Discussion. The structure of the molecule, the bond lengths and valence angles are shown in Figs. 1(*a*) and 1(*b*). The mean bond lengths in the diaza-18-crown-6 fragment are characteristic of the macrocyclic polyethers: C—C 1·517, C—N 1·476 and C—O 1·433 Å. The mean lengths of C—C and C—O bonds in the polyoxyethylene bridge are 1·468 and 1·441 Å, respectively. The C—C bonds in the cryptand $[3_{NON},2_O,2_O]$, like in other macrocyclic polyethers (Goldberg, 1980), are shorter than the standard value 1·54 Å. The C—S bond lengths 1·691 and 1·692 (7) Å in the almost planar thiourea groups are close in value to those found in other cyclic thioureas (Malinowsky, Kiritchenko, Simonov, Lukyanenko & Bogatsky, 1981; Ganin, Simonov, Lukyanenko, Bogatsky, Kiritchenko & Botoshansky, 1981), which is characteristic of the thione form of thiourea. The lengths of the C—N bonds are within the range 1·329–1·360 Å. The configurations of the C—N—C(=S)—N—C fragment in cyclic thiourea, *cis–cis*, *cis–trans* and *trans–trans*, may depend on the size of the ring. The torsion angles at the C(19)—N(20) and C(27)—N(26) bonds in cryptand $[3_{NON},2_O,2_O]$ are 8·2 and 5·5°, respectively. This testifies to the *cis* configuration of the fragments. Such a configuration is stabilized additionally by the intramolecular hydrogen bonds N(26)H···O(13) 2·830 Å [N—H 0·85, O···H 2·06 Å, angle N—H—O 150°] and N(20)H···O(16) 2·825 Å [N—H 1·12, O···H 1·85 Å, angle N—H—O 143°] (Fig. 2). The torsion angles are presented in Table 2. The most convenient *gauche, trans* and *trans* conformations are in the bridge N(20)C(21)···C(25)-N(26) *via* C—C, C—O and C—N bonds. Considerable conformational strains are observed in the diaza-18-crown-6 fragment. The conformation of this cycle is described by the sequence of $g^-, g^+, g^-, t, g^+, t, g^+, g^-, g^-, g^+, g^+, g^+, g^-, g^-, g^+, t, g^+, g^-$ rotamers. Convenient *trans* rotamers are realized only in three out of 12 C—O and C—N bonds. In contrast to the above, the convenient *trans–gauche–trans* conformations are realized *via* X—CH$_2$—CH$_2$—X bonds (X=O,N) (Dale, 1973; Herceg & Weiss, 1972; Moras, Metz, Herceg & Weiss, 1972; Weber, 1982) in a monocyclic diaza-18-crown-6 molecule.

The structure of the cryptand is molecular. The distances between the neighbouring molecules are equal to or exceed van der Waals radii.

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Sarcophinone at 115 K: a Cembranolide Diterpenoid from the South China Sea

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Abstract. $C_{20}H_{28}O_3$, $M_r = 316·44$, orthorhombic, $P2_12_12_1$, $a = 11·197$ (6), $b = 12·582$ (6), $c = 12·569$ (6) Å, $V = 1771$ Å^3, $Z = 4$, $D_x = 1·19$ g cm^{-3}, $\lambda(Mo K\alpha) = 0·7107$ Å, $\mu = 0·44$ cm^{-1}, $F(000) = 688$, $T = 115$ K, $R = 0·062$ for 1235 unique observed reflections. This cembranolide diterpenoid, isolated

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from the soft coral *Sarcophyton decaryi*, contains an unsaturated 14-membered ring, with slightly twisted *transoid* double bonds, a ketone function and a fused unsaturated lactone. It is isomeric with and conformationally similar to sarcophine, an epoxy cembranolide whose structure has previously been reported [Bernstein, Shmueli, Zadock & Kashman (1974). *Tetrahedron*, **30**, 2817–2824].

Introduction. Sarcophinone, a new diterpenoid, was isolated from soft coral collected in the South China Sea near Hai-nan Island in November 1980. The present compound, whose chemical formula is shown in (1), is isomeric with the cembranolide diterpenoid sarcophine* (Bernstein, Shmueli, Zadock & Kashman, 1974), isolated from the soft coral *Sarcophytum glaucum* of the Red Sea. The present structure (Fig. 1) differs from that of sarcophine (Fig. 2) only in the region of C(6) and C(7); sarcophine is an epoxide, with O(23) bridging C(6) and C(7), whereas in the present structure O(23) is doubly bonded to C(6). In addition, the configuration at C(7) in sarcophine is inverted with respect to that in the present molecule. We believe that the mild, acid-free conditions used in the isolation of sarcophinone would not have led to epimerization or the opening of an epoxide ring, and thus believe that the molecule whose structure is reported here is not an artifact of the isolation process. It is known that other closely related cembranolide diterpenoids occur in *S. glaucum* (Bernstein *et al.*, 1974), including sarcoglaucol (REFCODE SARCOL) (Albericci, Braekman, Daloze, Tursch, Declercq, Germain & Van Meersche, 1978). Other soft corals contain somewhat different cembranolide diterpenoids; several structures have been reported (*e.g.* Karlsson, 1977; Hossain, van der Helm, Matson & Weinheimer, 1979).
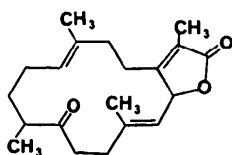
(1)

Experimental. Sarcophinone was extracted by cutting the soft coral into small pieces and extracting repeatedly with ethanol; the combined extracts were concentrated in a rotary evaporator, and then chromatographed on silica gel. In addition to sarcophinone, the known compounds sarcophine and gorgosterol (Hale, Leclercq, Tursch, Djerassi, Gross, Weinheimer, Gupta & Schleuer, 1970) were isolated.

* The REFCODE for sarcophine in the Cambridge Structural Data File (Allen, Bellard, Brice, Cartwright, Doubleday, Higgs, Hummelink, Hummelink-Peters, Kennard, Motherwell, Rodgers & Watson, 1979) is SARCOP.

Sarcophinone was crystallized from the fraction eluted with ethyl acetate/petroleum ether (1/4 v/v), and recrystallized from ethyl acetate; m.p. 414–415 K. The molecular weight was confirmed by high-resolution mass spectrometry, and the presence of the carbonyl group by IR spectroscopy. Colorless prismatic single crystal, $0.27 \times 0.27 \times 0.52$ mm; Syntex $P\bar{1}$ diffractometer equipped with a variable-temperature device (Strouse, 1976), graphite monochromator; unit-cell parameters by least-squares refinement of 15 reflections $(17.90 \leq 2\theta \leq 26.10°)$, θ–2θ scan, $6.0°$ min^{-1} from 1° below $K\alpha_1$ to 1° above $K\alpha_2$, $2\theta_{max} = 57°$ for range $0 \leq h \leq 15$, $0 \leq k \leq 16$, $0 \leq l \leq 16$, three reflections monitored every 97 reflections with maximum 5% intensity variation from average, 2597 measured intensities, 2577 unique data, 2234 with $I > 0$, 1235 with $I > 3\sigma(I)$ used in refinement, Lorentz and polarization but no absorption correction $(\mu = 0.44$ cm$^{-1})$; structure solved by direct methods using *RANTAN* (Yao Jia-xing, 1981), all atoms located on Fourier and difference Fourier maps except for three H atoms (on three different methyl groups). H atoms fixed in calculated positions based on those found, with C–H $= 1.09$ Å, H–C–H angle $= 109.5°$, and not refined; anisotropic displacement parameters for all non-H atoms; refined by full-matrix least-squares procedure based on F with a maximum $\sin\theta/\lambda = 0.67$ Å^{-1}; refinement of 208 parameters converged to $R = 0.062$, $wR = 0.060$, $w = 1/\sigma^2(F_o)$, goodness of fit $= 1.56$, max.
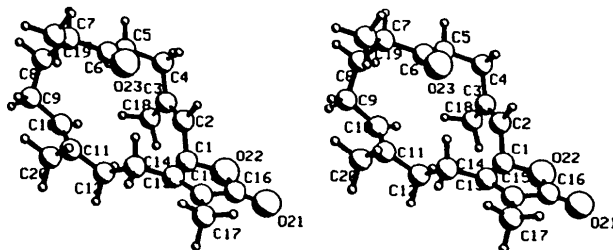

Fig. 1. A stereoview of sarcophinone. The molecular conformation is almost identical with that of sarcophine (Fig. 2).
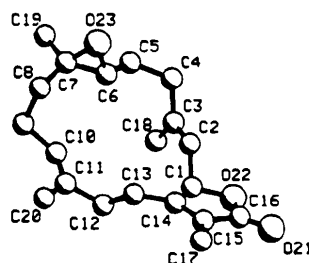

Fig. 2. The structure of sarcophine (Bernstein *et al.*, 1974). The numbering scheme is the same as that used for the present molecule. H atoms in this structure were not located.

Δ/σ in final cycle 0·36, average Δ/σ 0·04; maximum and minimum heights of 0·27 and −0·26 e Å⁻³ in final difference Fourier synthesis; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); all calculations on a DEC VAX 11/780 and VAX 11/750, using a locally edited profile-analysis program, *SHELX*76 (Sheldrick, 1976), a local molecular-geometry program, *MG*84, and a local molecular-motion program (Trueblood, 1978). Illustrations prepared with a locally edited version of *PLUTO*78 (Motherwell & Clegg, 1978).

Discussion. Atomic coordinates and equivalent isotropic displacement parameters are listed in Table 1* and bond lengths, bond angles and selected torsion angles are given in Table 2. There is no appreciable molecular motion; librational corrections to bond lengths ranged from 0·001 to 0·003 Å, only a small fraction of the e.s.d.'s, and were neglected. The anisotropic U values are of low quality by the rigid-bond test (Hirshfeld, 1976). On the whole, the molecular geometry is quite normal (Table 2). Although bond C(7)—C(19) seems unusually short for a bond between tetrahedral atoms [1·504 (7) Å], the C(7)—C(6) distance is longer than expected [1·532 (6) Å] for a bond between a trigonal and a tetrahedral atom; we suspect that the position of C(7) may be slightly in error. The conformation of sarcophinone (Fig. 1) is almost identical to that of sarcophine (Fig. 2) except in the region of C(6) and C(7). The lactone ring is planar (maximum deviation of any atom 0·005 Å), with the attached atoms C(17) and O(21) also essentially in the plane, the deviation of the latter [0·024 (5) Å] being at the border of significance. There are small twists around the double bonds (both of which are in the *E* or *transoid* conformation) in the 14-membered ring: 4·5 (6)° about C(2)—C(3) and 2·6 (7)° about C(10)—C(11). The normal to the least-squares plane through C(1), C(2), C(3), C(4) and C(18) makes an angle of 85° with the normal to the lactone ring. The shortest transannular distances (Fig. 1) are 3·19 Å between C(6) and C(10), 2·28 Å between the *unrefined* positions of H(10) and H(18B), and 2·18 Å between the *unrefined* positions of H(1) and H(12B). The shortest intermolecular distances are: C to O, 3·32 (1) Å from C(5) to O(22) at $-x, \frac{1}{2}+y, \frac{3}{2}-z$; C to C, 3·38 (1) Å from C(4) to C(19) at $\frac{1}{2}-x, 1-y, z-\frac{1}{2}$; C to H, 2·6 Å from C(11) to H(17B) at $-x, \frac{1}{2}+y, \frac{5}{2}-z$; O to H, 2·6 Å from O(21) to H(8A) at $x, y-1, z$; and H to H, 2·2 Å from H(8B) to H(17C) at $x-\frac{1}{2}, \frac{1}{2}-y, 2-z$.

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* Lists of anisotropic displacement parameters, H-atom parameters and geometry, additional torsion angles and structure-factor amplitudes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42584 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Position and displacement parameters for sarcophinone,* $C_{20}H_{28}O_3$

Displacement parameters are commonly called vibration parameters. Units of each e.s.d., in parentheses, are those of the least-significant digit of the corresponding parameter. Isotropic values are $1/8\pi^2 \times$ 'equivalent B value' defined by Hamilton (1959).

	x	y	z	$\langle u^2 \rangle (\text{Å}^2)$
C(1)	0·0484 (5)	0·0806 (5)	0·9452 (4)	0·029
C(2)	0·1000 (5)	0·1650 (5)	0·8760 (4)	0·028
C(3)	0·0408 (5)	0·2420 (5)	0·8256 (5)	0·028
C(4)	0·1069 (6)	0·3263 (5)	0·7649 (5)	0·031
C(5)	0·0940 (5)	0·4386 (6)	0·8110 (5)	0·031
C(6)	0·1662 (6)	0·4580 (6)	0·9117 (5)	0·031
C(7)	0·1553 (6)	0·5695 (5)	0·9597 (5)	0·037
C(8)	0·0265 (6)	0·5996 (5)	0·9959 (6)	0·048
C(9)	−0·0182 (6)	0·5480 (5)	1·1009 (6)	0·048
C(10)	−0·0318 (6)	0·4287 (5)	1·0920 (5)	0·040
C(11)	0·0070 (7)	0·3542 (5)	1·1581 (6)	0·040
C(12)	−0·0113 (5)	0·2385 (5)	1·1324 (5)	0·038
C(13)	0·1103 (5)	0·1804 (4)	1·1175 (4)	0·032
C(14)	0·1049 (5)	0·0800 (5)	1·0539 (5)	0·030
C(15)	0·1580 (5)	−0·0128 (5)	1·0707 (5)	0·028
C(16)	0·1414 (5)	−0·0805 (4)	0·9750 (5)	0·038
C(17)	0·2272 (6)	−0·0539 (5)	1·1649 (4)	0·043
C(18)	−0·0942 (4)	0·2521 (3)	0·8220 (3)	0·032
C(19)	0·2468 (6)	0·5890 (5)	1·0451 (5)	0·057
C(20)	0·0749 (4)	0·3747 (3)	1·2614 (4)	0·053
O(21)	0·1763 (5)	−0·1686 (5)	0·9569 (5)	0·055
O(22)	0·0759 (6)	−0·0246 (5)	0·9019 (5)	0·039
O(23)	0·2354 (4)	0·3934 (3)	0·9469 (3)	0·039

Table 2. *Distances* (Å), *angles* (°) *and selected torsion angles* (°) *for sarcophinone,* $C_{20}H_{28}O_3$, *at* 115 K

E.s.d.'s in parentheses are in units of the least-significant digit of the corresponding value.

C(1)—C(2)	1·489 (8)	C(1)—C(14)	1·506 (8)
C(1)—O(22)	1·464 (7)	C(2)—C(3)	1·334 (8)
C(3)—C(4)	1·502 (8)	C(3)—C(18)	1·518 (8)
C(4)—C(5)	1·534 (8)	C(5)—C(6)	1·522 (8)
C(6)—C(7)	1·532 (8)	C(6)—O(23)	1·207 (7)
C(7)—C(8)	1·559 (10)	C(7)—C(19)	1·504 (10)
C(8)—C(9)	1·554 (10)	C(9)—C(10)	1·513 (10)
C(10)—C(11)	1·326 (10)	C(11)—C(12)	1·505 (10)
C(11)—C(20)	1·526 (9)	C(12)—C(13)	1·557 (9)
C(13)—C(14)	1·496 (8)	C(14)—C(15)	1·327 (8)
C(15)—C(16)	1·486 (9)	C(15)—C(17)	1·507 (9)
C(16)—O(21)	1·197 (8)	C(16)—O(22)	1·370 (8)

C(2)—C(1)—C(14)	111·7 (5)	C(2)—C(1)—O(22)	110·2 (5)
C(14)—C(1)—O(22)	104·1 (4)	C(1)—C(2)—C(3)	127·0 (5)
C(2)—C(3)—C(4)	120·6 (5)	C(2)—C(3)—C(18)	124·7 (5)
C(4)—C(3)—C(18)	114·7 (5)	C(3)—C(4)—C(5)	114·3 (5)
C(4)—C(5)—C(6)	114·3 (5)	C(5)—C(6)—C(7)	115·6 (5)
C(5)—C(6)—O(23)	122·5 (5)	C(7)—C(6)—O(23)	121·6 (5)
C(6)—C(7)—C(8)	114·3 (5)	C(6)—C(7)—C(19)	112·1 (5)
C(8)—C(7)—C(19)	112·5 (5)	C(7)—C(8)—C(9)	116·4 (6)
C(8)—C(9)—C(10)	112·6 (6)	C(9)—C(10)—C(11)	128·5 (6)
C(10)—C(11)—C(12)	120·3 (6)	C(10)—C(11)—C(20)	125·2 (6)
C(12)—C(11)—C(20)	114·4 (5)	C(11)—C(12)—C(13)	111·2 (5)
C(12)—C(13)—C(14)	115·2 (5)	C(1)—C(14)—C(13)	119·8 (5)
C(1)—C(14)—C(15)	109·7 (5)	C(13)—C(14)—C(15)	129·8 (5)
C(14)—C(15)—C(16)	108·6 (5)	C(14)—C(15)—C(17)	131·1 (5)
C(16)—C(15)—C(17)	120·3 (5)	C(15)—C(16)—O(21)	130·1 (6)
C(15)—C(16)—O(22)	108·4 (5)	O(21)—C(16)—O(22)	121·5 (6)
C(1)—O(22)—C(16)	109·1 (4)		

C(2)—C(1)—C(14)—C(13)	52·5 (7)	C(14)—C(1)—C(2)—C(3)	−124·5 (7)
C(1)—C(2)—C(3)—C(4)	175·5 (6)	C(2)—C(3)—C(4)—C(5)	−114·7 (6)
C(3)—C(4)—C(5)—C(6)	74·9 (7)	C(4)—C(5)—C(6)—C(7)	179·9 (5)
C(5)—C(6)—C(7)—C(8)	61·9 (7)	C(6)—C(7)—C(8)—C(9)	75·4 (7)
C(7)—C(8)—C(9)—C(10)	−65·7 (8)	C(8)—C(9)—C(10)—C(11)	131·9 (8)
C(9)—C(10)—C(11)—C(12)	−177·4 (7)	C(10)—C(11)—C(12)—C(13)	116·6 (7)
C(11)—C(12)—C(13)—C(14)	−158·1 (6)	C(12)—C(13)—C(14)—C(1)	53·0 (7)
C(12)—C(13)—C(14)—C(15)	−137·6 (7)	C(1)—C(14)—C(15)—C(16)	0·2 (7)

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SHORT-FORMAT PAPERS

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Structure of 5,11-Bis(chloroformyl)-2,8-dimethyl-6H,12H-dibenzo[b,f][1,5]diazocine at 163 K

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Abstract. $C_{18}H_{16}Cl_2N_2O_2$, $M_r = 363 \cdot 24$, monoclinic, $P2_1/c$, $a = 10 \cdot 0742$ (15), $b = 9 \cdot 9129$ (18), $c = 8 \cdot 1320$ (12) Å, $\beta = 95 \cdot 581$ (10)°, $V = 808 \cdot 2$ (2) Å^3, $Z = 2$, $D_x = 1 \cdot 492$, $D_m(295 \text{ K}) = 1 \cdot 424 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0 \cdot 71079$ Å, $\mu = 4 \cdot 133 \text{ cm}^{-1}$, $F(000) = 376$, $R = 0 \cdot 0378$ for 1916 observed reflections. Molecules have a center of symmetry. The eight-membered central ring is in a chair conformation. The planes of the NCOCl groups make angles of 65·79 (4)° with the planes of the benzo bridges such that the Cl and O atoms are in van der Waals contact with the intramolecular H atoms [Cl···H(2), 3·02 (2); O···H(7B), 2·51 (2) Å]. The configuration at N is essentially planar and conjugation is evident in the shortened N—C(benzo) and N—C(carbonyl) bonds [1·439 (2) and 1·347 (2) Å respectively].

Experimental. Title compound prepared by method of Cooper & Partridge (1957). Data crystal obtained by cutting a corner from a large blocky crystal. A summary of data collection and structural refinement is given in Table 1.

Structure solved by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and Fourier techniques; refined by full-matrix least squares (*SHELX*76, Sheldrick, 1976); all atomic positional parameters refined as well as anisotropic thermal parameters for non-H atoms and isotropic for H atoms. Electron density difference map calculated at $R = 0 \cdot 06$ revealed H atoms as peaks of 0·67–1·02 e Å^{-3}. Scattering factors and anomalous-dispersion corrections for all non-H atoms from *International Tables for X-ray Crystallography* (1974); H scattering factors from Stewart, Davidson & Simpson (1965). Atomic parameters are in Table 2, bond lengths and angles in Table 3.† Atom labeling is shown in Fig. 1 and packing in Fig. 2. Principal computer programs given by Gadol & Davis (1982); program for least-squares-planes' calculations from Cordes (1983).

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