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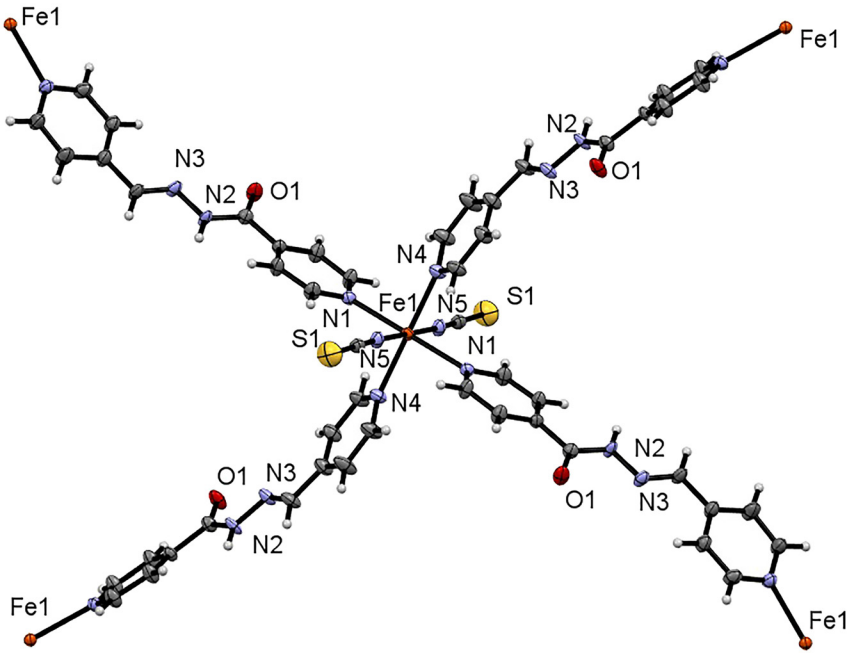
Crystal structure of poly[(bis(isothiocyano)-bis(μ_2-(*E*)-*N*′-(pyridin-4-ylmethylene) isonicotinohydrazide))iron(II) – methanol – 1,4-dioxane (1/2/2), $C_{36}H_{44}FeN_{10}O_8S_2$



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Abstract

$C_{36}H_{44}FeN_{10}O_8S_2$, monoclinic, $P2_1/c$ (no. 14), a = 8.7712(5) Å, b = 24.6087(12) Å, c = 9.6083(4) Å, β = 96.952(5)°, V = 2058.68(18) Å^3, Z = 2, $R_{gt}(F)$ = 0.0664, $wR_{ref}(F^2)$ = 0.1318, T = 173(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.35 × 0.21 × 0.12 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.53 mm^{-1}
Diffractometer, scan mode:	Xcalibur, φ and ω
θ_{max}, completeness:	29.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10,617, 4710, 0.054
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3405
$N(param)_{refined}$:	301
Programs:	CrysAlis^{PRO} [1], Olex2 [2, 3], Shelx [4], Mercury [5]

Source of material

The Schiff base ligand (*E*)-*N*′-(pyridin-4-ylmethylene) isonicotinohydrazide was prepared from the condensation of isonicotinaldehyde (CAS: 872-85-5) and isonicotinohydrazide (CAS: 54-85-3) under the reflux in methanol solution. The title

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
Fe1	0.500000	0.500000	1.000000	0.01685 (16)
S1	0.28616 (13)	0.45368 (5)	1.42892 (10)	0.0565 (3)
O1	0.1834 (3)	0.28025 (8)	0.57201 (19)	0.0312 (6)
N1	0.4082 (3)	0.42035 (10)	0.9076 (2)	0.0210 (6)
N2	0.1546 (3)	0.23825 (10)	0.7762 (2)	0.0245 (6)
H2	0.175378	0.237858	0.868171	0.029*
N3	0.0744 (3)	0.19640 (10)	0.7065 (2)	0.0254 (6)
N4	−0.2898 (3)	0.04243 (10)	0.5917 (2)	0.0253 (6)
N5	0.3980 (3)	0.48389 (10)	1.1815 (3)	0.0250 (6)
C1	0.3298 (4)	0.38493 (13)	0.9775 (3)	0.0293 (8)
H1	0.319283	0.392615	1.072786	0.035*
C2	0.2635 (4)	0.33798 (13)	0.9197 (3)	0.0270 (7)
H2A	0.210902	0.313844	0.974631	0.032*
C3	0.2749 (4)	0.32676 (12)	0.7809 (3)	0.0207 (7)
C4	0.3535 (4)	0.36306 (12)	0.7077 (3)	0.0283 (8)
H4	0.362441	0.356817	0.611469	0.034*
C5	0.4190 (4)	0.40831 (13)	0.7733 (3)	0.0273 (7)
H5	0.474792	0.432304	0.720800	0.033*
C6	0.2003 (4)	0.27971 (12)	0.7006 (3)	0.0228 (7)
C7	0.0363 (4)	0.15675 (13)	0.7807 (3)	0.0307 (8)
H7	0.072483	0.154582	0.877772	0.037*
C8	−0.0642 (4)	0.11483 (13)	0.7125 (3)	0.0299 (8)
C9	−0.1271 (4)	0.07564 (14)	0.7920 (3)	0.0412 (10)
H9	−0.093949	0.072911	0.889625	0.049*
C10	−0.2375 (4)	0.04068 (14)	0.7301 (3)	0.0360 (9)
H10	−0.278733	0.014163	0.786902	0.043*
C11	−0.2210 (4)	0.07825 (13)	0.5146 (3)	0.0297 (8)
H11	−0.249972	0.078460	0.416114	0.036*
C12	−0.1115 (4)	0.11452 (13)	0.5693 (3)	0.0302 (8)
H12	−0.068102	0.139388	0.509465	0.036*
C13	0.3507 (4)	0.47144 (12)	1.2850 (3)	0.0246 (7)
O2	−0.4672 (4)	0.23927 (14)	0.7816 (3)	0.0685 (9)
O3	−0.2452 (3)	0.18799 (12)	0.9740 (3)	0.0568 (8)
C14A[a]	−0.3108 (10)	0.2567 (5)	0.8070 (16)	0.065 (4)
H14A[a]	−0.304385	0.296103	0.787514	0.078*
H14B[a]	−0.248621	0.237124	0.743762	0.078*
C14B[b]	−0.361 (2)	0.2717 (6)	0.879 (2)	0.055 (5)
H14C[b]	−0.405568	0.278806	0.967229	0.066*
H14D[b]	−0.338333	0.306981	0.836316	0.066*
C15A[c]	−0.248 (2)	0.2455 (4)	0.958 (3)	0.060 (4)
H15A[c]	−0.143558	0.260770	0.979246	0.073*
H15B[c]	−0.315119	0.262189	1.022202	0.073*
C15B[d]	−0.219 (3)	0.2387 (7)	0.907 (3)	0.045 (4)
H15C[d]	−0.178250	0.231413	0.817412	0.054*
H15D[d]	−0.140973	0.259662	0.967671	0.054*
C16A[e]	−0.456 (2)	0.1808 (4)	0.805 (2)	0.070 (4)
H16A[e]	−0.384257	0.164996	0.743496	0.083*
H16B[e]	−0.557703	0.163905	0.780228	0.083*
C16B[f]	−0.509 (3)	0.1904 (10)	0.845 (3)	0.063 (6)
H16C[f]	−0.578571	0.168767	0.777708	0.075*
H16D[f]	−0.562757	0.198581	0.927597	0.075*
C17A[g]	−0.3977 (12)	0.1688 (7)	0.9572 (15)	0.056 (4)
H17A[g]	−0.460055	0.188147	1.020895	0.067*
H17B[g]	−0.401096	0.129356	0.976486	0.067*
C17B[h]	−0.3706 (19)	0.1610 (6)	0.887 (3)	0.090 (8)
H17C[h]	−0.330257	0.148258	0.800987	0.108*
H17D[h]	−0.398402	0.128223	0.938362	0.108*
O4	−0.0966 (4)	0.35938 (13)	0.7284 (3)	0.0689 (9)
H4A	−0.133784	0.343806	0.654137	0.103*
C18	−0.0410 (5)	0.41121 (19)	0.6959 (4)	0.0607 (12)
H18A	0.030177	0.407478	0.625382	0.091*
H18B	0.012612	0.427700	0.780939	0.091*
H18C	−0.127414	0.434389	0.658941	0.091*

[a]Occupancy: 0.63 (2), [b]Occupancy: 0.37 (2), [c]Occupancy: 0.59 (4), [d]Occupancy: 0.41 (4), [e]Occupancy: 0.60 (4), [f]Occupancy: 0.40 (4), [g]Occupancy: 0.54 (3), [h]Occupancy: 0.46 (3).

compound was prepared by the liquid-phase diffusion method. Firstly, 5 mL solution of (*E*)-*N*′-(pyridin-4-ylmethylene)isonicotinohydrazide (45.2 mg, 0.2 mmol) was put at the bottom of the test-tube, then, 5 mL mixed solution of methanol and 1,4-dioxane (V:V = 1:1) was layered on the previously mentioned solution. At last, 5 mL methanolic $Fe(NCS)_2$ (0.1 mmol/L, prepared according to the literature method [6]) solution was slowly layered on the top of test-tube. After one week, red block-shaped crystals of the title compound were obtained.

Experimental details

Restrained distances O3–C17A ≃ O3–C17B, C16A–C17A ≃ C16A–C17B, O2–C16A ≃ O2–C16B, C16A–C17A ≃ C16B–C17A, C16A–C17B ≃ C16B–C17B, O2–C14A ≃ O2–C14B, C15A–C14A ≃ C15A–C14B, O3–C15A ≃ O3–C15B, C15A–C14A ≃ C15B–C14A,C15A–C14B ≃ C15B–C14B with sigma of 0.02.

Comment

Schiff base derivatives have been widely studied as complex ligands [7, 8], or catalyst-active materials [9]. Its linear dipyridine derivatives were excellent linkers for polymeric complexes [10]. However, the coordination compounds of (*E*)-*N*′-(pyridin-4-ylmethylene)isonicotinohydrazide have not been reported yet. Herein, we report the crystal structure of a 2D-grid Fe(II) polymeric compound involving the Schiff base ligand (*E*)-*N*′-(pyridin-4-ylmethylene) isonicotinohydrazide.

The Fe(II) is bridged by two (*E*)-*N*′-(pyridin-4-ylmethylene)isonicotinohydrazide linkers to form the 2D-grid layer structure. Similar networks have been already reported [11, 12]. These layers are two-fold interpenetration.

The layers are interlinked to from 3D supramolecular networks by four intermolecular hydrogen bonds: N2–H2···O1 (symmetry code: x, $-y+1/2$, $z+1/2$) [length 2.00(1) Å, angle 164.37(2)°], C2–H2A···O1 (symmetry code: $x-1$, y, z) [length 2.52(2) Å, angle 148.80(4)°], C7–H7···O1 (symmetry code: x, $-y+1/2$, $z+1/2$) [length 2.56(2) Å, angle 137.11(4)°], C2–H2A···N3 (symmetry code: x, $-y+1/2$, $z+1/2$) [length 2.66(5) Å, angle 145.13(3)°] between the layers.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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