Table 1. *Experimental details*

Crystal dimensions	0·35 × 0·42 × 0·58 mm
Range of *h,k,l*	0→16, 0→8, −18→18
Standard reflections	22$\bar{2}$, 328
Reflections: all	2676
observed ($\|F\| > 6\sigma\|F\|$)	2334
Parameters refined	109
R, *wR* (all)	0·041, 0·058
R, *wR* (observed)	0·036, 0·055
S	1·830
$(\Delta/\sigma)_{max}$ in final cycle	0·1
$(\Delta\rho)_{min}/(\Delta\rho)_{max}$	−0·3/0·3 e Å^{-3}

Table 2. *Final atomic parameters* ($\times 10^4$) *with e.s.d.'s in parentheses*

$B_{eq} = \frac{1}{3}[B_{22} + 1/\sin^2\beta(B_{11} + B_{33} + 2B_{13}\cos\beta)]$.

	x	*y*	*z*	B_{eq} (Å^2)
P	−875·0 (3)	560·0 (7)	4961·0 (3)	2·80 (2)
Si(1)	498·0 (4)	1453·0 (8)	6816·0 (3)	3·40 (2)
Si(2)	−2909·0 (4)	−766·0 (9)	5441·0 (4)	3·90 (2)
S	−1503·0 (4)	3093·0 (8)	4578·0 (4)	4·20 (2)
N(1)	−1664 (1)	−1039 (2)	5273 (1)	3·60 (6)
N(2)	200 (1)	631 (2)	5706 (1)	3·20 (6)
C(1)	219 (3)	−581 (4)	7556 (2)	6·5 (1)
C(2)	1844 (2)	2023 (4)	7021 (2)	5·0 (1)
C(3)	−258 (2)	3727 (4)	6934 (2)	5·7 (1)
C(4)	−3745 (2)	132 (6)	4435 (2)	6·8 (1)
C(5)	−2981 (2)	957 (4)	6377 (2)	5·9 (1)
C(6)	−3263 (2)	−3327 (5)	5742 (3)	7·4 (2)

Table 3. *Selected interatomic distances* (Å) *and bond angles* (°)

P—S	1·930 (1)	S—P—N(1)	112·1 (1)
P—N(1)	1·635 (1)	S—P—N(2)	117·8 (1)
P—N(2)	1·695 (1)	S—P—N(2^i)	118·8 (1)
P—N(2^i)	1·684 (1)	N(1)—P—N(2)	110·7 (1)
Si(1)—N(2)	1·766 (1)	N(1)—P—N(2^i)	109·0 (1)
Si(1)—C(1)	1·843 (3)	N(2)—P—N(2^i)	85·8 (1)
Si(1)—C(2)	1·841 (3)	P—N(2)—P^i	94·2 (1)
Si(1)—C(3)	1·850 (3)	P—N(2)—Si(1)	133·4 (1)
Si(2)—N(1)	1·764 (2)	P^i—N(2)—Si(1)	132·2 (1)
Si(2)—C(4)	1·850 (3)	P—N(1)—Si(2)	132·0 (1)
Si(2)—C(5)	1·850 (3)	⟨N(2)—Si(1)—C⟩	108 (1)
Si(2)—C(6)	1·845 (3)	⟨N(1)—Si(2)—C⟩	108 (4)

Symmetry code: (i) $-x, -y, 1-z$.

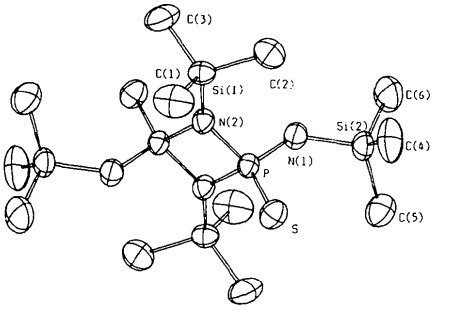

Fig. 1. A perspective view of the molecule.

diphosphazanes are reviewed by Hursthouse, Parkes, Shaw, Shaw & Watkins (1986) and Hursthouse, Ibrahim, Parkes, Shaw, Shaw & Watkins (1986).

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References

DOSTÁL, K., ŠIKOLA, J., MEISEL, M. & GRUNZE, H. (1986). *Z. Anorg. Allg. Chem.* **543**, 199–206.

HURSTHOUSE, M. B., IBRAHIM, E. H., PARKES, H. G., SHAW, L. S., SHAW, R. A. & WATKINS, D. A. (1986). *Phosphorous Sulfur*, **28**, 261–266.

HURSTHOUSE, M. B., PARKES, H. G., SHAW, L. S., SHAW, R. A. & WATKINS, D. A. (1986). *Phosphorus Sulfur*, **28**, 221–227.

SHAW, R. A. (1978). *Phosphorus Sulfur*, **4**, 101–121.

Syntex (1971). *XTL Structure Determination System.* Syntex Analytical Instruments, Cupertino, California, USA.

Acta Cryst. (1991). C**47**, 446–448

Structure of Ethyl 8-Dimethylamino-1-naphthalenecarboxylate–Picric Acid (1/1)

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Abstract. $C_{15}H_{18}NO_2^+.C_6H_2N_3O_7^-$, $M_r = 472{\cdot}41$, triclinic, $P\bar{1}$, $a = 7{\cdot}843$ (6), $b = 8{\cdot}839$ (2), $c = 15{\cdot}619$ (6) Å, $\alpha = 102{\cdot}93$ (2), $\beta = 92{\cdot}49$ (5), $\gamma = 96{\cdot}94$ (4)°, $V = 1045$ (2) Å^3, $Z = 2$, $D_x = 1{\cdot}502$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}0112$ mm^{-1}, $F(000) = 492$, $T = 293$ (1) K, $R = 0{\cdot}048$ for 2518 observed reflections with $I > 3\sigma(I)$. The naphthyl moiety is nearly planar [max. deviation 0·040 (3) Å]. The ethoxycarbonyl group is also planar and its carbonyl O atom is H bonded to the amino H

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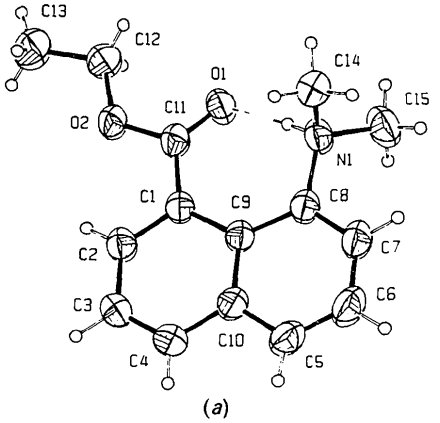
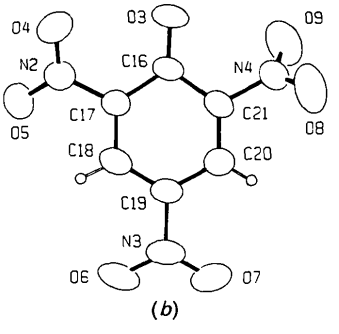
Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* ($Å^2$) *with e.s.d.'s in parentheses*

$$B_{eq} = (1/3)\sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O1	0·0967 (2)	0·1397 (2)	0·2189 (1)	4·49 (4)
O2	−0·1053 (2)	0·0038 (2)	0·1208 (1)	4·96 (4)
O3	0·2741 (2)	0·1893 (2)	0·6196 (1)	5·53 (5)
O4	0·3063 (3)	−0·0415 (2)	0·7036 (2)	7·36 (6)
O5	0·5256 (3)	−0·0017 (2)	0·7942 (1)	6·30 (5)
O6	0·6894 (2)	0·5036 (2)	0·9766 (1)	6·17 (5)
O7	0·6421 (3)	0·7081 (2)	0·9311 (1)	7·62 (6)
O8	0·1991 (3)	0·6082 (3)	0·6692 (2)	8·57 (6)
O9	0·3406 (4)	0·4864 (3)	0·5717 (1)	9·67 (8)
N1	0·1845 (3)	0·1101 (2)	0·3730 (1)	4·22 (5)
N2	0·4187 (3)	0·0466 (2)	0·7518 (1)	4·65 (5)
N3	0·6255 (3)	0·5663 (3)	0·9228 (1)	4·97 (6)
N4	0·2970 (3)	0·5132 (2)	0·6446 (1)	4·83 (5)
C1	0·0326 (3)	−0·1404 (3)	0·2042 (1)	3·28 (5)
C2	−0·0356 (3)	−0·2675 (3)	0·1380 (2)	3·92 (6)
C3	−0·0209 (3)	−0·4213 (3)	0·1406 (2)	4·27 (6)
C4	0·0593 (3)	−0·4508 (3)	0·2117 (2)	4·15 (6)
C5	0·2021 (3)	−0·3690 (3)	0·3578 (2)	4·53 (6)
C6	0·2682 (3)	−0·2593 (3)	0·4309 (2)	5·01 (6)
C7	0·2625 (3)	−0·1028 (3)	0·4316 (2)	4·62 (6)
C8	0·1916 (3)	−0·0583 (3)	0·3615 (2)	3·67 (5)
C9	0·1183 (3)	−0·1676 (3)	0·2828 (1)	3·28 (5)
C10	0·1273 (3)	−0·3275 (3)	0·2842 (2)	3·62 (5)
C11	0·0131 (3)	0·0140 (3)	0·1851 (1)	3·65 (5)
C12	−0·1287 (4)	0·1491 (3)	0·0947 (2)	5·43 (7)
C13	−0·2683 (4)	0·1118 (3)	0·0245 (2)	6·63 (8)
C14	0·0419 (4)	0·1612 (3)	0·4281 (2)	5·20 (7)
C15	0·3512 (4)	0·2110 (3)	0·4072 (2)	5·32 (7)
C16	0·3492 (3)	0·2711 (3)	0·6890 (2)	3·71 (5)
C17	0·4267 (3)	0·2138 (3)	0·7592 (2)	3·59 (5)
C18	0·5141 (3)	0·3086 (3)	0·8334 (2)	4·07 (6)
C19	0·5283 (3)	0·4685 (3)	0·8454 (2)	3·83 (6)
C20	0·4536 (3)	0·5353 (3)	0·7834 (2)	4·00 (6)
C21	0·3710 (3)	0·4400 (3)	0·7094 (2)	3·72 (5)



(*a*)



(*b*)

Fig. 1. *ORTEP* drawing of the title compound: (*a*) cation, (*b*) anion.

Table 2. *Bond distances* (Å) *and bond angles* (°)

O1—C11	1·214 (3)	C1—C11	1·485 (3)
O2—C11	1·318 (3)	C2—C3	1·387 (4)
O2—C12	1·461 (3)	C3—C4	1·344 (4)
O3—C16	1·239 (3)	C4—C10	1·417 (3)
O4—N2	1·211 (3)	C5—C6	1·358 (3)
O5—N2	1·220 (3)	C5—C10	1·408 (4)
O6—N3	1·227 (3)	C6—C7	1·386 (4)
O7—N3	1·222 (3)	C7—C8	1·363 (4)
O8—N4	1·216 (3)	C8—C9	1·431 (3)
O9—N4	1·184 (3)	C9—C10	1·429 (3)
N1—C8	1·467 (4)	C12—C13	1·468 (5)
N1—C14	1·493 (3)	C16—C17	1·446 (4)
N1—C15	1·495 (3)	C16—C21	1·443 (3)
N2—C17	1·449 (3)	C17—C18	1·369 (3)
N3—C19	1·444 (3)	C18—C19	1·374 (4)
N4—C21	1·452 (4)	C19—C20	1·385 (4)
C1—C2	1·380 (3)	C20—C21	1·355 (3)
C1—C9	1·454 (3)		
C11—O2—C12	116·7 (2)	C1—C9—C8	129·9 (2)
C8—N1—C14	111·4 (2)	C1—C9—C10	116·2 (2)
C8—N1—C15	114·2 (3)	C8—C9—C10	113·9 (2)
C14—N1—C15	111·0 (2)	C4—C10—C5	117·3 (2)
O4—N2—O5	121·7 (2)	C4—C10—C9	121·2 (2)
O4—N2—C17	119·1 (2)	C5—C10—C9	121·5 (2)
O5—N2—C17	119·3 (2)	O1—C11—O2	120·0 (2)
O6—N3—O7	123·1 (3)	O1—C11—C1	127·4 (2)
O6—N3—C19	118·8 (2)	O2—C11—C1	112·7 (2)
O7—N3—C19	118·2 (2)	O2—C12—C13	107·6 (3)
O8—N4—O9	122·0 (3)	O3—C16—C17	125·9 (2)
O8—N4—C21	118·1 (2)	O3—C16—C21	123·0 (2)
O9—N4—C21	119·7 (2)	C17—C16—C21	111·1 (2)
C2—C1—C9	118·9 (2)	N2—C17—C16	119·9 (2)
C2—C1—C11	114·4 (3)	N2—C17—C18	116·1 (2)
C9—C1—C11	126·6 (2)	C16—C17—C18	124·0 (2)
C1—C2—C3	123·4 (2)	C17—C18—C19	119·8 (2)
C2—C3—C4	119·4 (3)	N3—C19—C18	118·9 (2)
C3—C4—C10	121·0 (2)	N3—C19—C20	120·4 (2)
C6—C5—C10	121·7 (2)	C18—C19—C20	120·7 (3)
C5—C6—C7	118·4 (3)	C19—C20—C21	118·9 (2)
C6—C7—C8	121·5 (2)	N4—C21—C16	116·8 (2)
N1—C8—C7	115·9 (2)	N4—C21—C20	117·6 (2)
N1—C8—C9	121·0 (2)	C16—C21—C20	125·6 (2)
C7—C8—C9	123·0 (2)		

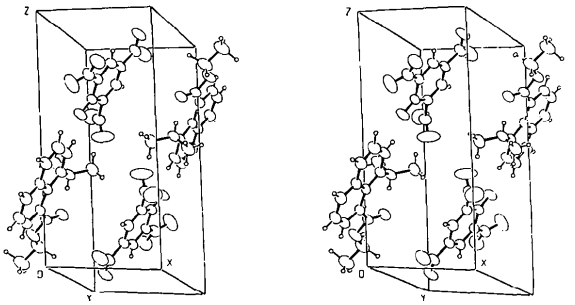


Fig. 2. Stereoview of the unit-cell packing.

atom (O···H 1·70 Å). The amine N atom is 0·424 (2) Å from the plane of its three bonded C atoms.

Experimental. Methyl 8-(*N*,*N*-dimethylamino)-1-naphthalenecarboxylate was synthesized using a published procedure (Schweizer, Proctor, Kaftory & Dunitz, 1978). Heating of this compound with an equimolar amount of picric acid in ethanol afforded the picrate of the ethyl ester as a consequence of ester interchange with the solvent. Recrystallizations from ethanol produced crystals of the title compound suitable for X-ray analysis (m.p. 408–411 K).

A crystal of approximate dimensions 0·25 × 0·30 × 0·42 mm was cut from a large block and used for data collection on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. The cell constants and orientation matrix were determined by least-squares refinement of the setting angles of 25 reflections in the 10–15° range. Intensity data were collected in the range $2 < \theta < 25°$ using the $\omega/2\theta$ scan method and variable scan speed (1·10–5·50° min^{-1}). The intensities of three standard reflections, monitored at regular intervals, did not show significant variations. 3095 unique reflections were collected (h 0→9, k −10→10, l −18→18), of which 2518 with $I > 3\sigma(I)$ were considered observed. Data were corrected for Lorentz and polarization effects; absorption was ignored.

The structure was solved by direct methods (*MULTAN*11/82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least-squares calculations on F's. H atoms were located from a difference map and included in the refinement fixed at these positions with the overall isotropic temperature factor B_{iso} = 5·0 Å^2; C, O and N had anisotropic temperature factors. The refinement converged completely with R = 0·048 and wR = 0·062, where $w = [\sigma^2(F_o) + (0{\cdot}010F_o)^2]^{-1}$; max. shift/e.s.d. in the last cycle of refinement was < 0·01 and goodness of fit, S = 1·383. A final difference map was devoid of significant features with $\Delta\rho$ in the range −0·40 to 0·48 e Å^{-3}. Scattering factors used in the calculations were taken from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965). Computer programs used in this study were from the Enraf–Nonius *Structure Determination Package* (B. A. Frenz & Associates, Inc., 1985) and *ORTEP*II (Johnson, 1976).

Final fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s are listed in Table 1.* Table 2 contains bond lengths and bond angles. Fig. 1 shows the molecular structure of the title compound. Fig. 2 is a stereoview of the unit-cell packing.

Related literature. 8-Methoxy- and 8-nitronaphthonitrile (Procter, Britton & Dunitz, 1981), *N,N*-dimethyl-8-nitro-1-naphthaleneamine (Egli, Wallis & Dunitz, 1986), and 8-dimethylamino-1-naphthonitrile (Parvez & Schuster, 1990).

* Lists of structure amplitudes, anisotropic temperature factors, least-squares-planes data, H-atom parameters and molecular dimensions involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53372 (35 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

B. A. Frenz & Associates, Inc. (1985). *SDP Structure Determination Package*. College Station, Texas, USA.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Egli, M., Wallis, J. D. & Dunitz, J. D. (1986). *Helv. Chim. Acta*, **69**, 255–266.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Parvez, M. & Schuster, I. I. (1990). *Acta Cryst.* C**46**, 947–948.

Procter, G., Britton, D. & Dunitz, J. D. (1981). *Helv. Chim. Acta*, **64**, 471–477.

Schweizer, W. B., Proctor, G., Kaftory, M. & Dunitz, J. D. (1978). *Helv. Chim. Acta*, **61**, 2783–2808.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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Structure of (±)-Cycleatjehenine, a New Bisbenzylisoquinoline Alkaloid from *Cyclea atjehensis*

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Abstract. 6,10,25-Trimethoxy-30-methyl-8,2,3-dioxa-15,30-diazaheptacylco[22.6.2.2^{9,6}.2^{18,21}.1^{3,7}.0^{12,35}.0^{27,31}]heptatriaconta-3,5,7(37),9,11,13,15,18,20,24,26,31,33,35-undecaen-32-ol–methanol (1/2), $C_{37}H_{36}N_2O_6.2CH_3OH$, M_r = 668·79, monoclinic, $P2_1/n$, a = 11·034 (3), b = 33·399 (3), c = 10·416 (2) Å, β = 114·25 (1)°, V = 3500 (2) Å^3, Z =

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