were located on a difference Fourier map at an advanced stage of anisotropic refinement and their coordinates refined. The use of more than one refinement block affords a method of origin fixing. $\sum w(\Delta F)^2$ minimized, $w = [\sigma^2(F_o) + 0{\cdot}00269(F_o)^2]^{-1}$, where σ is standard deviation of observed amplitudes, based on counting statistics; isotropic extinction parameter $X = 0{\cdot}0020$. In the last cycle $(\Delta/\sigma)_{max} = 0{\cdot}65$; $\Delta\rho$ from $-0{\cdot}24$ to $0{\cdot}39$ e Å^{-3}, $S = 1{\cdot}19$; final $R = 0{\cdot}044$, $wR = 0{\cdot}065$; scattering factors from *International Tables for X-ray Crystallography* (1974). All computations were performed on a Nova 4S computer and plots drawn on a Tektronix plotter with the *SHELXTL* system of programs.

Atomic coordinates are in Table 1.* A perspective molecular drawing and the atom labelling are displayed in Fig. 1. Bond distances and angles are listed in Table 2.

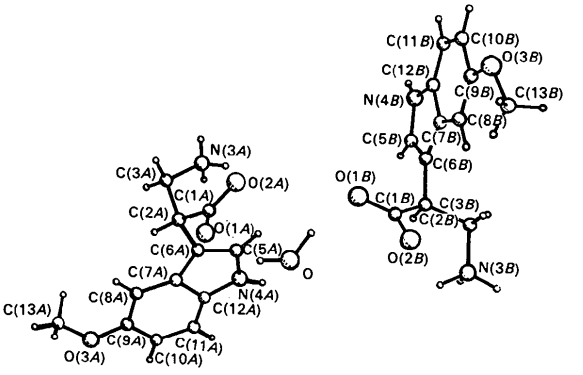

Fig. 1. The molecular structure of the title compound with atom numbering.

Related literature. The title compound has been prepared as part of an investigation on a series of tryptophan analogues having the carboxyl function at the β position and with antihypertensive activity (Safdy, Kurchacova, Schut, Vidrio & Hong, 1982). It was of interest to study the crystallographic structure of this compound in order to ascertain its conformation and molecular geometry.

We thank Mrs Cynthia Lesh de S. for technical assistance, and Dr Enrique Hong of Instituto Miles de Terapeutica Experimental, Mexico, for providing the sample.

* Lists of structure amplitudes, anisotropic thermal parameters, H-atom coordinates and least-squares-planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51837 (13 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 a 2,7-Dinitro-9-fluorenone and 2,2′-Bis-1,3-dithiole (DNF–TTF) Complex*

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Abstract. $C_{19}H_{10}N_2O_5S_4$, $M_r = 474{\cdot}5$, monoclinic, $P2_1$, $a = 7{\cdot}243$ (1), $b = 12{\cdot}135$ (3), $c = 11{\cdot}219$ (4) Å, $\beta = 103{\cdot}36$ (2)°, $V = 959$ (1) Å^3, $Z = 2$, $D_x = 1{\cdot}64$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 4{\cdot}817$ mm^{-1}, $F(000) = 484$, $T = 300$ K, $R = 0{\cdot}027$ for 1222 observed reflections. The bond lengths and angles of TTF agree with those of the un-

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Table 1. *Positional and equivalent isotropic thermal parameters and their e.s.d.'s*

$B_{eq} = (8\pi^2/3)[U_{22} + (1/\sin^2\beta)(U_{11} + U_{33} + 2U_{13}\cos\beta)]$.

	x	y	z	B_{eq}(Å^2)
S1	0·5948 (2)	0·0492	0·2488 (1)	4·02 (7)
S2	0·4204 (2)	0·2040 (2)	0·0524 (1)	3·87 (7)
S3	0·4254 (2)	0·3957 (2)	0·2530 (1)	4·02 (7)
S4	0·5960 (2)	0·2417 (2)	0·4519 (1)	3·65 (7)
O1	1·1096 (6)	0.0059 (4)	0·2569 (4)	4·5 (2)
O2	1·2104 (9)	0·2982 (5)	0·7649 (4)	7·1 (3)
O3	1·265 (1)	0·1309 (5)	0·7260 (4)	7·4 (3)
O4	0·8598 (8)	0·1077 (5)	−0·2234 (4)	6·6 (3)
O5	0·705 (1)	0·2584 (5)	−0·2582 (5)	7·7 (3)
N1	1·2077 (8)	0·2235 (6)	0·6914 (5)	4·7 (3)
N2	0·8069 (8)	0·1947 (6)	−0·1890 (5)	4·6 (3)
C1	0·556 (1)	0·0081 (8)	0·0986 (8)	4·8 (4)
C2	0·477 (1)	0·0744 (6)	0·0113 (7)	4·5 (3)
C3	0·5061 (8)	0·1843 (6)	0·2095 (5)	3·2 (3)
C4	0·5089 (8)	0·2636 (6)	0·2953 (6)	3·3 (3)
C5	0·484 (1)	0·4466 (6)	0·4018 (6)	4·0 (3)
C6	0·560 (1)	0·3794 (6)	0·4916 (6)	4·1 (3)
C7	1·0621 (8)	0·1011 (5)	0·2536 (5)	3·2 (3)
C8	1·0752 (7)	0·1774 (5)	0·3607 (5)	2·8 (3)
C9	1·1387 (9)	0·1562 (6)	0·4828 (5)	3·4 (3)
C10	1·1388 (8)	0·2437 (6)	0·5629 (5)	3·5 (3)
C11	1·0775 (9)	0·3480 (6)	0·5234 (6)	3·8 (3)
C12	1·0152 (9)	0·3679 (6)	0·3985 (5)	3·7 (3)
C13	1·0140 (7)	0·2833 (5)	0·3173 (5)	2·6 (2)
C14	0·9590 (7)	0·2801 (5)	0·1822 (5)	2·9 (2)
C15	0·8896 (9)	0·3591 (5)	0·0972 (5)	3·6 (3)
C16	0·844 (1)	0·3319 (6)	−0·0259 (6)	3·7 (3)
C17	0·8658 (8)	0·2238 (6)	−0·0609 (5)	3·3 (3)
C18	0·939 (1)	0·1417 (6)	0·0229 (6)	3·4 (3)
C19	0·9826 (8)	0·1707 (5)	0·1434 (4)	2·9 (3)

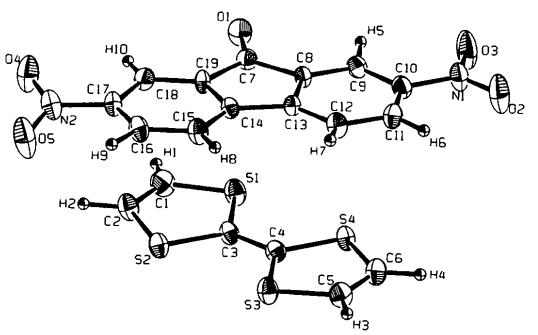

Fig. 1. The molecular conformation of the title compound, showing atom numbering. The thermal ellipsoids are drawn at the 50% probability level.

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

S1—C1	1·717 (9)	S1—C3	1·779 (7)
S2—C2	1·716 (8)	S2—C3	1·744 (6)
S3—C5	1·738 (7)	S3—C4	1·740 (7)
S4—C4	1·745 (6)	S4—C6	1·764 (8)
O1—C7	1·204 (7)	O2—N1	1·222 (8)
O3—N1	1·229 (8)	O4—N2	1·216 (8)
O5—N2	1·217 (8)	N1—C10	1·434 (8)
N2—C17	1·446 (8)	C1—C2	1·290 (10)
C3—C4	1·358 (7)	C5—C6	1·310 (10)
C7—C19	1·498 (8)	C7—C8	1·502 (8)
C8—C9	1·365 (8)	C8—C13	1·409 (8)
C9—C10	1·390 (10)	C10—C11	1·380 (10)
C11—C12	1·390 (8)	C12—C13	1·372 (8)
C13—C14	1·475 (8)	C14—C15	1·364 (8)
C14—C19	1·420 (9)	C15—C16	1·384 (8)
C16—C17	1·390 (10)	C17—C18	1·389 (9)
C18—C19	1·362 (8)		

C1—S1—C3	93·2 (4)	C2—S2—C3	95·4 (4)
C5—S3—C4	94·1 (3)	C4—S4—C6	94·0 (3)
O2—N1—O3	120·9 (6)	O2—N1—C10	119·9 (7)
O3—N1—C10	119·2 (6)	O4—N2—O5	122·6 (6)
O4—N2—C17	119·0 (7)	O5—N2—C17	118·4 (7)
C2—C1—S1	120·5 (7)	C1—C2—S2	117·2 (6)
C4—C3—S2	124·3 (4)	C4—C3—S1	122·1 (4)
S2—C3—S1	113·6 (4)	C3—C4—S3	120·8 (4)
C3—C4—S4	123·3 (4)	S3—C4—S4	115·9 (4)
C6—C5—S3	118·9 (6)	C5—C6—S4	117·0 (6)
O1—C7—C19	128·2 (6)	O1—C7—C8	127·0 (5)
C19—C7—C8	104·7 (5)	C9—C8—C13	121·7 (6)
C9—C8—C7	129·1 (6)	C13—C8—C7	109·2 (4)
C8—C9—C10	117·0 (6)	C11—C10—C9	122·7 (6)
C11—C10—N1	119·5 (6)	C9—C10—N1	117·8 (6)
C10—C11—C12	119·2 (6)	C13—C12—C11	119·3 (6)
C12—C13—C8	120·0 (5)	C12—C13—C14	131·3 (5)
C8—C13—C14	108·7 (5)	C15—C14—C19	119·6 (5)
C15—C14—C13	131·9 (6)	C19—C14—C13	108·4 (5)
C14—C15—C16	119·6 (6)	C15—C16—C17	119·4 (6)
C16—C17—C18	122·5 (6)	C16—C17—N2	119·0 (6)
C18—C17—N2	118·5 (6)	C19—C18—C17	116·9 (7)
C18—C19—C14	122·0 (6)	C18—C19—C7	129·1 (6)
C14—C19—C7	108·9 (5)		

complexed molecule. The overall TTF molecule is not planar but is slightly distorted: the dihedral angle between the S(3)—C(4)—S(4)—C(6)—C(5)/S(1)—C(1)—C(2)—S(2)—C(3) rings is 2·7 (1)°. The DNF molecule is planar. The nitro groups at C(10) and C(17) are twisted out of the DNF plane by 1·4 (3) and 15·9 (2)°, respectively. The DNF–TTF stack alternately in infinite columns, parallel to the *a* axis, the spacing and dihedral angle between the planes being 3·52 (1) Å and 2·6 (1)°, respectively.

Experimental. Crystals of the title compound were obtained by mixing 2,7-dinitro-9-fluorenone with equivalent amounts of 2,2′-bis-1,3-dithiole, in hot acetonitrile. Size of crystal 0·16 × 0·16 × 0·62 mm. Nicolet *R*3 four-circle diffractometer. Unit-cell parameters from 24 machine-centred reflections with $10{\cdot}9 < 2\theta < 28{\cdot}8°$, 2198 reflections measured with $3 < 2\theta < 110°$ for two octants, of which 1222 had $I > 3{\cdot}0\sigma(I)$ and used in the refinement. Index range h 0→7, k 0→12, l ±11, $2\theta/\theta$ scan mode, variable scan speed. Two standard reflections ($02\bar{1}$, $\bar{1}1\bar{1}$) monitored every 50 measurements, no significant variation. Intensities were corrected for Lorentz–polarization but not for absorption. $R_{int} = 0{\cdot}020$. Structure solved by direct methods using the *TEXSAN* (Molecular Structure Corporation, 1988) structure analysis package. Full-matrix least-squares refinement to minimize the function $\sum\sigma_F^2(|F_o| - |F_c|)^2$, in which $\sigma_F = \sigma_I/2F$ Lp, for the unique reflections. The results for the final refinement cycle were: 1222 independent observations; 311 variables; $R = 0{\cdot}027$; $wR = 0{\cdot}034$; $S = 4{\cdot}6$; $(\Delta/\sigma)_{max} - 0{\cdot}43$. Maximum and minimum peaks on the final electron density difference map had values +0·24 and −0·22 e Å^{-3}, respectively.

The isotropic extinction parameter $X = 4{\cdot}48 \times 10^{-5}$ (Zachariasen, 1968). Atomic scattering factors and anomalous-dispersion corrections from Cromer & Waber (1974). Atomic coordinates are in Table 1.* Fig. 1 shows the molecular conformation of the complex. Bond distances and angles are listed in Table 2.

Related literature. The discovery of metallic conductivity in the complex of 2,2′-bis-1,3-dithiole (tetrathiafulvalene) and tetracyanoquinodimethane (TTF–TCNQ) prompted a great interest in the synthesis and properties of related organic charge transfer complexes (Pal, Gruner, Janossy & Solyon, 1977; Alcacer, 1980).

We thank Mrs Cynthia Lesh de S. for technical assistance.

* Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51838 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(Chloro-5 indolyl-3)-2 Hydroxy-2 Indanedione-1,3 Oxyde de Diéthyle Monohydrate (1/1/1)

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Abstract. $C_{17}H_{10}ClNO_3.C_4H_{10}O.H_2O$, $M_r = 403{\cdot}9$, triclinic, $P\bar{1}$, $a = 8{\cdot}080$ (2), $b = 7{\cdot}167$ (2), $c = 19{\cdot}174$ (8) Å, $\alpha = 109{\cdot}00$ (4), $\beta = 93{\cdot}07$ (3), $\gamma = 99{\cdot}05$ (3)°, $V = 1030$ (2) Å^3, $Z = 2$, $D_x = 1{\cdot}302$ Mg m^{-3}, λ(Cu $K\bar{\alpha}$) = 1·5418 Å, $\mu = 1{\cdot}91$ mm^{-1}, $F(000) = 424$, $T = 295$ (1) K, $R = 0{\cdot}044$ for 2097 independent reflections. All molecular parameters agree with those obtained for related compounds. The indolyl group is planar but, in the indanedione moiety, the five-membered ring shows significant deviations from planarity. The dihedral angle between the least-squares planes of the two groups is 79·95 (7)°. The chain of the diethyl ether molecule is also planar. The structure can be regarded as constituted of layers which spread out along the (001) planes. The molecules belonging to the same layer are linked together by hydrogen bonds O—H···O and N—H···O. 2-(5-Chloro-3-indolyl)-2-hydroxy-1,3-indanedione has antiinflammatory properties. The present structure was solved in order to compare its molecular geometry with those of 2-azaarylindane-1,2-diones which have similar activity.

Partie expérimentale. Préparation par condensation du chloro-5 indole sur la dihydroxy-2,2 indanedione-1,3 en milieu hétérogène eau–oxyde de diéthyle. Cristaux jaunes fondant à 461 K. Cristal lamellaire: 0,34 × 0,37 × 0,04 mm. Dimensions de la maille déterminées avec 25 réflexions telles que $12{,}92 \le \theta \le 32{,}39°$. Diffractomètre Enraf–Nonius CAD-4. $0{,}023 \le (\sin\theta)/\lambda \le 0{,}550$ Å^{-1}. $-8 \le h \le 8$; $0 \le k \le 7$; $-21 \le l \le 19$. Réflexions de contrôle de l'intensité: 04$\bar{6}$, 025 et 035. Diminution de I au cours des mesures: 1,8%. $\sigma(I)/I$ moyen (contrôle): 0,0020. 2862 réflexions indépendantes mesurées, 765 réflexions inobservées [$I < 2\sigma(I)$]. Méthodes directes, programme *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). H liés à O(9), C(19) et C(23): série de Fourier des ΔF. Autres H:

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