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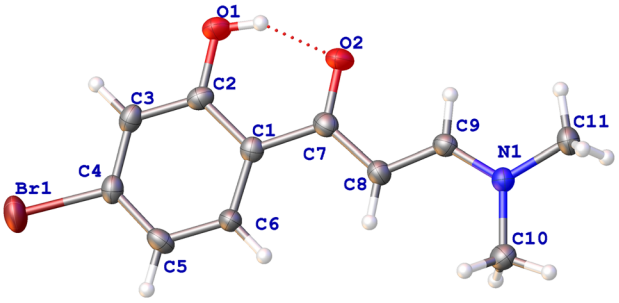
Synthesis and crystal structure (*E*)-1-(4-bromo-2-hydroxyphenyl)-3-(dimethylamino) prop-2-en-1-one, $C_{11}H_{12}BrNO_2$



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Abstract

$C_{11}H_{12}BrNO_2$, monoclinic, $P2_1/n$ (no. 14), a = 7.5143(12) Å, b = 11.8968(15) Å, c = 12.9852(13) Å, β = 105.575(14)°, V = 1118.2(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0607, w$R_{ref}(F^2)$ = 0.1615, T = 293.00(10) K.

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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.

1 Source of material

To a suspension of the (*E*)-1-(4-bromo-2-hydroxyphenyl)-3-(dimethylamino)prop-2-en-1-one (1.0 mmol, 0.86 g), in toluene (4 mL) was added *N*,*N*–dimethylformamide dimethyl acetal (1.05 mL, 8.0 mmol) at 80 °C and stirred for 6 h. Then, the crude product was subjected to flash column chromatography on silica gel (dichloromethane/ethyl acetate = 25:1) to afford the product.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.14 × 0.13 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	3.66 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	25.0°, 98 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	3793, 1923, 0.065
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1392
$N(param)_{refined}$:	140
Programs:	CrysAlis^{PRO} [1], Olex2 [2], SHELX [3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.60408 (9)	0.34817 (6)	0.02383 (4)	0.0722 (5)
O1	0.5602 (6)	0.1999 (3)	0.3877 (3)	0.0593 (11)
H1	0.573260	0.216583	0.452123	0.089*
O2	0.6404 (6)	0.3130 (3)	0.5522 (3)	0.0612 (11)
N1	0.8419 (5)	0.5746 (3)	0.7461 (3)	0.0395 (10)
C1	0.6671 (6)	0.3910 (4)	0.3899 (4)	0.0367 (11)
C2	0.6031 (6)	0.2888 (4)	0.3360 (4)	0.0400 (11)
C3	0.5835 (6)	0.2786 (4)	0.2271 (4)	0.0439 (12)
H3	0.541040	0.210133	0.191096	0.053*
C4	0.6261 (7)	0.3684 (4)	0.1721 (4)	0.0430 (12)
C5	0.6860 (7)	0.4690 (4)	0.2204 (4)	0.0458 (12)
H5	0.713781	0.530146	0.180306	0.055*
C6	0.7050 (7)	0.4794 (4)	0.3291 (4)	0.0418 (12)
H6	0.745121	0.549111	0.363050	0.050*
C7	0.6883 (7)	0.3972 (4)	0.5070 (4)	0.0416 (12)
C8	0.7604 (6)	0.4951 (4)	0.5669 (4)	0.0411 (11)
H8	0.792463	0.559849	0.533003	0.049*
C9	0.7819 (6)	0.4929 (4)	0.6753 (4)	0.0386 (11)
H9	0.749958	0.424307	0.703253	0.046*
C10	0.8916 (7)	0.6846 (4)	0.7133 (4)	0.0484 (13)
H10A	0.985486	0.675882	0.674109	0.073*
H10B	0.940964	0.731393	0.776673	0.073*
H10C	0.781734	0.720705	0.666994	0.073*
C11	0.8584 (7)	0.5556 (5)	0.8589 (4)	0.0495 (13)
H11A	0.777038	0.607759	0.883121	0.074*
H11B	0.986570	0.568283	0.900231	0.074*
H11C	0.822765	0.478129	0.869390	0.074*

2 Comment

Ketoamine compounds have good application potential in the field of organic synthesis, are intermediates [4, 5] used in many compounds, and have anti-inflammatory [6] and other pharmacological effects, therefore, the synthesis of arylketoamine has been a hot topic.

The title compound consisted of a basic structure similar to dopamine and a bromine atom, and a dimethylaminoacetone group. The bond lengths and angles derived from the title structure are within normal ranges and consistent with those reported previously in similar structures [7–9]. The C=C bond was determined at the distance of 1.372 (6) Å (C8–C9). The keto group was confirmed by the distance of 1.262(6) Å (C7–O2), and the alcohol hydroxyl group was identified by the distance of 1.338(6) Å (C2–O1), respectively. The bromine atom was determined at the distance of 1.902(5) Å (Br1–C4).

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

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