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Structure of Antischistosome Compounds. IV. (3-Bromopropyl)triphenylphosphonium Bromide

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Abstract. $C_{21}H_{21}BrP^+.Br^-$, $M_r = 464{\cdot}2$, monoclinic, $P2_1/c$, $a = 11{\cdot}165\ (2)$, $b = 10{\cdot}160\ (2)$, $c = 17{\cdot}614\ (3)$ Å, $\beta = 104{\cdot}99\ (1)^\circ$, $V = 1930{\cdot}08$ Å^3, $Z = 4$, $D_x = 1{\cdot}597$ g cm^{-3}, graphite-monochromatized Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å, $\mu = 61{\cdot}6$ cm^{-1}, $F(000) = 928$, $T = 292$ K. Final $R = 0{\cdot}052$ for 3255 observed reflections with $I > 3\sigma(I)$. Structure solved by direct methods. The bromopropyl moiety is in an extended conformation with the *gauche*$^+$ C—C—C—Br torsion angle orienting the C—Br vector in a direction similar to the direction of the N—Br vector observed in the (2-aminoethyl)triphenylphosphonium bromide hydrobromide structure and the cyano vector in the (3-cyanopropyl)triphenylphosphonium bromide structure.

Experimental. Sample provided by Professor G. Hillman, synthesized as described by McAllister, Dotson, Grim & Hillman (1980); colorless crystals (from ethanol), 0·50 × 0·50 × 0·20 mm; Enraf–Nonius CAD-4 diffractometer; cell parameters from 2θ values for 20 reflections from least-squares refinement with $6 < 2\theta < 47^\circ$; ω–2θ scan, width $(0{\cdot}80 + 0{\cdot}15\tan\theta)^\circ$, $[(\sin\theta)/\lambda]_{\max} = 0{\cdot}6092$ Å^{-1}, $0 \leq h \leq 13$, $0 \leq k \leq 12$, $-21 \leq l \leq 21$; intensities of three standard reflections monitored every 7200 s showed a linear decline in intensity of 0·8%, correction applied; 3670 unique reflections measured, 3255 reflections with $I > 3\sigma(I)$; Lp corrections; structure solved by *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) which revealed the P and two Br atoms; C and H atoms revealed by Fourier and difference Fourier syntheses; full-matrix least squares minimizing $\sum w(F_o - F_c)^2$ with unit weights; min. and max. absorption correction of 0·608 and 1·810, respectively, using program *DIFABS* (Walker & Stuart, 1983); final $R = 0{\cdot}052$, $wR = 0{\cdot}080$; all H atoms from ΔF map and refined isotropically; 302 variables with secondary-extinction correction, $g = 1{\cdot}46\ (19) \times 10^{-6}$ (Stout & Jensen, 1968), $S = 1{\cdot}89$; max. $\Delta/\sigma = 0{\cdot}00$ for non-H atoms, 0·02 for H atoms; no significant features in final ΔF synthesis with $\Delta\rho$ max. and min. $= 1{\cdot}32\ (2)$ and $-1{\cdot}30\ (2)$ e Å^{-3}, respectively, around the Br$^-$ ion; atomic scattering factors, f' and f'', from *International Tables for X-ray Crystallography* (1974); all calculations with a DEC PDP 11/44 computer using Enraf–Nonius *SDP-Plus* package (Frenz, 1985). Table 1* lists the fractional atomic coordinates of the non-H atoms and isotropic thermal parameters. Fig. 1 shows the structure of the molecule with the thermal-vibration ellipsoids of the non-H atoms. The packing of the molecules is shown in Fig. 2. All intermolecular distances

Table 1. *Positional and equivalent isotropic thermal parameters*

Anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as:

$$B_{\rm eq} = \tfrac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab\cos\gamma B_{12} + ac\cos\beta B_{13} + bc\cos\alpha B_{23}].$$

Numbers in parentheses are e.s.d.'s in the least-significant digits.

	x	y	z	$B_{\rm eq}$(Å^2)
Br1	0·88574 (3)	1·12915 (4)	0·33143 (2)	3·185 (8)
P	0·72582 (6)	0·80271 (7)	0·46238 (4)	1·96 (1)
C11	0·8247 (3)	0·9350 (3)	0·5108 (2)	2·45 (6)
C12	0·7772 (3)	0·9974 (3)	0·5767 (2)	3·01 (6)
C13	0·8464 (4)	1·1193 (4)	0·6093 (2)	3·39 (7)
Br14	0·80437 (6)	1·26475 (5)	0·53509 (3)	5·45 (1)
C21	0·8048 (3)	0·6979 (3)	0·4094 (2)	2·14 (5)
C22	0·8416 (3)	0·7519 (4)	0·3461 (2)	2·95 (6)
C23	0·9024 (3)	0·6734 (4)	0·3034 (2)	3·31 (7)
C24	0·9264 (3)	0·5438 (4)	0·3227 (2)	3·58 (7)
C25	0·8903 (3)	0·4890 (4)	0·3855 (2)	3·65 (8)
C26	0·8297 (3)	0·5653 (3)	0·4292 (2)	3·14 (7)
C31	0·6793 (3)	0·7081 (3)	0·5360 (2)	2·28 (5)
C32	0·7740 (3)	0·6567 (4)	0·5975 (2)	3·20 (7)
C33	0·7429 (4)	0·5838 (4)	0·6565 (2)	3·63 (7)
C34	0·6214 (4)	0·5654 (4)	0·6552 (2)	3·80 (7)
C35	0·5272 (4)	0·6155 (4)	0·5947 (2)	4·14 (8)
C36	0·5558 (3)	0·6883 (4)	0·5346 (2)	3·17 (7)
C41	0·5924 (3)	0·8679 (3)	0·3919 (2)	2·32 (6)
C42	0·5756 (3)	1·0021 (3)	0·3807 (2)	3·05 (7)
C43	0·4749 (4)	1·0479 (4)	0·3232 (2)	3·87 (8)
C44	0·3923 (3)	0·9610 (5)	0·2778 (2)	4·12 (8)
C45	0·4086 (3)	0·8280 (5)	0·2892 (2)	4·24 (9)
C46	0·5094 (3)	0·7798 (4)	0·3455 (2)	3·46 (7)

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and thermal parameters, bond lengths and angles involving H atoms, torsion angles and least-squares-planes' calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51083 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 2. *Bond distances* (Å), *angles* (°) *and selected torsion angles* (°)

Numbers in parentheses are e.s.d.'s in the least-significant digits.

P	C11	1·808 (3)	C31	C32	1·404 (4)
P	C21	1·791 (2)	C31	C36	1·387 (4)
P	C31	1·795 (2)	C32	C33	1·392 (4)
P	C41	1·801 (3)	C33	C34	1·365 (5)
C11	C12	1·533 (4)	C34	C35	1·386 (5)
C12	C13	1·494 (4)	C35	C36	1·395 (4)
C13	Br14	1·948 (3)	C41	C42	1·384 (4)
C21	C22	1·398 (4)	C41	C46	1·391 (4)
C21	C26	1·402 (3)	C42	C43	1·386 (4)
C22	C23	1·387 (4)	C43	C44	1·374 (5)
C23	C24	1·369 (5)	C44	C45	1·372 (6)
C24	C25	1·387 (5)	C45	C46	1·383 (4)
C25	C26	1·385 (4)			

C11	P	C21	111·4 (1)	P	C31	C32	117·1 (2)
C11	P	C31	108·2 (1)	P	C31	C36	122·5 (2)
C11	P	C41	110·3 (1)	C32	C31	C36	120·4 (2)
C21	P	C31	109·4 (1)	C31	C32	C33	119·4 (3)
C21	P	C41	106·9 (1)	C32	C33	C34	120·1 (3)
C31	P	C41	110·7 (1)	C33	C34	C35	121·0 (3)
P	C11	C12	112·1 (2)	C34	C35	C36	120·1 (3)
C11	C12	C13	112·8 (2)	C31	C36	C35	119·1 (3)
C12	C13	Br14	111·2 (2)	P	C41	C42	121·2 (2)
P	C21	C22	118·0 (2)	P	C41	C46	118·4 (2)
P	C21	C26	122·3 (2)	C42	C41	C46	120·3 (3)
C22	C21	C26	119·7 (2)	C41	C42	C43	119·3 (3)
C21	C22	C23	119·5 (3)	C42	C43	C44	120·3 (3)
C22	C23	C24	120·7 (3)	C43	C44	C45	120·3 (3)
C23	C24	C25	120·3 (3)	C44	C45	C46	120·4 (3)
C24	C25	C26	120·2 (3)	C41	C46	C45	119·3 (3)
C21	C26	C25	119·6 (3)				

C21	P	C11	C12	−161·2 (2)	P	C11	C12	C13	−171·3 (2)
C31	P	C11	C12	−40·9 (3)	C11	C12	C13	Br14	72·1 (3)
C41	P	C11	C12	80·3 (2)					

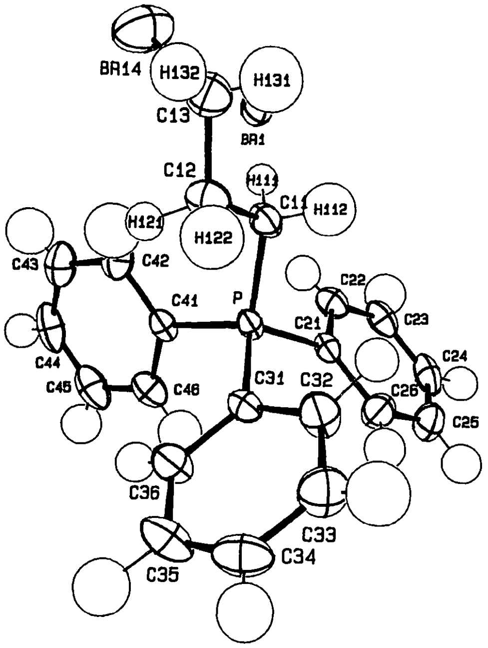

Fig. 1. A perspective view of the molecule showing the atom-labeling scheme. Only the cyanopropyl H atoms are labeled. Thermal ellipsoids are drawn at the 50% probability level.

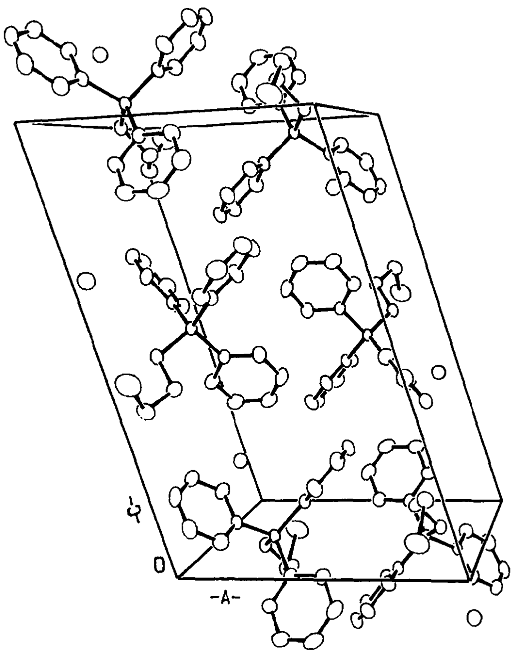

Fig. 2. The packing of the molecules in the unit cell. Perspective view is along the *b* axis following 10° rotations about and perpendicular to the *a* axis for clarity.

correspond to normal van der Waals interactions. Bond lengths and angles of the non-H atoms and selected torsion angles are given in Table 2. The phenyl-ring numbers and atom numbers are assigned as previously described (Czerwinski, 1986).

Related literature. This is the fourth crystal-structure determination in a series of seventeen triphenylphosphonium compounds which exhibit varying effects on the cholinergic nervous system of *Schistosoma mansoni* (McAllister *et al.*, 1980). We have previously reported the crystal structures of 2-aminoethyl- (Czerwinski, 1986), benzyl- (Ponnuswamy & Czerwinski, 1986) and 3-cyanopropyl- (Czerwinski & Ponnuswamy, 1988) triphenylphosphonium bromide. The 3-bromopropyl structure has no unusual features and is nearly identical to the 3-cyanopropyl structure. The crystals of these two compounds are isomorphous within 1% of the unit-cell parameters.

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Structure of *N*-[Di(2-pyridyl)methylene]aniline

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Abstract. $C_{17}H_{13}N_3$, $M_r = 259{\cdot}31$, triclinic, $P\bar{1}$, $a = 8{\cdot}217\,(4)$, $b = 8{\cdot}809\,(4)$, $c = 9{\cdot}847\,(4)$ Å, $\alpha = 81{\cdot}48\,(4)$, $\beta = 78{\cdot}86\,(4)$, $\gamma = 83{\cdot}71\,(4)°$, $V = 689\,(1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}250$ Mg m^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}07$ mm^{-1}, $F(000) = 272$, $T = 291\,(1)$ K, final $R = 0{\cdot}070$ for 1879 unique observed $[F \geq 3{\cdot}0\sigma(F)]$ diffractometer data. The crystal structure of a condensation product of di-2-pyridyl ketone with aniline was determined by the X-ray diffraction method. There are no unusual bond distances and angles in the molecule and no short contacts between the molecules.

Experimental. The title compound was prepared by reaction of 1 g of di-2-pyridyl ketone and 1·2 ml aniline in 90 ml boiling toluene with 3 mg *p*-toluenesulfonic acid as a catalyst (yield: 83%). In order to keep the reaction water free a watertrap was used. Yellow-orange needle-shaped crystals (m.p. 314–315 K) were obtained by multifold recrystallization from ethanol. The IR spectrum shows a C=N band at 1635 cm^{-1}. Crystal size ~ 0·38 × 0·26 × 0·38 mm; $\omega/2\theta$ scan, scan speed 1·5–14·6° min^{-1} in θ; Nicolet *R*3*m*/V diffractometer, graphite-monochromated Mo *Kα*; lattice parameters from least-squares fit with 25 reflections up to $2\theta = 24{\cdot}5°$; six standard reflections recorded every 2·5 h, only random deviations; 4999 reflections measured, $1{\cdot}5 \leq \theta \leq 25{\cdot}0°$, $-10 \leq h \leq 10$, $-11 \leq k \leq 11$, $-12 \leq l \leq 12$; after averaging ($R_{\text{int}} = 0{\cdot}024$): 2446 unique reflections, 1879 with $F \geq 3{\cdot}0\sigma(F)$; Lorentz–polarization correction, no absorption correction; structure solution in space group $P\bar{1}$ *via* direct methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H atoms, which were placed in geometrically calculated positions (C—H 0·96 Å), the phenyl group was refined as a rigid body (C—C 1·935 Å, C—C—C and C—C—H 120°); refinement on *F* with 1879 reflections and 170 refined parameters; $w = 1{\cdot}0/[\sigma^2(F) + (0{\cdot}0005F^2)]$; $S = 1{\cdot}94$, $R = 0{\cdot}070$, $wR = 0{\cdot}069$, $(\Delta/\sigma)_{\max} = 0{\cdot}04$, no extinction correction; largest peak in final ΔF map $\pm 0{\cdot}2\,(1)$ e Å^{-3},

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2 × 10^3)

$$U_{\text{eq}} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	*y*	*z*	U_{eq}
N(1)	0·3540 (3)	0·4905 (3)	0·8320 (2)	54
N(2)	0·1826 (3)	0·1337 (2)	0·9727 (2)	52
N(3)	0·1550 (4)	0·3230 (3)	0·6545 (3)	86
C(1)	0·2557 (3)	0·3828 (3)	0·8551 (3)	41
C(2)	0·2816 (3)	0·2487 (3)	0·9634 (3)	42
C(3)	0·3977 (4)	0·2441 (3)	1·0490 (3)	55
C(4)	0·4130 (4)	0·1174 (4)	1·1477 (3)	63
C(5)	0·3127 (4)	−0·0012 (3)	1·1581 (3)	58
C(6)	0·2003 (4)	0·0118 (3)	1·0689 (3)	56
C(7)	0·1138 (3)	0·3789 (3)	0·7800 (3)	44
C(8)	−0·0415 (3)	0·4268 (3)	0·8416 (4)	61
C(9)	−0·1670 (5)	0·4177 (5)	0·7683 (7)	115
C(10)	−0·1347 (9)	0·3609 (6)	0·6435 (9)	163
C(11)	0·0208 (9)	0·3157 (5)	0·5918 (5)	135
C(13)	0·1952 (2)	0·7358 (2)	0·7716 (2)	58
C(14)	0·1790 (2)	0·8736 (2)	0·6832 (2)	64
C(15)	0·2947 (2)	0·9019 (2)	0·5604 (2)	60
C(16)	0·4267 (2)	0·7924 (2)	0·5261 (2)	57
C(17)	0·4429 (2)	0·6546 (2)	0·6146 (2)	52
C(12)	0·3271 (2)	0·6263 (2)	0·7374 (2)	48

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