estimated standard deviations were all less than 0·3. The refinement was based on F_o, the quantity minimized being $\Sigma w(F_o - F_c)^2$. The weighting scheme used was based on counter statistics as defined by Corfield, Doedens & Ibers (1967); the value of p was 0·04. The scattering factors used were those of Hanson, Herman, Lea & Skillman (1964).*

Discussion. The final atomic coordinates and thermal parameters are given in Table 1. The standard deviations for atomic positions were taken from the least-squares matrix. Bond lengths and angles including those after thermal corrections (in square brackets) and estimated standard deviations (in parentheses) are given on the thermal ellipsoid plot of the molecule in Fig. 1. The molecule is virtually planar, with a maximum deviation of 0·04 Å from the least-squares plane. Fig. 2 shows the crystal packing where stacking of the benzene rings is observed. Though chemical equivalency allows for a mirror plane through O(9) bisecting bonds C(1)—C(2) and C(5)—C(4), no such plane is used in the crystal. Unlike tetrachlorophthalic anhydride (Rudman, 1971) and the perylene complex of pyromellitic dianhydride (Boeyens & Herbstein, 1965), where large discrepancies between the pairs of C=O and C—O

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32258 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

bonds in each molecule were reported, only small differences in bond lengths between chemically equivalent atoms in phthalic anhydride are noticeable. In this respect, phthalic anhydride is like the pyrene–pyromellitic dianhydride complex at 110K (Herbstein & Snyman, 1969).

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(25R)-Cholest-5-ene-3β,26-diol

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Abstract. $C_{27}H_{46}O_2$, orthorhombic, $P2_12_12_1$, $a = 19·182 (3)$, $b = 22·062 (5)$, $c = 11·605 (3)$ Å, $V = 4911 (2)$ Å³, $Z = 8$, $D_x = 1·089$ g cm⁻³. The analysis establishes the configuration at C(25) as (25R). The crystal is built up of molecules connected by hydrogen bonds, forming infinite chains running along **a**.

Introduction. The specimen was a clear, thin needle cut to 0·1 × 0·1 × 0·1 mm. Systematic absences were $h00$, h odd: $0k0$, k odd and $00l$, l odd. Intensities were measured in the θ–2θ mode on an automatic Syntex $P2_1$ four-circle diffractometer with Mo $K\alpha$ radiation ($\lambda = 0·71069$ Å) monochromatized by a graphite crystal.

With a 2° plus (α_1,α_2)-dispersion scan range for positive h,k,l and a maximum $2\theta = 45°$, 3595 unique reflexions were recorded of which 2349 were regarded as unobserved ($I < 3\sigma$) due to the small sample size and the high overall temperature factor ($B = 3\cdot9$ Å^2). No absorption correction was applied ($\mu = 0\cdot686$ cm^{-1}).

The structure (58 heavy atoms) was partially solved with *MULTAN* (Germain, Main & Woolfson, 1971). From 300 $|E|$'s $\geq 1\cdot6$, 2000 Σ_2 relationships, $\mathscr{H}_{hh'} \geq 7\cdot27$, a fragment of one molecule containing three six-membered rings could be recognized. The recycling procedure based on this fragment did not enlarge it. However, on insertion into *MULTAN* of those 23 phases with the highest α values obtained from the recycling procedure as known phases, a subsequent run clearly showed the corresponding fragment of the other molecule. At this stage 36 atoms had been found. The 22 missing atoms could be located from inspection of a difference map at $R = 0\cdot38$. Refinement was by full-matrix least squares with anisotropic temperature factors. Owing to the large number of parameters (524), refinement had to take place in overlapping cycles. H atoms were ignored. The reflexions were weighted with an adjustable Cruickshank scheme, the final weights being $w = 0\cdot28$ for $|F_o| \geq 50\cdot0$ and $w = 0\cdot00012|F_o|^2$ for $|F_o| < 50\cdot0$. An isotropic extinction factor (Zachariasen, 1963) was included (final value 6×10^{-7}). The refinement was stopped when the parameter shifts were well below the standard deviations. The final R_w was $0\cdot073$ or $0\cdot060$ with unobserved reflexions omitted.*

Discussion. For investigations on the biogenesis of spirostanole, (25R)-26-hydroxycholest-4-en-3-one was required. This compound could be synthesized from diosgenin (Brennecke, Saito, Töpfer & Tschesche, 1977). The (25S) compound was obtained earlier (Zaretskaya

et al., 1968; Schubert, Kaufmann & Budzikiewicz, 1969) and the configuration at C(25) was established by X-ray methods (Duchamp, Chidester, Wickramasinghe, Caspi & Yagen, 1971). Since the optical

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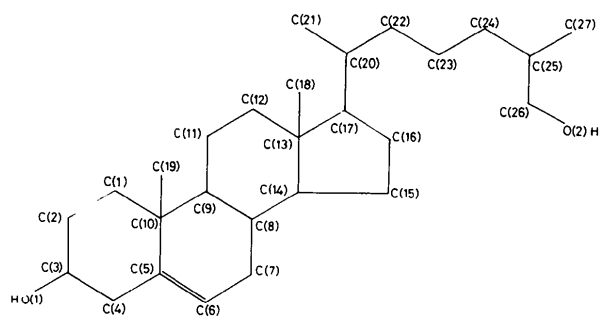

Fig. 1. Numbering scheme of atoms.

Table 1. *Positional parameters* ($\times 10^4$) *with standard deviations in parentheses*

	x	y	z
O(1)	1724 (4)	6064 (3)	3212 (8)
C(1)	9834 (6)	6100 (6)	4233 (11)
C(2)	637 (6)	6061 (6)	4345 (12)
C(3)	942 (6)	6176 (7)	3141 (13)
C(4)	819 (6)	6861 (6)	2831 (13)
C(5)	55 (7)	6951 (6)	2667 (12)
C(6)	9763 (7)	7215 (5)	1749 (12)
C(7)	8993 (5)	7362 (6)	1572 (11)
C(8)	8532 (5)	7268 (5)	2675 (10)
C(9)	8802 (5)	6656 (5)	3290 (10)
C(10)	9587 (6)	6740 (6)	3777 (11)
C(11)	8305 (6)	6483 (6)	4288 (10)
C(12)	7549 (6)	6373 (5)	3826 (10)
C(13)	7260 (6)	6937 (5)	3180 (11)
C(14)	7798 (5)	7102 (6)	2218 (11)
C(15)	7430 (6)	7589 (6)	1543 (11)
C(16)	6644 (6)	7378 (5)	1537 (11)
C(17)	6605 (7)	6858 (5)	2455 (11)
C(18)	7170 (6)	7489 (6)	4104 (10)
C(19)	9621 (6)	7241 (5)	4722 (11)
C(20)	5875 (6)	6850 (5)	3081 (12)
C(21)	5857 (6)	6376 (6)	4107 (10)
C(22)	5354 (6)	6689 (5)	2079 (11)
C(23)	4570 (6)	6603 (6)	2595 (11)
C(24)	4141 (6)	6491 (6)	1490 (9)
C(25)	3352 (6)	6470 (5)	1909 (11)
C(26)	2976 (6)	6221 (5)	763 (13)
C(27)	3053 (7)	7081 (6)	2246 (12)
O(2)	2231 (5)	6206 (4)	970 (8)
O(1')	1622 (4)	5724 (4)	9060 (8)
C(1')	9710 (5)	5916 (5)	9802 (6)
C(2')	522 (5)	6056 (5)	9815 (10)
C(3')	869 (6)	5534 (8)	9011 (13)
C(4')	643 (6)	5663 (6)	7744 (13)
C(5')	9824 (5)	5563 (4)	7672 (10)
C(6')	9548 (5)	5179 (5)	6917 (11)
C(7')	8751 (6)	5043 (6)	6786 (12)
C(8')	8362 (6)	5582 (6)	7283 (13)
C(9')	8618 (5)	5736 (5)	8556 (9)
C(10')	9406 (5)	5980 (5)	8507 (10)
C(11')	8153 (5)	6210 (5)	9241 (11)
C(12')	7367 (5)	6029 (6)	9230 (12)
C(13')	7159 (5)	5886 (5)	7953 (11)
C(14')	7600 (5)	5393 (5)	7434 (10)
C(15')	7237 (6)	5138 (6)	6383 (11)
C(16')	6420 (6)	5213 (5)	6713 (10)
C(17')	6363 (6)	5568 (5)	7853 (14)
C(18')	7118 (6)	6498 (5)	7176 (11)
C(19')	9476 (6)	6650 (4)	8078 (10)
C(20')	5726 (6)	5984 (5)	7937 (11)
C(21')	5637 (6)	6298 (5)	9140 (11)
C(22')	5059 (6)	5500 (6)	7736 (12)
C(23')	4421 (6)	5917 (6)	7372 (14)
C(24')	3809 (6)	5465 (6)	7111 (14)
C(25')	3144 (7)	5833 (8)	6589 (19)
C(26')	2554 (10)	5335 (8)	6498 (16)
C(27')	3320 (8)	6074 (6)	5400 (13)
O(2')	2429 (6)	5073 (5)	7579 (11)

activities of the two diastereomers are close to the limits of accuracy of determination (Duchamp *et al.*, 1971; Varma, Koreeda, Yagen, Nakanishi & Caspi, 1975), the (25R) form of the above compound could not be established unambiguously. Hence the configuration at C(25) had to be investigated by X-ray structure analysis. Owing to the better crystallization, the intermediate cholest-5-ene-3β,26-diol (Fig. 1) was used which is important for other subsequent syntheses.

Tables 1 and 2 give the final fractional atomic coordinates, bond distances, and angles of the molecules I and I'. The C—C single bonds vary from 1·50 to 1·68 Å with a mean of 1·57 (2) Å. These results are probably due to omission of the H atoms. However, C(5)—C(6), 1·34 (2), and C(5')—C(6'), 1·33 (2) Å, clearly establish them as C=C double bonds and hence confirm the chemical information.

Fig. 2. Stereoscopic view of the two molecules of the asymmetric unit.

Fig. 3. Stereoscopic view of the unit cell along c.

The C—O single bonds vary from 1·40 to 1·52 Å with a mean of 1·47 (2) Å, close to the expected value. An *ORTEP* drawing (Johnson, 1965) of the two molecules of the asymmetric unit is shown in Fig. 2. The molecules are tilted against each other at about 90°. The shortest distances between adjacent molecules are O(1)···O(2ⁱ), 3·08 (1), and O(2)···O(1ⁱⁱ), 2·72 (1) Å. These distances correspond to hydrogen bonds. Thus the structure can be described as consisting of infinite chains of molecules I and I' running along **a**. A stereoscopic view of the arrangement of the molecules in the unit cell, approximately along |001|, is shown in Fig. 3.

After completion of the refinement a final difference map showed no traces of the (25S) form. Hence the configuration of the title compound could be established unambiguously as (25R).

Table 2. *Interatomic distances* (Å) *and angles* (°) *for the molecules* I *and* I' *of the asymmetric unit*

The standard deviations are 0·02 Å and 1·0° respectively.

	I	I'		I	I'
C(1)—C(2)	1·55	1·59	C(12)—C(13)	1·55	1·57
C(1)—C(10)	1·58	1·62	C(13)—C(14)	1·56	1·50
C(2)—C(3)	1·54	1·62	C(13)—C(18)	1·63	1·63
C(3)—O(1)	1·52	1·50	C(13)—C(17)	1·52	1·68
C(3)—C(4)	1·57	1·56	C(14)—C(15)	1·50	1·51
C(4)—C(5)	1·49	1·59	C(15)—C(16)	1·58	1·62
C(5)—C(10)	1·64	1·56	C(16)—C(17)	1·57	1·54
C(5)—C(6)	1·34	1·33	C(17)—C(20)	-1·58	1·53
C(6)—C(7)	1·53	1·56	C(20)—C(21)	1·58	1·57
C(7)—C(8)	1·57	1·52	C(20)—C(22)	1·57	1·68
C(8)—C(9)	1·61	1·59	C(22)—C(23)	1·63	1·59
C(8)—C(14)	1·55	1·53	C(23)—C(24)	1·54	1·57
C(9)—C(11)	1·55	1·59	C(24)—C(25)	1·59	1·63
C(9)—C(10)	1·62	1·61	C(25)—C(27)	1·52	1·52
C(10)—C(19)	1·56	1·56	C(25)—C(26)	1·61	1·58
C(11)—C(12)	1·56	1·56	C(26)—O(2)	1·45	1·40

	I	I'		I	I'
C(2)—C(1)—C(10)	112	110	C(14)—C(13)—C(17)	100	100
C(1)—C(2)—C(3)	107	105	C(17)—C(13)—C(18)	111	105
O(1)—C(3)—C(4)	108	105	C(12)—C(13)—C(14)	107	112
O(1)—C(3)—C(2)	107	100	C(12)—C(13)—C(18)	108	112
C(2)—C(3)—C(4)	108	107	C(12)—C(13)—C(17)	118	112
C(3)—C(4)—C(5)	108	107	C(14)—C(13)—C(18)	111	114
C(4)—C(5)—C(10)	114	113	C(8)—C(14)—C(13)	114	113
C(4)—C(5)—C(6)	125	121	C(8)—C(14)—C(15)	116	117
C(6)—C(5)—C(10)	121	125	C(13)—C(14)—C(15)	103	109
C(5)—C(6)—C(7)	127	125	C(14)—C(15)—C(16)	104	103
C(6)—C(7)—C(8)	114	107	C(15)—C(16)—C(17)	105	109
C(7)—C(8)—C(9)	107	111	C(13)—C(17)—C(16)	105	102
C(7)—C(8)—C(14)	105	107	C(13)—C(17)—C(20)	119	118
C(9)—C(8)—C(14)	104	104	C(16)—C(17)—C(20)	111	114
C(8)—C(9)—C(10)	111	109	C(17)—C(20)—C(22)	103	103
C(8)—C(9)—C(11)	110	115	C(17)—C(20)—C(21)	112	114
C(10)—C(9)—C(11)	110	109	C(21)—C(20)—C(22)	113	109
C(1)—C(10)—C(5)	110	110	C(20)—C(22)—C(23)	110	105
C(1)—C(10)—C(9)	107	106	C(22)—C(23)—C(24)	102	105
C(1)—C(10)—C(19)	113	110	C(23)—C(24)—C(25)	105	110
C(5)—C(10)—C(9)	106	108	C(24)—C(25)—C(27)	114	110
C(5)—C(10)—C(19)	109	108	C(24)—C(25)—C(26)	101	104
C(9)—C(10)—C(19)	111	114	C(26)—C(25)—C(27)	110	110
C(9)—C(11)—C(12)	111	112	C(25)—C(26)—O(2)	108	110
C(11)—C(12)—C(13)	112	108			

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