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Lobosterol 4-*p*-Bromobenzoate

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Abstract. (24*S*)-24-Methylcholestane-3β,4β,5β,25-tetrol-6-one 4-*p*-bromobenzoate 25-monoacetate; monoclinic, $P2_1$; $a=20\cdot219$ (3), $b=13\cdot320$ (3), $c=6\cdot684$ (2) Å, $\beta=90\cdot30$ (2)°; $C_{37}O_7H_{53}Br$, $Z=2$, $D_x=1\cdot27$ g cm^{-3}.

Introduction. Intensities from a crystal, $0\cdot50\times0\cdot25\times0\cdot10$ mm, were collected on a four-circle PW1100 automatic diffractometer. The measurement of half the Cu sphere up to a Bragg angle of 60° yielded 2815 unique reflexions which were corrected for the Lp factor but not for absorption.

The structure was solved with *MULTAN*74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974) from the 230 highest E values and their 2000 strongest $\sum_2$ relationships. The electron density map corresponding to the solution of highest combined figure of merit revealed a five-atom fragment from which the entire structure was developed by successive structure-factor and Fourier calculations. Least-squares refinement

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Table 1. *Final positional* ($\times 10^4$) *and thermal parameters* ($\times 10^4$) *for lobosterol* 4-*p-bromobenzoate*

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br	3461 (1)	5662 (2)	−3112 (2)	642	845	1050	127	176	−172
C(1)	−388 (5)	10436 (8)	3014 (16)	533	331	483	−33	66	−4
C(2)	96 (6)	10157 (9)	4632 (14)	593	467	345	−125	−20	−93
C(3)	703 (5)	9606 (10)	3848 (14)	538	577	386	−151	−38	−32
C(4)	473 (5)	8687 (8)	2589 (14)	416	389	357	−19	−7	96
C(5)	−3 (4)	9004 (7)	924 (13)	389	321	296	−46	6	74
C(6)	−218 (4)	8077 (8)	−273 (12)	462	315	325	61	28	−25
C(7)	−637 (6)	7327 (9)	809 (16)	539	324	426	−22	115	−49
C(8)	−1244 (4)	7828 (8)	1754 (13)	325	361	359	8	28	−55
C(9)	−1045 (4)	8763 (7)	3006 (13)	420	267	317	22	−27	−24
C(10)	−625 (5)	9537 (8)	1751 (14)	511	305	371	45	24	13
C(11)	−1643 (5)	9229 (9)	4052 (16)	505	383	512	18	80	−15
C(12)	−2047 (5)	8450 (9)	5316 (14)	451	504	376	−79	66	−87
C(13)	−2241 (4)	7548 (8)	4094 (12)	327	391	347	37	29	30
C(14)	−1607 (4)	7105 (8)	3143 (13)	359	390	411	15	1	−31
C(15)	−1806 (5)	6081 (9)	2304 (18)	482	430	586	−13	69	−68
C(16)	−2328 (5)	5692 (11)	3856 (16)	438	517	639	−23	109	−19
C(17)	−2473 (4)	6598 (8)	5310 (12)	434	444	390	−29	−13	20
C(18)	−2762 (5)	7832 (10)	2456 (17)	480	554	530	70	−37	3
C(19)	−1045 (5)	9951 (9)	−13 (16)	507	502	535	43	38	101
C(20)	−3187 (4)	6531 (9)	6113 (13)	402	589	430	5	46	24
C(21)	−3420 (6)	7501 (11)	7245 (19)	583	707	621	−35	68	59
C(22)	−3218 (5)	5619 (13)	7525 (15)	422	682	587	−54	22	113
C(23)	−3917 (5)	5307 (8)	8170 (16)	462	589	638	40	22	68
C(24)	−3901 (5)	4391 (9)	9583 (17)	467	639	531	58	34	99
C(25)	−4654 (5)	4256 (9)	10733 (16)	550	612	771	−33	169	166
C(26)	−4490 (7)	3415 (11)	12362 (21)	1274	672	954	201	459	363
C(27)	−5150 (5)	4131 (11)	9399 (17)	561	802	1123	−56	160	−71
C(28)	−3690 (6)	3442 (10)	8565 (17)	777	775	840	134	188	87
C(29)	−5122 (5)	5426 (11)	12967 (15)	725	894	796	79	222	228
C(30)	−5044 (8)	6494 (11)	13907 (19)	923	770	964	75	157	−11

Table 1 (*cont.*)

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(31)	1417 (5)	7590 (9)	2505 (16)	447	571	523	−2	−10	54
C(32)	1942 (4)	7154 (8)	1159 (16)	457	491	543	−44	−30	22
C(33)	1973 (5)	7442 (9)	−828 (14)	504	675	605	112	−45	91
C(34)	2440 (5)	7000 (10)	−2092 (14)	588	790	589	178	18	62
C(35)	2855 (4)	6294 (9)	−1278 (16)	418	603	793	20	43	−50
C(36)	2823 (5)	5990 (8)	659 (15)	508	496	727	16	−37	10
C(37)	2366 (4)	6449 (9)	1898 (16)	463	520	703	−25	−67	60
O(1)	−4610 (3)	5217 (5)	11851 (10)	661	546	658	28	140	64
O(2)	1351 (3)	7368 (6)	4264 (9)	781	899	571	276	97	214
O(3)	1129 (4)	10423 (7)	2765 (11)	564	665	661	−190	18	4
O(4)	1022 (3)	8228 (6)	1528 (9)	437	575	415	66	21	118
O(5)	347 (3)	9653 (5)	−423 (9)	512	367	336	−37	54	72
O(6)	−61 (3)	8007 (5)	−2022 (8)	780	464	415	−44	109	−72
O(7)	−5586 (4)	4908 (9)	13356 (15)	1065	1010	1446	−32	599	−59

Table 1 (*cont.*)

	x	y	z	U
H(1*A*)	−885	10750	3230	752
H(1*B*)	0	10750	2126	1134
H(2*A*)	258	10750	5388	656
H(2*B*)	−90	9750	5497	958
H(3)	988	9338	4949	656
H(4)	236	8000	3379	521
H(7*A*)	−417	7000	1700	54
H(7*B*)	−774	6857	−148	321
H(8)	−1591	8084	1158	251
H(9)	−807	8552	3864	3
H(11*A*)	−1916	9500	3160	141
H(11*B*)	−1576	9750	4669	1187
H(12*A*)	−1751	8500	6156	98
H(12*B*)	−2485	8837	5928	458
H(14)	−1313	6928	3991	208
H(15*A*)	−1970	6230	1220	399
H(15*B*)	−1442	5757	2326	488
H(16*A*)	−2729	5522	3131	322
H(16*B*)	−2135	5037	4701	430
H(17)	−2290	6750	6550	528
H(18*A*)	−2795	7266	2004	432
H(18*B*)	−2520	8250	1830	807
H(18*C*)	−3200	7750	2700	1432
H(19*A*)	−676	10484	−774	727
H(19*B*)	−1370	10500	318	696
H(19*C*)	−1170	9250	−580	1297
H(20)	−3506	6500	4792	384
H(21*A*)	−3099	7566	7954	135
H(21*B*)	−3421	8078	6143	578
H(21*C*)	−3880	7250	7500	846
H(22*A*)	−2923	5750	8895	731
H(22*B*)	−2990	5000	6910	1080
H(23*A*)	−4143	5188	7023	589
H(23*B*)	−4200	5750	8530	754
H(24)	−3618	4548	10575	440
H(26*A*)	−4918	3250	13127	931
H(26*B*)	−4420	2750	11100	877
H(26*C*)	−3976	3262	12710	1029
H(27*A*)	−5504	4000	10319	645
H(27*B*)	−5082	3741	8523	679
H(27*C*)	−5200	4680	8880	288
H(28*A*)	−3921	3329	7430	465
H(28*B*)	−3200	3500	7810	1173
H(28*C*)	−3610	2750	9940	1359
H(30*A*)	−4650	6870	13310	841
H(30*B*)	−5470	6480	13900	628
H(30*C*)	−4905	6363	15554	654
H(33)	1639	7963	−1314	523
H(34)	2551	7250	−3457	553
H(36)	3246	5500	1521	1250
H(37)	2329	6000	3053	1413
H(O3)	1021	10250	1518	1246

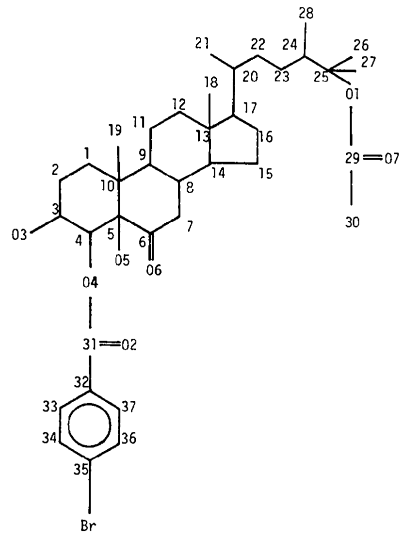

Fig. 1. The molecule of lobosterol 4-*p*-bromobenzoate.

Table 2. *Interatomic distances* (Å), *angles and torsion angles* (°) *for the lobosteryl part of the molecule*

C(1)—C(2)	1·502 (15)	C(13)–C(14)	1·549 (13)
–C(10)	1·541 (15)	–C(17)	1·577 (15)
C(2)—C(3)	1·526 (17)	–C(18)	1·562 (14)
C(3)—C(4)	1·555 (16)	C(14)–C(15)	1·528 (16)
–O(3)	1·412 (14)	C(15)–C(16)	1·571 (16)
C(4)—C(5)	1·527 (13)	C(16)–C(17)	1·578 (17)
–O(4)	1·455 (12)	C(17)–C(20)	1·545 (13)
C(5)—C(6)	1·532 (14)	C(20)–C(21)	1·571 (18)
–C(10)	1·548 (14)	–C(22)	1·540 (19)
–O(5)	1·438 (11)	C(22)–C(23)	1·536 (15)
C(6)—C(7)	1·499 (15)	C(23)–C(24)	1·543 (16)
–O(6)	1·216 (11)	C(24)–C(25)	1·559 (15)
C(7)—C(8)	1·536 (15)	–C(28)	1·498 (18)
C(8)—C(9)	1·552 (14)	C(25)–C(26)	1·568 (18)
–C(14)	1·528 (14)	–C(27)	1·489 (16)
C(9)—C(10)	1·580 (14)	–O(1)	1·485 (14)
–C(11)	1·531 (15)	C(29)–C(30)	1·562 (21)
C(10)–C(19)	1·551 (15)	–O(1)	1·309 (13)
C(11)–C(12)	1·570 (16)	–O(7)	1·195 (16)
C(12)–C(13)	1·503 (15)		

Table 2 (*cont.*)

C(2)—C(1)—C(10)	113·6 (9)	C(8)—C(9)—C(11)	111·6 (8)	C(13)-C(17)-C(16)	103·8 (7)
C(1)—C(2)—C(3)	113·2 (8)	C(10)-C(9)—C(11)	114·0 (8)	C(13)-C(17)-C(20)	120·5 (8)
C(2)—C(3)—C(4)	109·0 (9)	C(1)—C(10)-C(5)	107·5 (8)	C(16)-C(17)-C(20)	110·3 (8)
C(2)—C(3)—O(3)	112·4 (10)	C(1)—C(10)-C(9)	112·5 (8)	C(17)-C(20)-C(21)	113·7 (9)
C(4)—C(3)—O(3)	112·2 (8)	C(1)—C(10)-C(19)	107·9 (9)	C(17)-C(20)-C(22)	107·5 (8)
C(3)—C(4)—C(5)	111·2 (9)	C(5)—C(10)-C(9)	109·3 (8)	C(21)-C(20)-C(22)	109·9 (8)
C(3)—C(4)—O(4)	111·5 (8)	C(5)—C(10)-C(19)	109·5 (8)	C(20)-C(22)-C(23)	115·2 (9)
C(5)—C(4)—O(4)	103·9 (7)	C(9)—C(10)-C(19)	109·9 (8)	C(22)-C(23)-C(24)	111·7 (9)
C(4)—C(5)—C(6)	109·5 (8)	C(9)—C(11)-C(12)	113·0 (9)	C(23)-C(24)-C(25)	112·2 (9)
C(4)—C(5)—C(10)	112·1 (7)	C(11)-C(12)-C(13)	111·7 (8)	C(23)-C(24)-C(28)	113·2 (9)
C(4)—C(5)—O(5)	108·1 (7)	C(12)-C(13)-C(14)	108·3 (7)	C(25)-C(24)-C(28)	112·0 (10)
C(6)—C(5)—C(10)	109·1 (7)	C(12)-C(13)-C(17)	116·0 (7)	C(24)-C(25)-C(26)	110·2 (9)
C(6)—C(5)—O(5)	107·2 (7)	C(12)-C(13)-C(18)	111·1 (9)	C(24)-C(25)-C(27)	113·6 (9)
C(10)-C(5)—O(5)	110·6 (8)	C(14)-C(13)-C(17)	98·9 (8)	C(24)-C(25)-O(1)	101·9 (8)
C(5)—C(6)—C(7)	116·3 (7)	C(14)-C(13)-C(18)	111·2 (7)	C(26)-C(25)-C(27)	114·1 (10)
C(5)—C(6)—O(6)	119·3 (9)	C(17)-C(13)-C(18)	110·7 (8)	C(26)-C(25)-O(1)	105·7 (9)
C(7)—C(6)—O(6)	124·2 (9)	C(8)—C(14)-C(13)	114·2 (8)	C(27)-C(25)-O(1)	110·2 (9)
C(6)—C(7)—C(8)	111·3 (9)	C(8)—C(14)-C(15)	117·8 (8)	C(30)-C(29)-O(1)	110·2 (10)
C(7)—C(8)—C(9)	111·4 (8)	C(13)-C(14)-C(15)	105·9 (8)	C(30)-C(29)-O(7)	121·1 (11)
C(7)—C(8)—C(14)	111·3 (9)	C(14)-C(15)-C(16)	103·2 (9)	O(1)—C(29)-O(7)	128·6 (13)
C(9)—C(8)—C(14)	107·6 (7)	C(15)-C(16)-C(17)	106·4 (10)	C(25)-O(1)—C(29)	121·5 (9)
C(8)—C(9)—C(10)	112·0 (7)				

C(1)—C(2)—C(3)—C(4)	−54·1	C(10)-C(9)—C(8)—C(14)	176·6	C(13)-C(14)-C(15)-C(16)	−36·2
C(2)—C(3)—C(4)—C(5)	54·4	C(11)-C(9)—C(8)—C(7)	−176·5	C(14)-C(15)-C(16)-C(17)	9·9
C(3)—C(4)—C(5)—C(10)	−58·0	C(12)-C(13)-C(14)-C(15)	168·7	C(15)-C(16)-C(17)-C(13)	19·0
C(4)—C(5)—C(10)-C(1)	56·1	C(17)-C(13)-C(14)-C(8)	178·3	C(16)-C(17)-C(13)-C(14)	−39·6
C(5)—C(10)-C(1)—C(2)	−55·0	C(19)-C(10)-C(9)—C(11)	−65·8	C(17)-C(13)-C(14)-C(15)	47·3
C(10)-C(1)—C(2)—C(3)	56·5	C(19)-C(10)-C(9)—C(8)	63·8	C(19)-C(10)-C(5)—C(6)	−65·8
C(10)-C(5)—C(6)—C(7)	−55·5	C(19)-C(10)-C(1)—C(2)	−172·2	C(18)-C(13)-C(17)-C(20)	−48·1
C(5)—C(6)—C(7)—C(8)	52·5	C(19)-C(10)-C(5)—C(4)	172·4	C(18)-C(13)-C(17)-C(16)	78·0
C(6)—C(7)—C(8)—C(9)	−49·8	C(8)—C(9)—C(11)-C(12)	50·5	C(18)-C(13)-C(12)-C(11)	−69·3
C(7)—C(8)—C(9)—C(10)	53·2	C(9)—C(11)-C(12)-C(13)	−51·6	C(18)-C(13)-C(14)-C(8)	62·1
C(8)—C(9)—C(10)-C(5)	−56·0	C(11)-C(12)-C(13)-C(14)	53·9	C(18)-C(13)-C(14)-C(15)	−68·9
C(9)—C(10)-C(5)—C(6)	55·0	C(12)-C(13)-C(14)-C(8)	−60·2	C(20)-C(17)-C(13)-C(12)	79·1
C(4)—C(5)—C(10)-C(9)	−66·7	C(13)-C(14)-C(8)—C(9)	59·2	C(20)-C(17)-C(13)-C(14)	−165·7
C(6)—C(5)—C(10)-C(1)	177·8	C(14)-C(8)—C(9)—C(11)	−53·2	C(20)-C(17)-C(16)-C(15)	−150·7

allowed the O atoms to be distinguished without ambiguity and a difference synthesis revealed all but one H atom.

Except for the Br atom, the anisotropy of the thermal motion was not taken into account until most H atoms were found, these being given their parent C or O atom isotropic temperature factor.

One cycle of least-squares refinement was then carried out on the H temperature factors only (isotropic) and two more cycles on all non-hydrogen atoms (anisotropic). The final R is 6·5%.*

Table 1 lists the final parameters, Table 2 the interatomic distances, angles and torsion angles; Fig. 1 is a schematic diagram of the molecule.

Discussion. The original compound (lobosterol, $C_{30}O_6H_{50}$) was extracted from the alcyonacean *Lobophytum pauciflorum* collected at Anse Royale, Mahé, Seychelles Islands, as part of a study of the ecology of coral reefs (Tursch, Kaisin, Hootelé, Losman & Karlsson, 1976).

The available quantity of the compound being insufficient for a complete chemical elucidation, the bromobenzoate derivative was crystallized and a crystallographic study undertaken.

The 3,4,5,6-oxidation pattern is unprecedented in natural sterols and the *A/B-cis* ring fusion has not yet been reported for marine sterols. Alcyonaceans seem to be a promising source of the hitherto uncommon polyhydroxylated sterols.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31722 (12 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1 NZ, England.

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