

(Gilardi, George & Flippen-Anderson, 1989), 3,15-diacetoxy-7,8-dihydroxy-12,13-epoxytrichothec-9-ene (Hanson, 1986), Myrotoxin A and C (Jarvis, Cömezoglu, Lee, Flippen-Anderson, Gilardi & George, 1986), Verrucarin A (McPhail & Sim, 1966), Verrucarin B (Breitenstein, Tamm, Arnold & Clardy, 1979) and  $\alpha/\beta,8\beta,15$ -triacetoxy-12,13-epoxytrichothec-9-en-3 $\alpha$ -ol (Dillen, Gorst-Allman & Steyn, 1986).

This work was supported by USAMRIID contract #85MM5511.

#### References

- BREITENSTEIN, W., TAMM, C., ARNOLD, E. V. & CLARDY, J. (1979). *Helv. Chim. Acta*, **62**, 2697–2703.
- DILLEN, J. L. M., GORST-ALLMAN, C. P. & STEYN, P. S. (1986). *S. Afr. J. Chem.* **39**, 111.
- GILARDI, R., GEORGE, C. & FLIPPEN-ANDERSON, J. L. (1989). *Acta Cryst.* Accepted.
- HANSON, A. W. (1986). *Acta Cryst.* **C42**, 503–505.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JARVIS, B. B., CÖMEZOGLU, F. T., LEE, Y. M., FLIPPEN-ANDERSON, J. L., GILARDI, R. D. & GEORGE, C. F. (1986). *Bull. Soc. Chim. Belg.* **95**, 681–697.
- JARVIS, B. B., LEE, Y. M., YATAWARA, C. S., MAZZOCCHI, D. B., FLIPPEN-ANDERSON, J. L., GILARDI, R. D. & GEORGE, C. F. (1985). *Appl. Environ. Microbiol.* **50**, 1225–1228.
- MCPHAIL, A. T. & SIM, G. A. (1966). *J. Chem. Soc. C*, pp. 1394–1396.
- SHELDRIK, G. M. (1980). *SHELXTL80. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. Univ. of Göttingen, Federal Republic of Germany.

*Acta Cryst.* (1989). **C45**, 1665–1666

## Structure of a 1,3-Diphosphetane Derivative

BY PETER G. JONES

*Institut für Anorganische und Analytische Chemie der Technischen Universität, Hagenring 30, 3300 Braunschweig, Federal Republic of Germany*

GENE B. CARPENTER

*Department of Chemistry, Brown University, Providence, RI 02912, USA*

AND GEORGE M. SHELDRIK

*Institut für Anorganische Chemie der Universität, Tammannstrasse 4, 3400 Göttingen, Federal Republic of Germany*

(Received 28 November 1988; accepted 30 May 1989)

**Abstract.** 2,3,7,8-Tetramethyl-3,8-diaza-1,6-diphosphatricyclo[5.3.0.0<sup>2,6</sup>]decane,  $C_{10}H_{20}N_2P_2$ ,  $M_r = 230.23$ , monoclinic,  $P2_1/c$ ,  $a = 6.852$  (2),  $b = 13.683$  (4),  $c = 6.844$  (2) Å,  $\beta = 110.96$  (3)°,  $V = 599.2$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.276$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 2.9$  mm<sup>-1</sup>,  $F(000) = 248$ ,  $T = 293$  K. The structure was refined to  $R = 0.041$  for 948 unique observed reflections. The molecule possesses a crystallographic centre of symmetry. The P—C bond lengths in the four-membered ring are 1.888 (2) and 1.915 (2) Å, with ring bond angles 86.2 (1)° at P and 93.8 (1)° at C.

**Experimental.** A somewhat cloudy colourless block was mounted in a glass capillary. 1968 profile-fitted intensities (Clegg, 1981) were registered on a Stoe-Siemens four-circle diffractometer using graphite-monochromated Cu  $K\alpha$  radiation ( $2\theta_{\text{max}} = 130^\circ$ , one

hemisphere of data). An absorption correction was applied (crystal size  $0.5 \times 0.35 \times 0.2$  mm) that, on the basis of  $\psi$  scans, gave transmission factors of 0.59–0.86. Three check reflections decreased in intensity by ca 2% and an appropriate correction was applied. Merging equivalents gave 997 unique reflections ( $R_{\text{int}} = 0.059$ , index ranges after merging  $h - 7$  to 7,  $k 0$  to 15,  $l 0$  to 8, 948 of which with  $F > 4\sigma(F)$  were used for all calculations [program system *SHELX* (Sheldrick, 1976) locally modified by GMS]. Cell constants were refined from  $2\theta$  values of 54 reflections in the range 60–70°. Because the  $a$  and  $c$  axes are nearly equal, it is possible to construct a centred, metrically orthorhombic cell, but the true Laue symmetry is no higher than monoclinic.

The structure was solved by routine direct methods and subjected to anisotropic full-matrix least-squares refinement on  $F$  (H atoms located in

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )

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

	$x$	$y$	$z$	$U_{eq}$
P	5689.6 (8)	4622.3 (4)	3478.6 (8)	42 (1)
N	2776 (3)	3598 (1)	4398 (3)	44 (1)
C(2)	4749 (3)	4114 (2)	5550 (3)	40 (1)
C(4)	1616 (3)	4142 (2)	2495 (3)	51 (1)
C(5)	3147 (4)	4340 (2)	1402 (3)	54 (1)
C(6)	6243 (4)	3428 (2)	7140 (4)	51 (1)
C(7)	1496 (4)	3371 (2)	5642 (4)	58 (1)

difference synthesis, but included using a riding model). The final  $R$  value was 0.041, with  $wR = 0.056$ . The weighting scheme was  $w^{-1} = \sigma^2(F) + 0.00015F^2$ . 64 parameters,  $S = 3.3$ , max.  $\Delta/\sigma = 0.01$ , max.  $\Delta\rho = 0.25 \text{ e \AA}^{-3}$ . Atomic scattering factors as incorporated in *SHELX*.

Final atomic coordinates are given in Table 1,\* with derived bond lengths and angles in Table 2. The molecule is depicted in Fig. 1.

**Related literature.** The preparation of the compound from 2-(*N*-methylamino)ethylphosphine and dimethylacetamide dimethyl acetal has been described by Issleib, Schmidt & Leissring (1988), who also presented preliminary details of the structure.

We thank Professor Dr K. Issleib for providing the crystals and the Fonds der Chemischen Industrie for financial support.

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51950 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ )

P—C(2)	1.888 (2)	1.894*	P—C(2')	1.915 (2)	1.920
P—C(5)	1.853 (2)	1.859	N—C(2)	1.478 (3)	1.482
N—C(4)	1.463 (3)	1.468	N—C(7)	1.458 (3)	1.463
C(2)—C(6)	1.522 (3)	1.527	C(4)—C(5)	1.515 (4)	1.520
C(5)—P—C(2)	91.1 (1)		C(4)—N—C(2)	109.5 (2)	
C(7)—N—C(2)	114.6 (2)		C(7)—N—C(4)	112.3 (2)	
N—C(2)—P	105.5 (1)		C(6)—C(2)—P	116.2 (2)	
C(6)—C(2)—N	109.9 (2)		C(5)—C(4)—N	106.2 (2)	
C(4)—C(5)—P	106.6 (1)		C(2)—P—C(2')	86.2 (1)	
C(5)—P—C(2')	103.5 (1)		P—C(2)—P(i)	93.8 (1)	
N—C(2)—P(i)	112.9 (1)		C(6)—C(2)—P(i)	117.2 (1)	

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

\*The second values given for bond lengths are libration-corrected assuming rigid-body properties ( $R_{lib} = 0.046$ ).

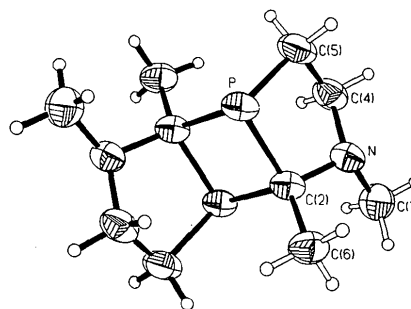


Fig. 1. The centrosymmetric molecule of the 1,3-diphosphetane derivative in the crystal, showing the numbering scheme of the asymmetric unit. Radii are arbitrary.

## References

- CLEGG, W. (1981). *Acta Cryst.* **A37**, 22–28.  
 ISSLEIB, K., SCHMIDT, H. & LEISSRING, E. (1988). *J. Organomet. Chem.* **355**, 71–77.  
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.