A diagram of a portion of the unit-cell packing is presented as Fig. 2. There is a distinct separation of hydrophobic and hydrophilic groups within the crystal. Nonbonded hydrophobic interactions between adjacent undecane chains undoubtedly contribute to crystal stabilization. In addition, there are two strong intermolecular hydrogen bonds involving both carboxyl groups interacting in a dimeric fashion across crystallographic inversion centers. The associated metrical parameters are $O(2)\cdots O(3) = 2\cdot648$ (2), $H1(O2)\cdots O(3) = 1\cdot67$ (3) Å, $O(2)-H1(O2)\cdots O(3) = 170$ (3)°, $O(4)\cdots O(5) = 2\cdot673$ (3), $H1(O4)\cdots O(5) = 1\cdot81$ (4) Å and $O(4)-H1(O4)\cdots O(5) = 176$ (3)°.

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Structure of Antischistosome Compounds. I. (2-Aminoethyl)triphenylphosphonium Bromide Hydrobromide

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Abstract. $C_{20}H_{22}NP^{2+}.2Br^-$, $M_r = 467\cdot2$, orthorhombic, $P2_12_12_1$, $a = 9\cdot262$ (2), $b = 12\cdot846$ (2), $c = 16\cdot845$ (3) Å, $V = 2004\cdot2$ (2) Å^3, $Z = 4$, $D_x = 1\cdot548$ g cm^{-3}, graphite-monochromated Cu $K\bar{\alpha}$ radiation, $\lambda = 1\cdot5418$ Å, $\mu = 59\cdot45$ cm^{-1}, $F(000) = 936$, $T = 292$ K, $R = 0\cdot034$ and $wR = 0\cdot035$ for 2158 unique reflections with $I > 3\sigma(I)$. The structure contains two non-bonded bromide ions and a divalent tetrahedral (aminoethyl)triphenylphosphonium cation. The four P–C distances are in the range $1\cdot790$ (4)–$1\cdot798$ (5) Å with a mean of $1\cdot795$ (3) Å. The six C–P–C angles range from $107\cdot5$ (2) to $111\cdot3$ (2)°. The aminoethyl group is extended with torsion angles of $-161\cdot1$ (3)° for P–C–C–N and $-175\cdot2$ (3), $-56\cdot6$ (6) and $66\cdot7$ (6)° for the C–P–C–C angles. The closest phosphorus–bromine distance is $4\cdot580$ (1) Å. The ammonium ion moiety interacts with four bromide ions to form a network which disperses and neutralizes the charges.

Introduction. The title compound is one of a series of alkyl-, alkylamino- and poly(methylene)bis(triphenylphosphonium) compounds which exhibit varying effects on the cholinergic nervous system of *Schistosoma mansoni* (McAllister, Dotson, Grim & Hillman, 1980). The biological effects of these compounds cannot be

explained solely on the basis of chemical differences in the substituted moiety. The effects may also be due to differences in the torsion angles in the substituted moiety. Therefore, the crystal structure determinations of the compounds in this series were initiated. This communication reports the first structure of this series to be completed.

Experimental. Title compound synthesized as described by McAllister et al. (1980) and was a gift from Dr G. R. Hillman; colorless parallelepiped crystal, $0.4 \times 0.5 \times 0.9$ mm, grown from ethanol solution by slow evaporation; systematic absences characteristic of $P2_12_12_1$; Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, cell parameters from least-squares refinement of 20 reflections with $4 < \theta < 46°$; $\omega/2\theta$ scan, ω width $(0.80 + 0.15\tan\theta)°$, $(\sin\theta/\lambda)_{max} = 0.6094$ Å^{-1}, $h = 0$ to 11, $k = 0$ to 15, $l = 0$ to 20; intensities of three standard reflections (011, 023, 208) monitored every 2 h showed no radiation decay or significant variation; 2184 unique measured intensities (systematically absent reflections not measured), 2158 with $I > 3\sigma(I)$; five reflections (10,6,2, 6,10,9, 0,9,15, 0,5,15, 0,6,16) with $I <$ background excluded; Lorentz–polarization corrections applied; structure solution from Patterson, Fourier and ΔF synthesis; full-matrix least-squares refinement minimizing $[w(F_o - F_c)^2]$ with unit weights and isotropic temperature factors converged at $R = 0.085$, $wR = 0.102$; min. and max. absorption correction of 0.842 and 1.322, using program DIFABS (Walker & Stuart, 1983), R and wR decreased to 0.066 and 0.070; atoms refined anisotropically to $R = 0.045$, $wR = 0.053$; 17 H atoms located from ΔF map, 5 placed in geometrically calculated positions, refined isotropically; full-matrix least-squares refinement with unit weights, 306 parameters with secondary-extinction correction, $g = 1.02$ (3) $\times 10^{-6}$ (Stout & Jensen, 1968), $R = 0.034$, $wR = 0.035$, $S = 1.43$, max. $\Delta/\sigma = 0.20$ for non-hydrogens, 0.47 for hydrogens; no significant features in final ΔF synthesis with max. $\Delta\rho = \pm 0.36$ e Å^{-3}, atomic scattering factors, f' and f'', from International Tables for X-ray Crystallography (1974); all calculations with DEC PDP 11/44 computer using the Enraf–Nonius Structure Determination Package Plus (Frenz, 1984) and ORTEP (Johnson, 1976).

Discussion. Table 1* contains the fractional atomic coordinates and equivalent isotropic thermal

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles and least-squares planes calculations for the phenyl rings and the N–Br complex have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42532 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters and their e.s.d.'s*

	x	y	z	B_{eq}(Å^2)
Br(1)	0.50183 (7)	0.08499 (5)	0.51083 (4)	3.80 (1)
Br(2)	0.54913 (7)	0.43481 (5)	0.47017 (4)	3.23 (1)
P	0.1023 (2)	0.1191 (1)	0.26268 (8)	2.00 (2)
C(11)	0.1203 (6)	0.1987 (4)	0.3497 (3)	2.6 (1)
C(12)	0.2734 (6)	0.2414 (5)	0.3635 (3)	2.8 (1)
N(13)	0.2828 (6)	0.2736 (4)	0.4497 (3)	3.3 (1)
C(21)	−0.0854 (6)	0.0864 (4)	0.2525 (3)	2.30 (9)
C(22)	−0.1742 (7)	0.0753 (5)	0.3185 (3)	3.4 (1)
C(23)	−0.3134 (7)	0.0414 (5)	0.3090 (4)	3.8 (1)
C(24)	−0.3673 (6)	0.0213 (5)	0.2352 (4)	3.8 (1)
C(25)	−0.2808 (8)	0.0337 (5)	0.1695 (4)	3.7 (1)
C(26)	−0.1387 (6)	0.0657 (5)	0.1778 (3)	3.1 (1)
C(31)	0.1591 (6)	0.1904 (4)	0.1764 (3)	2.3 (1)
C(32)	0.0777 (7)	0.2757 (5)	0.1535 (4)	3.2 (1)
C(33)	0.1187 (9)	0.3362 (5)	0.0901 (4)	4.3 (2)
C(34)	0.246 (1)	0.3122 (5)	0.0498 (4)	5.0 (2)
C(35)	0.3257 (8)	0.2279 (6)	0.0715 (4)	4.9 (2)
C(36)	0.2849 (7)	0.1661 (5)	0.1358 (4)	3.4 (1)
C(41)	0.2030 (6)	0.0010 (4)	0.2737 (3)	2.15 (9)
C(42)	0.2623 (7)	−0.0253 (4)	0.3471 (4)	2.9 (1)
C(43)	0.3352 (8)	−0.1208 (5)	0.3555 (4)	3.9 (1)
C(44)	0.3470 (8)	−0.1854 (5)	0.2909 (5)	4.2 (1)
C(45)	0.2911 (8)	−0.1586 (4)	0.2197 (4)	3.8 (1)
C(46)	0.2167 (7)	−0.0652 (4)	0.2101 (3)	3.0 (1)
H(111)	0.049 (7)	0.243 (5)	0.340 (3)	4 (1)*
H(112)	0.099 (7)	0.129 (5)	0.407 (4)	4 (2)*
H(121)	0.284 (6)	0.306 (4)	0.333 (3)	3 (1)*
H(122)	0.350 (7)	0.175 (5)	0.357 (4)	4 (2)*
H(131)	0.36 (1)	0.191 (7)	0.488 (6)	12 (3)*
H(132)	0.162 (9)	0.326 (7)	0.474 (5)	10 (3)*
H(133)	0.38 (1)	0.319 (7)	0.463 (6)	12 (3)*
H(22)	−0.138 (5)	0.079 (4)	0.356 (3)	2 (1)*
H(23)	−0.38 (1)	0.040 (8)	0.347 (6)	12 (3)*
H(24)	−0.468 (6)	0.001 (4)	0.232 (3)	3 (1)*
H(25)	−0.314 (8)	0.014 (6)	0.125 (4)	7 (2)*
H(26)	−0.070 (7)	0.068 (5)	0.130 (4)	5 (2)*
H(32)	−0.007 (7)	0.286 (4)	0.177 (3)	4 (1)*
H(33)	0.04 (1)	0.390 (7)	0.051 (5)	11 (3)*
H(34)	0.279 (8)	0.361 (6)	0.001 (4)	7 (2)*
H(35)	0.409 (7)	0.192 (5)	0.055 (4)	5 (2)*
H(36)	0.344 (8)	0.100 (6)	0.156 (4)	6 (2)*
H(42)	0.260 (9)	0.028 (5)	0.387 (4)	6 (2)*
H(43)	0.368 (7)	−0.149 (5)	0.415 (4)	4 (2)*
H(44)	0.391 (9)	−0.241 (6)	0.300 (5)	7 (2)*
H(45)	0.305 (6)	−0.198 (4)	0.190 (3)	3 (1)*
H(46)	0.186 (6)	−0.053 (4)	0.161 (3)	3 (1)*

* Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as $\frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i\cdot\mathbf{a}_j$.

parameters. Fig. 1 shows the structure of the molecule with the thermal vibration ellipsoids of the non-hydrogen atoms. Packing diagram is in Fig. 2. The bond lengths and angles of the non-hydrogen atoms are given in Table 2. The phenyl ring numbers (n) are assigned on the basis of the C(n1)–P–C(11)–C(12) torsion angles in order to facilitate comparisons with other structures of this type of compound. The phenyl ring *trans* to the substituted group in this series of compounds is designated as ring 2, ring 3 is *gauche*⁻ and ring 4 is *gauche*⁺. Least-squares planes calculations show that all phenyl rings are planar.

The phosphorus atom has a nearly perfect tetrahedral arrangement (Table 2). The average P–C bond length is 1.795 (3) Å. The $3.8°$ range of the C–P–C bond angles is comparable to the 2.1–$7.3°$ ranges observed in other triphenylphosphonium compounds (Skapski & Stephens, 1974; Bart, Bassi & Calcaterra, 1980, 1981; Archer, Modro & Nassimbeni, 1981; Kovacs & Parkanyi, 1982; Henichart, Houssin,

Vaccher, Foulon & Baert, 1983; Ponnuswamy & Czerwinski, 1986). The P—C(11)—C(12)—N(13) torsion angle indicates that the aminoethyl group is in the extended conformation (Table 2). This torsion angle is approximately 90° from the P—C—C(benzyl)—C(benzyl) torsion angles observed in the chloride, iodide and bromide structures of benzyltriphenylphosphonium halide (Skapski & Stephens, 1974; Archer *et al.*, 1981; Ponnuswamy & Czerwinski, 1986). The nitrogen atom interacts with four bromide ions to form an approximately square pyramid (Fig. 3, Table 2). The nitrogen atom is at the apex and is 0·786 (5) Å above the mean plane formed by the bromide ions. The

lengths of three of the nitrogen–bromide ion interactions are less than the sum of the van der Waals radii, indicating strong interactions (Pauling, 1960; Shrake & Rupley, 1973). ΔF synthesis shows that the three hydrogens bonded to the nitrogen are nearly coincident with these three N—Br vectors. The fourth bromide ion,
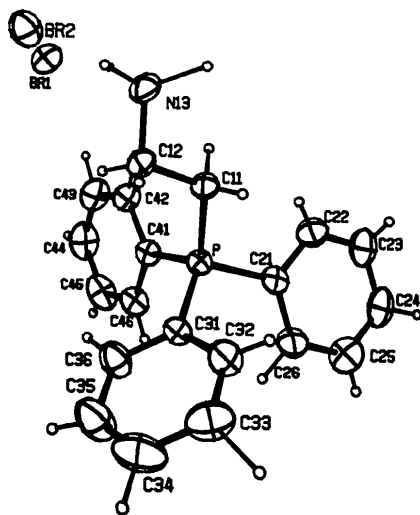

Fig. 1. Perspective view of the molecule showing the atom-labelling scheme. Thermal ellipsoids are depicted at the 50% probability level.
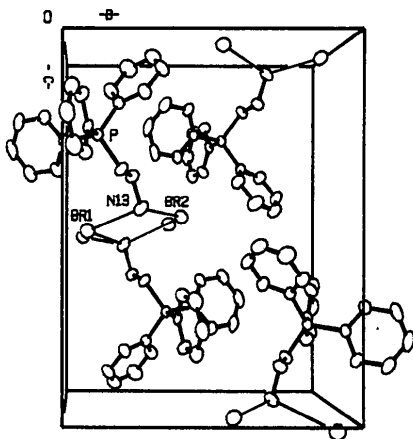

Fig. 2. Perspective view of the packing of the molecules in a unit cell along the a axis. N, Br and P atoms of molecule at x,y,z are labelled. Single lines show N–Br non-bonded interactions.

Table 2. *Bond distances* (Å), *bond angles* (°), *nonbonding angles* (°) *and selected torsion angles* (°) *for non-hydrogen atoms*

P	C(11)	1·796 (5)	C(31)	C(32)	1·385 (7)
P	C(21)	1·796 (5)	C(31)	C(36)	1·386 (7)
P	C(31)	1·798 (5)	C(32)	C(33)	1·373 (8)
P	C(41)	1·790 (4)	C(33)	C(34)	1·392 (10)
C(11)	C(12)	1·539 (7)	C(34)	C(35)	1·362 (10)
C(12)	N(13)	1·512 (6)	C(35)	C(36)	1·394 (8)
C(21)	C(22)	1·389 (7)	C(41)	C(42)	1·394 (7)
C(21)	C(26)	1·378 (6)	C(41)	C(46)	1·373 (7)
C(22)	C(23)	1·370 (8)	C(42)	C(43)	1·408 (7)
C(23)	C(24)	1·365 (9)	C(43)	C(44)	1·373 (9)
C(24)	C(25)	1·376 (9)	C(44)	C(45)	1·352 (9)
C(25)	C(26)	1·385 (8)	C(45)	C(46)	1·393 (8)

C(11)	P	C(21)	107·5 (2)	P	C(31)	C(32)	118·0 (4)
C(11)	P	C(31)	110·1 (2)	P	C(31)	C(36)	122·0 (4)
C(11)	P	C(41)	110·4 (2)	C(32)	C(31)	C(36)	119·9 (5)
C(21)	P	C(31)	109·0 (2)	C(31)	C(32)	C(33)	120·8 (6)
C(21)	P	C(41)	108·4 (2)	C(32)	C(33)	C(34)	119·2 (6)
C(31)	P	C(41)	111·3 (2)	C(33)	C(34)	C(35)	120·3 (6)
P	C(11)	C(12)	114·3 (4)	C(34)	C(35)	C(36)	120·9 (6)
C(11)	C(12)	N(13)	107·2 (4)	C(31)	C(36)	C(35)	118·8 (6)
P	C(21)	C(22)	121·4 (4)	P	C(41)	C(42)	120·2 (4)
P	C(21)	C(26)	118·6 (4)	P	C(41)	C(46)	119·5 (4)
C(22)	C(21)	C(26)	119·9 (5)	C(42)	C(41)	C(46)	120·3 (4)
C(21)	C(22)	C(23)	119·8 (6)	C(41)	C(42)	C(43)	119·3 (5)
C(22)	C(23)	C(24)	120·7 (6)	C(42)	C(43)	C(44)	119·0 (6)
C(23)	C(24)	C(25)	119·8 (5)	C(43)	C(44)	C(45)	121·3 (6)
C(24)	C(25)	C(26)	120·4 (6)	C(44)	C(45)	C(46)	120·7 (6)
C(21)	C(26)	C(25)	119·4 (5)	C(41)	C(46)	C(45)	119·3 (5)

Br(1)	N(13)	Br(1ⁱ)	146·6 (2)	Br(1ⁱ)	Br(2)	N(13)	38·34 (9)
Br(1)	N(13)	Br(2)	88·2 (1)	Br(2)	Br(1ⁱ)	Br(2ⁱ)	87·88 (1)
Br(2)	Br(1)	Br(2ⁱ)	99·57 (2)	Br(2)	Br(1ⁱ)	N(13)	38·29 (9)
Br(2)	Br(1)	N(13)	45·14 (9)	Br(1ⁱ)	N(13)	Br(2ⁱ)	82·0 (1)
Br(2ⁱ)	Br(1)	N(13)	57·32 (9)	Br(2)	N(13)	Br(2ⁱ)	150·4 (1)
Br(1)	Br(2)	Br(1ⁱ)	81·12 (1)	Br(2ⁱ)	Br(1ⁱ)	N(13)	53·31 (9)
Br(1)	N(13)	Br(2ⁱ)	73·5 (1)	Br(1)	Br(2ⁱ)	Br(1ⁱ)	91·41 (1)
Br(1ⁱ)	N(13)	Br(2)	103·4 (1)	Br(1)	Br(2ⁱ)	N(13)	49·16 (8)
Br(1)	Br(2)	N(13)	46·67 (9)	Br(1ⁱ)	Br(2ⁱ)	N(13)	44·67 (8)

C(21)	P	C(11)	C(12)	−175·2 (3)	C(41)	P	C(11)	C(12)	66·7 (6)
C(31)	P	C(11)	C(12)	−56·6 (6)	P	C(11)	C(12)	N(13)	−161·1 (3)

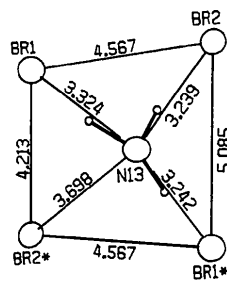Symmetry code: (i) $x-\frac{1}{2}, \frac{1}{2}-y, 1-z$.

Fig. 3. Perspective drawing down the c axis showing the N–Br interactions. Asterisks indicate Br atoms at symmetry position $x-\frac{1}{2}, \frac{1}{2}-y, 1-z$. Distances are in ångströms with N–Br and Br–Br standard deviations of 0·005 and 0·001 Å, respectively.

which is farthest from the nitrogen atom, is also the bromide ion closest to the phosphorus atom |4·580 (1) Å|. The ΔF map does not show any indication of a hydrogen atom in the region between this bromide ion and the phosphorus atom. This bromide ion is 'face' oriented with respect to the tetrahedral phosphonium center. This phosphorus to bromide ion arrangement is not significantly different from those of phosphorus to halide ions in other triphenylphosphonium structures (Archer *et al.*, 1981). Each bromide-ion pair also interacts with another nitrogen atom from a symmetry-related molecule, thus forming a network in the crystal which disperses and neutralizes the charges. The region occupied by this network is a channel parallel to the crystallographic *a* axis (Fig. 2). The charged atoms avoid the phenyl rings as expected (Archer *et al.*, 1981).

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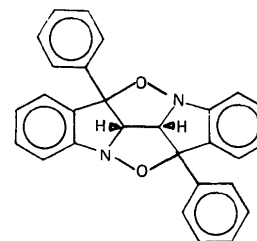# Structure of 1,3':1',3-Diepoxy-3,3'-diphenyl-2,2'-biindolinyl

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Abstract. $C_{28}H_{20}N_2O_2$, $M_r = 416\cdot48$, monoclinic, $C2/c$, $a = 23\cdot85$ (1), $b = 7\cdot90$ (1), $c = 13\cdot07$ (1) Å, $\beta = 120\cdot4$ (2)°, $V = 2124\cdot0$ Å^3, $Z = 4$, $D_x = 1\cdot303$, $D_m = 1\cdot309$ Mg m^{-3}, $\lambda(\text{Cu }K\alpha) = 1\cdot5418$ Å, $\mu = 0\cdot58$ mm^{-1}, $F(000) = 872$, $T = 293$ K, $R = 0\cdot056$ for 1751 unique observed reflexions. The molecule contains four five-membered rings fused into a dome. The O atoms lie out of plane of their rings to maximize the $O(1)\cdots O(1')$ distance. The phenyl groups are placed to minimize steric effects. The principal motion of the phenyl groups is a wag about the points of attachment to the molecule, $C(13)$ and $C(13')$.

Introduction. The title compound (I) was crystallized in *ca* 8% yield from the reaction of phenylacetylene and nitrosobenzene. The chemical properties and a partially

refined crystal structure (R 0·13) have been reported previously (Iball, Motherwell, Pollock & Tedder, 1968). The model has now been refined to convergence using the original data set.

(I)

Experimental. The colourless compound crystallizes in well formed diamond-shape prisms and the samples

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