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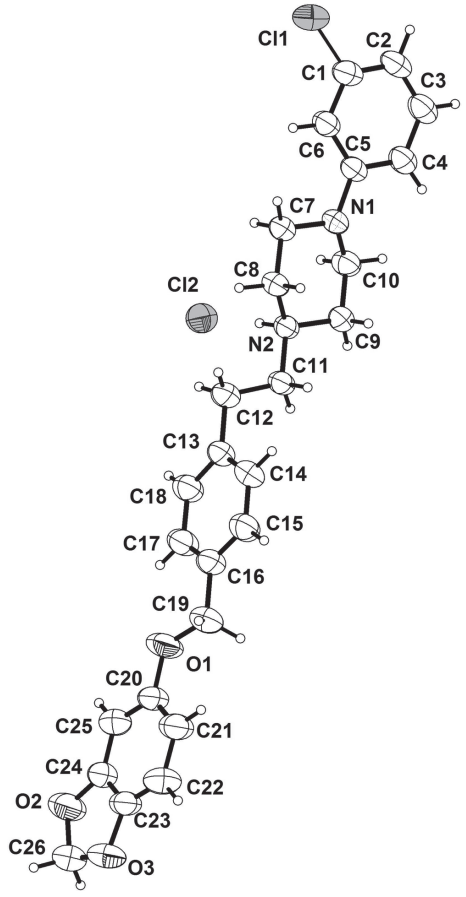
# Crystal structure of 1-(4-((benzo[*d*][1,3]dioxol-5-yloxy)methyl)phenethyl)-4-(3-chlorophenyl) piperazin-1-ium chloride, $C_{26}H_{28}Cl_2N_2O_3$

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## Abstract

$C_{26}H_{28}Cl_2N_2O_3$, monoclinic, $P2_1/c$ (no. 14), $a = 13.422(3)$ Å, $b = 7.0011(14)$ Å, $c = 26.249(5)$ Å, $\beta = 101.06(3)°$, $V = 2420.8(9)$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.0516$, $wR_{ref}(F^2) = 0.1370$, $T = 296$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Block, colorless |
| Size: | 0.30 × 0.10 × 0.10 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 9.5 mm$^{-1}$ |
| Diffractometer, scan mode: | Bruker P4, $\omega$-scans |
| $2\theta_{max}$, completeness: | 68.2°, >98% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 39188, 4439, 0.089 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 3123 |
| $N(param)_{refined}$: | 299 |
| Programs: | Bruker programs [1], SHELX [2] |

## Source of material

The title compound was synthesized from 2-(4-((benzo[*d*][1,3]dioxol-5-yloxy)methyl)phenyl)ethyl 4-methylbenzenesulfonate (65% yield) in form of colorless crystals. To a solution of 2-(4-((benzo[*d*][1,3]dioxol-5-yloxy)methyl)phenyl)ethyl 4-methylbenzenesulfonate (100 mg, 0.23 mmol) in acetonitrile ($CH_3CN$, 10 mL) was added 1-(3-chlorophenyl)piperazine (1.2 equiv) and potassium carbonate (6.0 equiv). The reaction mixture was stirred at reflux for 16 h. After cooling to ambient temperature, the reaction mixture was filtered through a Büchner funnel. After filtration the filtrate was concentrated in vacuo and the residue was purified by silica gel column chromatography using ethyl acetate/petroleum ether (1/5, v/v) as eluent to afford the product, and the title compound was recrystallized from trichloromethane and *n*-hexane [3].

## Experimental details

The hydrogen atoms were assigned with isotropic displacement factors $U_{iso}(H) = 1.2U_{eq}$ (N and imidazol C), or $U_{iso}(H) = 1.5U_{eq}$ (methyl C) and included in the final refinements by using geometrical restraints, with C—H = 0.93 Å (imidazol) or C–H = 0.96 Å (methyl), and N—H = 0.86 Å.

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

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.74454(6) | 0.60009(15) | 0.80257(3) | 0.0877(3) |
| Cl2 | 0.36791(6) | 0.23715(10) | 0.52937(3) | 0.0672(2) |
| O1 | 0.10602(19) | 0.8289(4) | 0.22125(8) | 0.0908(8) |
| O2 | −0.0435(2) | 0.6095(4) | 0.05155(8) | 0.0959(8) |
| O3 | −0.09168(18) | 0.9128(4) | 0.02059(8) | 0.0864(7) |
| N1 | 0.42623(15) | 0.6717(3) | 0.65995(7) | 0.0493(5) |
| N2 | 0.34226(14) | 0.6588(3) | 0.55024(7) | 0.0452(5) |
| H2 | 0.3504 | 0.5216 | 0.5449 | 0.054* |
| C1 | 0.6136(2) | 0.6255(4) | 0.78260(10) | 0.0577(7) |
| C2 | 0.5547(2) | 0.6326(4) | 0.81984(10) | 0.0603(7) |
| H2A | 0.5833 | 0.6298 | 0.8550 | 0.072* |
| C3 | 0.4514(2) | 0.6440(4) | 0.80275(10) | 0.0638(8) |
| H3 | 0.4092 | 0.6457 | 0.8270 | 0.077* |
| C4 | 0.4087(2) | 0.6530(4) | 0.75071(10) | 0.0574(7) |
| H4 | 0.3386 | 0.6613 | 0.7405 | 0.069* |
| C5 | 0.4695(2) | 0.6498(4) | 0.71292(9) | 0.0473(6) |
| C6 | 0.5737(2) | 0.6333(4) | 0.73020(9) | 0.0551(7) |
| H6 | 0.6166 | 0.6274 | 0.7063 | 0.066* |
| C7 | 0.49202(19) | 0.6530(4) | 0.62203(9) | 0.0525(6) |
| H7A | 0.5053 | 0.5187 | 0.6171 | 0.063* |
| H7B | 0.5564 | 0.7151 | 0.6353 | 0.063* |
| C8 | 0.44482(18) | 0.7409(4) | 0.57028(9) | 0.0493(6) |
| H8A | 0.4391 | 0.8779 | 0.5743 | 0.059* |
| H8B | 0.4884 | 0.7175 | 0.5454 | 0.059* |
| C9 | 0.27692(19) | 0.6832(4) | 0.58989(9) | 0.0535(6) |
| H9A | 0.2113 | 0.6245 | 0.5775 | 0.064* |
| H9B | 0.2662 | 0.8182 | 0.5952 | 0.064* |
| C10 | 0.3261(2) | 0.5928(4) | 0.64064(9) | 0.0564(7) |
| H10A | 0.2835 | 0.6134 | 0.6661 | 0.068* |
| H10B | 0.3318 | 0.4561 | 0.6358 | 0.068* |
| C11 | 0.2909(2) | 0.7439(4) | 0.49953(9) | 0.0502(6) |
| H11A | 0.2824 | 0.8801 | 0.5041 | 0.060* |
| H11B | 0.2239 | 0.6880 | 0.4894 | 0.060* |
| C12 | 0.3498(2) | 0.7121(5) | 0.45654(10) | 0.0683(8) |
| H12A | 0.4135 | 0.7814 | 0.4643 | 0.082* |
| H12B | 0.3651 | 0.5773 | 0.4544 | 0.082* |
| C13 | 0.2891(2) | 0.7794(5) | 0.40509(10) | 0.0593(7) |
| C14 | 0.2689(2) | 0.9716(5) | 0.39575(10) | 0.0680(8) |
| H14 | 0.2923 | 1.0606 | 0.4216 | 0.082* |
| C15 | 0.2143(2) | 1.0324(5) | 0.34838(11) | 0.0691(8) |
| H15 | 0.2012 | 1.1620 | 0.3428 | 0.083* |
| C16 | 0.1789(2) | 0.9027(5) | 0.30907(10) | 0.0623(7) |
| C17 | 0.1981(2) | 0.7126(5) | 0.31815(11) | 0.0678(8) |
| H17 | 0.1748 | 0.6240 | 0.2922 | 0.081* |
| C18 | 0.2524(2) | 0.6507(5) | 0.36587(10) | 0.0650(8) |
| H18 | 0.2642 | 0.5208 | 0.3715 | 0.078* |
| C19 | 0.1216(3) | 0.9784(5) | 0.25782(11) | 0.0778(9) |
| H19A | 0.0567 | 1.0300 | 0.2623 | 0.093* |
| H19B | 0.1601 | 1.0802 | 0.2456 | 0.093* |
| C20 | 0.0572(2) | 0.8698(5) | 0.17149(10) | 0.0678(8) |
| C21 | 0.0305(2) | 1.0506(5) | 0.15351(11) | 0.0749(9) |
| H21 | 0.0462 | 1.1551 | 0.1755 | 0.090* |
| C22 | −0.0204(3) | 1.0779(5) | 0.10207(12) | 0.0793(10) |
| H22 | −0.0396 | 1.1992 | 0.0895 | 0.095* |
| C23 | −0.0407(2) | 0.9218(5) | 0.07151(11) | 0.0660(8) |
| C24 | −0.0132(2) | 0.7428(5) | 0.08970(11) | 0.0675(8) |
| C25 | 0.0361(2) | 0.7112(5) | 0.13948(11) | 0.0761(9) |
| H25 | 0.0548 | 0.5888 | 0.1514 | 0.091* |
| C26 | −0.0928(3) | 0.7162(6) | 0.00771(13) | 0.0915(12) |
| H26A | −0.0582 | 0.6967 | −0.0211 | 0.110* |
| H26B | −0.1623 | 0.6728 | −0.0028 | 0.110* |

## Discussion

Compounds with arylpiperazine moieties have a wide range of activities including antiarrhythmic [4], diuretic [5], antiallergic [6], antidepressant [7], anxiolytic [8], antipsychotic [9], antimalarial [10], antiplasmodial [11] and anti-proliferative [12–14] properties. In addition, these compounds also display receptor-blocking properties [15–19]. Moreover, arylpiperazine derivatives have been reported as anticancer drugs for site-directed chemotherapy of prostate cancer in our previous work [20, 21], and some derivatives showed significant cytotoxic activity against the tested prostate cancer cell lines.

The molecule of the title compound shows an aryl, a piperazine and the benzo[*d*][1,3]dioxole dihydrosulfide moieties. The 2-chloro-phenyl moiety is connected to the 1,4-piperazine moiety *via* a C—N bond, then the above fragment further is connected by a $C_6H_4$ unit *via* an ethylene group. The termial fragment of molecule is the benzo[*d*][1,3]dioxole dihydrosulfide unit. The central aryl ring and benzo[*d*][1,3]dioxole dihydrosulfide ring are almost into the same plane. In the molecule, the N(1)—C(5), N(1)—C(7), N(1)—C(10), N(2)—C(11), N(2)—C(9), N(2)—N(8) bond lengths are found to be 1.452(3) Å, 1.456(3) Å, 458(3) Å, 1.500(3) Å, 1.494(3) Å and 1.491(3) Å, respectively, which are nearly equal to other typical single bonds. The bond lengths of C(24)—O(2) and C(26)—O(2) are found to be 1.372(4) and 1.424(4) Å, respectively. The bond angles C5—N1—C10, C5—N1—C7, C10—N1—C7, C9—N2—C11, C8—N2—C11 and C8—N2—C9 are 118.8(2)°, 118.4(2)°, 111.7(19)°, 109.97(18)°, 113.34(19)° and 109.64(18)°, respectively. N2—C11—C12 and C11—C12—C13 are 112.7(2)°, 110.5(3)°. Bond lengths and all other geometic parameters of the title molecule are in the expected ranges [22].

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