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Structure of 10-Benzyl-2-[(3,4-dimethoxyphenyl)methyl]phenothiazine-1-carbonitrile
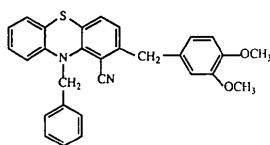
BY HONGMING ZHANG, JENIFER SELF, SUBHASH P. KHANAPURE AND ED BIEHL

Department of Chemistry, Southern Methodist University, Dallas, Texas 75275, USA

Abstract. $C_{29}H_{24}N_2O_2S$, $M_r = 464.58$, triclinic, $P\bar{1}$, $a = 9.967$ (3), $b = 10.912$ (3), $c = 12.258$ (3) Å, $\alpha = 71.07$ (2), $\beta = 67.26$ (2), $\gamma = 75.61$ (2)°, $V = 1151.4$ (8) Å^3, $Z = 2$, $D_x = 1.34$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.62$ cm^{-1}, $F(000) = 488$, $T = 230$ K, $R = 0.0375$ for 3734 observed reflections. The molecule adopts the *synclinal* conformation in which the 10-benzyl group occupies the pseudo axial position; the dihedral angle between the two benzo rings is 142.7 (1)°.

Experimental. The title compound (1) was prepared by the reaction of lithio-3,4-(dimethoxyphenyl)-acetonitrile and 10-benzyl-2-chlorophenothiazine with diisopropylamide in tetrahydrofuran. Colorless prismatic crystals were recrystallized from ethyl acetate. Unit-cell parameters by least-squares fit of

(1)

24 reflections in the range $21 < 2\theta < 30°$, crystal dimensions $0.35 \times 0.25 \times 0.20$ mm, space group $P\bar{1}$; Siemens $R3m/V$ diffractometer was used for data collection, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode, scan rate $3.0–15.0°$ min^{-1} depending upon the intensity, 5889 reflections measured in the range $3.0 \le 2\theta \le 56.0°$, $0 \le h \le 14$, -15

$\le k \le 15$, $-17 \le l \le 17$, 5579 independent, $R_{int} = 0.014$, 3734 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics. Three standard reflections remeasured after every 150 reflections showed a maximum fluctuation in intensity of $\pm 0.86\%$ during the data collection. Lorentz and polarizarion effects corrected, but no extinction or absorption correction. Structure was solved by direct methods with *SHELXTL-Plus* (Sheldrick, 1990), refined by full-matrix least squares, anisotropically for all non-H atoms. All H atoms were located in difference Fourier maps and refined isotropically. The function minimized was $\sum w(|F_o| - F_c)^2$ with $w = 1/[\sigma^2(F_o) + 0.00025(F_o)^2]$. Final $R = 0.0375$, $wR = 0.0436$, $S = 1.62$ for 3734 reflections with 403 parameters refined. In the last cycle, $(\Delta/\sigma)_{max} = 0.002$, the maximum and minimum difference Fourier residuals are 0.19 and -0.22 e Å^{-3}, respectively. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Positional and thermal parameters for non-H atoms are given in Table 1,* while the bond lengths and bond angles are given in Table 2. Fig. 1 is an *ORTEP* (Johnson, 1965) drawing of the title compound and Fig. 2 shows the packing in the unit cell.

$C_{29}H_{24}N_2O_2S$

Table 1. *Atomic coordinates* ($\times 10^5$) *and equivalent isotropic displacement coefficients* ($\text{Å}^2 \times 10^4$)

	x	y	z	U_{eq}*
C1	31442 (17)	46502 (15)	13095 (14)	330 (7)
C(2)	41408 (18)	37307 (16)	18307 (15)	377 (7)
C(3)	49654 (20)	41643 (18)	22763 (17)	439 (8)
C(4)	47905 (20)	54667 (19)	22462 (17)	428 (8)
S(5)	33564 (6)	79582 (5)	20020 (5)	456 (2)
C(6)	31710 (20)	100880 (17)	834 (19)	441 (8)
C(7)	27322 (20)	107633 (18)	−9131 (19)	486 (9)
C(8)	20461 (21)	101665 (18)	−13392 (19)	485 (8)
C(9)	17722 (20)	88864 (17)	−7627 (18)	422 (8)
N(10)	18295 (15)	69160 (13)	9060 (13)	362 (6)
C(4a)	37226 (18)	63594 (16)	18219 (15)	344 (7)
C(5a)	28861 (17)	88059 (16)	6802 (16)	361 (7)
C(9a)	21598 (17)	82032 (15)	2708 (15)	336 (7)
C(10a)	28797 (17)	59783 (15)	13366 (14)	313 (6)
C(11)	24863 (19)	41734 (16)	6970 (16)	397 (7)
N(12)	20493 (20)	37160 (17)	2026 (17)	569 (9)
C(13)	42991 (24)	22906 (18)	19316 (17)	454 (8)
C(14)	34270 (18)	15233 (15)	31798 (15)	347 (7)
C(15)	40622 (19)	2743 (15)	36627 (16)	355 (7)
C(16)	33179 (18)	−4933 (15)	47737 (15)	352 (7)
C(17)	18903 (18)	−140 (16)	54419 (15)	359 (7)
C(18)	12659 (20)	12251 (17)	49734 (17)	398 (8)
C(19)	20273 (20)	19904 (17)	38466 (17)	401 (8)
C(20)	4633 (19)	65744 (17)	9559 (17)	370 (7)
C(21)	−3599 (17)	58081 (15)	22127 (15)	334 (7)
C(22)	−9900 (21)	47467 (18)	23432 (18)	439 (8)
C(23)	−17972 (23)	40668 (21)	34841 (21)	547 (10)
C(24)	−19913 (24)	44451 (24)	45048 (21)	590 (10)
C(25)	−13902 (25)	55052 (25)	43892 (21)	617 (11)
C(26)	−5734 (22)	61789 (20)	32504 (19)	489 (9)
O(27)	38702 (13)	−17279 (11)	52954 (12)	475 (6)
C(28)	52738 (23)	−22652 (20)	46040 (21)	489 (9)
O(29)	12345 (13)	−8521 (12)	65274 (11)	474 (6)
C(30)	−2416 (23)	−4327 (26)	72000 (21)	513 (10)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.
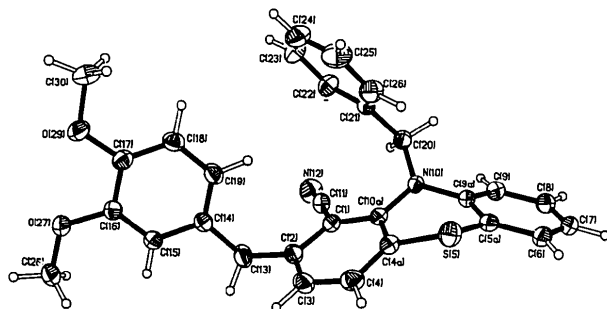

Fig. 1. Perspective view and atomic numbering of the title compound with thermal ellipsoids drawn at 40% probability level, H atoms with arbitrary radius.
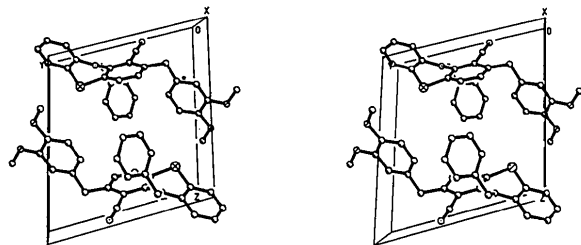

Fig. 2. Stereoview of the unit cell. H atoms are omitted for clarity.

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

C(1)—C(2)	1·407 (2)	C(1)—C(10a)	1·416 (2)
C(1)—C(11)	1·438 (3)	C(2)—C(3)	1·380 (3)
C(2)—C(13)	1·508 (3)	C(3)—C(4)	1·378 (3)
C(4)—C(4a)	1·392 (3)	S(5)—C(4a)	1·760 (3)
S(5)—C(5a)	1·761 (2)	C(6)—C(7)	1·377 (3)
C(6)—C(5a)	1·395 (2)	C(7)—C(8)	1·372 (4)
C(8)—C(9)	1·387 (3)	C(9)—C(9a)	1·390 (3)
N(10)—C(9a)	1·418 (2)	N(10)—C(10a)	1·402 (2)
N(10)—C(20)	1·473 (2)	C(4a)—C(10a)	1·397 (3)
C(5a)—C(9a)	1·393 (3)	C(11)—N(12)	1·146 (4)
C(13)—C(14)	1·517 (2)	C(14)—C(15)	1·395 (2)
C(14)—C(19)	1·382 (2)	C(15)—C(16)	1·376 (2)
C(16)—C(17)	1·406 (2)	C(16)—O(27)	1·368 (2)
C(17)—C(18)	1·378 (2)	C(17)—O(29)	1·367 (2)
C(18)—C(19)	1·392 (2)	C(20)—C(21)	1·508 (2)
C(21)—C(22)	1·387 (3)	C(21)—C(26)	1·382 (3)
C(22)—C(23)	1·381 (3)	C(23)—C(24)	1·372 (4)
C(24)—C(25)	1·375 (4)	C(25)—C(26)	1·380 (3)
O(27)—C(28)	1·420 (2)	O(29)—C(30)	1·423 (2)

C(2)—C(1)—C(10a)	121·2 (2)	C(2)—C(1)—C(11)	116·0 (2)
C(10a)—C(1)—C(11)	122·7 (2)	C(1)—C(2)—C(3)	118·8 (2)
C(1)—C(2)—C(13)	121·4 (2)	C(3)—C(2)—C(13)	119·8 (2)
C(2)—C(3)—C(4)	120·9 (2)	C(3)—C(4)—C(4a)	120·4 (2)
C(4a)—S(5)—C(5a)	98·9 (1)	C(7)—C(6)—C(5a)	120·0 (2)
C(6)—C(7)—C(8)	120·1 (2)	C(7)—C(8)—C(9)	120·4 (2)
C(8)—C(9)—C(9a)	120·3 (2)	C(9a)—N(10)—C(10a)	119·2 (2)
C(9a)—N(10)—C(20)	118·2 (1)	C(10a)—N(10)—C(20)	122·2 (1)
C(4)—C(4a)—S(5)	118·9 (2)	C(4)—C(4a)—C(10a)	121·0 (2)
S(5)—C(4a)—C(10a)	120·0 (1)	S(5)—C(5a)—C(6)	120·6 (2)
S(5)—C(5a)—C(9a)	119·1 (1)	C(6)—C(5a)—C(9a)	120·2 (2)
C(9)—C(9a)—N(10)	121·2 (2)	C(9)—C(9a)—C(5a)	118·8 (2)
N(10)—C(9a)—C(5a)	120·0 (2)	C(1)—C(10a)—N(10)	123·7 (2)
C(1)—C(10a)—C(4a)	117·4 (2)	N(10)—C(10a)—C(4a)	118·9 (2)
C(1)—C(11)—N(12)	174·8 (2)	C(2)—C(13)—C(14)	113·5 (2)
C(13)—C(14)—C(15)	118·1 (1)	C(13)—C(14)—C(19)	123·2 (1)
C(15)—C(14)—C(19)	118·6 (1)	C(14)—C(15)—C(16)	121·3 (1)
C(15)—C(16)—C(17)	119·7 (1)	C(15)—C(16)—O(27)	124·3 (1)
C(16)—C(17)—O(27)	116·0 (1)	C(16)—C(17)—C(18)	119·2 (1)
C(16)—C(17)—O(29)	115·4 (1)	C(18)—C(17)—O(29)	125·5 (1)
C(17)—C(18)—C(19)	120·7 (1)	C(14)—C(19)—C(18)	120·6 (2)
N(10)—C(20)—C(21)	112·5 (2)	C(20)—C(21)—C(22)	120·1 (2)
C(20)—C(21)—C(26)	121·4 (2)	C(22)—C(21)—C(26)	118·4 (2)
C(21)—C(22)—C(23)	120·8 (2)	C(22)—C(23)—C(24)	119·9 (2)
C(23)—C(24)—C(25)	119·9 (2)	C(24)—C(25)—C(26)	120·2 (3)
C(21)—C(26)—C(25)	120·7 (2)	C(16)—O(27)—C(28)	117·0 (1)
C(17)—O(29)—C(30)	117·2 (1)		

Related literature. The X-ray analysis reported herein confirms that the title compound was formed by a tandem-addition rearrangement mechanism (Pansegrau, Rieker & Meyers, 1988). The cyano and arylmethyl groups are suitably configured for further cyclization after proper modification (Crenshaw, Khanapure, Siriwardane & Biehl, 1988).

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