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Crystal structure of (*E*)-3',6'-bis(ethylamino)-2-((quinoxalin-2-ylmethylene)amino)spiro[isindoline-1,9'-xanthen]-3-one, $C_{35}H_{32}N_6O_2$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.20 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	φ and ω
$\theta_{\max}$, completeness:	25.0°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7651, 5201, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3198
$N(\text{param})_{\text{refined}}$:	393
Programs:	Bruker [1], SHELX [2, 3]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.61104 (17)	1.04092 (15)	0.37836 (14)	0.0552 (5)
O2	−0.04095 (15)	0.58048 (14)	0.09894 (13)	0.0473 (4)
N1	0.0233 (2)	1.0580 (2)	0.38800 (18)	0.0578 (6)
N2	0.3248 (2)	1.18152 (17)	0.52454 (16)	0.0463 (5)
N3	0.2911 (2)	0.92721 (17)	0.32126 (15)	0.0413 (5)
N4	0.36875 (19)	0.87883 (16)	0.28322 (15)	0.0404 (5)
N5	0.0054 (2)	0.3414 (2)	0.32119 (19)	0.0640 (6)
H5A	0.0620	0.3211	0.3601	0.077*
N6	−0.1451 (2)	0.7916 (2)	−0.13440 (18)	0.0589 (6)
H6A	−0.1143	0.8502	−0.1606	0.071*
C1	0.2344 (3)	1.2219 (2)	0.5523 (2)	0.0456 (6)
C2	0.2932 (3)	1.3266 (2)	0.6509 (2)	0.0632 (8)
H2	0.3931	1.3710	0.6948	0.076*
C3	0.2018 (4)	1.3627 (3)	0.6819 (3)	0.0744 (9)
H3	0.2404	1.4319	0.7473	0.089*
C4	0.0517 (4)	1.2976 (3)	0.6170 (3)	0.0718 (9)
H4	−0.0087	1.3228	0.6404	0.086*
C5	−0.0071 (3)	1.1980 (3)	0.5202 (2)	0.0594 (7)
H5	−0.1070	1.1561	0.4767	0.071*
C6	0.0835 (3)	1.1585 (2)	0.4860 (2)	0.0470 (6)
C7	0.1113 (3)	1.0210 (2)	0.3642 (2)	0.0531 (7)
H7	0.0732	0.9520	0.2987	0.064*
C8	0.2622 (2)	1.0803 (2)	0.43284 (19)	0.0407 (6)
C9	0.3558 (3)	1.0334 (2)	0.40332 (19)	0.0441 (6)
H9	0.4571	1.0784	0.4422	0.053*
C10	0.5176 (3)	0.9374 (2)	0.3064 (2)	0.0427 (6)
C11	0.5340 (2)	0.8499 (2)	0.22454 (18)	0.0401 (6)
C12	0.6620 (3)	0.8628 (2)	0.2135 (2)	0.0487 (6)

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Abstract

$C_{35}H_{32}N_6O_2$, triclinic, $P\bar{1}$ (no. 2), $a = 10.902(2)$ Å, $b = 12.434(2)$ Å, $c = 13.481(4)$ Å, $\alpha = 102.881(5)^\circ$, $\beta = 104.935(5)^\circ$, $\gamma = 114.542(3)^\circ$, $V = 1488.6(6)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0511$, $wR_{\text{ref}}(F^2) = 0.1429$, $T = 296$ K.

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The molecular structure is shown in the Figure (Hydrogen atoms are omitted for Clarity). Table 1 contains crystallographic data, and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H12	0.7533	0.9321	0.2625	0.058*
C13	0.6486 (3)	0.7692 (2)	0.1273 (2)	0.0529 (7)
H13	0.7324	0.7747	0.1179	0.063*
C14	0.5120 (3)	0.6669 (2)	0.0543 (2)	0.0516 (7)
H14	0.5055	0.6058	−0.0043	0.062*
C15	0.3847 (3)	0.6536 (2)	0.0666 (2)	0.0469 (6)
H15	0.2933	0.5841	0.0179	0.056*
C16	0.3981 (2)	0.7468 (2)	0.15363 (19)	0.0394 (6)
C17	0.2771 (2)	0.7532 (2)	0.18607 (18)	0.0379 (5)
C18	0.2031 (2)	0.6464 (2)	0.22364 (18)	0.0368 (5)
C19	0.2840 (3)	0.6225 (2)	0.30556 (19)	0.0457 (6)
H19	0.3855	0.6753	0.3379	0.055*
C20	0.2225 (3)	0.5253 (2)	0.3412 (2)	0.0464 (6)
C21	0.0690 (3)	0.4433 (2)	0.2904 (2)	0.0451 (6)
C22	−0.0137 (3)	0.4656 (2)	0.20948 (19)	0.0426 (6)
H22	−0.1152	0.4129	0.1761	0.051*
C23	0.0542 (2)	0.5662 (2)	0.17795 (18)	0.0367 (5)
C24	0.0172 (2)	0.6780 (2)	0.06275 (18)	0.0377 (5)
C25	−0.0867 (3)	0.6870 (2)	−0.01415 (19)	0.0430 (6)
H25	−0.1860	0.6301	−0.0363	0.052*
C26	−0.0430 (3)	0.7806 (2)	−0.05820 (19)	0.0449 (6)
C27	0.1076 (3)	0.8656 (2)	−0.0234 (2)	0.0470 (6)
C28	0.2059 (3)	0.8520 (2)	0.0520 (2)	0.0456 (6)
H28	0.3054	0.9075	0.0735	0.055*
C29	0.1652 (2)	0.7592 (2)	0.09859 (18)	0.0378 (5)
C30	0.3160 (3)	0.5055 (3)	0.4302 (2)	0.0717 (8)
H30A	0.4171	0.5691	0.4546	0.108*
H30B	0.3042	0.4223	0.4015	0.108*
H30C	0.2866	0.5124	0.4916	0.108*
C31	−0.1498 (3)	0.2675 (3)	0.2920 (3)	0.0738 (9)
H31A	−0.1725	0.1869	0.3007	0.089*
H31B	−0.2018	0.2490	0.2147	0.089*
C32	−0.2020 (4)	0.3341 (3)	0.3607 (3)	0.1016 (13)
H32A	−0.1448	0.3594	0.4376	0.152*
H32B	−0.3034	0.2778	0.3440	0.152*
H32C	−0.1916	0.4083	0.3449	0.152*
C33	−0.3008 (3)	0.7090 (3)	−0.1716 (2)	0.0653 (8)
H33A	−0.3278	0.7123	−0.1083	0.078*
H33B	−0.3268	0.6218	−0.2097	0.078*
C34	−0.3839 (4)	0.7492 (4)	−0.2489 (3)	0.1005 (12)
H34A	−0.3596	0.8349	−0.2106	0.151*
H34B	−0.4875	0.6929	−0.2734	0.151*
H34C	−0.3575	0.7455	−0.3117	0.151*
C35	0.1575 (3)	0.9693 (3)	−0.0690 (2)	0.0675 (8)
H35A	0.2629	1.0167	−0.0394	0.101*
H35B	0.1213	1.0255	−0.0485	0.101*
H35C	0.1203	0.9315	−0.1483	0.101*

product the title compound. Yield: ca. 88%. Single crystals of the title compound were obtained by slow evaporation of its methanol/DMF (1:1) solution at room temperature.

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [3]. All hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Comment

Rhodamine derivatives have attracted much attention in the design of chemosensors or chemodosimeters for metal ions [4]. Particularly, rhodamine carbohydrazones bearing N-containing heterocycle rings, such as pyrrole, pyridine and quinoline, have been proved to be excellent fluorescent probes for Cu²⁺ or Hg²⁺ [5–7]. Herein, we report the crystal structure of the title compound derived from rhodamine 6G hydrazone and 2-formylquinoxaline.

The asymmetric unit contains a neutral molecule in a ring-closed form (see the Figure). The C10=O1 bond distance is 1.217(3) Å, indicating the keto form of the amide. The bond length of C9–N3 is 1.280(3) Å, suggesting the existence of the Schiff base C=N moiety. All bond lengths are in the expected ranges and comparable with those of 2 directly related structures [8, 9]. The dihedral angle between the least-square plane of the benzamide and quinoline is 16.06°, while the torsion angle of N2–C8–C9–N3 is 172.4(2)°, which reveals that it is difficult for title compound to form chelate complexes in this ring closed structure. As expected, there are no classical hydrogen bonds and also no obvious π – π contacts in the solid state.

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Source of material

Rhodmine 6G hydrazide was condensed with equimolar 2-formylquinoxaline in boiling methanol and refluxed for 6 h, during which a white powder appeared, which was filtered off, washed with methanol/water (1:1, v/v) to

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