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Structure of 2,3-Dihydro-5,6-dimethoxy-3-(*o*-nitrobenzylidene)benzo[*b*]thiophen-2-one

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Abstract. $C_{17}H_{13}NO_5S$, $M_r = 343{\cdot}4$, triclinic, $P\bar{1}$, $a = 7{\cdot}706$ (4), $b = 9{\cdot}785$ (6), $c = 11{\cdot}470$ (10) Å, $\alpha = 66{\cdot}34$ (6), $\beta = 78{\cdot}04$ (6), $\gamma = 84{\cdot}44$ (5)°, $V = 774{\cdot}9$ Å^3, $D_m = 1{\cdot}45$ (3), $D_x = 1{\cdot}458$ (2) g cm^{-3}, $Z = 2$, λ(Mo $K\bar{\alpha}$) = 0·71069 Å, $\mu = 1{\cdot}89$ cm^{-1}, $F(000) = 356$, $T = 295$ K, m.p. 448–449 K, $R = 0{\cdot}0275$ for 1553 observed reflections with $F_o \geq 4{\cdot}5\sigma(F_o)$. The methoxy groups are in the plane of the benzene ring and steric hindrance causes an increase, by more than 4·3° from the theoretical 120°, in the O(2)–C(5)–C(4) and O(3)–C(6)–C(7) angles.

Experimental. The compound was provided by Dr Catsoulakos. Red transparent crystals from a 1:1 chloroform–methanol solution. Density measured by flotation. Syntex $P2_1$ diffractometer. Crystal size 0·28 × 0·30 × 0·48 mm. Lattice parameters from 15 automatically centered reflections, $20 \leq 2\theta \leq 22°$. Graphite-monochromatized Mo $K\bar{\alpha}$ radiation, θ–2θ scan. $2\theta \leq 43°$, h −7→7, k −10→10, l 0→11. Variable scan speed 1·0–10·0° min^{-1}. Scan range 1·8° (2θ) plus α_1–α_2 separation, background counting time 0·5 of scan time. Three reflections monitored periodically showed less than 3% intensity fluctuation. Lp corrections applied; no correction for absorption. Data collected/unique/R_{int}, 1879/1710/0·0076; 1558 reflections with $F_o \geq 4{\cdot}5\sigma(F_o)$; five reflections affected by extinction were given zero weight during final refinement cycles. Structure solved by *MULTAN* (Germain, Main & Woolfson, 1971). Refinement based on F

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Table 1. *Positional and equivalent isotropic thermal parameters* (×10^4) *of the non-*H *atoms*

E.s.d.'s are in parentheses. $U_{eq} = (U_{11} + U_{22} + U_{33})/3$.

	x	y	z	U_{eq}(Å^2)
C(1)	507 (3)	4647 (2)	2255 (2)	435
C(2)	810 (2)	3153 (2)	2170 (2)	363
C(3)	1869 (2)	3286 (2)	920 (2)	357
C(4)	2254 (3)	2193 (2)	402 (2)	380
C(5)	3187 (3)	2533 (2)	−827 (2)	394
C(6)	3764 (3)	4000 (2)	−1603 (2)	404
C(7)	3367 (3)	5095 (2)	−1111 (2)	409
C(8)	2428 (3)	4728 (2)	135 (2)	388
S	1744·9 (7)	6024·2 (5)	863·8 (5)	493
O(1)	−398 (2)	4909 (1)	3135 (1)	594
O(2)	3623 (2)	1523 (1)	−1399 (1)	518
C(9)	3017 (4)	47 (2)	−669 (2)	602
O(3)	4668 (2)	4221 (1)	−2812 (1)	410
C(10)	5160 (4)	5710 (2)	−3659 (2)	563
C(11)	96 (3)	1970 (2)	3192 (2)	383
C(12)	433 (2)	401 (2)	3364 (2)	334
C(13)	−874 (2)	−612 (2)	3587 (2)	347
N	−2739 (2)	−134 (2)	3674 (2)	452
O(4)	−3105 (2)	1177 (2)	3113 (1)	596
O(5)	−3857 (2)	−1081 (2)	4301 (2)	692
C(14)	−497 (3)	−2064 (2)	3713 (2)	451
C(15)	1242 (3)	−2554 (2)	3638 (2)	486
C(16)	2564 (3)	−1612 (2)	3469 (2)	431
C(17)	2161 (3)	−160 (2)	3342 (2)	402

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

Maximum e.s.d.'s are 0·003 Å for distances and 0·2° for angles.

C(1)–O(1)	1·206	C(8)–S	1·767
C(1)–C(2)	1·497	S–C(1)	1·779
C(2)–C(3)	1·457	C(2)–C(11)	1·335
C(3)–C(8)	1·387	C(11)–C(12)	1·471
C(3)–C(4)	1·401	C(12)–C(13)	1·394
C(4)–C(5)	1·365	C(13)–N	1·463
C(5)–O(2)	1·372	N–O(4)	1·218
O(2)–C(9)	1·416	N–O(5)	1·217
C(5)–C(6)	1·409	C(13)–C(14)	1·377
C(6)–O(3)	1·357	C(14)–C(15)	1·376
C(3)–C(10)	1·425	C(15)–C(16)	1·372
C(6)–C(7)	1·382	C(16)–C(17)	1·379
C(7)–C(8)	1·381	C(17)–C(12)	1·390

O(1)–C(1)–C(2)	126·4	C(7)–C(8)–C(3)	122·1
O(1)–C(1)–S	123·6	C(7)–C(8)–S	124·3
C(2)–C(1)–S	110·0	C(3)–C(8)–S	113·5
C(1)–C(2)–C(3)	110·9	C(8)–S–C(1)	91·8
C(1)–C(2)–C(11)	117·6	C(2)–C(11)–C(12)	125·6
C(3)–C(2)–C(11)	131·5	C(11)–C(12)–C(13)	124·8
C(2)–C(3)–C(4)	128·5	C(11)–C(12)–C(17)	119·2
C(2)–C(3)–C(8)	113·4	C(13)–C(12)–C(17)	116·0
C(4)–C(3)–C(8)	117·9	C(12)–C(13)–N	119·8
C(3)–C(4)–C(5)	120·9	C(12)–C(13)–C(14)	122·9
C(4)–C(5)–O(2)	124·3	N–C(13)–C(14)	117·3
C(4)–C(5)–C(6)	120·4	C(13)–N–O(4)	119·1
O(2)–C(5)–C(6)	115·3	C(13)–N–O(5)	117·9
C(5)–O(2)–C(9)	117·2	O(4)–N–O(5)	123·0
C(5)–C(6)–C(7)	119·3	C(13)–C(14)–C(15)	118·9
C(5)–C(6)–O(3)	115·7	C(14)–C(15)–C(16)	120·0
O(3)–C(6)–C(7)	125·0	C(15)–C(16)–C(17)	120·2
C(6)–O(3)–C(10)	117·3	C(16)–C(17)–C(12)	121·8
C(6)–C(7)–C(8)	119·4		

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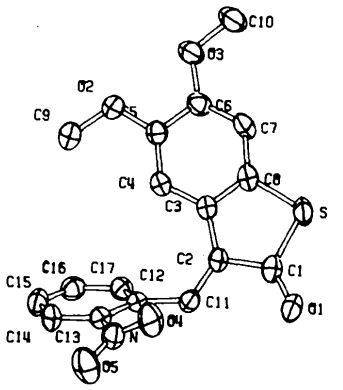

Fig. 1. An *ORTEP* diagram with 50% probability thermal ellipsoids, H atoms not included.

using *SHELX*76 (Sheldrick, 1976). H positions located from difference map but refined riding on C atoms at 0·98 Å. Least-squares refinement minimized $\sum w\Delta^2$, $1/w = \sigma^2(F_o) + 0{\cdot}00095|F_o|^2$. Non-H atoms anisotropic. $|\Delta/\sigma|_{max} = 0{\cdot}003$. $\Delta\rho_{max}/\Delta\rho_{min}$, 0·14/−0·19 e Å^{-3}. R/wR, 0·0275/0·0345 for 1553 observed data; R/wR, 0·0311/0·0371 for all data. Atomic scattering factors from *SHELX*76. The final atomic parameters of the non-H atoms are given in Table 1.*

Bond distances and angles are given in Table 2. A view of the molecule is shown in Fig. 1 (Johnson, 1965).

Related literature. The structures of two other heterocyclic analogues of benzothiazinone have been published (Salem, Filippakis, Hountas & Terzis, 1986).

* Lists of observed and calculated structure factors, anisotropic thermal parameters of the non-H atoms and positional and isotropic thermal parameters of the H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44407 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 9-Methoxy-11-demethylellipticine

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Abstract. 9-Methoxy-5-methyl-6*H*-pyrido[4,3-*b*]-carbazole, $C_{17}H_{14}N_2O$, $M_r = 262{\cdot}31$, monoclinic, $P2_1/c$, $a = 6{\cdot}326$ (2), $b = 23{\cdot}535$ (6), $c = 9{\cdot}141$ (3) Å, $\beta = 99{\cdot}87$ (3)°, $V = 1340{\cdot}8$ (16) Å^3, $Z = 4$, $D_x = 1{\cdot}299$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}571$ mm^{-1}, $F(000) = 552$, $R = 0{\cdot}066$ for 1487 observed reflections at 298 K. The geometry and dimensions of the planar ring system are not significantly different from those of ellipticine and its derivatives. Crystal packing is also very similar: the molecules are stacked along the *a* axis and weakly hydrogen bonded, N(6)—H···N(2) 2·999 (4) Å. The presence or absence of a substituent at positions 9 or 11 does not significantly influence the structure.

Experimental. Two light-yellow crystals of the title compound (Gouyette, Reynaud, Sadet, Baaillargé, Gansser, Cros, Le Goffic, Le Pecq, Paoletti & Viel, 1980) were obtained with difficulty from methanol solution. Crystal dimensions: 0·25 × 0·12 × 0·10 mm. Enraf–Nonius CAD-4 diffractometer; graphite-monochromated Cu $K\alpha$ radiation, ω–2θ scan. $\theta_{max} = 60°$ ($-7 \le h \le 7$, $0 \le k \le 26$, $0 \le l \le 10$). Lattice parameters from least-squares refinement of 25

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