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5′-*tert*-Butoxycarbonyl-4′-methoxycarbonylethyl-3′,4-dimethyl-3-ethyl-2,2′-methylenedipyrrol-5(2*H*)-one

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Abstract. $C_{22}H_{32}N_2O_5$, monoclinic, $P2_1/c$, a = 13·369 (3), b = 14·502 (8), c = 11·751 (4) Å, β = 95·02 (3)°, M_r = 404·5, Z = 4, D_x = 1·26 g cm^{-3}. The interplanar angle between the ring systems is 99·6°. The molecules are linked together through two intermolecular N–H···O hydrogen bonds of length 2·86 and 2·85 Å.

Introduction. Cell dimensions were obtained from a least-squares fit to the settings for 15 reflexions (±*hkl*) on a Syntex $P2_1$ diffractometer (Cu K, λ = 1·54178 Å). Intensity measurements were carried out in the θ–2θ mode (3·5 ≤ 2θ ≤ 135·0°) with graphite-monochromated Mo $K\alpha$ radiation, at scan speeds varying linearly between 2·93° min^{-1} (150 counts s^{-1} and below) and 29·30° min^{-1} (5000 counts s^{-1} and above). Scan and background times were equal. Lorentz and polarization but no absorption [μ(Cu $K\alpha$) = 6·03 cm^{-1}] corrections were applied. After application of the acceptance criterion $I > 1{\cdot}5\sigma(I)$, 3590 unique reflexions were retained for use in the refinement. The structure was solved by direct methods (*SHELX* 76, G. M. Sheldrick) and refined by blocked full-matrix least squares with anisotropic temperature factors for all the non-hydrogen atoms. The H positional parameters were freely refined with individual isotropic temperature factors and a joint factor for the ethyl and *tert*-butyl groups. The terminal value of $R_G[=(\Sigma w\Delta^2/\Sigma wF_o^2)^{1/2}]$ was 0·049, with R_w (= $\Sigma w^{1/2}\Delta/\Sigma w^{1/2}|F_o|$) 0·049 and R 0·051. The weights adopted were those from the counting statistics. Complex neutral-atom scattering factors were employed (Cromer & Waber, 1965; Cromer & Liberman, 1970). Tables 1 and 2 list the final atomic parameters.* The atom-numbering scheme is given in Fig. 1.

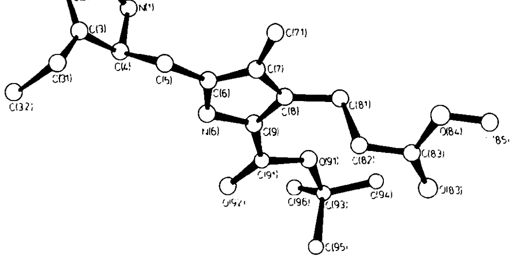

Fig. 1. The molecule of (I) in perspective view with the numbering system.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32619 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Positional parameters* (×10⁴)

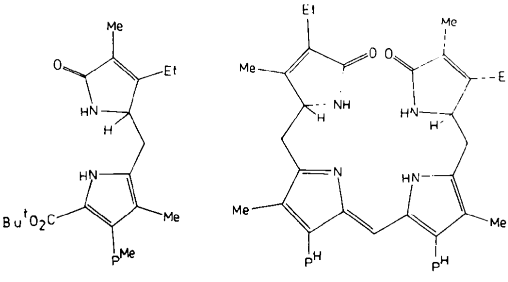
	x	*y*	*z*
N(1)	11832 (1)	1876 (1)	844 (1)
C(1)	12376 (1)	1936 (1)	9942 (1)
C(2)	13311 (1)	1407 (1)	227 (1)
C(3)	13304 (1)	1089 (1)	1290 (1)
C(4)	12341 (1)	1359 (1)	1782 (1)
C(5)	11746 (1)	517 (1)	2121 (1)
N(6)	10607 (1)	1355 (1)	3363 (1)
C(6)	10750 (1)	697 (1)	2569 (1)
C(7)	9866 (1)	198 (1)	2368 (1)
C(8)	9162 (1)	578 (1)	3065 (1)
C(9)	9638 (1)	1298 (1)	3673 (1)
O(11)	12152 (1)	2354 (1)	9042 (1)
C(21)	14077 (1)	1302 (1)	9378 (2)
C(31)	14112 (1)	543 (1)	1967 (2)
C(32)	14887 (2)	1138 (2)	2595 (3)
C(71)	9693 (1)	9399 (1)	1565 (2)
C(81)	8118 (1)	218 (1)	3165 (1)
C(82)	8085 (1)	9614 (1)	4231 (2)
C(83)	7037 (1)	9398 (1)	4501 (2)
O(83)	6667 (1)	9644 (1)	5340 (1)
O(84)	6525 (1)	8928 (1)	3681 (1)
C(85)	5501 (2)	8685 (2)	3877 (3)
C(91)	9323 (1)	1921 (1)	4537 (1)
O(91)	8326 (1)	1885 (1)	4612 (1)
O(92)	9877 (1)	2427 (1)	5118 (1)
C(93)	7840 (1)	2434 (1)	5467 (1)
C(94)	6742 (1)	2172 (2)	5199 (3)
C(95)	8238 (2)	2147 (2)	6656 (2)
C(96)	7981 (2)	3457 (1)	5262 (2)

Table 2. *Hydrogen atoms: positional parameters and isotropic temperature factors* (×10³)

	x	*y*	*z*	*U*
H(11)	1119 (2)	207 (2)	84 (2)	77 (7)
H(211)	1440 (3)	191 (3)	925 (4)	197 (17)
H(212)	1466 (3)	88 (3)	970 (3)	169 (14)
H(213)	1379 (2)	108 (2)	870 (3)	147 (13)
H(311)	1379 (2)	10 (2)	253 (3)	128 (3)
H(312)	1438 (2)	7 (2)	130 (3)	128 (3)
H(321)	1532 (2)	138 (2)	185 (3)	128 (3)
H(322)	1541 (2)	83 (2)	314 (3)	128 (3)
H(323)	1487 (2)	172 (2)	279 (3)	128 (3)
H(41)	1247 (1)	177 (1)	249 (2)	48 (5)
H(51)	1163 (2)	12 (1)	138 (2)	64 (7)
H(52)	1218 (1)	20 (1)	275 (2)	60 (6)
H(61)	1104 (1)	177 (1)	365 (2)	65 (7)
H(711)	1030 (2)	919 (2)	130 (3)	126 (11)
H(712)	945 (2)	888 (2)	199 (3)	138 (12)
H(713)	923 (3)	953 (2)	98 (3)	166 (14)
H(811)	790 (1)	985 (1)	242 (2)	59 (6)
H(812)	764 (1)	74 (1)	323 (2)	60 (6)
H(821)	846 (2)	990 (2)	498 (2)	95 (9)
H(822)	841 (2)	904 (2)	406 (2)	85 (8)
H(851)	552 (3)	828 (2)	471 (3)	139 (13)
H(852)	513 (3)	925 (3)	426 (3)	156 (16)
H(853)	517 (3)	850 (3)	321 (4)	189 (21)
H(941)	642 (2)	229 (2)	436 (3)	128 (3)
H(942)	663 (2)	153 (2)	525 (3)	128 (3)
H(943)	634 (2)	249 (2)	584 (3)	128 (3)
H(951)	911 (2)	245 (2)	684 (3)	128 (3)
H(952)	783 (2)	246 (2)	722 (3)	128 (3)
H(953)	827 (2)	152 (2)	670 (3)	128 (3)
H(961)	753 (2)	381 (2)	582 (3)	128 (3)
H(962)	882 (2)	365 (2)	554 (3)	128 (3)
H(963)	777 (2)	363 (2)	447 (3)	128 (3)

Discussion. The title compound (1) may be regarded as a useful model for certain bile pigments, and in particular for mesourobilin IXa (2) (Fig. 2), with regard to the relative orientation of the constituent rings of the 2,2′-methylenedipyrrole system about the central methylene bridge. (2) (formerly *i*-urobilin) has been isolated from natural sources both as a racemate and as the dextrorotatory enantiomer (*d*-urobilin of *M* 590) (Petryka, Weimer, Lightner, Chedekel, Bovey, Moscowitz & Watson, 1975). We have studied (1), whose synthesis has recently been described (Gossauer & Kühne, 1977), as the centrosymmetric racemic mixture.

Bond lengths are given in Table 3, bond angles in Table 4.



(1) (2)

Fig. 2. Structures of (1) and mesourobilin IXa (2).

Table 3. *Bond lengths* (Å)

C(1)–N(1)	1·339 (3)	C(4)–N(1)	1·451 (2)
C(2)–C(1)	1·480 (2)	O(11)–C(1)	1·233 (3)
C(3)–C(2)	1·332 (3)	C(21)–C(2)	1·499 (3)
C(4)–C(3)	1·509 (3)	C(31)–C(3)	1·508 (3)
C(5)–C(4)	1·529 (3)	C(6)–C(5)	1·497 (3)
C(6)–N(6)	1·359 (2)	C(9)–N(6)	1·379 (2)
C(7)–C(6)	1·389 (3)	C(8)–C(7)	1·412 (3)
C(71)–C(7)	1·499 (3)	C(9)–C(8)	1·387 (3)
C(81)–C(8)	1·505 (2)	C(91)–C(9)	1·449 (3)
C(32)–C(31)	1·492 (4)	C(82)–C(81)	1·533 (3)
C(83)–C(82)	1·498 (3)	O(83)–C(83)	1·196 (3)
O(84)–C(83)	1·321 (3)	C(85)–O(84)	1·451 (3)
O(91)–C(91)	1·344 (2)	O(92)–C(91)	1·211 (2)
C(93)–O(91)	1·477 (3)	C(94)–C(93)	1·523 (3)
C(95)–C(93)	1·509 (4)	C(96)–C(93)	1·517 (3)
H(11)–N(1)	0·91 (2)	H(41)–C(4)	1·03 (2)
H(51)–C(5)	1·05 (2)	H(52)–C(5)	1·00 (2)
H(61)–N(6)	0·88 (2)	H(211)–C(21)	1·00 (4)
H(212)–C(21)	1·03 (4)	H(213)–C(21)	0·92 (3)
H(311)–C(31)	1·05 (3)	H(312)–C(31)	1·12 (3)
H(321)–C(32)	1·15 (3)	H(322)–C(32)	1·02 (3)
H(323)–C(32)	0·87 (3)	H(711)–C(71)	0·95 (3)
H(712)–C(71)	0·97 (3)	H(713)–C(71)	0·90 (4)
H(811)–C(81)	1·04 (2)	H(812)–C(81)	1·00 (2)
H(821)–C(82)	1·06 (3)	H(822)–C(82)	0·97 (2)
H(851)–C(85)	1·14 (4)	H(852)–C(85)	1·08 (4)
H(853)–C(85)	0·91 (4)	H(941)–C(94)	1·05 (3)
H(942)–C(94)	0·94 (3)	H(943)–C(94)	1·07 (4)
H(951)–C(95)	1·25 (3)	H(952)–C(95)	1·00 (3)
H(953)–C(95)	0·90 (3)	H(961)–C(96)	1·06 (3)
H(962)–C(96)	1·18 (3)	H(963)–C(96)	0·98 (3)

Table 4. *Bond angles* (°)

C(4)–N(1)–C(1)	112·6 (1)	C(2)–C(1)–N(1)	107·0 (2)
O(11)–C(1)–N(1)	127·1 (2)	O(11)–C(1)–C(2)	125·9 (2)
C(3)–C(2)–C(1)	108·5 (2)	C(21)–C(2)–C(1)	121·0 (2)
C(21)–C(2)–C(3)	130·5 (2)	C(4)–C(3)–C(2)	110·2 (2)
C(31)–C(3)–C(2)	127·8 (2)	C(31)–C(3)–C(4)	122·0 (2)
C(3)–C(4)–N(1)	101·7 (1)	C(5)–C(4)–N(1)	112·9 (1)
C(5)–C(4)–C(3)	111·9 (2)	C(6)–C(5)–C(4)	116·9 (2)
C(9)–N(6)–C(6)	109·4 (1)	N(6)–C(6)–C(5)	123·2 (2)
C(7)–C(6)–C(5)	128·3 (2)	C(7)–C(6)–N(6)	108·2 (2)
C(8)–C(7)–C(6)	107·4 (2)	C(71)–C(7)–C(6)	126·3 (2)
C(71)–C(7)–C(8)	126·3 (2)	C(9)–C(8)–C(7)	107·1 (2)
C(81)–C(8)–C(7)	125·4 (2)	C(81)–C(8)–C(9)	127·4 (2)
C(8)–C(9)–N(6)	108·0 (2)	C(91)–C(9)–N(6)	119·0 (2)
C(91)–C(9)–C(8)	133·0 (2)	C(32)–C(31)–C(3)	113·1 (2)
C(82)–C(81)–C(8)	110·8 (2)	C(83)–C(82)–C(81)	112·8 (2)
O(83)–C(83)–C(82)	125·1 (2)	O(84)–C(83)–C(82)	112·8 (2)
O(84)–C(83)–O(83)	122·0 (2)	C(85)–O(84)–C(83)	116·4 (2)
O(91)–C(91)–C(9)	111·8 (2)	O(92)–C(91)–C(9)	124·9 (2)
O(92)–C(91)–O(91)	123·3 (2)	C(93)–O(91)–C(91)	121·4 (1)
C(94)–C(93)–O(91)	101·4 (2)	C(95)–C(93)–O(91)	109·9 (2)
C(95)–C(93)–C(94)	112·2 (3)	C(96)–C(93)–O(91)	110·5 (2)
C(96)–C(93)–C(94)	110·0 (2)	C(69)–C(93)–C(95)	112·2 (2)

The interplanar angle in (1) is 99·6°. This is similar to those of 98 and 107° between the methylene-bridged ring systems in bilirubin (Bonnett, Davies & Hursthouse, 1976) and in a biladiene-*ac* dihydrobromide derivative (Struckmeier, Thewalt & Engel, 1976) respectively. An angle of 71·9° has been observed for a model 2,2′-methylenedipyrrole (Bonnett, Hursthouse & Neidle, 1972). In bilirubin six intramolecular N–H···O hydrogen bonds stabilize this arrangement. In (1) the molecules are linked together through linear intermolecular N(1)–H(11)···O(92) and N(6)–H(61)···O(11) hydrogen bonds of 2·86 and 2·85 Å respectively. The C(91)–O(92) carbonyl group is *syn* to the N(6)–H(61) vector. Surprisingly it is twisted somewhat out of the plane of the pyrrole ring [C(91) 0·039, O(92) 0·235, O(91) −0·192 Å from this plane], presumably as a result of steric contacts between O(91) and C(81) and C(82) [O(91)···C(81) 2·95, O(91)···C(82) 3·34 Å].

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4-Methylheptane-3,5-dionato(2–)-C^2,C^6-tellurium(II)*

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Abstract. $C_8H_{12}O_2Te$, monoclinic, $P2_1$, $a = 8{\cdot}80$ (1), $b = 4{\cdot}92$ (1), $c = 11{\cdot}04$ (2) Å, $\beta = 93{\cdot}3$ (1)°, $M_r = 267{\cdot}8$, $U = 477{\cdot}2$ Å^3, $Z = 2$, $D_m = 1{\cdot}87$, $D_c = 1{\cdot}86$ g cm^{-3}, $F(000) = 256$. Mo $K\alpha$ radiation ($\lambda = 0{\cdot}7107$ Å; $\mu = 28{\cdot}5$ cm^{-1}). The structure was solved by the heavy-atom method from 789 two-circle diffractometer reflexions and refined by full-matrix least squares to R 0·048. In the molecules of the compound each Te^{II} atom is bonded to one bivalent 'bidentate chelate ligand' *via* the C^2 and C^6 carbon atoms [Te–C 2·18 (1) Å; C–Te–C 89·3 (6)°] forming a heterocyclic ring of chair conformation with the three methyl substituents all in equatorial positions.

Introduction. We have previously reported the crystal structures of compounds *A*, *B*, *C*, and *D*.

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* Studies on Tellurium–Carbon Bonded Compounds. IV. Part III: Dewan & Silver (1977*c*).

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