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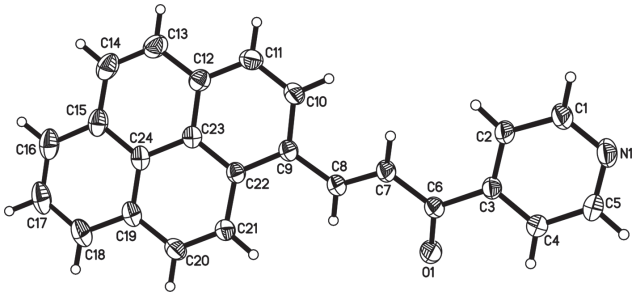
Crystal structure of (*E*)-3-(pyren-1-yl)-1-(pyridin-4-yl)prop-2-en-1-one, $C_{24}H_{15}NO$



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Abstract

$C_{24}H_{15}NO$, monoclinic, $P2_1/n$ (no. 14), $a = 10.3161(3)$ Å, $b = 5.53330(10)$ Å, $c = 29.2019(7)$ Å, $\beta = 97.341(1)°$, $V = 1653.24(7)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0423$, $wR_{ref}(F^2) = 0.1344$, $T = 294(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	red block
Size:	0.23 × 0.21 × 0.13 mm
Wavelength:	Cu $K\alpha$ radiation (1.54178 Å)
μ:	0.64 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
$2\theta_{max}$, completeness:	68.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	18178, 3026, 0.025
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2697
$N(param)_{refined}$:	235
Programs:	Bruker programs [1], SHELX [2]

Source of material

1-Pyrenecarboxaldehyde (230 mg, 1.0 mmol) and methanol (30 mL) were placed in a round-bottom flask and stirred at room temperature for 10 min. Then 4-acetylpyridine (145 mg, 1.2 mmol) and NaOH (5 mol/L, 10 mL) were added. The resulting reaction mixture was stirred at room temperature for 30 min. The mixture was extracted with CH_2Cl_2. The solvent was removed under reduced pressure to give the crude product, which was purified by column chromatography (petroleum ether:ethyl acetate = 1:4) to give the title compound as a red solid (isolated yield 50%). The isolated product was crystallized from $CHCl_3$ to give red single crystals.

Experimental details

All hydrogen H atoms were positioned geometrically and refined using a riding model, with d(C—H) = 0.93 Å and $U_{iso}(H) = 1.2\,U_{eq}(C)$.

Discussion

Pyrenes are considered to be excellent organic fluorophores and they are applied as environmental probes with distinct morphology and tunable emission in various micro environments due to their polarity dependent vibronic emission, high charge carrier mobility, fluorescence lifetime and chemical stability [3–6]. Up to now, numerous pyrene derivatives with good fluorescence properties have been reported. For example, (2-pyrene-1-yl-vinyl)pyridine has reversible luminescence and nonlinear optical properties [7], 4-((pyren-1-yl)methyleneamino)-1,2-dihydro-2,3-dimethyl-1-phenylpyrazol-5-one exhibits notable aggregation induced emission (AIE) phenomena in both solution and solid state [5]. 1-Methyl benzoate-pyrene displays pure blue monomer photoluminescence and electrogenerated chemiluminescence emissions in non-aqueous media [8]. In order to search for more pyrene-based compounds, in this paper we report a new one.

There is one molecule in the asymmetric unit (*cf*. the figure). Bond lengths and angles are all in the expected ranges [9]. The non-H atoms of the pyrene ring and pyridine ring are nearly planar, respectively. But the pyrene unit is not coplanar

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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}
C1	1.13266(18)	0.4802(3)	0.69654(5)	0.0734(5)
H1A	1.1843	0.6183	0.7000	0.088*
C2	1.05016(17)	0.4520(3)	0.65563(5)	0.0662(4)
H2A	1.0471	0.5692	0.6327	0.079*
C3	0.97281(11)	0.2493(2)	0.64914(4)	0.0432(3)
C4	0.98248(14)	0.0860(3)	0.68488(5)	0.0595(4)
H4A	0.9320	−0.0538	0.6824	0.071*
C5	1.06724(16)	0.1301(3)	0.72436(5)	0.0684(4)
H5A	1.0719	0.0166	0.7480	0.082*
C6	0.88559(12)	0.1946(2)	0.60518(4)	0.0469(3)
C7	0.85779(12)	0.3846(2)	0.57063(4)	0.0488(3)
H7A	0.8927	0.5378	0.5769	0.059*
C8	0.78414(11)	0.3453(2)	0.53054(4)	0.0456(3)
H8A	0.7435	0.1955	0.5266	0.055*
C9	0.76119(11)	0.5157(2)	0.49218(4)	0.0427(3)
C10	0.84922(12)	0.7051(2)	0.48882(4)	0.0514(3)
H10A	0.9188	0.7246	0.5121	0.062*
C11	0.83639(13)	0.8630(2)	0.45240(5)	0.0554(3)
H11A	0.8967	0.9874	0.4516	0.066*
C12	0.73426(12)	0.8399(2)	0.41647(4)	0.0484(3)
C13	0.72046(15)	0.9974(3)	0.37723(5)	0.0630(4)
H13A	0.7810	1.1207	0.3756	0.076*
C14	0.62250(16)	0.9714(3)	0.34280(5)	0.0668(4)
H14A	0.6167	1.0762	0.3177	0.080*
C15	0.52691(13)	0.7862(3)	0.34387(4)	0.0570(4)
C16	0.42289(16)	0.7567(4)	0.30861(5)	0.0712(4)
H16A	0.4145	0.8614	0.2835	0.085*
C17	0.33301(16)	0.5749(4)	0.31061(5)	0.0763(5)
H17A	0.2650	0.5575	0.2867	0.092*
C18	0.34197(14)	0.4180(3)	0.34758(5)	0.0669(4)
H18A	0.2801	0.2963	0.3483	0.080*
C19	0.44338(12)	0.4404(3)	0.38405(4)	0.0516(3)
C20	0.45603(12)	0.2846(3)	0.42343(4)	0.0550(3)
H20A	0.3938	0.1647	0.4254	0.066*
C21	0.55567(12)	0.3066(2)	0.45787(4)	0.0493(3)
H21A	0.5606	0.2014	0.4829	0.059*
C22	0.65441(10)	0.4888(2)	0.45682(4)	0.0405(3)
C23	0.64225(11)	0.6509(2)	0.41870(4)	0.0418(3)
C24	0.53697(11)	0.6257(2)	0.38224(4)	0.0459(3)
N1	1.14263(13)	0.3243(2)	0.73074(4)	0.0640(3)
O1	0.84364(12)	−0.01056(18)	0.59898(4)	0.0743(4)

with the pyridine ring (dihedral angle = 12.96(6)°). In addition, there are intermolecular C—H···O distances, which may be considered as very weak hydrogen bonds with the bond length of 3.4554(16) Å and bond angle of 145.8° [10]. Molecules are linked leading to one-dimensional chain along the *b* direction. Two adjacent chains are further connected by another possible C—H···O hydrogen bond with the bond length of 3.457(2) Å and a bond angle of 146.0° forming a double chain. The double chains are stacked by van der Waals forces resulting in a three-dimensional supramolecular architecture in the solid state.

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