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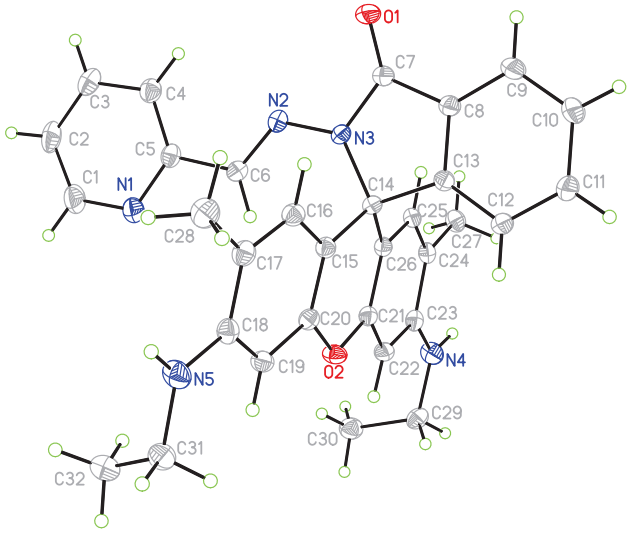
Crystal structure of (*E*)-3′,6′-bis(ethylamino)-2′,7′-dimethyl-2-((pyridin-2-ylmethylene)amino)spiro[isoindoline-1,9′-xanthen]-3-one, $C_{32}H_{31}N_5O_2$



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Abstract

$C_{32}H_{31}N_5O_2$, triclinic, $P\bar{1}$ (no. 2), $a = 9.9226(4)$ Å, $b = 11.1446(7)$ Å, $c = 12.6719(6)$ Å, $\alpha = 90.693(4)°$, $\beta = 109.858(4)°$, $\gamma = 95.382(4)°$, $Z = 2$, $V = 1310.72(12)$ Å^3, $R_{gt}(F) = 0.0497$, $wR_{ref}(F^2) = 0.1419$, $T = 173(10)$ 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.

Source of material

All of the starting materials and solvents were reagent grade and were used without purification. The title compound was obtained from Rhodamine 6G by a two-step reaction.

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Table 1: Data collection and handling.

Crystal:	Colorless rod-like
Size:	0.40 × 0.30 × 0.10 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	0.67 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	76.8°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8793, 5295, 0.024
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4778
$N(param)_{refined}$:	356
Programs:	OLEX2 [1], SHELX [2, 3], CrysAlis^{PRO} [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O2	0.62952(11)	0.42340(9)	0.52425(8)	0.0301(2)
O1	0.64105(13)	0.19506(11)	0.99411(9)	0.0408(3)
N3	0.66350(12)	0.26114(10)	0.82731(9)	0.0257(2)
N2	0.78010(12)	0.19989(11)	0.84103(10)	0.0280(3)
N4	0.30616(14)	0.09943(13)	0.30566(11)	0.0367(3)
H4	0.2468	0.0372	0.3031	0.044*
N5	1.00517(15)	0.72836(13)	0.71075(12)	0.0399(3)
H5	1.0648	0.7613	0.7728	0.048*
N1	1.03909(15)	0.16870(14)	0.71182(12)	0.0423(3)
C26	0.53032(13)	0.27276(12)	0.62230(11)	0.0241(3)
C20	0.71318(14)	0.47944(12)	0.62597(11)	0.0257(3)
C15	0.70087(14)	0.44633(12)	0.72746(11)	0.0251(3)
C13	0.47804(14)	0.38260(12)	0.77614(11)	0.0255(3)
C25	0.44520(14)	0.16212(12)	0.60991(11)	0.0264(3)
H25	0.4372	0.1275	0.6742	0.032*
C21	0.54415(14)	0.31894(12)	0.52489(11)	0.0248(3)
C14	0.59497(14)	0.34197(12)	0.73478(11)	0.0241(3)
C22	0.47296(15)	0.26201(13)	0.41981(11)	0.0278(3)
H22	0.4846	0.2958	0.3563	0.033*
C23	0.38425(14)	0.15456(13)	0.40892(12)	0.0279(3)
C19	0.81302(15)	0.57225(13)	0.61875(12)	0.0290(3)
H19	0.8182	0.5919	0.5490	0.035*
C24	0.37278(14)	0.10188(13)	0.50770(12)	0.0277(3)
C16	0.79453(15)	0.51148(13)	0.82389(11)	0.0286(3)
H16	0.7879	0.4916	0.8932	0.034*
C7	0.60180(15)	0.25416(13)	0.91108(11)	0.0289(3)
C17	0.89611(15)	0.60368(13)	0.82101(12)	0.0307(3)
C18	0.90547(15)	0.63610(13)	0.71564(12)	0.0299(3)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C5	0.97640(15)	0.14832(13)	0.78891(12)	0.0307(3)
C8	0.48382(15)	0.33203(13)	0.87663(11)	0.0285(3)
C6	0.84644(15)	0.21117(13)	0.77075(11)	0.0286(3)
H6	0.8134	0.2583	0.7089	0.034*
C12	0.37338(16)	0.45832(14)	0.72485(12)	0.0312(3)
H12	0.3688	0.4927	0.6574	0.037*
C4	1.02854(17)	0.07489(15)	0.87850(13)	0.0365(3)
H4A	0.9813	0.0628	0.9301	0.044*
C27	0.28226(16)	−0.01633(14)	0.49949(13)	0.0336(3)
H27A	0.2895	−0.0405	0.5735	0.050*
H27B	0.3159	−0.0767	0.4628	0.050*
H27C	0.1836	−0.0072	0.4569	0.050*
C29	0.31836(17)	0.14032(14)	0.20117(12)	0.0350(3)
H29A	0.3209	0.2275	0.2017	0.042*
H29B	0.2332	0.1076	0.1398	0.042*
C11	0.27577(18)	0.48093(16)	0.77781(14)	0.0399(4)
H11	0.2046	0.5311	0.7449	0.048*
C3	1.15248(18)	0.01975(16)	0.88987(15)	0.0425(4)
H3	1.1895	−0.0304	0.9487	0.051*
C9	0.38677(18)	0.35506(16)	0.93018(13)	0.0388(4)
H9	0.3921	0.3212	0.9980	0.047*
C28	0.99480(18)	0.67037(16)	0.92720(14)	0.0409(4)
H28A	0.9757	0.7533	0.9257	0.061*
H28B	1.0931	0.6659	0.9328	0.061*
H28C	0.9785	0.6344	0.9908	0.061*
C10	0.28229(19)	0.42992(17)	0.87916(15)	0.0431(4)
H10	0.2156	0.4465	0.9127	0.052*
C31	1.01471(18)	0.77284(14)	0.60603(15)	0.0406(4)
H31A	1.0584	0.8557	0.6191	0.049*
H31B	0.9181	0.7726	0.5520	0.049*
C30	0.4508(2)	0.10433(16)	0.17964(14)	0.0425(4)
H30A	0.4540	0.1365	0.1105	0.064*
H30B	0.4465	0.0180	0.1746	0.064*
H30C	0.5358	0.1358	0.2403	0.064*
C2	1.21884(18)	0.04142(19)	0.81171(17)	0.0498(4)
H2	1.3024	0.0068	0.8169	0.060*
C32	1.1013(2)	0.69960(18)	0.55593(16)	0.0485(4)
H32A	1.0965	0.7294	0.4841	0.073*
H32B	1.0623	0.6164	0.5468	0.073*
H32C	1.1998	0.7066	0.6052	0.073*
C1	1.1587(2)	0.1158(2)	0.72517(17)	0.0531(5)
1	1.2046	0.1299	0.6729	0.064*

Rhodamine 6G hydrozone is prepared according to the literature method [5]. To a 50 mL flask, Rhodamine 6G (1 mmol, 0.479 g) was dissolved in 20 mL ethanol. 2.0 mL (excess) hydrazine monohydrate (80%) was then added dropwise under stirring at room temperature. After the addition, the stirred mixture was refluxed for 3 h, during which the pink precipitate appeared. The precipitate was filtered, washed 3 times with methanol/water (1:1). Yield: *ca* 80%.

The synthesis of the title compound. Rhodamine 6G hydrozone (0.5 mmol, 0.214 g) was dissolved in boiling methanol, and 2-pyridinecarbaldehyde (0.5 mmol, 0.5 mL) was dropped to the above solution under stirring. Then, the mixture was refluxed for 5 h with vigorous stirring, and white precipitates appeared. The precipitate was filtered, washed 3 times with methanol/water (1:1) and dried over P_2O_5 under vacuum to obtain white powder. Yield: *ca* 85%. The white powder (0.05 mmol, 25.9 mg) was dissolved in methanol/dichloromethane (1:1) (10 mL) under stirring to give a colorless solution. Afterwards, ether (20 mL) was slowly diffused to the above solution in a sealed monotube. After 3 days, colourless rod-like crystals were obtained.

Experimental details

The H atoms were added using riding models. Their U_{iso} values were set to $1.2U_{eq}$ of the parent atoms.

Comment

Rhodamine derivatives have attracted great attention in the field of material and analytical chemistry [6–8], because of the excellent spectroscopic properties such as high fluorescence quantum yield and long wavelength emission. Rhodamine derivatives have been widely investigated as fluorescent probe and fluorescence chemosensors [9–11].

In this paper, we report a rhodamine 6G pyridyl aroylhydrozone compound. The asymmetric unit contains a neutral molecule in a ring-closed form. The amide C=O bond distance is 1.2162(18) Å, indicative of the keto form. The aroylhydrozone plane and the pyridyl plane are nearly coplanar with the dihedral angle of 4.8°. The dihedral angle between the xanthene plane and pyridyl aroylhydrozone group is 84.8°.

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