Table 2. *Bond lengths* (Å), *bond angles* (°) *and intermolecular distances* ≤3.6 Å *with e.s.d.'s in parentheses*

| | | | |
|---|---|---|---|
| O—C(7) | 1.225 (2) | N(4)—C(13) | 1.470 (3) |
| N(1)—N(3) | 1.398 (2) | C(1)—C(2) | 1.386 (2) |
| N(1)—C(1) | 1.417 (2) | C(1)—C(6) | 1.387 (3) |
| N(1)—C(7) | 1.401 (3) | C(2)—C(3) | 1.384 (2) |
| N(2)—C(7) | 1.378 (2) | C(3)—C(4) | 1.385 (3) |
| N(2)—C(8) | 1.315 (2) | C(4)—C(5) | 1.379 (3) |
| N(3)—C(8) | 1.339 (2) | C(5)—C(6) | 1.382 (2) |
| N(3)—C(12) | 1.471 (3) | C(8)—C(9) | 1.486 (2) |
| N(4)—C(10) | 1.464 (3) | C(9)—C(10) | 1.526 (3) |
| N(4)—C(11) | 1.460 (2) | C(11)—C(12) | 1.511 (3) |

| | | | |
|---|---|---|---|
| C(1)—N(1)—N(3) | 120.9 (1) | C(2)—C(3)—C(4) | 120.5 (2) |
| C(1)—N(1)—C(7) | 125.6 (1) | C(3)—C(4)—C(5) | 119.4 (2) |
| N(3)—N(1)—C(7) | 106.1 (1) | C(4)—C(5)—C(6) | 120.9 (2) |
| C(7)—N(2)—C(8) | 106.1 (2) | C(5)—C(6)—C(1) | 119.3 (2) |
| N(1)—N(3)—C(8) | 105.6 (1) | O—C(7)—N(1) | 123.9 (1) |
| N(1)—N(3)—C(12) | 121.0 (1) | O—C(7)—N(2) | 128.0 (2) |
| C(8)—N(3)—C(12) | 124.8 (1) | N(1)—C(7)—N(2) | 108.1 (1) |
| C(10)—N(4)—C(11) | 113.0 (1) | N(2)—C(8)—N(3) | 113.6 (1) |
| C(10)—N(4)—C(13) | 109.6 (2) | N(2)—C(8)—C(9) | 125.6 (2) |
| C(11)—N(4)—C(13) | 109.2 (1) | N(3)—C(8)—C(9) | 120.8 (2) |
| N(1)—C(1)—C(2) | 118.7 (2) | C(8)—C(9)—C(10) | 115.7 (1) |
| N(1)—C(1)—C(6) | 120.9 (1) | N(4)—C(10)—C(9) | 113.8 (2) |
| C(2)—C(1)—C(6) | 120.3 (1) | N(4)—C(11)—C(12) | 114.3 (2) |
| C(1)—C(2)—C(3) | 119.5 (2) | N(3)—C(12)—C(11) | 111.6 (2) |

| Atom in molecule 1 at *x*, *y*, *z* | Atom in molecule 2 | Distance (Å) | Molecule 2 at |
|---|---|---|---|
| N(2) | C(6) | 3.354 (2) | $-x, -y, 1-z$ |
| O | C(9) | 3.371 (2) | $1-x, -y, 1-z$ |
| C(7) | C(8) | 3.372 (2) | $1-x, -y, 1-z$ |
| O | C(3) | 3.429 (2)* | $-x, -1-y, -z$ |
| N(1) | N(2) | 3.436 (1) | $1-x, -y, 1-z$ |
| N(2) | C(7) | 3.487 (2) | $1-x, -y, 1-z$ |
| O | C(11) | 3.520 (2)* | $x, -1+y, z$ |
| C(6) | C(8) | 3.530 (2) | $-x, -y, 1-z$ |
| O | C(8) | 3.543 (2) | $1-x, -y, 1-z$ |
| N(4) | C(4) | 3.543 (2)* | $1+x, 1+y, 1+z$ |
| N(2) | N(2) | 3.589 (1) | $1-x, -y, 1-z$ |

* Intercolumnar distance.

≤3.6 Å are given in Table 2. Fig. 1 shows the atom-numbering scheme of the molecule. The crystal packing is illustrated in Fig. 2.

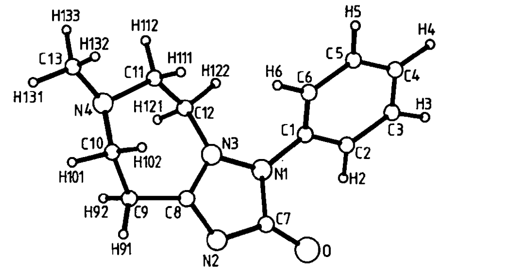


Fig. 1. Perspective view of the molecule showing the atom-numbering scheme. Radii are arbitrary.

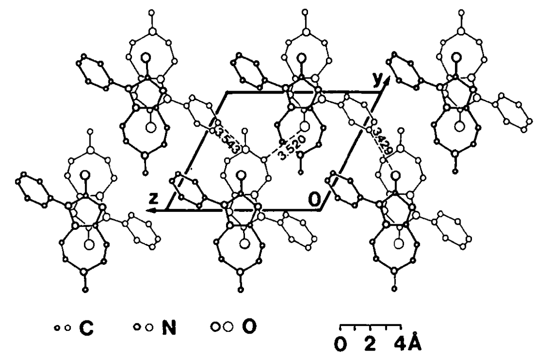


Fig. 2. Packing plot projected down *x*. H atoms omitted for clarity.

Support for this work by the Fonds zur Förderung der wissenschaftlichen Forschung through grants P 2718 and P 6804-CH is gratefully acknowledged.

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*Acta Cryst.* (1992). C**48**, 581–583

# 8,12-Diethyl-2,3,7,13,17,18-hexamethyl-20-phenylporphyrin

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(*Received* 10 *December* 1990; *accepted* 27 *August* 1991)

**Abstract.** $C_{36}H_{38}N_4$, $M_r = 526.7$, orthorhombic, *Iba*2, $a = 12.870$ (6), $b = 55.71$ (3), $c = 7.837$ (2) Å, $V = 5619$ (4) Å$^3$, $Z = 8$, $D_x = 1.245$ Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.069$ mm$^{-1}$, $F(000) = 2256$, $T = 130$ K, $R = 0.076$ for 1428 independent reflections [$I > 2\sigma(I)$]. The title compound consists of the slightly

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0108-2701/92/030581-03$03.00 

Table 1. *Atomic coordinates and isotropic temperature factors* ($Å^2 \times 10^3$)

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

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| N(21) | 0.3468 (5) | 0.1484 (1) | 0.4742 | 27 (3) |
| N(22) | 0.3986 (6) | 0.1000 (1) | 0.6420 (16) | 23 (3) |
| N(23) | 0.1868 (6) | 0.0905 (1) | 0.6579 (16) | 28 (3) |
| N(24) | 0.1352 (6) | 0.1382 (1) | 0.4894 (17) | 24 (3) |
| C(1) | 0.3151 (7) | 0.1699 (2) | 0.4108 (19) | 23 (3) |
| C(2) | 0.4064 (8) | 0.1848 (2) | 0.3752 (19) | 28 (3) |
| C(21) | 0.4125 (7) | 0.2103 (2) | 0.3027 (19) | 36 (3) |
| C(3) | 0.4893 (8) | 0.1713 (2) | 0.4126 (19) | 30 (3) |
| C(31) | 0.6018 (7) | 0.1772 (2) | 0.3887 (20) | 34 (3) |
| C(4) | 0.4516 (7) | 0.1485 (2) | 0.4759 (20) | 24 (4) |
| C(5) | 0.5181 (8) | 0.1312 (2) | 0.5375 (18) | 28 (3) |
| C(6) | 0.4982 (9) | 0.1094 (2) | 0.6151 (18) | 25 (3) |
| C(7) | 0.5698 (7) | 0.0925 (2) | 0.6819 (19) | 26 (3) |
| C(71) | 0.6845 (7) | 0.0963 (2) | 0.6862 (20) | 36 (3) |
| C(8) | 0.5171 (8) | 0.0734 (2) | 0.7489 (19) | 28 (3) |
| C(81) | 0.5608 (8) | 0.0519 (2) | 0.8345 (20) | 36 (3) |
| C(82) | 0.5890 (10) | 0.0311 (2) | 0.7228 (23) | 70 (4) |
| C(9) | 0.4085 (7) | 0.0784 (2) | 0.7193 (18) | 24 (2) |
| C(10) | 0.3239 (7) | 0.0642 (2) | 0.7594 (19) | 30 (3) |
| C(11) | 0.2201 (7) | 0.0697 (2) | 0.7308 (19) | 26 (4) |
| C(12) | 0.1328 (7) | 0.0549 (2) | 0.7759 (18) | 25 (2) |
| C(121) | 0.1403 (8) | 0.0300 (2) | 0.8494 (18) | 38 (3) |
| C(122) | 0.1569 (8) | 0.0113 (2) | 0.7099 (21) | 44 (3) |
| C(13) | 0.0465 (7) | 0.0670 (2) | 0.7324 (19) | 28 (3) |
| C(131) | −0.0658 (7) | 0.0601 (2) | 0.7540 (20) | 35 (3) |
| C(14) | 0.0818 (8) | 0.0892 (2) | 0.6565 (17) | 22 (4) |
| C(15) | 0.0150 (9) | 0.1065 (2) | 0.5894 (18) | 30 (4) |
| C(16) | 0.0392 (7) | 0.1281 (2) | 0.5103 (18) | 25 (4) |
| C(17) | −0.0343 (7) | 0.1448 (2) | 0.4451 (19) | 26 (3) |
| C(171) | −0.1514 (7) | 0.1404 (2) | 0.4453 (19) | 28 (3) |
| C(18) | 0.0169 (7) | 0.1644 (2) | 0.3864 (19) | 20 (2) |
| C(181) | −0.0350 (6) | 0.1854 (1) | 0.2995 (19) | 27 (3) |
| C(19) | 0.1246 (7) | 0.1606 (2) | 0.4147 (19) | 24 (2) |
| C(20) | 0.2100 (7) | 0.1757 (2) | 0.3831 (17) | 20 (2) |
| C(201) | 0.1844 (7) | 0.2008 (2) | 0.3238 (17) | 21 (2) |
| C(202) | 0.1627 (7) | 0.2187 (2) | 0.4404 (18) | 27 (3) |
| C(203) | 0.1365 (8) | 0.2415 (2) | 0.3845 (18) | 38 (3) |
| C(204) | 0.1366 (7) | 0.2471 (2) | 0.2121 (17) | 34 (3) |
| C(205) | 0.1577 (7) | 0.2292 (2) | 0.0950 (18) | 34 (3) |
| C(206) | 0.1816 (7) | 0.2061 (2) | 0.1513 (19) | 28 (3) |

Table 2. *Bond lengths* (Å) *and bond angles* (°) *between non*-H *atoms with e.s.d.'s in parentheses*

| | | | |
|---|---|---|---|
| N(21)—C(1) | 1.360 (12) | N(21)—C(4) | 1.349 (11) |
| N(22)—C(6) | 1.401 (13) | N(22)—C(9) | 1.354 (13) |
| N(23)—C(11) | 1.361 (13) | N(23)—C(14) | 1.353 (12) |
| N(24)—C(16) | 1.367 (12) | N(24)—C(19) | 1.382 (13) |
| C(1)—C(2) | 1.466 (14) | C(1)—C(20) | 1.407 (13) |
| C(2)—C(21) | 1.532 (14) | C(2)—C(3) | 1.339 (14) |
| C(3)—C(31) | 1.496 (14) | C(3)—C(4) | 1.448 (14) |
| C(4)—C(5) | 1.377 (14) | C(5)—C(6) | 1.381 (14) |
| C(6)—C(7) | 1.419 (15) | C(7)—C(71) | 1.493 (13) |
| C(7)—C(8) | 1.366 (14) | C(8)—C(81) | 1.485 (15) |
| C(8)—C(9) | 1.444 (13) | C(81)—C(82) | 1.502 (18) |
| C(9)—C(10) | 1.381 (13) | C(10)—C(11) | 1.390 (13) |
| C(11)—C(12) | 1.439 (14) | C(12)—C(121) | 1.504 (14) |
| C(12)—C(13) | 1.342 (14) | C(121)—C(122) | 1.527 (18) |
| C(13)—C(131) | 1.506 (13) | C(13)—C(14) | 1.445 (14) |
| C(14)—C(15) | 1.396 (14) | C(15)—C(16) | 1.391 (14) |
| C(16)—C(17) | 1.419 (14) | C(17)—C(171) | 1.527 (13) |
| C(17)—C(18) | 1.356 (14) | C(18)—C(181) | 1.510 (14) |
| C(18)—C(19) | 1.419 (13) | C(19)—C(20) | 1.406 (13) |
| C(20)—C(201) | 1.509 (13) | C(201)—C(202) | 1.383 (16) |
| C(201)—C(206) | 1.384 (20) | C(202)—C(203) | 1.383 (14) |
| C(203)—C(204) | 1.386 (19) | C(204)—C(205) | 1.380 (16) |
| C(205)—C(206) | 1.399 (14) | | |

| | | | |
|---|---|---|---|
| C(1)—N(21)—C(4) | 107.5 (8) | C(6)—N(22)—C(9) | 108.3 (8) |
| C(11)—N(23)—C(14) | 105.7 (8) | C(16)—N(24)—C(19) | 109.4 (8) |
| N(21)—C(1)—C(2) | 109.2 (8) | N(21)—C(1)—C(20) | 123.1 (8) |
| C(2)—C(1)—C(20) | 127.7 (9) | C(1)—C(2)—C(21) | 129.6 (9) |
| C(1)—C(2)—C(3) | 106.2 (9) | C(21)—C(2)—C(3) | 124.2 (9) |
| C(2)—C(3)—C(31) | 128.4 (9) | C(2)—C(3)—C(4) | 107.6 (9) |
| C(31)—C(3)—C(4) | 124.0 (9) | N(21)—C(4)—C(3) | 109.6 (8) |
| N(21)—C(4)—C(5) | 128.4 (9) | C(3)—C(4)—C(5) | 121.8 (9) |
| C(4)—C(5)—C(6) | 131.0 (10) | N(22)—C(6)—C(5) | 124.4 (10) |
| N(22)—C(5)—C(7) | 106.0 (8) | C(5)—C(6)—C(7) | 128.8 (10) |
| C(6)—C(7)—C(71) | 123.8 (9) | C(6)—C(7)—C(8) | 109.7 (9) |
| C(71)—C(7)—C(8) | 126.4 (10) | C(7)—C(8)—C(81) | 127.9 (9) |
| C(7)—C(8)—C(9) | 105.6 (9) | C(81)—C(8)—C(9) | 126.5 (9) |
| C(8)—C(81)—C(82) | 116.4 (13) | N(22)—C(9)—C(8) | 109.5 (8) |
| N(22)—C(9)—C(10) | 122.5 (9) | C(8)—C(9)—C(10) | 128.0 (9) |
| C(9)—C(10)—C(11) | 126.4 (10) | N(23)—C(11)—C(10) | 124.0 (9) |
| N(23)—C(11)—C(12) | 110.3 (8) | C(10)—C(11)—C(12) | 125.7 (10) |
| C(11)—C(12)—C(121) | 125.0 (9) | C(11)—C(12)—C(13) | 107.2 (9) |
| C(121)—C(12)—C(13) | 127.8 (9) | C(12)—C(121)—C(122) | 111.4 (11) |
| C(12)—C(13)—C(131) | 129.6 (10) | C(12)—C(13)—C(14) | 105.9 (8) |
| C(131)—C(13)—C(14) | 124.5 (9) | N(23)—C(14)—C(13) | 111.0 (8) |
| N(23)—C(14)—C(15) | 125.5 (9) | C(13)—C(14)—C(15) | 123.6 (9) |
| C(14)—C(15)—C(16) | 129.0 (10) | N(24)—C(16)—C(15) | 127.8 (9) |
| N(24)—C(16)—C(17) | 106.9 (8) | C(15)—C(16)—C(17) | 125.2 (2) |
| C(16)—C(17)—C(171) | 123.6 (9) | C(16)—C(17)—C(18) | 108.9 (8) |
| C(171)—C(17)—C(18) | 127.5 (9) | C(17)—C(18)—C(181) | 124.2 (8) |
| C(17)—C(18)—C(19) | 107.6 (9) | C(181)—C(18)—C(19) | 128.1 (8) |
| N(24)—C(19)—C(18) | 107.2 (8) | N(24)—C(19)—C(20) | 122.4 (9) |
| C(18)—C(19)—C(20) | 130.3 (9) | C(1)—C(20)—C(19) | 126.0 (9) |
| C(1)—C(20)—C(201) | 118.0 (8) | C(19)—C(20)—C(201) | 115.9 (8) |
| C(20)—C(201)—C(202) | 120.7 (12) | C(20)—C(201)—C(206) | 120.3 (10) |
| C(202)—C(201)—C(206) | 119.0 (9) | C(201)—C(202)—C(203) | 120.2 (13) |
| C(202)—C(203)—C(204) | 121.0 (11) | C(203)—C(204)—C(205) | 119.1 (10) |
| C(204)—C(205)—C(206) | 119.5 (13) | C(201)—C(206)—C(205) | 120.7 (11) |

ruffled porphyrin macrocycle with the phenyl substituent oriented 84.6° out of the plane of the N atoms.

**Experimental.** The porphyrin was prepared by acid-catalyzed condensation of 8,12-diethyl-2,3,7,13,17,-18-hexamethyl-*a*,*c*-biladiene dihydrobromide with benzaldehyde in the presence of air. Crystals were obtained by liquid diffusion from $CH_2Cl_2$/*n*-hexane. Red, hexagonal plates, 0.325 × 0.275 × 0.125 mm. Data collected using the Wyckoff scan technique; scan rate 8.08° $min^{-1}$ (in $\omega$); 2432 intensities recorded on a Syntex $P2_1$ diffractometer with graphite-monochromated Mo $K\alpha$ radiation, $2\theta_{max}$ = 50°, $0 \le h \le 15$, $0 \le k \le 49$, $0 \le l \le 9$. Two check reflections measured every 198 reflections, 2% intensity change. All of the 2432 reflections collected were unique, 1428 with $I > 2.0\sigma(I)$ used for all calculations (program system *SHELXTL-Plus*; Sheldrick, 1990). Cell constants refined from 20 reflections in the range $12 < 2\theta < 16°$. An absorption correction was performed using the program *XABS* (Hope & Moezzi, 1987), transmission factors from 0.71 to 0.85. Structure solution by random-start multisolution direct methods. $z$ position of N(21) fixed. The final cycle of refinement on $|F|$ included 181 variable parameters and converged with $R = 0.076$, $wR = 0.074$ and $S = 1.37$ (only the N atoms were refined anisotropically); H atoms were inserted at calculated positions with a riding model with C—H = 0.96 Å and $U_{iso}(H) = 0.04$ $Å^2$. Table 1 lists atomic coordinates and isotropic temperature factors and Table 2 gives bond lengths and bond angles.* The molecular

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a least-squares-plane calculation have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54575 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

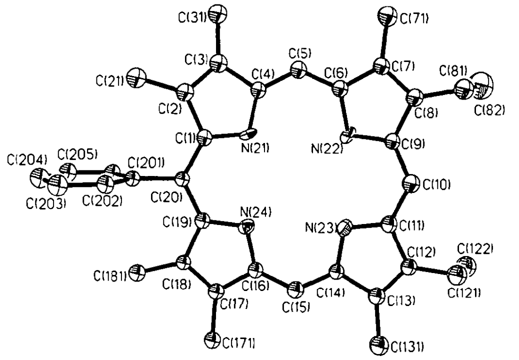


Fig. 1. Molecular structure and numbering scheme for the title compound.

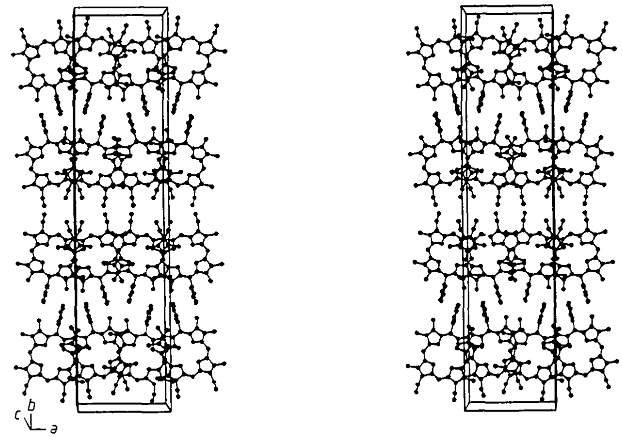

Fig. 2. View of the molecular packing.

structure is shown in Fig. 1. Fig. 2 gives a stereoview of the molecular packing in the unit cell. Weighting scheme defined as $w^{-1} = \sigma^2(F) + 0.0005F^2$, $\Delta/\sigma$ 0.180, data/parameter ratio 7.9:1, $(\Delta\rho)_{max} = 0.49$, $(\Delta\rho)_{min} = -0.36$ e Å$^{-1}$. Atomic scattering factors from Cromer & Waber (1974).

**Related literature.** This study presents part of an accumulating body of information on conformational details of the porphyrin macrocycle (Scheidt & Lee, 1987; Barkigia, Berber, Fajer, Medforth, Renner & Smith, 1990). The conformational flexibility is of central importance for the biological role of these chromophores in photosynthesis and electron transport (Barkigia, Chantranupong, Smith & Fajer, 1988). Although the $\beta$-substituents are small and only one *meso* position is substituted the molecule shows a slight degree of conformational distortion with the $\beta$-pyrrole atoms pushed alternately above and below the mean plane. The *meso* C atom bearing the phenyl substituent is displaced slightly above the mean plane and the plane of the phenyl substituent deviates 5.4° from an orthogonal orientation with respect to the plane of the four N atoms. The molecules are tightly packed in parallel layers with each layer connected to the neighboring one by $\pi - \pi$ stacking of one of the pyrrole rings.

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*Acta Cryst.* (1992). C**48**, 583–585

# Structure of a Styrylbenzoxazole Derivative

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(*Received* 8 *July* 1991; *accepted* 17 *September* 1991)

**Abstract.** 2-(2-Chloro-5-nitrostyryl)benzoxazole (nsbo), $C_{15}H_9ClN_2O_3$, $M_r = 300.70$, monoclinic, $P2_1/c$, $a = 7.030$ (3), $b = 13.55$ (1), $c = 15.40$ (1) Å, $\beta = 114.77$ (3)°, $V = 1332$ (3) Å$^3$, $Z = 4$, $D_x = 1.498$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.71073 Å, $\mu = 2.94$ cm$^{-1}$, $F(000) = 616$, $T = 298$ K, final $R = 0.043$ for 1244 unique observed reflections. The molecule is almost planar, with a dihedral angle of 6.8 (6)° between the benzoxazole and phenyl rings.

**Experimental.** The oxazole derivative nsbo (1) ($X$ = O) was prepared as part of a study of substitution in

0108-2701/92/030583-03$03.00