Table 2. *Bond distances* (Å), *bond angles* (°) *and torsion angles* (°)

S(1)—C(2)	1.790 (3)	C(8)—C(14)	1.513 (4)
S(1)—C(10)	1.767 (3)	C(9)—C(10)	1.339 (3)
O(1)—C(2)	1.212 (4)	C(9)—C(11)	1.457 (3)
O(2)—C(17)	1.440 (4)	C(11)—C(12)	1.326 (4)
O(2)—C(20)	1.347 (3)	C(12)—C(13)	1.498 (4)
O(3)—C(20)	1.202 (5)	C(13)—C(14)	1.544 (4)
C(2)—C(3)	1.454 (5)	C(13)—C(17)	1.526 (4)
C(3)—C(5)	1.333 (4)	C(13)—C(18)	1.561 (4)
C(5)—C(6)	1.502 (5)	C(14)—C(15)	1.531 (4)
C(5)—C(10)	1.458 (3)	C(15)—C(16)	1.528 (4)
C(6)—C(7)	1.523 (5)	C(16)—C(17)	1.545 (5)
C(7)—C(8)	1.526 (4)	C(18)—C(22)	1.510 (6)
C(8)—C(9)	1.530 (4)	C(20)—C(21)	1.458 (5)

C(2)—S(1)—C(10)	92.0 (2)	C(9)—C(11)—C(12)	123.9 (3)
C(17)—O(2)—C(20)	117.4 (4)	C(11)—C(12)—C(13)	121.3 (2)
S(1)—C(2)—O(1)	122.6 (3)	C(12)—C(13)—C(14)	107.6 (2)
S(1)—C(2)—C(3)	108.5 (2)	C(12)—C(13)—C(17)	116.0 (2)
O(1)—C(2)—C(3)	128.9 (3)	C(12)—C(13)—C(18)	110.1 (3)
C(2)—C(3)—C(5)	115.5 (3)	C(14)—C(13)—C(17)	98.9 (3)
C(3)—C(5)—C(6)	128.4 (3)	C(14)—C(13)—C(18)	112.4 (2)
C(3)—C(5)—C(10)	114.3 (3)	C(17)—C(13)—C(18)	111.3 (2)
C(6)—C(5)—C(10)	117.3 (3)	C(8)—C(14)—C(13)	113.4 (3)
C(5)—C(6)—C(7)	111.7 (3)	C(8)—C(14)—C(15)	120.9 (2)
C(6)—C(7)—C(8)	110.8 (4)	C(13)—C(14)—C(15)	103.1 (2)
C(7)—C(8)—C(9)	110.0 (2)	C(14)—C(15)—C(16)	103.3 (3)
C(7)—C(8)—C(14)	114.3 (3)	C(15)—C(16)—C(17)	106.3 (3)
C(9)—C(8)—C(14)	108.5 (2)	O(2)—C(17)—C(13)	111.1 (3)
C(8)—C(9)—C(10)	118.9 (2)	O(2)—C(17)—C(16)	113.4 (3)
C(8)—C(9)—C(11)	118.4 (2)	C(13)—C(17)—C(16)	104.7 (3)
C(10)—C(9)—C(11)	122.7 (3)	C(13)—C(18)—C(22)	115.9 (3)
S(1)—C(10)—C(5)	109.7 (2)	O(2)—C(20)—O(3)	122.6 (3)
S(1)—C(10)—C(9)	125.5 (2)	O(2)—C(20)—C(21)	111.3 (4)
C(5)—C(10)—C(9)	124.8 (3)	O(3)—C(20)—C(21)	126.1 (4)

C(9)—C(10)—C(5)—C(6)	3.0 (4)	C(13)—C(14)—C(15)—C(16)	37.5 (3)
C(10)—C(5)—C(6)—C(7)	24.1 (4)	C(14)—C(15)—C(16)—C(17)	−11.6 (3)
C(5)—C(6)—C(7)—C(8)	−53.8 (3)	C(15)—C(16)—C(17)—C(13)	−18.7 (3)
C(6)—C(7)—C(8)—C(9)	56.5 (3)	C(16)—C(17)—C(13)—C(14)	40.7 (3)
C(7)—C(8)—C(9)—C(10)	−30.3 (4)	C(18)—C(13)—C(17)—O(2)	45.0 (3)
C(8)—C(9)—C(10)—C(5)	0.5 (4)	C(13)—C(17)—O(2)—C(20)	160.5 (2)
C(14)—C(8)—C(9)—C(11)	23.2 (3)	C(16)—C(17)—O(2)—C(20)	−81.8 (3)
C(8)—C(9)—C(11)—C(12)	2.3 (4)	C(17)—O(2)—C(20)—C(21)	178.9 (3)
C(9)—C(11)—C(12)—C(13)	2.5 (5)	C(17)—O(2)—C(20)—O(3)	−0.9 (5)
C(11)—C(12)—C(13)—C(14)	−31.2 (4)	C(12)—C(13)—C(18)—C(22)	40.0 (4)
C(12)—C(13)—C(14)—C(8)	58.1 (3)	C(14)—C(13)—C(18)—C(22)	159.9 (3)
C(13)—C(14)—C(8)—C(9)	−53.9 (3)	C(17)—C(13)—C(18)—C(22)	−90.1 (3)
C(17)—C(13)—C(14)—C(15)	−48.5 (3)		

98.9 (3)°. A similar bond angle was found in the following steroids: 6α-methyl-3,20-oxo-1,4,9(11)-pregnatrien-17α-yl acetate (Gałdecki, Grochulski, Wawrzak, Duax & Strong, 1990); 3β,20α-bis(dimethylamino)pregn-5-en-18-ol (Radhakrishnan, Viswamitra, Bhutani & Vaid, 1988). The plane of the OAc side chain and that of the ethyl side chain of C(13), C(18) and C(22) make angles of 80.3 (1) and 78.1 (2)°, respectively, with the steroid skeleton.

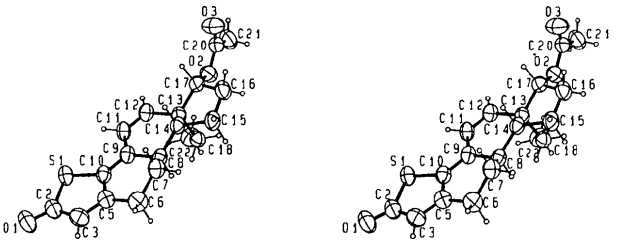

Fig. 1. A stereoscopic view of the molecules with the atom numbering viewed along the *c* axis. Thermal ellipsoids of the C and O atoms are drawn at 50% probability level. H atoms are shown with arbitrary radii.

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1-[3-(3,4-Methylenedioxyphenoxy)propyl]-4-phenylpiperazine Monohydrochloride

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Abstract. 1-[3-(Benzo[1,3]dioxol-5-yloxy)propyl]-4-phenylpiperazinium chloride, $C_{20}H_{25}N_2O_3^+.Cl^-$, M_r = 376.5, orthorhombic, *Pbca*, a = 17.615 (1), b = 15.400 (2), c = 13.786 (2) Å, V = 3739.9 (8) Å^3, Z = 8, D_x = 1.339 Mg m^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, μ = 1.998 mm^{-1}, $F(000)$ = 1600, T = 293 K, final R =

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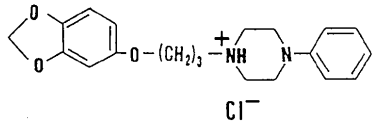
0.063 for 2799 independent reflections. The piperazine ring adopts a chair conformation. Both the propyl and the phenyl groups are in equatorial positions. An N^+—H···Cl^- hydrogen bond of 3.037 (3) Å was found.

Experimental. Colorless cubic crystals were obtained by slow evaporation of a methanol solution at room temperature. Crystal of dimensions 0.5 × 0.4 ×



0.1 mm. Rigaku AFC-5 diffractometer, ω–2θ scanning technique ($2\theta_{max} = 126°$), graphite-monochromatized Cu $K\alpha$ radiation, unit-cell dimensions by least-squares procedure based on 2θ values ($30 < 2\theta < 55°$) of 24 reflections, intensity fluctuation (< 1.3%) monitored periodically by three reflections ($\bar{6}4\bar{2}$, 045, $20\bar{6}$); 3408 unique reflections ($0 \le h \le 19$, $0 \le k \le 17$, $0 \le l \le 15$). Lorentz and polarization corrections, but none for absorption. Structure solved using *SHELXS*86 (Sheldrick, 1986); structure refined by full-matrix least-squares method using *SHELX*76 (Sheldrick, 1976), with anisotropic temperature factors for all non-H atoms; all H atoms were located from a difference Fourier map and geometrical calculations were made with *MOLCON* (Fujii, 1979). The final refinement, including all H atoms with isotropic temperature factors, reduced R to 0.063 ($wR = 0.057$, $S = 0.305$) for 2799 observed reflections [$|F_o| \ge 3\sigma(F_o)$] and 335 variables; function minimized: $\sum w(|F_o| - k|F_c|)^2$, $w = 1.0/[\sigma(|F_o|)^2 - 0.429|F_o| + 0.0203|F_o|^2]$ for $F_o \neq 0$, $w = 0.0$ for $|F_o| = 0$. Highest and lowest residuals in the final difference Fourier map: 0.34 and −0.24 e Å^{-3}, respectively; $(\Delta/\sigma)_{max} = 0.026$ for non-H atoms. Scattering factors from *International Tables for X-ray Crystallography* (1962, Vol. III); program system *HBLS-V* (Ashida, 1979). All calculations were carried out on the ACOS930 computer of the Research Center for Protein Engineering, Institute for Protein Research, Osaka University.

The molecular structure with atomic numbering is shown in Fig. 1. Atomic coordinates and the equivalent isotropic thermal parameters of the non-H atoms are listed in Table 1.* Selected bond lengths, angles and torsion angles are given in Table 2. The crystal packing viewed along the c axis is shown in Fig. 2.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55919 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0505]

Table 1. *Atomic coordinates* (× 10^4) *and thermal parameters* (Å^2) *of the non-H atoms with e.s.d.'s in parentheses*

$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij} \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
C(1)	485 (1)	5241 (2)	3352 (2)	2.34 (6)
C(2)	275 (1)	4367 (2)	3290 (2)	2.37 (6)
C(3)	−431 (1)	4171 (2)	3660 (2)	2.43 (6)
C(4)	−908 (1)	4785 (2)	4044 (2)	2.74 (6)
C(5)	−709 (2)	5638 (2)	4096 (2)	3.11 (7)
C(6)	6 (2)	5860 (2)	3742 (2)	2.92 (7)
C(7)	−1462 (2)	3491 (2)	4212 (2)	3.91 (8)
C(8)	1738 (2)	4932 (2)	2770 (2)	3.21 (7)
C(9)	2491 (2)	5405 (2)	2717 (2)	3.07 (7)
C(10)	2671 (1)	5798 (2)	3697 (2)	2.62 (6)
C(11)	3539 (1)	6601 (2)	4752 (2)	2.66 (6)
C(12)	4323 (1)	6977 (2)	4877 (2)	2.59 (6)
C(13)	4838 (1)	5946 (2)	3765 (2)	2.88 (6)
C(14)	4064 (1)	5559 (2)	3593 (2)	2.55 (6)
C(15)	5628 (1)	6470 (2)	5106 (2)	2.37 (6)
C(16)	6280 (2)	6306 (2)	4560 (2)	3.02 (7)
C(17)	6996 (2)	6420 (2)	4972 (2)	3.72 (8)
C(18)	7075 (2)	6716 (2)	5904 (2)	3.92 (8)
C(19)	6430 (2)	6901 (2)	6444 (2)	3.85 (8)
C(20)	5713 (2)	6775 (2)	6058 (2)	3.00 (7)
N(1)	3445 (1)	6198 (1)	3768 (1)	2.05 (5)
N(2)	4891 (1)	6301 (1)	4746 (1)	2.47 (5)
O(1)	1168 (1)	5552 (1)	3014 (2)	3.03 (5)
O(2)	−1582 (1)	4400 (1)	4326 (2)	4.13 (6)
O(3)	−764 (1)	3366 (1)	3692 (2)	3.73 (5)
Cl	3441 (1)	7587 (1)	2204 (1)	3.57 (2)

Table 2. *Selected bond lengths* (Å), *angles* (°) *and torsion angles* (°) *with e.s.d.'s in parentheses*

C(1)—O(1)	1.376 (3)	C(10)—N(1)	1.500 (3)
C(3)—C(4)	1.371 (4)	C(11)—C(12)	1.508 (4)
C(3)—O(3)	1.373 (3)	C(11)—N(1)	1.500 (3)
C(4)—O(2)	1.383 (4)	C(12)—N(2)	1.455 (3)
C(7)—O(2)	1.425 (4)	C(13)—C(14)	1.506 (4)
C(7)—O(3)	1.436 (4)	C(13)—N(2)	1.462 (4)
C(8)—C(9)	1.515 (4)	C(14)—N(1)	1.489 (3)
C(8)—O(1)	1.426 (4)	C(15)—N(2)	1.414 (3)
C(9)—C(10)	1.514 (4)		

C(2)—C(3)—O(3)	126.5 (2)	C(14)—C(13)—N(2)	110.6 (2)
C(4)—C(3)—O(3)	110.4 (2)	C(13)—C(14)—N(1)	112.0 (2)
C(3)—C(4)—O(2)	109.9 (2)	C(10)—N(1)—C(11)	109.2 (2)
C(5)—C(4)—O(2)	128.3 (3)	C(10)—N(1)—C(14)	112.5 (2)
O(2)—C(7)—O(3)	108.3 (3)	C(11)—N(1)—C(14)	109.9 (2)
C(9)—C(8)—O(1)	107.8 (2)	C(12)—N(2)—C(13)	109.8 (2)
C(8)—C(9)—C(10)	109.4 (2)	C(1)—O(1)—C(8)	117.5 (2)
C(9)—C(10)—N(1)	114.4 (2)	C(4)—O(2)—C(7)	105.2 (2)
C(12)—C(11)—N(1)	111.3 (2)	C(3)—O(3)—C(7)	105.1 (2)
C(11)—C(12)—N(2)	110.0 (2)		

C(6)—C(1)—O(1)—C(8)	168.8 (2)	C(12)—C(11)—N(1)—C(10)	177.3 (2)
C(9)—C(8)—O(1)—C(1)	−163.1 (2)	C(11)—C(12)—N(2)—C(15)	−162.2 (2)
O(1)—C(8)—C(9)—C(10)	61.3 (3)	C(14)—C(13)—N(2)—C(15)	163.3 (2)
C(8)—C(9)—C(10)—N(1)	173.4 (2)	C(13)—C(14)—N(1)—C(10)	−174.3 (2)
C(9)—C(10)—N(1)—C(11)	177.2 (2)	C(16)—C(15)—N(2)—C(12)	−133.5 (3)
C(9)—C(10)—N(1)—C(14)	−60.5 (3)	C(16)—C(15)—N(2)—C(13)	−0.1 (4)
N(1)—C(11)—C(12)—N(2)	−58.5 (3)		

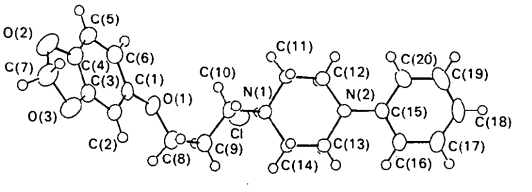


Fig. 1. The atom-numbering scheme with the thermal ellipsoids of all atoms drawn by *ORTEP* (Johnson, 1965). The thermal vibration ellipsoids are set to 50% probability.

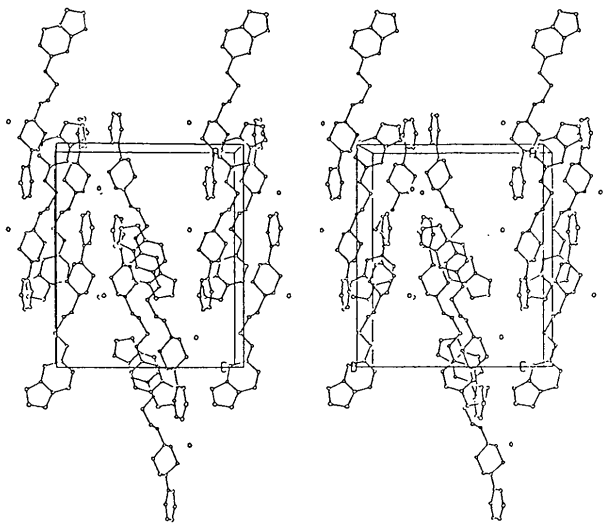


Fig. 2. The crystal packing viewed along the *c* axis.

Related literature. Pharmacological study demonstrates that this substance is a selective 5-HT_{1A} (serotonin$_{1A}$) receptor agonist (Matsuda, Seong, Aono, Kanda, Baba, Saito, Tobe & Iwata, 1989).

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Structure of 2,2-Bis(3,5-di-*tert*-butylphenyl)propane-2′,2″-dithiol

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Abstract. $C_{31}H_{48}S_2$, $M_r = 484.86$, monoclinic, $C2/c$, $a = 18.479$ (2), $b = 9.294$ (1), $c = 18.126$ (2) Å, $\beta = 99.41$ (1)°, $V = 3071.3$ Å^3, $Z = 4$, $D_x = 1.049$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 1.627$ mm^{-1}, $F(000) = 1064$, $T = 293$ K. The final R value was 0.055 for 2413 significant [$I > 3\sigma(I)$] reflections. The crystal structure confirms the close relationship of the thiol groups (S···S′ = 4.29 Å) which explains the observed elimination of hydrogen sulfide leading to the formation of the corresponding thioxanthene.

Experimental. The compound was synthesized by a Newman–Kwart rearrangement (Newman & Karnes, 1966; Kwart & Evans, 1966). In contrast to the very stable 2,4,6-tri-*tert*-butylbenzenethiol (Rundel, 1968), the title compound proved to be rather sensitive, in that it readily eliminates hydrogen sulfide, for instance when heated to its melting point (440 K). Crystals were grown by slowly cooling a warm saturated ethyl acetate solution of the pure (crystallized twice from ethyl acetate) compound. A yellow single crystal of approximate dimensions 0.15 × 0.15 ×

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