Supplementary Material (ESI) for CrystEngComm

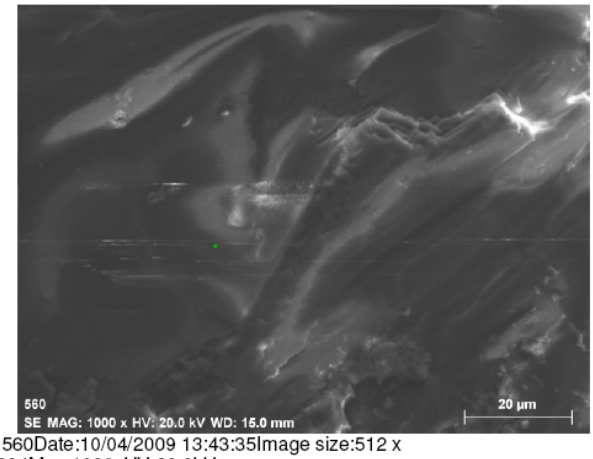
Supporting Information

Preparation of one dimensional Group 14 metal sulfides: Different roles of metal-amino complexes

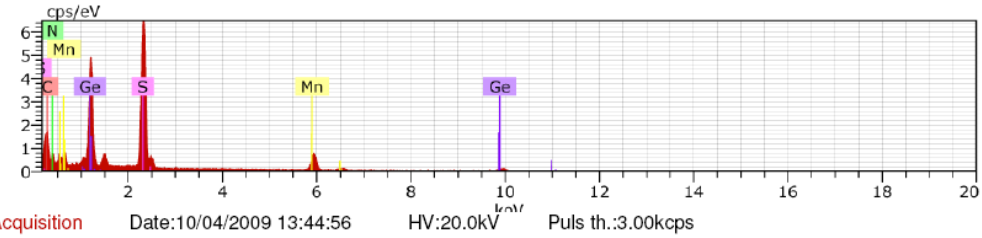
Zhenqing Wang,[a,b] Gongfeng Xu,[a] Yanfeng Bi,[a,b] Cheng Wang*[a]

[a] *State Key Laboratory of Rare Earth Resource Utilization, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, 5625 Renmin Street, Changchun, Jilin 130022, P.R. China. Fax:(+86)431-85698041; Tel:(+86)431-85262770; E-mail: cwang@ciac.jl.cn*

[b] *Graduate School of the Chinese Academy of Sciences, Beijing, 100039, P. R. China.*



560Date:10/04/2009 13:43:35Image size:512 x 384Mag:1000xHV:20.0kV



Acquisition Date:10/04/2009 13:44:56 HV:20.0kV Puls th.:3.00kcps

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [%]
C	6	K-series	17.58	17.79	26.52	3.1
N	7	K-series	45.28	45.82	58.59	9.5
S	16	K-series	16.51	16.71	9.33	0.6
Mn	25	K-series	8.72	8.82	2.88	0.3
Ge	32	K-series	10.74	10.87	2.68	0.6
		Total:	98.83	100.00	100.00	

Spectrum: Acquisition

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [%]
C	6	K-series	17.58	17.79	26.52	3.1
N	7	K-series	45.28	45.82	58.59	9.5
S	16	K-series	16.51	16.71	9.33	0.6
Mn	25	K-series	8.72	8.82	2.88	0.3
Ge	32	K-series	10.74	10.87	2.68	0.6

Fig.S1 The energy dispersive X-ray analysis (EDS) of compound **1**.

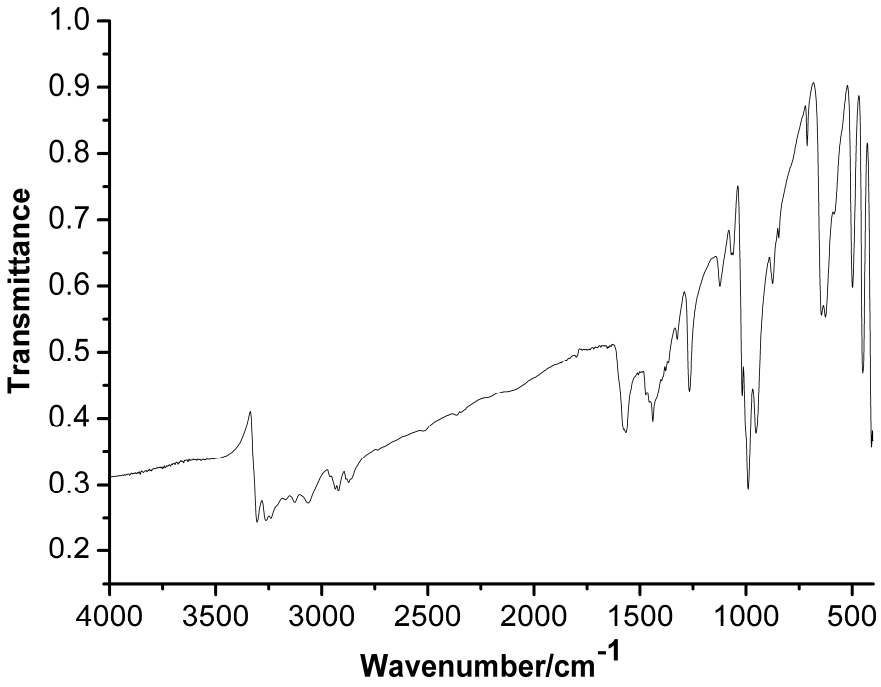
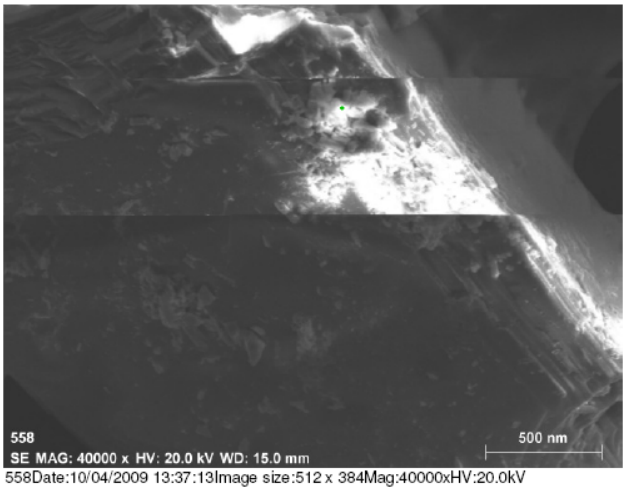
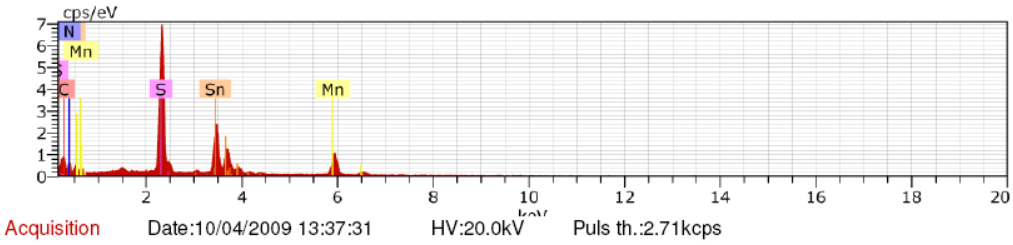


Fig.S2 The IR spectrum of compound **1** (400-4000 cm^{-1}).





El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [%]
C	6	K-series	13.84	15.14	43.15	3.2
N	7	K-series	3.15	3.45	8.43	5.8
S	16	K-series	22.97	25.14	26.84	0.9
Mn	25	K-series	14.60	15.97	9.95	0.5
Sn	50	L-series	36.83	40.30	11.62	1.2
		Total:	91.39	100.00	100.00	

Spectrum: Acquisition

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [%]
C	6	K-series	13.84	15.14	43.15	3.2
N	7	K-series	3.15	3.45	8.43	5.8
S	16	K-series	22.97	25.14	26.84	0.9
Mn	25	K-series	14.60	15.97	9.95	0.5
Sn	50	L-series	36.83	40.30	11.62	1.2

Fig.S3 The energy dispersive X-ray analysis (EDS) of compound **2**.

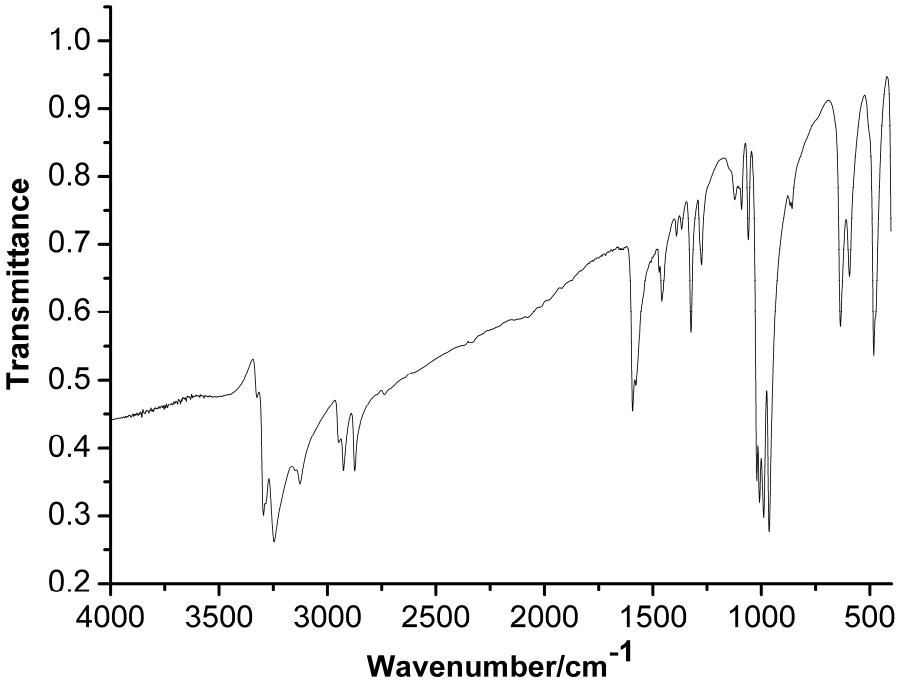


Fig.S4 The IR spectrum of compound **2** (400-4000 cm^{-1}).

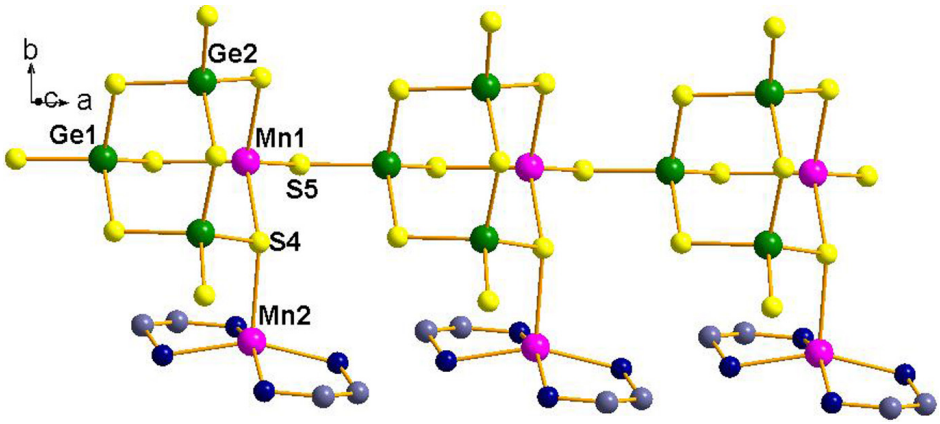


Fig. S5 Another possible structure of compound **1**. Green, Ge; pink, Mn; yellow, S; red, O; dark blue, N; blue grey, C. The disorders of Mn2 and C2 in $[Mn(en)_2]^{2+}$ are not shown for clarity. Hydrogen atoms are omitted for clarity. Symmetry transformations used to generate equivalent atoms: # 1 x-1, y, z; # 2 x, -y+3/2, z; # 3 -x+1, -y+1, -z; # 4 x+1, y, z.

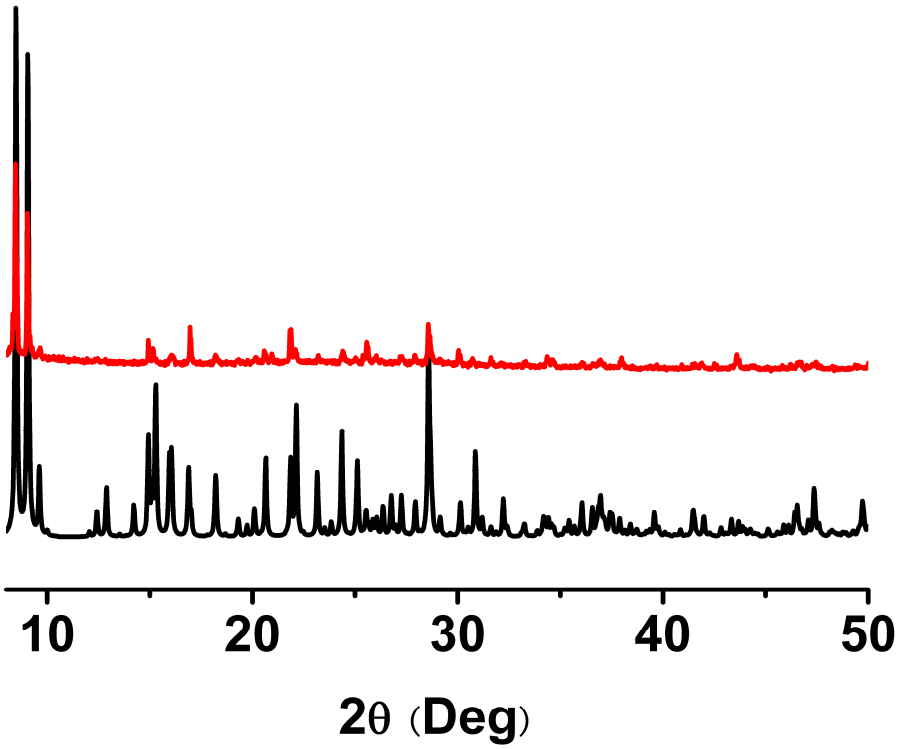


Fig. S6 XRD patterns for **1**: (top) taken at room temperature; (bottom) calculated on the basis of the structure determined by single-crystal X-ray diffraction.

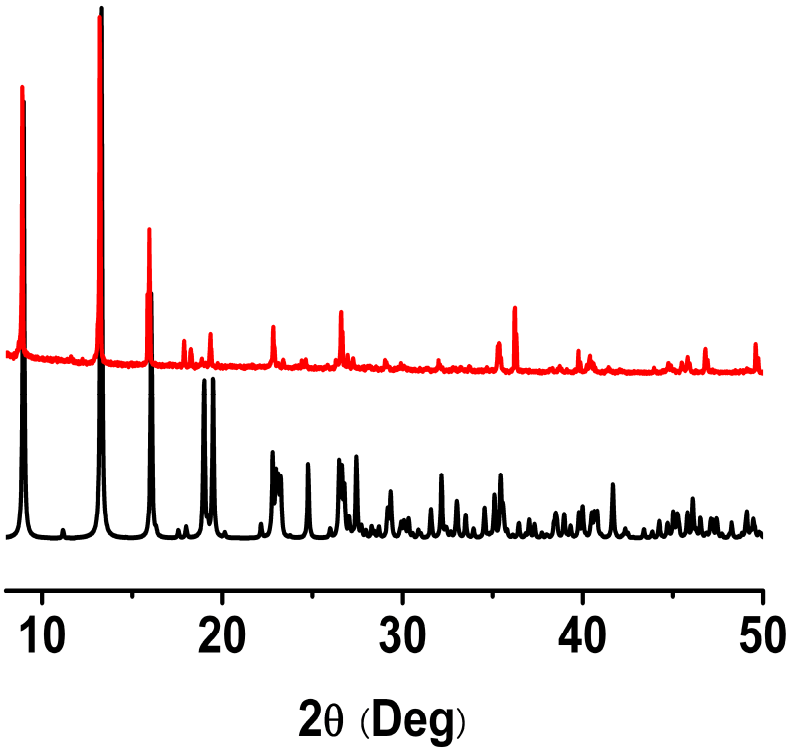


Fig. S7 XRD patterns for **2**: (top) taken at room temperature; (bottom) calculated on the basis of

the structure determined by single-crystal X-ray diffraction.

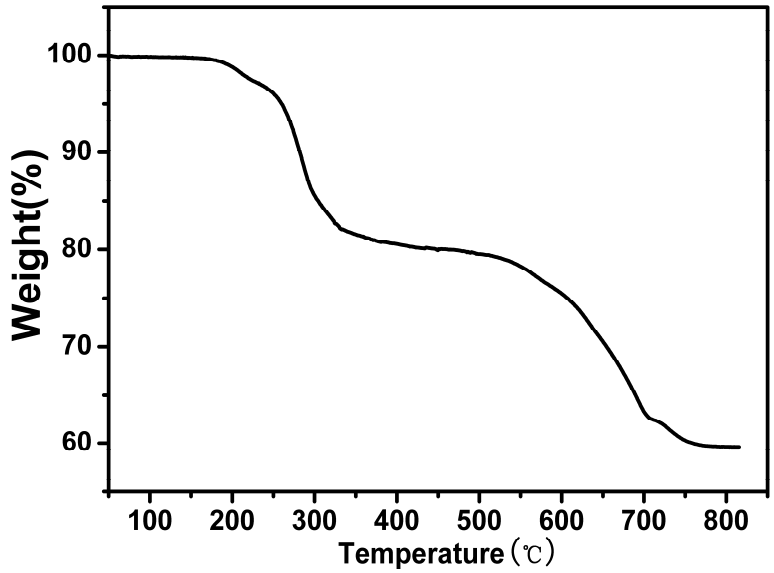


Fig. S8 TG curve for compound **1**.

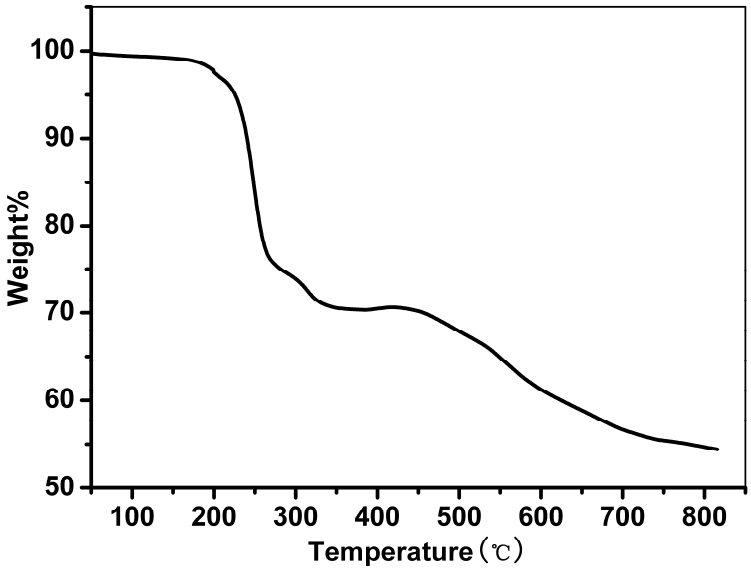


Fig. S9 TG curve for compound **2**.

Table S1. Selected bond lengths (Å) and angles (°) in compounds **1**[a,b] and **2**[c].

compound **1**			
Mn(1)-S(3)	2.447(1)	Mn(1)-S(4)	2.454(1)
Mn(1)-S(4)#2	2.454 (1)	Mn(1)-S(5)#1	2.446(1)
Mn(2)-N(1)	2.192(4)	Mn(2)-N(2)	2.180(2)
Mn(2)-N(1)#3	2.272(4)	Mn(2)-N(2)#3	2.218(2)
Mn(2)-S(4)	2.537(2)	Mn(3)-N(3)	2.258(2)
Mn(3)-N(4)	2.211(2)	Mn(3)-N(3)#2	2.258(2)
Mn(3)-N(4)#2	2.211(2)	Mn(3)-O(1)	2.185(5)
Ge(1)-S(2)	2.245(1)	Ge(1)-S(3)	2.186(1)
Ge(1)-S(5)	2.198(1)	Ge(1)-S(2)#2	2.245(1)
Ge(2)-S(1)	2.247(1)	Ge(2)-S(2)	2.265(1)
Ge(2)-S(4)	2.201(1)	Ge(2)-S(6)	2.131(1)
S(3)-Mn(1)-S(4)	109.35(3)	S(3)-Mn(1)-S(4)#2	109.35(3)
S(3)-Mn(1)-S(5)#1	101.64(5)	S(4)-Mn(1)-S(4)#2	111.05(5)
S(4)-Mn(1)-S(5)#1	112.50(3)	S(4)#2-Mn(1)-S(5)#1	112.50(3)
N(1)-Mn(2)-N(1)#3	157.07(7)	N(2)-Mn(2)-N(2)#3	156.65(6)
S(4)-Mn(2)-S(4)#3	179.66(5)	N(1)-Mn(2)-N(2)	97.10(12)
N(1)-Mn(2)-N(2)#3	80.41(12)	N(2)-Mn(2)-N(1)#3	79.47(12)
N(1)#3-Mn(2)-N(2)#3	93.76(12)	O(1)-Mn(3)-N(3)	97.80(9)
O(1)-Mn(3)-N(4)	100.19(9)	O(1)-Mn(3)-N(3)#2	97.80(9)
O(1)-Mn(3)-N(4)#2	100.19(9)	N(3)-Mn(3)-N(4)	79.30(6)
N(3)-Mn(3)-N(3)#2	99.57(10)	N(4)-Mn(3)-N(4)#2	96.18(10)
N(3)#2-Mn(3)-N(4)#2	79.30(6)	S(2)-Ge(1)-S(3)	113.77(3)
S(2)-Ge(1)-S(5)	105.07(3)	S(3)-Ge(1)-S(5)	110.86(5)
S(3)-Ge(1)-S(2)#2	113.77(3)	S(5)-Ge(1)-S(2)#2	105.07(3)
S(1)-Ge(2)-S(2)	108.28(4)	S(1)-Ge(2)-S(4)	110.73(4)
S(1)-Ge(2)-S(6)	104.21(4)	S(2)-Ge(2)-S(4)	111.04(4)
S(2)-Ge(2)-S(6)	107.35(4)	S(4)-Ge(2)-S(6)	114.82(4)
S(2)-Ge(1)-S(2)#2	107.58(5)		

compound **2**			
Mn(1)-N(1)	2.256(9)	Mn(1)-N(2)	2.295(9)
Mn(1)-N(3)	2.282(10)	Mn(1)-N(4)	2.269(10)
Mn(1)-S(2)	2.578(3)	Mn(1)-S(2)#2	2.589(3)
Sn(1)-S(1)	2.317(3)	Sn(1)-S(2)	2.356(2)
Sn(1)-S(3)	2.446(3)	Sn(1)-S(3)#1	2.454(3)
N(1)-Mn(1)-N(4)	165.80(32)	N(2)-Mn(1)-S(2)	174.47(25)
N(3)-Mn(1)-S(2)#2	174.85(26)	N(2)-Mn(1)-N(3)	92.26(36)
N(2)-Mn(1)-S(2)#2	87.97(23)	N(3)-Mn(1)-S(2)	93.17(30)
S(2)-Mn(1)-S(2)#2	86.53(9)	S(1)-Sn(1)-S(2)	115.12(10)
S(1)-Sn(1)-S(3)	112.65(10)	S(1)-Sn(1)-S(3)#1	115.97(10)
S(2)-Sn(1)-S(3)	112.77(9)	S(2)-Sn(1)-S(3)#1	104.54(9)
S(3)-Sn(1)-S(3)#1	93.70(8)		

[a] Estimated standard deviations are given in parentheses. [b]Symmetry transformations used to generate equivalent atoms: #1 x-1, y, z; #2 x, -y+3/2, z; #3 -x+1, -y+1, -z; #4 x+1, y, z. [c]Symmetry transformations used to generate equivalent atoms: #1 -x+1, -y+2, -z; #2 -x+1, -y+2, -z+1.

Table S2. Selected hydrogen bond distances (Å) and angles (^{o}) in compounds **1** and **2**.

compound **1**				
D-H	A	d(H···A)	d(D···A)	<(DHA)
O1— H1E	S4	2.79	3.510(4)	143
O1— H1F	S1	2.75	3.591(5)	165
N4—H4C	S6	2.72	3.549(2)	153
N4—H4D	S3	2.69	3.555(2)	162
compound **2**				
N(1) —H(1A)	S(3)	2.76	3.5756	151
N(2) —H(2A)	S(1)	2.60	3.4927	170
N(3) —H(3A)	S(1)	2.74	3.6225	166

Supplementary Material (ESI) for CrystEngComm