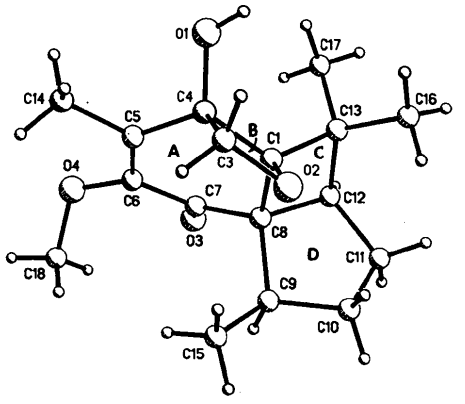

Fig. 1. Molecular structure of the title compound with atom numbering.

45°, of which 991 with $I > 2{\cdot}5\sigma(I)$ were used in the analysis. Index range $h\ 0 \rightarrow 14$, $k\ 0 \rightarrow 15$, $l\ 0 \rightarrow 8$, ω scan and variable scan speed. Two standard reflections ($0\bar{2}0$, $\bar{1}12$) monitored every 50 measurements, no significant variation. Lp correction, absorption ignored, $R_{int} = 0{\cdot}032$. Structure solved by direct methods using *SHELXTL5* (Sheldrick, 1985). Non-H atoms were treated anisotropically in least squares; H atoms in calculated positions riding on bonded C with a fixed isotropic temperature factor, $U = 0{\cdot}06 Å^2$, hydroxyl H-atom positions (from ΔF map) refined. $\sum w(\Delta F)^2$ minimized, $w = [\sigma^2(F_o) + 0{\cdot}002(F_o)^2]^{-1}$, where σ is standard deviation of observed amplitudes, based on counting statistics; isotropic extinction parameter $X = 0{\cdot}0038$. In the last cycle $(\Delta/\sigma)_{max} = 0{\cdot}080$; $\Delta\rho$ from $-0{\cdot}16$ to $0{\cdot}20$ e $Å^{-3}$, $S = 1{\cdot}07$; final $R = 0{\cdot}049$, $R_w = 0{\cdot}054$ and $wR = 0{\cdot}063$. Scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on a Nova 4S computer and plots drawn on a Tektronix plotter with the *SHELXTL* system of programs.

The atomic coordinates are given in Table 1.* A perspective molecular drawing, together with the atom-numbering scheme, is displayed in Fig. 1. Bond distances, angles and selected torsion angles are listed in Table 2.

Related literature. As part of our studies on the ultraviolet irradiation of *O*-methylmethoxyperezone (Barrera, Barrios & Walls, 1980), the title compound was prepared. Its structure was investigated by chemical methods and could not be established unambiguously by ^{13}C NMR (Barrios, Salazar, Díaz, Walls & Joseph-Nathan, 1986).

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 51835 (9 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 3-Amino-2-(6-methoxy-3-indolyl)propionic Acid Hemihydrate*

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Abstract. $C_{12}H_{14}N_2O_3.0{\cdot}5H_2O$, $M_r = 243{\cdot}3$, orthorhombic, $Pna2_1$, $a = 9{\cdot}986\ (2)$, $b = 37{\cdot}117\ (7)$, $c = 6{\cdot}578\ (1)$ Å, $V = 2438\ (1)$ Å^3, $Z = 8$, $D_x = 1{\cdot}32$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0{\cdot}780$ mm^{-1}, $F(000) = 1032$, $T = 293$ K, $R = 0{\cdot}044$ for 1588 observed reflections. The distances and angles of the indole rings agree well with those

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0108-2701/89/091440-03$03.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($Å^2 \times 10^3$)

$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$.

	x	y	z	U_{eq}
O(1*A*)	7916 (3)	7219 (1)	1563 (7)	55 (1)
O(2*A*)	6099 (4)	7530 (1)	828 (9)	65 (2)
C(1*A*)	6767 (4)	7249 (1)	817 (10)	43 (2)
C(2*A*)	6157 (5)	6912 (1)	−190 (9)	35 (2)
C(3*A*)	5349 (5)	7007 (2)	−2102 (9)	42 (2)
N(3*A*)	4124 (4)	7216 (1)	−1534 (8)	46 (2)
N(4*A*)	3787 (4)	6575 (1)	3685 (8)	53 (2)
C(5*A*)	4440 (5)	6846 (2)	2724 (9)	44 (2)
C(6*A*)	5321 (4)	6704 (1)	1321 (9)	36 (2)
C(7*A*)	5187 (4)	6321 (1)	1409 (8)	36 (1)
C(8*A*)	5808 (4)	6036 (1)	378 (9)	39 (1)
C(9*A*)	5470 (5)	5685 (1)	921 (10)	49 (2)
C(10*A*)	4532 (6)	5621 (2)	2465 (11)	58 (2)
C(11*A*)	3896 (5)	5896 (2)	3464 (11)	64 (2)
C(12*A*)	4225 (5)	6247 (2)	2941 (9)	47 (2)
O(3*A*)	5996 (4)	5382 (1)	49 (8)	63 (2)
C(13*A*)	6879 (5)	5427 (1)	−1614 (11)	72 (2)
O(1*B*)	1360 (4)	6802 (1)	−4251 (7)	60 (1)
O(2*B*)	2134 (3)	6700 (1)	−1174 (6)	65 (1)
C(1*B*)	1216 (5)	6717 (1)	−2452 (9)	38 (2)
C(2*B*)	−214 (4)	6613 (1)	−1711 (8)	34 (2)
C(3*B*)	−191 (5)	6480 (1)	485 (9)	42 (2)
N(3*B*)	138 (4)	6775 (1)	1902 (7)	42 (1)
N(4*B*)	−2213 (3)	6129 (1)	−5549 (6)	47 (1)
C(5*B*)	−1973 (4)	6418 (1)	−4316 (8)	43 (2)
C(6*B*)	−851 (4)	6359 (1)	−3155 (7)	36 (1)
C(7*B*)	−377 (4)	6006 (1)	−3718 (8)	36 (1)
C(8*B*)	723 (4)	5798 (1)	−3082 (9)	43 (2)
C(9*B*)	911 (5)	5465 (1)	−3966 (9)	47 (2)
C(10*B*)	28 (6)	5336 (2)	−5454 (10)	58 (2)
C(11*B*)	−1066 (5)	5534 (1)	−6071 (10)	53 (2)
C(12*B*)	−1241 (4)	5871 (1)	−5196 (7)	44 (1)
O(3*B*)	1934 (3)	5236 (1)	−3531 (6)	63 (1)
C(13*B*)	2779 (5)	5328 (1)	−1846 (11)	73 (2)
O	2610 (4)	7486 (1)	−4635 (6)	63 (1)

observed in other indole structures. Both of the indole rings show small distortions from planarity with maximum out-of-plane deviations of 0·012 (6), 0·013 (7), 0·022 (5), 0·028 (7) Å for C(11*B*), C(10*B*), C(6*A*), C(10*A*), respectively. The side chains at C(6) are twisted out of the plane of the indole rings, the torsion angles are C(3*A*)—C(2*A*)—C(6*A*)—C(5*A*) = −82·8 (7)° and C(3*B*)—C(2*B*)—C(6*B*)—C(5*B*) = 118·3 (5)°, while the C(9)—methoxy groups are nearly coplanar with C(8*A*)—C(9*A*)—O(3*A*)—C(13*A*) = −4·4 (8)° and C(8*B*)—C(9*B*)—O(3*B*)—C(13*B*) = 8·4 (7)°. The angle between the best planes through the indole ring and the carboxyl group in molecules *A* and *B* is 71·6 (2) and 97·2 (1)°, respectively. The crystal structure is stabilized by six intermolecular hydrogen bonds: N(3*B*)···O(1*A*) ($-1+x$, y, z) 2·774 (5); N(3*B*)···O(1*B*), N(4*A*)···O(1*B*)(x, y, $1+z$) 2·811 (6), 2·902 (6); N(3*B*)···O(2*A*), N(3*A*)···O(1*A*) ($-0{\cdot}5+x$, $1{\cdot}5-y$, z) 2·843 (6), 3.162 (6); O···O(1*A*) ($-0{\cdot}5+x$, $1{\cdot}5-y$, $-1{\cdot}0+z$) 2·746 (6) Å and seven intermolecular C—H···O and C—H···N interactions < 3·41 Å: C(11*B*)···O(3*B*) ($-x$, $1-y$, $-0{\cdot}5+z$) 3·398 (6); C(1*A*)···O ($0{\cdot}5+x$, $1{\cdot}5-y$, $1+z$) 3·258 (7); C(13*A*)···O(3*B*) ($1-x$, $1-y$, $0{\cdot}5+z$) 3·402 (7); C(3*A*)···O ($0{\cdot}5+x$, $1{\cdot}5-y$, z) 3·377 (7); C(8*A*)···N(4*B*), C(7*A*)···N(4*B*) ($1-x$, y, $1+z$) 3·347 (6), 3·354 (6); and C(3*B*)···O(1*A*) ($-1+x$, y, z) 3·406 (6) Å.

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

	Molecule *A*	Molecule *B*
O(1)—C(1)	1·252 (6)	1·233 (7)
O(2)—C(1)	1·236 (6)	1·245 (6)
C(1)—C(2)	1·541 (7)	1·558 (7)
C(2)—C(3)	1·535 (8)	1·527 (8)
C(2)—C(6)	1·512 (7)	1·483 (7)
C(3)—N(3)	1·495 (7)	1·474 (7)
N(4)—C(5)	1·354 (7)	1·367 (7)
N(4)—C(12)	1·386 (7)	1·383 (6)
C(5)—C(6)	1·380 (8)	1·374 (6)
C(6)—C(7)	1·427 (7)	1·439 (6)
C(7)—C(8)	1·402 (7)	1·406 (6)
C(7)—C(12)	1·420 (7)	1·393 (6)
C(8)—C(9)	1·394 (7)	1·379 (7)
C(9)—C(10)	1·402 (9)	1·402 (8)
C(9)—O(3)	1·365 (7)	1·360 (6)
C(10)—C(11)	1·371 (9)	1·377 (8)
C(11)—C(12)	1·385 (9)	1·388 (7)
O(3)—C(13)	1·415 (8)	1·434 (7)
O(1)—C(1)—O(2)	124·5 (5)	125·2 (5)
O(1)—C(1)—C(2)	117·3 (4)	118·0 (5)
O(2)—C(1)—C(2)	118·2 (4)	116·8 (5)
C(1)—C(2)—C(3)	111·9 (4)	111·2 (4)
C(1)—C(2)—C(6)	110·6 (5)	110·5 (4)
C(3)—C(2)—C(6)	111·5 (4)	114·0 (4)
C(2)—C(3)—N(3)	110·2 (5)	111·2 (4)
C(5)—N(4)—C(12)	109·7 (5)	108·7 (4)
N(4)—C(5)—C(6)	109·5 (5)	110·2 (4)
C(2)—C(6)—C(5)	126·5 (5)	127·0 (4)
C(2)—C(6)—C(7)	126·0 (5)	127·1 (4)
C(5)—C(6)—C(7)	107·2 (5)	105·8 (4)
C(6)—C(7)—C(8)	133·7 (5)	132·7 (4)
C(6)—C(7)—C(12)	106·6 (4)	107·7 (4)
C(8)—C(7)—C(12)	119·7 (4)	119·6 (4)
C(7)—C(8)—C(9)	118·4 (5)	118·2 (5)
C(8)—C(9)—C(10)	120·4 (5)	121·0 (5)
C(8)—C(9)—O(3)	124·7 (5)	125·0 (5)
C(10)—C(9)—O(3)	114·9 (5)	114·0 (4)
C(9)—C(10)—C(11)	122·0 (6)	121.5 (5)
C(10)—C(11)—C(12)	118·1 (6)	117·3 (5)
N(4)—C(12)—C(7)	107·0 (5)	107·6 (4)
N(4)—C(12)—C(11)	131·7 (5)	130·0 (4)
C(7)—C(12)—C(11)	121·3 (5)	122·4 (4)
C(9)—O(3)—C(13)	117·9 (4)	117·1 (4)

Experimental. The title compound was recrystallized from methanol and gave colourless crystals. Size of crystal 0·18 × 0·20 × 0·28 mm. Nicolet *R*3 four-circle diffractometer, Ni-filtered Cu $K\alpha$ radiation. Lattice parameters from 25 machine-centred reflections with $5{\cdot}7 < 2\theta < 24{\cdot}6°$. 1696 reflections with $3 < 2\theta < 110°$ for one octant, 1588 independent with $I > 2{\cdot}8\sigma(I)$, index range $h\ 0\rightarrow 10$, $k\ 0\rightarrow 39$, $l\ 0\rightarrow 6$, $2\theta/\theta$ scan mode, variable scan speed. Two standard reflections (0,0,10, 024) monitored every 50 measurements, no significant variation. Intensities were corrected for Lorentz–polarization but not for absorption. Data adjusted to an approximate absolute scale (overall $U = 0{\cdot}049$ Å^2). Structure solved by combination of direct methods and partial structure expansion by an iterative *E*-Fourier procedure using *SHELXTL5* (Sheldrick, 1985). Blocked-cascade least-squares refinement with all non-H atoms treated anisotropically; H atoms of CH, CH_2 and CH_3 were allowed to ride on bonded C with a fixed isotropic $U = 0{\cdot}06$ Å^2. The H atoms bonded to N and O atoms

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.

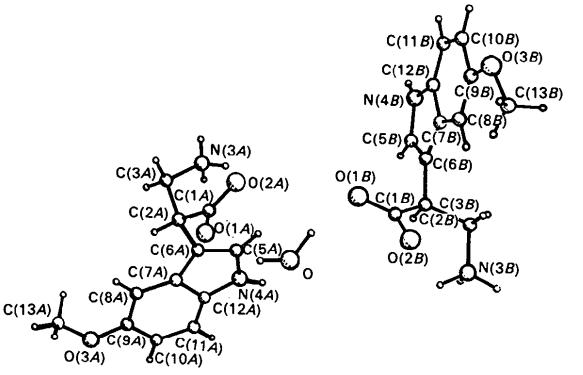

Fig. 1. The molecular structure of the title compound with atom numbering.

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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