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The crystal structure of (Z)-3-((2-(2-(2-aminophenoxy)ethoxy)phenyl)amino)-1-phenylbut-2-en-1-one, $C_{24}H_{24}N_2O_3$

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Abstract

$C_{24}H_{24}N_2O_3$, monoclinic, $P2_1/n$ (no. 14), $a = 13.674(3)$ Å, $b = 7.3900(14)$ Å, $c = 20.862(4)$ Å, $\beta = 106.728(7)^\circ$, $V = 2018.9(7)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0633$, $wR_{ref}(F^2) = 0.1804$, $T = 293$ 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.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.23 × 0.21 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	Bruker Apex-II, φ and ω
θ_{max} , completeness:	25.0°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	13143, 3530, 0.062
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1844
$N(param)_{refined}$:	264
Programs:	Bruker [1], SHELX [2], Olex2 [3]

Source of material

To the solution of 2,2'-(ethane-1,2-diylbis(oxy))dianiline (0.244 g 1 mmol) in ethanol (20 ml) was added the solution of benzoylacetone 0.162 g (1 mmol) in ethanol (20 ml) dropwisely. The mixture was refluxed for 8 h then cooled and filtered to get a white solid. The solid was washed with ethanol for three times, dried and purified by column chromatography (the developing agent was dichloromethane: Petroleum ether = 1:1) to get (Z)-3-((2-(2-(2-aminophenoxy)ethoxy)phenyl)amino)-1-phenylbut-2-en-1-one (0.194 g, the yield 50%). The single crystal suitable for X-ray diffraction was obtained by recrystallization in diethyl ether.

Experimental details

The data were scaled and corrected for absorption using SADABS-2016/2 (Bruker, APEX-II CCD). The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Comment

α,β -Unsaturated ketones [4–5] are highly appreciated because they have shown promising biological activities such as anti-inflammatory [6–7], antimicrobial [8],

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.6986 (2)	0.4610 (4)	0.33550 (15)	0.0508 (8)
C2	0.7324 (3)	0.4662 (4)	0.40478 (16)	0.0614 (9)
H2	0.6850	0.4717	0.4289	0.074*
C3	0.8348 (3)	0.4635 (5)	0.43842 (17)	0.0703 (10)
H3	0.8558	0.4685	0.4849	0.084*
C4	0.9066 (3)	0.4535 (5)	0.40386 (18)	0.0737 (10)
H4	0.9758	0.4515	0.4269	0.088*
C5	0.8746 (2)	0.4462 (5)	0.33398 (17)	0.0655 (10)
H5	0.9222	0.4378	0.3100	0.079*
C6	0.7722 (2)	0.4516 (4)	0.30107 (15)	0.0516 (8)
C7	0.7976 (2)	0.4539 (5)	0.19064 (14)	0.0549 (8)
H7A	0.8390	0.3451	0.1967	0.066*
H7B	0.8423	0.5586	0.2002	0.066*
C8	0.7259 (2)	0.4633 (4)	0.12108 (14)	0.0537 (8)
H8A	0.6749	0.3685	0.1145	0.064*
H8B	0.6913	0.5794	0.1138	0.064*
C9	0.7329 (2)	0.4139 (4)	0.00945 (15)	0.0508 (8)
C10	0.6318 (2)	0.4615 (5)	−0.01889 (16)	0.0598 (9)
H10	0.5954	0.5170	0.0070	0.072*
C11	0.5851 (2)	0.4265 (5)	−0.08572 (17)	0.0652 (10)
H11	0.5171	0.4585	−0.1046	0.078*
C12	0.6382 (2)	0.3447 (4)	−0.12453 (16)	0.0632 (9)
H12	0.6059	0.3206	−0.1694	0.076*
C13	0.7390 (2)	0.2982 (4)	−0.09730 (15)	0.0557 (8)
H13	0.7743	0.2425	−0.1239	0.067*
C14	0.7884 (2)	0.3334 (4)	−0.03082 (15)	0.0459 (7)
C15	0.9706 (2)	0.3223 (4)	−0.08844 (14)	0.0567 (9)
H15A	1.0372	0.3589	−0.0901	0.085*
H15B	0.9231	0.4193	−0.1041	0.085*
H15C	0.9491	0.2180	−0.1164	0.085*
C16	0.9745 (2)	0.2762 (4)	−0.01741 (14)	0.0445 (7)
C17	1.0673 (2)	0.2308 (4)	0.02690 (14)	0.0483 (8)
H17	1.1241	0.2295	0.0109	0.058*
C18	1.0824 (2)	0.1860 (4)	0.09499 (15)	0.0470 (8)
C19	1.1877 (2)	0.1466 (4)	0.14006 (15)	0.0465 (8)
C20	1.2758 (2)	0.1954 (4)	0.12423 (16)	0.0588 (9)
H20	1.2710	0.2508	0.0834	0.071*
C21	1.3707 (3)	0.1622 (5)	0.16862 (18)	0.0717 (10)
H21	1.4294	0.1960	0.1577	0.086*
C22	1.3785 (3)	0.0790 (5)	0.22905 (18)	0.0719 (10)
H22	1.4423	0.0571	0.2590	0.086*
C23	1.2914 (3)	0.0287 (4)	0.24488 (16)	0.0622 (9)
H23	1.2965	−0.0286	0.2854	0.075*
C24	1.1973 (3)	0.0624 (4)	0.20133 (15)	0.0550 (8)
H24	1.1390	0.0287	0.2128	0.066*
N1	0.5959 (2)	0.4638 (4)	0.29936 (14)	0.0720 (9)
H1A	0.5756	0.3541	0.2902	0.086*
H1B	0.5613	0.5132	0.3232	0.086*
N2	0.89046 (18)	0.2845 (3)	0.00320 (12)	0.0500 (7)
H2A	0.9001	0.2550	0.0444	0.060*
O1	0.73090 (15)	0.4513 (3)	0.23229 (10)	0.0643 (7)
O2	0.78584 (14)	0.4406 (3)	0.07565 (9)	0.0585 (6)
O3	1.00963 (16)	0.1834 (3)	0.12094 (10)	0.0664 (7)

anti-parasitic [9], antibacterial [10], antimalarial [11], antitumor [12], anti-nematodal [13], antioxidant [14] and anti-tubulin [15] activities. The synthesis and activity research of the novel α,β -unsaturated ketones have drawn more and more attentions in recent decades.

This contribution is part of a study on amine-based organic molecules [16]. The title compound (*Z*)-3-((2-(2-aminophenoxy)ethoxy)phenyl)amino)-1-phenylbut-2-en-1-one was synthesized by condensation reaction using 2, 2'-(ethane-1,2-diylbis(oxy))dianiline as the starting material. There is one molecule in the asymmetric unit (see the figure). The angles of O3–C18–C19, C16–N2–C14 and C6–O1–C7 are 117.4(3), 131.5(2) and 119.3(2)° respectively, which are in normal range. Hydrogen bonds were observed as following: N1–H1B...O3 hydrogen bond ($d(\text{H1B}\cdots\text{O3}) = 2.13 \text{ \AA}$, N2–H2A...O2 hydrogen bond ($d(\text{H2A}\cdots\text{O2}) = 2.31 \text{ \AA}$, N2–H2A...O3 hydrogen bond ($d(\text{H2A}\cdots\text{O3}) = 1.92 \text{ \AA}$).

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

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