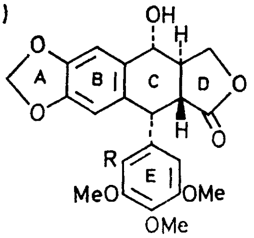
Crystal Structure and Absolute Configuration of 2′-Bromopodophyllotoxin–0.5 Ethyl Acetate

By **Trevor J. Petcher,* H. P. Weber, M. Kuhn, and A. von Wartburg**, Pharmaceutical Chemical Laboratories, Sandoz Ltd., Basle, Switzerland

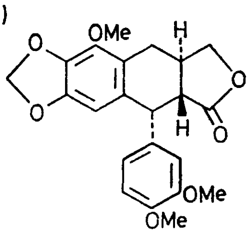
The crystal structure of the title compound (I; R = Br) has been determined in order to provide conclusive evidence for the absolute configuration of podophyllotoxin (I; R = H) and related lignans. The structure was determined from diffractometer data by the heavy-atom method and refined by least-squares techniques to *R* 0·098 for 2043 observed reflections. Crystals are monoclinic, space group $P2_1$ with $Z = 4$ in a unit cell of dimensions $a = 1321(4)$, $b = 1558(2)$, $c = 1168(5)$ pm, $\beta = 102{\cdot}0(2)°$.

PODOPHYLLOTOXIN (I; R = H), is an antimitotic[1] lignan isolated from the species *Podophyllum*. Its structure and configuration have previously been established by chemical methods;[2] a series of chemical interconversions and comparisons of optical rotations revealed a spatial correlation with several related natural lignans of the diarylbutane and aryltetrahydronaphthalene groups.[3] It was also possible to correlate guaiaretic acid dimethyl ether, a key intermediate in these stereochemical deductions, with L-3,4-dihydroxyphenylalanine and thus establish the absolute configuration of podophyllotoxin and related lignans[4] as that shown in (I). Recently, the first *X*-ray crystal-structure analysis of one of these lignans, 5′-demethoxy-β-peltatin (II) has been published.[5] While confirming the chemically assigned structure,[6] the authors cast some doubt on the assignment of absolute configuration. By use of the anomalous scattering of oxygen for Cu-K_α radiation ($\Delta f'' = 0{\cdot}04$ e) and Hamilton's *R*-factor ratio test,[7] the authors claimed a reversal of the previously published absolute configuration with a probability of 99·5%. (The validity of this claim will be discussed later.) Measurement of Friedel pairs, however, gave inconclusive results. We have undertaken a crystal-structure analysis of 2′-bromopodophyllotoxin (I; R = Br) which is easily accessible by a procedure described by Kofod and Jørgensen.[8] The position of the bromine atom and retention of the configuration at the asymmetric centres were unequivocally determined by chemical degradations and by n.m.r. spectroscopy before the *X*-ray study.

(I)



(II)



EXPERIMENTAL

The sample used was twice recrystallised from ethyl acetate as clear tabular plates of $C_{22}H_{21}BrO_8,\frac{1}{2}C_4H_8O_2$, and a dried analytical sample had m.p. 184—185°, $[\alpha]_D^{22}$ −110° (*c* 1·093 in $CHCl_3$) and $[\alpha]_D^{22}$ −91° (*c* 0·968 in EtOH).

Crystal Data.—$C_{22}H_{21}BrO_8,\frac{1}{2}C_4H_8O_2$, $M = 493{\cdot}31 + 44$. Monoclinic, $a = 1321(4)$, $b = 1558(2)$, $c = 1168(5)$ pm, $\beta = 102{\cdot}0(2)°$, $U = 2351 \times 10^6$ pm^3, $D_m = 1{\cdot}56$—$1{\cdot}60$ (by flotation), $Z = 4$, $D_c = 1{\cdot}52$. Space group $P2_1$ (C_2^2, No. 4). Mo-K_α radiation, $\lambda = 71{\cdot}07$ pm, μ(Mo-K_α) = 10·61 cm^{-1}.

---

[1] Mitosis-inhibiting natural products 25. For paper 24 see C. Keller-Juslén, M. Kuhn, A. von Wartburg, and H. Stähelin, *J. Medicin. Chem.*, 1971, **14**, 936.

[2] J. L. Hartwell and A. W. Schrecker, *Fortschr. Chem. org. Naturstoffe*, 1958, **15**, 83.

[3] A. W. Schrecker and J. L. Hartwell, *J. Amer. Chem. Soc.*, 1955, **77**, 432, 6725.

[4] A. W. Schrecker and J. L. Hartwell, *J. Org. Chem.*, 1956, **21**, 381; *J. Amer. Chem. Soc.*, 1957, **79**, 3827.

[5] R. B. Bates and J. B. Wood, *J. Org. Chem.*, 1972, **37**, 562.

[6] E. Bianchi, K. Sheth, and J. R. Cole, *Tetrahedron Letters*, 1969, 2759.

[7] W. C. Hamilton, *Acta Cryst.*, 1965, **18**, 502.

[8] H. Kofod and C. Jørgensen, *Acta Chem. Scand.*, 1955, **9**, 1327.

Unit-cell and space-group data were determined from precession photographs and three-dimensional intensity data were collected on a Hilger and Watts linear diffractometer by use of graphite monochromatised Mo-K_α radiation. A total of 3588 measurements for $\sin\theta/\lambda \leqslant 0{\cdot}55$ yielded 2043 observed symmetry-independent reflections with $I \geqslant 3\sigma(I)$. The structure was solved by straightforward application of the heavy-atom method and refined by block-diagonal least-squares employing anisotropic thermal parameters for all non-hydrogen atoms. Refinement, neglecting the effect of anomalous scattering, terminated at a conventional residual, R 0·105. Absorption and extinction corrections were not applied, nor have hydrogen atoms been located. At the termination of this refinement, structure factors were calculated for both enantiomers, taking into account the dispersion of the bromine atoms ($\Delta f' = 0{\cdot}3$, $\Delta f'' = 2{\cdot}6$e). The residuals were 0·1020 and 0·1054 respectively, yielding a ratio of 1·0333. At the same time, a calculation was performed to indicate those reflections which would show the maximum significant intensity differences between Friedel pairs. Twelve such pairs were carefully measured on a four-circle diffractometer, together with a consecutive group of reflections having $h = 6$ for further confirmation. The results (Table 1) clearly indicate the correct enantiomer to be that with R 0·102. Three further cycles of refinement including the effect of anomalous scattering reduced R to a final value of 0·098. By use of the previously determined R ratio of 1·0333, we may also, by application of the Hamilton ratio test,[7] reject the one-dimensional null hypothesis having 1440 degrees of freedom, that the absolute configuration is the opposite to that shown in the Figures, at the significance level of considerably $<0{\cdot}005$. At the 0·005 significance level, the R ratio is required to be $>1{\cdot}00283$ for such a hypothesis. This it clearly is: we may say that the absolute configuration is correct with a probability much greater than 99·5%. Bates and Wood,[5] however, have claimed a probability of 99·5% for the opposite configuration, using as evidence an R ratio of 1·00228 for a hypothesis with 1431 degrees of freedom. For such a case, the ratio is required to be $>1{\cdot}00285$ at the 0·005 significance level. Their calculations are clearly in error.

TABLE 1

Measurements of Friedel pairs of integrated intensities

h	k	l	I [a]	ΔI [b]	$\Delta(F_c^2)/(F_c^2)$ [c]
$\bar{4}$	1	1	20760 (720)	−3624	−0·22
3	1	1	57624 (1187)	−9600	−0·21
5	1	2	7224 (429)	2833	0·23
$\bar{2}$	1	3	36576 (952)	−11928	−0·33
$\bar{3}$	1	3	40334 (999)	7560	0·27
1	1	4	37128 (966)	−5256	−0·20
3	1	4	16680 (644)	4176	0·22
$\bar{5}$	1	5	17472 (662)	4316	0·32
5	2	1	7008 (425)	1968	0·23
$\bar{4}$	2	2	64334 (1253)	9416	0·20
$\bar{1}$	2	2	68369 (1296)	15743	0·18
4	2	3	82656 (1418)	−2544	−0·09

h	k	l	Δ_o [d]	Δ_c	h	k	l	Δ_o	Δ_c
6	1	$\bar{6}$	0·17	0·25	6	3	$\bar{3}$	−0·03	−0·02
6	1	$\bar{5}$	−0·14	0·07	6	3	$\bar{2}$	0·11	0·07
6	1	$\bar{4}$	0·22	0·25	6	3	$\bar{1}$	−0·03	−0·02
6	1	$\bar{3}$	−0·02	0·00	6	3	0	−0·01	0·00
6	1	$\bar{2}$	−0·20	−0·22	6	3	1	−0·02	0·21
6	1	$\bar{1}$	0·02	−0·06	6	3	2	−0·01	−0·04
6	1	0	0·11	0·09	6	3	3	−0·35	−0·33
6	2	$\bar{6}$	0·62	0·63	6	3	4	0·06	−0·04
6	2	$\bar{3}$	−0·41	−0·42	6	3	5	−0·29	−0·19
6	2	$\bar{2}$	−0·17	−0·17	6	4	$\bar{6}$	0·25	0·32
6	2	$\bar{1}$	0·02	0·02	6	4	$\bar{5}$	0·13	0·11
6	2	0	−0·39	−0·40	6	4	$\bar{4}$	−0·05	−0·09
6	2	1	−0·21	−0·23	6	4	$\bar{3}$	−0·40	−0·67
6	2	2	−0·02	−0·01	6	4	$\bar{2}$	0·18	0·10
6	2	4	−0·04	−0·04	6	4	0	0·06	0·04
6	2	5	−0·17	−0·16	6	4	1	0·25	0·04
6	3	$\bar{6}$	−0·02	0·20	6	4	2	0·01	−0·14
6	3	$\bar{5}$	−0·20	−0·10	6	4	3	0·00	−0·06
6	3	$\bar{4}$	−0·25	−0·30	6	4	4	−0·03	−0·16

[a] I is the measured net intensity of hkl, uncorrected for absorption. The figures in parentheses are standard deviations derived from counting statistics only. [b] $\Delta I = I(hkl) - I(\bar{h}\bar{k}\bar{l})$. [c] F_c refers to hkl and is calculated on the basis of the positions of the correct enantiomer, $(\Delta F_c^2) = F_c^2(hkl) - F_c^2(\bar{h}\bar{k}\bar{l})$. [d] $\Delta_{obs} = \Delta I/I$ as defined in (b), Δ_c corresponds to $\Delta(F_c^2)/(F_c^2)$ as in (c).

DISCUSSION

Description of the Structure.—The two molecules in the asymmetric unit have very similar conformations and are shown in stereo-pairs in Figure 1. Figure 2 shows intramolecular distances and angles for both molecules and also illustrates the numbering scheme used. Final positions and thermal parameters for all 68 non-hydrogen atoms are presented in Table 2; Table 3 lists sufficient torsion angles for both molecules for the construction of accurate molecular models, and Table 4 lists some selected intermolecular distances. Structure factor tables have been deposited as Supplementary Publication No. SUP 20561 (17 pp., 1 microfiche).* Figure 3 shows the molecular packing projected down the b axis; for clarity only half the cell in the b direction is drawn.

In both independent molecules, the C–D ring junction is *trans*, the 13-hydroxy-substituent is equatorial to the C-ring, and the E-ring substituent at C(7) is pseudo-axial to the C-ring, which displays an envelope [flap C(12)] conformation. The dioxolen (A) ring is coplanar with ring B, and the lactone ring (D) exhibits the expected envelope conformation with C(12) the flap and C(8)–C(9)–O(10)–C(11)–O(18) coplanar. The absolute configuration of the bromo-derivative, following the convention of Cahn, Ingold, and Prelog[9] is (7S,8R,12R,13R) with our numbering scheme. For the native lignan this changes to (7R,8R,12R,13R) or (1R,2R,3R,4R) in terms of the conventional numbering of ref. 4.

The plane of ring E is almost normal to the mean plane through rings A, B, C, and D in both molecules, but the CH_3 groups of the methoxy-substituents take up different orientations in the two molecules. None are coplanar with the associated benzene ring because of repulsive interactions with bromine. The torsion angles describing their orientations are collected for convenience in a subgroup of Table 3. Apart from such minor differences of methoxy-group orientation, the conformations of the two independent molecules are almost

---

* For details see Notice to Authors No. 7 in *J. Chem. Soc.* (*A*), 1970, Issue No. 20.

[9] R. S. Cahn, C. K. Ingold, and V. Prelog, *Experientia*, 1956, **12**, 81; *Angew. Chem.*, 1966, **8**, 413.

TABLE 2

Final positions (× 10^4),[a] vibrations (× 10^5),[b] and estimated standard deviations

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
O(1)	5409(9)	−5055(8)	−0985(10)	942(131)	1082(86)	592(132)	07(106)	107(107)	87(112)
C(2)	6538(14)	−5106(15)	−1104(16)	714(189)	1726(219)	485(205)	−363(186)	−121(155)	−79(195)
O(3)	7125(9)	−4924(8)	0142(10)	703(112)	909(79)	1115(158)	66(96)	180(107)	352(117)
C(4)	6351(13)	−4889(11)	0857(15)	700(174)	813(114)	898(223)	39(147)	260(159)	−64(168)
C(5)	6600(12)	−4780(12)	2081(14)	531(160)	904(130)	568(194)	42(126)	318(141)	93(140)
C(6)	5751(12)	−4810(10)	2535(14)	529(154)	607(103)	628(191)	130(114)	−422(139)	−86(129)
C(7)	5977(12)	−4722(9)	3965(14)	538(153)	423(91)	688(198)	105(95)	162(144)	52(111)
C(8)	4919(12)	−4879(12)	4370(13)	381(139)	1197(137)	278(164)	−77(140)	273(126)	−112(153)
C(9)	4933(14)	−4533(10)	5551(15)	739(180)	522(83)	1031(250)	171(117)	−26(178)	−245(140)
O(10)	3890(9)	−4328(7)	5583(11)	1011(135)	712(74)	1015(162)	171(85)	416(120)	36(97)
C(11)	3225(13)	−4460(13)	4421(16)	641(184)	1091(136)	624(211)	99(141)	81(157)	129(163)
C(12)	4021(13)	−4366(10)	3629(15)	566(159)	692(103)	551(194)	122(117)	196(143)	−125(133)
C(13)	3707(12)	−4845(14)	2442(14)	374(149)	1383(167)	461(188)	31(150)	148(134)	105(170)
C(14)	4704(13)	−4831(11)	1916(14)	425(145)	907(120)	512(183)	81(128)	−113(132)	322(147)
C(15)	4483(13)	−4954(11)	0596(15)	779(178)	707(97)	698(301)	24(141)	315(150)	27(154)
C(16)	5386(13)	−5052(11)	0215(15)	681(176)	892(113)	562(193)	207(140)	−25(148)	450(158)
O(17)	2894(9)	−4356(8)	1666(10)	610(110)	935(80)	850(148)	32(83)	247(103)	−63(99)
O(18)	5605(9)	−4434(8)	6419(10)	741(122)	1016(81)	692(139)	180(90)	249(106)	112(101)
C(19)	6569(13)	−3883(11)	4360(15)	658(164)	617(98)	664(206)	261(119)	123(148)	433(133)
C(20)	6209(11)	−3017(10)	3947(15)	314(139)	557(90)	845(215)	124(101)	224(140)	175(128)
C(21)	6686(12)	−2329(10)	4444(14)	367(138)	593(97)	641(195)	−172(102)	335(135)	−44(120)
C(22)	7593(13)	−2372(11)	5414(15)	516(158)	835(117)	578(200)	147(119)	210(144)	66(136)
C(23)	8066(13)	−3155(8)	5771(14)	1096(193)	258(73)	552(192)	184(107)	477(158)	125(107)
C(24)	7478(13)	−3931(9)	5236(15)	889(184)	421(85)	566(202)	−176(118)	68(155)	16(122)
O(25)	6367(9)	−1513(6)	4165(10)	822(120)	301(47)	1153(153)	−66(68)	−17(109)	133(81)
C(26)	5710(13)	−1438(8)	2996(15)	688(169)	140(59)	973(215)	142(93)	−464(155)	−227(111)
O(27)	8038(9)	−1700(7)	5873(11)	733(124)	574(64)	1658(187)	128(76)	68(123)	21(99)
C(28)	8172(14)	−1628(12)	7211(15)	931(204)	802(120)	580(199)	123(132)	37(164)	222(138)
O(29)	8921(8)	−3279(7)	6648(10)	573(109)	921(82)	816(148)	89(86)	−295(102)	81(99)
C(30)	9807(12)	−3201(13)	6198(17)	273(147)	1277(166)	1055(253)	163(140)	199(158)	47(175)
Br(31)	8033(1)	−5000(0)	5879(2)	914(20)	636(9)	1079(24)	−71(15)	−91(17)	−55(17)
O(1′)	3343(10)	−2125(8)	3575(10)	1264(148)	1125(96)	497(128)	164(109)	203(110)	−58(107)
C(2′)	2184(17)	−2182(14)	3047(16)	1496(245)	931(145)	772(234)	−151(163)	446(199)	−173(170)
O(3′)	1659(9)	−2093(7)	4024(10)	854(117)	793(74)	1037(155)	53(90)	112(110)	−29(105)
C(4′)	2402(13)	−2120(11)	5061(16)	523(157)	538(95)	1245(244)	57(115)	130(161)	68(148)
C(5′)	2323(12)	−2169(12)	6203(14)	469(149)	799(118)	597(188)	38(120)	282(136)	−103(140)
C(6′)	3204(12)	−2243(10)	7083(13)	536(151)	624(106)	370(168)	55(107)	220(129)	−46(117)
C(7′)	3045(12)	−2249(10)	8310(14)	595(160)	559(104)	593(192)	155(112)	−171(143)	31(125)
C(8′)	4115(13)	−2273(8)	9145(14)	750(173)	356(82)	836(214)	05(103)	406(158)	−106(116)
C(9′)	4321(15)	−2538(9)	10466(17)	1300(237)	395(85)	1144(274)	−125(130)	224(202)	100(138)
O(10′)	5243(9)	−2807(7)	10842(10)	894(130)	854(80)	853(157)	−174(88)	−38(114)	−206(101)
C(11′)	5813(14)	−2594(12)	9927(17)	616(177)	991(133)	976(251)	84(136)	224(169)	−37(166)
C(12′)	4956(12)	−2704(11)	8787(16)	363(151)	851(123)	958(235)	−305(120)	246(152)	−44(151)
C(13′)	5229(13)	−2289(9)	7698(15)	714(171)	543(99)	727(213)	211(113)	−70(151)	−231(123)
C(14′)	4202(13)	−2237(9)	6817(15)	815(180)	500(98)	640(204)	134(113)	123(152)	151(126)
C(15′)	4279(13)	−2166(10)	5597(14)	786(175)	590(98)	660(203)	143(121)	207(153)	172(132)
C(16′)	3410(14)	−2130(11)	4804(16)	1095(210)	645(106)	900(230)	27(138)	281(179)	116(146)
O(17′)	6007(9)	−2704(7)	7278(10)	627(108)	747(74)	1069(158)	73(78)	461(105)	−60(96)
O(18′)	3579(10)	−2626(8)	11070(10)	1091(138)	866(77)	666(147)	−181(91)	30(113)	−110(97)
C(19′)	2408(12)	−3097(10)	8511(16)	468(152)	524(93)	826(218)	−4(104)	−290(146)	−188(126)
C(20′)	2587(12)	−3859(10)	7980(15)	702(176)	424(92)	862(229)	−98(112)	34(161)	41(125)
C(21′)	2001(14)	−4639(11)	8115(15)	691(179)	697(115)	728(216)	216(118)	−146(158)	45(133)
C(22′)	1166(13)	−4574(10)	8844(16)	693(178)	524(91)	994(237)	184(110)	148(165)	272(134)
C(23′)	0993(13)	−3826(12)	9308(16)	772(183)	754(115)	819(231)	364(129)	186(165)	−120(143)
C(24′)	1579(15)	−3154(11)	9126(17)	1303(226)	632(106)	826(237)	−279(135)	290(189)	521(141)
O(25′)	2264(10)	−5364(6)	7621(12)	856(126)	432(56)	1556(187)	194(72)	230(125)	34(89)
C(26′)	1541(15)	−5488(14)	6442(18)	831(213)	1172(150)	926(255)	146(154)	−71(187)	262(173)
O(27′)	0796(11)	−5337(8)	8959(13)	985(143)	1134(100)	1405(192)	405(97)	252(134)	83(116)
C(28′)	1250(16)	−5728(12)	10055(19)	1307(260)	739(128)	1570(332)	188(149)	72(237)	−196(170)
O(29′)	0263(10)	−3778(9)	9921(13)	1008(141)	1213(105)	1077(160)	258(106)	673(123)	292(117)
C(30′)	−0743(13)	−4002(14)	9386(18)	380(161)	1175(144)	1421(298)	399(143)	121(174)	342(189)
Br(31′)	1193(1)	−2029(1)	9736(2)	988(21)	928(13)	1070(24)	−149(16)	380(19)	154(19)
C(101)	8542(21)	−2750(19)	2134(25)	2148(394)	1686(264)	2538(483)	508(249)	879(354)	935(300)
C(102)	9449(16)	−3215(10)	2577(19)	1245(236)	448(102)	1697(327)	19(135)	198(227)	−160(155)
O(103)	9205(10)	−4025(7)	2751(14)	753(121)	484(56)	2294(229)	−85(80)	−173(135)	−131(106)
C(104)	9846(14)	−4550(13)	3139(21)	383(174)	1151(151)	2190(370)	137(142)	−392(209)	−180(214)
C(105)	9583(17)	−5435(14)	3312(20)	1068(251)	1543(197)	1360(312)	−289(183)	492(225)	−503(216)
O(106)	10694(12)	−4274(10)	3672(18)	820(155)	1508(143)	3574(362)	102(125)	−61(194)	−369(194)

[a] Molecule (1) is C(1)–Br(31), the second, independent molecule is labelled C(1′)–Br(31′), and numbers over 100 refer to the solvent molecule. [b] Anisotropic temperature factors are of the form exp − $(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})$.

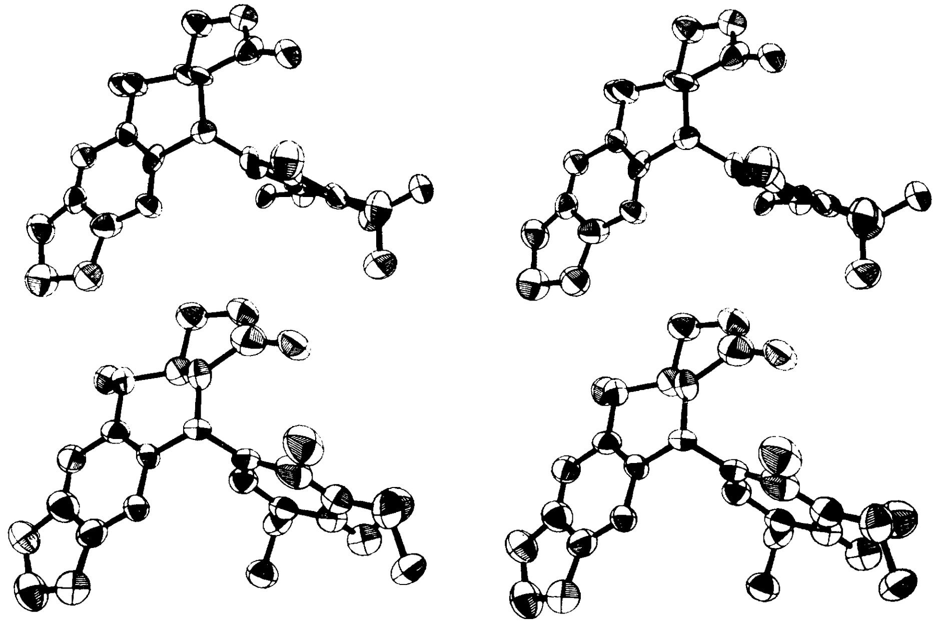


FIGURE 1 Stereo-pairs of the two independent molecules of bromopodophyllotoxin, viewed along the same relative direction; the lower molecule is represented as C(1′) · · · Br(31′) in Table 2

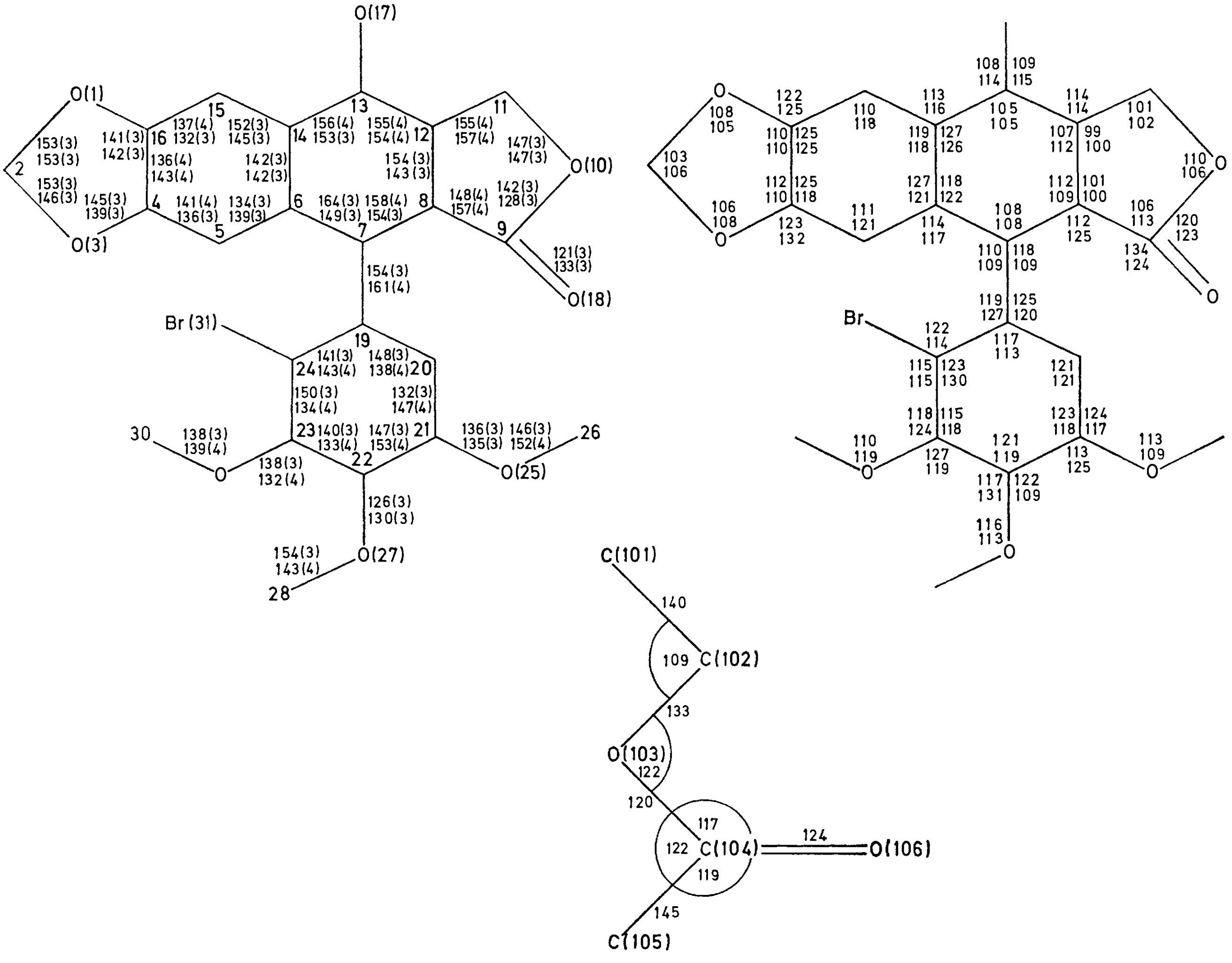


FIGURE 2 Bond lengths (pm) and angles (deg.) in the two independent molecules, and in the solvent molecule; σ for bond angles all *ca.* 2°

TABLE 3

Some selected torsion angles (deg.), with standard deviations in parentheses

	Molecule (1)	Molecule (2)
C(6)–C(7)–C(8)–C(12)	−47(2)	−32(3)
C(7)–C(8)–C(12)–C(13)	76(2)	62(3)
C(8)–C(12)–C(13)–C(14)	−60(2)	−52(3)
C(8)–C(9)–C(10)–C(11)	−5(3)	11(3)
C(9)–O(10)–C(11)–C(12)	−24(2)	−34(2)
O(10)–C(11)–C(12)–C(8)	42(2)	45(2)
C(11)–C(12)–C(8)–C(9)	−46(2)	−36(2)
C(12)–C(8)–C(9)–O(10)	32(2)	19(3)
C(11)–C(12)–C(13)–O(17)	76(3)	71(3)
C(15)–C(14)–C(13)–O(17)	−42(3)	−33(3)
C(8)–C(7)–C(19)–C(20)	−68(3)	−82(3)
C(6)–C(7)–C(19)–C(24)	−129(2)	−138(2)
C(20)–C(21)–O(25)–C(26)	−22(3)	−97(3)
C(21)–C(22)–O(27)–C(28)	126(3)	99(3)
C(22)–C(23)–O(29)–C(30)	−88(3)	−58(4)
Solvent		
C(101)–C(102)–O(103)–C(104)	179(3)	
C(102)–O(103)–C(104)–O(105)	179(4)	
C(102)–O(103)–C(104)–O(106)	6(4)	

TABLE 4

Some selected intermolecular distances (pm)

O(17) ··· O(18′)	297	O(103) ··· O(3)	391
O(18) ··· O(17′)	288	O(103) ··· C(5)	357
		O(103) ··· O(29′)	387
C(101) ··· C(11′)	398	O(103) ··· C(30′)	395
C(101) ··· O(25′)	389		
C(101) ··· C(26′)	391	C(105) ··· C(5′)	382
C(102) ··· C(2′)	389	O(106) ··· C(11)	329
C(102) ··· O(3′)	352	O(106) ··· C(30)	378
C(102) . . . O(29′)	360	O(106) ··· C(2′)	395
C(102) ··· C(30′)	388	O(106) ··· O(3′)	362
		O(106) ··· C(26′)	372

identical and, moreover, very similar to that of 5′-demethoxy-β-peltatin. Each molecule forms one hydrogen bond (to judge from O ··· O separations) O(17)–H ··· O(18′) which links the symmetry-independent lignan molecules in chains roughly parallel to the crystallographic *c* axis. No hydrogen bonds are formed to the ethyl acetate solvent molecule, which appears simply to fill interstices left by the packing of the lignan.

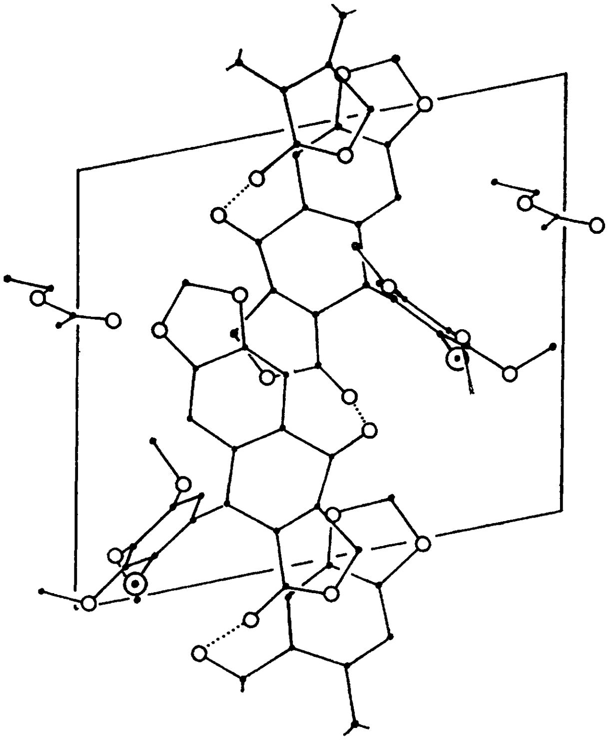


FIGURE 3 Projection of the crystal packing down the *b*-axis. For clarity only half of the cell content is shown

We thank Scintag, Wetzikon for use of a Syntex *P*1̄ diffractometer for measurements of Friedel pairs.

[2/1936 *Received*, 11*th August*, 1972]