Tableau 3. *Distances caractéristiques* (Å) *des sites actifs*

	Présent travail	Pullman *et al.*	Gadret *et al.*
Azote N(15)–oxygène O(13)	2,836 (5)	2,8–2,9	2,89
Azote N(15)–centre du noyau benzénique	5,040 (7)	5,1–5,2	5,15
Oxygène O(13)–centre du noyau benzénique	3,740 (7)	3,6–3,7	3,68

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Structure of 9,10-Bis(*p*-nitrobenzeneazo)-*cis*-decalin* (PNPAD), $C_{22}H_{24}N_6O_4$

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Abstract. $M_r = 436{\cdot}47$, monoclinic, $P2_1/c$, $a = 7{\cdot}520\,(2)$, $b = 20{\cdot}180\,(4)$, $c = 14{\cdot}823\,(3)$ Å, $\beta = 105{\cdot}02\,(1)^\circ$, $V = 2172{\cdot}592$ Å^3, $Z = 4$, $D_m = 1{\cdot}301$, $D_x = 1{\cdot}334$ Mg m^{-3}, m.p. = 435–438 K, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}0998$ mm^{-1}, $F(000) = 920$, $T = 298$ K, final $R = 0{\cdot}061$ for 707 independent non-zero reflections. The molecule is made up of two almost planar *p*-nitrobenzeneazo ligands attached to the two central C atoms of the *cis*-Decalin®. The interatomic distances are normal, the two N=N double bonds both being 1·22 (2) Å; C—C bond lengths in the Decalin® ring system average 1·54 Å.

Introduction. The crystal structure analysis of the title compound (PNPAD) was carried out within the framework of a detailed structure investigation of a family of 9,10-bis-arylazodecalins recently prepared at the Organic Chemistry Laboratory of the University of Thessaloniki (Malamidou-Xenikaki & Alexandrou, 1982).

The first structure of the series, that of 9,10-bis(*p*-chlorobenzeneazo)-*cis*-decalin (PCPAD), shows interesting stereochemical features (Kavounis & Rentzeperis, 1983). The structure determination of PNPAD was undertaken in order to study the changes induced by the substitution of the Cl atoms in PCPAD by NO_2 groups.

Experimental. Pure red crystals, 0·20 × 0·13 × 0·10 mm. D_m measured by flotation in KBr solution, computer-controlled Philips PW 1100 four-circle single-crystal diffractometer, three-dimensional data, graphite-monochromated Mo $K\alpha$, $\theta/2\theta$ scan mode; cell parameters and standard deviations by least-squares analysis with *LATCON* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) of the θ angles of 57 strong reflections; intensity statistics indicated $P2_1/c$; 2875 measured reflections, $\theta = 3$–24°, index range h: 0→8, k: 0→22, l: −16→16, 707 considered observed $[I > 2\sigma(I)]$. Three standard reflections monitored after every 97 reflections (2 h) showed < 1·7% intensity fluctuation throughout data collection. Correction for decrease in intensity, for Lorentz and polarization factors with *DATRED* (Main, 1970), no absorption correction; structure solved by direct methods with *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977); full-matrix least squares using F magnitudes, *XRAY76* (Stewart *et al.*, 1976), H atoms kept fixed at calculated positions with isotropic temperature factors as those of bonded C atoms, 6 C atoms (see Table 1) treated isotropically, other non-H atoms anisotropically, $w = 1/\{1 + [(F-B)/A]^2\}$ with $A = 30$, $B = 70$; $R = 0{\cdot}061$, $R_w = 0{\cdot}050$, $S = 1{\cdot}77$, $(\Delta/\sigma)_{\max} = 1{\cdot}795$ [for C(17)] and $(\Delta/\sigma)_{\rm mean} = 0{\cdot}242$, residual electron density in final difference map $-1{\cdot}2$–$0{\cdot}8$ e Å^{-3}; scattering factors from *International Tables for X-ray Crystallography* (1974).

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* Bis(*p*-nitrobenzeneazo)-4a,8a-decahydronaphthalene.

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Table 1. *Fractional atomic coordinates and isotropic temperature factors* (Å^2) *for the non*-H *atoms in PNPAD*

Atoms marked with an asterisk were refined isotropically, the others were refined anisotropically. For these, $B_{eq} = \frac{8}{3}\pi^2 \mathrm{trace}\, \tilde{\mathbf{U}}$.

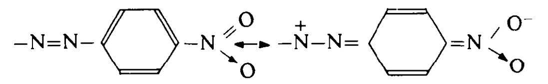
	x	y	z	B_{eq}/B_{iso}
N(1)	0·2167 (16)	0·1758 (6)	0·1749 (8)	3·6
N(2)	0·1384 (14)	0·1875 (6)	0·0937 (8)	3·4
N(3)	−0·3710 (16)	−0·0066 (7)	−0·0916 (10)	3·6
N(4)	0·3155 (16)	0·1733 (6)	0·3659 (7)	3·0
N(5)	0·3817 (16)	0·1313 (6)	0·4245 (8)	4·0
N(6)	−0·0449 (25)	0·0402 (10)	0·6637 (11)	6·9
O(1)	−0·4447 (16)	0·0066 (6)	−0·1745 (8)	6·1
O(2)	−0·4004 (16)	−0·0526 (6)	−0·0480 (9)	6·9
O(3)	0·0299 (22)	−0·0019 (8)	0·7195 (9)	10·5
O(4)	−0·1894 (22)	0·0645 (10)	0·6548 (10)	11·4
C(1)	0·5867 (20)	0·1498 (7)	0·3023 (9)	3·9
C(2)	0·7190 (20)	0·1794 (9)	0·2467 (10)	5·1
C(3)	0·6054 (20)	0·2047 (8)	0·1529 (10)	4·7
C(4)	0·4675 (17)	0·2560 (7)	0·1638 (8)	3·2
C(5)	0·2012 (19)	0·2852 (6)	0·2415 (9)	2·8
C(6)	0·3036 (22)	0·3400 (7)	0·3055 (11)	4·5
C(7)	0·4114 (20)	0·3115 (8)	0·3984 (10)	4·1
C(8)	0·5478 (20)	0·2572 (7)	0·3853 (9)	3·5
C(9)*	0·4455 (18)	0·2025 (6)	0·3177 (9)	1·0 (3)
C(10)*	0·3383 (20)	0·2320 (7)	0·2242 (9)	1·1 (3)
C(11)*	0·0156 (18)	0·1331 (7)	0·0519 (10)	1·0 (3)
C(12)	−0·0479 (19)	0·1407 (6)	−0·0469 (10)	3·8
C(13)*	−0·1757 (18)	0·0927 (7)	−0·0933 (9)	1·0 (3)
C(14)*	−0·2336 (18)	0·0440 (7)	−0·0436 (10)	1·2 (4)
C(15)	−0·1668 (19)	0·0370 (7)	0·0516 (10)	3·6
C(16)	−0·0405 (18)	0·0826 (7)	0·0959 (8)	2·6
C(17)	0·2634 (18)	0·1055 (7)	0·4784 (9)	3·7
C(18)*	0·3415 (16)	0·0633 (6)	0·5496 (8)	0·9 (3)
C(19)	0·2356 (22)	0·0406 (7)	0·6077 (9)	4·2
C(20)	0·0592 (20)	0·0605 (8)	0·5946 (9)	3·9
C(21)	−0·0254 (21)	0·1034 (8)	0·5235 (11)	5·2
C(22)	0·0906 (19)	0·1258 (6)	0·4678 (9)	3·2

Table 2. *Interatomic distances* (Å) *and angles* (°)

N(1)–N(2)	1·22 (2)	C(5)–C(6)	1·53 (2)
N(1)–C(10)	1·52 (2)	C(6)–C(7)	1·52 (2)
N(2)–C(11)	1·47 (2)	C(7)–C(8)	1·55 (2)
N(3)–O(1)	1·24 (2)	C(8)–C(9)	1·55 (2)
N(3)–O(2)	1·18 (2)	C(9)–C(10)	1·53 (2)
N(3)–C(14)	1·50 (2)	C(11)–C(12)	1·43 (2)
N(4)–N(5)	1·22 (2)	C(11)–C(16)	1·34 (2)
N(4)–C(9)	1·47 (2)	C(12)–C(13)	1·41 (2)
N(5)–C(17)	1·44 (2)	C(13)–C(14)	1·36 (2)
N(6)–O(3)	1·22 (2)	C(14)–C(15)	1·38 (2)
N(6)–O(4)	1·17 (3)	C(15)–C(16)	1·36 (2)
N(6)–C(20)	1·50 (3)	C(17)–C(18)	1·37 (2)
C(1)–C(2)	1·57 (2)	C(17)–C(22)	1·33 (2)
C(1)–C(9)	1·56 (2)	C(18)–C(19)	1·39 (2)
C(2)–C(3)	1·52 (2)	C(19)–C(20)	1·35 (2)
C(3)–C(4)	1·50 (2)	C(20)–C(21)	1·38 (2)
C(4)–C(10)	1·56 (2)	C(21)–C(22)	1·42 (2)
C(5)–C(10)	1·56 (2)		

C(10)–N(1)–N(2)	114·0 (1·2)	C(4)–C(10)–C(5)	114·7 (1·1)
N(1)–N(2)–C(11)	111·2 (1·1)	C(4)–C(10)–C(9)	112·3 (1·1)
O(1)–N(3)–O(2)	127·8 (1·3)	C(4)–C(10)–N(1)	110·1 (1·1)
O(1)–N(3)–C(14)	113·6 (1·3)	C(5)–C(10)–N(1)	104·6 (1·1)
O(2)–N(3)–C(14)	118·6 (1·2)	C(5)–C(10)–C(9)	109·5 (1·1)
N(5)–N(4)–C(9)	114·9 (1·1)	N(2)–C(11)–C(12)	111·1 (1·2)
N(4)–N(5)–C(17)	116·6 (1·2)	N(2)–C(11)–C(16)	127·6 (1·2)
O(3)–N(6)–O(4)	128·5 (2·1)	C(16)–C(11)–C(12)	121·3 (1·2)
O(3)–N(6)–C(20)	115·1 (1·7)	C(11)–C(12)–C(13)	115·5 (1·3)
O(4)–N(6)–C(20)	116·3 (1·7)	C(12)–C(13)–C(14)	120·2 (1·2)
C(9)–C(1)–C(2)	111·5 (1·2)	N(3)–C(14)–C(15)	116·2 (1·3)
C(1)–C(2)–C(3)	109·1 (1·2)	N(3)–C(14)–C(13)	120·9 (1·2)
C(2)–C(3)–C(4)	111·9 (1·2)	C(13)–C(14)–C(15)	122·9 (1·3)
C(3)–C(4)–C(10)	113·5 (1·2)	C(14)–C(15)–C(16)	116·6 (1·4)
C(6)–C(5)–C(10)	110·7 (1·1)	C(15)–C(16)–C(11)	123·3 (1·2)
C(5)–C(6)–C(7)	110·7 (1·2)	N(5)–C(17)–C(18)	116·8 (1·2)
C(6)–C(7)–C(8)	111·7 (1·3)	N(5)–C(17)–C(22)	123·3 (1·2)
C(7)–C(8)–C(9)	110·5 (1·1)	C(18)–C(17)–C(22)	119·6 (1·4)
N(4)–C(9)–C(1)	111·1 (1·1)	C(17)–C(18)–C(19)	118·9 (1·2)
N(4)–C(9)–C(8)	104·5 (1·1)	C(18)–C(19)–C(20)	120·7 (1·2)
N(4)–C(9)–C(10)	109·5 (1·0)	N(6)–C(20)–C(19)	119·1 (1·3)
C(1)–C(9)–C(8)	109·6 (1·0)	N(6)–C(20)–C(21)	118·3 (1·5)
C(1)–C(9)–C(10)	110·5 (1·1)	C(19)–C(20)–C(21)	122·4 (1·5)
C(8)–C(9)–C(10)	111·4 (1·1)	C(20)–C(21)–C(22)	114·3 (1·4)
N(1)–C(10)–C(9)	104·8 (1·1)	C(17)–C(22)–C(21)	124·1 (1·2)

Discussion. Atomic parameters are given in Table 1.* Interatomic distances and angles are given in Table 2. A clinographic projection of the molecule with atomic numbering is shown in Fig. 1. As in the case of PCPAD (Kavounis & Rentzeperis, 1983), the main characteristic of the molecule is the central Decalin® system with the two cyclohexane rings in chair conformations. The C—C distances in these rings are normal with a mean of 1·54 Å. The least-squares planes through the cyclohexane chairs form an angle of 114·7 (1)°. The *p*-nitrobenzeneazo ligands bonded to the two central atoms of the Decalin® ring [C(9) and C(10)] are in *cis* relationship; the geometries about the azo groups are, as in PCPAD, antiperiplanar and the ligands are planar to a good approximation. The two N=N bond lengths are 1·22 Å, the same as the corresponding bond in *anti*-azobenzene 2-thiocyanate (Kakati & Chaudhuri, 1968) and do not deviate significantly from the values 1·23 and 1·24 Å found in PCPAD. The difference in bond length between the N atoms of the azo groups linked to the Decalin® ring (1·47, 1·52 Å) and those linked to the benzene rings (1·44, 1·47 Å) may indicate partial conjugation between the azo and *p*-nitrophenyl groups, of the type:



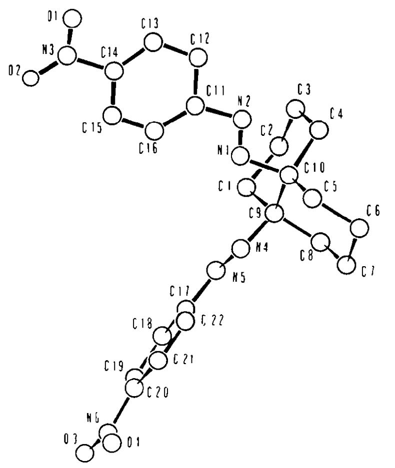


Fig. 1. Clinographic projection of the molecule.

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* Lists of structure factors, anisotropic thermal parameters for non-H atoms, coordinates and isotropic temperature factors for H atoms and least-squares-plane calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39389 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

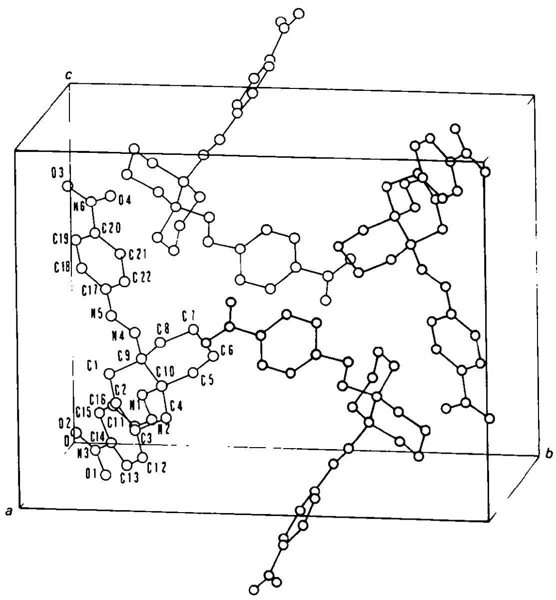

Fig. 2. Clinographic projection of the unit cell showing the molecular packing.

The geometrical features of the benzene rings are normal with average C–C distances 1·35 Å and 1·37 Å and with average bond angles 120°. The two rings form an angle of 66·3 (1)° with each other.

The N–O distances in the NO_2 groups, 1·17, 1·18, 1·22 and 1·24 Å, are in good agreement with the values 1·14–1·21 (4) Å given in *International Tables for X-ray Crystallography* (1968) and the values 1·19 (2) –1·25 (2) Å in dimethyl 3-(*p*-nitrophenyl)-4-oxo-6,6a-diphenyl-3a,4-dihydro-6a*H*-cyclopenta[2,3-*d*]-isoxazole-3a,5-dicarboxylate (Stergiou, Kokkou & Rentzeperis, 1978).

A clinographic projection of the structure, showing the molecular packing in the unit cell, is given in Fig. 2.

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(±)-Norphenylephrine Hydrochloride (*m*-Octopamine Hydrochloride), $C_8H_{12}NO_2^+.Cl^-$*

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Abstract. $M_r = 189·5$, monoclinic, $P2_1/n$, $a = 11·071$ (2), $b = 5·879$ (2), $c = 14·310$ (3) Å, $\beta = 104·00$ (2)°, $V = 903·7$ Å^3, $Z = 4$, $D_m = 1·42$, $D_x = 1·39$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·70926$ Å, $\mu = 4·0$ cm^{-1}, $F(000) = 400$, $T = 293$ K, $R = 0·032$ for 1293 observed reflections. The aminoethanol side chain is in the unusual *gauche* conformation with CC–CN and

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* Norphenylephrine is 2-amino-1-(3-hydroxyphenyl)ethanol.

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