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Crystal structure of *N',N'''*-((1*E*,1'*E*)-((decane-1,10-diylbis(oxy))bis(2,1-phenylene))bis(methanelylidene))di(isonicotinohydrazide), $C_{36}H_{40}N_6O_4$

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Abstract

$C_{36}H_{40}N_6O_4$, monoclinic, $P2_1/c$ (no. 14), $a = 11.094(6)$ Å, $b = 17.694(11)$ Å, $c = 8.698(5)$ Å, $\beta = 103.56(2)^\circ$, $V = 1659.9(16)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0470$, $wR_{ref}(F^2) = 0.1375$, $T = 273$ 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:	Colourless block
Size:	0.25 × 0.22 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffraction, scan mode:	D8/APEX2, φ and ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	30844, 2926, 0.081
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1983
$N(param)_{refined}$:	208
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

Source of material

A solution of 2,2'-(decane-1,10-diylbis(oxy))dibenzaldehyde (0.76 g, 2.0 mmol) in ethanol (50 mL) was added to a stirred ethanol solution (50 mL) of isonicotinohydrazide (0.55 g, 4.0 mmol). The reaction mixture was refluxed for 8 h, then the mixture was cooled to room temperature. The white precipitated residue was removed by filtration and then washed with ethanol three times. The white solids

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (²/Å²).

Atom	x	y	z	U _{iso} */U _{eq}
C1	0.0084 (2)	0.16008 (15)	0.1557 (4)	0.0806 (8)
H1	−0.071239	0.163079	0.091178	0.097*
C2	0.1064 (2)	0.18228 (13)	0.0925 (3)	0.0678 (6)
H2A	0.092771	0.197627	−0.012384	0.081*
C3	0.22485 (19)	0.18116 (11)	0.1887 (2)	0.0516 (5)
C4	0.2390 (2)	0.15567 (12)	0.3411 (3)	0.0600 (6)
H4	0.317066	0.153934	0.409752	0.072*
C5	0.1352 (3)	0.13264 (14)	0.3908 (3)	0.0776 (7)
H5	0.146514	0.114368	0.493395	0.093*
C6	0.33254 (19)	0.20559 (11)	0.1242 (2)	0.0512 (5)
C7	0.58877 (19)	0.31823 (12)	0.2782 (2)	0.0532 (5)
H7	0.564944	0.330439	0.370833	0.064*
C8	0.70311 (18)	0.35032 (12)	0.2499 (2)	0.0537 (5)
C9	0.7602 (2)	0.32037 (14)	0.1379 (3)	0.0697 (7)
H9	0.726954	0.277761	0.080786	0.084*
C10	0.8667 (2)	0.35349 (17)	0.1101 (3)	0.0820 (8)
H10	0.904385	0.333250	0.034448	0.098*
C11	0.9159 (2)	0.41611 (17)	0.1946 (3)	0.0785 (7)
H11	0.986951	0.438267	0.175326	0.094*
C12	0.8623 (2)	0.44654 (14)	0.3070 (3)	0.0696 (6)
H12	0.896764	0.488946	0.363811	0.084*
C13	0.75605 (18)	0.41364 (13)	0.3356 (2)	0.0562 (5)
C14	0.7509 (2)	0.49630 (13)	0.5546 (3)	0.0657 (6)
H14A	0.760950	0.543301	0.501521	0.079*
H14B	0.831819	0.479380	0.613280	0.079*
C15	0.6654 (2)	0.50723 (13)	0.6638 (3)	0.0664 (6)
H15A	0.584805	0.523297	0.602462	0.080*
H15B	0.697894	0.547081	0.738637	0.080*
C16	0.6498 (3)	0.43612 (13)	0.7540 (3)	0.0762 (7)
H16A	0.726171	0.426769	0.832536	0.091*
H16B	0.636621	0.393841	0.680962	0.091*
C17	0.5431 (2)	0.43894 (14)	0.8367 (3)	0.0741 (7)
H17A	0.466142	0.444668	0.757177	0.089*
H17B	0.539445	0.390976	0.889436	0.089*
C18	0.5515 (2)	0.50107 (13)	0.9557 (3)	0.0657 (6)
H18A	0.548821	0.549311	0.902034	0.079*
H18B	0.630832	0.497592	1.031572	0.079*
N1	0.0205 (2)	0.13490 (13)	0.3016 (3)	0.0849 (7)
N2	0.41619 (15)	0.24825 (9)	0.22356 (19)	0.0526 (4)
H2	0.404775	0.259711	0.315175	0.063*
N3	0.52103 (15)	0.27385 (10)	0.1786 (2)	0.0544 (4)
O1	0.33980 (14)	0.18754 (8)	−0.00953 (17)	0.0646 (4)
O2	0.69441 (14)	0.44041 (9)	0.44244 (18)	0.0718 (5)

(0.1 mmol) were dissolved in a solution mixture (20.0 mL, CH₃OH/DMF, 9:1, v/v) within three days. Crystals were obtained by slow evaporation at room temperature.

Experimental details

Using Olex2 [1], the structure was solved using Charge Flipping and refined with the ShelXL [3] refinement. All

hydrogen atoms were positioned geometrically, with the $d(\text{C-H}) = 0.97\text{--}0.99$ Å, $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{parent atom})$.

Comment

A bis-pyridylhydrazone Schiff base ligand was synthesized. The nitrogen atoms on the pyridine ring have strong nucleophilic ability [5]. Acylhydrazone Schiff base compounds can be easily assembled with different transition metal ions in different coordination modes to form novel complexes [6].

In the crystal structure of the title compound, the asymmetric unit of the title compound consists of one target molecule. The title molecule shows a slightly twisted “Z” shape (see the Figure). The bond lengths and bond angles are in the normal ranges [6]. The amide forms an intermolecular hydrogen bond with the amide N on another molecule. The intermolecular N–H...O hydrogen bonds in the target compound extend the framework into a 2D structure.

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

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