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.

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

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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

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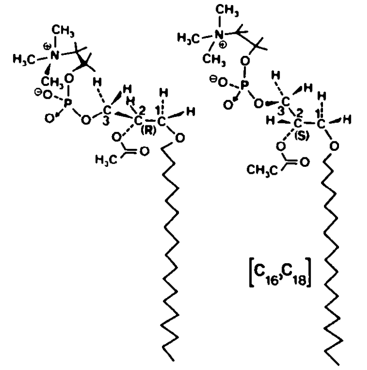
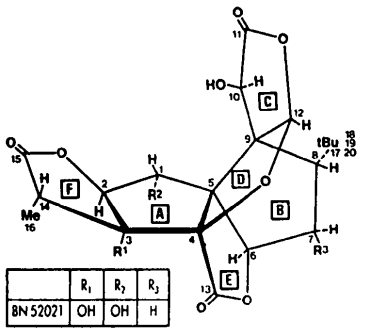
isolated guinea-pig heart. In addition, it induces marked extravasation, hypotension and shock.



(1) PAF–acether.

Ginkgolide B (BN 52021) (2) (Braquet, Spinnewyn, Braquet, Bourgain, Taylor, Etienne & Drieu, 1985) is a highly specific PAF receptor antagonist.



(2) BN 52021.

The first isolation of the terpenoid was achieved from *Ginkgo* by Furukawa (1932). However it was only at the end of the 1960's that the Japanese team of Nakanishi identified its chemical structure (Maruyama, Terahara, Nakadaira, Woods, Takagi & Nakanishi, 1967).

Experimental. Ginkgolide B (BN 52021) was prepared from a standardized extract (GBE 761) prepared from dried and stabilized leaves of *Ginkgo biloba* L. (IHP – IPSEN Research Laboratories, Paris, France). The extraction was performed according to the procedure previously described by Okabe, Yamada, Yamarura & Takada (1967). Further details concerning the isolation, purification and characterization (500 MHz NMR, IR, UV) of BN 52021 have been published elsewhere (Braquet, 1984). BN 52021 was crystallized by slow evaporation of a methanol–acetone–water solution. Crystal: colourless, 0·54 × 0·66 × 0·36 mm; m.p. 574 K.

Cell parameters determined by least-squares refinement from 14 reflections ($12{\cdot}0 < \theta < 15{\cdot}0°$). Siemens diffractometer, 5085 intensities collected ($3 < 2\theta < 55°$), Mo $K\alpha$ radiation with monochromator ($2\theta = 12°$), ω scan mode. 4870 unique reflections; $0 < h < 9$, $-14 < k < 14$, $-16 < l < 16$; $R_{\rm int} = 0{\cdot}03$; 3834 considered as

Table 1. *Fractional coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses and equivalent isotropic temperature factors* (Å^2) *of non-hydrogen atoms*

$B_{\rm eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$. $\sigma(B_{\rm eq}) \sim 0{\cdot}2$ Å^2.
The anisotropic thermal correction $q(hkl) = \exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{23}kl + \beta_{13}hl + \beta_{12}hk)]$.

Atoms C(1),...,C(20), and O(1),...,O(10) refer to molecule *A*; C(21),...,C(40), O(11),...,O(20) are the corresponding atoms of molecule *B*.

	x	y	z	$B_{\rm eq}$
C(1)	44	9	37	3·3
C(2)	1035 (11)	1315 (9)	−216 (6)	3·8
C(3)	−448 (12)	1761 (8)	−684 (6)	3·7
C(4)	−1935 (10)	534 (7)	−1227 (5)	3·0
C(5)	−1438 (9)	−560 (7)	−860 (5)	2·7
C(6)	−737 (12)	−1137 (9)	−1838 (6)	3·9
C(7)	−1981 (14)	−2494 (9)	−2022 (6)	4·1
C(8)	−3891 (11)	−2425 (8)	−1689 (6)	3·2
C(9)	−3391 (9)	−1513 (7)	−665 (5)	2·4
C(10)	−3482 (10)	−2024 (7)	365 (5)	2·7
C(11)	−3686 (9)	−975 (7)	1144 (5)	2·8
C(12)	−4572 (9)	−643 (7)	−479 (6)	2·8
C(13)	−1843 (11)	358 (9)	−2410 (6)	3·5
C(14)	−1054 (13)	2385 (9)	297 (7)	4·0
C(15)	659 (15)	2798 (9)	1000 (7)	4·6
C(16)	−1920 (20)	3403 (11)	171 (9)	5·8
C(17)	−5444 (14)	−3712 (8)	−1711 (7)	4·0
C(18)	−7268 (13)	−3542 (10)	−1343 (9)	5·0
C(19)	−4887 (19)	−4617 (10)	−1126 (9)	5·7
C(20)	−5799 (18)	−4264 (10)	−2878 (8)	5·6
C(21)	6 (9)	−1930 (7)	4813 (6)	2·8
C(22)	−933 (10)	−3267 (8)	4339 (6)	3·4
C(23)	587 (10)	−3734 (7)	3847 (5)	2·8
C(24)	2025 (9)	−2559 (7)	3534 (5)	2·4
C(25)	1526 (8)	−1433 (7)	4040 (5)	2·3
C(26)	851 (10)	−910 (8)	3124 (6)	3·0
C(27)	2039 (12)	397 (8)	3163 (6)	3·4
C(28)	3969 (10)	429 (7)	3568 (5)	2·6
C(29)	3447 (9)	−445 (6)	4436 (5)	2·3
C(30)	3514 (10)	142 (7)	5578 (5)	2·7
C(31)	3721 (9)	−848 (8)	6184 (6)	2·9
C(32)	4656 (9)	−1279 (6)	4508 (5)	2·2
C(33)	1974 (11)	−2467 (7)	2358 (5)	2·9
C(34)	1218 (11)	−4265 (7)	4734 (6)	3·3
C(35)	−509 (14)	−4643 (9)	5365 (7)	4·3
C(36)	2071 (16)	−5306 (9)	4486 (8)	4·5
C(37)	5447 (12)	1695 (7)	3773 (6)	3·2
C(38)	7293 (12)	1600 (8)	4212 (9)	4·5
C(39)	4878 (16)	2664 (8)	4505 (9)	5·0
C(40)	5842 (17)	2212 (9)	2722 (8)	5·2
O(1)	1348 (8)	−628 (7)	90 (5)	4·5
O(2)	339 (9)	2615 (6)	−1395 (5)	4·4
O(3)	−1967 (8)	−2380 (6)	599 (4)	3·6
O(4)	−3286 (9)	−765 (6)	2054 (4)	4·1
O(5)	−4405 (8)	−262 (5)	643 (4)	3·4
O(6)	−3804 (7)	409 (5)	−982 (4)	3·2
O(7)	−1073 (9)	−509 (7)	−2706 (4)	4·4
O(8)	−2377 (11)	878 (7)	−3025 (5)	5·1
O(9)	1041 (13)	3549 (9)	1755 (7)	7·1
O(10)	1819 (9)	2175 (7)	685 (5)	5·0
O(100)	7434 (17)	3199 (10)	7513 (8)	7·9
O(11)	−1281 (7)	−1292 (6)	4931 (5)	3·9
O(12)	−278 (8)	−4628 (5)	2979 (4)	3·8
O(13)	1972 (8)	512 (6)	5824 (4)	3·6
O(14)	3320 (9)	−1028 (7)	7052 (4)	4·2
O(15)	4515 (8)	−1586 (5)	5589 (4)	3·2
O(16)	3900 (6)	−2385 (4)	3841 (4)	2·6
O(17)	1210 (8)	−1629 (6)	2167 (4)	3·5
O(18)	2558 (10)	−3050 (7)	1697 (5)	4·6
O(19)	−822 (14)	−5328 (8)	6008 (7)	6·7
O(20)	−1710 (9)	−4052 (6)	5113 (5)	4·6
O(200)	3395 (20)	2175 (14)	7487 (12)	13·0

observed $[I > 2{\cdot}5\sigma(I)]$. Standard reflections: $\bar{5}40$ $(1373 < F_o < 1485)$ and 004 $(875 < F_o < 965)$. Absorption corrections by method of North, Phillips & Mathews (1968): minimum and maximum coefficients, 0·867 and 0·996; structure solved by Patterson method with *SHELXS*84 and *PATSEE* (Egert & Sheldrick, 1985) and *DIRDIF* (Beurskens, Bosman, Doesburg, Gould, van den Hark & Prick, 1980). Starting model built with *EUCLID* (Essen, 1983) using distances and angles measuring on a Dreiding stereomodel. Because of the large number of parameters (585), refinement on F, blocked diagonal matrix approximation, one block (9 x 9) per atom, with *NRC*10 (Ahmed, 1970). Anisotropic temperature factors for all non-H atoms, H atoms at calculated positions. Two of the six H (OH) positions and those of H water were estimated from difference map and hydrogen-bond considerations. H(O1) and H(O2) could not be placed. Final conventional $R(F)$ 0·089, wR 0·118, $w = 1/\sigma^2(F_o)$ $[\sigma(F_o)$ based on counting statistics]. This relatively high value of R is possibly the result of the poor quality of the crystals. In final cycle of least-squares refinement, max. $\Delta/\sigma < 0{\cdot}7$, maximum and minimum heights in final ΔF synthesis +0·4 and −0·4 e Å^{-3}; atomic scattering factors taken from *International Tables for X-ray Crystallography* (1962).

Table 2. *Bond lengths* (Å) *with e.s.d.'s in parentheses*

	Molecule *A*	Molecule *B*
C(2)–C(1)	1·527 (10)	1·522 (12)
C(5)–C(1)	1·543 (7)	1·549 (10)
O(1)–C(1)	1·418 (7)	1·400 (10)
C(3)–C(2)	1·534 (13)	1·538 (11)
O(10)–C(2)	1·424 (11)	1·433 (11)
C(4)–C(3)	1·580 (12)	1·544 (10)
C(14)–C(3)	1·529 (12)	1·510 (11)
O(2)–C(3)	1·422 (11)	1·424 (9)
C(5)–C(4)	1·511 (11)	1·538 (10)
C(13)–C(4)	1·525 (10)	1·539 (10)
O(6)–C(4)	1·427 (10)	1·427 (9)
C(6)–C(5)	1·535 (11)	1·550 (10)
C(9)–C(5)	1·584 (10)	1·576 (10)
C(7)–C(6)	1·534 (14)	1·481 (12)
O(7)–C(6)	1·472 (11)	1·482 (9)
C(8)–C(7)	1·545 (14)	1·548 (12)
C(9)–C(8)	1·550 (10)	1·568 (10)
C(17)–C(8)	1·569 (13)	1·521 (11)
C(10)–C(9)	1·515 (10)	1·541 (9)
C(12)–C(9)	1·547 (10)	1·539 (10)
C(11)–C(10)	1·523 (10)	1·507 (11)
O(3)–C(10)	1·388 (10)	1·399 (10)
O(4)–C(11)	1·191 (9)	1·191 (10)
O(5)–C(11)	1·346 (10)	1·362 (10)
O(5)–C(12)	1·457 (9)	1·481 (8)
O(6)–C(12)	1·405 (9)	1·398 (8)
O(7)–C(13)	1·332 (12)	1·323 (10)
O(8)–C(13)	1·196 (11)	1·198 (10)
C(15)–C(14)	1·498 (15)	1·523 (13)
C(16)–C(14)	1·542 (17)	1·540 (14)
O(9)–C(15)	1·189 (13)	1·190 (13)
O(10)–C(15)	1·343 (13)	1·364 (13)
C(18)–C(17)	1·535 (15)	1·557 (13)
C(19)–C(17)	1·521 (15)	1·545 (14)
C(20)–C(17)	1·552 (14)	1·544 (13)

Discussion. The atomic parameters are listed in Table 1.* The atom numbering is defined in formula (2) and Fig. 1. O(100) and O(200) belong to water molecules. There are no significant differences between the two independent molecules *A* (Fig. 1) and *B*. The 'best' rotation to relate the two sets of coordinates is 179·2 (5)° (calculated with *BMFIT*: Nyburg, 1980), but the water molecule positions are not consistent with this pseudo twofold rotation. The possibility of a higher symmetry than *P*1 has been eliminated after careful investigations. Bond lengths (Table 2) and angles are normal. Cremer–Pople parameters calculated with the program *PUCK*2 (Luger & Bülow, 1983) show that rings *A* and *B* have a twist (or half-chair) conformation, ring *C* is intermediate between twist and envelope, and rings *D*, *E* and *F* have envelope conformations. The most flexible ring (*B*) exhibits the largest puckering amplitude deviations (ΔQ) between molecules *A* and *B*. Some torsion angles (°) of rings *A* and *B* may be compared with the NMR data of Perly & Foldu (1986):

	Molcule *A*	Molecule *B*	NMR
H(1α)–C(1)–C(2)–H(2)	154	152	120
H(6)–C(6)–C(7)–H(7β)	−81	−83	−90
H(7α)–C(7)–C(8)–H(8)	−168	−163	−170
H(7β)–C(7)–C(8)–H(8)	−48	−42	−50
H(6)–C(6)–C(7)–H(7α)	42	38	30
H(7α)–C(7)–C(8)–C(17)	−56	−56	−50
H(7β)–C(7)–C(8)–C(17)	64	65	70

The cohesion of the crystal is a result of van der Waals interactions and O—H···O hydrogen bonds involving water molecules: O(2)···H—O(200) $(x,\ y,\ -1+z) = 2{\cdot}898\ (17)$ Å, O(2)—H···O(100) $(-1+x,\ y,\ -1+z) = 2{\cdot}919\ (14)$ Å, O(3)—H···O(17) = 2·985 (14) Å, O(8)···H—O(100) $(-1+x,\ y,\ -1+z) = 2{\cdot}727\ (13)$ Å, O(100)—H···O(19) $(1+x,\ 1+y,\ z) = 2{\cdot}796\ (14)$ Å, O(200)—H···O(13) = 2·666 (13) Å.

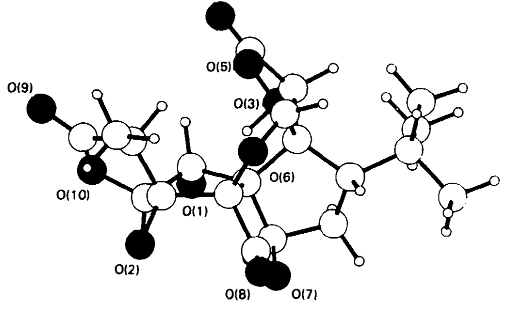


Fig. 1. Structure of molecule *A*. Atoms H(O1) and H(O2) could not be placed.

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* Lists of structure factors, bond angles, anisotropic thermal parameters, H-atom positions, Cremer–Pople parameters, bond angles and selected torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43120 (45 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The molecular structure of BN 52021 provides a rigid model which can serve as reference for comparison of PAF–acether and specific antagonist structures.

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Structure of an Intermediate in the Synthesis of C_{20}-Gibberellins

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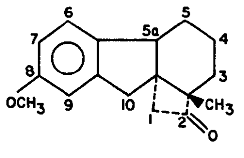
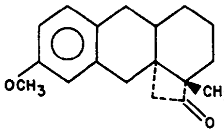
Abstract. 8-Methoxy-2aβ-methyl-2a,3,4,5,5aβ,10-hexahydro-1*H*-cyclobuta[*j*]fluoren-2-one, $C_{17}H_{20}O_2$, $M_r = 256{\cdot}34$, orthorhombic, *Pbca*, $a = 7{\cdot}757$ (2), $b = 17{\cdot}005$ (3), $c = 21{\cdot}321$ (7) Å, $V = 2812{\cdot}40$ (13) Å^3, $Z = 8$, $D_x = 1{\cdot}210$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu(\text{Cu } K\alpha) = 6{\cdot}21$ cm^{-1}, $F(000) = 1104$, $T = 293$ K, $R = 0{\cdot}041$ for 1669 reflections. The aromatic ring is planar and the cyclohexane ring adopts a distorted chair conformation. The five-membered ring is in an envelope conformation and the cyclobutanone ring has an angle of 157·0° between the planes through C(9), C(17), C(16) and C(9), C(14), C(16). Both the ring junctions *B–C* and *C–D* are *cis*-fused.

Introduction. The title compound (I), an important intermediate in the total synthesis of C_{20}-gibberellins (Ghatak, Ghosh & Sanyal, 1980), was obtained by stereoselective catalytic hydrogenation of unsaturated hydrocyclobutafluorenone *via* an acid-induced decomposition of a rigid polycyclic $\beta\gamma$-unsaturated diazomethyl ketone. The structure determination of this highly rigid polycyclic ketone (I) was undertaken in order to confirm its structure and stereochemistry which had been assigned by analogy with the corresponding hydrophenanthrene (II) of established stereochemistry.



(I)



(II)

Experimental. Transparent colourless plate-like crystal obtained by crystallization from petroleum ether solution, crystal dimensions: 0·2 × 0·3 × 0·3 mm. Syntex $P2_1$ four-circle diffractometer, unit-cell parameters by least-squares calculations from angular setting of 15 independent reflections, $9 < \theta < 16°$. 1885 unique reflections measured, $2\theta \leq 114°$, $8 \geq h \geq 0$, $18 \geq k \geq 0$, $23 \geq l \geq 0$, graphite-monochromatized Cu $K\alpha$ radiation, ω-scan technique, no

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