

this diminution in the size of anion would lead to a less stable structure as the cations would then be in contact with each other. In the present compound the empirical formula of the anion $[\text{Cu}_{1.25}\text{Cl}_{4.25}]$ leads to a greater effective volume for the anion which will be much nearer to the volume of the $[\text{CuCl}_5]^{3-}$ ion.

The structure shows clearly how twinning can occur since over 60% of the scattering material (based on f^2) lies on the diagonal (110) plane ($x=y$) conforming to the higher symmetry $Fd\bar{3}m$. Two twin fragments related by reflexion across (110) differ only in the arrangement of the nitrogen atoms and the terminal $\text{Cl}(3)$ atoms, corresponding to rotations of the CuCl_3 groups and $[\text{Co}(\text{NH}_3)_6]^{3+}$ ions. These rotations are unlikely to have much effect on the rest of the structure so that this form of twinning is highly probable.

The structure also gives insight into the possible formulae and structures of the solid solutions formed between $[\text{Co}(\text{NH}_3)_6]\text{CuCl}_5$ and the title compound. A single crystal of an intermediate phase has been in-

vestigated and it is hoped to publish the structure in a future paper.

References

- BARNET, M. T., CRAVEN, B. M., FREEMAN, H. C., KIME, N. E. & IBERS, J. A. (1966). *Chem. Commun.* pp. 307–308.
 BRITTON, D. (1972). *Acta Cryst. A* **28**, 296–297.
 DAY, P. (1968). *J. Chem. Soc. (A)*, pp. 1835–1838.
 IBERS, J. A. (1968). *International Tables for X-ray Crystallography*. Vol. III. Birmingham: Kynoch Press.
International Tables for X-ray Crystallography (1952). Vol. I, p. 343. (1959). Vol. II, p. 95. Birmingham: Kynoch Press.
 MORI, M. (1960). *Bull. Chem. Soc. Japan*, **33**, 985–988.
 MURRAY-RUST, P. (1963). Part II Thesis, Oxford.
 MURRAY-RUST, P., DAY, P. & PROUT, C. K. (1966). *Chem. Commun.* pp. 277–278.
 RAYMOND, K. N., MEEK, D. W. & IBERS, J. A. (1968). *Inorg. Chem.* **7**, 1111–1117.
 ZALKIN, A., FORRESTER, J. D. & TEMPLETON, D. H. (1964). *Acta Cryst.* **17**, 1408–1412.

Acta Cryst. (1973). **B29**, 2566

The Crystal and Molecular Structure of the Diterpenoid Baccatin V, a Naturally Occurring Oxetan with a Taxane Skeleton

BY E. E. CASTELLANO AND O. J. R. HODDER

Chemical Crystallography Laboratory, South Parks Road, Oxford OX1 3QS, England

(Received 12 March 1973; accepted 6 June 1973)

The structure of a new diterpenoid, baccatin V, $\text{C}_{31}\text{H}_{38}\text{O}_{11}$, isolated from *Taxus baccata* L., has been determined by X-ray diffraction techniques. The crystals are orthorhombic, space group $P2_12_1$ with cell dimensions $a = 25.75 \pm 0.01$, $b = 9.31 \pm 0.01$, $c = 12.34 \pm 0.02$ Å and $Z = 4$. The structure was solved by application of the symbolic addition procedure for phase determination. The molecule has been shown to contain an oxetan ring and to be $5\beta,20$ -epoxy- $1\beta,2\alpha,4\alpha,7\alpha,10\beta,13\alpha$ -hexahydroxytax-11-en-9-one $4\alpha,10\beta$ -diacetate 2α -benzoate. The final R value is 9.14% for 2459 observed reflexions, refining positional and anisotropic thermal parameters of non-hydrogen atoms and including 28 hydrogen positions, obtained from a ΔF synthesis, in the final structure-factor calculations.

Introduction

In recent structure investigations on a series of taxane derivatives, a new diterpenoid, baccatin V, $\text{C}_{31}\text{H}_{38}\text{O}_{11}$, was isolated from *Taxus baccata* L. An insufficient amount of substance was available for detailed chemical studies, so an X-ray three-dimensional crystal-structure determination was carried out. The present paper reports on the structural characteristics of the compound. Details of its n.m.r. spectrum have been published elsewhere (Della Casa de Marcano, Halsall, Castellano & Hodder, 1970).

Experimental

Unit-cell dimensions were measured on oscillation and Weissenberg photographs and refined later on a Hilger and Watts linear diffractometer. Crystal data are as follows: $a = 25.75 \pm 0.01$, $b = 9.31 \pm 0.01$, $c = 12.34 \pm 0.02$ Å; $U = 2958.3$ Å³; $D_m = 1.308$ g cm⁻³ (by flotation), $D_c = 1.317$ g cm⁻³; $Z = 4$. Systematic absences, $h00$ when $h \neq 2n$, $k00$ when $k \neq 2n$, and $l00$ when $l \neq 2n$; space group $P2_12_1$.

From a crystal mounted along the c axis 3364 reflexions of the layers zero to 12 were collected on a Hilger

and Watts linear diffractometer with Mo radiation. 2462 of these were taken as significantly above background. Corrections were made for Lorentz and polarization factors but not for absorption or extinction.

Unitary structure factors were derived in the usual way. The overall temperature factor given by the Wilson plot was $B=3.0 \text{ \AA}^2$.

Phase determination and refinement

Table 1. Starting set for the tangent-formula refinement

Origin-fixing reflexions					
<i>h</i>	<i>k</i>	<i>l</i>	<i>E</i>	Phase	
7	4	0	2.966	90°	
23	3	0	2.539	90	
14	0	11	2.513	90	
Enantiomorph selection					
<i>h</i>	<i>k</i>	<i>l</i>	<i>E</i>	Phase	
11	0	2	2.426	0°	
Trial set					
<i>h</i>	<i>k</i>	<i>l</i>	<i>E</i>	Phase	
11	4	8	2.584	90°	
9	4	9	2.385	135	
5	4	7	2.832	-135	
3	2	0	2.629	-90	

Phases were determined by the symbolic addition procedure for non-centrosymmetric crystals (Karle & Karle, 1966) with the aid of a program developed by one us (O.J.R.H.) for the Oxford KDF-9 computer. A preliminary manual generation of the Σ_2 relations suggested that a good development of the phases should result from the starting set shown in Table 1. Four of these reflexions were chosen to define the origin and enantiomorph so that their phases could be fixed (Hauptman & Karle, 1956). The phases of two of the other reflexions were indicated to be close to $\mp\pi/2$ from the manual calculations. Choice of the quadrants for the phases of the two remaining reflexions gave 64 combinations of trial starting-phase sets, each of which

Table 2. Space parameters and anisotropic thermal parameters (all $\times 10^4$) with standard deviations in parentheses

The temperature factor is defined by $T = \exp[-2\pi(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + B_{23}klb^*c^* + B_{13}hla^*c^* + B_{12}hka^*b^*)]$.

	<i>x</i>	<i>y</i>	<i>z</i>	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
O(1)	3513 (2)	3998 (5)	-143 (5)	625 (35)	176 (24)	575 (41)	40 (50)	100 (62)	-131 (50)
O(2)	3469 (2)	2260 (5)	1507 (4)	306 (23)	211 (23)	312 (30)	-103 (40)	54 (41)	53 (38)
O(20)	2662 (2)	3202 (6)	1389 (5)	431 (28)	435 (32)	388 (35)	116 (55)	120 (49)	299 (51)
O(4)	4083 (2)	35 (6)	2213 (5)	411 (27)	373 (28)	337 (34)	-3 (49)	-25 (46)	167 (50)
O(40)	4545 (2)	-1648 (8)	1347 (6)	484 (33)	685 (43)	666 (50)	-175 (79)	-100 (64)	483 (66)
O(5)	3079 (3)	-1577 (7)	2819 (6)	774 (42)	403 (33)	552 (45)	259 (61)	498 (70)	-168 (63)
O(7)	3901 (3)	-2939 (7)	-185 (6)	732 (41)	476 (36)	546 (45)	-286 (63)	-54 (67)	511 (64)
O(9)	2935 (2)	-1458 (7)	-1908 (6)	581 (36)	490 (35)	577 (45)	-176 (64)	-549 (64)	-152 (60)
O(10)	3668 (3)	-419 (7)	-3066 (5)	718 (39)	515 (36)	362 (38)	-273 (60)	-156 (62)	467 (62)
O(100)	4062 (3)	-2513 (8)	-3478 (6)	662 (41)	893 (51)	583 (49)	-798 (83)	-376 (70)	790 (81)
O(13)	5162 (2)	1684 (10)	85 (6)	373 (30)	1257 (66)	498 (47)	253 (92)	-226 (58)	-230 (79)
C(1)	3765 (3)	2628 (8)	-272 (7)	355 (35)	273 (36)	374 (50)	76 (66)	31 (64)	-134 (62)
C(2)	3425 (3)	1626 (7)	459 (6)	253 (29)	221 (32)	269 (42)	-59 (58)	94 (54)	-74 (33)
C(3)	3575 (2)	-11 (7)	545 (6)	195 (27)	221 (32)	329 (42)	-150 (58)	170 (52)	-150 (53)
C(4)	3604 (3)	-489 (8)	1714 (7)	272 (32)	231 (34)	437 (51)	133 (62)	-63 (61)	87 (54)
C(5)	3477 (4)	-2089 (8)	2070 (8)	705 (57)	261 (40)	493 (59)	287 (74)	69 (94)	142 (79)
C(6)	3268 (5)	-3117 (10)	1244 (10)	1038 (83)	288 (48)	696 (80)	12 (92)	167 (127)	-189 (104)
C(7)	3371 (4)	-2660 (8)	116 (9)	715 (59)	214 (38)	565 (65)	-97 (76)	97 (94)	-7 (77)
C(8)	3228 (3)	-1035 (8)	-130 (7)	304 (35)	264 (35)	467 (54)	-70 (68)	171 (66)	-46 (60)
C(9)	3282 (3)	-987 (9)	-1382 (7)	566 (48)	365 (42)	262 (49)	-230 (70)	-381 (71)	250 (75)
C(10)	3790 (3)	-590 (10)	-1938 (8)	398 (40)	565 (51)	322 (51)	-240 (82)	67 (69)	343 (77)
C(11)	4048 (3)	747 (9)	-1557 (7)	358 (38)	448 (45)	334 (50)	35 (72)	208 (67)	58 (71)
C(12)	4535 (3)	734 (11)	-1172 (7)	247 (34)	709 (60)	318 (52)	-34 (83)	189 (60)	-33 (77)
C(13)	4739 (3)	2030 (13)	-595 (9)	299 (38)	921 (75)	537 (65)	123 (115)	30 (74)	-365 (93)
C(14)	4316 (3)	2720 (10)	108 (7)	398 (40)	623 (56)	319 (52)	-95 (84)	28 (68)	-515 (82)
C(15)	3728 (3)	2141 (9)	-1485 (7)	434 (40)	372 (42)	311 (49)	-10 (69)	170 (69)	-41 (68)
C(16)	3167 (3)	2023 (9)	-1838 (8)	409 (41)	420 (43)	381 (52)	-88 (73)	-160 (70)	156 (71)
C(17)	3970 (4)	3361 (11)	-2210 (9)	534 (52)	680 (62)	509 (66)	347 (101)	268 (90)	-106 (98)
C(18)	4901 (4)	-504 (14)	-1309 (10)	428 (49)	1218 (105)	684 (82)	-381 (151)	259 (97)	613 (121)
C(19)	2640 (3)	-865 (10)	119 (10)	289 (36)	571 (55)	818 (77)	-303 (109)	14 (84)	-296 (80)
C(20)	3153 (3)	-109 (9)	2535 (7)	470 (42)	374 (42)	320 (48)	106 (70)	328 (69)	-74 (75)
C(21)	3176 (3)	3702 (8)	2935 (7)	485 (43)	267 (38)	302 (49)	-51 (62)	280 (72)	35 (64)
C(22)	3633 (4)	3491 (12)	3495 (9)	736 (65)	761 (70)	393 (64)	-329 (106)	-164 (98)	-155 (113)
C(23)	3711 (5)	4110 (14)	4431 (12)	931 (84)	886 (88)	758 (95)	-670 (151)	-125 (141)	73 (148)
C(24)	3328 (5)	4857 (13)	4958 (10)	1023 (85)	630 (66)	618 (81)	-461 (122)	-187 (129)	-69 (132)
C(25)	2859 (5)	5079 (11)	4452 (9)	1257 (93)	409 (53)	483 (70)	-248 (94)	398 (123)	254 (124)
C(26)	2777 (4)	4500 (9)	3411 (9)	865 (68)	290 (43)	504 (65)	-115 (79)	232 (106)	209 (88)
C(27)	3066 (3)	3069 (7)	1885 (7)	432 (38)	131 (31)	445 (51)	152 (59)	-24 (70)	-41 (56)
C(28)	4516 (3)	-642 (11)	1957 (9)	378 (41)	597 (57)	581 (65)	179 (98)	-217 (81)	99 (82)
C(29)	4977 (4)	29 (17)	2528 (12)	480 (50)	1160 (98)	911 (88)	31 (168)	-860 (105)	59 (134)
C(30)	3830 (3)	-1479 (12)	-3746 (9)	446 (45)	751 (68)	427 (61)	-341 (100)	-138 (77)	254 (95)
C(31)	3651 (5)	-1119 (17)	-4901 (10)	1063 (92)	1404 (124)	395 (78)	-669 (153)	-607 (129)	799 (185)

Table 2 (cont.)

Hydrogen attached to atom	x	y	z	U
C(2)	3081	1753	241	300
C(3)	3893	-18	240	300
C(5)	3739	-2484	2558	500
C(6)	2949	-3102	1488	900
C(6)	3349	-3972	1588	900
C(7)	3097	-3243	-257	600
C(10)	4042	-1368	-1505	500
C(13)	4866	2913	-1186	900
C(14)	4407	3753	167	600
C(14)	4260	2253	787	600
C(16)	3159	1512	-2496	400
C(16)	2959	1392	-1246	400
C(16)	3069	3132	-1946	400
C(17)	4406	3550	-2033	600
C(17)	3646	3640	-2353	600
C(17)	4016	2770	-3033	600
C(18)	5269	17	-1578	800
C(18)	4599	-782	-1878	800
C(18)	5109	-502	-698	800
C(19)	2613	-1038	-727	600
C(19)	2433	-1588	-407	600
C(19)	2513	-38	-227	600
C(20)	2925	356	2129	500
C(20)	3205	576	3009	500
C(22)	3927	3134	3002	700
C(25)	2555	5710	4826	400
C(26)	2432	4559	2949	800
O(7)	4017	-2328	422	600

was developed by the tangent formula program in a trial run on the 240 reflexions with E values exceeding 1.6. The correct solution turned out to be the one with the lowest Karle R value (16.78%, the second smallest being 22.97%). An E map computed with the 225 phased structure factors showed a nearly complete molecule although some peaks were ill resolved and two spurious ones were present. The oxygen of the oxetan ring was the 30th in the list of peak heights but it was left out in the calculation of an F_o synthesis to-

Table 3. Interatomic distances (Å) with standard deviations in parentheses

O(1)—C(1)	1.441 (11)	O(2)—C(2)	1.426 (9)
O(2)—C(27)	1.365 (10)	O(20)—C(27)	1.212 (11)
O(4)—C(4)	1.463 (10)	O(4)—C(28)	1.319 (13)
O(40)—C(28)	1.203 (14)	O(5)—C(5)	1.460 (12)
O(5)—C(20)	1.423 (11)	O(7)—C(7)	1.438 (13)
O(9)—C(9)	1.188 (11)	O(10)—C(10)	1.436 (12)
O(10)—C(30)	1.361 (13)	O(100)—C(30)	1.180 (13)
O(13)—C(13)	1.413 (15)	C(1)—C(2)	1.566 (11)
C(1)—C(14)	1.496 (75)	C(1)—C(15)	1.567 (12)
C(2)—C(3)	1.577 (10)	C(3)—C(4)	1.512 (11)
C(3)—C(8)	1.549 (12)	C(4)—C(5)	1.586 (13)
C(4)—C(20)	1.580 (12)	C(5)—C(6)	1.499 (16)
C(6)—C(7)	1.480 (16)	C(7)—C(8)	1.587 (14)
C(8)—C(9)	1.552 (12)	C(8)—C(19)	1.553 (15)
C(9)—C(10)	1.523 (13)	C(10)—C(11)	1.488 (13)
C(11)—C(12)	1.340 (13)	C(11)—C(15)	1.540 (12)
C(12)—C(13)	1.497 (15)	C(12)—C(18)	1.500 (16)
C(13)—C(14)	1.535 (76)	C(15)—C(16)	1.514 (13)
C(15)—C(17)	1.576 (14)	C(21)—C(22)	1.378 (14)
C(21)—C(26)	1.398 (14)	C(21)—C(27)	1.452 (12)
C(22)—C(23)	1.307 (19)	C(23)—C(24)	1.371 (20)
C(24)—C(25)	1.375 (18)	C(25)—C(26)	1.409 (17)
C(28)—C(29)	1.518 (19)	C(30)—C(31)	1.535 (19)

Table 4. Interbond angles (°) with standard deviations in parentheses

C(2)—O(2)—C(27)	118.5 (6)
C(5)—O(5)—C(20)	93.6 (7)
C(2)—C(1)—O(1)	102.2 (6)
C(31)—C(30)—O(100)	126.2 (11)
C(15)—C(1)—C(15)	112.0 (29)
C(3)—C(2)—O(2)	108.6 (6)
C(2)—C(3)—C(4)	111.2 (6)
C(2)—C(3)—C(8)	114.7 (6)
C(3)—C(4)—O(4)	110.2 (6)
C(3)—C(4)—C(5)	122.1 (7)
C(6)—C(5)—O(5)	112.7 (9)
C(5)—C(6)—C(7)	113.0 (10)
C(6)—C(7)—O(7)	111.3 (9)
C(3)—C(8)—C(7)	110.5 (7)
C(9)—C(8)—C(19)	106.3 (8)
C(8)—C(9)—O(9)	117.8 (8)
C(10)—C(9)—O(9)	119.3 (8)
C(11)—C(10)—O(10)	108.2 (7)
C(10)—C(11)—C(12)	121.5 (8)
C(12)—C(11)—C(15)	119.2 (8)
C(11)—C(12)—C(18)	123.7 (9)
C(12)—C(13)—O(13)	111.7 (9)
C(13)—C(13)—O(14)	107.9 (29)
C(1)—C(15)—C(11)	105.5 (7)
C(11)—C(15)—C(16)	115.7 (7)
C(4)—C(20)—O(5)	92.4 (6)
C(22)—C(21)—C(27)	123.8 (9)
C(21)—C(22)—C(23)	120.8 (11)
C(23)—C(24)—C(25)	119.6 (12)
C(21)—C(26)—C(25)	118.4 (10)
C(21)—C(27)—O(20)	125.3 (8)
O(4)—C(28)—O(40)	125.1 (10)
C(29)—C(28)—O(40)	124.2 (11)
C(31)—C(30)—O(10)	108.8 (10)
C(4)—O(4)—C(28)	116.9 (7)
C(10)—O(10)—C(30)	116.8 (8)
C(14)—C(1)—O(1)	110.1 (29)
C(2)—C(1)—C(15)	110.1 (7)
C(1)—C(2)—O(2)	103.4 (6)
C(1)—C(2)—C(3)	118.6 (6)
C(4)—C(3)—C(8)	111.2 (6)
C(20)—C(4)—O(4)	105.9 (6)
C(5)—C(4)—C(20)	83.2 (6)
C(4)—C(5)—O(5)	90.8 (7)
C(4)—C(5)—C(6)	119.0 (9)
C(6)—C(7)—C(8)	114.4 (9)
C(8)—C(7)—O(7)	110.1 (8)
C(7)—C(8)—C(19)	106.6 (8)
C(3)—C(8)—C(9)	117.8 (7)
C(8)—C(9)—C(10)	122.1 (7)
C(9)—C(10)—O(10)	106.0 (7)
C(9)—C(10)—C(11)	116.4 (8)
C(10)—C(11)—C(15)	119.0 (8)
C(11)—C(12)—C(13)	119.4 (9)
C(13)—C(12)—C(18)	116.8 (9)
C(12)—C(13)—C(14)	110.9 (29)
C(1)—C(14)—C(13)	118.2 (49)
C(1)—C(15)—C(17)	108.0 (7)
C(16)—C(15)—C(17)	105.5 (7)
C(22)—C(21)—C(26)	119.6 (9)
C(26)—C(21)—C(27)	116.5 (8)
C(22)—C(23)—C(24)	122.1 (13)
C(24)—C(25)—C(26)	119.2 (12)
O(2)—C(27)—O(20)	122.4 (8)
C(21)—C(27)—O(2)	112.3 (7)
C(29)—C(28)—O(4)	110.7 (10)
O(10)—C(30)—O(11)	125.0 (10)

gether with the three methyl groups C(18), C(29) and C(31). The electron-density map showed the entire

molecule and only one of the atoms obtained from the E map was shown to be in a wrong position.

After three cycles of isotropic and two of anisotropic refinement with unit weights the R value was 10.6%. A difference map showed 28 of the 38 hydrogens of the

molecule. Inclusion of these hydrogens in the refinement and the introduction of the weighting scheme

$$w = \begin{cases} 1 & \text{if } F_o < a \\ 1/F_o & \text{if } F_o > a \end{cases}$$

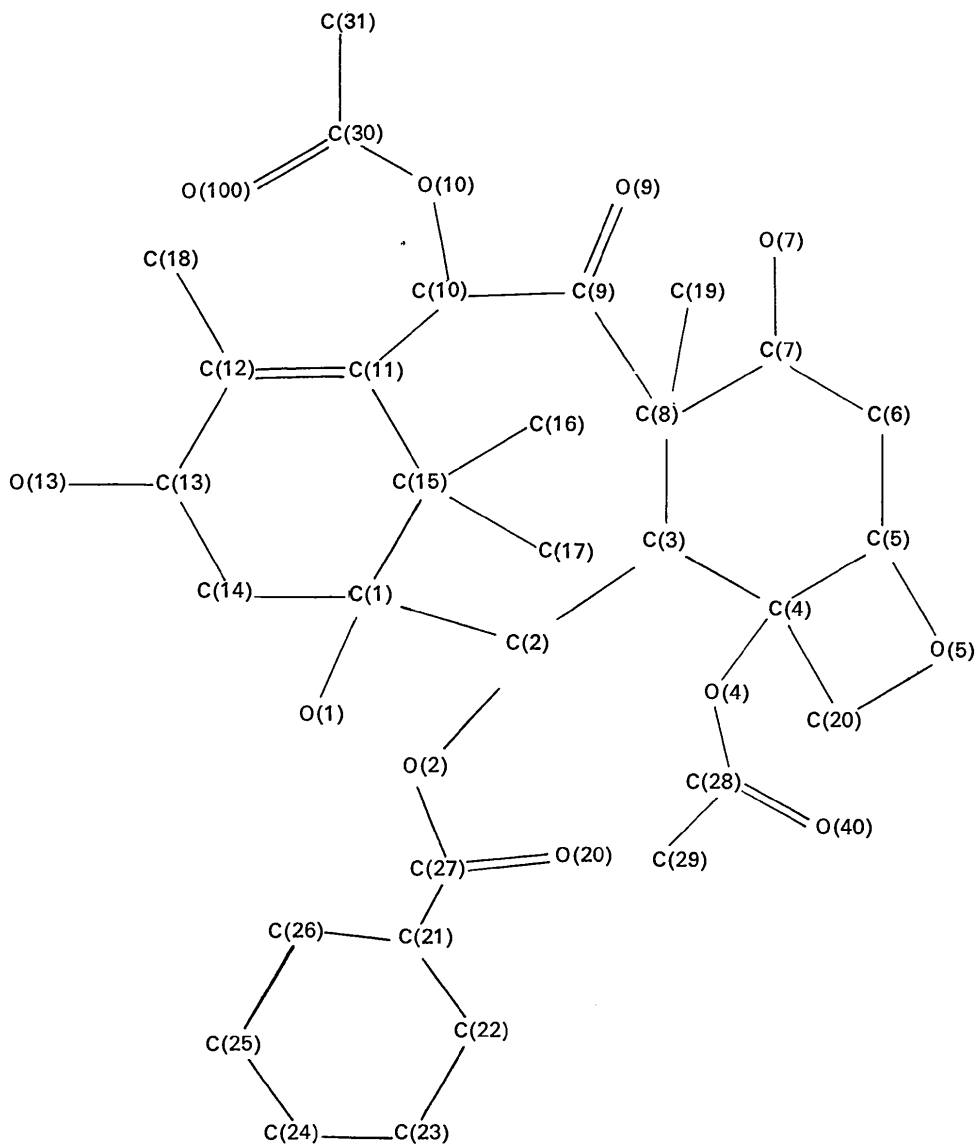


Fig. 1. Atomic numbering, interatomic distances and interbond angles.

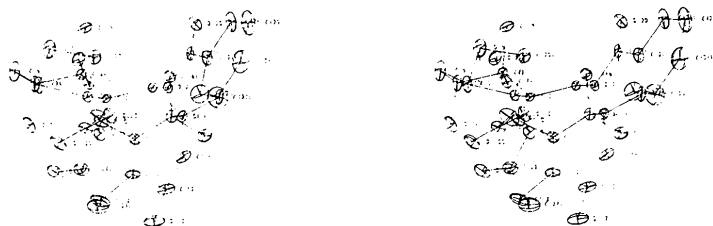


Fig. 2. Stereoscopic projection of bacatin V as viewed down the c axis.

with $a=25$ on an absolute scale gave, after four more cycles of anisotropic refinement, the final R value of 9.16%.* The refinement matrix was split into 10 blocks, one of 2×2 for the scale and overall temperature factor and nine of 42×42 for the parameters x/a , y/b , z/c , B_{11} , B_{22} , B_{33} , B_{23} , B_{13} and B_{12} respectively.

Description of the structure

The final space coordinates and thermal parameters are given in Table 2. Interatomic distances and angles calculated from the final positional parameters are given in Tables 3 and 4 respectively and are also shown in Fig. 1 together with the atomic numbering. Inspection of Table 2 shows that the majority of the B_{ii} are in the range commonly encountered. The rather large values for atoms C(22) to C(26) suggest a high degree of flapping and perhaps explain the somewhat abnormal C-C distances found in the benzene ring of the benzoate system.

The oxetan ring is planar to within experimental accuracy. The r.m.s. deviation of its four atoms from the best least-squares plane calculated through them is 0.009 Å.

The C(3) to C(8) six-membered ring is in a very distorted chair conformation with atoms C(3) and C(6) displaced 0.437 and -0.403 Å from the least-squares plane through C(4), C(5), C(7) and C(8), these latter being planar to within 0.177 Å. The distortion is mainly due to the planarity requirements of the oxetan ring, which forces the torsion angle C(20)-C(4)-C(5)-O(5) to adopt a value near to zero, rather than that of about 60° normally found in undistorted chair conformation rings.

The C(1), C(11) to C(15) six-membered ring is in a boat conformation somewhat distorted by the presence of the double bond C(11)-C(12) and by the steric requirements of the eight-membered ring. The fold angle about C(13)-C(15) is 48.2°.

The acyl oxygen of the 4- α -acetate group is strongly hydrogen bonded to that of the 7- α -hydroxyl, their separation being 2.77 Å.

Before this crystal structure analysis had been carried out, it was discovered that the proton at C(3) could not oxidize, contrary to expectation. The reason for this appeared clearly after solving the structure, as the hy-

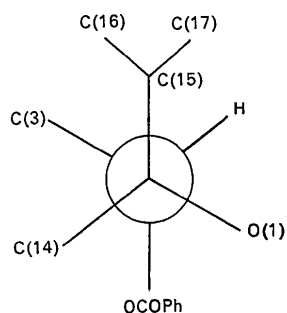


Fig. 3. Newman projection down the bond C(1)-C(2) showing the configuration of the molecule assumed by the authors.

drogen at C(3) is completely shielded by neighbouring atoms of the molecule.

Fig. 2 shows a stereoscopic projection of the molecule as viewed down the c axis and Fig. 3 is a Newman projection down the bond C(1)-C(2). It is interesting that the conformations of the oxygen atoms attached to C(5) and C(7) (β and α respectively) are opposite to those in other taxane derivatives previously found. In a recent paper (Wani, Taylor, Wall, Coggon & McPhail, 1971), the structure of another taxane derivative from *Taxus brevifolia* has been reported. It has also a naturally occurring oxetan with the oxygen at C(5) in the same configuration as that in baccatin V but its oxygen at C(7) is again in the β configuration normally found.

This new taxane derivative has been found to have antileukemic and tumor-inhibitory properties (Wani *et al.*, 1971).

All calculations were carried out on the Oxford University KDF 9 computer. All Fourier syntheses, least-squares refinement and interatomic distances and angles calculations were performed with the Rollett, Hodder & Ford (1968: unpublished) systems of programs. The observed structure amplitudes and structure factors calculated from the final atomic parameters (Table 2) have been deposited at the National Lending Library, England. Thanks are due to the Consejo Nacional de Investigaciones Científicas y Técnicas (Argentina) for support to one of us (E.E.C.).

References

- DELLA CASA DE MARCANO, D. P., HALSALL, T. G., CASTELLANO, E. E. & HODDER, O. J. R. (1970). *Chem. Commun.* pp. 1382-1383.
 HAUPTMAN, H. & KARLE, J. (1956). *Acta Cryst.* **9**, 45-55.
 KARLE, J. & KARLE, I. L. (1966). *Acta Cryst.* **21**, 849-859.
 WANI, M. C., TAYLOR, H. L., WALL, M. E., COGGO, P. & MCPHAIL, A. T. (1971). *J. Amer. Chem. Soc.* **9**, 2325-2327.

* A list of observed and calculated structure factors has been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30157 (23 pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.