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Crystal structure of 4-((1,3-dioxoisindolin-2-yl)methyl)phenethyl 4-methylbenzenesulfonate, $C_{24}H_{21}NO_5S$

Table 1: Data collection and handling.

Crystal:	Prism, colorless
Size:	$0.2 \times 0.1 \times 0.1$ mm
Wavelength:	Cu $K\alpha$ radiation (1.54178 Å)
μ :	1.64 mm^{-1}
Diffractometer, scan mode:	Bruker P4, ω -scans
θ_{max} , completeness:	68.2° , $>98\%$
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	34088, 3896, 0.049
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2702
$N(\text{param})_{\text{refined}}$:	281
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

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Abstract

$C_{24}H_{21}NO_5S$, monoclinic, $P2_1/c$ (no. 14), $a = 7.7790(16)$ Å, $b = 22.726(5)$ Å, $c = 12.755(3)$ Å, $\beta = 107.15(3)^\circ$, $V = 2154.6(9)$ Å³, $Z = 4$, $R_{\text{gt}}(F^2) = 0.0453$, $wR_{\text{ref}}(F^2) = 0.1371$, $T = 293.15$ K.

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

Source of material

To a mixture of 2-[[4-(2-hydroxyethyl)phenyl]methyl]-1*H*-isoindole-1,3(2*H*)-dione (2.81 g, 10 mmol), potassium carbonate K_2CO_3 (4.15 g, 30 mmol) and 60 mL dry CH_2Cl_2 , a solution

of tosyl chloride (2.10 g, 11 mmol) in 30 mL CH_2Cl_2 was added dropwise at about 0°C . The reaction mixture was stirred and maintained at a certain heated temperature for 1 to 4 h, until TLC indicated the end of reaction. The solvent was removed under reduced pressure. The residue was purified by silica gel column chromatography using ethyl acetate/petroleum ether (1:8, v/v) as the eluent. The title compound was afforded as a white solid.

Experimental details

All H atoms were placed in geometrically idealized positions and refined using a riding model, with $C-H = 0.97$ (methylene), 0.93 Å (benzene), and with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$ for H atoms on methylene and benzene. The hydrogens on terminal methyls were constrained with $C-H = 0.97$ Å and $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(C)$.

Discussion

The arylpiperazine moiety is an important pharmacophore, which is widely demonstrated to have pharmacological activities [4, 5]. Naftopidil is a derivative of arylpiperazine ether and a specific antagonist targeted for the adrenergic receptor [6], which has already been regarded as one of the most commonly utilized antagonists targeted for the adrenergic receptor to treat the benign prostatic hyperplasia (BPH) in Japan [7]. The title compound is a pivotal intermediate for some arylpiperazine derivatives [8, 9].

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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}
S1	0.65559(9)	0.34733(3)	0.24221(5)	0.0699(2)
O1	0.4767(2)	0.36163(7)	0.15066(13)	0.0701(5)
O2	0.2664(3)	0.68074(9)	0.33844(17)	0.0888(6)
O3	0.6635(3)	0.64986(9)	0.14344(17)	0.0947(6)
O4	0.7887(3)	0.34565(9)	0.18642(18)	0.0937(6)
O5	0.6302(3)	0.29745(7)	0.30401(15)	0.0820(5)
N1	0.4295(3)	0.66102(8)	0.21802(16)	0.0631(5)
C1	0.3084(4)	0.35763(11)	0.1777(2)	0.0716(7)
H1A	0.3219	0.3749	0.2492	0.086*
H1B	0.2739	0.3167	0.1797	0.086*
C2	0.1663(4)	0.39033(11)	0.0910(2)	0.0753(7)
H2A	0.0491	0.3814	0.0994	0.090*
H2B	0.1673	0.3766	0.0192	0.090*
C3	0.1937(3)	0.45621(11)	0.0970(2)	0.0652(6)
C4	0.2810(4)	0.48491(12)	0.0334(2)	0.0750(7)
H4	0.3217	0.4636	−0.0169	0.090*
C5	0.3099(4)	0.54502(12)	0.0424(2)	0.0763(7)
H5	0.3714	0.5632	−0.0012	0.092*
C6	0.2499(3)	0.57843(11)	0.1140(2)	0.0658(6)
C7	0.2808(4)	0.64403(11)	0.1223(2)	0.0797(8)
H7A	0.3061	0.6578	0.0564	0.096*
H7B	0.1716	0.6633	0.1264	0.096*
C8	0.4073(3)	0.68039(10)	0.3166(2)	0.0659(6)
C9	0.5884(3)	0.69838(10)	0.3864(2)	0.0605(6)
C10	0.7083(3)	0.68803(10)	0.3277(2)	0.0617(6)
C11	0.6082(4)	0.66426(11)	0.2186(2)	0.0661(6)
C12	0.8890(4)	0.69958(12)	0.3721(3)	0.0829(8)
H12	0.9700	0.6928	0.3324	0.099*
C13	0.9460(4)	0.72160(13)	0.4778(3)	0.0937(10)
H13	1.0676	0.7293	0.5102	0.112*
C14	0.8263(5)	0.73219(13)	0.5355(3)	0.0947(10)
H14	0.8682	0.7474	0.6062	0.114*
C15	0.6434(4)	0.72084(12)	0.4910(2)	0.0810(8)
H15	0.5621	0.7281	0.5303	0.097*
C16	0.1563(4)	0.55021(12)	0.1761(2)	0.0772(7)
H16	0.1110	0.5719	0.2239	0.093*
C17	0.1292(4)	0.48979(12)	0.1677(2)	0.0764(7)
H17	0.0665	0.4715	0.2105	0.092*
C18	0.6882(3)	0.40947(10)	0.32704(19)	0.0636(6)
C19	0.7033(4)	0.46419(12)	0.2835(2)	0.0824(8)
H19	0.6936	0.4680	0.2094	0.099*
C20	0.7328(4)	0.51305(12)	0.3503(2)	0.0877(9)
H20	0.7431	0.5498	0.3205	0.105*
C21	0.7473(4)	0.50863(11)	0.4598(2)	0.0764(7)
C22	0.7303(4)	0.45350(12)	0.5019(2)	0.0843(8)
H22	0.7385	0.4497	0.5758	0.101*
C23	0.7014(4)	0.40377(11)	0.4360(2)	0.0752(7)
H23	0.6912	0.3670	0.4655	0.090*
C24	0.7813(5)	0.56234(13)	0.5339(3)	0.1015(10)
H24A	0.9063	0.5640	0.5749	0.152*
H24B	0.7098	0.5598	0.5836	0.152*
H24C	0.7491	0.5972	0.4900	0.152*

The asymmetric unit contains only one molecule. The title compound contained three aryl and a pyrrole moiety. Ring (C9—C10—C12—C13—C14—C15) and ring (C8—C9—C10—C11—N1) are coplanar. The dihedral angles made by the planes of (C9—C10—C12—C13—C14—C15) and (C3—C4—C5—C6—C16—C17), (C9—C10—C12—C13—C14—C15) and (C18—C23), (C9—C10—C12—C13—C14—C15) and (C18—C23) are 114.93(9)°, 33.86(10)° and 103.52(10)°, respectively. The packing shows no classical hydrogen bonds. There is at least one non-classical hydrogen bond between O2 and C12ⁱ (symmetry code (i): $-1 + x, y, z$) with $D \cdots A = 3.119$ Å. Weak π - π interactions were observed between ring (C3—C4—C5—C6—C16—C17) and ringⁱⁱ (C3—C4—C5—C6—C16—C17) with symmetry code (ii) $-x, 1 - y, -z$.

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