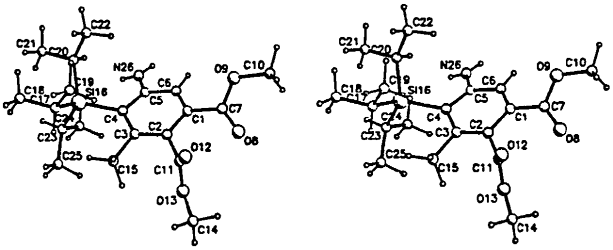

Fig. 1. Stereoscopic view of the molecule (*PLUTO*; Motherwell & Clegg, 1978) showing the atomic numbering scheme.

atomic parameters are given in Table 1.* The bond lengths and the bond angles are listed in Table 2. Fig. 1 shows a stereoscopic view of the molecule.

Related literature. Strong deviations from planarity of non-condensed, non-bridged benzene or pyridine derivatives have been reported for *tert*-butyl 1,2,3-tris(*tert*-butyl)-4,6-bis(methoxycarbonyl)-5-benzoate (I) and 2,3,4-tris(*tert*-butyl)-5-*tert*-butoxycarbonyl-6-ethoxycarbonylpyridine (II) (Maas, Fink, Wingert, Blatter & Regitz, 1987). In those cases the steric overcrowding is caused by *vic*-tri-*tert*-butyl substitution of the aromatic ring which results in unsymmetrical boat conformations with bow and stern angles as high as 30·1 and 11·6° (I) and 25·5 and 12·9° (II).

For silyl aromatic compounds with both *ortho* positions substituted, endocyclic torsion angles similar to those reported here have been observed in the tri-*tert*-butylphenyl moiety of 1,1-difluoro-2,2-di-mesityl-1-(2,4,6-tri-*tert*-butylphenyl)disilane (Weidenbruch, Kramer, Pohl & Saak, 1986).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52465 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Ethyl *p*-Nitrophenylsulfonylacetate

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Abstract. $C_{10}H_{11}NO_6S$, $M_r = 273{\cdot}26$, monoclinic, $P2_1/c$, $a = 11{\cdot}440\,(1)$, $b = 13{\cdot}239\,(2)$, $c = 8{\cdot}131\,(1)$ Å, $\beta = 92{\cdot}69\,(1)°$, $V = 1230$ Å^3, $Z = 4$, $D_x = 1{\cdot}475$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54178$ Å, $\mu = 25{\cdot}04$ cm^{-1}, $F(000) = 568$, $T = 293$ K, $R = 0{\cdot}060$, $wR = 0{\cdot}058$ for 1302 reflections. All bond lengths and angles lie within normal ranges. The ethyl acetate substituent is almost planar [maximum deviation of 0·14 (1) Å for the terminal —CH_2CH_3 group] and makes a dihedral angle of 93·5 (1)° with the plane of the phenyl ring. The NO_2 group lies nearly in the plane of the phenyl ring [max. deviation of

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

	x	y	z	B_{eq}* (Å^2)
S(1)	0·92180 (9)	0·1295 (1)	0·1715 (1)	3·24 (2)
O(1)	0·7708 (4)	−0·2443 (3)	0·6569 (5)	7·6 (1)
O(2)	0·6887 (4)	−0·1205 (4)	0·7776 (5)	8·6 (1)
O(3)	1·0332 (2)	0·0942 (3)	0·1196 (4)	4·0 (1)
O(4)	0·9127 (3)	0·2318 (3)	0·2289 (4)	4·5 (1)
O(5)	0·6633 (3)	0·1855 (4)	0·1410 (5)	8·3 (1)
O(6)	0·6325 (3)	0·1184 (3)	−0·1046 (4)	5·7 (1)
N(1)	0·7472 (3)	−0·1563 (4)	0·6710 (5)	5·5 (1)
C(1)	0·7951 (4)	−0·0814 (4)	0·5526 (5)	3·6 (1)
C(2)	0·8676 (4)	−0·1205 (4)	0·4348 (5)	3·8 (1)
C(3)	0·9075 (4)	−0·0538 (4)	0·3193 (5)	3·3 (1)
C(4)	0·8740 (3)	0·0455 (3)	0·3251 (5)	2·7 (1)
C(5)	0·8032 (4)	0·0831 (4)	0·4451 (5)	3·5 (1)
C(6)	0·7633 (4)	0·0174 (4)	0·5604 (5)	3·7 (1)
C(7)	0·8219 (3)	0·1091 (4)	0·0000 (5)	3·5 (1)
C(8)	0·6977 (4)	0·1423 (4)	0·0259 (5)	4·1 (1)
C(9)	0·5099 (4)	0·1499 (6)	−0·1108 (7)	6·8 (2)
C(10)	0·4521 (5)	0·0950 (6)	−0·2511 (9)	9·7 (2)

$$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

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

S(1)—O(3)	1·439 (3)	N(1)—C(1)	1·504 (6)
S(1)—O(4)	1·438 (4)	C(1)—C(2)	1·396 (6)
S(1)—C(4)	1·776 (4)	C(1)—C(6)	1·360 (7)
S(1)—C(7)	1·782 (4)	C(2)—C(3)	1·382 (7)
O(1)—N(1)	1·202 (7)	C(3)—C(4)	1·371 (6)
O(2)—N(1)	1·216 (7)	C(4)—C(5)	1·389 (6)
O(5)—C(8)	1·180 (6)	C(5)—C(6)	1·373 (6)
O(6)—C(8)	1·306 (5)	C(7)—C(8)	1·511 (6)
O(6)—C(9)	1·462 (6)	C(9)—C(10)	1·482 (9)
O(3)—S(1)—C(4)	118·7 (3)	C(1)—C(2)—C(3)	117·1 (5)
O(3)—S(1)—C(4)	108·1 (2)	C(2)—C(3)—C(4)	119·2 (4)
O(3)—S(1)—C(7)	105·4 (2)	S(1)—C(4)—C(3)	118·7 (4)
O(4)—S(1)—C(4)	109·4 (2)	S(1)—C(4)—C(5)	118·6 (4)
O(4)—S(1)—C(7)	110·0 (2)	C(3)—C(4)—C(5)	122·7 (5)
C(4)—S(1)—C(7)	104·4 (2)	C(4)—C(5)—C(6)	118·4 (5)
C(8)—O(6)—C(9)	118·0 (4)	C(1)—C(6)—C(5)	118·7 (4)
O(1)—N(1)—O(2)	125·5 (5)	S(1)—C(7)—C(8)	114·8 (3)
O(1)—N(1)—C(1)	119·2 (4)	O(5)—C(8)—O(6)	124·2 (4)
O(2)—N(1)—C(1)	115·3 (5)	O(5)—C(8)—C(7)	127·1 (5)
N(1)—C(1)—C(2)	116·3 (4)	O(6)—C(8)—C(7)	108·8 (4)
N(1)—C(1)—C(6)	119·9 (4)	O(6)—C(9)—C(10)	106·3 (5)
C(2)—C(1)—C(6)	123·8 (4)		

0·171 (4) Å for the O(1) atom]. The crystal structure contains some short C—H···O contacts.

Experimental. The title compound was obtained following Borchardt, Janota & Zakrzewski (1986). Colourless crystal of approximate dimensions 0·3 × 0·3 × 0·3 mm mounted on glass fibre. Enraf–Nonius CAD-4 four-circle diffractometer, graphite-monochromated Cu *Kα*, cell dimensions from 25 reflections ($9 < \theta < 19°$), data collection $2 \leq \theta \leq 60°$ ($-9 \leq h \leq 9$, $0 \leq k \leq 12$, $0 \leq l \leq 14$), ω–2θ scans, ω-scan angle $(0{\cdot}65 + 0{\cdot}14\tan\theta)°$, three reference reflections measured every hour showed no significant intensity decay (total loss of intensity < 1%), total number of reflections measured 1716, 1707 unique (R_{int} = 0·01), Lorentz–polarization correction but not for absorption. Structure solved by direct methods (*MULTAN*11/12; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), full-matrix least-squares refinement on $|F|$ for 1302 reflections with $F_o \geq 2\sigma(F)$. Parameters refined: one scale factor, positional parameters and anisotropic temperature factors for non-H atoms (positions of H atoms calculated with C—H = 0·95 Å and B_{iso} = 5·0 Å^2 were not refined). R = 0·060, wR = 0·058, S = 1·14, unit weights; $(\Delta/\sigma)_{max}$ = 0·03; $(\Delta\rho)_{max}$ = 0·35 and $(\Delta\rho)_{min}$ = −0·26 e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974) as well as anomalous-dispersion terms; all calculations performed with *SDP* system (Frenz, 1978), *PARST* (Nardelli, 1983) and *ORTEP* (Johnson, 1976). Final atomic coordinates are given in Table 1* and bond lengths and angles in Table 2. The molecule with atomic numbering scheme is shown in Fig. 1.

Related literature. Ethyl *p*-nitrophenylsulfonylacetate is an example of a *p*-disubstituted benzene derivative with two electronegative substituents. The Huheey (1966) electronegativities for NO_2 and SO_2R are 4·65 and 3·2°, respectively, leading to the deformation of the geometry of the ring in agreement with the Walsh rule (Bent, 1961; Krygowski 1984) which predicts an increase of both *ipso* angles and a positive or close to

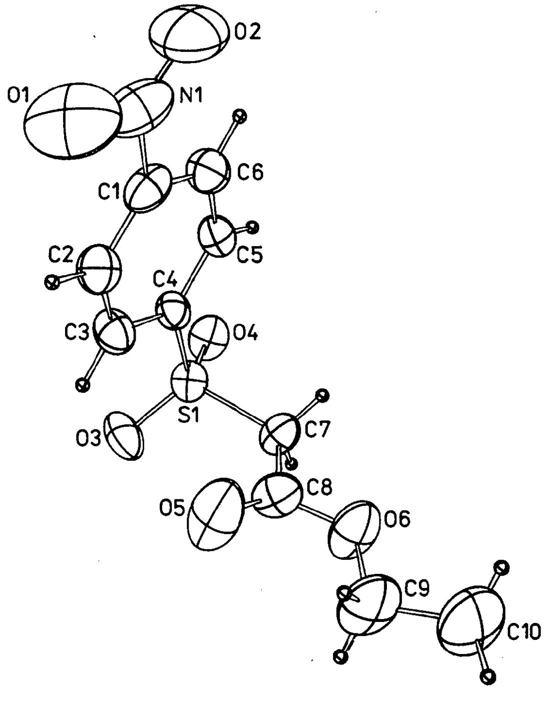

Fig. 1. *ORTEP* (Johnson, 1976) drawing of the molecule.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52434 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

zero Δ ($= b - a$) value (where a and b are the *ipso–ortho* and *ortho–meta* C—C bond lengths respectively). In the crystal structure there are close non-bonded C—H···O intermolecular contacts, C(2)···O(1) $(x, -\frac{1}{2} - y, -\frac{1}{2} + z)$ 3·050 (6) Å and C(7)···O(4) $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$ 3·254 (6) Å, somewhat longer than those reported for phenacyl phenyl sulfone (Krawiec, Krygowski & Zakrzewski, 1989).

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Structure of the Tetrabutylammonium Salt of 2,4,4,6,6-Pentachloro-1,3,5,2λ^5,4λ^5,6λ^5-triazatriphosphorinine 2-Oxide

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Abstract. $[N(C_4H_9)_4][N_3P_3Cl_5O]$, $M_r = 570·67$, monoclinic, $P2_1/c$, $a = 10·184\,(2)$, $b = 18·072\,(3)$, $c = 16·448\,(2)$ Å, $\beta = 106·88\,(2)°$, $V = 2896·8\,(9)$ Å^3, $Z = 4$, $D_x = 1·308$ g cm^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71073 Å, $\mu = 6·8$ cm^{-1}, $F(000) = 1192$, $T = 298$ K, $R_F = 0·082$, $wR = 0·069$ for 2089 unique observed reflections with $I \geq 1·5\sigma(I)$ and 263 parameters. The inorganic ring skeleton is nearly flat, tending somewhat to a crown conformation. The P—N bond lengths (mean values) are: N—P(OCl) 1·606 (5) Å and N—P(Cl$_2$) 1·540 (5), 1·58 (1) Å.

Experimental. Suitable crystals were obtained by recrystallization from a heptane–chloroform mixture. A block-shaped, transparent, colourless crystal (0·07 × 0·15 × 0·20 mm) was glued on top of a glass fibre and transferred to an Enraf-Nonius CAD-4F diffractometer interfaced to a MicroVAX 2000 computer. Graphite-monochromated Mo $K\bar{\alpha}$ radiation used to obtain 4957 reflections (h $-12 \rightarrow 0$, k $0 \rightarrow 22$, l $-12 \rightarrow 19$); $1·13 < \theta < 26·0°$; $[(\sin\theta)]_{\max}/\lambda = 0·6168$ Å^{-1}; $\omega/2\theta$ scan, $\Delta\omega = (1·0 + 0·35\,\mathrm{tg}\,\theta)°$. Unit-cell parameters and their e.s.d.'s were derived from setting angles of 22 reflections ($13·84 < \theta < 16·87°$) in four alternate settings (de Boer & Duisenberg, 1984). Reduced cell calculations did not indicate any higher lattice symmetry (Spek, 1988). Three reference reflections measured every 2 h (012: r.m.s.d. 2·6%, $\bar{2}00$: r.m.s.d. 2·5%, $\bar{1}04$: r.m.s.d. 2·9%) showed a linear decay of 11·8% during the 89·5 h of X-ray exposure time. A 360° ψ scan for a reflection close to axial ($\overline{42}\bar{1}$) showed a variation in intensity of up to 10% about the mean value. The intensity data were corrected for this decay, for Lorentz and polarization effects, but not for absorption. Standard deviations in the intensities based on counting statistics were increased according to an analysis of the excess variance (McCandlish, Stout &

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