Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title compound, with thermal ellipsoids drawn at the 40% probability level.
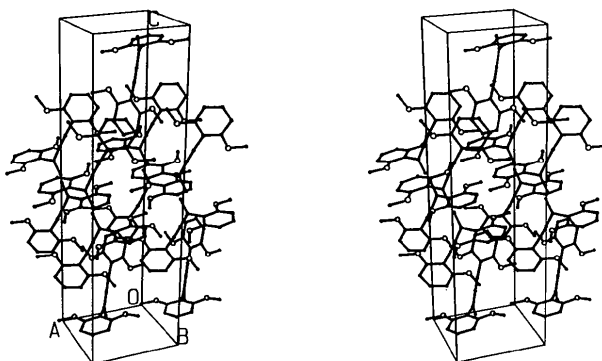

Fig. 2. Stereoview of the unit cell, viewed slightly oblique to the *b* axis, with *a* horizontal.

References

ABRAMS, S. R., QUAIL, J. W. & DELBAERE, L. T. J. (1983). *Can. J. Chem.* **61**, 2449–2454.

BEGLEY, M. J., KNIGHT, D. W. & PATTENDEN, G. (1975). *J. Chem. Soc. Perkin Trans.* 2, pp. 1863–1866.

CHURCHILL, M. R. & JULIS, S. A. (1981). *Cryst. Struct. Commun.* **10**, 1375–1379.

CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

DESTRO, R., MERATI, F. & ORTOLEVA, E. (1989). *J. Mol. Struct.* **193**, 121–131.

EVANS, K. L., HORN, G. W., FRONCZEK, F. R., GANDOUR, R. D. & WATKINS, S. F. (1990). *Acta Cryst.* C46, 331–332.

FRENZ, B. A. & OKAYA, Y. (1980). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

FUKUYAMA, T., KUCHITSU, K. & MORINO, Y. (1969). *Bull. Chem. Soc. Jpn*, **42**, 379–382.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

LARSON, A. C. (1969). *Crystallographic Computing*, edited by F. R. AHMED, S. R. HALL & C. P. HUBER, pp. 291–294. Copenhagen: Munksgaard.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

TROST, B. M., CHEN, C. & RUHTER, G. (1987). *J. Am. Chem. Soc.* **109**, 3486–3487.

Methyl 2-(2,6-Dimethoxyphenyl)benzofuran-4-carboxylate

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Abstract. $C_{18}H_{16}O_5$, $M_r = 312.3$, orthorhombic, *Pbca*, $a = 11.4064$ (7), $b = 13.098$ (2), $c = 20.848$ (2) Å, $V = 3114.8$ (6) Å^3, $Z = 8$, $D_x = 1.332$ g cm^{-3} at 293 K, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 7.68$ cm^{-1}, $F(000) = 1312$, 3203 unique data measured, final $R = 0.040$ for 2738 reflections with $I > 3.0\sigma(I)$. Maximum deviation from planarity of the ring containing the two methoxyl substituents is 0.004 (2) Å, and the maximum deviation from planarity of the benzofuran is 0.0259 (12) Å. The dihedral angle formed between the benzofuran and the ring bearing the two methoxyl groups is 62.39 (3)°. Both of the methoxyl substituents are nearly coplanar with the ring, and *anti* to the benzofuran. The torsional deviations are 2.3 (2)° for the methoxyl group nearest the benzofuran oxygen and 5.8 (2)° for the other. The ester substituent is nearly coplanar with the benzofuran ring system with a

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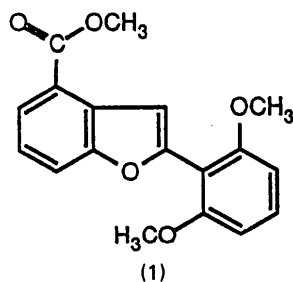* To whom correspondence should be addressed.

$C_{18}H_{16}O_5$

Table 1. *Coordinates and isotropic thermal parameters*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å²)
O1	0.5030 (1)	0.23191 (8)	0.22157 (5)	5.50 (2)
O2	0.58793 (8)	−0.06049 (8)	0.10297 (5)	4.85 (2)
O3	0.36702 (7)	−0.00809 (6)	0.16321 (5)	3.69 (2)
O4	0.0253 (1)	0.2099 (1)	0.00188 (7)	7.63 (3)
O5	0.1939 (1)	0.27106 (8)	0.03931 (5)	5.21 (2)
C1	0.5874 (1)	0.1643 (1)	0.20319 (7)	4.17 (3)
C2	0.7053 (1)	0.1700 (1)	0.22142 (8)	5.44 (3)
C3	0.7824 (1)	0.0971 (2)	0.20006 (8)	5.77 (4)
C4	0.7476 (1)	0.0187 (1)	0.16145 (8)	5.06 (3)
C5	0.6308 (1)	0.0128 (1)	0.14268 (6)	3.85 (3)
C6	0.5493 (1)	0.0853 (1)	0.16335 (6)	3.44 (2)
C7	0.4262 (1)	0.07859 (9)	0.14299 (6)	3.21 (2)
C8	0.3568 (1)	0.13996 (9)	0.10832 (6)	3.35 (2)
C9	0.2438 (1)	0.09109 (9)	0.10499 (6)	3.33 (2)
C10	0.2556 (1)	0.0007 (1)	0.13942 (6)	3.53 (2)
C11	0.1689 (1)	−0.0715 (1)	0.14727 (8)	4.65 (3)
C12	0.0624 (1)	−0.0510 (1)	0.11747 (9)	5.25 (3)
C13	0.0466 (1)	0.0373 (1)	0.08246 (8)	4.87 (3)
C14	0.1348 (1)	0.1098 (1)	0.07536 (6)	3.88 (3)
C15	0.1105 (1)	0.2007 (1)	0.03520 (7)	4.60 (3)
C16	0.5353 (2)	0.3083 (2)	0.2671 (1)	7.39 (5)
C17	0.6683 (2)	−0.1337 (2)	0.0772 (1)	6.71 (4)
C18	0.1799 (2)	0.3612 (1)	0.0001 (1)	6.96 (5)

torsional deviation of 9.8 (2)° for the carbonyl oxygen. The C=C double-bond distance is 1.340 (2) Å.

Experimental. A colorless crystal of (1) was grown by evaporation from acetonitrile from the crude reaction of methyl 2-[(2,6-dimethoxyphenyl)ethynyl]-3-methoxybenzoate with boron trichloride. Crystal size

(1)

0.22 × 0.35 × 0.38 mm, space group from systematic absences 0*kl* with *k* odd, *h0l* with *l* odd, and *hk*0 with *h* odd, cell dimensions from setting angles of 25 reflections having 25 < θ < 30°. Data collection on an Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 25\sigma(I)$, subject to max. scan time = 90s, scan rates varied 0.61–3.3° min⁻¹. One octant of data having 2 < θ < 75°, 0 ≤ *h* ≤ 14, 0 ≤ *k* ≤ 16, 0 ≤ *l* ≤ 26 measured. Data corrected for background, Lorentz and polarization effects. The standard reflections 200, 040, 006 varied randomly, and no decay correction was applied. Absorption corrections were based on ψ scans, with relative transmission coefficients ranging from 0.9259 to 0.9998. 3203 unique data were collected with 2738 observed with *I*

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

O1—C1	1.363 (2)	C4—C5	1.391 (2)
O1—C16	1.428 (2)	C5—C6	1.396 (2)
O2—C5	1.358 (2)	C6—C7	1.469 (2)
O2—C17	1.431 (2)	C7—C8	1.340 (2)
O3—C7	1.387 (1)	C8—C9	1.440 (2)
O3—C10	1.369 (2)	C9—C10	1.391 (2)
O4—C15	1.200 (2)	C9—C14	1.410 (2)
O5—C15	1.328 (2)	C10—C11	1.379 (2)
O5—C18	1.444 (2)	C11—C12	1.391 (2)
C1—C2	1.399 (2)	C12—C13	1.380 (2)
C1—C6	1.397 (2)	C13—C14	1.392 (2)
C2—C3	1.372 (2)	C14—C15	1.481 (2)
C3—C4	1.364 (3)		

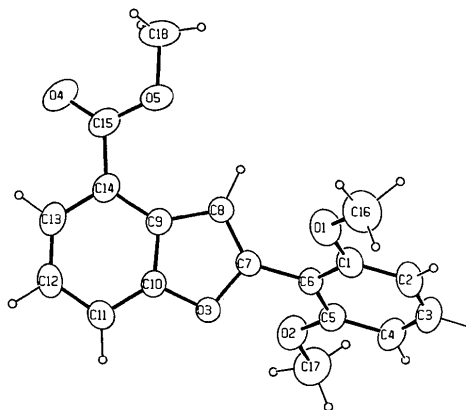|			
C1—O1—C16	117.4 (1)	C6—C7—C8	133.2 (1)
C5—O2—C17	118.2 (1)	C7—C8—C9	106.8 (1)
C7—O3—C10	105.85 (9)	C8—C9—C10	105.5 (1)
C15—O5—C18	116.8 (1)	C8—C9—C14	137.1 (1)
O1—C1—C2	124.6 (1)	C10—C9—C14	117.4 (1)
O1—C1—C6	115.4 (1)	O3—C10—C9	110.4 (1)
C2—C1—C6	120.0 (1)	O3—C10—C11	124.4 (1)
C1—C2—C3	119.4 (2)	C9—C10—C11	125.2 (1)
C2—C3—C4	121.9 (1)	C10—C11—C12	116.2 (1)
C3—C4—C5	119.2 (1)	C11—C12—C13	120.8 (1)
O2—C5—C4	123.7 (1)	C12—C13—C14	122.3 (1)
O2—C5—C6	115.4 (1)	C9—C14—C13	118.2 (1)
C4—C5—C6	120.8 (1)	C9—C14—C15	123.6 (1)
C1—C6—C5	118.7 (1)	C13—C14—C15	118.2 (1)
C1—C6—C7	120.9 (1)	O4—C15—O5	123.2 (1)
C5—C6—C7	120.4 (1)	O4—C15—C14	124.0 (1)
O3—C7—C6	115.2 (1)	O5—C15—C14	112.8 (1)
O3—C7—C8	111.5 (1)		

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title compound, with thermal ellipsoids drawn at the 40% probability level.
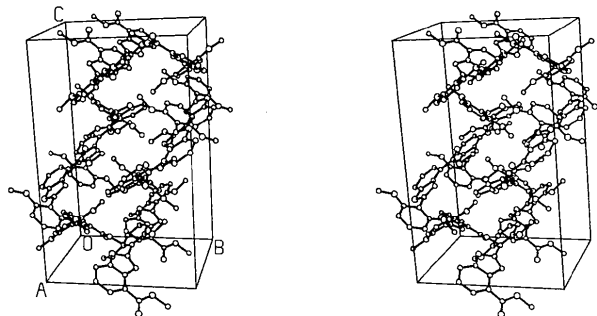
Fig. 2. Stereoview of the unit cell, viewed slightly oblique to the *a* axis, with *b* horizontal.

$> 3\sigma(I)$. Structure solved by direct methods, using *RANTAN* (Yao, 1981), refined by full-matrix least-squares based upon F with weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using the Enraf–Nonius package *SDP/VAX* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-H atoms refined anisotropically; H atoms located from difference maps and refined isotropically. Final $R = 0.040$ (0.047 for all data), $wR = 0.056$, $S = 3.057$ for 273 variables. Maximum shift 0.02σ in the final cycle, max. residual density 0.13, min. -0.15 e Å^{-3}, extinction coefficient (Larson, 1969) $g = 1.16 (5) \times 10^{-6}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c; maximum correction 33.8% for the 024 reflection.

Coordinates* and equivalent isotropic thermal parameters are given in Table 1; bond distances and angles are given in Table 2. The molecule is illustrated in Fig. 1, and the unit cell is shown in Fig. 2.

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* Lists of H-atom positional parameters, bond distances and angles involving H, torsion angles, least-squares planes, anisotropic thermal parameters, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54144 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Structures with 2-arylbenzofuran skeleton: 2-(2′-hydroxy-4′-methoxyphenyl)benzofuran-6-ol: Jurd & Wong (1984); albanol A methyl ether: Rama Rao, Deshpande, Shastri, Tavale & Dhaneshwar (1983); daphnodorin A pentamethyl ether: Baba, Takeuchi, Doi, Inoue & Kozawa (1986).

References

BABA, K., TAKEUCHI, K., DOI, M., INOUE, M. & KOZAWA, M. (1986). *Chem. Pharm. Bull.* **34**, 1540–1545.

CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

FRENZ, B. A. & OKAYA, Y. (1980). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

JURD, L. & WONG, R. Y. (1984). *Aust. J. Chem.* **37**, 1127–1133.

LARSON, A. C. (1969). *Crystallographic Computing*, edited by F. R. AHMED, S. R. HALL & C. P. HUBER, pp. 291–294. Copenhagen: Munksgaard.

RAMA RAO, A. V., DESHPANDE, V. H., SHASTRI, R. K., TAVALE, S. S. & DHANESHWAR, N. N. (1983). *Tetrahedron Lett.* **24**, 3013–3016.

YAO, J. (1981). *Acta Cryst.* A**37**, 642–644.

Acta Cryst. (1991). C**47**, 2733–2735

Crystal Studies of Heterocyclic Compounds Containing One Oxygen and Two Nitrogen Atoms. XVI. 6,8-Ditosyl-6,7,8,9-tetrahydrodibenzo[*b,h*][1,4,6]oxadiazonine

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Abstract. $C_{28}H_{26}N_2O_5S_2$, $M_r = 534\cdot7$, orthorhombic, $P2_12_12_1$, $a = 8\cdot939 (1)$, $b = 11\cdot67 (1)$, $c = 24\cdot968 (2)$ Å, $V = 2604\cdot6 (6)$ Å^3, $Z = 4$, $D_x = 1\cdot364 (1)$ g cm^{-3}, λ(Cu $K\alpha$) $= 1\cdot54178$ Å, $\mu = 20\cdot82$ cm^{-1}, $F(000) = 1120$, room temperature, $R = 0\cdot0395$ for 3767 reflections with $I > 3\sigma(I)$. The nine-membered heterocyclic ring is in a chair-like conformation. There is an *exo, exo* conformation of tosyl groups.

Experimental. Colourless crystals $0\cdot2 \times 0\cdot3 \times 0\cdot4$ mm from ethanol at room temperature. CAD-4 diffractometer using θ–2θ scan technique, graphite-monochromatized Cu $K\alpha$ radiation, data not corrected for absorption. Unit-cell parameters from 25 reflections in the θ range $16\cdot4$–$45\cdot0°$. Total of 4558 independent reflections measured to $(\sin\theta)/\lambda = 0\cdot63$ Å^{-1}, $R_{\text{int}} = 3\cdot52\%$, range of h, k, l: -10 to 10, 0 to 12, 0 to 26, respectively; standard reflection 1,3,10, max. change $2\cdot4\%$, 3767 reflections with $I > 3\sigma(I)$ used in calculation. Solution by direct methods using *SHELX*76 (Sheldrick, 1976), all H atoms located from a difference map, refinement by a full-matrix least-squares procedure on F magnitudes, 438 parameters. Refinement to final $R = 0\cdot0395$, unit weights, $S = 0\cdot880$. Absolute configuration of the