3·335 (3) Å (Hill & Hope, 1974), while in the 1,5-dithiacyclooctane–diiodine adduct, the distance is 3·44 (1) Å (Nichols, 1978). A possible explanation for this apparent anomaly may be attributed to an increased C—C—C angle found in the diacetal. The presence of the five-membered 1,3-dioxolane ring requires the O—C—O angle to be smaller than tetrahedral. This forces the C—C—C angle to increase with a corresponding increase in the transannular S···S distance. These results suggest that there is little if any sulfur–sulfur interaction in the neutral eight-membered-ring dithioether. If the structure is not altered appreciably in the mixed-valence ruthenium complex, the interaction that produces the intervalence transition must occur over a distance of ~3·6 Å. This is the frontier of the van der Waals radii of two S atoms (3·7 Å).

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17-Methoxy-16,17-seco-8α,13α-androsta-4,9(11)-diene-3,15,17-trione

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Abstract. $C_{20}H_{26}O_4$, $M_r = 330·4$, orthorhombic, *Pbca*, $a = 22·968 (18)$, $b = 12·807 (4)$, $c = 12·017 (5)$ Å, $V = 3535$ Å^3, $Z = 8$, $\rho_x = 1·24$ Mg m^{-3}, $T \sim 298$ K, $F(000) = 1424$, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å. Intensities for 2307 unique reflections were collected on a diffractometer. A block-diagonal least-squares refinement gave a final R of 0·051 for 1435 observed $[I \geq 3\sigma(I)]$ reflections. The title compound exists as a racemic mixture.

(I)

Introduction. The title compound (I) was prepared in good yield by an SnCl$_4$-catalysed addition of methyl (Z)-2-methyl-4-oxo-2-pentenoate to 9-methyl-1-vinyl-3,4,6,7,8,9-hexahydro-6-naphthalenone (Das, 1978). The establishment of the structure and stereochemistry of adduct (I) revealed the geometric and electronic requirements of this complex Diels–Alder reaction and made it possible to develop short total syntheses of androstane-type steroids (Kakushima, Allain, Dickinson, White & Valenta, 1979; Kakushima, Das, Reid, White & Valenta, 1979).
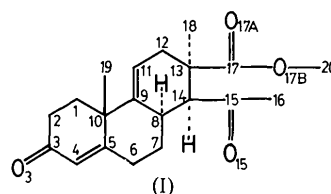
Crystallographic data were measured on a specimen crystal of approximate dimensions 0·83 × 0·20 × 0·13 mm using a Picker FACS-I diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Preliminary photographic work had shown the crystal to be orthorhombic, and the space group (*Pbca*) was uniquely determined by the systematic absences 0kl: k odd, h0l: l odd and hk0: h odd. The lattice parameters were refined by a least-squares fit of cell dimensions and an orientation matrix to the centred diffractometer settings for 12 Friedel pairs of reflections in the interval $33° \leq 2\theta \leq 37°$. Of the 2307 independent reflections measured in the range $2\theta \leq 45°$, 872 (37·8%) were

considered unobserved $[I < 3\sigma(I)]$ and were not included in subsequent calculations.

Data reduction and structure refinement utilized the NRC PDP-8e Crystal Structure System (Larson & Gabe, 1978). Initial positional parameters for non-hydrogen atoms were determined using Larson &

Table 1. *Fractional atomic coordinates* ($\times 10^4$ *for the non-hydrogen atoms,* $\times 10^3$ *for* H; *e.s.d.'s in parentheses) and isotropic thermal parameters* ($U_{eq} \times 10^4$ *for non-hydrogen atoms,* $U \times 10^2$ *for* H; *e.s.d.'s for* U *in parentheses*)

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}/U (Å^2)
C(1)	3621 (2)	4002 (3)	4353 (4)	941
C(2)	3253 (2)	3025 (4)	4143 (4)	1159
C(3)	3207 (2)	2825 (3)	2913 (4)	860
C(4)	3118 (1)	3751 (3)	2245 (3)	656
C(5)	3172 (1)	4730 (2)	2604 (3)	519
C(6)	3011 (1)	5641 (2)	1880 (2)	559
C(7)	3293 (1)	6670 (3)	2204 (2)	527
C(8)	3890 (1)	6506 (2)	2726 (2)	447
C(9)	3844 (1)	5837 (2)	3766 (2)	475
C(10)	3375 (1)	4986 (3)	3786 (3)	572
C(11)	4202 (1)	5983 (3)	4615 (3)	569
C(12)	4657 (1)	6809 (3)	4707 (3)	585
C(13)	4792 (1)	7337 (2)	3594 (2)	486
C(14)	4228 (1)	7529 (2)	2928 (2)	456
C(15)	3839 (1)	8369 (2)	3441 (3)	517
C(16)	3607 (2)	9210 (3)	2697 (3)	769
C(17)	5100 (1)	8373 (2)	3763 (3)	532
C(18)	5214 (2)	6650 (3)	2908 (3)	643
C(19)	2833 (2)	5390 (4)	4427 (3)	808
C(20)	5760 (2)	9329 (3)	4894 (3)	755
O(3)	3213 (1)	1947 (2)	2521 (4)	1192
O(15)	3724 (1)	8376 (2)	4423 (2)	772
O(17A)	5060 (1)	9107 (2)	3155 (2)	856
O(17B)	5443 (1)	8373 (2)	4654 (2)	698
H(1A)	404 (2)	380 (3)	408 (4)	14 (2)
H(1B)	374 (2)	414 (3)	514 (4)	14 (2)
H(2A)	285 (4)	318 (6)	449 (6)	23 (3)
H(2B)	345 (2)	243 (3)	454 (3)	10 (1)
H(4)	301 (2)	368 (3)	148 (4)	10 (1)
H(6A)	312 (1)	542 (2)	115 (2)	6 (1)
H(6B)	259 (1)	574 (2)	187 (2)	7 (1)
H(7A)	330 (1)	706 (2)	162 (2)	6 (1)
H(7B)	303 (1)	702 (2)	269 (2)	5 (1)
H(8)	412 (1)	609 (2)	219 (2)	5 (1)
H(11)	419 (1)	552 (2)	523 (3)	7 (1)
H(12A)	452 (2)	741 (3)	526 (3)	8 (1)
H(12B)	503 (2)	646 (2)	498 (3)	9 (1)
H(14)	435 (1)	774 (2)	221 (2)	4 (1)
H(16A)	402 (3)	964 (4)	252 (5)	19 (2)
H(16B)	354 (2)	888 (3)	201 (3)	12 (1)
H(16C)	332 (2)	964 (4)	299 (4)	14 (2)
H(18A)	526 (1)	691 (2)	214 (3)	8 (1)
H(18B)	561 (1)	663 (3)	320 (3)	10 (1)
H(18C)	507 (2)	594 (2)	291 (3)	8 (1)
H(19A)	251 (1)	476 (3)	449 (3)	11 (1)
H(19B)	292 (2)	554 (3)	512 (3)	9 (1)
H(19C)	270 (2)	602 (3)	412 (4)	13 (2)
H(20A)	603 (4)	952 (5)	427 (5)	21 (3)
H(20B)	604 (3)	906 (4)	538 (6)	21 (3)
H(20C)	555 (2)	992 (5)	494 (5)	19 (2)

Gabe's adaptation of the *MULTAN* system (Germain, Main & Woolfson, 1971).

The H-atom positions were calculated where possible or located from selected-area difference electron density maps prepared at an intermediate stage of least-squares refinement of structural parameters. In the final cycles of block-diagonal-matrix least-squares refinement, positional parameters for all atoms, anisotropic thermal vibration parameters for the non-hydrogen atoms and isotropic ones for the H atoms were varied. Using the weighting scheme $w = 1/[\sigma^2(F) + kF_o^2]$ where $\sigma(F)$ was estimated from counting statistics (Corfield, Doedens & Ibers, 1967), and with $k = 0.0075$, refinement converged to $R = 0.051$, $R_w = 0.076$, where $R = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$, for the 1435 reflections. The function minimized by the procedure was $\Sigma w(|F_o| - |F_c|)^2$. The average shift/error for the final cycle was 0.06 with a maximum value of 0.45 associated with the x coordinate of H(20C). A ΔF synthesis showed a maximum electron density of 0.17 e Å^{-3} and a minimum electron density of -0.19 e Å^{-3}, indicating no misplaced or uncounted atoms. The atomic parameters are listed in Table 1.* Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). No absorption correction was attempted because of the low value of μ(Mo $K\alpha$) for an organic compound.

Discussion. Compound (I) exists as a racemic mixture; the crystallographically observed structure of the enantiomer corresponding to the formula given is shown in Fig. 1 and intramolecular bond lengths and angles are given in Tables 2 and 3. The molecule's 26 C–H bonds were found to have distances in the range 0.86 (3) to 1.11 (7) Å with a mean value of 0.98 Å. No short intermolecular contacts were observed.

The bond lengths and angles compare well with average values for a number of Δ^4-3-one structures (Duax, Weeks & Rohrer, 1976). However, in this
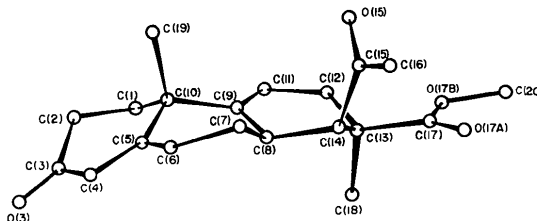

Fig. 1. Perspective view of the molecular structure. The endocyclic torsion angles at the double bonds are C(3)–C(4)–C(5)–C(10) = 3·7(7) and C(8)–C(9)–C(11)–C(12) = 3·3(6)°.

Table 2. *Bond lengths* (Å) *for non-hydrogen atoms*
(estimated standard deviations in parentheses)

C(1)—C(2)	1·529 (6)	C(9)—C(11)	1·326 (4)
C(1)—C(10)	1·540 (5)	C(10)—C(19)	1·551 (5)
C(2)—C(3)	1·504 (7)	C(11)—C(12)	1·491 (5)
C(3)—C(4)	1·447 (5)	C(12)—C(13)	1·532 (4)
C(3)—O(3)	1·219 (5)	C(13)—C(14)	1·543 (4)
C(4)—C(5)	1·331 (5)	C(13)—C(17)	1·517 (4)
C(5)—C(6)	1·502 (5)	C(13)—C(18)	1·547 (4)
C(5)—C(10)	1·530 (4)	C(14)—C(15)	1·527 (4)
C(6)—C(7)	1·519 (5)	C(15)—C(16)	1·498 (5)
C(7)—C(8)	1·522 (4)	C(15)—O(15)	1·209 (4)
C(8)—C(9)	1·519 (4)	C(17)—O(17A)	1·194 (4)
C(8)—C(14)	1·542 (4)	C(17)—O(17B)	1·329 (4)
C(9)—C(10)	1·534 (4)	O(17B)—C(20)	1·452 (4)

Table 3. *Bond angles* (°) *for non-hydrogen atoms*
(estimated standard deviations in parentheses)

C(2)—C(1)—C(10)	113·2 (3)	C(5)—C(10)—C(19)	106·8 (3)
C(1)—C(2)—C(3)	109·9 (4)	C(1)—C(10)—C(19)	110·4 (3)
C(2)—C(3)—C(4)	114·5 (3)	C(9)—C(11)—C(12)	126·3 (3)
C(2)—C(3)—O(3)	122·4 (4)	C(11)—C(12)—C(13)	113·0 (3)
C(4)—C(3)—O(3)	123·0 (4)	C(12)—C(13)—C(14)	110·7 (2)
C(3)—C(4)—C(5)	125·4 (4)	C(12)—C(13)—C(17)	111·3 (2)
C(4)—C(5)—C(6)	121·4 (3)	C(12)—C(13)—C(18)	109·9 (3)
C(4)—C(5)—C(10)	122·1 (3)	C(14)—C(13)—C(17)	108·8 (2)
C(10)—C(5)—C(6)	116·5 (3)	C(14)—C(13)—C(18)	109·9 (2)
C(5)—C(6)—C(7)	114·8 (3)	C(17)—C(13)—C(18)	106·1 (3)
C(6)—C(7)—C(8)	111·8 (3)	C(13)—C(14)—C(8)	111·6 (2)
C(7)—C(8)—C(9)	110·7 (2)	C(13)—C(14)—C(15)	113·1 (2)
C(7)—C(8)—C(14)	113·7 (3)	C(8)—C(14)—C(15)	111·6 (2)
C(9)—C(8)—C(14)	112·6 (2)	C(14)—C(15)—C(16)	118·2 (3)
C(8)—C(9)—C(10)	117·5 (2)	C(14)—C(15)—O(15)	121·9 (3)
C(8)—C(9)—C(11)	120·8 (3)	O(15)—C(15)—C(16)	119·9 (3)
C(10)—C(9)—C(11)	121·6 (3)	C(13)—C(17)—O(17A)	124·7 (3)
C(9)—C(10)—C(5)	110·6 (2)	C(13)—C(17)—O(17B)	112·6 (3)
C(9)—C(10)—C(1)	109·3 (3)	O(17A)—C(17)—O(17B)	122·6 (3)
C(9)—C(10)—C(19)	109·6 (3)	C(17)—O(17B)—C(20)	117·2 (3)
C(5)—C(10)—C(1)	110·3 (3)		

compound C(9) is sp^2 hybridized which gives rise to a shortening of the adjacent bonds [C(9)—C(10), 1·534(4); C(9)—C(8), 1·519(4) Å] and an increase in the bond angles around C(9).

The C(3)—C(4) bond distance of 1·447 (5) Å is short for a formal single bond, but both C(3) and C(4) are involved in π bonding and the torsion angle defined by O(3), C(3), C(4), C(5) is −172·0° which would indicate some delocalization of the π system along C(3)—C(4). The review by Duax *et al.* corroborates a shorter C(3)—C(4) bond distance in similar compounds, but gives a mean value of 1·455 (3) Å which is a less marked contraction than in compound (I). However, thermal parameters for the atoms in ring A of our structure are noticeably higher than those for the remainder of the structure, and this will also cause some foreshortening of bond lengths.

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Refinement of the Structure of *N*-(2-Hydroxyethyl)taurine

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Abstract. $C_4H_{11}NO_4S$, $M_r = 169·2$, orthorhombic, *Pbca*, $a = 9·675$ (3), $b = 11·636$ (4), $c = 12·752$ (6) Å, $V = 1435·6$ Å³, $Z = 8$, $D_m = 1·560$, $D_c = 1·566$ Mg m^{-3}, μ(Mo $K\alpha$) = 0·403 mm^{-1}. $R = 0·036$ for 2114 reflections. Crystals consist of $HOCH_2CH_2NH_2^+$-$CH_2CH_2SO_3^-$ zwitterions connected by hydrogen bonds of 2·731 (2), 2·812 (2) and 2·855 (2) Å. Torsion angles S—C—C—N and N—C—C—O are 176·1 (1) and −58·7 (2)°.

Introduction. The structure of the title compound obtained on the basis of film data with an anisotropic refinement of the non-hydrogen atoms ($R = 0·120$) was reported by Galešić, Herceg, Matković, Šljukić, Trupčević & Zelenko (1974). In the present work diffractometer data are used, H atom positions determined and more precise bond lengths and angles reported.

Intensities were collected from a crystal ground to a