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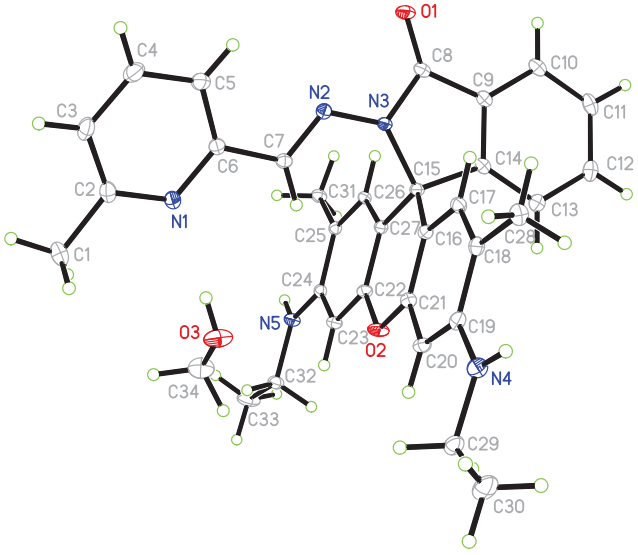
Crystal structure of (*E*)-2-(3,6-bis(ethylamino)-2,7-dimethyl-9*H*-xanthen-9-yl)-*N*′-((6-methylpyridin-2-yl)methylene)benzohydrazide – methanol (1/1), $C_{34}H_{37}N_5O_3$



https://doi.org/10.1515/ncrs-2019-0285
Received April 21, 2019; accepted June 4, 2019; available online July 20, 2019

Abstract

$C_{34}H_{37}N_5O_3$, triclinic, $P\bar{1}$ (no. 2), $a = 8.9446(3)$ Å, $b = 13.4223(4)$ Å, $c = 13.4559(4)$ Å, $\alpha = 83.371(3)°$, $\beta = 71.724(3)°$, $\gamma = 75.383(3)°$, $Z = 2$, $V = 1483.18(9)$ Å^3, $R_{gt}(F) = 0.0383$, $wR_{ref}(F^2) = 0.0981$, $T = 105.6(5)$ K.

CCDC no.: 1920651

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

The title compound was synthesized according to an analogous method reported previously in the literature [5]. Rhodamine 6G hydrozone (1 mmol, 0.428 g) was dissolved in boiling methanol. A methanol solution (20 mL) of 6-methyl-2-pyridinecarboxaldehyde (1.0 mmol, 0.121 g) was added to the above rhodamine 6G hydrozone methanol solution. The mixture was refluxed for 6 h under stirring. After cooling to room temperature, a pale pink precipitate was obtained. The precipitate was filtered and washed with methanol/water (1:1 v/v), and dried over P_2O_5 under vacuum to obtain pale pink powder. Yield: 90%. The pale pink powder (0.05 mmol, 26.6 mg) was dissolved in methanol/dichloromethane (1:1) (10 mL) to give a colorless solution to evaporate at room temperature. After one week, colorless block crystals appeared. Yield: ca. 75%.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.10 × 0.10 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	0.66 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω-scans
θ_{max}, completeness:	71.5°, >94% (up to 76.7°, >97%)
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9338, 5418, 0.014
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5089
$N(param)_{refined}$:	386
Programs:	CrysAlis^{PRO} [1], OLEX2 [2], SHELX [3, 4]

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 not only in the field of analytical chemistry [6–8] but also in the field of coordination chemistry [9, 10], because of the excellent spectroscopic properties and coordination ability to transition metals and rare-earth metals.

The asymmetric unit of the title compound contains a ring-closed 6-methyl-2-pyridinecarbaldehyde rhodamine 6G hydrazone molecule and one methanol solvent molecule. The amide C=O bond distance is 1.2107(15) Å, indicating the keto form. The bond length of C7—N2 is 1.2850(15) Å, which shows that the existence of schiff base C=N [11]. The dihedral angle between the aroylhydrozone plane and the pyridyl plane is 25.48°. While the xanthene least-square plane and pyridyl aroylhydrozone group are almost perpendicular, the dihedral

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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}
O1	0.78386(12)	0.47412(7)	0.54353(7)	0.0266(2)
O2	0.77145(10)	0.76464(6)	0.12868(6)	0.01997(19)
O3	0.44038(13)	0.95999(8)	0.25160(8)	0.0348(2)
H3A	0.4280	0.9445	0.3139	0.052*
N1	0.38453(12)	0.91078(7)	0.46786(8)	0.0182(2)
N2	0.62141(12)	0.65638(7)	0.47149(7)	0.0160(2)
N3	0.74429(12)	0.59006(7)	0.40691(7)	0.0146(2)
N4	0.37058(13)	0.63569(8)	0.05455(8)	0.0215(2)
H4	0.3199	0.5872	0.0634	0.026*
N5	1.16146(12)	0.92098(7)	0.17823(8)	0.0176(2)
H5	1.2300	0.9227	0.2104	0.021*
C1	0.21819(18)	1.08508(10)	0.48125(12)	0.0311(3)
H1A	0.3172	1.1067	0.4475	0.047*
H1B	0.1522	1.1304	0.5366	0.047*
H1C	0.1612	1.0871	0.4311	0.047*
C2	0.25584(15)	0.97695(9)	0.52603(10)	0.0220(3)
C3	0.15981(16)	0.94651(10)	0.62174(10)	0.0265(3)
H3	0.0706	0.9933	0.6605	0.032*
C4	0.19843(17)	0.84564(11)	0.65883(10)	0.0282(3)
H4A	0.1342	0.8238	0.7223	0.034*
C5	0.33309(16)	0.77750(10)	0.60088(10)	0.0233(3)
H5A	0.3626	0.7099	0.6252	0.028*
C6	0.42299(14)	0.81305(9)	0.50518(9)	0.0168(2)
C7	0.56251(14)	0.74536(9)	0.43523(9)	0.0162(2)
H7	0.6072	0.7664	0.3663	0.019*
C8	0.81581(14)	0.49841(9)	0.45108(9)	0.0177(2)
C9	0.93277(14)	0.43968(9)	0.36154(9)	0.0178(2)
C10	1.03258(16)	0.34189(9)	0.36205(10)	0.0242(3)
H10	1.0373	0.3055	0.4245	0.029*
C11	1.12463(16)	0.30063(10)	0.26638(11)	0.0264(3)
H11	1.1937	0.2357	0.2642	0.032*
C12	1.11502(15)	0.35534(10)	0.17296(10)	0.0240(3)
H12	1.1749	0.3252	0.1094	0.029*
C13	1.01753(15)	0.45403(9)	0.17326(9)	0.0194(2)
H13	1.0129	0.4909	0.1110	0.023*
C14	0.92740(14)	0.49584(8)	0.26939(9)	0.0153(2)
C15	0.81318(14)	0.60140(8)	0.29052(8)	0.0144(2)
C16	0.68656(14)	0.61754(8)	0.23389(9)	0.0150(2)
C17	0.58373(14)	0.54987(9)	0.25232(9)	0.0166(2)
H17	0.5864	0.4997	0.3059	0.020*
C18	0.47887(14)	0.55399(9)	0.19499(9)	0.0176(2)
C19	0.47227(14)	0.63214(9)	0.11445(9)	0.0176(2)
C20	0.57027(14)	0.70199(9)	0.09663(9)	0.0176(2)
H20	0.5645	0.7546	0.0456	0.021*
C21	0.67724(14)	0.69304(9)	0.15538(9)	0.0159(2)
C22	0.87978(14)	0.76126(9)	0.18385(9)	0.0153(2)
C23	0.96438(14)	0.83886(9)	0.15551(9)	0.0163(2)
H23	0.9450	0.8878	0.1035	0.020*
C24	1.07831(14)	0.84380(8)	0.20474(8)	0.0146(2)
C25	1.10494(14)	0.76893(8)	0.28505(9)	0.0151(2)
C26	1.01853(14)	0.69316(8)	0.31014(8)	0.0145(2)
H26	1.0367	0.6439	0.3622	0.017*
C27	0.90423(14)	0.68681(8)	0.26084(8)	0.0141(2)
C28	0.37572(15)	0.47716(9)	0.21456(10)	0.0216(3)
H28A	0.2637	0.5119	0.2384	0.032*
H28B	0.4014	0.4268	0.2670	0.032*
H28C	0.3960	0.4436	0.1508	0.032*
C29	0.34590(16)	0.71770(10)	−0.02254(10)	0.0231(3)
H29A	0.4481	0.7195	−0.0753	0.028*
H29B	0.3048	0.7834	0.0110	0.028*
C30	0.22594(18)	0.70018(12)	−0.07400(11)	0.0337(3)
H30A	0.2107	0.7545	−0.1254	0.050*
H30B	0.1243	0.6997	−0.0217	0.050*
H30C	0.2671	0.6352	−0.1073	0.050*
C31	1.22237(15)	0.77448(9)	0.34244(9)	0.0176(2)
H31A	1.1851	0.8371	0.3799	0.026*
H31B	1.3269	0.7734	0.2930	0.026*
H31C	1.2300	0.7165	0.3910	0.026*
C32	1.13707(15)	0.99926(9)	0.09795(9)	0.0197(2)
H32A	1.0250	1.0371	0.1173	0.024*
H32B	1.1602	0.9669	0.0323	0.024*
C33	1.2456(2)	1.07267(11)	0.08399(12)	0.0340(3)
H33A	1.2244	1.1258	0.0326	0.051*
H33B	1.3566	1.0358	0.0610	0.051*
H33C	1.2248	1.1032	0.1495	0.051*
C34	0.55310(18)	1.02285(12)	0.21651(12)	0.0344(3)
H34A	0.5852	1.0289	0.1414	0.052*
H34B	0.6464	0.9925	0.2397	0.052*
H34C	0.5045	1.0900	0.2446	0.052*

angle of them is 85.99°. The hydrogen bond between N1 atom on pyridine and H3a atom on the hydroxyl group of methanol molecule has a length of 2.003 Å.

Acknowledgements: This work was supported by the project of scientific and technological in Henan Province (Project No. 172102310433).

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