 2\sigma(I)$) [a]	0.0202	0.0447
Final $wR(F^2)$ values ($I > 2\sigma(I)$) [a]	0.0552	0.1167
Final R_1 values (all data) [a]	0.0210	0.0568
Final $wR(F^2)$ values (all data) [a]	0.0559	0.1238
Goodness of fit on F^2	1.050	1.046
CCDC number	896280	896281

[a] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, $wR = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$.

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Table S2 Selected interatomic distances (Å) and angles (deg) for **1** and **2**[a]

1

Mn1—O1	2.1828(14)	O3—Mn1—O3^{ii}	88.35(6)	O3—Mn1—O1	89.67(5)
Mn1—O3	2.1585(12)	O3—Mn1—O2^{v}	82.77(4)	O1^{ii}—Mn1—O1	95.99(8)
Mn1—O2	2.1790(12)	O3^{ii}—Mn1—O2^{v}	107.35(5)	C1—O1—Mn1	129.23(11)
S1—O2	1.4715(12)	O2^{v}—Mn1—O2^{iv}	166.20(7)	S1—O3—Mn1	132.61(7)
S1—O3	1.4835(12)	O3—Mn1—O1^{ii}	164.94(5)	O2^{iii}—S1—O2	110.11(10)
C1—O1	1.436(2)	O2^{v}—Mn1—O1^{ii}	83.58(5)	O2—S1—O3	109.33(7)
C1—C1^{i}	1.506(3)	O2^{iv}—Mn1—O1^{ii}	87.19(5)	O3—S1—O3^{iii}	109.71(10)

2

Mn1—O11^{ii}	2.108(2)	S1—O3	1.483(2)	O14—Mn1—O8	94.98(10)	O1—S1—O3	107.98(16)
Mn1—O14	2.114(2)	S1—O4	1.463(2)	O8—Mn1—O18	78.9(1)	O4—S1—O3	108.98(15)
Mn1—O8	2.143(2)	S3—O10	1.463(2)	O11^{ii}—Mn1—O14	95.6(1)	O7—S2—O5	112.40(15)
Mn1—O18	2.215(3)	S3—O12	1.463(2)	O14—Mn1—O13	145.96(8)	O7—S2—O8	107.41(13)
Mn1—O13	2.256(2)	S3—O13	1.481(2)	O18—Mn1—O12	154.47(10)	O5—S2—O6	105.10(13)
Mn1—O12	2.305(2)	S2—O5	1.463(2)	O13—Mn1—O12	61.72(8)	O8—S2—O6	109.97(14)
Mn2—O10	2.103(2)	S2—O8	1.465(2)	O4—Mn2—O6	166.97(9)	O11—S3—O10	107.24(13)
Mn2—O4	2.106(2)	S2—O6	1.486(2)	O10—Mn2—O7^{i}	169.97(10)	O11—S3—O12	111.74(15)
Mn2—O7^{i}	2.145(2)	S2—O7	1.460(2)	O4—Mn2—O7^{i}	89.6(1)	O10—S3—O12	112.15(15)
Mn2—O9	2.155(2)	S3—O11	1.462(2)	O9—Mn2—O5	158.01(9)	O12—S3—O13	105.26(13)
Mn2—O6	2.252(2)	S4—O16	1.453(3)	O6—Mn2—O5	61.13(8)	O16—S4—O15	111.12(15)
Mn2—O5	2.349(2)	S4—O15	1.471(2)	O9—Mn2—O6	98.06(9)	O16—S4—O17	109.68(16)
S1—O1	1.450(3)	S4—O17	1.471(2)	O2—S1—O1	109.8(2)	O15—S4—O14	108.07(14)
S1—O2	1.447(3)	S4—O14	1.476(2)	O2—S1—O4	109.95(16)	O17—S4—O14	107.63(15)

[a] Symmetry transformations used to generate equivalent atoms. for **1**: i: -x, 2-y, 1-z; ii: 1/4-x, 9/4-y, z; iii: 1/4-x, y, 1/4-z; iv: -1/4+x, 1/4+y, 1/2-z; v: 1/2-x, 2-y, 1/2-z ; for **2**: (i) 1-x, 1-y, -z; (ii) 2-x, 1-y, -z.

Characterization

Powder X-ray diffraction (PXRD) data were obtained using a Bruker D8 Advance diffractometer with Cu Kα radiation (λ =1.54056 Å), with a step speed of 0.2° per second. FT-IR spectra were recorded on a Nicolet Impact 410 spectrometer between 400 and 4000 cm^{-1} using the KBr pellet method. Thermogravimetric analyses (TGA) were conducted on a Perkin-Elmer Pyris I thermogravimetric analyzer with a heating rate of 20°C·min^{-1} in an N_2 atmosphere. The temperature-dependent magnetic susceptibility measurements of compounds **1** and **2** were performed on the powdered samples in the temperature range of 1.8–300 K at a 2000 Oe external field with a Quantum Design MPMS-XL7 SQUID magnetometer.

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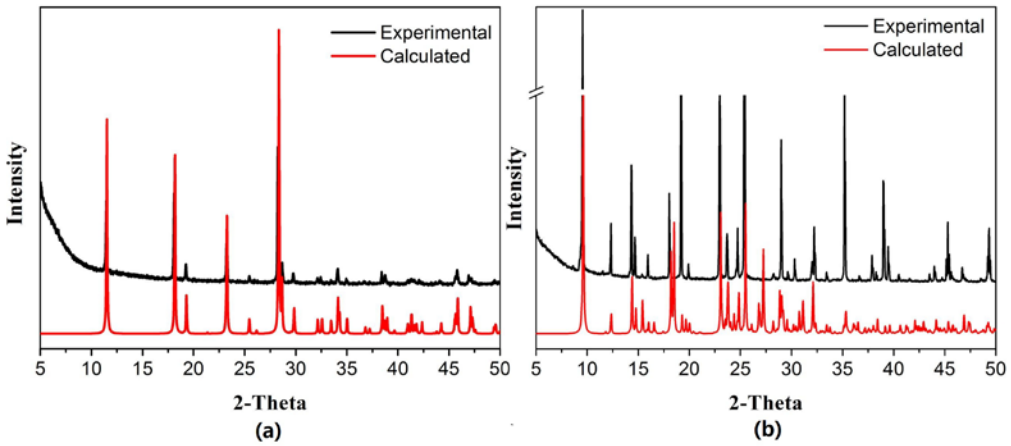

Fig. S1 Powder X-ray diffraction (PXRD) patterns of **1** (a) and **2** (b).

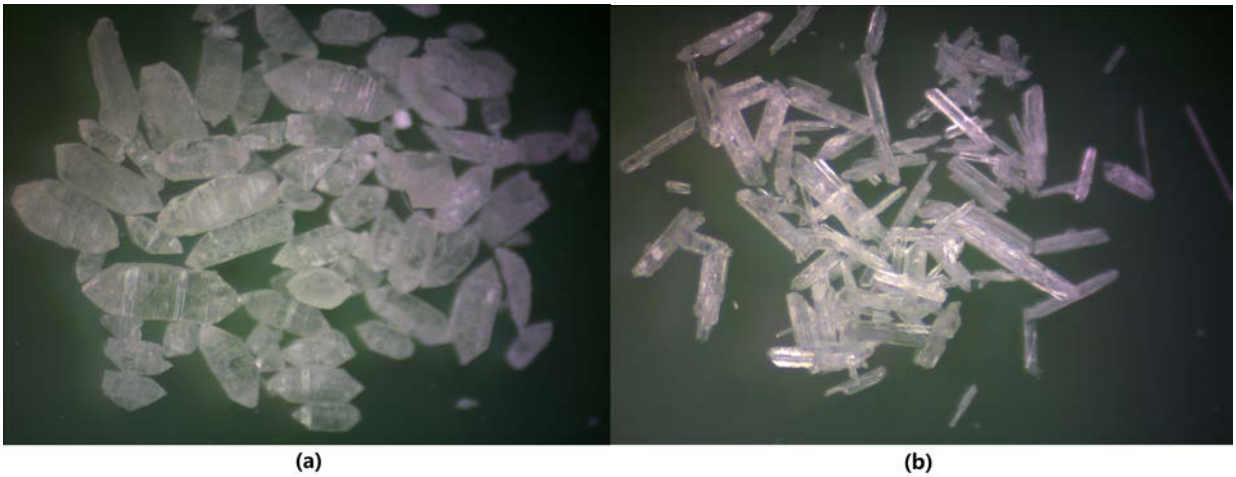

Fig. S2 The microscopy images of crystals of **1** (a, ×100) and **2** (b, ×200)**.**

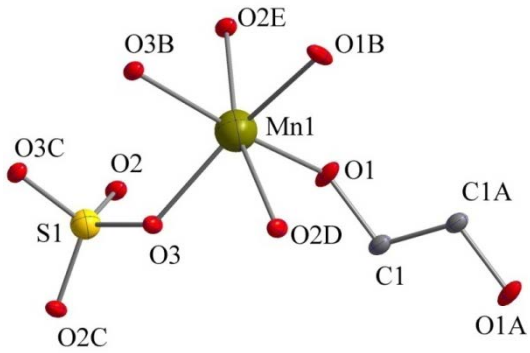

Fig. S3 ORTEP drawing of the coordination environment of Mn in **1** (A: -x, 2-y, 1-z; B: 1/4-x, 9/4-y, z; C: 1/4-x, y, 1/4-z; D: -1/4+x, 1/4+y, 1/2-z; E: 1/2-x, 2-y, 1/2-z).

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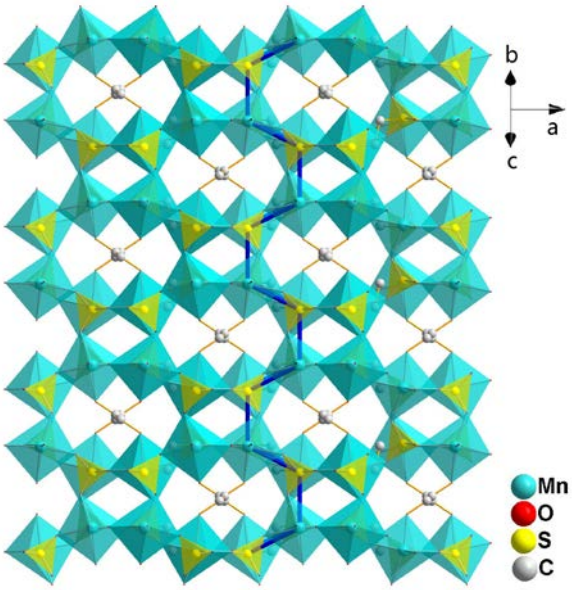

Fig. S4 Polyhedral presentations of **1** along the [011] direction, showing the 8-MR channels and the residing EG molecules (all the H atoms are omitted for clarity).

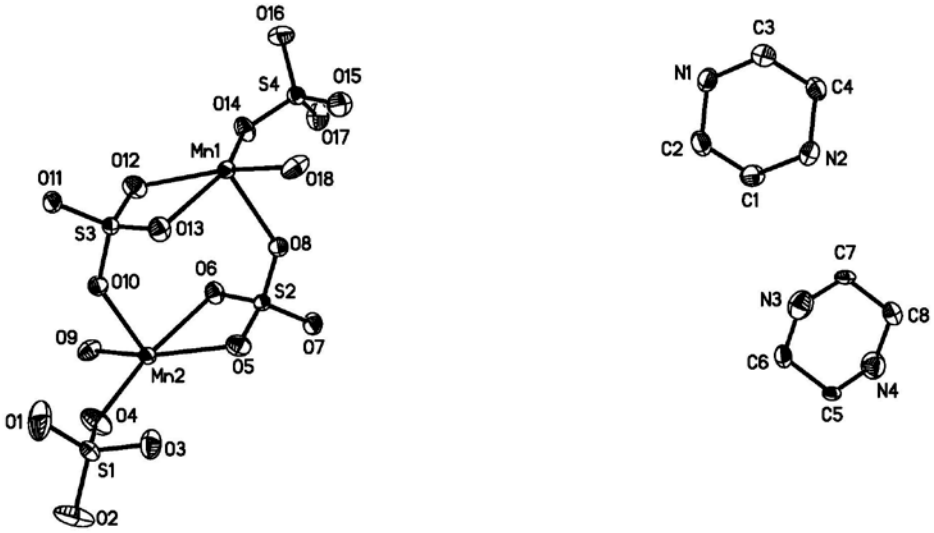

Fig. S5 ORTEP drawing of the asymmetric unit of **2** with ellipsoids drawn at the 50% probability.

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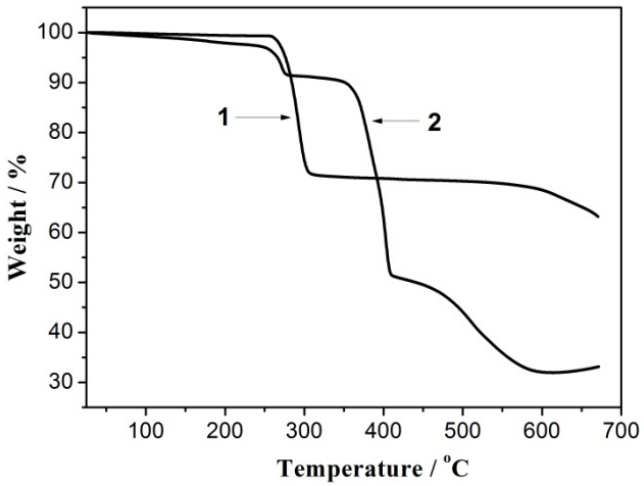

Fig. S6 TG curves for **1** and **2** measured in a N_2 atmosphere

References:

[1] SMART and SADABS, Bruker AXS Inc., Madison, Wisconsin, USA.

[2] G. M. Sheldrick, *Acta Crystallogr.*, 2008, **A64**, 112–122.