Qin-Juan Xu*

Crystal structure of *N*-(5-(2-(benzyl(1-(4-methoxyphenyl)propan-2-yl)amino)-1-hydroxyethyl)-2-(benzyloxy)phenyl)formamide, $C_{33}H_{36}N_2O_4$

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Abstract

$C_{33}H_{36}N_2O_4$, orthorhombic, *Pbca* (no. 61),
 $a = 15.3543(17)$ Å, $b = 14.7958(17)$ Å, $c = 25.496(3)$ Å,
 $V = 5792.1(11)$ Å³, $Z = 8$, $R_{gt}(F) = 0.0490$, $wR_{ref}(F^2) = 0.1402$,
 $T = 296(2)$ 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.

Source of material

The title compound was prepared by the reaction of 1-(3-amino-4-(benzyloxy)phenyl)-2-(benzyl(1-(4-methoxy

Table 1: Data collection and handling.

Crystal:	Colorless sheet
Size:	0.23 × 0.19 × 0.05 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	φ and ω
θ_{max} , completeness:	27.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	26962, 6305, 0.028
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4395
$N(param)_{refined}$:	364
Programs:	Bruker [1], SHELX [2], Diamond [3]

-phenyl)propan-2-yl)amino)ethan-1-ol (R1) with formic acid protected by N_2 . 6.5 g R1 was dissolved in 30 mL $ClCH_2CH_2Cl$. 4.49 g $EtN=C=N(CH_2)_3NMe_2$ hydrochloric acid salt was dissolved in 25 mL $ClCH_2CH_2Cl$ and mixed with the former solution cooled to 10 °C. Then, 1.2 g anhydrous formic acid was added slowly and reacted for 2 h. After that, the organic phase was washed with 10% sodium carbonate hydrous solution and 20% sodium chloride hydrous solution for twice, respectively. The organic phase was concentrated at 40–50 °C to give rise to dark-red stickiness substance (yield 68%). This method was referenced to a patent [4]. The colorless sheet-shaped crystals suitable for X-ray diffraction of the title compound were obtained by slow evaporation of a MeOH solution.

*Corresponding author: Qin-Juan Xu, Weihai Ocean Vocational College, Rongcheng, Shandong 264300, P. R. China, E-mail: 15662370667@163.com. <https://orcid.org/0000-0002-2910-209X>

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
N1	0.46246 (8)	0.12243 (8)	0.83595 (5)	0.0406 (3)
N2	0.09567 (9)	0.27618 (9)	0.98248 (5)	0.0464 (3)
O1	0.10064 (8)	0.14121 (8)	1.04744 (5)	0.0613 (3)
O2	0.87256 (10)	0.16534 (14)	0.87061 (8)	0.1029 (6)
O3	0.13010 (9)	0.39210 (10)	0.92729 (6)	0.0751 (4)
O4	0.46809 (7)	0.18781 (9)	0.93622 (5)	0.0563 (3)
C1	0.38441 (9)	0.11814 (11)	0.86881 (6)	0.0418 (3)
H1A	0.3813	0.0595	0.8857	0.050*
H1B	0.3331	0.1252	0.8470	0.050*
C2	0.38579 (9)	0.19203 (10)	0.91047 (6)	0.0426 (3)
H2B	0.3795	0.2511	0.8935	0.051*
C3	0.49060 (11)	0.03357 (10)	0.81525 (6)	0.0479 (4)
H3A	0.5270	0.0459	0.7845	0.058*
C4	0.54882 (12)	−0.01433 (12)	0.85524 (7)	0.0599 (5)
H4B	0.5191	−0.0161	0.8888	0.072*
H4C	0.5580	−0.0762	0.8440	0.072*
C5	0.45420 (10)	0.19040 (11)	0.79426 (6)	0.0500 (4)
H5A	0.4279	0.2445	0.8087	0.060*
H5B	0.4156	0.1674	0.7673	0.060*
C6	0.41669 (13)	−0.02737 (13)	0.79671 (7)	0.0641 (5)
H6A	0.3817	0.0045	0.7715	0.096*
H6B	0.3813	−0.0443	0.8261	0.096*
H6C	0.4405	−0.0807	0.7808	0.096*
C7	0.31346 (9)	0.17943 (10)	0.94947 (6)	0.0418 (3)
C8	0.24075 (9)	0.23555 (10)	0.94766 (5)	0.0402 (3)
H8A	0.2390	0.2828	0.9237	0.048*
C9	0.17098 (9)	0.22176 (10)	0.98123 (5)	0.0400 (3)
C10	0.17362 (10)	0.15047 (11)	1.01712 (6)	0.0464 (4)
C11	0.24661 (11)	0.09621 (12)	1.01994 (6)	0.0548 (4)
H11A	0.2493	0.0499	1.0445	0.066*
C12	0.31582 (10)	0.11084 (11)	0.98608 (6)	0.0511 (4)
H12A	0.3646	0.0738	0.9881	0.061*
C13	0.09412 (12)	0.06297 (13)	1.08000 (7)	0.0603 (5)
H13A	0.1346	0.0676	1.1091	0.072*
H13B	0.1085	0.0093	1.0600	0.072*
C14	0.08184 (12)	0.35480 (12)	0.95767 (7)	0.0527 (4)
C15	0.00311 (11)	0.05635 (12)	1.10022 (7)	0.0547 (4)
C16	−0.06654 (16)	0.0518 (2)	1.06691 (10)	0.0966 (8)
H16A	−0.0574	0.0550	1.0309	0.116*
C17	−0.15039 (18)	0.0426 (2)	1.08605 (18)	0.1218 (12)
H17A	−0.1972	0.0378	1.0630	0.146*
C18	−0.1640 (2)	0.0407 (2)	1.13904 (19)	0.1232 (13)
H18A	−0.2201	0.0344	1.1523	0.148*
C19	−0.0951 (2)	0.0480 (3)	1.17226 (14)	0.1300 (13)
H19A	−0.1045	0.0480	1.2083	0.156*
C20	−0.01182 (15)	0.05523 (18)	1.15302 (9)	0.0873 (7)
H20A	0.0348	0.0594	1.1762	0.105*
C21	0.54001 (12)	0.21462 (11)	0.76982 (7)	0.0564 (4)
C22	0.55370 (18)	0.20232 (16)	0.71676 (9)	0.0865 (7)
H22A	0.5097	0.1784	0.6959	0.104*
C23	0.6331 (3)	0.2257 (2)	0.69471 (15)	0.1281 (14)
H23A	0.6420	0.2166	0.6590	0.154*
C24	0.6976 (2)	0.2613 (2)	0.7238 (2)	0.1343 (16)
H24A	0.7501	0.2778	0.7083	0.161*

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C25	0.68524 (16)	0.27284 (19)	0.77621 (17)	0.1163 (12)
H25A	0.7299	0.2964	0.7967	0.140*
C26	0.60637 (13)	0.24967 (15)	0.79948 (10)	0.0787 (6)
H26A	0.5986	0.2580	0.8353	0.094*
C27	0.63538 (11)	0.03058 (11)	0.86225 (7)	0.0525 (4)
C28	0.65541 (13)	0.08140 (13)	0.90562 (7)	0.0608 (5)
H28A	0.6151	0.0849	0.9328	0.073*
C29	0.73312 (14)	0.12755 (14)	0.91056 (7)	0.0678 (5)
H29A	0.7444	0.1615	0.9405	0.081*
C30	0.79341 (12)	0.12296 (14)	0.87107 (8)	0.0668 (5)
C31	0.77609 (13)	0.07094 (15)	0.82747 (8)	0.0696 (5)
H31A	0.8172	0.0663	0.8008	0.084*
C32	0.69846 (12)	0.02608 (14)	0.82328 (7)	0.0631 (5)
H32A	0.6876	−0.0084	0.7935	0.076*
C33	0.8949 (2)	0.2210 (3)	0.91319 (16)	0.1465 (14)
H33A	0.9522	0.2453	0.9079	0.220*
H33B	0.8940	0.1861	0.9449	0.220*
H33C	0.8537	0.2695	0.9159	0.220*
H1	0.0248 (12)	0.3816 (12)	0.9669 (6)	0.056 (5)*
H4	0.5056 (16)	0.1731 (16)	0.9129 (9)	0.092 (8)*
H2	0.0570 (12)	0.2609 (13)	1.0066 (8)	0.065 (6)*

Experimental details

The H atoms were calculated geometrically and refined as riding, with C–H = 0.93–0.98 Å with *U*_{iso}(H) = 1.2 *U*_{eq} (parent atom).

Comment

N-(5-(2-(Benzyl(1-(4-methoxyphenyl)propan-2-yl)amino)-1-hydroxyethyl)-2-(benzyloxy)phenyl)formamide is an important precursor to prepare Formoterol, which is a widely used pharmaceutical active compound [5–7]. Herein, we report the crystal structure of the title compound. In the title compound, all the bond lengths are within normal ranges [8–10]. The molecules of the title compound are interlinked to form 3D supramolecular networks by two classic intermolecular H-bondings O4–H4–N1 (length 2.735(2) Å, angle 120.18(4)°) and N2–H2–O4 (*x* − 1/2, −*y* + 1/2, −*z* + 2) (length 2.910(3) Å, angle 144.42(19)°).

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