Supplementary Material (ESI) for Chemical Communications

Supporting Information

Nanoporosity of an interpenetrated NbO-type molecular framework studied by single crystal X-ray diffraction

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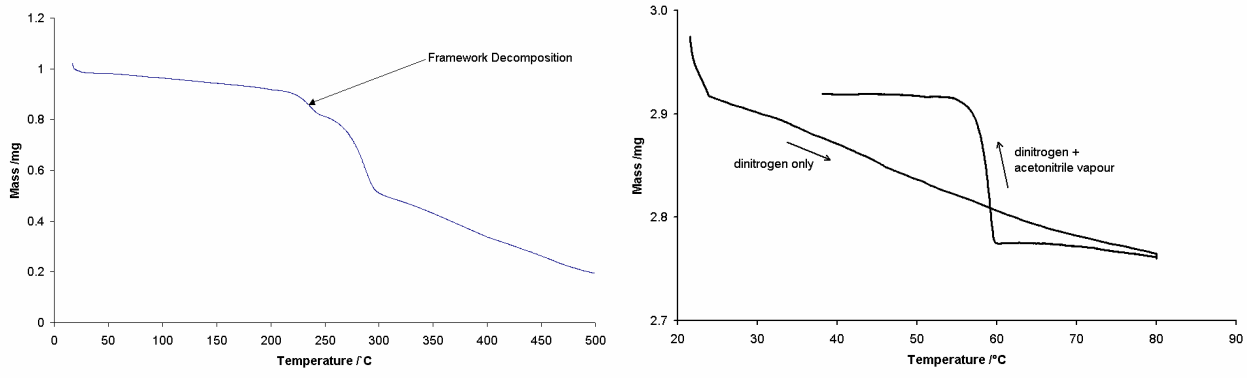
Thermogravimetry

Thermogravimetric analyses (TGA) were performed using a TA Instruments Hi-Res TGA2850 Thermogravimetric Analyser. Decomposition analysis was performed by heating at 1°C/min to 500°C in a nitrogen gas atmosphere, primarily to determine the temperature range of solvent loss. Desolvation-resolvation studies were performed by heating slowly under nitrogen until guest loss occurred, then cooling in an atmosphere containing the solvent vapour (achieved by bubbling nitrogen through the solvent). Measurements were analysed using Universal Analysis 2000 for Windows. Desorption/decomposition and desorption/sorption data are shown in Fig. S1.



(a) (b)

Fig. S1 (a) Thermogravimetry of **A·⅔MeCN** to high temperature under a stream of dry dinitrogen, showing loss of unbound MeCN in the temperature range 30 - 200 °C (complete removal of MeCN occurs at 80 °C under dry dinitrogen although the kinetics of guest loss are very slow at this temperature) and framework decomposition above 200 °C, and (b) reversible desorption and sorption of MeCN by heating in dry dinitrogen followed by cooling under a mixiture of MeCN vapour and dinitrogen. The weight loss below 25 °C in each case corresponds to the loss of surface solvent.

Single Crystal X-ray Diffraction

Single crystal X-ray diffraction (SCXRD) data for structure determination were collected on a Bruker-AXS SMART 1000 CCD diffractometer equipped with an Oxford Cryosystems Cryostream nitrogen gas stream. Single crystals were found by inspection under a polarising microscope and were attached with a film of paratone N oil to a mohair fibre mounted on a copper pin. The pin was then mounted on the goniometer and the crystal quench cooled to 150 K under the nitrogen gas stream. Diffraction patterns were generated using an incident beam of graphite monochromated Mo Kα radiation. For each crystal, 2000 frames of intensity data (exposure time between 20 s and 40 s per frame) were collected over a range of incident angles covering the entire sphere. Unit cells were determined by means of 3 × 15 frames of intensity data with the exposure times between 10 s and 20 s per frame. For twinned crystals, unit cells of the twin components were determined using GEMINI. Data integration and reduction was undertaken using SAINT and XPREP, or SAINT, GEMINI and XPREP for some twinned crystals. The structures were solved by direct methods and refined using SHELXL-97 and difference Fourier synthesis. An ORTEP diagram of **A·⅔MeCN** at 150 K is given in Fig. S2.

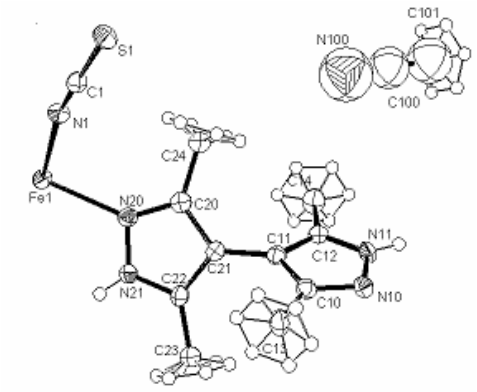


Fig. S2 ORTEP diagram of the asymmetric unit of **A·⅔MeCN** at 150 K showing 50% thermal ellipsoids.

For *in*-situ guest desorption investigations the desorbed framework structure was determined by SCXRD on single crystals mounted with a thin smear of grease within open-ended capillary tubes such that the open end of the capillary was immersed in the nitrogen cryostream. Crystals were desolvated *in situ* by heating to 375 K, during which time unit cell determinations were performed (see Fig. S3). Three full structures were determined at this temperature (**A·0.40MeCN**, **A·0.33MeCN** and **A·0.27MeCN**) before cooling to 150 K to obtain a data set on **A·0.27MeCN**. A single crystal of **A** was obtained by *ex-situ* desolvation by heating in an oven at 400 K in air before being attached within a capillary; the capillary was then sealed and SCXRD intensity data collected at 375 K. A summary of crystallographic collection and refinement data for the three 150 K structures is given in Table S1, and full crystallographic details for these structures are given at the end of this document. Fourier electron difference maps for the three 150 K structures are given in Figure S4, clearly showing the difference in cavity electron density in each of the three structures. A framework diagram showing the presence of the 1-D channels is given in Figure S5. Calculations leading to a comparison of the shifts in relative atomic positions within **A·⅔MeCN** and **A** at 150 K are given in Table S2. Crystallographic information files (cifs) for all six structures (at 150 K: **A·⅔MeCN**, **A·0.27MeCN** and **A**, and at 375 K: **A·0.40MeCN**, **A·0.33MeCN** and **A·0.27MeCN**) have been deposited with the Cambridge Crystallographic Database.

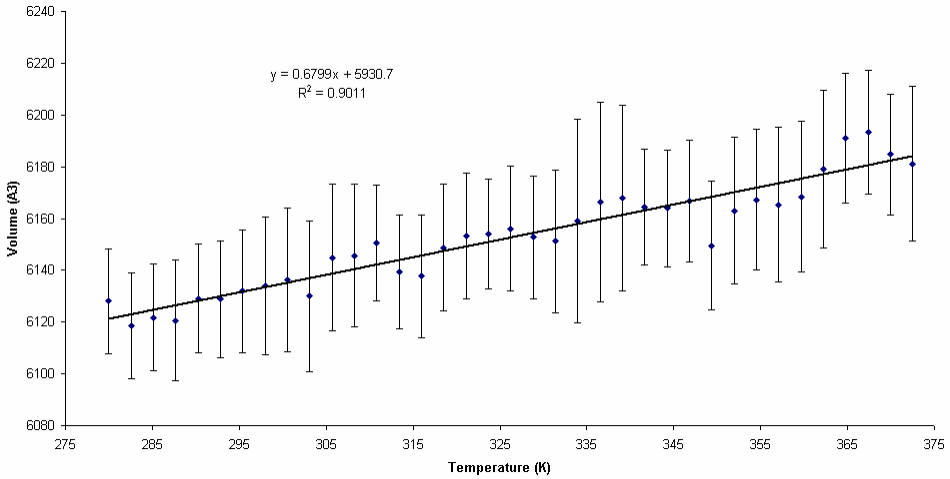


Fig. S3 Temperature dependence of the unit cell volume of **A·x MeCN** ($0 \le x \le ⅔$) as obtained during heating of a fully solvated crystal of **A·⅔MeCN** from 280 to 375 K at 20 K h^{-1} under dry dinitrogen in an open-ended glass capillary.

Table S1 Summary of structural details for **A·⅔MeCN**, **A·0.27MeCN** and **A** at 150 K.

Compound	**A·⅔MeCN**	**A·0.27MeCN**	**A**
Formula	$Fe(tmbpz)_2(NCS)_2$. $⅔CH_3CN$	$Fe(tmbpz)_2(NCS)_2$. $0.27CH_3CN$	$Fe(tmbpz)_2(NCS)_2$
FW/gmo1^{-1}	579.88	563.69	552.51
T/K	150(2)	150(2)	150(2)
Crystal System	Trigonal	Trigonal	Trigonal
Space Group	$R\bar{3}$ (no. 148)	$R\bar{3}$ (no. 148)	$R\bar{3}$ (no. 148)
a/Å	25.283(17)	25.3021(14)	25.2831(14)
c/Å	10.962(11)	10.9672(13)	10.9463(13)
V/Å^3	6068(8)	6080.5(9)	6059.8(9)
Z	9	9	9
ρ_{calc}/Mgm^{-3}	1.428	1.385	1.363
μ/mm^{-1}	0.749	0.745	0.746
Data/restraints/ parameters	3193/0/171	3110/0/168	3241/0/161
R(*F*)/% ($I>2\sigma(I)$, all data)	0.0575, 0.1016	0.0371, 0.0701	0.0423, 0.0824
tmbpz interplanar angle/°	54.7(6)	54.5(4)	54.4(4)
Fe(1)–N(1)–C(1); N(1)–C(1)–S(1)/°	166.1(3); 178.7(4)	165.5(2) 178.6(2)	165.4(2) 178.6(3)

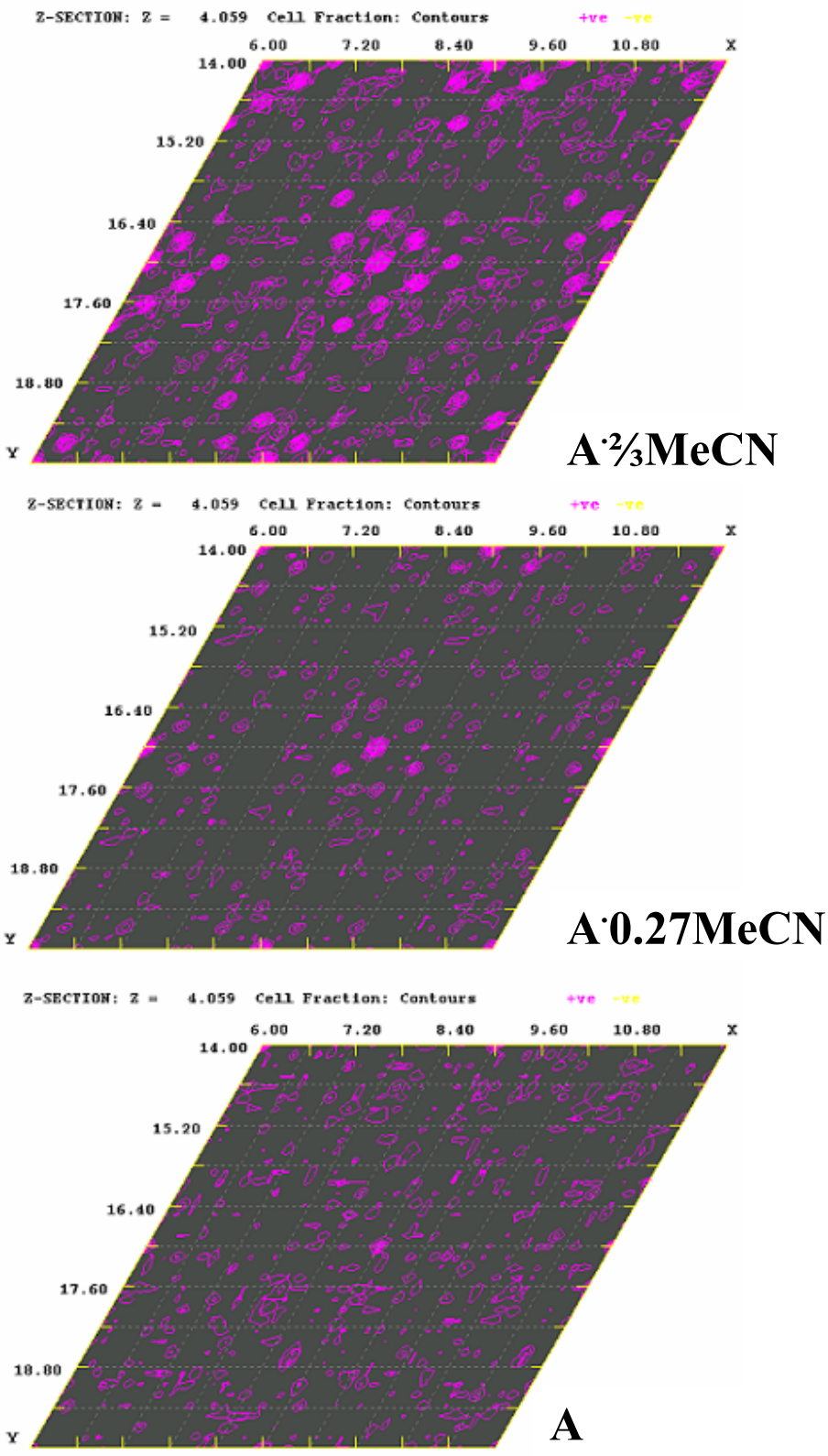


Fig. S4 Positive electron density difference maps for **A·⅔MeCN**, **A·0.27MeCN** and **A**, viewed down (001) with pore located in the centre of the diagram (x and y axes in Å; contour interval 0.2 eA^{-3}).

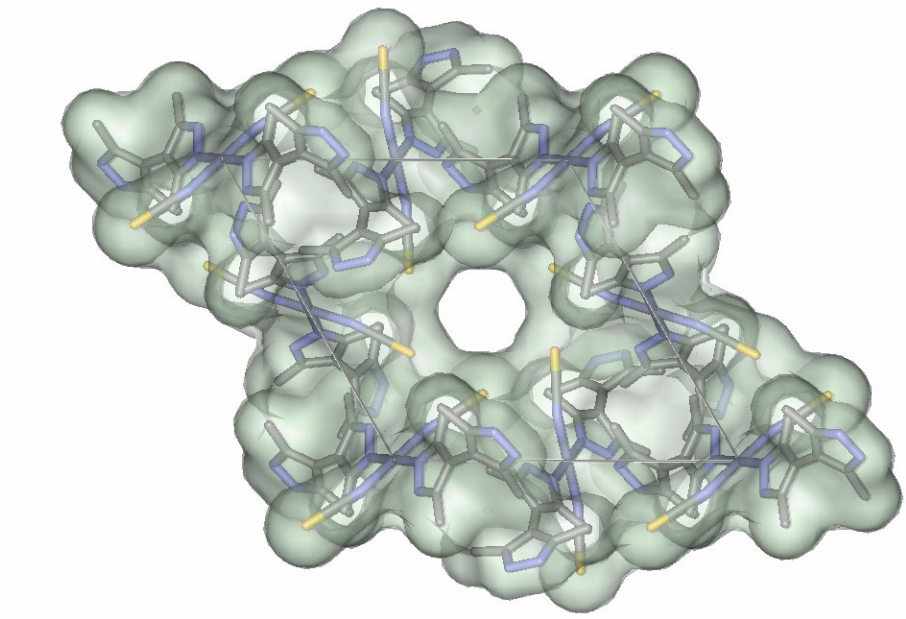


Fig. S5 The porous framework structure of **A** viewed down the *c* axis; addition of a vdW surface highlights the presence of empty 1-D channels within the structure. Hydrogen atoms are omitted for clarity, although the framework vdW surface has been generated with consideration of these.

Table S2 Atomic shift calculations, comparing the atomic positions in **A·⅔MeCN** and **A** at 150 K.

POSITION	A·⅔MeCN				A											
	x	y	z		x	y	z	Del(X + Yc	DelYsin12(	DelZ		a	b	c	pythag displacement	
Fe1	0.5	0.5	0.5		0.5	0.5	0.5	0	0	0		25.283	25.283	10.96	0	
S1	0.330218	0.526167	0.624328		0.329839	0.524733	0.623106	0.000338	0.001242	0.001222		25.283	25.283	10.96	0.035189	
C1	0.391657	0.522601	0.595661		0.391576	0.521469	0.595129	0.000485	0.00098	0.000532		25.283	25.283	10.96	0.028261	
N1	0.435285	0.519912	0.572889		0.435737	0.519609	0.572669	0.000604	0.000262	0.00022		25.283	25.283	10.96	0.016812	
N10	0.54818	0.527803	0.680923		0.54824	0.527482	0.681894	0.00022	0.000278	0.000971		25.283	25.283	10.96	0.013919	
N11	0.548998	0.576799	0.732956		0.548989	0.576664	0.734282	5.85E-05	0.000117	0.001326		25.283	25.283	10.96	0.014904	
C10	0.584738	0.51637	0.752512		0.584723	0.516234	0.753106	5.3E-05	0.000118	0.000594		25.283	25.283	10.96	0.007283	
C11	0.60904	0.559304	0.849704		0.609	0.558749	0.850499	0.000237	0.000481	0.000795		25.283	25.283	10.96	0.016114	
C12	0.583871	0.596328	0.834327		0.584084	0.596177	0.835051	0.000289	0.000131	0.000724		25.283	25.283	10.96	0.011274	
C13	0.596568	0.464846	0.729494		0.596333	0.464686	0.728867	0.000155	0.000139	0.000627		25.283	25.283	10.96	0.008652	
C14	0.590064	0.648324	0.907333		0.590128	0.648119	0.908262	0.000166	0.000178	0.000929		25.283	25.283	10.96	0.011897	
N20	0.734892	0.610251	1.075014		0.734727	0.610219	1.075684	0.000149	2.77E-05	0.00067		25.283	25.283	10.96	0.008283	
N21	0.694068	0.55167	1.110366		0.694501	0.552	1.110483	0.000268	0.000286	0.000117		25.283	25.283	10.96	0.009988	
C20	0.645068	0.524396	1.036796		0.645039	0.52407	1.0376	0.000134	0.000282	0.000804		25.283	25.283	10.96	0.011835	
C21	0.652543	0.56556	0.946558		0.652702	0.565577	0.947227	0.00015	1.47E-05	0.000669		25.283	25.283	10.96	0.008269	
C22	0.70952	0.618642	0.974989		0.70931	0.618363	0.974831	7.05E-05	0.000242	0.000158		25.283	25.283	10.96	0.006595	
C23	0.59338	0.461153	1.058829		0.59409	0.461026	1.060692	0.000773	0.00011	0.001863		25.283	25.283	10.96	0.02841	
C24	0.741098	0.676817	0.904336		0.74081	0.67667	0.904829	0.000215	0.000127	0.000493		25.283	25.283	10.96	0.008305	
															0.013666	
ESD	A·⅔MeCN				A											
	x	y	z		x	y	z	d(x + ycos	dysin120	dz		sum errors	esd (sqrt)		**shift/esd**	
Fe1	0	0	0		0	0	0	0	0	0		0	0		**0**	
S1	0.00004	0.00005	0.00009		0.00003	0.00003	0.00006	0.002001	0.001277	0.001186		0.000146134	0.002076417		**16.94699**	
C1	0.00017	0.00017	0.0003		0.00013	0.00012	0.0002	0.008041	0.004556	0.003952		0.000469152	0.008300234		**3.404886**	
N1	0.00015	0.00015	0.0003		0.00011	0.0001	0.00019	0.006982	0.003947	0.003892		0.000284208	0.008452515		**1.988998**	
N10	0.00014	0.00014	0.0003		0.0001	0.0001	0.0002	0.006525	0.003767	0.003952		0.000209813	0.007536991		**1.846743**	
N11	0.00014	0.00014	0.0003		0.0001	0.0001	0.00019	0.006525	0.003767	0.003892		0.000154695	0.005189673		**2.871875**	
C10	0.00018	0.00017	0.0003		0.00012	0.00012	0.0002	0.0081	0.004556	0.003952		0.000100296	0.006885363		**1.057792**	
C11	0.00017	0.00017	0.0003		0.00012	0.00012	0.0002	0.007892	0.004556	0.003952		0.000274372	0.008513639		**1.89269**	
C12	0.00017	0.00018	0.0003		0.00013	0.00013	0.0002	0.008218	0.004862	0.003952		0.000214743	0.009523898		**1.183748**	
C13	0.0002	0.00019	0.0004		0.00014	0.00013	0.0003	0.009083	0.005041	0.00548		0.000181823	0.010507759		**0.823374**	
C14	0.0002	0.0002	0.0004		0.00015	0.00014	0.0003	0.009407	0.005345	0.00548		0.000238779	0.010035282		**1.185517**	
N20	0.00014	0.00014	0.0003		0.0001	0.0001	0.00019	0.006525	0.003767	0.003892		0.000111598	0.006736694		**1.229508**	
N21	0.00014	0.00014	0.0003		0.0001	0.0001	0.0002	0.006525	0.003767	0.003952		0.000152995	0.007658742		**1.304166**	
C20	0.00018	0.00017	0.0003		0.00013	0.00013	0.0002	0.008319	0.004686	0.003952		0.000192908	0.008149599		**1.452273**	
C21	0.00017	0.00017	0.0003		0.00012	0.00012	0.0002	0.007892	0.004556	0.003952		0.000121398	0.007340384		**1.12653**	
C22	0.00017	0.00017	0.0003		0.00012	0.00013	0.0002	0.007966	0.004686	0.003952		9.9337E-05	0.00753119		**0.875697**	
C23	0.0002	0.00019	0.0004		0.00014	0.00014	0.0003	0.009156	0.005168	0.00548		0.000610638	0.010747069		**2.643465**	
C24	0.00019	0.00019	0.0004		0.00014	0.00014	0.0003	0.008951	0.005168	0.00548		0.000189566	0.011413323		**0.727624**	
														AVERAGE	2.50364	
															1.506758	neglecting S1

Magnetic Susceptibility

Magnetic susceptibility data were collected at Monash University, Melbourne, with the assistance of Professor Keith S. Murray and Dr Boujemaa Moubaraki using a Quantum Design MPMS SQUID magnetometer with an applied field of 1 T. Samples were contained in a gelatine capsule held in a plastic straw. Figure S6 shows that the iron(II) centres in **A·⅔MeCN** remain high-spin from 5 – 300 K.

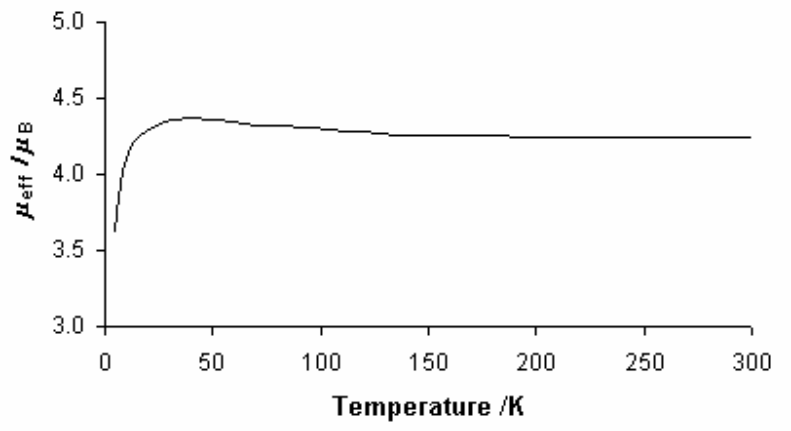


Fig. S6 Temperature dependent magnetic susceptibility of **A·⅔MeCN**.

Full X-ray Crystallographic Tables

Table 1. Crystal data and structure refinement for A·⅔MeCN at 150 K.

Identification code	A·⅔MeCN
Empirical formula	C23.33 H30 Fe N10.67 S2
Formula weight	579.88
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system, space group	Hexagonal, R-3
Unit cell dimensions	a = 25.283(17) Å α = 90 ° b = 25.283(17) Å β = 90 ° c = 10.962(11) Å γ = 120 °
Volume	6068(8) $Å^3$
Z, Calculated density	9, 1.428 Mg/m^3
Absorption coefficient	0.749 mm^{-1}
F(000)	2724
Crystal size	(twinned)
Theta range for data collection	1.61 to 28.06 °
Limiting indices	-33<=h<=33, -33<=k<=33, -14<=l<=14
Reflections collected / unique	18070 / 3193 [R(int) = 0.0771]
Completeness to theta = 28.06	97.1 %
Absorption correction	Empirical
Max. and min. transmission	1.000000 and 0.265124
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3193 / 0 / 171
Goodness-of-fit on F^2	1.087
Final R indices [I>2sigma(I)]	R1 = 0.0575, wR2 = 0.1324
R indices (all data)	R1 = 0.1016, wR2 = 0.1673
Largest diff. peak and hole	0.660 and -0.900 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4), equivalent isotropic displacement parameters ($Å^2$ x 10^4) and site occupation parameters for A·⅔MeCN at 150 K. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)	Occ
Fe(1)	0	5000	5000	212(2)	1000
S(1)	1960(1)	5262(1)	6243(1)	264(3)	1000
N(1)	846(2)	5199(2)	5729(3)	231(7)	1000
C(1)	1309(2)	5226(2)	5957(3)	214(8)	1000
N(10)	1246(1)	3898(1)	-750(3)	224(7)	1000
N(11)	1424(1)	4483(1)	-1104(3)	228(7)	1000
C(10)	1207(2)	4756(2)	-368(3)	226(8)	1000
C(11)	870(2)	4344(2)	534(3)	212(8)	1000
C(12)	909(2)	3814(2)	250(3)	229(8)	1000
C(14)	643(2)	3232(2)	957(4)	294(9)	1000
C(13)	1322(2)	5389(2)	-588(4)	338(10)	1000
N(20)	204(1)	4722(1)	3191(3)	224(7)	1000
N(21)	-278(1)	4232(1)	2670(3)	230(7)	1000
C(20)	684(2)	4836(2)	2475(3)	235(8)	1000
C(21)	497(2)	4407(2)	1503(3)	216(8)	1000
C(22)	-125(2)	4037(2)	1657(3)	223(8)	1000
C(23)	1317(2)	5352(2)	2705(4)	298(9)	1000
C(24)	-583(2)	3517(2)	927(4)	331(10)	1000
C(101)	3333	6667	-1259(18)	1320(60)	1000
C(100)	3063(13)	6324(12)	-210(30)	1040(80)	330
N(100)	2776(16)	6082(16)	710(30)	1720(120)	330

Table 3. Bond lengths [Å] and angles [°] for A·⅔MeCN at 150 K.

Fe(1)-N(1)#1	2.096(3)
Fe(1)-N(1)	2.096(3)
Fe(1)-N(20)#1	2.248(3)
Fe(1)-N(20)	2.248(3)
Fe(1)-N(10)#2	2.385(3)
Fe(1)-N(10)#3	2.385(3)
S(1)-C(1)	1.631(4)
N(1)-C(1)	1.166(5)
N(10)-C(12)	1.340(5)
N(10)-N(11)	1.371(4)
N(10)-Fe(1)#4	2.385(3)
N(11)-C(10)	1.344(5)
C(10)-C(11)	1.379(5)
C(10)-C(13)	1.494(6)
C(11)-C(12)	1.428(5)
C(11)-C(21)	1.479(5)
C(12)-C(14)	1.492(5)
N(20)-C(20)	1.349(5)
N(20)-N(21)	1.355(4)
N(21)-C(22)	1.349(5)
C(20)-C(21)	1.423(5)
C(20)-C(23)	1.497(6)
C(21)-C(22)	1.380(5)
C(22)-C(24)	1.479(6)
C(101)-C(100)	1.40(3)
C(100)-N(100)	1.21(4)
C(100)-C(100)#5	1.37(4)
C(100)-C(100)#6	1.37(4)

N(1)#1-Fe(1)-N(1)	180.00(9)
N(1)#1-Fe(1)-N(20)#1	92.60(13)
N(1)-Fe(1)-N(20)#1	87.40(13)
N(1)#1-Fe(1)-N(20)	87.40(13)
N(1)-Fe(1)-N(20)	92.60(13)
N(20)#1-Fe(1)-N(20)	180.0
N(1)#1-Fe(1)-N(10)#2	84.49(12)
N(1)-Fe(1)-N(10)#2	95.51(12)
N(20)#1-Fe(1)-N(10)#2	81.01(12)
N(20)-Fe(1)-N(10)#2	98.99(12)
N(1)#1-Fe(1)-N(10)#3	95.51(12)
N(1)-Fe(1)-N(10)#3	84.49(12)
N(20)#1-Fe(1)-N(10)#3	98.99(12)
N(20)-Fe(1)-N(10)#3	81.01(12)
N(10)#2-Fe(1)-N(10)#3	180.00(12)
C(1)-N(1)-Fe(1)	166.1(3)
N(1)-C(1)-S(1)	178.7(4)
C(12)-N(10)-N(11)	103.9(3)
C(12)-N(10)-Fe(1)#4	133.6(2)
N(11)-N(10)-Fe(1)#4	121.6(2)
C(10)-N(11)-N(10)	112.8(3)
N(11)-C(10)-C(11)	107.5(3)
N(11)-C(10)-C(13)	122.7(3)
C(11)-C(10)-C(13)	129.8(4)
C(10)-C(11)-C(12)	104.2(3)
C(10)-C(11)-C(21)	128.3(4)

C(12)-C(11)-C(21)	127.1(3)
N(10)-C(12)-C(11)	111.6(3)
N(10)-C(12)-C(14)	121.2(3)
C(11)-C(12)-C(14)	127.2(3)
C(20)-N(20)-N(21)	104.6(3)
C(20)-N(20)-Fe(1)	140.2(3)
N(21)-N(20)-Fe(1)	114.6(2)
C(22)-N(21)-N(20)	112.9(3)
N(20)-C(20)-C(21)	110.7(3)
N(20)-C(20)-C(23)	123.0(3)
C(21)-C(20)-C(23)	126.3(4)
C(22)-C(21)-C(20)	104.9(3)
C(22)-C(21)-C(11)	125.8(3)
C(20)-C(21)-C(11)	129.3(3)
N(21)-C(22)-C(21)	106.9(3)
N(21)-C(22)-C(24)	122.0(3)
C(21)-C(22)-C(24)	131.1(3)
N(100)-C(100)-C(100)#5	123(2)
N(100)-C(100)-C(100)#6	111(3)
C(100)#5-C(100)-C(100)#6	60.000(3)
N(100)-C(100)-C(101)	169(4)
C(100)#5-C(100)-C(101)	60.6(9)
C(100)#6-C(100)-C(101)	60.6(9)

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

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^4) for A·⅔MeCN at 150 K. The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
Fe(1)	210(4)	234(4)	228(4)	-29(3)	-19(3)	138(3)
S(1)	214(5)	290(5)	327(5)	15(4)	4(4)	154(4)
N(1)	224(17)	220(17)	254(16)	2(13)	-17(13)	114(14)
C(1)	250(20)	181(18)	234(18)	-9(14)	8(15)	126(16)
N(10)	228(17)	220(17)	238(16)	42(13)	25(13)	122(14)
N(11)	206(16)	227(17)	245(16)	53(13)	71(13)	102(14)
C(10)	205(19)	222(19)	227(18)	-16(15)	-13(15)	89(16)
C(11)	204(19)	235(19)	202(18)	-13(14)	-19(14)	113(16)
C(12)	207(19)	230(20)	257(19)	-1(15)	-2(15)	110(16)
C(14)	350(20)	270(20)	280(20)	59(16)	126(17)	167(19)
C(13)	430(30)	260(20)	350(20)	63(17)	70(19)	190(20)
N(20)	226(17)	206(16)	248(16)	-25(13)	-7(13)	113(14)
N(21)	197(16)	226(17)	262(16)	-29(13)	27(13)	103(14)
C(20)	250(20)	220(20)	236(19)	13(15)	-16(15)	126(17)
C(21)	232(19)	220(19)	205(18)	-8(14)	-1(14)	118(16)
C(22)	250(20)	232(19)	217(18)	2(15)	29(15)	142(17)
C(23)	280(20)	270(20)	300(20)	-29(16)	6(17)	102(18)
C(24)	250(20)	320(20)	350(20)	-92(18)	31(17)	91(19)

Table 5. Hydrogen coordinates (x 10^3), isotropic displacement parameters ($Å^2$ x 10^3) and site occupation parameters for A·⅔MeCN at 150 K.

	x	y	z	U(eq)	Occ
H(11)	166	466	-174	27	1000
H(14A)	74	295	55	44	500
H(14B)	20	305	100	44	500
H(14C)	82	332	178	44	500
H(14D)	43	326	167	44	500
H(14E)	97	316	122	44	500
H(14F)	36	289	44	44	500
H(13A)	158	556	-131	51	500
H(13B)	153	564	12	51	500
H(13C)	93	538	-72	51	500
H(13D)	111	549	4	51	500
H(13E)	117	541	-140	51	500
H(13F)	176	568	-55	51	500
H(21)	-65	406	296	28	1000
H(23A)	132	558	343	45	500
H(23B)	146	563	200	45	500
H(23C)	159	519	283	45	500
H(23D)	159	535	208	45	500
H(23E)	146	530	351	45	500
H(23F)	132	574	268	45	500
H(24A)	-99	336	130	50	500
H(24B)	-48	320	90	50	500
H(24C)	-59	366	10	50	500
H(24D)	-38	345	23	50	500
H(24E)	-89	361	63	50	500
H(24F)	-78	315	144	50	500
H(10A)	302	656	-189	197	170
H(10B)	365	658	-156	197	170
H(10C)	352	710	-107	197	170
H(10D)	377	694	-112	197	170
H(10E)	315	691	-146	197	170
H(10F)	327	639	-194	197	170

Table 6. Hydrogen bonds for A·⅔MeCN at 150 K [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(21)-H(21)...N(1)#1	0.88	2.60	3.046(5)	112.8
N(21)-H(21)...S(1)#7	0.88	2.80	3.625(4)	156.8
N(11)-H(11)...S(1)#8	0.88	2.57	3.391(4)	156.0
N(11)-H(11)...N(1)#9	0.88	2.61	3.063(5)	113.3

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z+1 #2 -y+1/3,x-y+2/3,z+2/3 #3 y-1/3,-x+y+1/3,-z+1/3
#4 -x+y-1/3,-x+1/3,z-2/3 #5 -y+1,x-y+1,z #6 -x+y,-x+1,z
#7 -y+1/3,x-y+2/3,z-1/3 #8 x,y,z-1 #9 x-y+2/3,x+1/3,-z+1/3

Table 7. Crystal data and structure refinement for A·0.27MeCN at 150 K.

Identification code	A·0.27MeCN
Empirical formula	C22.54 H28.82 Fe N10.27 S2
Formula weight	563.69
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system, space group	Hexagonal, R-3
Unit cell dimensions	a = 25.3021(14) Å α = 90 ° b = 25.3021(14) Å β = 90 ° c = 10.9672(13) Å γ = 120 °
Volume	6080.5(9) $Å^3$
Z, Calculated density	9, 1.385 Mg/m^3
Absorption coefficient	0.745 mm^{-1}
F(000)	2646
Crystal size	0.27 x 0.12 x 0.11 mm
Theta range for data collection	2.08 to 27.96 °
Limiting indices	-32<=h<=32, -32<=k<=32, -14<=l<=14
Reflections collected / unique	19731 / 3110 [R(int) = 0.0422]
Completeness to theta = 27.96	95.4 %
Absorption correction	Empirical
Max. and min. transmission	1.0000 and 0.874582
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3110 / 0 / 168
Goodness-of-fit on F^2	1.076
Final R indices [I>2sigma(I)]	R1 = 0.0371, wR2 = 0.0733
R indices (all data)	R1 = 0.0701, wR2 = 0.0878
Largest diff. peak and hole	0.550 and -0.414 e.$Å^{-3}$

Table 8. Atomic coordinates (x 10^4), equivalent isotropic displacement parameters ($Å^2$ x 10^4) and site occupation parameters for A·0.27MeCN at 150 K. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)	Occ
Fe(1)	5000	5000	5000	145(1)	1000
S(1)	3296(1)	5245(1)	6236(1)	207(2)	1000
C(1)	3914(1)	5216(1)	5954(2)	158(5)	1000
N(1)	4355(1)	5196(1)	5728(2)	173(4)	1000
N(10)	5484(1)	5275(1)	6816(2)	167(4)	1000
N(11)	5491(1)	5765(1)	7335(2)	170(4)	1000
C(10)	5848(1)	5161(1)	7526(2)	155(5)	1000
C(11)	6091(1)	5589(1)	8500(2)	155(5)	1000
C(12)	5842(1)	5963(1)	8342(2)	167(5)	1000
C(13)	5966(1)	4648(1)	7285(2)	226(6)	1000
C(14)	5904(1)	6483(1)	9074(3)	267(6)	1000
C(20)	6453(1)	5240(1)	10367(2)	166(5)	1000
C(21)	6528(1)	5657(1)	9470(2)	149(5)	1000
C(22)	7094(1)	6185(1)	9745(2)	155(5)	1000
C(23)	5942(1)	4608(1)	10596(3)	260(6)	1000
C(24)	7408(1)	6767(1)	9045(2)	236(6)	1000
N(20)	7349(1)	6103(1)	10754(2)	151(4)	1000
N(21)	6946(1)	5520(1)	11104(2)	161(4)	1000
C(101)	3333	6667	-1830(20)	1000	410(12)
C(100)	3260(30)	6399(18)	-700(40)	1000	137(4)
N(100)	3190(30)	6210(20)	250(50)	1500	137(4)

Table 9. Bond lengths [Å] and angles [°] for A·0.27MeCN at 150 K.

Fe(1)-N(1)#1	2.087(2)
Fe(1)-N(1)	2.087(2)
Fe(1)-N(10)	2.258(2)
Fe(1)-N(10)#1	2.258(2)
Fe(1)-N(20)#2	2.386(2)
Fe(1)-N(20)#3	2.386(2)
S(1)-C(1)	1.633(3)
C(1)-N(1)	1.167(3)
N(10)-C(10)	1.344(3)
N(10)-N(11)	1.357(3)
N(11)-C(12)	1.347(3)
C(10)-C(11)	1.423(3)
C(10)-C(13)	1.494(3)
C(11)-C(12)	1.387(3)
C(11)-C(21)	1.481(3)
C(12)-C(14)	1.480(4)
C(20)-N(21)	1.352(3)
C(20)-C(21)	1.383(3)
C(20)-C(23)	1.492(4)
C(21)-C(22)	1.417(3)
C(22)-N(20)	1.347(3)
C(22)-C(24)	1.490(3)
N(20)-N(21)	1.364(3)
N(20)-Fe(1)#4	2.386(2)
C(101)-C(100)	1.39(4)
C(100)-C(100)#5	1.05(7)
C(100)-C(100)#6	1.05(6)
C(100)-N(100)	1.12(5)
C(100)-N(100)#5	1.75(6)
C(100)-N(100)#6	1.77(6)
N(100)-C(100)#6	1.75(6)
N(100)-N(100)#5	1.76(8)
N(100)-N(100)#6	1.76(8)
N(100)-C(100)#5	1.77(6)
N(1)#1-Fe(1)-N(1)	180.0
N(1)#1-Fe(1)-N(10)	92.46(8)
N(1)-Fe(1)-N(10)	87.54(8)
N(1)#1-Fe(1)-N(10)#1	87.54(8)
N(1)-Fe(1)-N(10)#1	92.46(8)
N(10)-Fe(1)-N(10)#1	180.0
N(1)#1-Fe(1)-N(20)#2	84.62(7)
N(1)-Fe(1)-N(20)#2	95.38(7)
N(10)-Fe(1)-N(20)#2	80.91(7)
N(10)#1-Fe(1)-N(20)#2	99.09(7)
N(1)#1-Fe(1)-N(20)#3	95.38(7)
N(1)-Fe(1)-N(20)#3	84.62(7)
N(10)-Fe(1)-N(20)#3	99.09(7)
N(10)#1-Fe(1)-N(20)#3	80.91(7)
N(20)#2-Fe(1)-N(20)#3	180.00(6)
N(1)-C(1)-S(1)	178.7(2)
C(1)-N(1)-Fe(1)	165.5(2)
C(10)-N(10)-N(11)	104.83(19)
C(10)-N(10)-Fe(1)	140.34(17)
N(11)-N(10)-Fe(1)	114.14(15)

C(12)-N(11)-N(10)	112.9(2)
N(10)-C(10)-C(11)	110.7(2)
N(10)-C(10)-C(13)	122.7(2)
C(11)-C(10)-C(13)	126.6(2)
C(12)-C(11)-C(10)	104.7(2)
C(12)-C(11)-C(21)	125.4(2)
C(10)-C(11)-C(21)	129.9(2)
N(11)-C(12)-C(11)	106.8(2)
N(11)-C(12)-C(14)	122.2(2)
C(11)-C(12)-C(14)	131.0(2)
N(21)-C(20)-C(21)	106.6(2)
N(21)-C(20)-C(23)	122.6(2)
C(21)-C(20)-C(23)	130.8(2)
C(20)-C(21)-C(22)	104.9(2)
C(20)-C(21)-C(11)	127.7(2)
C(22)-C(21)-C(11)	127.3(2)
N(20)-C(22)-C(21)	111.4(2)
N(20)-C(22)-C(24)	121.0(2)
C(21)-C(22)-C(24)	127.6(2)
C(22)-N(20)-N(21)	103.93(19)
C(22)-N(20)-Fe(1)#4	133.64(16)
N(21)-N(20)-Fe(1)#4	121.36(14)
C(20)-N(21)-N(20)	113.13(19)
C(100)#5-C(100)-C(100)#6	60.000(17)
C(100)#5-C(100)-N(100)	110(5)
C(100)#6-C(100)-N(100)	108(5)
C(100)#5-C(100)-C(101)	67.8(14)
C(100)#6-C(100)-C(101)	67.8(14)
N(100)-C(100)-C(101)	175(5)
C(100)#5-C(100)-N(100)#5	37(3)
C(100)#6-C(100)-N(100)#5	74(4)
N(100)-C(100)-N(100)#5	72(4)
C(101)-C(100)-N(100)#5	105(3)
C(100)#5-C(100)-N(100)#6	72(4)
C(100)#6-C(100)-N(100)#6	37(2)
N(100)-C(100)-N(100)#6	71(4)
C(101)-C(100)-N(100)#6	104(3)
N(100)#5-C(100)-N(100)#6	60(2)
C(100)-N(100)-C(100)#6	35(3)
C(100)-N(100)-N(100)#5	71(4)
C(100)#6-N(100)-N(100)#5	60(3)
C(100)-N(100)-N(100)#6	72(3)
C(100)#6-N(100)-N(100)#6	37.1(17)
N(100)#5-N(100)-N(100)#6	60.000(7)
C(100)-N(100)-C(100)#5	34(3)
C(100)#6-N(100)-C(100)#5	35(2)
N(100)#5-N(100)-C(100)#5	36.9(17)
N(100)#6-N(100)-C(100)#5	59(3)

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

Table 10. Anisotropic displacement parameters ($Å^2$ x 10^4) for A·0.27MeCN at 150 K. The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
Fe(1)	139(3)	183(3)	116(2)	-18(2)	-10(2)	82(2)
S(1)	168(3)	263(4)	214(3)	11(3)	15(3)	127(3)
C(1)	186(13)	125(11)	116(11)	-5(9)	-18(9)	43(10)
N(1)	177(11)	183(11)	142(10)	7(8)	9(8)	76(9)
N(10)	167(10)	182(11)	143(10)	-23(8)	-6(8)	80(9)
N(11)	189(11)	203(11)	145(10)	-18(8)	-34(8)	118(9)
C(10)	147(12)	162(12)	124(11)	15(9)	15(9)	53(10)
C(11)	157(12)	158(12)	114(11)	-8(9)	-13(9)	53(10)
C(12)	166(12)	200(13)	120(12)	-12(10)	-16(9)	81(10)
C(13)	282(15)	231(14)	180(13)	-33(11)	-52(11)	139(12)
C(14)	352(16)	297(15)	226(14)	-89(12)	-117(12)	219(14)
C(20)	189(13)	188(13)	135(12)	-6(10)	8(10)	105(11)
C(21)	177(12)	181(12)	95(11)	-4(9)	-1(9)	94(10)
C(22)	162(12)	182(12)	127(11)	-13(9)	-14(9)	91(10)
C(23)	259(15)	215(14)	227(14)	35(11)	-12(11)	60(12)
C(24)	233(14)	231(14)	179(13)	32(11)	-81(11)	69(12)
N(20)	165(10)	164(10)	121(10)	16(8)	13(8)	80(9)
N(21)	185(11)	174(11)	129(10)	32(8)	-10(8)	94(9)

Table 11. Hydrogen coordinates (x 10^3), isotropic displacement parameters ($Å^2$ x 10^3) and site occupation parameters for A·0.27MeCN at 150 K.

	x	y	z	U(eq)	Occ
H(11)	529	594	704	20	1000
H(13A)	574	443	655	34	500
H(13B)	583	437	798	34	500
H(13C)	640	481	716	34	500
H(13D)	624	465	791	34	500
H(13E)	615	470	648	34	500
H(13F)	558	426	731	34	500
H(14A)	566	665	870	40	500
H(14B)	633	680	910	40	500
H(14C)	576	634	990	40	500
H(14D)	617	655	977	40	500
H(14E)	550	639	937	40	500
H(14F)	608	685	857	40	500
H(23A)	604	444	1131	39	500
H(23B)	589	435	989	39	500
H(23C)	556	462	1074	39	500
H(23D)	562	450	998	39	500
H(23E)	577	459	1141	39	500
H(23F)	610	432	1055	39	500
H(24A)	779	705	945	35	500
H(24B)	715	695	901	35	500
H(24C)	749	668	822	35	500
H(24D)	716	674	833	35	500
H(24E)	781	684	878	35	500
H(24F)	746	710	957	35	500
H(21)	700	534	1174	19	1000
H(10A)	304	637	-241	150	68(2)
H(10B)	375	681	-212	150	68(2)
H(10C)	326	701	-178	150	68(2)
H(10D)	366	709	-179	150	68(2)
H(10E)	295	665	-208	150	68(2)
H(10F)	344	645	-243	150	68(2)

Table 12. Hydrogen bonds for A·0.27MeCN at 150 K [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(11)-H(11)...N(1)	0.88	2.60	3.049(3)	113.0
N(11)-H(11)...S(1)#7	0.88	2.77	3.598(2)	156.8
N(21)-H(21)...S(1)#8	0.88	2.56	3.384(2)	155.4
N(21)-H(21)...N(1)#4	0.88	2.60	3.059(3)	113.6

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+1 #2 x-y+1/3,x-1/3,-z+5/3 #3 -x+y+2/3,-x+4/3,z-2/3
#4 -y+4/3,x-y+2/3,z+2/3 #5 -y+1,x-y+1,z #6 -x+y,-x+1,z
#7 x-y+2/3,x+1/3,-z+4/3 #8 -x+1,-y+1,-z+2

Table 13. Crystal data and structure refinement for A at 150 K.

Identification code	A
Empirical formula	C22 H28 Fe N10 S2
Formula weight	552.51
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system, space group	Hexagonal, R-3
Unit cell dimensions	a = 25.2831(14) Å α = 90 ° b = 25.2831(14) Å β = 90 ° c = 10.9463(13) Å γ = 120 °
Volume	6059.8(9) $Å^3$
Z, Calculated density	9, 1.363 Mg/m^3
Absorption coefficient	0.746 mm^{-1}
F(000)	2592
Crystal size	0.20 x 0.18 x 0.15 mm
Theta range for data collection	1.61 to 27.99 °
Limiting indices	-33<=h<=33, -33<=k<=33, -14<=l<=14
Reflections collected / unique	17321 / 3241 [R(int) = 0.0412]
Completeness to theta = 27.99	99.4 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.804385
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3241 / 0 / 160
Goodness-of-fit on F^2	1.033
Final R indices [I>2sigma(I)]	R1 = 0.0423, wR2 = 0.0878
R indices (all data)	R1 = 0.0824, wR2 = 0.1036
Largest diff. peak and hole	0.588 and -0.461 $e.Å^{-3}$

Table 14. Atomic coordinates (x 10^4), equivalent isotropic displacement parameters ($Å^2$ x 10^4) and site occupation parameters for A at 150 K. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)	Occ
Fe(1)	5000	5000	5000	147(1)	1000
S(1)	3298(1)	5247(1)	6231(1)	214(2)	1000
C(1)	3916(1)	5215(1)	5951(2)	162(6)	1000
N(1)	4357(1)	5196(1)	5727(2)	170(5)	1000
N(10)	5482(1)	5275(1)	6819(2)	173(5)	1000
N(11)	5490(1)	5767(1)	7343(2)	168(5)	1000
C(10)	5847(1)	5162(1)	7531(2)	166(5)	1000
C(11)	6090(1)	5587(1)	8505(2)	152(5)	1000
C(12)	5841(1)	5962(1)	8351(2)	169(6)	1000
C(13)	5963(1)	4647(1)	7289(3)	224(6)	1000
C(14)	5901(2)	6481(1)	9083(3)	267(7)	1000
C(20)	6450(1)	5241(1)	10376(2)	177(6)	1000
C(21)	6527(1)	5656(1)	9472(2)	152(5)	1000
C(22)	7093(1)	6184(1)	9748(2)	168(6)	1000
C(23)	5941(1)	4610(1)	10607(3)	272(7)	1000
C(24)	7408(1)	6767(1)	9048(3)	240(6)	1000
N(20)	7347(1)	6102(1)	10757(2)	156(5)	1000
N(21)	6945(1)	5520(1)	11105(2)	165(5)	1000

Table 15. Bond lengths [Å] and angles [°] for A at 150 K.

Fe(1)-N(1)#1	2.079(2)
Fe(1)-N(1)	2.079(2)
Fe(1)-N(10)	2.255(2)
Fe(1)-N(10)#1	2.255(2)
Fe(1)-N(20)#2	2.385(2)
Fe(1)-N(20)#3	2.385(2)
S(1)-C(1)	1.633(3)
C(1)-N(1)	1.167(4)
N(10)-C(10)	1.342(3)
N(10)-N(11)	1.361(3)
N(11)-C(12)	1.345(3)
C(10)-C(11)	1.417(4)
C(10)-C(13)	1.496(4)
C(11)-C(12)	1.385(4)
C(11)-C(21)	1.477(4)
C(12)-C(14)	1.480(4)
C(20)-N(21)	1.348(3)
C(20)-C(21)	1.384(4)
C(20)-C(23)	1.487(4)
C(21)-C(22)	1.418(4)
C(22)-N(20)	1.344(3)
C(22)-C(24)	1.490(4)
N(20)-N(21)	1.360(3)
N(20)-Fe(1)#4	2.385(2)
N(1)#1-Fe(1)-N(1)	180.0
N(1)#1-Fe(1)-N(10)	92.55(8)
N(1)-Fe(1)-N(10)	87.45(8)
N(1)#1-Fe(1)-N(10)#1	87.45(8)
N(1)-Fe(1)-N(10)#1	92.55(8)
N(10)-Fe(1)-N(10)#1	180.0
N(1)#1-Fe(1)-N(20)#2	95.48(8)
N(1)-Fe(1)-N(20)#2	84.52(8)
N(10)-Fe(1)-N(20)#2	99.00(8)
N(10)#1-Fe(1)-N(20)#2	81.00(8)
N(1)#1-Fe(1)-N(20)#3	84.52(8)
N(1)-Fe(1)-N(20)#3	95.48(8)
N(10)-Fe(1)-N(20)#3	81.00(8)
N(10)#1-Fe(1)-N(20)#3	99.00(8)
N(20)#2-Fe(1)-N(20)#3	180.0
N(1)-C(1)-S(1)	178.6(3)
C(1)-N(1)-Fe(1)	165.4(2)
C(10)-N(10)-N(11)	104.6(2)
C(10)-N(10)-Fe(1)	140.39(18)
N(11)-N(10)-Fe(1)	114.32(16)
C(12)-N(11)-N(10)	112.7(2)
N(10)-C(10)-C(11)	111.0(2)
N(10)-C(10)-C(13)	122.5(2)
C(11)-C(10)-C(13)	126.5(2)
C(12)-C(11)-C(10)	104.7(2)
C(12)-C(11)-C(21)	125.3(2)
C(10)-C(11)-C(21)	130.0(2)
N(11)-C(12)-C(11)	107.0(2)
N(11)-C(12)-C(14)	121.7(2)
C(11)-C(12)-C(14)	131.3(2)

N(21)-C(20)-C(21)	106.5(2)
N(21)-C(20)-C(23)	122.7(2)
C(21)-C(20)-C(23)	130.7(3)
C(20)-C(21)-C(22)	104.7(2)
C(20)-C(21)-C(11)	127.7(3)
C(22)-C(21)-C(11)	127.4(2)
N(20)-C(22)-C(21)	111.5(2)
N(20)-C(22)-C(24)	120.9(2)
C(21)-C(22)-C(24)	127.6(2)
C(22)-N(20)-N(21)	103.9(2)
C(22)-N(20)-Fe(1)#4	133.49(18)
N(21)-N(20)-Fe(1)#4	121.54(16)
C(20)-N(21)-N(20)	113.4(2)

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

Table 16. Anisotropic displacement parameters ($Å^2$ x 10^4) for A at 150 K.
The anisotropic displacement factor exponent takes the form:
$-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
Fe(1)	134(3)	183(3)	123(2)	-17(2)	-10(2)	79(2)
S(1)	171(4)	270(4)	223(3)	15(3)	19(3)	127(3)
C(1)	180(14)	143(13)	126(12)	-4(10)	-1(10)	53(11)
N(1)	183(12)	180(12)	137(11)	13(9)	12(9)	84(10)
N(10)	189(12)	177(12)	155(11)	-10(9)	-15(9)	92(10)
N(11)	195(12)	175(12)	156(11)	-27(9)	-34(9)	108(10)
C(10)	159(13)	163(13)	147(12)	18(10)	15(10)	59(12)
C(11)	157(13)	160(13)	116(12)	-7(10)	-7(10)	64(11)
C(12)	168(14)	188(14)	131(12)	-11(10)	-19(10)	73(12)
C(13)	277(16)	226(15)	198(14)	-31(11)	-43(12)	148(13)
C(14)	347(18)	291(17)	221(15)	-88(13)	-112(13)	203(15)
C(20)	188(14)	212(14)	156(12)	-9(11)	14(11)	118(12)
C(21)	170(13)	189(14)	116(12)	-3(10)	6(10)	104(12)
C(22)	183(14)	204(14)	133(12)	2(10)	-9(10)	110(12)
C(23)	278(17)	219(16)	240(15)	42(12)	-15(13)	65(14)
C(24)	222(15)	244(16)	186(14)	32(12)	-62(12)	66(13)
N(20)	166(11)	166(12)	137(10)	13(9)	2(9)	83(10)
N(21)	175(12)	175(12)	151(11)	33(9)	-14(9)	92(10)

Table 17. Hydrogen coordinates (x 10^3), isotropic displacement parameters ($Å^2$ x 10^3) and site occupation parameters for A at 150 K.

	x	y	z	U(eq)	Occ
H(11)	529	594	705	20	1000
H(13A)	574	443	656	34	500
H(13B)	583	437	799	34	500
H(13C)	640	481	716	34	500
H(13D)	624	464	792	34	500
H(13E)	615	470	648	34	500
H(13F)	558	426	731	34	500
H(14A)	566	664	871	40	500
H(14B)	633	680	911	40	500
H(14C)	575	634	991	40	500
H(14D)	617	655	978	40	500
H(14E)	550	639	938	40	500
H(14F)	608	685	857	40	500
H(23A)	604	444	1133	41	500
H(23B)	589	435	990	41	500
H(23C)	556	462	1075	41	500
H(23D)	562	450	999	41	500
H(23E)	577	459	1142	41	500
H(23F)	610	432	1056	41	500
H(24A)	779	705	946	36	500
H(24B)	715	695	901	36	500
H(24C)	749	669	822	36	500
H(24D)	716	674	833	36	500
H(24E)	781	684	878	36	500
H(24F)	746	710	957	36	500
H(21)	700	534	1174	20	1000

Table 18. Hydrogen bonds for A at 150 K [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(11)-H(11)...N(1)	0.88	2.60	3.046(3)	112.6
N(11)-H(11)...S(1)#5	0.88	2.77	3.596(2)	157.1
N(21)-H(21)...S(1)#6	0.88	2.57	3.384(2)	154.9
N(21)-H(21)...N(1)#4	0.88	2.59	3.055(3)	113.7

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