Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Symmetry codes are defined in the figure caption.

P(1)—C(1)	1·805 (4)	C(4)—C(5)	1·384 (7)
C(1)—C(2)	1·390 (6)	C(5)—C(6)	1·371 (7)
C(1)—C(6)	1·393 (6)	P(2)—F(1)	1·555 (5)
C(2)—C(3)	1·383 (7)	P(2)—F(2)	1·556 (3)
C(3)—C(4)	1·371 (8)		
P(1)—C(1)—C(2)	122·0 (3)	C(3)—C(4)—C(5)	120·5 (4)
P(1)—C(1)—C(6)	118·1 (3)	C(4)—C(5)—C(6)	119·7 (5)
C(2)—C(1)—C(6)	120·0 (4)	C(1)—C(6)—C(5)	120·2 (4)
C(1)—P(1)—C(1ⁱ)	107·2 (3)	F(1)—P(2)—F(2)	90·0 (2)
C(1)—P(1)—C(1ⁱⁱ)	110·6 (1)	F(1)—P(2)—F(1ⁱⁱ)	180·0
C(1)—P(1)—C(1ⁱⁱⁱ)	110·6 (1)	F(2)—P(2)—F(2ⁱ)	180·0 (1)
C(1)—C(2)—C(3)	119·1 (5)	F(2)—P(2)—F(2ⁱⁱ)	90·0 (1)
C(2)—C(3)—C(4)	120·6 (4)	F(2)—P(2)—F(2ⁱⁱⁱ)	90·0 (1)

(Motherwell & Clegg, 1978). Atomic parameters are in Table 1,* bond lengths and angles in Table 2. The atomic numbering is shown in Fig. 1.

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* Lists of structure amplitudes, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51019 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The title compound consists of two much used counterions. A search of the Cambridge Structural Database (Allen *et al.*, 1979) during February 1988 shows that the PPh₄⁺ cation and the PF₆⁻ anion have been determined in 252 and 580 different crystal structures, respectively, with mean P—C bond length 1·798 Å, and mean P—F 1·551 Å.

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Structure of 9,10-Dihydro-10,9-propenoanthracene

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Abstract. $C_{17}H_{14}$, $M_r = 218·30$, orthorhombic, *Iba*2, $a = 22·796$ (5), $b = 14·145$ (4), $c = 7·477$ (2) Å, $V = 2411$ (1) Å³, $Z = 8$, $D_x = 1·203$ g cm⁻³, Mo $K\alpha$ ($\lambda = 0·71073$ Å), $\mu = 0·63$ cm⁻¹, $F(000) = 928$, $T = 298$ K, $R = 0·0352$ for 1128 reflections $[F_o \geq 6\sigma(|F_o|)]$. The molecule consists of a dihydro-anthracene unit bridged by a propeno unit between C(9) and C(10). The dihedral angle between the two phenyl rings is 134·6° and they form almost equal dihedral angles, 67·9 and 67·1°, with the plane of atoms C(9),C(10),C(11),C(12) and C(13). Angles C(10)—C(11)—C(12), C(11)—C(12)—C(13) and C(9)—C(13)—C(12) are larger than the expected sp^2 and sp^3 angles of 120 and 109·5°, indicating that the molecule in this region is under angular strain.

Experimental. This compound (1) was isolated as the sole product (m.p. 441–443 K; 60%) of the attempted amination of 11-bromo-9,10-dihydro-9,10-propano-anthracene (2) with 3-*N*,*N*-dimethylaminopropyl-amine, potassium iodide and potassium *tert*-butoxide in refluxing dioxane. Treatment of 10,11-dihydro-5,10-ethano-5*H*-dibenzo[*a,d*]cycloheptane (3) with *N*-bromosuccinimide and benzoyl peroxide in refluxing carbon tetrachloride for one hour in the presence of a strong light afforded (2) (b.p. 391 K, 6·7 Pa; 65%; structure consistent with ¹³C NMR and ¹H NMR) instead of the expected 11-bromo-10,11-dihydro-5,10-ethano-5*H*-dibenzo[*a,d*]cycloheptane. A modification of the cyclidehydration of *trans*-4-benzyl-1-tetralol by Low & Roberts (1973) was used to prepare (3).
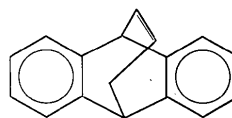
(1)

Table 1. *Atomic coordinates* (× 10⁴) *and equivalent isotropic displacement parameters* ($Å^2 × 10^3$)

	x	y	z	U_{eq}
C(1)	1044 (1)	3938 (2)	7168 (7)	66 (1)
C(2)	691 (2)	4269 (3)	5841 (7)	80 (1)
C(3)	452 (2)	3663 (3)	4627 (7)	84 (1)
C(4)	569 (1)	2696 (3)	4734 (5)	68 (1)
C(4A)	930 (1)	2355 (2)	6071 (5)	52 (1)
C(5)	2088 (1)	545 (2)	5979 (6)	63 (1)
C(6)	2672 (1)	492 (3)	6482 (6)	70 (1)
C(7)	2898 (2)	1114 (3)	7688 (6)	68 (1)
C(8)	2549 (1)	1799 (2)	8439 (5)	63 (1)
C(8A)	1960 (1)	1862 (2)	7980	50 (1)
C(9)	1544 (1)	2573 (2)	8766 (5)	56 (1)
C(9A)	1166 (1)	2985 (2)	7304 (5)	51 (1)
C(10)	1081 (1)	1323 (2)	6356 (6)	56 (1)
C(10A)	1732 (1)	1223 (2)	6745 (5)	51 (1)
C(11)	738 (1)	953 (2)	7964 (7)	71 (1)
C(12)	788 (2)	1317 (3)	9621 (7)	77 (1)
C(13)	1152 (2)	2102 (3)	10172 (6)	75 (1)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)–C(2)	1·361 (5)	C(1)–C(9A)	1·380 (4)
C(2)–C(3)	1·362 (6)	C(3)–C(4)	1·395 (6)
C(4)–C(4A)	1·383 (4)	C(4A)–C(9A)	1·391 (4)
C(4A)–C(10)	1·514 (4)	C(5)–C(6)	1·387 (5)
C(5)–C(10A)	1·380 (4)	C(6)–C(7)	1·362 (5)
C(7)–C(8)	1·375 (5)	C(8)–C(8A)	1·388 (4)
C(8A)–C(9)	1·503 (4)	C(8A)–C(10A)	1·392 (4)
C(9)–C(9A)	1·508 (4)	C(9)–C(13)	1·533 (5)
C(10)–C(10A)	1·520 (4)	C(10)–C(11)	1·527 (5)
C(11)–C(12)	1·346 (6)	C(12)–C(13)	1·447 (6)
C(9A)–C(1)–C(2)	120·7 (4)	C(3)–C(2)–C(1)	120·3 (3)
C(4)–C(3)–C(2)	120·2 (3)	C(4A)–C(4)–C(3)	119·8 (4)
C(9A)–C(4A)–C(4)	119·1 (3)	C(10)–C(4A)–C(4)	125·0 (3)
C(10)–C(4A)–C(9A)	115·9 (2)	C(10A)–C(5)–C(6)	119·3 (3)
C(7)–C(6)–C(5)	120·5 (3)	C(8)–C(7)–C(6)	120·5 (3)
C(8A)–C(8)–C(7)	120·3 (3)	C(9)–C(8A)–C(8)	123·8 (3)
C(10A)–C(8A)–C(8)	118·9 (3)	C(10A)–C(8A)–C(9)	117·3 (2)
C(9A)–C(9)–C(8A)	109·6 (2)	C(13)–C(9)–C(8A)	110·2 (3)
C(13)–C(9)–C(9A)	109·4 (2)	C(4A)–C(9A)–C(1)	119·9 (3)
C(9)–C(9A)–C(1)	123·1 (3)	C(9)–C(9A)–C(4A)	117·0 (2)
C(10A)–C(10)–C(4A)	109·7 (2)	C(11)–C(10)–C(4A)	109·0 (2)
C(11)–C(10)–C(10A)	108·5 (3)	C(8A)–C(10A)–C(5)	120·5 (2)
C(10)–C(10A)–C(5)	124·0 (3)	C(10)–C(10A)–C(8A)	115·5 (2)
C(12)–C(11)–C(10)	123·3 (3)	C(13)–C(12)–C(11)	127·2 (3)
C(12)–C(13)–C(9)	118·2 (3)		

Fig. 1. View of the title compound showing the atom-labelling scheme (50% thermal ellipsoids).

Crystals of the title compound were grown by sublimation in vacuum and a colorless plate of dimensions 0·18 × 0·24 × 0·45 mm was chosen for X-ray diffraction. Data were collected on a Nicolet *R*3/*V* diffractometer equipped with a graphite monochromator utilizing Mo *K*α radiation ($\lambda = 0·71073$ Å). 30 reflections with $17·01 \le 2\theta \le 29·81°$ were used to refine the cell parameters. 2975 reflections (*h*, 0→29; *k*, 0→18; *l*, −9→9) were collected using the ω-scan method, 1509 unique reflections, $R_{int} = 0·0198$; 2θ range 0→55°, 1° ω-scan at 3–6° min⁻¹, depending upon intensity. Four reflections (121, 213, 222, 231) were measured every 96 reflections to monitor instrument and crystal stability. Absorption corrections were not applied because of the small size of the crystal and the value of the absorption coefficient ($\mu = 0·63$ cm⁻¹).

The structure was solved by direct methods (*SHELXTL PLUS;* Nicolet XRD Corporation, 1987) from which the locations of all C atoms were obtained. The structure was refined using full-matrix least squares and the positions of all H atoms were determined from the difference Fourier map. C atoms were treated anisotropically, whereas H atoms were refined with isotropic thermal parameters. 209 parameters were refined and $\sum w(|F_o| - |F_c|)^2$ was minimized; $w = 1/\sigma(|F_o|)^2$, $\sigma(F_o) = 0·5kI^{-1/2}\{[\sigma(I)]^2 + (0·005I)^2\}^{1/2}$, *I*(intensity) = ($I_{peak} - I_{background}$)(scan rate), and $\sigma(I) = (I_{peak} + I_{background})^{1/2}$(scan rate), *k* is the correction due to decay and Lp effects, 0·005 is a factor used to downweight intense reflections and to account for instrument instability. Final $R = 0·0352$, $wR = 0·0443$ for 1128 reflections having $F_o \ge 6\sigma(F_o)$, and goodness of fit = 1·224. Maximum $\Delta/\sigma < 0·1$ in the final refinement cycle and the minimum and maximum peaks in the ΔF map were −0·15 and 0·10 e Å⁻³ respectively. Scattering factors were obtained from Cromer & Waber (1974). Positional parameters and the equiv-

alent thermal parameters of the C atoms are listed in Table 1, and the bond lengths and angles of non-hydrogen atoms in Table 2.* The atom-labelling scheme is shown in Fig 1.

Related literature. Rearrangements between 10,11-dihydro-5,10-ethano-5*H*-dibenzo[a,d]cycloheptenes and 9,10-dihydro-9,10-propanoanthracenes have been studied extensively (Cioranescu, Banciu, Jelescu, Rentzea, Elian & Nenitzescu, 1969a,b; Cristol & Noreen, 1976; Cristol, Sequeira & Mayo, 1968; Cristol, Stull & Daussin, 1978; Stanescu, Mihal, Stanescu & Cioranescu, 1979). An efficient func-

tionalization of the parent hydrocarbon, avoiding rearrangement between the two systesm, leads to analogs of tetracyclic amine antidepressants which may be of interest (El-Zohry, Li, Tyllick & Roberts, 1988).

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Structure of a Diamino-s-tetrazine

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Abstract. 3,6-Bis(β-fluoro-β,β-dinitroethylamino)-1,2,4,5-tetrazine, $C_6H_6F_2N_{10}O_8$, $M_r = 384.17$, monoclinic, $P2_1/n$, $a = 6.569$ (2), $b = 5.821$ (2), $c = 18.153$ (6) Å, $\beta = 91.75$ (3)°, $V = 694.0$ (4) Å^3, $Z = 2$, $D_x = 1.839$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71079 Å, $\mu = 0.17$ mm^{-1}, $F(000) = 388$, $T = 225$ K, final $R = 0.051$, 1181 unique reflections. This is the first reported structural study of a diaminotetrazine. The center of the heterocyclic ring lies on a center of symmetry. Crowding in the vicinity of the nitro groups is evidenced by the eclipsed geometry [F—C(6)—N(8)—O(8b) = 3.4 (4), F—C(6)—N(7)—O(7b) = −30.4 (4)°] and close intramolecular approaches of the F atom [F···O(7b) at 2.537 (5) and F···O(8b) at 2.494 (5) Å]. Packing is influenced by the presence of an N(4)···N(2) intermolecular hydrogen bond [N—H = 0.88 (4), H···N = 2.07 (4), N···N = 2.94 (2) Å, and N—H···N = 169.6 (9)°].

Experimental. Translucent orange data crystal, 0.34 × 0.23 × 0.44 mm, synthesized by Robert Schmitt of SRI International, Palo Alto, California. Automated Nicolet R3m diffractometer with incident-beam graphite monochromator; 25 centered reflections within $28 \leq 2\theta \leq 35°$ for determining cell parameters. Data corrected for Lorentz and polarization effects, but not for absorption. $2\theta_{max} = 50°$; range of hkl: $-7 \leq h \leq 7$,

$0 \leq k \leq 6$, $0 \leq l \leq 14$; standards, $\overline{4}00$, 040, 006, monitored every 100 reflections with random variation of 3.0% over data collection, $\theta/2\theta$ mode, scan width $[2\theta(K\alpha_1) - 1.0]$ to $[2\theta(K\alpha_2) + 1.0]°$, scan rate a function of count rate (6° min^{-1} minimum, 30° min^{-1} maximum); 1515 reflections measured, 1181 unique ($R_{int} = 0.015$), 1080 observed $[F_o > 3\sigma(F_o)]$. Structure solved by direct methods. Full-matrix least-squares refinement on F with MicroVax version of the SHELXTL system (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0.00025$. 130 parameters refined: atom coordinates for all atoms, anisotropic temperature factors for non-H atoms and isotropic temperature factors for H atoms; $(\Delta/\sigma)_{max} = 0.01$, $R = 0.051$, $wR = 0.089$, $S = 3.95$. Final difference Fourier excursions 0.26 and -0.29 e Å^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for Tables 1 and 2, atom coordinates, bond distances and bond angles, follows that in Fig. 1.