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Crystal structure of (*E*)-*N'*-((1,6-dihydropyren-1-yl)methylene)isonicotinohydrazide – methanol (1/1), C₂₄H₁₉N₃O₂

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Abstract

C₂₄H₁₉N₃O₂, monoclinic, *P*₂₁/*c* (no. 14), *a* = 11.0629(2) Å, *b* = 10.4583(2) Å, *c* = 16.0432(3) Å, β = 92.373(2)°, *V* = 1854.59(7) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0435, *wR*_{ref}(*F*²) = 0.1269, *T* = 116.2(3) K.

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The asymmetric unit of the cocrystal title 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.

Source of material

All solvents and starting materials are commercially available and used without further treatment. The target compound was facily synthesized by one-step condensation reaction according to reference [5].

Pyrene-1-carbaldehyde (1 mmol, 230 mg) was dispersed with appropriate amount of ethanol. Isonicotinohydrazide (1 mmol, 137 mg) was dissolved with appropriate amount of absolute ethanol to obtain a colorless solution. Then the two solutions were added under stirring. The stirred mixture was refluxed for 4 h to appear yellow precipitation immediately.

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Table 1: Data collection and handling.

Crystal:	Dark yellow cube
Size:	0.40 × 0.20 × 0.10 mm
Wavelength:	Cu Kα radiation (1.54184 Å)
μ:	0.71 mm ^{−1}
Diffractometer, scan mode:	SuperNova, ω
θ _{max} , completeness:	71.6°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	6518, 3501, 0.024
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3098
<i>N</i> (<i>param</i>) _{refined} :	264
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3, 4]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.42268(9)	0.10172(9)	0.69591(7)	0.0324(3)
N1	0.01551(11)	0.11005(12)	0.81951(7)	0.0297(3)
N2	0.37482(10)	0.31257(11)	0.68270(7)	0.0238(3)
H2	0.3228	0.3716	0.6915	0.029*
N3	0.48052(10)	0.34115(11)	0.64438(7)	0.0245(3)
C1	0.16219(12)	0.26467(13)	0.77868(8)	0.0259(3)
H1	0.1851	0.3501	0.7760	0.031*
C2	0.05517(13)	0.23032(14)	0.81389(9)	0.0286(3)
H2A	0.0076	0.2950	0.8351	0.034*
C3	0.08805(13)	0.01907(14)	0.79100(9)	0.0307(3)
H3	0.0634	−0.0656	0.7954	0.037*
C4	0.19749(13)	0.04323(13)	0.75535(8)	0.0275(3)
H4	0.2450	−0.0237	0.7371	0.033*
C5	0.23513(12)	0.16935(13)	0.74738(8)	0.0230(3)
C6	0.35336(12)	0.19035(13)	0.70646(8)	0.0232(3)
C7	0.49086(12)	0.45959(13)	0.62524(8)	0.0235(3)
H7	0.4305	0.5168	0.6391	0.028*
C8	0.59554(12)	0.50589(12)	0.58216(8)	0.0217(3)
C9	0.67768(12)	0.41868(12)	0.54982(8)	0.0241(3)
H9	0.6650	0.3315	0.5570	0.029*
C10	0.77645(12)	0.45904(12)	0.50781(8)	0.0244(3)
H10	0.8295	0.3989	0.4871	0.029*
C11	0.79845(12)	0.58999(12)	0.49563(8)	0.0219(3)
C12	0.90103(12)	0.63484(14)	0.45253(8)	0.0259(3)
H12	0.9555	0.5758	0.4324	0.031*
C13	0.92043(13)	0.76079(14)	0.44049(8)	0.0281(3)
H13	0.9879	0.7870	0.4122	0.034*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C14	0.83888(13)	0.85459(13)	0.47052(8)	0.0262(3)
C15	0.85508(14)	0.98650(14)	0.45752(9)	0.0325(3)
H15	0.9208	1.0148	0.4283	0.039*
C16	0.77467(16)	1.07425(14)	0.48755(10)	0.0374(4)
H16	0.7866	1.1609	0.4778	0.045*
C17	0.67646(14)	1.03539(14)	0.53204(10)	0.0335(3)
H17	0.6237	1.0960	0.5524	0.040*
C18	0.65625(13)	0.90504(13)	0.54649(8)	0.0263(3)
C19	0.55628(13)	0.86126(13)	0.59229(9)	0.0281(3)
H19	0.5044	0.9208	0.6148	0.034*
C20	0.53539(12)	0.73495(13)	0.60370(8)	0.0251(3)
H20	0.4689	0.7097	0.6333	0.030*
C21	0.61399(12)	0.63889(12)	0.57105(8)	0.0208(3)
C22	0.71619(11)	0.68008(12)	0.52723(8)	0.0205(3)
C23	0.73715(12)	0.81372(12)	0.51454(8)	0.0223(3)
O2	−0.23393(9)	0.05521(10)	0.79425(7)	0.0357(3)
H2B	−0.1638	0.0658	0.8113	0.054*
C24	−0.26909(16)	0.15720(19)	0.74123(12)	0.0464(4)
H24A	−0.2144	0.1631	0.6965	0.070*
H24B	−0.2672	0.2356	0.7723	0.070*
H24C	−0.3497	0.1424	0.7188	0.070*

The product was filtered, washed several times with ethanol/water (1:1) to obtain yellow powder. Yield: ca. 80%.

The yellow powder (0.05 mmol, 17.5 mg) was dissolved in methanol/dichloromethane (4:1) (10 mL) under stirring to give a pale yellow solution. This solution was filtered and evaporated slowly at room temperature. After one week, yellow cube crystals appeared. Yield: ca. 60%.

Experimental details

The H atoms were added geometrically using riding models and refined isotropically. Their *U*_{iso} values were set to 1.2*U*_{eq} of the parent C and N atoms, and 1.5*U*_{eq} of the parent O atoms.

Comment

The application of fluorescent probe technology has been widely focused on by more and more scientists in biochemistry, analytical chemistry and medicine engineering in recent years [6–9]. The fluorescent probes based on pyrene derivatives have been reported [5, 10]. We herein report a

new pyrene-derived compound, which has good coordination ability to transition metal and rare-earth metal ions.

The asymmetric unit of the title compound contains a neutral molecule and a methanol solvent molecule. The bond length of C7–N3 is 1.2824(18) Å, which indicate that the target compound was synthesized successfully. Methanol molecules link the adjacent molecules into a chain structure. Hydrogen bond length between N1 of pyridine ring and O2 on methanol hydroxyl group is 2.831 Å. Hydrogen bond length between N2 of imine and O2 on methanol hydroxyl group is 3.009 Å (*cf.* the figure).

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