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Diethyl {3-[β-(2,4-dichlorophenyl)-vinyl]-*N*-(phenylsulfonyl)indol-2-ylmethyl}phosphonate and diethyl {3-[β-(4-bromophenyl)vinyl]-*N*-(phenylsulfonyl)indol-2-ylmethyl}-phosphonate

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The title compounds, $C_{27}H_{26}Cl_2NO_5PS$, (I), and $C_{27}H_{27}BrNO_5PS$, (II), respectively, crystallize in the centrosymmetric space group $P2_1/n$ with one molecule in the asymmetric unit in each case. The dihedral angle between the benzene and pyrrole rings is 2.1 (1)° in (I) and 0.9 (2)° in (II). The phenylsulfonyl group is orthogonal to the halophenyl moiety, with a dihedral angle of 82.0 (1)° in (I) and 78.7 (2)° in (II). In both compounds, the molecular structures and packing are stabilized by C—H···O and C—H···halogen interactions. The intermolecular hydrogen bonds in (I) form cyclic dimers with graph-set descriptors $R_2^1(10)$ and $R_2^2(8)$ about a 2_1 axis, and those in (II) form a $C_2^2(20)$ chain.

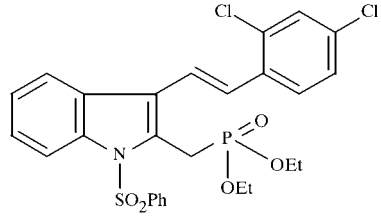
Comment

Organophosphorus compounds have attracted considerable interest because of their applications as insecticides, bactericides, flame retardants, lubricants, *etc*. (Ismail, 1975). Phosphate esters are present in many enzymes, nucleic acids, bacteria, vitamins and viruses. Organophosphorus compounds are also reported as effective antitumour compounds.

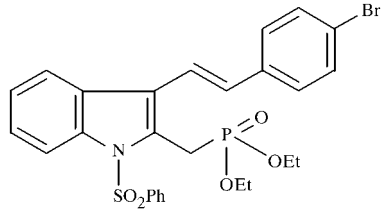
The indole ring system is present in a number of natural products, many of which are found to possess antibacterial (Okabe & Adachi, 1998), antitumour (Schollmeyer *et al.*, 1995), antidepressant (Papenstasion & Newmeyer, 1972), psychotropic (Grinev *et al.*, 1978), hypertensive (Merk, 1971), antimicrobial (El-Sayed *et al.*, 1986; Gadaginamath & Patil, 1999) or anti-inflammatory activities (Rodriguez *et al.*, 1985; Polletto *et al.*, 1974). Indoles also intercalate with DNA (Sivaraman *et al.*, 1996), and this intercalation between the base pairs in DNA has been implicated for their medicinal properties. The indole system occurs in plants (Nigović *et al.*, 2000), for example, indole-3-acetic acid is a naturally occurring plant-growth hormone that controls several plant-growth activities (Moore, 1989; Fargasova, 1994). Indoles have also been proven to display high aldose reductase inhibitory activity (Rajeswaran *et al.*, 1999).

Sulfonamide-containing drugs acts as diuretics, and sulthiame, as a carbonic anhydrase inhibitor, has been shown to possess anticonvulsant activity (Crawford & Kennedy, 1959; Camerman & Camerman, 1975; Tanimukai *et al.*, 1965). Sulfonamides inhibit the growth of bacterial organisms and are also useful for treating urinary and gastrointestinal infections.

Both title compounds are excellent intermediates for elaborating the substituent at the 2-position of the indole ring. Compounds (I) and (II) could be converted into 2-vinyl derivatives by the Wittig reaction. These compounds have also been converted into analogues of the anticancer alkaloid ellipticine (Srinivasan & Mohanakrishnan, 1995). Similarly, compounds of this type have been shown to undergo a Diels–Alder reaction to give 2-(quinolin-2-yl)indoles (Srinivasan & Elango, 1999). Against this background, and in order to obtain detailed information on their molecular conformation in the solid state, X-ray studies of the title compounds, (I) and (II), have been carried out and the results are presented here.



(I)



(II)

Figs. 1 and 2 show the molecular structures of (I) and (II), respectively, with the atom-numbering schemes. The mean P—O single-bond distance of 1.565 (3) Å for (I) and (II) is in agreement with the reported value of 1.564 (5) Å for the structure of diethyl (1-hydroxy-2-butynyl)phosphonate (Sanders *et al.*, 1996). The P1═O1 double-bond distances in (I) and (II) are comparable with the reported values of 1.464 (2), 1.459 (3) and 1.454 (4) Å [Naidu *et al.* (1992), Boehlow *et al.* (1997) and Yokota *et al.* (1990), respectively]. The P1—C15 single-bond lengths in both compounds are in good agreement with reported values of 1.791 (2) and 1.806 (6) Å (Weichsel & Lis, 1996; Liu *et al.*, 1995; Perales & García-Blanco, 1977; Howells *et al.*, 1973).

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In (I) and (II), the average values of the S1═O2 and S1═O3 distances [1.417 (3) and 1.426 (3) Å, respectively] are comparable with the literature value of 1.427 (4) Å (Datta *et al.*, 1993; Ghosh *et al.*, 1989; Seetharaman & Rajan, 1995). The S1—N1 and S1—C9 bond distances in (I) and (II) compare well with the literature values of 1.64 (2) and 1.758 (18) Å, respectively (Allen *et al.*, 1987).

The relatively large values of the C—N distances in the indole moiety (N1—C1 and N1—C4) are due to the electron-withdrawing character of the phenylsulfonyl group (Govindasamy *et al.*, 1997, 1998). As in similar structures (Hazel & Collin, 1972; Ezra & Collin, 1973; Sanders *et al.*, 1996), short C—C bond distances [C18—C19 in (I) and (II)], along with high thermal motion, are observed for the ethyl C atoms. The Csp^2—X bond distances [X is Cl in (I) and Br in (II)] are comparable with reported values of 1.734 (19) and 1.883 (15) Å, respectively (Allen *et al.*, 1987). Selected geometric parameters for (I) and (II) are given in Tables 1 and 3, respectively.

Atom P1 adopts a distorted tetrahedral configuration in both compounds, and the widening of the O1—P1—O4 angles, and the resulting narrowing of the O5—P1—C15 angles from the ideal tetrahedral value, is attributed to the Thorpe–Ingold effect (Bassindale, 1984). The P1—C15 bond is (−)synclinal to the C1—C2 bond in (I), whereas it is (+)synclinal to C1—C2 in (II). The conformation about P1—C15 is, as expected, staggered in (I) and (II).

In both compounds, the indole system is not strictly planar, and the dihedral angle formed by the pyrrole and benzene planes is 2.1 (1)° in (I) and 0.9 (2)° in (II). The O2—S1—N1—C4 and O2—S1—C9—C14 torsion angles in both compounds describe the conformation of the phenylsulfonyl moiety with respect to the indole system, which causes the best planes of the indole and phenyl rings to form a dihedral angle of 77.3 (1)° in (I) and 71.6 (2)° in (II), as observed in similar structures (Yokum & Fronczek, 1997; Sankaranarayanan *et al.*, 2000).

The phenyl rings of the dichlorophenyl moiety in (I) and the bromophenyl moieties in (II) are orthogonal to the phenyl ring of the sulfonyl substituent in each, forming a dihedral

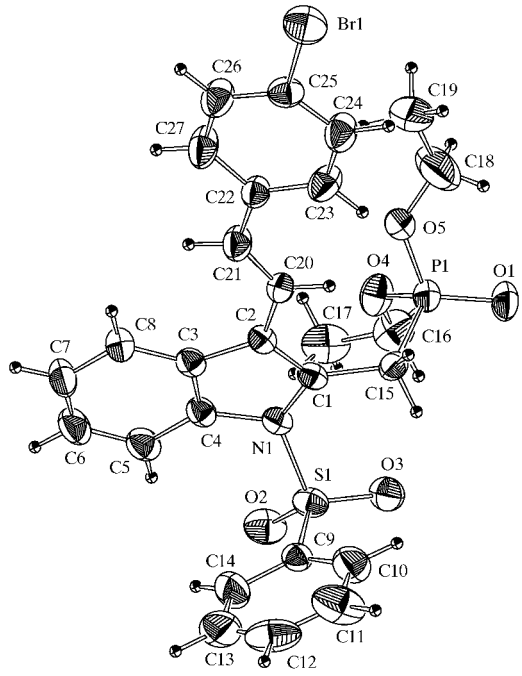

Figure 2
The molecular structure of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 35% probability level and H atoms are shown as small spheres of arbitrary radii.

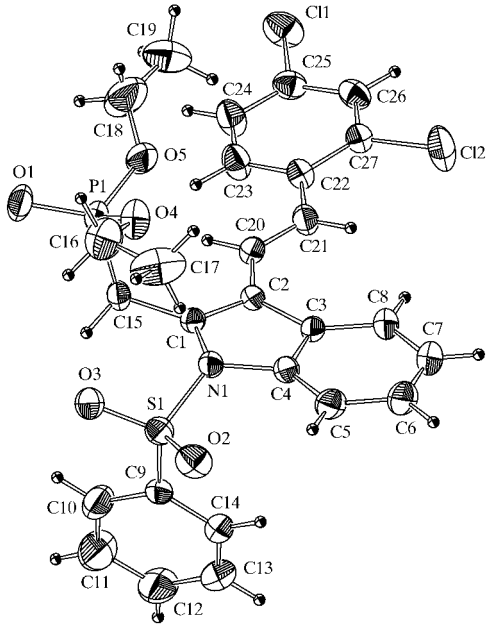

Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 35% probability level and H atoms are shown as small spheres of arbitrary radii.

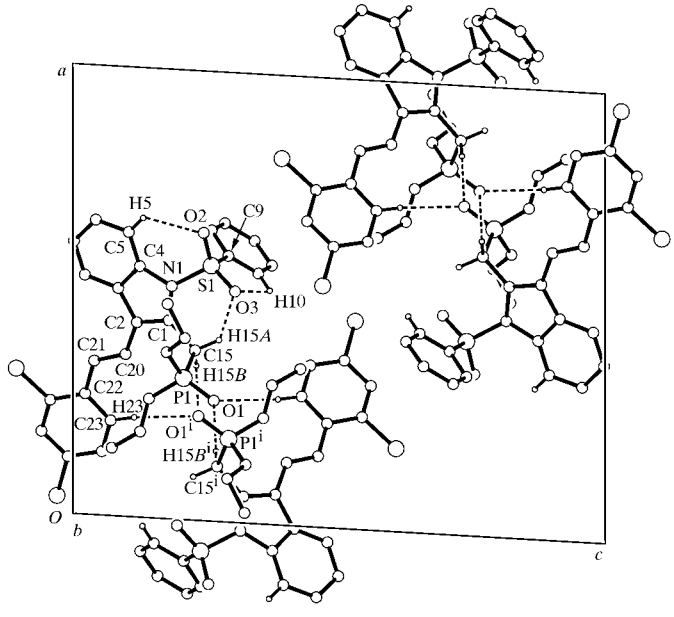

Figure 3
The crystal structure of (I), with the hydrogen-bonding scheme shown as dashed lines [symmetry code: (i) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$].

angle of 82.0 (1)° in (I) and 78.7 (2)° in (II). The dihedral angle formed by the weighted least-squares planes through the pyrrole ring and the halophenyl group is 12.0 (1)° in (I) and 11.4° in (II). Atoms C15 and C20 are out of the indole plane by 0.205 (4) and 0.057 (4) Å, respectively, in (I), and by 0.097 (4) and 0.038 (4) Å, respectively, in (II), all on one side, while atom S1 is out of the plane on the other side, by 0.730 (1) Å in (I) and 0.812 Å in (II).

In the benzene ring of the indole system, the endocyclic angles at C3 and C5 are contracted to 118.7 (4) and 116.9 (5)°, respectively, in (I), and 118.9 (4) and 117.5 (5)°, respectively, in (II), while those at C4, C6 and C7 are expanded to 122.2 (4), 121.9 (4) and 120.7 (3)°, respectively, in (I), and 122.3 (4), 121.3 (5) and 121.1 (5)°, respectively, in (II). This would appear to be a real effect caused by the fusion of the smaller pyrrole ring to the six-membered benzene ring, and the strain is taken up by angular distortion rather than by bond-length distortions (Allen, 1981). A similar effect has also been observed by both Varghese *et al.* (1986) and Sankaranarayanan *et al.* (2000).

The angular disposition of the bond about atom S1 shows a significant deviation from that of a regular tetrahedron, with the largest deviation for the O—S—O angle. The widening of the O2—S1—O3 angle, to 119.1 (2)° in (I) and 119.5 (2)° in (II), from the ideal tetrahedral value is presumably the result of the repulsive interaction between the short S═O bonds, similar to that observed in related structures (Rodriguez *et al.*, 1985; Beddoes *et al.*, 1986). The orientation of the indole substituent is influenced by a weak C5—H5···O2 interaction, defined by the torsion angle C5—C4—N1—S1 in (I) and C4—N1—S1—O2 in (II), while the orientation of the phenyl ring bound to the sulfonyl group is governed by a C10—H10···O3 interaction, defined by the N1—S1—C9—C10 torsion angle in both compounds.

In addition to van der Waals interactions, the molecular structures and packing are stabilized by C—H···O and C—H···halogen interactions (Tables 2 and 4).

In compound (I), the hydrogen bonds form cyclic dimers with graph-set descriptors (Bernstein *et al.*, 1995) $R_2^1(10)$ and $R_2^2(8)$ about a 2_1 axis. This system of rings is joined through the O5—C19—C18 ethoxy chain by a hydrogen bond to the Cl1 atom of a molecule shifted by **b** in the [010] direction. For $R_2^1(10)$, the ring is C15—H15*B*···O1^{i}···H23—C23—C22—C21—C20—C2—C1, and for $R_2^2(8)$, the ring is C15—H15*B*···O1^{i}—P1^{i}—C15^{i}—H15*B*^{i}···O1···P1 [symmetry code: (i) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$]. There are three intramolecular rings in (I), namely, *S*(6) C5—H5···O2—S1—N1—C4, *S*(6) C15—H15*A*···O3—S1—N1—C1 and *S*(5) C10—H10···O3—S1—C9 (Fig. 3).

In (II), there is a $C_2^2(20)$ chain, O1^{i}···H15*A*—C15—C1—N1—S1—C9—C14—H14···Br1^{ii}—C25^{ii}—C24^{ii}—C23^{ii}—C22^{ii}—C21^{ii}—C20^{ii}—C2^{ii}—C1^{ii}—C15^{ii}—H15^{ii} [symmetry code: (ii) $x-\frac{1}{2}, -\frac{1}{2}-y, \frac{1}{2}+z$]. There are also two intramolecular rings in (II), namely, *S*(6) C5—H5···O2—S1—N1—C4 and *S*(6) C15—H15*A*···O3—S1—N1—C1 (Fig. 4).

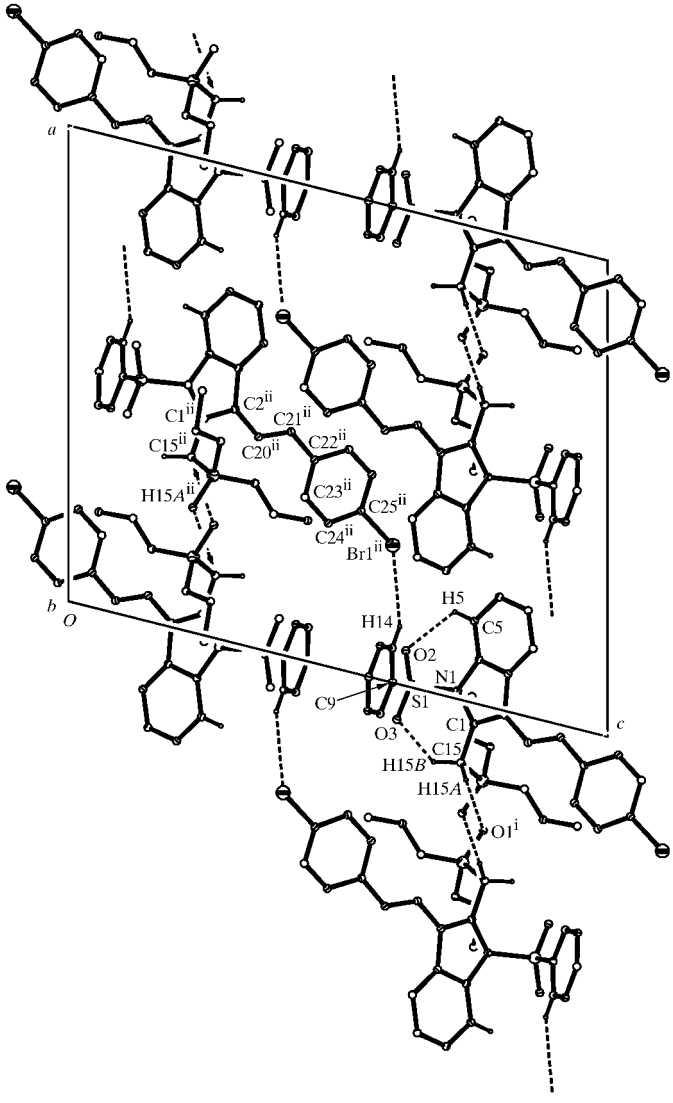

Figure 4
The crystal structure of (II), with the hydrogen-bonding scheme shown as dashed lines [symmetry codes: (i) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $x-\frac{1}{2}, -\frac{1}{2}-y, \frac{1}{2}+z$].

Experimental

For each compound, a mixture of the appropriate 2-bromomethyl-1-phenylsulfonyl-3-(β-arylvinyl)indole (5 mmol) and triethyl phosphite (1.5 g, 9 mmol) was heated under nitrogen at 543 K for 3 h. The sticky oil was then poured over ice (200 g) and acidified with concentrated HCl (1 ml). The solid which precipitated was filtered off and dried over calcium chloride. The crude products were each recrystallized from ethyl acetate to give the phosphonate esters, (I) and (II), as colourless crystalline solids.

Compound (I)

Crystal data

$C_{27}H_{26}Cl_2NO_5PS$
M_r = 578.42
Monoclinic, $P2_1/n$
a = 16.608 (2) Å
b = 8.357 (1) Å
c = 19.647 (5) Å
β = 93.30 (1)°
V = 2722.4 (9) Å^3
Z = 4
D_x = 1.411 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 2.1–25.0°
μ = 0.41 mm^{-1}
T = 293 (2) K
Tablet, colourless
0.36 × 0.36 × 0.21 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.865, T_{max} = 0.917
4904 measured reflections
4759 independent reflections
2932 reflections with $I > 2\sigma(I)$

R_{int} = 0.030
θ_{max} = 25°
$h = -19 \rightarrow 19$
$k = -9 \rightarrow 0$
$l = 0 \rightarrow 23$
3 standard reflections
every 100 reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.181
S = 1.01
4759 reflections
336 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for (I).

P1—O1	1.463 (3)	S1—N1	1.670 (3)
P1—O4	1.566 (3)	S1—C9	1.754 (4)
P1—O5	1.564 (3)	Cl1—C25	1.739 (4)
P1—C15	1.798 (4)	Cl2—C27	1.735 (4)
S1—O2	1.422 (3)	N1—C1	1.424 (5)
S1—O3	1.427 (3)	N1—C4	1.433 (5)
O1—P1—O4	116.1 (2)	O3—S1—N1	107.3 (2)
O1—P1—O5	116.0 (2)	O2—S1—C9	109.6 (2)
O1—P1—C15	113.2 (2)	O3—S1—C9	108.3 (2)
O4—P1—C15	106.9 (2)	C16—O4—P1	125.4 (3)
O5—P1—C15	103.0 (2)	C18—O5—P1	130.8 (4)
O2—S1—N1	106.5 (2)		
O2—S1—N1—C4	38.7 (4)	O3—S1—C9—C10	2.0 (4)
C9—S1—N1—C4	−77.6 (3)	N1—S1—C9—C10	−112.4 (4)
O1—P1—O4—C16	27.0 (5)	O2—S1—C9—C14	−45.5 (4)
O5—P1—O4—C16	152.5 (4)	C2—C1—C15—P1	−83.8 (5)
O4—P1—O5—C18	−114.8 (8)	O1—P1—C15—C1	−164.6 (3)
S1—N1—C1—C15	40.1 (5)	O4—P1—C15—C1	−35.5 (3)
S1—N1—C4—C5	−35.6 (6)	O5—P1—C15—C1	69.4 (3)

Table 2
C—H···O and C—H···halogen interactions (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O2	0.93	2.32	2.896 (6)	120
C10—H10···O3	0.93	2.47	2.867 (7)	106
C15—H15A···O3	0.97	2.20	2.915 (5)	130
C15—H15B···O1^{i}	0.97	2.40	3.347 (5)	165
C23—H23···O1^{i}	0.93	2.46	3.382 (5)	173
C19—H19B···Cl1^{ii}	0.96	2.87	3.826 (7)	172

Symmetry codes: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $x, 1 + y, z$.

Compound (II)

Crystal data

$C_{27}H_{27}BrNO_5PS$
M_r = 588.44
Monoclinic, $P2_1/n$
a = 16.837 (1) Å
b = 8.323 (1) Å
c = 19.611 (1) Å
β = 104.11 (1)°
V = 2665.3 (3) Å^3
Z = 4

D_x = 1.466 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 1.4–24.9°
μ = 1.72 mm^{-1}
T = 293 (2) K
Prism, colourless
0.3 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.461, T_{max} = 0.709
4827 measured reflections
4683 independent reflections
2474 reflections with $I > 2\sigma(I)$

R_{int} = 0.060
θ_{max} = 25°
$h = -20 \rightarrow 19$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 23$
3 standard reflections
every 100 reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.144
S = 1.01
4683 reflections
327 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0637P)^2 + 1.5731P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.012
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 3
Selected geometric parameters (Å, °) for (II).

Br1—C25	1.897 (5)	P1—O5	1.564 (4)
S1—O2	1.412 (3)	P1—O4	1.567 (4)
S1—O3	1.425 (3)	P1—C15	1.792 (4)
S1—N1	1.685 (4)	N1—C4	1.420 (5)
S1—C9	1.753 (5)	N1—C1	1.430 (5)
P1—O1	1.456 (3)		
O2—S1—N1	105.8 (2)	C18—O5—P1	127.5 (4)
O1—P1—O5	116.0 (2)	C4—C3—C8	118.9 (4)
O1—P1—O4	116.5 (2)	C3—C4—C5	122.3 (4)
O1—P1—C15	112.8 (2)	C6—C5—C4	117.5 (5)
O5—P1—C15	103.6 (2)	C5—C6—C7	121.3 (5)
C16—O4—P1	124.3 (3)	C8—C7—C6	121.1 (5)
O2—S1—N1—C4	−46.4 (4)	O3—S1—C9—C10	−20.6 (5)
O1—P1—O4—C16	−29.7 (5)	N1—S1—C9—C10	93.9 (4)
O5—P1—O4—C16	−154.8 (4)	C2—C1—C15—P1	85.9 (5)
S1—N1—C1—C15	−42.0 (5)	O1—P1—C15—C1	163.6 (3)
S1—N1—C4—C5	34.2 (6)	O5—P1—C15—C1	−70.3 (4)
O2—S1—C9—C14	27.7 (5)	O4—P1—C15—C1	34.0 (4)

Table 4
C—H···O and C—H···halogen interactions (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O2	0.93	2.31	2.893 (6)	121
C15—H15B···O3	0.97	2.17	2.904 (6)	132
C15—H15A···O1^{i}	0.97	2.35	3.301 (5)	167
C14—H14···Br1^{ii}	0.93	2.88	3.660 (7)	142

Symmetry codes: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $x - \frac{1}{2}, -\frac{1}{2} - y, \frac{1}{2} + z$.

All the H atoms of both compounds were fixed geometrically and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, and with U_{iso}(H) = 1.5$_{eq}$(C) for methyl H atoms and 1.2U_{eq}(C) for all other H atoms.

For both compounds, data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 2000); software used to prepare material for publication: *SHELX*97 and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1565). Services for accessing these data are described at the back of the journal.

References

Allen, F. H. (1981). *Acta Cryst.* B**37**, 900–906.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Bassindale, A. (1984). *The Third Dimension in Organic Chemistry*, ch. 1, p. 11. New York: John Wiley and Sons.
Beddoes, R. L., Dalton, L., Joule, J. A., Mills, O. S., Street, J. O. & Watt, C. I. F. (1986). *J. Chem. Soc. Perkin Trans.* 2, pp. 787–797.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Boehlow, T., De la Cruz, A., Rath, N. P. & Spilling, C. D. (1997). *Acta Cryst.* C**53**, 1947–1949.
Camerman, A. & Camerman, N. (1975). *Can. J. Chem.* **53**, 2194–2198.
Crawford, J. D. & Kennedy, G. C. (1959). *Nature* (*London*), **183**, 891–892.
Datta, M., Das, A. K., Mazumdar, B., Talapatra, S. K. & Bocelli, G. (1993). *J. Crystallogr. Spectrosc. Res.* **23**, 519–522.
El-Sayed, K., Barnhart, D. M., Ammon, H. L. & Wassel, G. M. (1986). *Acta Cryst.* C**42**, 1383–1385.
Enraf–Nonius (1994). *CAD*-4 *EXPRESS*. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.
Ezra, F. S. & Collin, R. L. (1973). *Acta Cryst.* B**29**, 1398–1403.
Fargasova, A. (1994). *Bull. Environ. Contam. Toxicol.* **52**, 706–711.
Gadaginamath, G. S. & Patil, S. A. (1999). *Indian J. Chem. Sect. B*, **38**, 1070–1074.
Ghosh, M., Basak, A. K., Mazumdar, S. K. & Sheldrick. B. (1989). *J. Crystallogr. Spectrosc. Res.* **19**, 289–298.
Govindasamy, L., Velmurugan, D., Ravikumar, K. & Mohanakrishnan, A. K. (1997). *Acta Cryst.* C**53**, 929–931.
Govindasamy, L., Velmurugan, D., Ravikumar, K. & Mohanakrishnan, A. K. (1998). *Acta Cryst.* C**54**, 635–637.
Grinev, A. N., Trofimkin, Yu. I., Lomanova, E. V., Andreeva, N. I. & Mashkovskii, M. D. (1978). *Khim. Farm. Zh.* **18**, 159–163.
Harms, K. & Wocadlo, S. (1995). *XCAD*4. University of Marburg, Germany.
Hazel, J. P. & Collin, R. L. (1972). *Acta Cryst.* B**28**, 2951–2957.
Howells, M. A., Howells, R. D., Baenziger, N. C. & Burton, D. J. (1973). *J. Am. Chem. Soc.* **95**, 5366–5370.
Ismail, R. (1975). German Patent 1 543 539; *Chem. Abstr.* (1975), **83**, 97419q.
Liu, X.-L., Zhou, Y., Li, W.-Z., Fan, Z., Miao, F.-M., Mao, L.-J. & Chen, R.-Y. (1995). *Acta Cryst.* C**51**, 2350–2352.
Merk, P. (1971). *J. Appl. Phys.* **21**, 62–73.
Moore, C. T. (1989). *Biochemistry and Physiology of Plant Harmones*, ch. 2, p. 33. New Delhi: Narosa Publishing House.
Naidu, S. M., Krishnaiah, M. & Sivakumar, K. (1992). *Acta Cryst.* C**48**, 483–485.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Nigović, B., Antolić, S., Kojić-Prodić, B., Kiralj, R., Magnus, V. & Salopek-Sondi, B. (2000). *Acta Cryst.* B**56**, 94–111.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Okabe, N. & Adachi, Y. (1998). *Acta Cryst.* C**54**, 386–387.
Papenstasion, Z. W. & Newmeyer, J. L. (1972). US Patent 3 674 809; *Chem. Abstr.* (1972). **77**, 126425.
Perales, A. & García-Blanco, S. (1977). *Acta Cryst.* B**33**, 1935–1938.
Polletto, J. P., Allen, G. R. & Weiss, M. J. (1974). US Patent 3 801 594; *Chem. Abstr.* (1974). **81**, 3769.
Rajeswaran, W. G., Labroo, R. B. & Cohen, L. A. (1999). *J. Org. Chem.* **64**, 1369–1371.
Rodriguez, J. G., Temprano, F., Esteban-Calderon, C., Martinez-Ripoll, M. & García-Blanco, S. (1985). *Tetrahedron*, **41**, 3813–3823.
Sanders, T. C., Hammond, G. B., Golen, J. A. & Williard, P. G. (1996). *Acta Cryst.* C**52**, 667–669.
Sankaranarayanan, R., Velmurugan, D., Shanmuga Sundara Raj, S., Fun, H.-K., Babu, G. & Perumal, P. T. (2000). *Acta Cryst.* C**56**, 475–476.
Schollmeyer, D., Fischer, G. & Pindur, U. (1995). *Acta Cryst.* C**51**, 2572–2575.
Seetharaman, J. & Rajan, S. S. (1995). *Acta Cryst.* C**51**, 78–80.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Sivaraman, J., Subramanian, K., Velmurugan, D., Subramanian, E. & Seetharaman, J. (1996). *J. Mol. Struct.* **385**, 123–128.
Spek, A. L. (2000). *PLATON*2000. University of Utrecht, The Netherlands.
Srinivasan, P. C. & Elango, S. (1999). *Synth. Commun.* **29**, 2043–2051.
Srinivasan, P. C. & Mohanakrishnan, A. K. (1995). *J. Org. Chem.* **60**, 1939–1946.
Tanimukai, H., Inoni, M., Hariguchi, S. & Kaneko, Z. (1965). *Biochem. Pharmacol.* **14**, 961–970.
Varghese, B., Srinivasan, S., Padmanabhan, P. V. & Ramadas, S. R. (1986). *Acta Cryst.* C**42**, 1544–1546.
Weichsel, A. & Lis, T. (1996). *Acta Cryst.* C**52**, 97–101.
Yokota, Y., Tsukihara, T., Sakaguchi, K.-I., Hamada, Y. & Takeuchi, I. (1990). *Acta Cryst.* C**46**, 167–168.
Yokum, T. S. & Fronczek, F. R. (1997). *Acta Cryst.* C**53**, 362–363.
Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.