Table 3. *Hydrogen-bonding distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Donor	H	Acceptor	*D*—H	H···*A*	*D*···*A*	Angle
Intermolecular						
N(1)	H(18^{i})	Cl(16)	0·91 (3)	2·38 (3)	3·240 (2)	157 (2)
N(4)	H(21^{iii})	Cl(16)	0·88 (3)	2·39 (3)	3·240 (2)	162 (2)
Intramolecular						
N(1)	H(17)	Cl(16)	0·84 (2)	2·58 (2)	3·340 (2)	150 (2)
N(2)	H(20)	Cl(16)	0·89 (3)	2·47 (3)	3·280 (2)	152 (3)

References

CASSEL, D. & PFEUFFER, T. (1978). *Proc. Natl Acad. Sci. USA*, **75**, 2669–2673.

CROMER, D. T. & WABER, J. T. (1974). In *International Tables for X-ray Crystallography*, Vol. IV, edited by J. A. IBERS & W. C. HAMILTON, p. 99. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

GOFF, C. G. (1974). *J. Biol. Chem.* **249**, 6181–6190.

JOHNSON, C. K. (1970). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

KARCHER, B. A. (1981). *ABSN*. PhD dissertation, Iowa State Univ.

LAPP, R. L. & JACOBSON, R. A. (1979). *ALLS*. US Department of Energy Report IS-4708. Iowa State Univ.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MOSS, J. & VAUGHAN, M. (1977). *J. Biol. Chem.* **252**, 2455–2457.

NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

POWELL, D. R. & JACOBSON, R. A. (1980). *FOUR*. US Department of Energy Report IS-4737. Iowa State Univ.

PURNELL, M. R., STONE, P. R. & WHISH, W. J. D. (1980). *Biochem. Soc. Trans.* **8**, 215–227.

SOMAN, G., TOMER, K. B. & GRAVES, D. J. (1983). *Anal. Biochem.* **134**, 101–110.

Acta Cryst. (1986). C**42**, 1757–1759

4-Methyl-1,2,6-triphosphatricyclo[2.2.1.$0^{2,6}$]heptane

BY HELMUTH ZIMMERMANN* AND MARTIN GOMM

Institut für Angewandte Physik, Lehrstuhl für Kristallographie der Universität Erlangen-Nürnberg, Bismarckstrasse 10, *D*-8520 *Erlangen, Federal Republic of Germany*

AND EBERHARD KÖCK AND JOCHEN ELLERMANN

Institut für Anorganische Chemie der Universität Erlangen-Nürnberg, Egerlandstrasse 1, *D*-8520 *Erlangen, Federal Republic of Germany*

(*Received* 17 *March* 1986; *accepted* 29 *May* 1986)

Abstract. $C_5H_9P_3$, $M_r = 162{\cdot}05$, hexagonal, $R\bar{3}$, $a = 9{\cdot}281\,(1)$, $c = 15{\cdot}386\,(2)$ Å, $V = 1147{\cdot}7\,(2)$ Å^3, $Z = 6$, $D_x = 1{\cdot}41$ g cm^{-3}, λ(Mo $K\alpha$) = 0·70926 Å, $\mu = 6{\cdot}7$ cm^{-1}, $F(000) = 504$, room temperature, final $R = 0{\cdot}043$ for 563 reflections (including five unobserved). The molecule consists of three P atoms which form a regular triangle [P—P: 2·200(1) Å] and the organic group [P—C: 1·851(2) Å]. Literature data of bond lengths and bond angles are in good agreement with our results. In the crystal the molecules are held together by van der Waals forces.

Introduction. The title compound is especially suited for the study of phosphorus bonding because the molecule possesses a single P_3 unit. The electronic structure was calculated on the basis of its photoelectron spectrum (Gleiter, Köppel, Hofmann, Schmidt & Ellermann, 1985). Here we describe the solid-state structure determined by X-ray analysis. The analogous Sb compound was reported recently (Ellermann, Köck & Burzlaff, 1985).

Experimental. Colourless crystals recrystallized from heptane. Spherical crystal, $r = 0{\cdot}21\,(2)$ mm, Philips PW 1100 diffractometer (graphite monochromator, Mo $K\alpha$ radiation), ω–2θ scans, scan width: $(1{\cdot}000 + 0{\cdot}345\tan\theta)^\circ$, 38 reflections ($15{\cdot}6 < \theta < 18{\cdot}3^\circ$) used in least-squares program *GIVER* (Krogmann, 1966) for refinement of lattice parameters; spherical absorption correction applied: $\mu R = 0{\cdot}14\,(1)$, absorption correction factor $A = 1{\cdot}225\,(1)$; max. $(\sin\theta)/\lambda = 0{\cdot}640$ Å^{-1}, index range $h\,0\rightarrow11$, $k\,-10\rightarrow9$, $l\,0\rightarrow19$; six standard reflections used for control of stability (intensity variation: 0·3%), 3354 reflections measured, after averaging equivalents 563 reflections remained [including 5 unobserveds with $I \leq 3\sigma(I)$], $R_{\text{int}} = 0{\cdot}013$; structure solved by Patterson techniques, subsequent Fourier map revealed all C atoms and all H atoms found in a difference Fourier map; least squares based on F and weights $w = 1/\sigma^2(|F_o|)$; final cycles refined

---

* Author for correspondence.

0108-2701/86/121757-03$01.50

scale factor, coordinates, anisotropic temperature factors for non-hydrogen atoms and isotropic for H atoms. $R = 0{\cdot}043$, $wR = 0{\cdot}040$, max. $\Delta/\sigma = 0{\cdot}02$, $\Delta\rho = -0{\cdot}12$ to $0{\cdot}06\ \text{e Å}^{-3}$. Atomic scattering factors taken from *International Tables for X-ray Crystallography* (1974). All calculations were carried out with the program system *CRYSTAN* (Burzlaff, Böhme & Gomm, 1977) and plots with *ORTEP*II (Johnson, 1977).

Discussion. Atomic coordinates and temperature factors are listed in Table 1 and bond lengths and angles in Table 2.* The molecule with atomic-numbering scheme is shown in Fig. 1.

By spectroscopic methods the symmetry of the molecule is indicated to be C_{3v} ($3m$) (Ellermann & Demuth, 1984). In the crystal structure this symmetry is reduced to the subgroup $C_3 \equiv 3$. The molecule is built up of three P atoms in the shape of a regular triangle and the organic group $CH_3C(CH_2)_3$. The P—C [1·851 (2) Å] and C—C (mean value: 1·524 Å) distances are in good agreement with previous results (Hahn, Baudler, Krüger & Tsay, 1982). The P—P distances [2·200 (1) Å] are also close to values previously found for P_3 units: 2·235 Å in P_4S_3 (Leung, Waser, van Houten, Vos, Wiegers & Wiebenga, 1957), 2·25 Å in P_4Se_3 (Keulen & Vos, 1959), 2·202 Å in $P_4(SiMe_2)_3$* (Hönle & von Schnering, 1978), and 2·196 Å in (t-BuP)$_3$ (Hahn *et al.*, 1982). The angles show no significant differences.

In the crystal (Fig. 2) the molecules are oriented along the $\bar{3}$ symmetry axis. At the inversion centre two molecules build up a trigonal P antiprism [P···P: 4·013 (1) Å (see Table 2)] with their P_3 units. Both molecules have three further molecules as nearest neighbours [P···P: 4·304 (1) Å]. This arrangement makes possible a three-dimensional network of the molecules by P···P interactions.

An important difference between the title compound and the structures of the analogous As (Thiele, Zoubek,

Table 1. *Atomic coordinates* (× 10^4) *with estimated standard deviations; equivalent isotropic thermal parameters for* P *and* C *atoms and isotropic thermal parameters for* H *atoms* (Å^2 × 10^2)

$$U_{eq} = 1/(6\pi^2)\ \textstyle\sum_i\sum_j\beta_{ij}(\mathbf{a}_i.\mathbf{a}_j).$$

	x	y	z	U_{eq}/U_{iso}
P	920 (1)	1573 (1)	1237 (1)	8·27
C(1)	1013 (2)	1740 (2)	2437 (1)	6·89
C(2)	0	0	2815 (2)	5·60
C(3)	0	0	3813 (2)	8·60
H(1)	2126 (23)	2233 (22)	2639 (11)	4·58
H(2)	571 (22)	2398 (24)	2618 (12)	4·48
H(3)	−519 (26)	688 (26)	4042 (13)	5·42

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

E.s.d.'s are given in parentheses.

P—P′	2·200 (1)	C(3)—H(3)	1·04 (3)
P—C(1)	1·851 (2)		
C(1)—C(2)	1·521 (2)	P···P	4·013 (1) (i)*
C(2)—C(3)	1·534 (4)	P···P	4·013 (1) (ii)*
C(1)—H(1)	0·95 (2)	P···P	4·304 (1) (iii)*
C(1)—H(2)	0·93 (3)	P···P	4·304 (1) (iv)*
P′—P—P″	60·00	C(1)—C(2)—C(3)	112·5 (2)
P′—P—C(1)	93·68 (5)	P—C(1)—H(1)	111·2 (6)
P″—P—C(1)	93·54 (4)	P—C(1)—H(2)	109·6 (8)
P—C(1)—C(2)	108·4 (1)	H(3)—C(3)—C(2)	110 (1)
C(1)—C(2)—C(1′)	106·3 (1)	H(1)—C(1)—H(2)	108 (2)

Symmetry codes:
(a) Intramolecular
(′) $y-x, -x, z$
(″) $-y, x-y, z.$

(b) Intermolecular
(i) $x-y, x, -z;$
(ii) $y, y-x, -z;$
(iii) $\frac{2}{3}+x-y, \frac{1}{3}+x, \frac{1}{3}-z;$
(iv) $-\frac{1}{3}+y, \frac{1}{3}+y-x, \frac{1}{3}-z.$

* For intermolecular distances see Fig. 2.

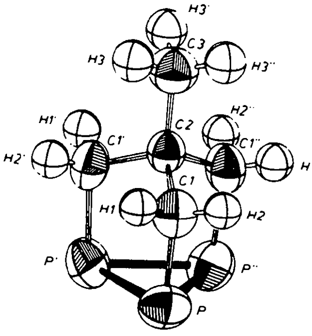

Fig. 1. The $CH_3C(CH_2P)_3$ molecule showing atom labelling and 50% probability thermal ellipsoids.

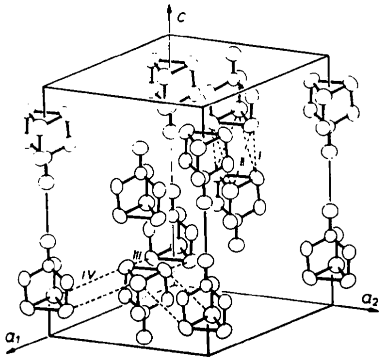

Fig. 2. Molecular packing in the crystal of $CH_3C(CH_2P)_3$; H atoms have been omitted for clarity.

---

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43122 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* (Hexamethyl)trisila(tetraphospha)nortricyclene.

Lindner & Ellermann, 1978) and Sb compounds (Ellermann *et al.*, 1985) is that in these structures the dimeric unit builds up a distorted *E* octahedron (*E* = As, Sb), As···As: 3·88 Å, Sb···Sb: 4·012 (3) Å. In this arrangement the molecules of the dimers have contacts only with two additional molecules [As···As: 4·01 Å, Sb···Sb: 3·966 (2) Å].

We should like to thank Dr A. A. M. Demuth for the preparation of the compound. The work was supported by the Deutsche Forschungsgemeinschaft, the Verband der Chemischen Industrie, and the Fonds der Chemischen Industrie.

References

BURZLAFF, H., BÖHME, R. & GOMM, M. (1977). *CRYSTAN. A Crystallographic Program System for Minicomputers.* Univ. of Erlangen.

ELLERMANN, J. & DEMUTH, A. A. M. (1984). *Angew. Chem. Int. Ed. Engl.* **23**, 805–806.

ELLERMANN, J., KÖCK, E. & BURZLAFF, H. (1985). *Acta Cryst.* C**41**, 1437–1439.

GLEITER, R., KÖPPEL, H. HOFMANN, P., SCHMIDT, H. R. & ELLERMANN, J. (1985). *Inorg. Chem.* **24**, 4020–4023.

HAHN, J., BAUDLER, M., KRÜGER, C. & TSAY, Y.-H. (1982). *Z. Naturforsch. Teil B*, **37**, 797–805.

HÖNLE, W. & VON SCHNERING, H. G. (1978). *Z. Anorg. Allg. Chem.* **440**, 171–182.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JOHNSON, C. K. (1977). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.

KEULEN, E. & VOS, A. (1959). *Acta Cryst.* **12**, 323–329.

KROGMANN, K. (1966). *GIVER. Algol-Programm zur Verfeinerung von Gitterkonstanten und einiger Dinge mehr.* Univ. of Stuttgart.

LEUNG, Y. C., WASER, J., VAN HOUTEN, S., VOS, A., WIEGERS, G. A. & WIEBENGA, E. H. (1957). *Acta Cryst.* **10**, 574–582.

THIELE, G., ZOUBEK, G., LINDNER, H. A. & ELLERMANN, J. (1978). *Angew. Chem. Int. Ed. Engl.* **17**, 135–136.

Acta Cryst. (1986). C**42**, 1759–1762

Structure of Ginkgolide B (BN 52021) Monohydrate, a Highly Specific PAF/Acether Receptor Antagonist Isolated from *Ginkgo biboba* L.

BY L. DUPONT AND O. DIDEBERG

Laboratoire de Cristallographie, Institut de Physique B5, Université de Liège au Sart Tilman, B – 4000 *Liège, Belgium*

G. GERMAIN

Laboratoire de Chimie quantique, Université Catholique de Louvain, Place Pasteur, 1,
B – 1348 *Louvain-la-Neuve, Belgium*

AND P. BRAQUET

IHB – Research Laboratories, Avenue Descartes, 17, *F* – 92350 *Le Plessis-Robinson, France*

(*Received* 10 *December* 1985; *accepted* 29 *May* 1986)

Abstract. 3-(1,1-Dimethylethyl)hexahydro-4,7b,11-trihydroxy-8-methyl-9*H*-1,7a-(epoxymethano)-1*H*,6a*H*-cyclopenta[*c*]furo[2,3-*b*]furo[3′,2′:3,4]cyclopenta[1,2-*d*]-furan-5,9,12(4*H*)-trione monohydrate, $C_{20}H_{24}O_{10}.H_2O$, $M_r = 442{\cdot}4$, triclinic, $P1$, $a = 7{\cdot}627\ (4)$, $b = 11{\cdot}514\ (9)$, $c = 12{\cdot}941\ (8)$ Å, $\alpha = 97{\cdot}05\ (5)$, $\beta = 90{\cdot}27\ (5)$, $\gamma = 108{\cdot}71\ (5)°$, $V = 1067{\cdot}1\ (8)$ Å^3, $Z = 2$, $D_x = 1{\cdot}377$ g cm^{-3}, m.p. 574 K, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 1{\cdot}215$ cm^{-1}, $T = 290$ K, $F(000) = 468$, final $R = 0{\cdot}089$ for 3834 observed reflections. The two independent molecules (but not the H_2O molecules) are related by a ‘best fit’ rotation angle of 179·2 (5)°. The cohesion of the crystal is the result of van der Waals interactions and O–H···O hydrogen bonds involving water molecules.

Introduction. Platelet activating factor (PAF, PAF–acether, AGEPC or APRL) is a naturally occurring etherphospholipid [1-*O*-alkyl-2(*R*)-acetylglyceryl-3-phosphorylcholine] (1) that is generated by specific activation of rabbit basophils (Benveniste, Henson & Cochrane, 1972), human and rabbit polymorphonuclears (PMNLs) (Betz & Henson, 1980), human macrophages (Arnoux, Grimfeld, Duroux & Denjean, 1983) and eosinophils (Ching, Lenihan, Malone, Roddy & Wasserman, 1984). Generation of PAF–acether during anaphylaxis and/or shock leads to chemotaxis, aggregation and degranulation of polymorphonuclear leukocytes, induces bronchoconstriction and contraction of smooth muscle and reduces coronary blood flow and contractile force of

0108-2701/86/121759-04$01.50