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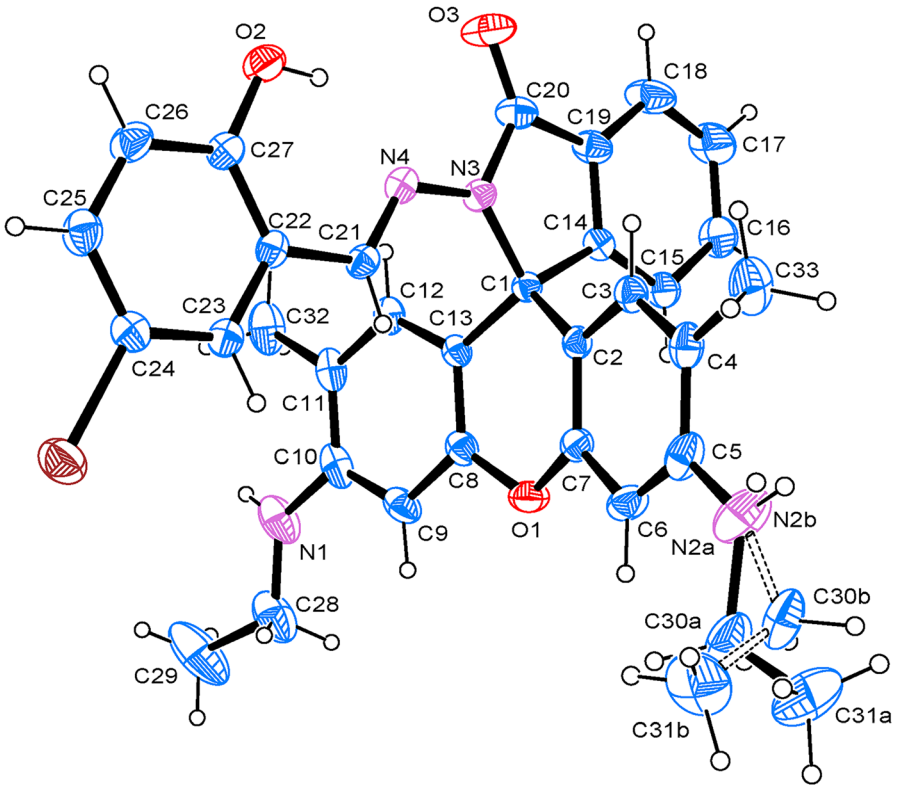
The crystal structure of (*E*)-2-((5-bromo-2-hydroxybenzylidene)amino)-3′,6′-bis(ethylamino)-2′, 7′-dimethylspiro [isoindoline-1,9′-xanthen]-3-one, $C_{33}H_{31}BrN_4O_3$



https://doi.org/10.1515/ncrs-2022-0432
Received August 28, 2022; accepted October 10, 2020; published online October 20, 2022

Abstract

$C_{33}H_{31}BrN_4O_3$, monoclinic, $P2_1/c$ (no. 14), a = 9.3451(7) Å, b = 17.3057(13) Å, c = 18.4402(13) Å, β = 98.4470(10)°, V = 2949.9(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0409, $wR_{ref}(F^2)$ = 0.1106, T = 296(2) K.

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CCDC no.: 2203806

The molecular 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 chemicals were purchased from commercial sources and used as received. A mixture of rhodamine 6G hydrazide (1 mmol), 5-bromo-2-hydroxybenzaldehyde (1 mmol), and CH_3OH (50 mL) was added in a round-bottomed flask (100 mL), and refluxed for 5.0 h. The solution was cooled and filtered. Finally, the title compound was precipitated by controlling solvent volatilization.

Table 1: Data collection and handling.

Crystal:	Yellow acicular
Size:	0.20 × 0.13 × 0.12 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.43 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	15988, 5797, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3968
$N(param)_{refined}$:	399
Programs:	Bruker [1], SHELX [2, 3], WinGX/ORTEP [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.31205 (4)	0.91976 (2)	1.11742 (2)	0.07085 (14)
O1	0.5752 (2)	0.69126 (10)	0.81706 (10)	0.0547 (5)
O2	0.2741 (2)	1.06985 (10)	0.82071 (10)	0.0577 (5)
H2	0.313358	1.043836	0.792112	0.087*
O3	0.3878 (3)	1.00532 (11)	0.63994 (11)	0.0787 (6)
N1	0.0904 (3)	0.60231 (16)	0.79538 (16)	0.0815 (8)
H1	0.002097	0.605472	0.774351	0.098*
N3	0.4413 (2)	0.90494 (10)	0.72138 (10)	0.0389 (5)
N4	0.3947 (2)	0.94480 (11)	0.77698 (11)	0.0407 (5)
C1	0.4998 (2)	0.82461 (12)	0.72406 (12)	0.0345 (5)
C2	0.6392 (2)	0.81804 (13)	0.77737 (11)	0.0361 (5)
C3	0.7439 (2)	0.87569 (14)	0.78466 (12)	0.0429 (6)
H3	0.724964	0.920649	0.757307	0.051*
C4	0.8738 (3)	0.86982 (16)	0.83013 (14)	0.0517 (7)
C5	0.9007 (3)	0.80160 (19)	0.87181 (14)	0.0583 (7)
C6	0.7977 (3)	0.74366 (17)	0.86594 (13)	0.0562 (7)
H6	0.814932	0.698861	0.893707	0.067*
C7	0.6691 (3)	0.75223 (14)	0.81886 (12)	0.0421 (6)
C8	0.4325 (3)	0.70393 (14)	0.78684 (13)	0.0448 (6)
C9	0.3348 (3)	0.64854 (15)	0.80387 (15)	0.0565 (7)
H9	0.367504	0.606577	0.833215	0.068*
C10	0.1895 (3)	0.65577 (16)	0.77727 (15)	0.0582 (8)
C11	0.1418 (3)	0.71854 (16)	0.73117 (15)	0.0538 (7)
C12	0.2432 (3)	0.77179 (15)	0.71618 (13)	0.0458 (6)
H12	0.211785	0.813561	0.686287	0.055*
C13	0.3896 (2)	0.76651 (13)	0.74323 (12)	0.0375 (5)
C14	0.5273 (2)	0.81718 (13)	0.64492 (12)	0.0391 (5)
C15	0.5844 (3)	0.75520 (15)	0.61209 (13)	0.0504 (6)
H15	0.610054	0.709890	0.637875	0.060*
C16	0.6022 (3)	0.76300 (19)	0.53896 (15)	0.0666 (8)
H16	0.641110	0.722053	0.515635	0.080*
C17	0.5642 (4)	0.8293 (2)	0.50021 (16)	0.0827 (10)
H17	0.576815	0.832538	0.451205	0.099*
C18	0.5077 (4)	0.89073 (19)	0.53323 (16)	0.0787 (10)
H18	0.482456	0.936016	0.507322	0.094*
C19	0.4888 (3)	0.88390 (15)	0.60630 (13)	0.0516 (6)
C20	0.4322 (3)	0.94026 (15)	0.65365 (14)	0.0513 (6)
C21	0.4037 (3)	0.91669 (15)	0.84127 (14)	0.0397 (5)
C22	0.3505 (2)	0.96055 (13)	0.89921 (12)	0.0385 (5)
C23	0.3570 (3)	0.92774 (14)	0.96815 (13)	0.0428 (6)
H23	0.398708	0.879237	0.977062	0.051*
C24	0.3024 (3)	0.96589 (15)	1.02368 (13)	0.0484 (6)
C25	0.2414 (3)	1.03789 (16)	1.01127 (15)	0.0560 (7)
H25	0.205221	1.063915	1.048795	0.067*
C26	0.2342 (3)	1.07128 (15)	0.94304 (15)	0.0557 (7)
H26	0.192793	1.119962	0.934924	0.067*
C27	0.2872 (3)	1.03384 (14)	0.88648 (13)	0.0438 (6)
C28	0.1292 (4)	0.5416 (2)	0.8480 (2)	0.0981 (13)
H28A	0.179139	0.563645	0.893144	0.118*
H28B	0.194453	0.505782	0.829151	0.118*
C29	−0.0053 (6)	0.4983 (3)	0.8640 (3)	0.154 (2)
H29A	−0.068668	0.533502	0.883987	0.232*
H29B	0.022915	0.457717	0.898631	0.232*
H29C	−0.054715	0.476473	0.819345	0.232*
C32	−0.0150 (3)	0.7267 (2)	0.69822 (18)	0.0793 (10)
H32A	−0.028001	0.773915	0.670666	0.119*
H32B	−0.073949	0.727756	0.736674	0.119*
H32C	−0.042777	0.683737	0.666354	0.119*
C33	0.9831 (3)	0.9336 (2)	0.83504 (19)	0.0775 (10)
H33A	0.943300	0.976617	0.806015	0.116*
H33B	1.068440	0.915505	0.817057	0.116*
H33C	1.007599	0.949429	0.885216	0.116*
N2A[a]	1.0306 (3)	0.79465 (19)	0.91838 (15)	0.0913 (9)
H2A[a]	1.097608	0.828959	0.920167	0.110*
C30A[a]	1.0480 (9)	0.7206 (7)	0.9675 (6)	0.085 (3)
H30A[a]	0.978192	0.721099	1.001571	0.102*
H30B[a]	1.032084	0.674635	0.937313	0.102*
C31A[a]	1.1983 (9)	0.7210 (5)	1.0082 (5)	0.128 (4)
H31A[a]	1.265446	0.731678	0.974808	0.192*
H31B[a]	1.219639	0.671497	1.030645	0.192*
H31C[a]	1.206410	0.760222	1.045473	0.192*
N2B[b]	1.0306 (3)	0.79465 (19)	0.91838 (15)	0.0913 (9)
H2B[b]	1.070804	0.838165	0.931429	0.110*
C30B[b]	1.1076 (12)	0.7314 (9)	0.9478 (7)	0.078 (4)
H30C[b]	1.211026	0.739507	0.950629	0.094*
H30D[b]	1.080284	0.685019	0.919596	0.094*
C31B[b]	1.0634 (15)	0.7274 (8)	1.0219 (6)	0.117 (5)
H31D[b]	1.086359	0.775278	1.047168	0.176*
H31E[b]	1.114067	0.685908	1.049093	0.176*
H31F[b]	0.961069	0.718321	1.017210	0.176*
H21	0.437 (2)	0.8715 (14)	0.8549 (12)	0.035 (6)*

[a]Occupancy: 0.580 (10), [b]Occupancy: 0.420 (10).

Experimental details

All H-atoms bonded to C atoms were placed geometrically and refined using a riding model with common isotropic displacement factors U_{iso}(H) = 1.2 or 1.5 U_{eq}(parent C-atom).

Comment

Rhodamine derivatives have attracted much attention in the design of chemosensors or chemodosimeters for metal ions [5]. Particularly, rhodamine hydrazones bearing a 2-hydroxybenzaldehyde unit may act as fluorescent probes for Cu^{2+} or Hg^{2+} [6, 7].

The ORTEP diagram is presented in the figure. Bond lengths and angles are all in the expected ranges [8]. It can be seen from Figure 1 that C1, C4, C19, C20, and N4 atoms form a five-membered heterocyclic ring, indicating that the title compound exists in the form of a closed spiro ring. The C=N of the title molecule exhibits an *E* configuration. The bond length of C21=N4 is 1.269(3) Å, which is similar to those reported in the literature [8, 9]. The oxygen atom O2 provides one intramolecular hydrogen bond to N4 (O2···N4 = 2.637(3) Å).

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was supported by the Hunan Provincial Natural Science Foundation of China (No. 2022JJ30096), and College Students Innovation and Entrepreneurship Training Program (No. NYD202211).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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