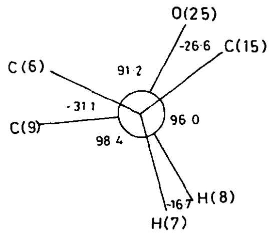

Fig. 2. Newman projection along the C(7)—C(8) bond.

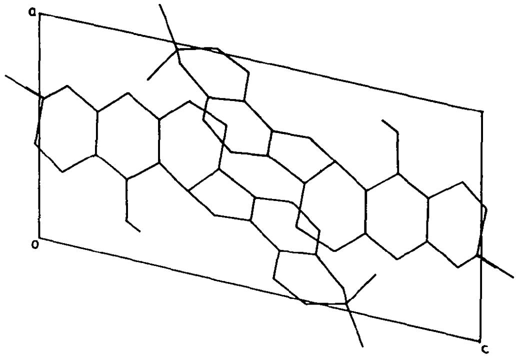

Fig. 3. Molecular packing of gangetinin in the unit cell viewed down the *b* axis.

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.

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3-(Triphenylphosphonio)indole-2-thiolate

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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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gen bonds to its symmetry independent counterpart thus forming dimers with almost mutually perpendicular indolyl groups.

Table 1. *Final coordinates* ($\times 10^4$) *and* U_{eq} *values* ($\times 10^3$) *for non-*H *atoms, with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq} (Å^2)
N1	−993 (4)	637 (2)	−1423 (2)	40 (2)
C2	−1603 (5)	390 (2)	−925 (3)	37 (2)
C3	−459 (5)	235 (2)	−318 (3)	38 (2)
C4	904 (5)	405 (2)	−474 (3)	37 (2)
C5	2395 (6)	381 (2)	−106 (3)	46 (3)
C6	3393 (6)	593 (3)	−451 (4)	57 (3)
C7	2977 (6)	830 (2)	−1141 (4)	60 (4)
C8	1528 (6)	860 (2)	−1511 (3)	49 (3)
C9	500 (5)	654 (2)	−1168 (3)	38 (2)
S10	−3458 (2)	305 (1)	−1100 (1)	43 (1)
P11	−762 (2)	−132 (1)	397 (1)	37 (1)
C12	−1669 (6)	232 (2)	1001 (3)	44 (3)
C13	−1901 (8)	22 (3)	1648 (4)	63 (4)
C14	−2636 (8)	301 (3)	2082 (4)	75 (5)
C15	−3069 (9)	802 (4)	1882 (4)	84 (5)
C16	−2771 (11)	1035 (3)	1259 (4)	91 (5)
C17	−2103 (8)	744 (2)	814 (4)	65 (4)
C18	−1736 (6)	−731 (2)	71 (3)	47 (3)
C19	−2462 (14)	−1002 (4)	491 (4)	122 (7)
C20	−2982 (15)	−1508 (4)	270 (5)	141 (5)
C21	−2861 (10)	−1699 (3)	−376 (4)	78 (3)
C22	−2251 (8)	−1416 (3)	−821 (4)	68 (4)
C23	−1680 (7)	−927 (2)	−605 (3)	55 (3)
C24	992 (6)	−337 (2)	996 (3)	47 (3)
C25	1705 (7)	−45 (3)	1591 (4)	68 (4)
C26	3111 (9)	−188 (5)	1999 (4)	90 (6)
C27	3769 (10)	−610 (5)	1808 (6)	99 (7)
C28	3055 (11)	−908 (4)	1217 (8)	112 (7)
C29	1680 (9)	−767 (3)	812 (5)	82 (3)
N31	1334 (5)	3443 (2)	3443 (2)	43 (2)
C32	1863 (5)	3211 (2)	2900 (3)	37 (2)
C33	1886 (6)	2666 (2)	3032 (3)	38 (2)
C34	1377 (5)	2576 (2)	3691 (3)	36 (2)
C35	1189 (6)	2142 (2)	4125 (3)	48 (3)
C36	661 (7)	2221 (2)	4744 (3)	56 (3)
C37	326 (7)	2719 (3)	4951 (3)	60 (3)
C38	525 (6)	3152 (2)	4550 (3)	51 (3)
C39	1046 (5)	3075 (2)	3931 (3)	39 (3)
S40	2365 (2)	3547 (1)	2198 (1)	48 (7)
P41	2613 (1)	2238 (< 1)	2474 (1)	34 (< 1)
C42	4482 (4)	2416 (2)	2521 (2)	41 (2)
C43	5110 (8)	2343 (4)	1955 (4)	123 (6)
C44	6543 (9)	2480 (6)	2012 (5)	153 (8)
C45	7377 (7)	2681 (3)	2657 (4)	86 (4)
C46	6797 (6)	2757 (2)	3227 (4)	60 (3)
C47	5332 (6)	2627 (2)	3168 (3)	58 (3)
C48	2596 (5)	1588 (2)	2867 (2)	42 (2)
C49	1268 (6)	1317 (2)	2717 (3)	56 (3)
C50	1194 (7)	835 (2)	3056 (3)	71 (3)
C51	2417 (9)	624 (2)	3510 (4)	84 (4)
C52	3737 (8)	881 (3)	3647 (4)	89 (4)
C53	3818 (6)	1370 (2)	3321 (3)	65 (3)
C54	1557 (5)	2170 (2)	1536 (2)	39 (2)
C55	321 (5)	2472 (2)	1262 (3)	49 (3)
C56	−524 (6)	2377 (3)	544 (3)	66 (3)
C57	−168 (7)	1996 (3)	111 (3)	71 (4)
C58	1075 (7)	1691 (2)	385 (3)	70 (4)
C59	1908 (6)	1778 (2)	1095 (3)	59 (3)

Introduction. The crystal structure of 3-(triphenylphosphonio)indole-2-thiolate has been determined as part of the study of intramolecular cyclization of isothiocyanates. The new method of preparation of indole derivatives described by Gonda, Kristian & Imrich (1987) will be used for the production of some biologically active heterocyclic compounds.

The molecular structure determination was required to confirm the identity of the reaction product. An additional reason for this study was to investigate the influence of different substituents on the structure of the indolyl moiety.

Experimental. The title compound was prepared by adding a solution of triethylamine in acetonitrile to a solution of 2-isothiocyanatobenzyltriphenylphosphonium bromide in acetonitrile. The crystals were filtered and washed successively in water, methanol and ether (Gonda, Kristian & Imrich, 1987). The substance decomposes at temperatures in the interval 578–582 K. The final bright yellow crystals were recrystallized from the acetone and methanol solution. The density was measured pycnometrically in CCl_4 and toluene. Unit-cell parameters were determined by least squares from positions of ten reflexions (in the interval 8–21° 2θ) centered on a CAD-4 diffractometer at room temperature. Systematic absences khl: $h + k = 2n$, $h0l$ $l = 2n$ were checked by ψ scan. Approximately cubic crystal with side 0·6 mm was used for intensity measurement. Range of h −11/9, k 0/30, l 0/21. Total of 3773 intensities of independent reflexions with $\sin\theta/\lambda$ less

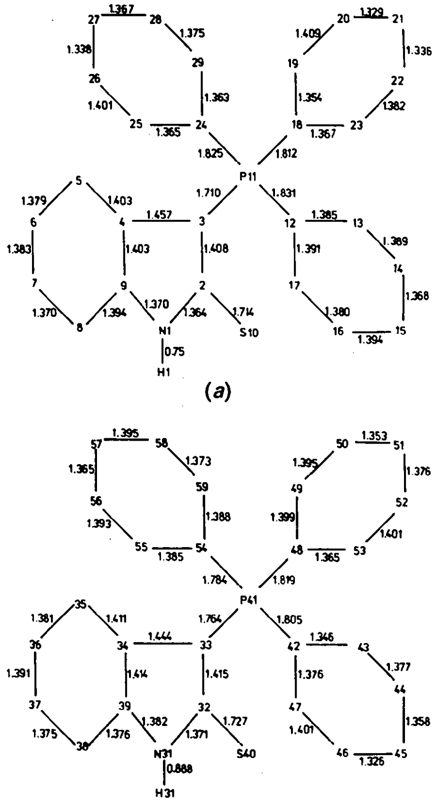

Fig. 1. The numbering scheme and interatomic distances (Å) of non-H atoms in both symmetry independent molecules. E.s.d.'s of distances in the indolyl moiety and around phosphorus are from 0·004 to 0·008 Å, in outer parts of the phenyl rings from 0·009 to 0·014 Å.

than 0·5876 Å^{-1} were determined by $\omega/2\theta$ scan, graphite monchromator. 389 were classified as unobserved according to the criterion $I < 3{\cdot}92\sigma(I)$. No corrections for absorption or extinction were made. The structure was solved by direct methods with *SIMPEL* (Schenk, 1982). H atoms could be localized from the difference map. The function minimized was $\sum w\Delta^2$, where $\Delta = ||F_o| - |F_c||$ and $w = [\sigma^2(F_o) + 0{\cdot}0005|F_o|^2]^{-1}$. The refinement was made by *SHELX76* (Sheldrick, 1976) in two blocks up to $R = 0{\cdot}037$, $wR = 0{\cdot}038$ for 3384 observed reflexions and 683 refined parameters (all non-H atoms with anisotropic and H atoms with isotropic temperature factors). The refinement was stopped when the ratio shift/e.s.d. had dropped below 0·3 for all parameters. Eight highest peaks in the final difference map from 0·18 to 0·27 e Å^{-3} lay in pairs about 1·0 Å from P11, P41, S10 and S40 near the supposed positions of lone electron pairs. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The geometry was calculated with *PARST* (Nardelli, 1983) and *IMC* (Hašek, 1981) programs.

Table 2. *Mean planes through indolyl moieties and benzene rings, χ^2 values and dihedral angles between planes in the same molecule*

$$\chi^2 = \sum(\Delta/\sigma)^2.$$

	Plane	χ^2
a	N1, C2, C3, C4, C5, C6, C7, C8, C9, S10	67
b	C12, C13, C14, C15, C16, C17	53
c	C24, C25, C26, C27, C28, C29	1
d	C18, C19, C20, C21, C22, C23	79
e	N31, C32, C33, C34, C35, C36, C37, C38, C39, S40	148
f	C42, C43, C44, C45, C46, C47	2
g	C48, C49, C50, C51, C52, C53	14
h	C54, C55, C56, C57, C58, C59	4

Dihedral angles (°)*

Molecule 1		Molecule 2	
a–b	67·3	*e–f*	73·9
a–c	96·5	*e–g*	82·0
a–d	108·5	*e–h*	113·3
b–c	107·9	*f–g*	103·7
b–d	44·6	*f–h*	53·9
c–d	87·3	*g–h*	74·3

* All e.s.d.'s are 0·2°.

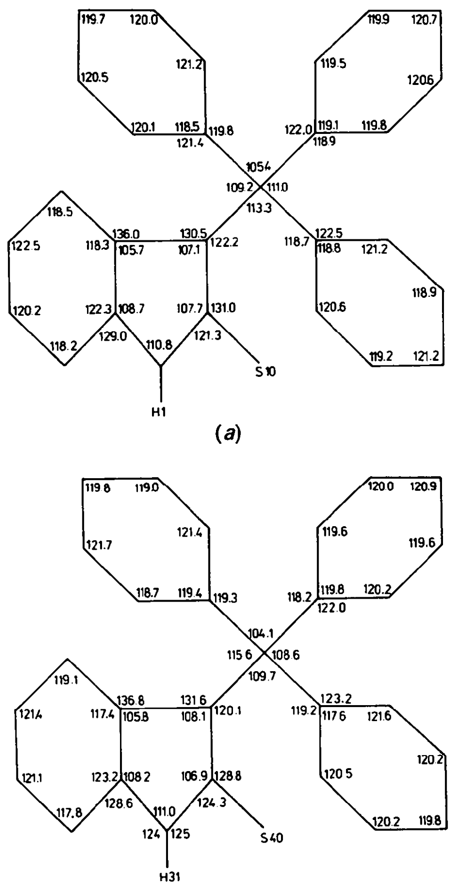

Fig. 2. The bond angles (°) in both symmetry independent molecules. E.s.d.'s are from 0·2 to 0·5° for angles containing at least one atom of the indolyl moiety or phosphorus, and in the range 0·5–0·9° in the phenyl rings.

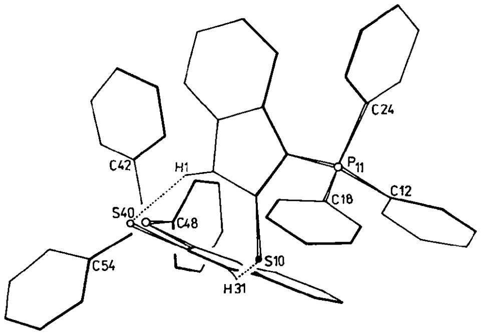

Fig. 3. Projection of two symmetry independent molecules of 3-(triphenylphosphonio)indole-2-thiolate along the *b* axis.

Discussion. The numbering of non-H atoms, and bond distances are given in Fig. 1. The numbering of H atoms corresponds to the numbering of bonded carbons. The refined coordinates of non-H atoms, with U_{eq}'s, are given in Table 1.* The temperature parameters U_{eq} indicate wider peaks of an average electron density at the outer parts of all the six phenyl rings. This agrees with the higher e.s.d.'s of bond distances and angles in the phenyl ring parts most distant from phosphorus (Figs. 1 and 2).

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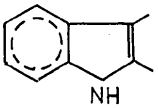
* Tables of anisotropic thermal parameters and H-atom parameters, a view of the molecular packing along the *b* axis and the list of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52236 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The average P—C (benzene) bond, 1·813 Å, is longer than P—C (indolyl) 1·737 Å. The bond distances and angles show high delocalization of π electrons over the whole indolyl moiety. All the indolyl distances are typical of partially conjugated bonds. The bond lengths C2—C3 and C32—C33 (average 1·412 Å) are longer than the aromatic C—C bond. In any case the description of the indolyl moiety by the resonance structure with the double bond between C2—C3



would be misleading. The six-membered ($\chi^2 = 21$, 13) and five-membered rings ($\chi^2 = 1$, 10) of the indolyl moieties form dihedral angles of 0·5 (1) and 0·9 (1)° for molecules 1 and 2 respectively. The deviations from the mean plane of the five-membered rings are: for phosphorus 0·015 (1), −0·020 (2) Å, and for sulfur 0·122 (1), −0·109 (1) Å. The torsion angles are S10—C2—C3—P11 4·1 (5), S40—C32—C33—P41 4·6 (5) and S10—C2—C3—C4 179·1 (3), S40—C32—C33—C34 175·3 (3)°.

Comparison of intramolecular dihedral angles (Table 2) shows a similar conformation for both molecules. Projection of the crystal structure along the *b* axis (Fig. 3) shows that the dominant feature of the crystal structure is dimerization of two symmetry independent molecules, formed by two nonequivalent hydrogen bonds $N1^i—H1^i\cdots S40$ and $N31—H31\cdots S10^i$, where (i) = $x + \frac{1}{2}$, $-y + \frac{1}{2}$, $z + \frac{1}{2}$. The corresponding characteristics are $N1^i\cdots S40$ 3·366 (3), $N1^i—H1^i$ 0·75 (4) Å, $N1^i—H1^i\cdots S40$ 164 (5)°, $N31\cdots S10^i$ 3·308 (2), N31—H31 0·89 (5) Å, $N31—H31\cdots S10^i$ 169 (4)°.

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Structure of Hexamethylenediammonium Terephthalate Dihydrate

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Abstract. $C_6H_{18}N_2^{2+}.C_8H_4O_4^{2-}.2H_2O$, $M_r = 318·38$, monoclinic, $P2_1/n$, $a = 20·541$ (2), $b = 7·2953$ (5), $c = 11·376$ (1) Å, $\beta = 100·588$ (6)°, $V = 1675·8$ (2) Å^3, $Z = 4$, $D_m = 1·267$ (1), $D_x = 1·262$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1·5418$ Å, $\mu = 0·83$ mm^{-1}, $F(000) = 688$, $T = 295$ K, $R = 0·056$ for 2672 unique reflections. Hexamethylenediammonium cations and terephthalate anions are held together by two kinds of N—H···O hydrogen bonds to form a ribbon of these ions along **a**. The ribbons are stacked along **b** to form a sheet parallel to (001) and the sheets are joined by N—H···O and O—H···O hydrogen bonds to form a three-dimensional network.

Introduction. Hexamethylenediammonium terephthalate (6T) is one of the intermediates in the manufacture of Nylon 6T (Morgan & Kwolek, 1975; Pineault & Brisse, 1983*a*). The structure of 6T has been studied in order to elucidate a characteristic feature of the molecular arrangement in the crystals of the Nylon salt composed of the aliphatic cation and aromatic anion.

Experimental. Crystals obtained from an aqueous solution by slow evaporation at room temperature: colorless prisms elongated along **b**. D_m by flotation in benzene–CCl_4. Systematic absences: $h0l$ for $h + l$ odd, $0k0$ for k odd. A crystal with dimensions 0·20 × 0·28 × 0·28 mm mounted on a Rigaku AFC-5 four-circle diffractometer. Lattice parameters determined with 15 reflections in the range $39 < 2\theta < 41°$ by the

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