O3—C15	1.209 (2)	C6—C7	1.544 (3)
O4—C15	1.336 (2)	C8—C9	1.487 (2)
O4—C16	1.443 (2)	C9—C10	1.394 (2)
N—C1	1.470 (2)	C9—C14	1.383 (2)
N—C5	1.465 (2)	C10—C11	1.381 (3)
C1—C2	1.551 (2)	C11—C12	1.384 (3)
C1—C7	1.530 (2)	C12—C13	1.384 (3)
C2—C3	1.533 (2)	C13—C14	1.388 (3)
C2—C15	1.504 (2)		
C3—O1—C8	116.8 (1)	C5—C6—C7	103.9 (1)
C15—O4—C16	116.1 (1)	C1—C7—C6	104.1 (1)
C1—N—C5	102.2 (1)	O1—C8—O2	123.7 (2)
N—C1—C2	113.2 (1)	O1—C8—C9	112.0 (1)
N—C1—C7	101.2 (1)	O2—C8—C9	124.3 (1)
C2—C1—C7	111.0 (1)	C8—C9—C10	118.0 (1)
C1—C2—C3	109.4 (1)	C8—C9—C14	122.0 (1)
C1—C2—C15	111.0 (1)	C10—C9—C14	120.0 (2)
C3—C2—C15	111.2 (1)	C9—C10—C11	119.9 (2)
O1—C3—C2	106.7 (1)	C10—C11—C12	120.2 (2)
O1—C3—C4	109.6 (1)	C11—C12—C13	119.8 (2)
C2—C3—C4	113.7 (1)	C12—C13—C14	120.4 (2)
C3—C4—C5	109.9 (1)	C9—C14—C13	119.7 (2)
N—C5—C4	110.9 (1)	O3—C15—O4	122.9 (2)
N—C5—C6	101.7 (1)	O3—C15—C2	125.7 (2)
C4—C5—C6	112.6 (1)	O4—C15—C2	111.4 (1)

Data were processed and refined using the *MolEN* software package (Fair, 1990). The structure was solved using the *MULTAN*11/82 series of programs (Main *et al.*, 1982).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: LI1093). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A New Crystal Form of Methyl Bacteriopheophorbide *a*

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Abstract

The structure of a new crystal form of methyl bacteriopheophorbide *a*, $C_{36}H_{40}N_4O_6$, which does not contain benzene molecules of crystallization, has been determined at 200 K. The molecule is slightly ruffled with maximum displacements from planarity of ± 0.21 Å in all five rings. The crystal packing is dominated by π–π interactions between pyrrole rings I and III of adjacent molecules, which are separated by 3.58 Å.

Comment

Bacteriochlorophylls (BChls) and their demetallated analogs, bacteriopheophytins (BPheos), are prosthetic groups of antenna (Tronrud, Schmid & Matthews, 1986) and reaction-center (Allen, Feher, Yeates, Komiya & Rees, 1988; Deisenhofer & Michel, 1989; El-Kabbani, Chang, Tiede, Norris & Schiffer, 1991) proteins, whose structures have been determined by X-ray methods. The BChls in antenna complexes harvest light into reaction centers where a network of BChls and BPheos effect the primary charge separation which eventually drives the biochemistry of the organisms; for a review of this subject see Kirmaier & Holten (1987). Recently, we reported a high-precision structure (Barkigia, Gottfried, Boxer & Fajer, 1989) of methyl bacteriopheophorbide *a* (MeBPheo), a molecule lacking only the phytyl chain of BPheo, that was crystallized from CH_2Cl_2/benzene and consequently contained benzene of solvation. Since structural data clearly demonstrate that crystal packing as well as steric and protein constraints (Deisenhofer, Epp, Miki, Huber & Michel, 1984, 1985; Barkigia, Fajer, Chang & Young, 1984; Waditschatka, Kratky, Jaun, Heinzer & Eschenmoser, 1985; Tronrud *et al.*, 1986; Michel, Epp & Deisenhofer, 1986; Scheidt & Lee, 1987) cause porphyrin conformational variations which in turn modulate redox, optical and charge-transfer prop-

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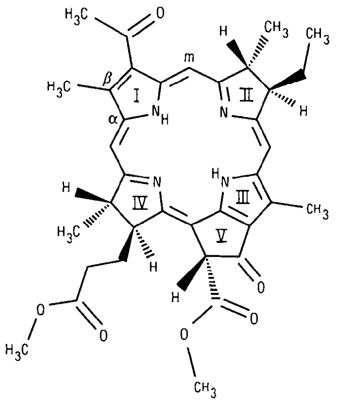
erties (Barkigia, Chantranupong, Smith & Fajer, 1988; Forman *et al.*, 1989; Barkigia *et al.*, 1989, 1993; Fajer *et al.*, 1990; Barkigia & Fajer, 1993) *in vivo* and *in vitro*, we attempted to prepare different crystal forms of MeBPheo in a search for different conformers.



(1)

The present sample of the title compound (1) was obtained from mixtures of CH_2Cl_2/methanol and does not occlude a solvent. The existence of this form was suggested by powder diffraction data (Gottfried & Boxer, 1992). Its molecular structure (Fig. 1) is typical of a bacteriochlorin (Barkigia & Fajer, 1993, and references therein) with elongated Cα—Cβ and Cβ—Cβ bonds in the pyrroline rings (II and IV) compared with those in the pyrrole rings (I and III). Unlike the other polymorph, the central H atoms were clearly located on rings I and III from a difference Fourier map.

As in other hydroporphyrins, the distances from the center of the molecule (Ct) to the N atoms are longer for the reduced rings (2.116 and 2.161 Å for Ct···N2 and Ct···N4, *cf* 2.064 and 2.001 Å for Ct···N1 and Ct···N3) and are indicative of an expanded core (Barkigia *et al.*, 1984, 1992; Chang, Barkigia, Hanson & Fajer, 1986). In general, the distances are symmetric about the N1···N3 direction with steric perturbations, imposed by ring V, in the vicinity of ring III. Likewise, the bonds in ring V are quite asymmetrical, probably as a result of the partial conjugation of the O1 keto group with the porphyrin. The C13—C28 bond is short [1.436 (14) Å] whereas C15—C29 is long and C28—C29 exceptionally long [1.518 (12) and 1.578 (12) Å, respectively], but consonant with values observed in all other chlorophyll derivatives (Chow, Serlin & Strouse, 1975; Serlin, Chow & Strouse, 1975; Kratky & Dunitz, 1975, 1977; Kratky, Isenring & Dunitz, 1977; Senge & Smith, 1992). The good internal consistency between the four C=O bond lengths clearly establishes that the C28—O1 bond [1.233 (10) Å] is keto rather than enol in nature.

The effect of saturation in rings II and IV is also evident from the bond angles which display fairly good agreement across the N1···N3 axis, except adjacent to ring III. The average Cα–Cβ–Cβ angles of 102.1 (8)° in rings II and IV are significantly smaller than the average of 108.1 (8)° in rings I and III, as noted previously (Barkigia & Fajer, 1993, and references therein). Unlike metallohydroporphyrins whose Cα—N—Cα angles are wider in the reduced rings, the breadth of the Cα—N—Cα angles seems to be controlled by the positions of the central H atoms. This applies not only here, but also in free-base porphyrin and chlorins (Hoard, 1975; Barkigia *et al.*, 1992). The angles are more obtuse in rings I and III [112.2 (8), 109.9 (7)°] than in rings II and IV [107.8 (8), 106.4 (8)°]. This effect is counteracted by smaller N—Cα—Cβ angles [mean values in the pyrrole and pyrroline rings of 106.5 (8) and 113.9 (8)°, respectively]. The N—Cα—Cm and N—Cα—Cβ angles around ring III are grossly different, presumably for the same reasons as the asymmetry in the distances.

Fig. 2 presents a comparison of the displacements of the atoms of the macrocycle from the plane of the N

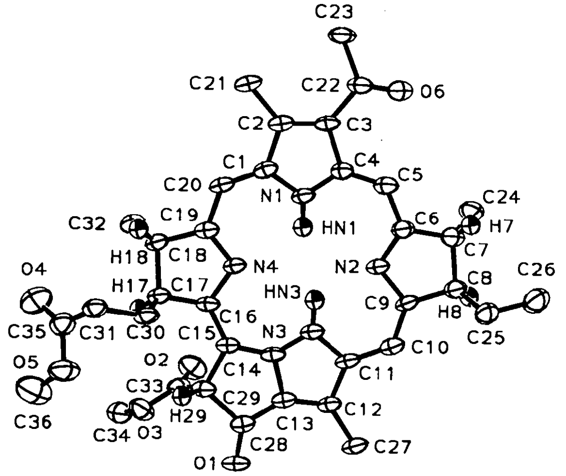


Fig. 1. Molecular structure of MeBPheo at 200 K. Probability ellipsoids are drawn at 50%, except for the H atoms which are not to scale. H atoms at the *meso* positions and on the side chains are omitted for clarity.

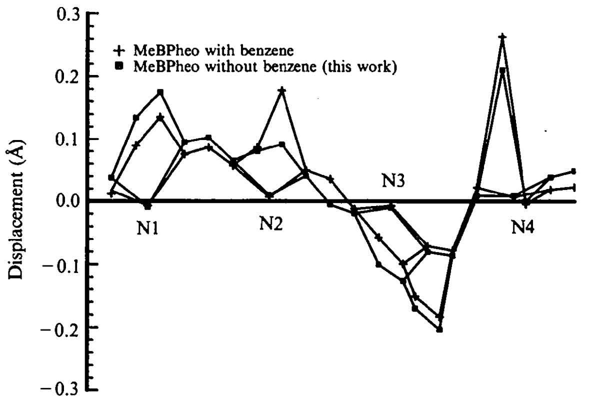


Fig. 2. Linear display of the displacements of the atoms of the macrocycle from the plane of the four N atoms for the two forms of MeBPheo. The ring order is I, II, III, V and IV from left to right (the horizontal axis is not to scale).

atoms in the two forms of MeBPheo. Their conformations are quite similar, except at C8 which deviates by 0.091 (10) Å in the present structure and by 0.178 Å in the other form. The maximum displacements occur at C17 in both structures and the orientation of the acetyl group [as judged by the C2—C3—C22—O6 torsion angle of 159 (1)°] is identical. When considered individually, rings I, II and III are flat while ring IV is twisted about C17—C18 [C16—C17—C18—C19 torsion angle −13 (1)°].

Although the *c* axis shrinks from ~17 Å in the solvated form (Barkigia *et al.*, 1989; Barkigia, Fajer, Smith & Williams, 1981) to ~15 Å in this structure, as was predicted from powder diffraction studies (Gottfried & Boxer, 1992), the same molecular aggregation is maintained. Rings I and III of successive molecules adhere through π–π contacts with perpendicular separations of 3.58 Å and center-to-center distances of 8.06 Å. Adjacent layers are 7.17 Å apart.

Experimental

Crystal data

$C_{36}H_{40}N_4O_6$
$M_r = 624.74$
Triclinic
$P1$
$a = 7.170\ (4)$ Å
$b = 8.057\ (2)$ Å
$c = 14.953\ (14)$ Å
$\alpha = 84.87\ (4)°$
$\beta = 89.04\ (6)°$
$\gamma = 69.85\ (4)°$
$V = 807.5\ (12)$ Å^3
$Z = 1$
$D_x = 1.29$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 9$–$21°$
$\mu = 0.678$ mm^{-1}
$T = 200$ K
Thin plate
0.32 × 0.31 × 0.10 mm
Blackish green

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: analytical
$T_{\min} = 0.814$, $T_{\max} = 0.938$
3451 measured reflections
3451 independent reflections

2998 observed reflections $[F_o > 3\sigma(F_o)]$
$\theta_{\max} = 75°$
$h = 0 \rightarrow 8$
$k = -9 \rightarrow 10$
$l = -18 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity variation: 1.1%

Refinement

Refinement on F
$R = 0.076$
$wR = 0.092$
$S = 1.85$
2998 reflections
412 parameters
All H-atom parameters included but not refined
$w = 4F_o^2/[\sigma^2(F_o^2)+(0.05F^2)^2]$

$(\Delta/\sigma)_{\max} < 0.01$
$\Delta\rho_{\max} = 0.44$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
O1	0.254 (1)	0.5456 (7)	−0.0020 (5)	4.1 (1)
O2	0.143 (1)	0.1966 (8)	−0.0992 (5)	4.9 (2)
O3	0.308 (1)	0.3349 (8)	−0.1893 (5)	4.5 (2)
O4	0.878 (1)	−0.1467 (9)	−0.3198 (6)	7.0 (2)
O5	0.839 (1)	0.1175 (8)	−0.2720 (5)	5.1 (2)
O6	0.814 (1)	−0.9057 (8)	0.4281 (5)	4.5 (2)
N1	0.661	−0.510	0.208	2.9 (1)
N2	0.445 (1)	−0.2183 (7)	0.3308 (5)	2.9 (1)
N3	0.416 (1)	0.0288 (7)	0.1871 (5)	2.9 (1)
N4	0.634 (1)	−0.2451 (7)	0.0580 (5)	3.0 (1)
C1	0.761 (1)	−0.6174 (9)	0.1444 (6)	2.9 (2)
C2	0.832 (1)	−0.7948 (9)	0.1881 (6)	2.9 (2)
C3	0.778 (1)	−0.7882 (9)	0.2760 (6)	2.9 (2)
C4	0.667 (1)	−0.6021 (9)	0.2905 (6)	2.8 (2)
C5	0.587 (1)	−0.5384 (9)	0.3694 (6)	3.3 (2)
C6	0.484 (1)	−0.362 (1)	0.3893 (6)	3.3 (2)
C7	0.408 (1)	−0.323 (1)	0.4823 (6)	3.7 (2)
C8	0.316 (1)	−0.118 (1)	0.4742 (6)	3.2 (2)
C9	0.349 (1)	−0.0700 (9)	0.3770 (6)	3.1 (2)
C10	0.287 (1)	0.100 (1)	0.3364 (6)	3.2 (2)
C11	0.318 (1)	0.1517 (9)	0.2467 (6)	3.0 (2)
C12	0.255 (1)	0.3236 (9)	0.1981 (6)	3.0 (2)
C13	0.320 (1)	0.2971 (9)	0.1114 (6)	3.1 (2)
C14	0.419 (1)	0.1134 (9)	0.1043 (6)	2.8 (2)
C15	0.494 (1)	0.0685 (9)	0.0203 (6)	2.8 (2)
C16	0.604 (1)	−0.0991 (9)	−0.0043 (6)	2.9 (2)
C17	0.710 (1)	−0.1421 (9)	−0.0905 (6)	2.9 (2)
C18	0.773 (1)	−0.3495 (9)	−0.0823 (6)	3.0 (2)
C19	0.735 (1)	−0.3889 (9)	0.0155 (6)	3.0 (2)
C20	0.793 (1)	−0.5645 (9)	0.0577 (6)	3.1 (2)
C21	0.952 (1)	−0.9523 (9)	0.1390 (6)	3.4 (2)
C22	0.821 (1)	−0.9306 (9)	0.3502 (6)	3.5 (2)
C23	0.876 (2)	−1.121 (1)	0.3248 (7)	4.3 (2)
C24	0.263 (2)	−0.413 (1)	0.5148 (7)	5.8 (3)
C25	0.421 (1)	−0.036 (1)	0.5410 (6)	3.9 (2)
C26	0.378 (2)	−0.078 (1)	0.6375 (7)	4.9 (2)
C27	0.143 (1)	0.4948 (9)	0.2361 (6)	3.7 (2)
C28	0.326 (1)	0.3869 (9)	0.0247 (6)	3.1 (2)
C29	0.434 (1)	0.2403 (9)	−0.0405 (6)	3.2 (2)
C30	0.892 (1)	−0.0872 (9)	−0.0935 (6)	3.3 (2)
C31	1.020 (1)	−0.131 (1)	−0.1781 (6)	3.5 (2)
C32	0.651 (2)	−0.412 (1)	−0.1471 (7)	4.3 (2)
C33	0.281 (1)	0.250 (1)	−0.1123 (6)	3.7 (2)
C34	0.158 (2)	0.369 (1)	−0.2574 (7)	4.9 (3)
C35	0.912 (1)	−0.062 (1)	−0.2612 (7)	4.0 (2)
C36	0.733 (2)	0.203 (1)	−0.3518 (9)	7.1 (3)

Table 2. *Selected geometric parameters* (Å, °)

N1—C1	1.369 (11)	C2—C3	1.364 (14)
N1—C4	1.375 (11)	C7—C8	1.549 (13)
N2—C6	1.340 (12)	C12—C13	1.379 (14)
N2—C9	1.394 (10)	C17—C18	1.566 (11)
N3—C11	1.389 (12)	C4—C5	1.367 (13)
N3—C14	1.362 (12)	C5—C6	1.411 (12)
N4—C16	1.391 (11)	C9—C10	1.373 (13)
N4—C19	1.345 (11)	C10—C11	1.405 (14)
C1—C2	1.440 (12)	C14—C15	1.387 (14)
C3—C4	1.469 (12)	C15—C16	1.387 (11)
C6—C7	1.506 (14)	C19—C20	1.420 (12)
C8—C9	1.506 (14)	C20—C1	1.369 (13)
C11—C12	1.432 (13)	C13—C28	1.436 (14)
C13—C14	1.417 (11)	C15—C29	1.518 (12)
C16—C17	1.491 (13)	C28—C29	1.578 (12)
C18—C19	1.510 (13)	C28—O1	1.233 (10)
C1—N1—C4	112.2 (8)	C4—C5—C6	129.9 (9)
C6—N2—C9	107.8 (8)	C9—C10—C11	126.5 (8)
C11—N3—C14	109.9 (7)	C14—C15—C16	127.4 (8)
C16—N4—C19	106.4 (8)	C19—C20—C1	127.8 (8)
N1—C1—C2	106.2 (8)	C1—C2—C3	108.7 (8)
N1—C4—C3	105.1 (8)	C2—C3—C4	107.9 (8)

N2—C6—C7	114.0 (8)	C6—C7—C8	103.0 (8)
N2—C9—C8	112.9 (8)	C7—C8—C9	102.3 (8)
N3—C11—C12	107.5 (9)	C11—C12—C13	106.2 (8)
N3—C14—C13	107.0 (8)	C12—C13—C14	109.5 (8)
N4—C16—C17	114.2 (7)	C16—C17—C18	101.3 (7)
N4—C19—C18	114.5 (7)	C17—C18—C19	101.8 (7)
N1—C1—C20	126.4 (8)	C2—C1—C20	127.4 (8)
N1—C4—C5	128.4 (8)	C3—C4—C5	126.5 (9)
N2—C6—C5	125.2 (9)	C5—C6—C7	120.8 (9)
N2—C9—C10	122.9 (9)	C8—C9—C10	124.3 (8)
N3—C11—C10	121.9 (8)	C10—C11—C12	130.5 (8)
N3—C14—C15	137.6 (8)	C13—C14—C15	115.4 (8)
N4—C16—C15	118.9 (8)	C15—C16—C17	126.8 (8)
N4—C19—C20	123.0 (9)	C18—C19—C20	122.4 (8)

The data do not discriminate between enantiomorphs. The enantiomorph was chosen to be consistent with all other known chlorophyll and bacteriochlorophyll structures. The coordinates of N1 were fixed to define the origin.

Data collection: CAD-4 (Enraf-Nonius, 1977). Cell refinement: CAD-4. Data reduction: *MolEN* (Fair, 1990). Structure solution: *MULTAN*80 (Main *et al.*, 1980). Molecular graphics: *ORTEP*II (Johnson, 1976) and *GENPLOT* (Computer Graphics Service, 1989).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1097). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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8-Chloro-3,6-dioxaoctyl (*E*,*E*)-2-Chloro-3-(3,5-dichloro-2-hydroxyphenylazo)-2-butenoate

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Abstract

The title molecule, $C_{16}H_{18}Cl_4N_2O_5$, shows an *E*,*E* configuration. An intramolecular three-center hydrogen bond is formed and stacking between the molecules indicates the presence of weak π-electron interactions.

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