BERNSTEIN, J., COHEN, M. D. & LEISEROWITZ, L. (1974). *The Chemistry of Quinonoid Compounds*, edited by S. PATAI, pp. 37–110. New York: John Wiley.
BERNSTEIN, J., GOLDSTEIN, E. & GOLDBERG, I. (1980). *Cryst. Struct. Commun.* **9**, 295–299, 301–305.
BLOCK, S., WEIR, C. E. & PIERMARINI, G. J. (1970). *Science*, **169**, 586–587.
BRAUN, P. B., HORNSTRA, J. & LEENHOUTS, J. I. (1969). *Philips Res. Rep.* **24**, 85–118.
BURKE-LAING, M. & LAING, M. (1976). *Acta Cryst.* B**32**, 3216–3224.
DUNITZ, J. D. & SEILER, P. (1973). *Acta Cryst.* B**29**, 589–595.
EISENSTEIN, M. (1979). *Acta Cryst.* B**35**, 2614–2625.
GOLDBERG, I. (1975). *Acta Cryst.* B**31**, 754–762, 2592–2600.
GOLDBERG, I. (1980). *Cryst. Struct. Commun.* **9**, 329–333.
GOLDBERG, I., BERNSTEIN, J. KOSOWER, E. M., GOLDSTEIN, E. & PAZHENCHEVSKY, B. (1982). Unpublished results.
GOLDBERG, I. & KOSOWER, E. M. (1982). *J. Phys. Chem.* **86**, 332–335.
GOLDBERG, I. & SHMUELI, U. (1973). *Acta Cryst.* B**29**, 432–440.
HEHRE, W. J., LANTHAN, W. A., DITCHFIELD, R., NEWTON, N. W. & POPLE, J. A. (1976). *Quantum Chemistry Program Exchange*. Indiana Univ., Bloomington, Indiana. Program No. 236.
HIRSHFELD, F. L. (1977). *Isr. J. Chem.* **16**, 226–229.
HUPPERT, D., DODIUK, H., KANETY, H. & KOSOWER, E. M. (1979). *Chem. Phys. Lett.* **65**, 164–168.
International Tables for X-ray Crystallography (1974). Vol. IV, pp. 71–98. Birmingham: Kynoch Press.
KOSOWER, E. M., HERMOLIN, J., PAZHENCHEVSKY, B. & GOLDBERG, I. (1981). Unpublished results.
KOSOWER, E. M. & PAZHENCHEVSKY, B. (1980). *J. Am. Chem. Soc.* **102**, 4983–4993.
KOSOWER, E. M., PAZHENCHEVSKY, B., DODIUK, H., KANETY, H. & FAUST, D. (1981). *J. Org. Chem.* **46**, 1666–1673.
KOSOWER, N. S., KOSOWER, E. M., ZIPSER, Y., FALTIN, Z. & SHOMRAT, R. (1981). *Biochim. Biophys. Acta*, **640**, 748–759.
KOSOWER, N. S., NEWTON, G. L., KOSOWER, E. M. & RANNEY, H. M. (1980). *Biochim. Biophys. Acta*, **622**, 201–209.
LAING, M., SOMMERVILLE, P. & PIACENZA, L. P. L. (1977). *Acta Cryst.* B**33**, 2464–2471.
MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1974). *MULTAN* 74. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
PIERMARINI, G. J., MIGHELL, A. D., WEIR, C. E. & BLOCK, S. (1969). *Science*, **165**, 1250–1255.
PROUT, C. K. & KAMENAR, B. (1973). *Molecular Complexes*. Vol. I, ch. 4, edited by R. FOSTER. London: Elek Science.
SCHOMBERG, D. (1980). *J. Am. Chem. Soc.* **102**, 1055–1058.
SINGH, T. P. & VIJAYAN, M. (1973). *Acta Cryst.* B**29**, 714–720.
SINGH, T. P. & VIJAYAN, M. (1974). *Acta Cryst.* B**30**, 557–562.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1982). B**38**, 2000–2004

Structures of Inorganic Rings as Antitumor Agents. III.* Structures of the Monoclinic and Orthorhombic Forms of 2,2,4,4,6,6-Hexa(1-aziridinyl)cyclotri(phosphazene), $N_3P_3az_6$

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Abstract

$P_3N_3[N(CH_2)_2]_6$, $C_{12}H_{24}N_9P_3$, $M_r = 387{\cdot}3$; form (I): orthorhombic, $a = 7{\cdot}981\ (2)$, $b = 13{\cdot}641\ (3)$, $c = 16{\cdot}589\ (3)$ Å, $Z = 4$, $D_c = 1{\cdot}424$ g cm^{-3}, $\mu = 2{\cdot}93$ cm^{-1}, space group $P2_12_12_1$, 1030 observed reflections, $R = 0{\cdot}027$; form (II): monoclinic, $a = 9{\cdot}626\ (2)$, $b = 8{\cdot}099\ (3)$, $c = 23{\cdot}67\ (1)$ Å, $\beta = 101{\cdot}39\ (3)°$, $Z = 4$, $D_c = 1{\cdot}420$ g cm^{-3}, $\mu = 2{\cdot}92$ cm^{-1}, $P2_1/c$, 998 observed reflections, $R = 0{\cdot}053$. Monochromatic Mo $K\alpha_1$ radiation ($\lambda = 0{\cdot}70926$ Å) was used. The structures were solved by the direct method. The molecule in each of the two forms has a very similar conformation with the geminal aziridinyl groups adopting the *trans* arrangement.

* Part II: Cameron, Labarre & Graffeuil (1982).
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Introduction

The aziridinylcyclotri(phosphazenes) have been shown to have powerful antitumor activity (Labarre *et al.*, 1978; Labarre, Faucher, Levy, Sournies, Cros & Francois, 1979); of these, hexaaziridinylcyclotri(phosphazene) is one of the more promising examples. This compound crystallizes in a number of different forms (Galy, Enjalbert & Labarre, 1980; Cameron, Labarre & Graffeuil, 1982); reported here is the structure of the compound which has been recrystallized from *m*-xylene to give an orthorhombic form (I) and from carbon disulphide to give a monoclinic form (II).

Systematic extinctions [crystal (I): $h00$, $h = 2n + 1$; $0k0$, $k = 2n + 1$; $00l$, $l = 2n + 1$; crystal (II): $h0l$, $l = 2n + 1$; $0k0$, $k = 2n + 1$] are consistent with space groups $P2_12_12_1$ [crystal (I)] and $P2_1/c$ [crystal (II)]. 1835 [2494 for crystal (II)] independent reflections, of which 1030 [998] had $I > 3\sigma(I)$, were measured on a CAD-4 diffractometer (2θ: 4–46°).

Both crystal forms readily produce large crystals. The orthorhombic crystals from *m*-xylene were needle-shaped and suitable for X-ray structure analysis. Those from carbon disulphide were prismatic, forming a central 'lump' from which a cluster of very fine needles extended. The needles were far too thin for X-ray work, cross section ~0·001 mm^2, but the central lump when cleaved and cleaned gave a diffraction pattern. This pattern showed extensive loom-line streaking, thermal diffuse scatter and multiple reflections on the Weissenberg photographs. Powdered crystals of the monoclinic form were sealed in contact with a saturated CS_2 solution and the temperature of the solution was continuously raised and then lowered between ambient temperature and 313 K for periods of a day, several days, and up to three weeks. The time for a full temperature cycle was approximately 2 h. This process did produce larger crystals but the compound appeared to have decomposed. Attempts were also made to zone-refine the crystal using the CAD-4 variable-temperature attachment to melt the crystal partially in the capillary and allow it to resolidify. This was not successful, and there seemed to be some slight evidence from the diffraction pattern that the crystal was changing to the orthorhombic form. Therefore, a powdered sample was examined with a Guinier–Lenné X-ray camera. [The powder pattern is projected through a narrow slit onto an X-ray film, while the film cassette is moving synchronously with a smoothly varying sample temperature (Lenné, 1961).] The evidence of this scan was not conclusive, but it appeared that the material progressively decomposed as the temperature was raised, and the small portion that did not decompose formed the orthorhombic sample as it cooled. No method, however, produced crystals of the monoclinic form that were substantially better than those deposited originally from the CS_2 solution. The resultant structure of the monoclinic form is not good (see below) and we report it here only for completeness.

Solution and refinement of the structures

The data for both compounds were processed by routine methods (Cameron & Cordes, 1979) and the structures were solved by the application of the multi-solution tangent-refinement procedure in *SHELX* (Sheldrick, 1976). The solution proceeded without incident, and produced the positions of all non-hydrogen atoms which were refined with isotropic temperature factors by full-matrix least squares on $\sum w(\Delta F)^2$ to $R = 0{\cdot}069$ and 0·075 for forms (I) and (II) respectively. The difference Fourier syntheses for both forms showed the positions of the H atoms, and the refinement was continued with anisotropic temperature factors on the heavy atoms and isotropic ones on the H atoms until it converged at $R = 0{\cdot}027$ (I) and 0·053 (II) for the observed reflections. Weights were given by $w = (\sigma^2 |F_o| + xF_o^2)^{-1}$, where x is 0·0089 for form (I) and 0·0047 for form (II), and σ is the e.s.d. for each reflection as derived from the diffractometer counting statistics. The atomic parameters are given in Tables 1 and 2; the interatomic distances for form (I) are given

Table 1. *Orthorhombic form: atomic parameters* ($\times 10^4$)

Mod $(U) = (U_{11} + U_{22} + U_{33})/3$.

	x	y	z	Mod (U) (Å^2)
P(1)	4859 (2)	−966 (1)	9051 (1)	280 (7)
P(2)	3472 (2)	904 (1)	9231 (1)	267 (7)
P(3)	4699 (2)	−88 (1)	10576 (1)	290 (7)
N(1)	5379 (6)	−918 (3)	9978 (3)	350 (23)
N(2)	4076 (6)	16 (4)	8682 (3)	351 (24)
N(3)	3853 (6)	850 (3)	10168 (2)	315 (23)
N(11)	3628 (7)	−1948 (3)	8922 (3)	353 (25)
N(12)	6446 (7)	−1296 (3)	8449 (3)	412 (28)
N(21)	1400 (5)	1017 (3)	9106 (3)	369 (24)
N(22)	4261 (6)	1979 (3)	8898 (3)	319 (24)
N(31)	6204 (7)	321 (4)	11199 (3)	423 (30)
N(32)	3443 (7)	−616 (3)	11250 (3)	380 (27)
C(111)	1850 (7)	−1843 (4)	9121 (3)	410 (31)
C(112)	2377 (9)	−1959 (4)	8289 (3)	485 (33)
C(121)	7863 (8)	1848 (5)	8781 (4)	506 (36)
C(122)	8099 (7)	−830 (5)	8523 (4)	491 (34)
C(211)	562 (7)	1935 (4)	9216 (4)	492 (34)
C(212)	688 (8)	1522 (5)	8409 (4)	519 (35)
C(221)	6081 (7)	2044 (4)	8767 (4)	461 (34)
C(222)	4914 (10)	2066 (4)	8078 (3)	499 (36)
C(311)	7854 (8)	556 (4)	10866 (4)	535 (34)
C(312)	7636 (9)	−304 (4)	11395 (4)	559 (38)
C(321)	2211 (9)	1 (5)	11681 (3)	555 (34)
C(322)	1645 (8)	−681 (4)	11054 (3)	492 (33)

Table 2. *Monoclinic form: atomic parameters* ($\times 10^4$)

Mod $(U) = (U_{11} + U_{22} + U_{33})/3$.

	x	y	z	Mod (U) (Å^2)
P(1)	4348 (3)	2481 (3)	3906 (1)	355 (14)
P(2)	2638 (3)	1089 (3)	2933 (1)	344 (15)
P(3)	1634 (3)	1424 (3)	3955 (1)	344 (14)
N(1)	3096 (8)	2221 (10)	4244 (4)	419 (49)
N(2)	4092 (8)	1866 (10)	3267 (3)	420 (49)
N(3)	1491 (8)	734 (10)	3319 (3)	342 (46)
N(11)	4673 (9)	4509 (9)	3897 (4)	470 (53)
N(12)	5788 (9)	1692 (9)	4315 (4)	432 (50)
N(21)	1845 (8)	2248 (9)	2380 (3)	403 (48)
N(22)	3009 (9)	−545 (9)	2556 (3)	467 (50)
N(31)	258 (8)	2694 (10)	3945 (4)	470 (49)
N(32)	1243 (9)	30 (11)	4422 (4)	493 (56)
C(111)	5639 (13)	5320 (12)	4370 (5)	672 (84)
C(112)	6024 (13)	5139 (13)	3818 (6)	786 (93)
C(121)	6876 (10)	902 (14)	4038 (5)	627 (73)
C(122)	5947 (11)	−114 (12)	4308 (5)	577 (74)
C(211)	1830 (10)	4050 (11)	2473 (5)	524 (68)
C(212)	2751 (10)	3389 (14)	2120 (4)	601 (70)
C(221)	1968 (10)	−1895 (11)	2418 (4)	467 (64)
C(222)	3268 (10)	−2173 (12)	2829 (4)	542 (67)
C(311)	361 (10)	3909 (14)	4420 (5)	624 (74)
C(312)	467 (10)	4456 (12)	3863 (5)	553 (68)
C(321)	221 (10)	−1322 (14)	4226 (4)	614 (71)
C(322)	1795 (12)	−1681 (12)	4399 (4)	589 (70)

Table 3. *Interatomic distances* (Å) *and angles* (°) *for the orthorhombic form*

P(1)–N(1)	1·594 (5)	N(12)–C(122)	1·469 (8)
P(1)–N(2)	1·600 (5)	N(21)–C(211)	1·431 (8)
P(1)–N(11)	1·675 (5)	N(21)–C(212)	1·461 (8)
P(1)–N(12)	1·675 (5)	N(22)–C(221)	1·472 (7)
P(2)–N(2)	1·591 (5)	N(22)–C(222)	1·462 (8)
P(2)–N(3)	1·585 (5)	N(31)–C(311)	1·463 (8)
P(2)–N(21)	1·673 (5)	N(31)–C(312)	1·463 (8)
P(2)–N(22)	1·688 (5)	N(32)–C(321)	1·479 (8)
P(3)–N(1)	1·600 (5)	N(32)–C(322)	1·475 (8)
P(3)–N(3)	1·597 (5)	C(111)–C(112)	1·451 (8)
P(3)–N(31)	1·680 (5)	C(121)–C(122)	1·465 (9)
P(3)–N(32)	1·664 (5)	C(211)–C(212)	1·455 (9)
N(11)–C(111)	1·464 (7)	C(221)–C(222)	1·474 (9)
N(11)–C(112)	1·449 (8)	C(311)–C(312)	1·476 (9)
N(12)–C(121)	1·466 (8)	C(321)–C(322)	1·468 (9)

N(1)–P(1)–N(2)	115·9 (2)	C(121)–N(12)–C(122)	59·9 (4)
N(1)–P(1)–N(11)	108·0 (2)	P(2)–N(21)–C(211)	121·8 (4)
N(1)–P(1)–N(12)	112·9 (2)	P(2)–N(21)–C(212)	121·7 (4)
N(2)–P(1)–N(11)	113·1 (3)	C(211)–N(21)–C(212)	60·4 (4)
N(2)–P(1)–N(12)	107·0 (3)	P(2)–N(22)–C(221)	118·0 (4)
N(11)–P(1)–N(12)	98·7 (2)	P(2)–N(22)–C(222)	120·5 (4)
N(2)– P(2)–N(3)	117·8 (2)	C(221)–N(22)–C(222)	60·3 (4)
N(2)–P(2)–N(21)	107·4 (2)	P(3)–N(31)–C(311)	118·9 (4)
N(2)–P(2)–N(22)	111·2 (2)	P(3)–N(31)–C(312)	120·1 (4)
N(3)–P(2)–N(21)	108·4 (2)	C(311)–N(31)–C(312)	60·6 (4)
N(3)–P(2)–N(22)	106·8 (2)	P(3)–N(32)–C(321)	118·6 (4)
N(21)–P(2)–N(22)	104·4 (2)	P(3)–N(32)–C(322)	117·7 (4)
N(1)–P(3)–N(3)	116·5 (2)	C(321)–N(32)–C(322)	59·6 (4)
N(1)–P(3)–N(31)	112·0 (2)	N(11)–C(111)–C(112)	59·6 (4)
N(1)–P(3)–N(32)	108·3 (2)	N(11)–C(112)–C(111)	60·7 (4)
N(3)–P(3)–N(31)	107·3 (2)	N(12)–C(121)–C(122)	60·1 (4)
N(3)–P(3)–N(32)	112·1 (2)	N(12)–C(122)–C(121)	60·0 (4)
N(31)–P(3)–N(32)	99·3 (3)	N(21)–C(211)–C(212)	60·8 (4)
P(1)–N(1)–P(3)	122·6 (3)	N(21)–C(212)–C(211)	58·8 (4)
P(1)–N(2)–P(2)	122·5 (3)	N(22)–C(221)–C(222)	59·5 (4)
P(2)–N(3)–P(3)	122·4 (3)	N(22)–C(222)–C(221)	60·2 (4)
P(1)–N(11)–C(111)	117·4 (4)	N(31)–C(311)–C(312)	59·7 (4)
P(1)–N(11)–C(112)	120·3 (4)	N(31)–C(312)–C(311)	59·7 (4)
C(111)–N(11)–C(112)	59·7 (4)	N(32)–C(321)–C(322)	60·1 (4)
P(1)–N(12)–C(121)	119·8 (4)	N(32)–C(322)–C(321)	60·4 (4)
P(1)–N(12)–C(122)	120·9 (4)		

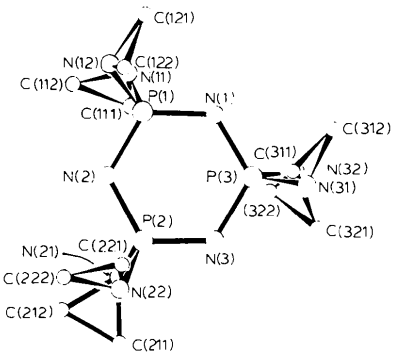

Fig. 1. The molecule viewed perpendicular to the P_3N_3 ring.

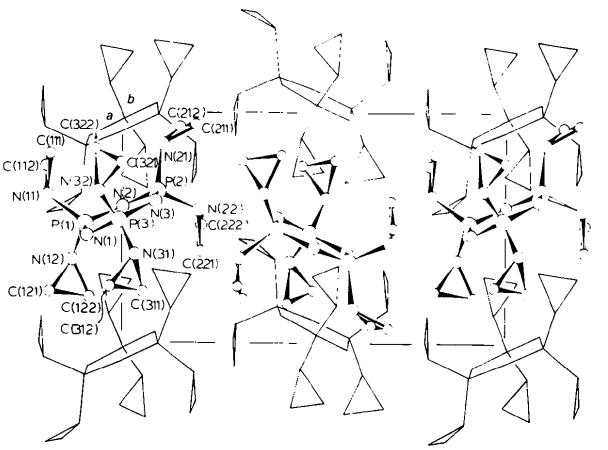

Fig. 2. The unit cell of form (I) (orthorhombic) viewed along **c**.

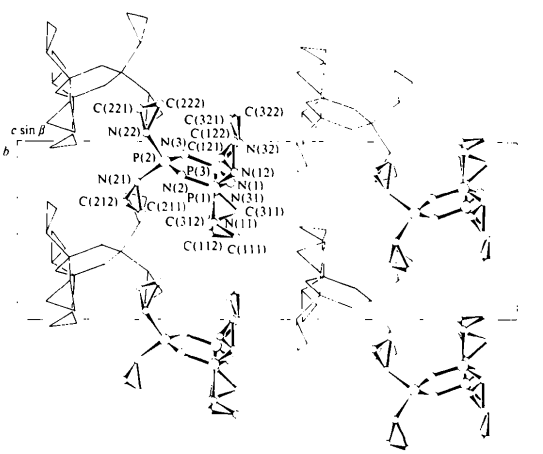

Fig. 3. The unit cell of form (II) (monoclinic) viewed along **a**.

in Table 3 and those for form (II) have been deposited.* The unique molecule for form (I) is shown in Fig. 1,

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters for (I) and (II) and torsion angles for (I) and bond distances, bond angles and torsion angles for (II) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36771 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

and the unit cells for forms (I) and (II) are given in Figs. 2 and 3 respectively.

Results and discussion

Both crystal forms are constructed from isolated molecules of hexaaziridinylcyclotri(phosphazene). The conformations of the molecules in the two crystal forms are shown in projection on the P_3N_3 plane in Fig. 4. It can be seen that the conformations are almost identical, and since there is no significant difference in the dimensions of the two forms, only those for the more accurately determined orthorhombic structure will be discussed.

The conformation of the aziridinyl groups is similar to that observed in the CCl_4 clathrate (Galy *et al.*, 1980) (Fig. 4*c*), and is quite different from that observed in the benzene clathrate (Cameron *et al.*, 1982) (Fig. 4*d*). Within the P_3N_3 ring, the P–N bond lengths are in the range 1·585 (5)–1·600 (5) Å [mean 1·596 (3) Å], with no two lengths significantly different. These lengths are normal for this type of compound, and are significantly longer than those found in $P_3N_3Cl_6$ (Bullen, 1971). The ring itself is not planar; with N(1), N(2) and N(3) 0·17, 0·07, and 0·01 (5) Å from the plane through the three P atoms. Thus the ring has a very slight crown configuration. The exocyclic P–N bond lengths are in the range 1·664 (5)–1·688 (5) Å, with a mean of 1·676 (4) Å. These lengths too are normal for this type of compound and are slightly longer than those [1·652 (4) Å] found in the exocyclic P–N bonds in hexakis(dimethylamino)cyclotri(phosphazene) (Rettig & Trotter, 1973), where the dimethylamino groups are essentially planar.

The aziridinyl groups are exceptionally pyramidal. The sums of the interbond angles at the N atoms are in the range 295·9 (4)–303·9 (4)°, with a mean of 300 (2)°. The group has a prominent electron lone-pair orbital on the N atom which occupies space within the crystal lattice and the efficient packing of the molecules in the crystal must take into account both the inter- and intramolecular forces acting on this lone pair.

The conformation that is adopted by the aziridinyl groups is worth examining. In the benzene clathrate the lone pairs of the two geminal aziridinyl groups adopt a *cis* conformation with the two lone-pair orbitals eclipsed when they are viewed directly along the line joining the two N atoms (Cameron *et al.*, 1982). In this compound, the axes of the lone-pair orbitals are inclined at angles of 96·7 [N(11)···N(12)], 134·0 [N(21)···N(22)], and 102·6° [N(31)···N(41)] and so adopt the more normal *trans* configuration.

In the aziridinyl groups themselves, five groups have very similar conformations with respect to the P_3N_3 ring; the sixth group –N(21)–C(211)–C(212) has a different orientation (Fig. 1). The N–C bond lengths in the five groups are not significantly different and have a mean value of 1·466 (7) Å, which compares well with 1·466 (3) and 1·467 (2) Å in the CCl_4 and benzene clathrates respectively (Galy *et al.*, 1980; Cameron *et al.*, 1982); the N–C lengths in the sixth group are 1·431 (8) and 1·461 (8) Å, which are significantly different. A similar trend in distances is observed in the monoclinic form, but that structure is too ill defined for detailed comparison. The C–C distances in the aziridinyl groups do not have such a wide range of lengths [mean 1·465 (4) Å]; they do not differ significantly among themselves or from those observed elsewhere (Galy *et al.*, 1980, 1982; Cameron, Chan, Labarre & Graffeuil, 1980).

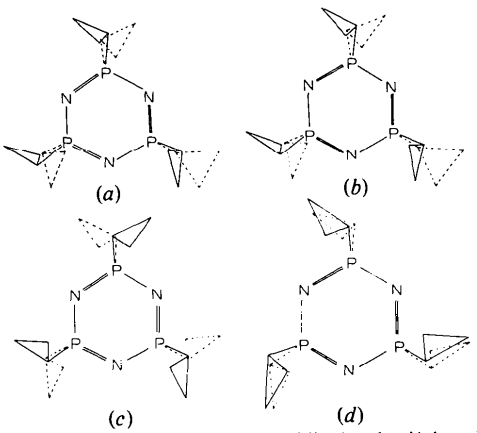

Fig. 4. The conformations of hexaaziridinylcyclotri(phosphazene) in four different structures: (*a*) form (I), (*b*) form (II), (*c*) carbon tetrachloride clathrate, (*d*) benzene clathrate.

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References

Bullen, G. J. (1971). *J. Chem. Soc. A*, pp. 1450–1453.
Cameron, T. S., Chan, C., Labarre, J.-F. & Graffeuil, M. (1980). *Z. Naturforsch. Teil B*, **35**, 784–785.
Cameron, T. S. & Cordes, R. E. (1979). *Acta Cryst.* B**35**, 748–750.
Cameron, T. S., Labarre, J.-F. & Graffeuil, M. (1982). *Acta Cryst.* B**38**, 168–171.
Galy, J., Enjalbert, R. & Labarre, J.-F. (1980). *Acta Cryst.* B**36**, 392–397.
Galy, J., Labarre, J.-F. & Graffeuil, M. (1982). To be published.

LABARRE, J.-F., CROS, S., FAUCHER, J.-P., FRANCOIS, G., LEVY, G., PAOLETTI, C. & SOURNIES, F. (1978). 2nd Int. Symp. on Inorganic Ring Systems (IRIS), Göttingen. Proceedings, p. 44.

LABARRE, J.-F., FAUCHER, J.-P., LEVY, G., SOURNIES, F., CROS, S. & FRANCOIS, G. (1979). *Eur. J. Cancer*, **15**, 637–643.

LENNÉ, H.-U. (1961). *Z. Kristallogr.* **116**, 3–6.

RETTIG, S. J. & TROTTER, J. (1973). *Can. J. Chem.* **51**, 1295–1302.

SHELDRICK, G. M. (1976). *SHELX*. X-ray system report, Univ. Chemical Laboratory, Lensfield Road, Cambridge, England.

Acta Cryst. (1982). B**38**, 2004–2008

The Orientation of a $T = 1$ Assembly of Alfalfa Mosaic Virus Coat Protein in a Hexagonal Crystalline Array

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Abstract

X-ray diffraction data for $T = 1$ alfalfa mosaic virus protein aggregates, crystallized as type I hexagonal crystals, have been collected to 4·5 Å resolution. A rotation function reveals the particle orientation in the unit cell. Packing considerations show the particles possibly to have small protrusions along their twofold axes.

Introduction

Alfalfa mosaic virus (AMV) is an RNA-containing plant virus whose genome is divided into four segments (Jaspars, 1974). Each RNA molecule is encapsidated by the same type of coat protein subunits into bacillus-shaped particles, whose lengths vary from 260 to 590 Å (Hull, 1969; Hull, Hills & Markham, 1969). The particles have an external diameter of 180 Å, with icosahedral $T = 1$ ends [$T = 1$ is the notation of Caspar & Klug (1962)]. The central portions have protein subunits arranged in a hexagonal ($P6$) lattice (Gibbs, Nixon & Woods, 1963; Hull *et al.*, 1969; Mellema & van den Berg, 1974). The single protein in the coat has a molecular weight of 24 250 (Van Beynum, De Graaf, Castel, Kraal & Bosch, 1977), and it is stable as a dimer over a wide range of conditions. These protein dimers are able to aggregate, in the absence of RNA, into spherical $T = 1$ shells containing 30 dimers each (Driedonks, Krijgsman & Mellema, 1977). Similar spherical particles are obtained in the presence of nucleic acid (Hull, 1970; Lebeurier, Fraenkel-Conrat, Wurtz & Hirth, 1971; Driedonks, Krijgsman & Mellema, 1978).

We have recently reported (Fukuyama, Abdel-Meguid & Rossmann, 1981) the crystallization of reassembled AMV coat protein particles which were partially digested with trypsin to remove the first 26 amino acid residues. These particles are empty icosahedral, $T = 1$ protein shells built with 60 AMV protein subunits, and are about 190 Å in diameter. In the hexagonal type I crystals ($a = 199·8$, $c = 314·5$ Å, $P6_3$), which have been utilized in this study, there are two virus particles per unit cell, each being situated on a crystallographic threefold axis. The orientation of the particle about this threefold axis remained unknown.

Two approaches have been utilized in determining the orientation of the non-crystallographic symmetry axes of virus particles from X-ray diffraction data. In one, Caspar (1956) utilized spikes of high-intensity reflections on precession photographs to determine the orientation of tomato bushy stunt virus (TBSV). These spikes, which extend from the reciprocal-lattice origin, represent the positions of particle symmetry axes. The angular relation between these spikes allows the identification of the symmetry of each axis and, thus, the orientation of the particle in the unit cell. In the second approach, first utilized on satellite tobacco necrosis virus (STNV; Åkervall *et al.*, 1972; Lentz & Strandberg, 1974), the orientations of the non-crystallographic axes are determined from three-dimensional data by means of a rotation function (Rossmann & Blow, 1962).

In this paper, details are reported of the data collection as well as the determination of the AMV

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