There is localization of the double bonds C(6)=C(7) and C(8)=C(9), confirmed by their respective lengths, 1·345 (4) and 1·357 (3) Å. The furanone ring is planar within experimental error, with a maximum deviation of 0·018 (2) Å from the mean plane for C(1); the keto oxygen O(10) lies in this plane. The pyridinone ring is also planar, the maximum deviation being $-0{\cdot}014$ (2) Å for C(6); atoms O(11) and C(12) lie in the same plane. The dihedral angle between these two planes, 1·02 (6)°, means that the whole molecule, except the two methyl groups substituted at C(1), lie in a plane. These two methyl groups are on either side of this molecular plane with deviations of $-1{\cdot}188$ (2) and 1·320 (2) Å for C(13) and C(14) respectively.

The packing of the molecules in the unit cell viewed down the *c* axis is shown in Fig. 2. There are two intermolecular hydrogen bonds of the C—H···O type observed in this structure. C(6)···O(11$^{\rm i}$) = 3·409 (3), C(6)—H(6) = 1·04, H(6)···O(11$^{\rm i}$) 2·41 Å, C(6)—H(6)···O(11$^{\rm i}$) = 162°; C(7)···O(2$^{\rm ii}$) = 3·299 (3), C(7)—H(7) = 0·98, H(7)···O(2$^{\rm ii}$) = 2·35 Å, C(7)—H(7)··· O(2$^{\rm ii}$) = 162° [symmetry code: (i) $-x+\frac{1}{2},y-\frac{1}{2},z$; (ii) $-x+1,y-\frac{1}{2},z+\frac{1}{2}$]. These C—H···O hydrogen bonds are within the limits specified by Taylor & Kennard (1982).

The authors thank Dr Adibatty, Department of Pharmacology, Bangalore Medical College, Bangalore for providing the compound and Dr Balakrishna, Captain Srinivasamurthy Drug Research Institute, Madras, for useful discussions. The authors also thank RSIC, IIT, Madras, for data collection. One of the authors (KS) acknowledges financial assistance from UGC (India), New Delhi.

References

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P & Woolfson, M. M. (1980). *MULTAN*80. *A System of Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1986). *SHELXS*86. Program for crystal structure solution. Univ. of Göttingen, Federal Republic of Germany.

Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.

Acta Cryst. (1990). C**46**, 841–843

Structure of Gangetinin

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(*Received* 28 *June* 1989; *accepted* 15 *August* 1989)

Abstract. $C_{26}H_{26}O_5$, $M_r = 418{\cdot}5$, monoclinic, $P2_1$, $a = 7{\cdot}315$ (1), $b = 10{\cdot}774$ (1), $c = 14{\cdot}760$ (2) Å, $\beta = 102{\cdot}85$ (2)°, $V = 1134{\cdot}1$ (4) Å^3, $Z = 2$, $D_x = 1{\cdot}226$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 6{\cdot}47$ cm^{-1}, $F(000) = 444$, $T = 297$ K, final R value is 0·035 for 1780 observed reflections. Gangetinin contains six fused rings *A–F*. The pyrone rings *A* and *F* adopt half-chair conformations while the pyrone ring *C* is a distorted sofa. There are two planar benzene rings *B* and *E* and a five-membered furan ring *D* in an envelope conformation. The ring junction *C*/*D* shows *cis* fusion which induces a large (*ca* 37°) bend in the hexacyclic framework.

Introduction. Gangetinin, a pterocarpanoid, has been isolated from the roots of the plant *Desmodium gangeticum* (Purushothaman, Chandrasekharan, Balakrishna & Connolly, 1975) along with the other pterocarpanoids gangetin and desmodin (Purushothaman, Kishore, Narayanaswami & Connolly, 1971). These pterocarpanoids show anti-implantation and anti-inflammatory activities (Pillai, Alam & Purushothaman, 1981).

Experimental. Crystals from methanol, Enraf–Nonius CAD-4 diffractometer, crystal size 0·3 × 0·3 × 0·5 mm, graphite-monochromated Cu $K\alpha$ radiation, cell parameters from least-squares treatment of the setting angles of 25 reflections in the range $30 < \theta < 45°$; $\omega/2\theta$ scan technique, intensity variation of two standard reflections monitored every 98 reflections showed no significant variation, total number of reflections measured 2157 with $2\theta \leq 120°$, 1780 observed reflections with $I > 3\sigma(I)$, range of hkl: $h = -8$ to 8, $k = 0$ to 12, $l = 0$ to 17, intensity data corrected for Lp but not for absorption. Structure solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq &

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Table 1. *Positional parameters and equivalent isotropic thermal parameters*

$$B = \tfrac{8}{3}\pi^2(U_{11} + U_{22} + U_{33}).$$

	x	y	z	B_{eq}(Å^2)
O(1)	0·3000 (3)	0·4408	0·9380 (2)	5·33 (5)
C(2)	0·3826 (5)	0·4827 (4)	0·8685 (2)	4·40 (7)
C(3)	0·2745 (4)	0·5550 (4)	0·7992 (2)	4·22 (5)
C(4)	0·3558 (4)	0·6005 (4)	0·7299 (2)	3·94 (5)
O(5)	0·2432 (3)	0·6673 (4)	0·6609 (2)	4·53 (5)
C(6)	0·3113 (4)	0·6782 (4)	0·5774 (2)	4·39 (8)
C(7)	0·5091 (4)	0·7327 (4)	0·5970 (2)	4·06 (7)
C(8)	0·6394 (4)	0·6464 (4)	0·6649 (2)	3·83 (5)
C(9)	0·5468 (4)	0·5805 (4)	0·7306 (2)	3·55 (5)
C(10)	0·6469 (4)	0·5071 (4)	0·8023 (2)	3·88 (5)
C(11)	0·5712 (4)	0·4573 (4)	0·8715 (2)	4·18 (5)
C(12)	0·6772 (5)	0·3870 (5)	0·9501 (2)	5·25 (9)
C(13)	0·5877 (6)	0·3314 (5)	1·0077 (2)	5·76 (8)
C(14)	0·3777 (6)	0·3319 (5)	0·9909 (2)	5·77 (9)
C(15)	0·5947 (4)	0·7253 (4)	0·5136 (2)	3·90 (5)
C(16)	0·5792 (5)	0·7984 (4)	0·4345 (3)	4·63 (9)
C(17)	0·6817 (5)	0·7658 (5)	0·3690 (2)	4·69 (9)
C(18)	0·7956 (4)	0·6622 (5)	0·3822 (2)	4·25 (5)
O(19)	0·9037 (4)	0·6390 (4)	0·3192 (2)	5·22 (5)
C(20)	0·9678 (6)	0·5126 (5)	0·3105 (3)	5·69 (10)
C(21)	1·0146 (5)	0·4478 (5)	0·4017 (2)	4·88 (9)
C(22)	0·9411 (4)	0·4816 (4)	0·4724 (2)	4·25 (5)
C(23)	0·8128 (4)	0·5862 (4)	0·4603 (2)	3·84 (5)
C(24)	0·7096 (4)	0·6220 (4)	0·5244 (2)	3·59 (5)
O(25)	0·7135 (3)	0·5588 (4)	0·6052 (1)	3·86 (5)
O(26)	0·8354 (3)	0·4867 (4)	0·8041 (1)	4·46 (5)
C(27)	0·8720 (5)	0·3683 (5)	0·7675 (3)	5·43 (10)
C(28)	0·3137 (7)	0·3416 (6)	1·0813 (3)	8·37 (15)
C(29)	0·2966 (8)	0·2190 (6)	0·9353 (3)	8·21 (13)
C(30)	1·1446 (8)	0·5315 (6)	0·2717 (4)	9·32 (18)
C(31)	0·8111 (9)	0·4458 (5)	0·2434 (3)	9·51 (18)

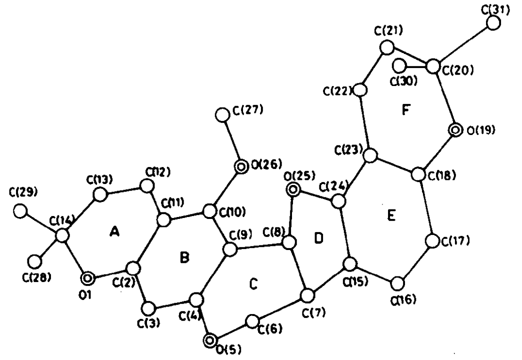

Fig. 1. Numbering scheme for the molecule.

Woolfson, 1980). An E map computed with the second largest CFOM (2·197) gave a partial structure, which revealed 21 out of 31 non-H atoms, remaining atoms from successive difference Fourier maps, refinement on F by full-matrix least-squares methods (Sheldrick, 1976), H atoms from difference Fourier map, anisotropic thermal parameters for non-H and isotropic for H atoms, H-atom positions not refined, final $R = 0{\cdot}035$, $wR = 0{\cdot}044$, $w = 1{\cdot}0/[\sigma^2(|F_o|) + 0{\cdot}00054F_o^2]$, $S = 1{\cdot}43$, $(\Delta/\sigma)_{\max} = 0{\cdot}003$, final difference map featureless, $\Delta\rho$ within $\pm 0{\cdot}52$ e Å^{-3}, no correction for secondary extinction, atomic scattering factors for all atoms were as in *SHELX*76 (Sheldrick, 1976), geometrical calculations using *PARST* (Nardelli, 1983), calculations performed on an IBM 360/44 computer.

Table 2. *Bond lengths* (Å) *and angles* (°) *involving non-*H *atoms, and endocyclic torsion angles* (°)

O(1)—C(2)	1·377 (5)	C(13)—C(14)	1·501 (6)
O(1)—C(14)	1·453 (6)	C(14)—C(28)	1·513 (6)
C(2)—C(3)	1·385 (4)	C(14)—C(29)	1·513 (8)
C(2)—C(11)	1·397 (5)	C(15)—C(16)	1·392 (6)
C(3)—C(4)	1·382 (5)	C(15)—C(24)	1·382 (5)
C(4)—O(5)	1·364 (4)	C(16)—C(17)	1·394 (6)
C(4)—C(9)	1·411 (4)	C(17)—C(18)	1·381 (7)
O(5)—C(6)	1·433 (4)	C(18)—O(19)	1·371 (5)
C(6)—C(7)	1·528 (4)	C(18)—C(23)	1·397 (5)
C(7)—C(8)	1·534 (5)	O(19)—C(20)	1·455 (7)
C(7)—C(15)	1·502 (5)	C(20)—C(21)	1·487 (6)
C(8)—C(9)	1·482 (5)	C(20)—C(30)	1·540 (8)
C(8)—O(25)	1·474 (5)	C(20)—C(31)	1·519 (7)
C(9)—C(10)	1·392 (5)	C(21)—C(22)	1·327 (5)
C(10)—C(11)	1·374 (5)	C(22)—C(23)	1·452 (5)
C(10)—O(26)	1·391 (4)	C(23)—C(24)	1·390 (5)
C(11)—C(12)	1·456 (5)	C(24)—O(25)	1·368 (4)
C(12)—C(13)	1·326 (6)	O(26)—C(27)	1·434 (6)

C(2)—O(1)—C(14)	118·7 (3)	C(13)—C(14)—C(28)	111·1 (3)
O(1)—C(2)—C(11)	120·4 (2)	O(1)—C(14)—C(29)	107·4 (3)
O(1)—C(2)—C(3)	117·5 (2)	O(1)—C(14)—C(28)	104·9 (4)
C(3)—C(2)—C(11)	122·1 (2)	C(28)—C(14)—C(29)	111·9 (4)
C(2)—C(3)—C(4)	118·5 (3)	C(7)—C(15)—C(24)	107·6 (3)
C(3)—C(4)—C(9)	121·9 (3)	C(7)—C(15)—C(16)	133·3 (3)
C(3)—C(4)—O(5)	116·9 (3)	C(16)—C(15)—C(24)	119·1 (3)
O(5)—C(4)—C(9)	121·2 (3)	C(15)—C(15)—C(17)	118·9 (4)
C(4)—O(5)—C(6)	114·7 (3)	C(16)—C(17)—C(18)	120·4 (3)
O(5)—C(6)—C(7)	111·4 (2)	C(17)—C(18)—C(23)	122·1 (3)
C(6)—C(7)—C(15)	112·0 (3)	C(17)—C(18)—O(19)	118·2 (4)
C(6)—C(7)—C(8)	108·4 (3)	O(19)—C(18)—C(23)	119·6 (3)
C(8)—C(7)—C(15)	101·0 (3)	C(18)—O(19)—C(20)	118·4 (4)
C(7)—C(8)—O(25)	104·8 (2)	O(19)—C(20)—C(31)	106·8 (4)
C(7)—C(8)—C(9)	114·2 (3)	O(19)—C(20)—C(30)	102·9 (4)
C(9)—C(8)—O(25)	111·5 (3)	O(19)—C(20)—C(21)	111·9 (4)
C(4)—C(9)—C(8)	121·2 (3)	C(30)—C(20)—C(31)	113·2 (4)
C(8)—C(9)—C(10)	122·0 (3)	C(21)—C(20)—C(31)	110·9 (4)
C(4)—C(9)—C(10)	116·4 (3)	C(21)—C(20)—C(30)	110·8 (4)
C(9)—C(10)—O(26)	117·1 (3)	C(20)—C(21)—C(22)	122·1 (4)
C(9)—C(10)—C(11)	123·8 (3)	C(21)—C(22)—C(23)	118·7 (3)
C(11)—C(10)—O(26)	119·1 (3)	C(13)—C(23)—C(22)	119·6 (3)
C(2)—C(11)—C(10)	117·3 (3)	C(22)—C(23)—C(24)	124·5 (3)
C(10)—C(11)—C(12)	124·3 (3)	C(13)—C(23)—C(24)	115·9 (3)
C(2)—C(11)—C(12)	118·3 (3)	C(15)—C(24)—C(23)	123·6 (3)
C(11)—C(12)—C(13)	119·6 (3)	C(23)—C(24)—O(25)	123·1 (3)
C(12)—C(13)—C(14)	121·7 (3)	C(15)—C(24)—O(25)	113·3 (3)
O(1)—C(14)—C(13)	110·8 (3)	C(8)—O(25)—C(24)	105·3 (3)
C(13)—C(14)—C(29)	110·5 (4)	C(10)—O(26)—C(27)	113·6 (3)

Ring *A*		Ring *C*	
C(2)—O(1)—C(14)—C(13)	37·6 (5)	O(5)—C(4)—C(9)—C(8)	9·3 (5)
C(14)—O(1)—C(2)—C(11)	−25·3 (5)	C(9)—C(4)—O(5)—C(6)	19·9 (5)
O(1)—C(2)—C(11)—C(12)	−0·6 (5)	C(4)—O(5)—C(6)—C(7)	−55·5 (4)
C(2)—C(11)—C(12)—C(13)	11·3 (6)	O(5)—C(6)—C(7)—C(8)	60·0 (4)
C(11)—C(12)—C(13)—C(14)	4·0 (6)	C(6)—C(7)—C(8)—C(9)	−31·1 (4)
C(12)—C(13)—C(14)—O(1)	−27·3 (6)	C(7)—C(8)—C(9)—C(4)	−1·5 (5)
Ring *D*		Ring *F*	
C(8)—C(7)—C(15)—C(24)	17·0 (4)	O(19)—C(18)—C(23)—C(22)	−1·7 (5)
C(15)—C(7)—C(8)—C(25)	−26·6 (3)	C(23)—C(18)—O(19)—C(20)	26·9 (6)
C(7)—C(8)—O(25)—C(24)	27·4 (3)	C(18)—O(19)—C(20)—C(21)	−36·6 (5)
C(7)—C(15)—C(24)—O(25)	−0·4 (4)	O(19)—C(20)—C(21)—C(22)	23·8 (6)
C(15)—C(24)—O(25)—C(8)	−17·4 (4)	C(20)—C(21)—C(22)—C(23)	−0·8 (6)
		C(21)—C(22)—C(23)—C(18)	−11·5 (5)

Discussion. Gangetinin has a framework of six fused rings *A–F*; the atomic numbering scheme and ring identifiers are given in Fig. 1. The final positional and equivalent isotropic thermal parameters for the non-H atoms are given in Table 1.* Bond lengths

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, distances and angles involving H atoms, torsion angles, least-squares planes and intermolecular distances less than 3·5 Å have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52528 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

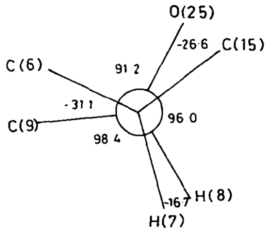

Fig. 2. Newman projection along the C(7)—C(8) bond.

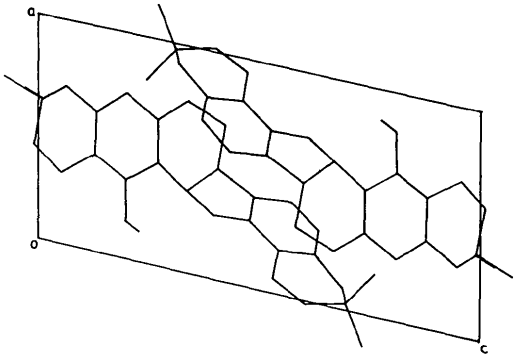

Fig. 3. Molecular packing of gangetinin in the unit cell viewed down the *b* axis.

and valence angles involving non-H atoms and endocyclic torsion angles characterizing the ring conformations are given in Table 2.

The pyrone ring *A* is in the half-chair conformation, with the best rotational axis bisecting the O(1)—C(14) and C(11)—C(12) bonds $\{\Delta C_2[C(1)—C(14)] = 3·6°\}$. The benzene ring *B* is planar ($\chi^2 = 36·4$) and O(26) of the methoxy group is coplanar with it. The pyrone ring *C* adopts a distorted sofa conformation, the asymmetry parameter $\Delta C_s[C(6)] = 8·3°$ (Duax, Weeks & Rohrer, 1976). The five-membered ring *D* is a perfect envelope with the mirror plane bisecting the C(15)—C(24) bond. The *C*/*D* junction shows *cis* fusion. Fig. 2 shows the Newman projection along the C(7)—C(8) bond. The molecule bends considerably at this junction: the dihedral angle between the best planes through the rings *C* and *D* is 36·9 (2)°. The benzene ring *E* is planar ($\chi^2 = 3·3$). The pyrone ring *F* is a half chair; the rotation axis bisects C(19)—C(20) and C(22)—C(23) $\{\Delta C_2[C(19)—C(20)] = 2·8°\}$. Molecular packing, viewed along *b*, is illustrated in Fig. 3.

The authors thank Dr K. Balakrishna, Captain Srinivasamurthy Drug Research Institute, Madras, for providing the sample and also RSIC, IIT, Madras, for data collection.

References

Duax, W. L., Weeks, C. M. & Rohrer, D. C. (1976). *Topics in Stereochemistry*, Vol. 9, edited by N. L. Allinger & E. L. Eliel, pp. 271–383. New York: John Wiley.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Pillai, N. R., Alam, M. & Purushothaman, K. K. (1981). *J. Res. Ayurveda Sidda*, **2**, 349–356.
Purushothaman, K. K., Chandrasekharan, S., Balakrishna, K. & Connolly, J. D. (1975). *Phytochemistry*, **14**, 1129–1130.
Purushothaman, K. K., Kishore, V. M., Narayanaswami, V. & Connolly, J. D. (1971). *J. Chem. Soc. C*, pp. 2420–2422.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1990). **C46**, 843–846

3-(Triphenylphosphonio)indole-2-thiolate

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(*Received* 24 *December* 1987; *accepted* 16 *May* 1989)

Abstract. Two symmetry independent molecules, $C_{26}H_{20}NPS$, $M_r = 409·48$, monoclinic, *Cc*, $a = 9·4497$ (6), $b = 25·590$ (2), $c = 18·600$ (1) Å, $\beta = 104·70$ (1)°, $V = 4350·6$ (5) Å^3, $Z = 8$, $D_x = 1·250$, $D_m = 1·251$ (1) g cm^{-3}, Cu $K\alpha$, $\lambda = 1·54178$ Å, $\mu = 20·72$ cm^{-1}, $F(000) = 1712$, room temperature, $R = 0·037$ for 3384 independent observed reflexions. Every molecule is connected by two S···H—N hydro-

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