Table 1. Crystal Data and Structure Refinement for Compounds **1**–**4**

	1	**2**	**3**	**4**
empirical formula	$C_5H_{26}MnN_2O_{14}S_2$	$C_5H_{26}FeN_2O_{14}S_2$	$C_5H_{26}CoN_2O_{14}S_2$	$C_5H_{26}NiN_2O_{14}S_2$
formula weight	457.33	458.24	461.32	461.08
temperature (K)	293(2)	293(2)	293(2)	293(2)
crystal system	monoclinic	monoclinic	monoclinic	monoclinic
space group	$P2_1/n$	$P2_1/c$	$P2_1/n$	$P2_1/c$
a (Å)	6.6385(10)	10.9273(2)	6.5710(2)	10.8328(2)
b (Å)	11.0448(2)	7.8620(10)	10.9078(3)	7.8443(10)
c (Å)	12.6418(2)	11.7845(3)	12.5518(3)	11.6790(2)
ß (deg)	101.903(10)	116.733(10)	101.547(2)	116.826(10)
V(Å^3)	906.98(3)	904.20(3)	881.44(4)	885.63(2)
Z	2	2	2	2
D_{calc}, (g·cm^{-3})	1.675	1.683	1.738	1.729
$\lambda_{(MoK\alpha)}$ (Å)	0.71073	0.71073	0.71073	0.71073
crystal form, color	prism-white	prism-dark green	prism-red	prism-clear green
crystal size (mm)	0.31 × 0.07 × 0.03	0.30 × 0.11 × 0.07	0.22 × 0.14 × 0.06	0.1 × 0.21 × 0.08
μ (mm^{-1})	1.026	1.133	1.280	1.401
Θ ranges (deg)	2.91 – 27.48	2.91 – 30.50	2.91 – 27.48	2.91 – 27.48
unique data	2073	2750	2025	2012
observed data [$I > 2\sigma(I)$]	1893	2290	1862	1955
F(000)	478	480	482	484
goodness-of-fit on F^2	1.085	1.060	1.141	1.050
R_1, wR_2 $[I > 2\sigma(I)]^{\alpha}$	0.0398, 0.1009	0.0431, 0.1070	0.0426, 0.1156	0.0414, 0.1173
no. parameters	199	122	199	140
transmission factors	0.8045 – 0.9106	0.6722 – 0.8767	0.5494 – 0.8135	0.4936 – 0.7624
$(\Delta\rho)_{min}$, $(\Delta\rho)_{max}$, eÅ^{-3}	– 0.366 and 0.505	– 0.644 and 0.831	– 0.888 and 0.742	– 0.479 and 0.430

Table 2. Selected Bond Distances (Å) and Angles (deg) for Compounds **1–4**[a]

Octahedron around Mn (**1**)			
Mn(1)–OW(1)	2.2013(2)	OW(3)–Mn(1)–OW(1)	88.38(7)
Mn(1)–OW(2)	2.1856(2)	OW(3)–Mn(1)–OW(1)$^{\#1}$	91.62(7)
Mn(1)–OW(3)	2.1602(2)	OW(3)–Mn(1)–OW(2)	86.48(8)
OW(2)–Mn(1)–OW(1)	91.82(8)	OW(3)–Mn(1)–OW(2)$^{\#1}$	93.52(8)
OW(2)–Mn(1)–OW(1)$^{\#1}$	88.18(8)		
Octahedron around Fe (**2**)			
Fe(1)–OW(1)	2.1036(2)	OW(3)–Fe(1)–OW(1)	91.87(7)
Fe(1)–OW(2)	2.1403(2)	OW(3)–Fe(1)–OW(1)$^{\#2}$	88.13(7)
Fe(1)–OW(3)	2.1263(2)	OW(3)–Fe(1)–OW(2)	86.59(7)
OW(2)–Fe(1)–OW(1)	89.99(7)	OW(3)–Fe(1)–OW(2)$^{\#2}$	93.41(7)
OW(2)–Fe(1)–OW(1)$^{\#2}$	90.01(7)		
Octahedron around Co (**3**)			
Co(1)–OW(1)	2.1105(2)	OW(3)–Co(1)–OW(1)	92.59(8)
Co(1)–OW(2)	2.0787(2)	OW(3)–Co(1)–OW(1)$^{\#1}$	87.41(8)
Co(1)–OW(3)	2.0963(2)	OW(3)–Co(1)–OW(2)	86.00(8)
OW(2)–Co(1)–OW(1)	87.14(7)	OW(3)–Co(1)–OW(2)$^{\#1}$	94.00(8)
OW(2)–Co(1)–OW(1)$^{\#1}$	92.86(7)		
Octahedron around Ni (**4**)			
Ni(1)–OW(1)	2.064(2)	OW(3)–Ni(1)–OW(1)	87.67(9)
Ni(1)–OW(2)	2.065(2)	OW(3)–Ni(1)–OW(1)$^{\#2}$	92.33(9)
Ni(1)–OW(3)	2.056(2)	OW(3)–Ni(1)–OW(2)	89.11(10)
OW(2)–Ni(1)–OW(1)	85.98(9)	OW(3)–Ni(1)–OW(2)$^{\#2}$	90.89(10)
OW(2)–Ni(1)–OW(1)$^{\#2}$	94.02(9)		

[a] Symmetry transformations used to generate equivalent atoms: #1 –x, –y, –z ; #2 –x, –y+1, –z.

Table 3. Selected Bond Distances (Å) and Angles (deg) for [SO_4] tetrahedron in **1**–**4**

	Mn (**1**)	Fe (**2**)	Co (**3**)	Ni (**4**)
S(1)–O(1)	1.4736(2)	1.4850(2)	1.479(2)	1.471(2)
S(1)–O(2)	1.459(2)	1.4652(2)	1.452(2)	1.490(2)
S(1)–O(3)	1.449(2)	1.4689(2)	1.466(2)	1.464(2)
S(1)–O(4)	1.464(2)	1.4696(2)	1.464(3)	1.468(2)
O(3)–S(1)–O(2)	108.72(2)	109.15(10)	109.10(15)	110.97(15)
O(3)–S(1)–O(4)	109.22(2)	110.23(2)	108.95(19)	109.15(16)
O(2)–S(1)–O(4)	108.74(2)	109.15(2)	108.82(18)	108.10(14)
O(3)–S(1)–O(1)	112.43(1)	108.95(9)	109.26(13)	109.39(14)
O(2)–S(1)–O(1)	109.43(1)	110.97(2)	112.15(13)	108.91(13)
O(4)–S(1)–O(1)	109.43(1)	108.39(10)	108.50(14)	110.32(16)