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Crystal structure of 1-(3-chlorophenyl)-4-(4-(((2,3-dihydro-1*H*-inden-5-yl)oxy)methyl)phenethyl)piperazine, C₂₈H₃₁ClN₂O

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Abstract

C₂₈H₃₁ClN₂O, monoclinic, *P* $\bar{1}$ (no. 2), *a* = 21.9309(11) Å, *b* = 9.9648(5) Å, *c* = 11.0049(7) Å, β = 93.403(6)°, *V* = 2400.7(2) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0566, *wR*_{ref}(*F*²) = 0.1355, *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.

Source of material

The title compound was synthesized from 4-(((2,3-dihydro-1*H*-inden-5-yl)oxy)methyl)phenethyl 4-methylbenzenesulfonate in form of light yellow crystals. To a solution of 4-(((2,3-dihydro-1*H*-inden-5-yl)oxy)methyl)phenethyl 4-methylbenzenesulfonate (100 mg, 0.23 mmol) in acetonitrile (CH₃CN, 10 mL) was added 1-(3-chlorophenyl)piperazine (54 mg, 0.27 mmol) and potassium carbonate (190 mg,

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.18 × 0.15 × 0.13 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.18 mm ^{−1}
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	25.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	12758, 4213, 0.028
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 2973
<i>N</i> (<i>param</i>) _{refined} :	289
Programs:	SHELX [1], Olex2 [2], CrysAlis ^{PRO} [3]

1.38 mmol). The reaction mixture was stirred at reflux for 16 h. The mixture was filtered before cooling to ambient. The filtrate was concentrated *in vacuo* and the residue was purified by silica gel column chromatography using ethyl acetate/petroleum ether (1/15, v/v) as eluent to obtain the product as a light yellow solid. Yield: 71%; **Mp.** 83–84 °C (HCl salt); ¹H NMR (500 MHz, CDCl₃) δ [ppm] 7.40 (d, *J* = 7.9 Hz, 2H), 7.28 (d, *J* = 2.3 Hz, 1H), 7.20 (t, *J* = 8.1 Hz, 1H), 7.15 (d, *J* = 8.2 Hz, 1H), 6.92 (t, *J* = 2.0 Hz, 1H), 6.90 (s, 1H), 6.86–6.79 (m, 3H), 5.04 (s, 2H), 3.35–3.18 (m, 4H), 2.99–2.81 (m, 6H), 2.71 (dd, *J* = 16.0, 7.0 Hz, 6H), 2.16–2.05 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ [ppm] 157.81, 152.29, 145.75, 139.72, 136.48, 135.29, 134.99, 130.05, 128.92, 127.74, 124.76, 119.36, 115.81, 113.91, 112.84, 110.94, 70.10, 60.30, 53.00, 48.62, 33.21, 32.02, 29.73, 25.86; **HRMS** (ESI) *m/z* [*M* + 1]⁺: calcd. for C₂₈H₃₁ClN₂O, 447.2198, found, 447.2196.

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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}
Cl1	0.95801(3)	0.13164(8)	0.32362(9)	0.0820(3)
O1	0.30527(8)	0.36018(18)	0.92112(17)	0.0575(5)
N1	0.75550(8)	0.3461(2)	0.42215(18)	0.0448(5)
N2	0.65779(9)	0.3809(2)	0.58260(18)	0.0482(5)
C1	0.05755(13)	0.1458(4)	1.0455(3)	0.0779(10)
H1A	0.0207	0.1734	0.9990	0.094*
H1B	0.0481	0.0677	1.0934	0.094*
C2	0.10706(12)	0.1124(3)	0.9606(3)	0.0633(8)
H2A	0.1223	0.0222	0.9755	0.076*
H2B	0.0916	0.1191	0.8764	0.076*
C3	0.15654(11)	0.2140(2)	0.9881(2)	0.0471(6)
C4	0.21119(11)	0.2312(3)	0.9320(2)	0.0497(6)
H4	0.2211	0.1751	0.8686	0.060*
C5	0.25040(11)	0.3321(3)	0.9713(2)	0.0475(6)
C6	0.23540(11)	0.4163(3)	1.0657(2)	0.0502(7)
H6	0.2619	0.4847	1.0916	0.060*
C7	0.18104(12)	0.3982(3)	1.1210(2)	0.0529(7)
H7	0.1711	0.4544	1.1842	0.063*
C8	0.14149(11)	0.2969(3)	1.0826(2)	0.0498(6)
C9	0.08040(13)	0.2580(3)	1.1277(3)	0.0719(9)
H9A	0.0848	0.2279	1.2116	0.086*
H9B	0.0524	0.3334	1.1225	0.086*
C10	0.32434(11)	0.2709(3)	0.8299(2)	0.0533(7)
H10A	0.2936	0.2676	0.7628	0.064*
H10B	0.3292	0.1813	0.8633	0.064*
C11	0.38385(11)	0.3184(2)	0.7847(2)	0.0463(6)
C12	0.40245(11)	0.2679(3)	0.6751(2)	0.0508(7)
H12	0.3778	0.2068	0.6313	0.061*
C13	0.45739(11)	0.3079(3)	0.6307(2)	0.0525(7)
H13	0.4690	0.2723	0.5574	0.063*
C14	0.49538(11)	0.3990(3)	0.6920(3)	0.0507(7)
C15	0.47636(12)	0.4489(3)	0.8006(3)	0.0602(8)
H15	0.5008	0.5107	0.8441	0.072*
C16	0.42141(12)	0.4086(3)	0.8465(3)	0.0591(7)
H16	0.4100	0.4434	0.9202	0.071*
C17	0.55386(12)	0.4451(3)	0.6399(3)	0.0621(8)
H17A	0.5448	0.4760	0.5572	0.074*
H17B	0.5699	0.5208	0.6871	0.074*
C18	0.60224(11)	0.3379(3)	0.6388(3)	0.0596(8)
H18A	0.6128	0.3104	0.7219	0.072*
H18B	0.5855	0.2604	0.5951	0.072*
C19	0.70695(11)	0.2854(3)	0.6097(3)	0.0619(8)
H19A	0.6950	0.1979	0.5781	0.074*
H19B	0.7142	0.2776	0.6972	0.074*
C20	0.76509(11)	0.3295(3)	0.5540(2)	0.0571(7)
H20A	0.7787	0.4139	0.5903	0.069*
H20B	0.7968	0.2632	0.5715	0.069*
C21	0.70578(10)	0.4401(3)	0.3939(2)	0.0470(6)
H21A	0.6983	0.4467	0.3064	0.056*
H21B	0.7171	0.5284	0.4248	0.056*
C22	0.64837(11)	0.3936(3)	0.4506(2)	0.0495(6)
H22A	0.6158	0.4574	0.4315	0.059*
H22B	0.6360	0.3076	0.4162	0.059*
C23	0.80744(10)	0.3531(2)	0.3537(2)	0.0426(6)
C24	0.85340(11)	0.2564(2)	0.3714(2)	0.0469(6)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H24	0.8505	0.1916	0.4316	0.056*
C25	0.90288(11)	0.2568(3)	0.3000(2)	0.0501(7)
C26	0.90951(12)	0.3506(3)	0.2096(3)	0.0559(7)
H26	0.9431	0.3495	0.1619	0.067*
C27	0.86455(12)	0.4456(3)	0.1928(2)	0.0555(7)
H27	0.8680	0.5102	0.1325	0.067*
C28	0.81439(11)	0.4480(3)	0.2628(2)	0.0479(6)
H28	0.7848	0.5140	0.2490	0.057*

Experimental details

The hydrogen atoms were placed in calculated positions and refined using a riding model on attached atoms with isotropic thermal parameters 1.2 times those of their carrier atoms.

Comment

Arylpiperazines are great important kinds of drug molecules, and have various bioactivities such as antiarrhythmic, diuretic, antiallergic, antidepressant, anxiolytic, antipsychotic, antimalarial, and antiplasmodial [4–12]. Some of these compounds have good receptor-blocking properties [13]. According to experiments of the testprostate cancer cells, arylpiperazine derivatives have displayed significant cytotoxic [14]. Although some crystal structures of arylpiperazine derivatives have been reported recently [15, 16], the different functional groups and organic ligands would lead to different properties. Therefore, to further study and explore the new arylpiperazine, we report the crystal structure of the title compound.

The molecule of the title compound displays the 2,3-dihydro-1*H*-indene, piperazine ring, and two benzene ring groups. One nitrogen atom of piperazine connects the phenylethyl moiety via C–N bond, and the other N links to the carbon atom of chlorobenzene. Furthermore, the above mentioned phenylethyl extends connection of 2,3-dihydro-1*H*-indene group a C–O bond (see the figure). The 2,3-dihydro-1*H*-indene group, piperazine unit, and two aryl moieties are not in the same plane. The whole molecule displays a twisty conformation. In the molecule, the Cl1–C25, O1–C10, O1–C5, N1–C20, N1–C23, N1–C21, N2–C22, N2–C19, and N2–C18 bond lengths are found to be 1.746(3) Å, 1.423(3) Å, 1.382(3) Å, 1.464(3) Å, 1.404(3) Å, 1.457(3) Å, 1.461(3) Å, 1.455(3) Å, and 1.463(3) Å, respectively, which are nearly equal to other typical single bonds. The bond angles C5–O1–C10, C23–N1–C20, C23–N1–C21, C21–N1–C20, C22–N2–C18, C19–N2–C22, C19–N2–C18, N2–C22–C21, N1–C20–C19, C24–C23–N1, C28–C23–N1, N1–C21–C22, C24–C25–Cl1, C26–C25–Cl1, N2–C19–C20,

N2—C18—C17, O1—C10—C11, O1—C5—C6, 115.3(2)°, and C4—C5—O1 are 117.05(19)°, 117.67(19)°, 118.31(19)°, 110.22(19)°, 112.0(2)°, 108.7(2)°, 110.32(19)°, 111.4(2)°, 111.1(2)°, 119.4(2)°, 123.1(2)°, 110.3(2)°, 118.4(2)°, 119.32(19)°, 111.0(2)°, 113.6(2)°, 109.6(2)°, and 115.3(2)°, respectively.

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