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Non-Steroidal Anti-inflammatory Drugs. I. Structure of (*E*)-2,6-Di-*tert*-butyl-4-[2-(2-thienyl)ethenyl]phenol

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Abstract. $C_{20}H_{26}OS$, $M_r = 314.48$, orthorhombic, *Iba*2, $a = 17.655$ (4), $b = 21.155$ (4), $c = 9.978$ (2) Å, $V = 3727$ (1) Å^3, $Z = 8$, $D_m = 1.119$ (3) (flotation), $D_x = 1.121$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 0.166$ mm^{-1}, $F(000) = 1360$, $T = 294$ K, final $R = 0.046$ for 1544 reflections [$F_o \geq 3\sigma(F_o)$]. The phenyl and the thienyl ring systems are planar. The dihedral angle between the phenyl ring plane and the plane of the thienyl ring is 10.32 (1)°. The orientations of the thienyl ring and the phenyl ring relative to the double bond (C5═C6) are 174.5 (6) and 174.4 (5)° respectively.

Experimental. Greenish yellow cube-shaped crystals were kindly supplied by Dr Edward S. Lazer, Department of Medicinal Chemistry, Boehringer Ingelheim Pharmaceuticals, Inc., Ridefield, Connecticut, USA. Crystal 0.2 × 0.25 × 0.35 mm was used on an automated Siemens *R*3*m*/*V* diffractometer with incident-beam graphite-monochromated Mo $K\alpha$ radiation; 25 centered reflections within $20 < 2\theta < 35°$ were used for determining cell parameters. Data corrected for Lorentz and polarization effects, absorption ignored. $2\theta_{max} = 48°$, range of *hkl*: $0 \leq h \leq 12$, $0 \leq k \leq 21$, $0 \leq l \leq 25$. Standards 143, 220 and 055 monitored every 97 reflections with random variation of 2.1% over data collection. 2θ–θ scan mode; scan speed variable, 3.9–29.3° min^{-1}; scan width $[1.0 + 0.6(2\theta K\alpha_2 - 2\theta K\alpha_1)]°$; 3288 reflections measured, 1964 unique, $R_{merge} = 0.051$, 1544 observed [$F_o > 3\sigma(F_o)$].

Structure solved by direct methods using *SHELXTL-Plus* (Sheldrick, 1990). Refinement, minimizing $\sum w||F_o| - |F_c||^2$, by full-matrix least-squares calculations (on *F*) with anisotropic thermal parameters; in the final cycles of refinement, H atoms were included in calculated positions as riding atoms and with a common isotropic temperature factor. The final refinement included 200 variable parameters, $R = 0.046$, $wR = 0.048$, $w = 1.0/[\sigma^2(F_o) + 0.0011F_o^2]$, $S = 1.43$ with maximum shift/e.s.d. = 0.014. The highest and lowest residuals in the final difference Fourier map were 0.29 and −0.14 e Å^{-3}. Atomic scattering factors used were those in *SHEXLTL-*

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å^2 × 10^3) *with e.s.d.'s in parentheses*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	*y*	*z*	U_{eq}
S1	0.0975 (1)	0.0790 (1)	0.5319	96 (1)
O1	−0.2054 (2)	0.4011 (1)	0.6467 (4)	83 (1)
C1	0.1447 (7)	0.0154 (2)	0.5932 (7)	80 (2)
C2	0.1270 (3)	0.0063 (3)	0.7176 (9)	118 (3)
C3	0.0745 (4)	0.0506 (3)	0.7696 (8)	126 (3)
C4	0.0535 (2)	0.0938 (2)	0.6777 (6)	69 (2)
C5	0.0021 (3)	0.1471 (2)	0.6979 (5)	71 (2)
C6	−0.0133 (2)	0.1908 (2)	0.6123 (5)	60 (2)
C7	−0.0648 (2)	0.2450 (2)	0.6251 (5)	55 (2)
C8	−0.0672 (2)	0.2889 (2)	0.5237 (6)	57 (2)
C9	−0.1130 (2)	0.3423 (2)	0.5287 (6)	57 (1)
C10	−0.1583 (2)	0.3493 (2)	0.6430 (5)	53 (2)
C11	−0.1579 (2)	0.3064 (2)	0.7499 (5)	54 (2)
C12	−0.1101 (2)	0.2543 (2)	0.7380 (5)	52 (1)
C13	−0.1131 (3)	0.3907 (2)	0.4140 (6)	71 (2)
C14	−0.0590 (3)	0.3730 (3)	0.3011 (7)	108 (3)
C15	−0.0869 (3)	0.4553 (2)	0.4679 (8)	108 (3)
C16	−0.1909 (3)	0.3964 (3)	0.3484 (6)	87 (2)
C17	−0.2069 (3)	0.3162 (3)	0.8760 (5)	67 (2)
C18	−0.1885 (4)	0.3802 (2)	0.9422 (6)	95 (2)
C19	−0.2913 (3)	0.3123 (3)	0.8421 (7)	94 (2)
C20	−0.1922 (3)	0.2655 (3)	0.9810 (6)	95 (2)

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

S—C1	1.697 (6)	S1—C4	1.679 (6)
O1—C10	1.377 (5)	C1—C2	1.294 (9)
C2—C3	1.417 (9)	C3—C4	1.346 (9)
C4—C5	1.462 (7)	C5—C6	1.288 (7)
C6—C7	1.468 (6)	C7—C8	1.374 (7)
C7—C12	1.396 (7)	C8—C9	1.390 (5)
C9—C10	1.401 (7)	C9—C13	1.535 (8)
C10—C11	1.400 (7)	C11—C12	1.393 (6)
C11—C17	1.540 (7)	C13—C14	1.524 (8)
C13—C15	1.541 (8)	C13—C16	1.526 (7)
C17—C18	1.541 (7)	C17—C19	1.530 (7)
C17—C20	1.522 (8)		

C1—S1—C4	93.6 (3)	S1—C1—C2	110.2 (5)
C1—C2—C3	114.3 (7)	C2—C3—C4	112.3 (6)
S1—C4—C3	109.7 (4)	C3—C4—C5	126.9 (6)
S1—C4—C5	123.4 (4)	C4—C5—C6	126.4 (5)
C5—C6—C7	129.3 (5)	C6—C7—C12	122.3 (4)
C6—C7—C8	118.9 (4)	C8—C7—C12	118.7 (4)
C7—C8—C9	122.7 (5)	C8—C9—C13	121.0 (4)
C8—C9—C10	116.5 (4)	C10—C9—C13	122.4 (3)
O1—C10—C9	116.7 (4)	C9—C10—C11	123.3 (4)
O1—C10—C11	119.9 (4)	C10—C11—C17	122.2 (4)
C10—C11—C12	116.7 (4)	C12—C11—C17	121.0 (4)
C7—C12—C11	121.9 (4)	C9—C13—C16	112.0 (4)
C9—C13—C15	109.3 (5)	C9—C13—C14	112.8 (4)
C15—C13—C16	110.5 (4)	C14—C13—C16	105.4 (5)
C14—C13—C15	106.7 (5)	C11—C17—C20	111.8 (4)
C11—C17—C19	111.0 (4)	C11—C17—C18	110.5 (4)
C19—C17—C20	106.3 (4)	C18—C17—C20	106.8 (4)
C18—C17—C19	110.3 (4)		

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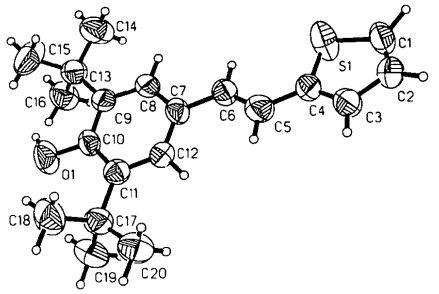

Fig. 1. Structure of the title compound. The ellipsoids are shown at 50% probability level.

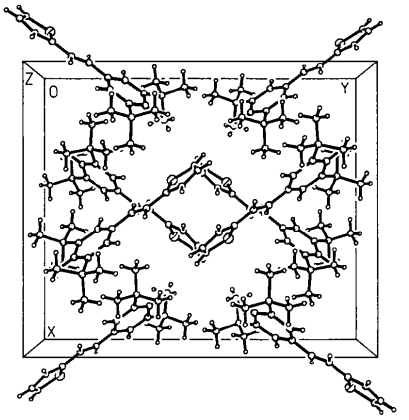

Fig. 2. The unit cell viewed down the *c* axis.

Plus. The calculations of geometrical data and crystal packing were computed using the program *PARST* (Nardelli, 1983). All computations were carried out on a VAX 3100 work station. Table 1 gives the atomic coordinates and equivalent isotropic thermal parameters* and Table 2 lists bond distances and angles. Fig. 1 shows a perspective view of the molecule with the adopted numbering scheme and Fig. 2 gives a view of the unit cell along the *c* axis.

Related literature. The 2,6-di-*tert*-butylphenol derivatives appear to represent a new class of non-steroidal anti-inflammatory drugs with antioxidant properties (Ikuta, Shirota, Kobayashi, Yamagishi, Yamada, Yamatsu & Katayama, 1987). The title compound is one of a series of related compounds prepared by Lazer, Wong, Possanza, Graham & Farina (1989) that have anti-inflammatory activity. In all essential details the geometry of the molecule in terms of bond lengths and angles shows normal values (Tenon, Ebby, Voglozin, Degny, N'Guessan, Baldy, Pierrot & Bodot, 1989; Bernstein, 1975; Tirado-Rives, Fronczek & Gandour, 1985).

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* 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 54789 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

BERNSTEIN, J. (1975). *Acta Cryst.* B**31**, 1268–1271.
IKUTA, S., SHIROTA, H., KOBAYASHI, S., YAMAGISHI, Y., YAMADA, K., YAMATSU, I. & KATAYAMA, K. (1987). *J. Med. Chem.* **30**, 1995–1998.
LAZER, E. S., WONG, H. C., POSSANZA, G. J., GRAHAM, A. & FARINA, P. R. (1989). *J. Med. Chem.* **32**, 100–104.
NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.
SHELDRICK, G. M. (1990). *SHELXTL-Plus*. Revision 4.11/*V*. Siemens Analytical X-ray Instruments, Inc., Application Laboratory, Single Crystal, Siemens AG, AUT V353, Karlsruhe, Germany.
TENON, A. J., EBBY, N., VOGLOZIN, A., DEGNY, E., N'GUESSAN, T. Y., BALDY, A., PIERROT, M. & BODOT, H. (1989). *Acta Cryst.* C**45**, 622–625.
TIRADO-RIVES, J., FRONCZEK, F. R. & GANDOUR, R. D. (1985). *Acta Cryst.* C**41**, 1327–1329.

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Structure of (3*R*,6*S*,12b*R*)-6-Cyanomethyl-3-ethyl-2-oxo-1,2,3,4,7,7a,12a,12b-octahydro-6*H*-indolo[2,3-*a*]quinolizine

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Abstract. $C_{19}H_{21}N_3O$, $M_r = 307.40$, monoclinic, $P2_1/c$, $a = 12.200\ (7)$, $b = 16.795\ (2)$, $c = 16.655\ (1)$ Å, $\beta = 104.18\ (3)°$, $V = 3308\ (3)$ Å^3, $Z = 8$, $D_x = 1.234$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.73$ cm^{-1}, $F(000) = 1312$, $T = 296$ K, final $R = 0.051$ for 2227 independent observed reflections. The two

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