Crystal Structure of Isozygosporin A *p*-Bromobenzoate

By (Miss) **Y. Tsukuda** and **H. Koyama,*** Shionogi Research Laboratory, Shionogi & Co. Ltd., Fukushima-ku, Osaka, Japan

The crystal structure of the title compound (III) has been determined by three-dimensional *X*-ray diffraction methods from diffractometer data. The crystals are orthorhombic, $a = 22.535$, $b = 12.332$, $c = 13.917$ Å, $Z = 4$, space group, $P2_12_12_1$. The structure was solved by heavy-atom and three-dimensional Fourier methods, and refined by least-squares calculations to a final R of 0·098 for 2318 independent observed reflexions. The absolute configuration was determined by the anomalous dispersion method.

ZYGOSPORIN A, a cytotoxic antibiotic, which was isolated[1] from the culture filtrate of *Zygosporium Masonii*, is a crystalline solid of empirical formula $C_{30}H_{37}O_6N$. From the results of our preliminary *X*-ray analysis[2] of isozygosporin A *p*-bromobenzoate (III) and various chemical information, Minato[3] assigned the formulae (I) to zygosporin A and (II) to isozygosporin A. Zygosporin A (I) is identical with the structure suggested[4] for cytochalasin D.

We now describe a further *X*-ray analysis of the structure (III), including the determination of the absolute configuration.

EXPERIMENTAL

Crystals of isozygosporin A *p*-bromobenzoate (m.p. 242—245 °C) from isopropyl alcohol are colourless prisms elongated along the *b* axis. Precession and Weissenberg photographs were taken with Cu-K_α ($\lambda = 1.5418$ Å) radiation;

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[1] S. Hayakawa, T. Matsushima, T. Kimura, H. Minato, and K. Katagiri, *J. Antibiotics*, 1968, **21**, 523.

[2] Y. Tsukuda, M. Matsumoto, H. Minato, and H. Koyama, *Chem. Comm.*, 1969, 41.

[3] H. Minato and M. Matsumoto, *J. Chem. Soc.* (*C*), 1970, 38.

[4] D. C. Aldridge and W. B. Turner, *J. Chem. Soc.* (*C*), 1969, 923.

space group and the unit-cell dimensions were determined from the precession photographs and on the automatic diffractometer.

Crystal Data.—$C_{37}H_{40}BrNO_7$, C_3H_8O, $M = 751$, Orthorhombic, $a = 22{\cdot}535 \pm 0{\cdot}004$, $b = 12{\cdot}332 \pm 0{\cdot}002$, $c = 13{\cdot}917 \pm 0{\cdot}002$ Å, $U = 3867$ Å^3, $D_m = 1{\cdot}281$ (by flotation), $Z = 4$, $D_c = 1{\cdot}289$, $F(000) = 1576$. Space group, $P2_12_12_1$ (D_2^4, No. 19) from systematic absences: $h00$ when h is odd, $0k0$ when k is odd, $00l$ when l is odd. Mo-K_α radiation, $\lambda = 0{\cdot}7107$ Å; μ(Mo-K_α) = 11·80 cm^{-1}.

The three-dimensional intensity data were collected on a Hilger and Watts automatic four-circle diffractometer Y 290 controlled by a PDP 8 computer. Integrated intensities were measured for $\theta \leqslant 27{\cdot}5°$ by the θ—2θ scan technique using Mo-K_α radiation and a scintillation counter (with a zirconium filter and pulse-height analyser). Each reflexion was integrated in 80 steps of intervals of 0·01°. In this way 3606 independent reflexions were recorded, of which 2318 had counts sufficiently above background for use in the subsequent analysis. All the intensities were corrected for Lorentz and polarization factors, and the structure amplitudes derived. No absorption corrections were applied since the specimen was considered to be sufficiently small (0·30 × 0·30 × 0·30 mm).

TABLE 1

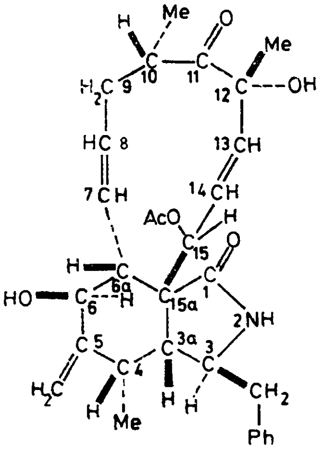
Final fractional atomic co-ordinates and anisotropic thermal parameters (all $\times 10^4$),* with estimated standard deviations in parentheses

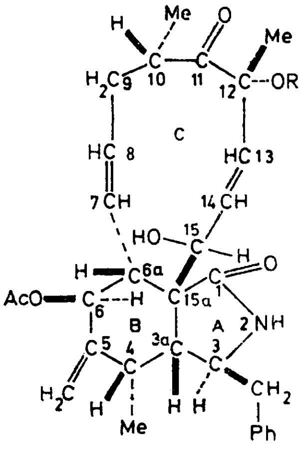
Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Br	4134(1)	−8231(1)	7213(1)	33(0)	112(1)	124(1)	17(0)	10(0)	−20(1)
O(1)	4877(4)	−6185(7)	2906(7)	25(2)	49(7)	58(6)	1(3)	−0(3)	−25(6)
O(2)	5832(5)	−6571(8)	−15(7)	32(3)	71(8)	72(7)	5(5)	−0(4)	17(7)
O(3)	5811(7)	−8237(12)	598(14)	58(5)	110(13)	220(20)	−31(8)	18(9)	−12(15)
O(4)	6596(4)	−6063(9)	4362(7)	25(2)	73(9)	67(6)	1(4)	7(3)	−5(7)
O(5)	5961(4)	−4579(8)	5142(6)	15(2)	80(8)	53(6)	1(3)	−0(3)	7(6)
O(6)	6570(5)	−5128(10)	6296(7)	29(2)	95(11)	61(6)	19(5)	−13(3)	4(7)
O(7)	5652(4)	−3090(7)	1844(7)	20(2)	38(6)	74(6)	−5(3)	8(3)	−16(6)
N(2)	4123(5)	−5362(7)	2096(8)	20(2)	30(7)	66(7)	4(4)	10(4)	−19(6)
C(1)	4704(5)	−5533(10)	2342(9)	16(2)	50(10)	35(7)	−6(4)	−1(3)	25(8)
C(3)	4023(6)	−4606(12)	1351(11)	19(3)	85(13)	65(9)	4(6)	−0(5)	1(9)
C(3a)	4646(6)	−4280(11)	988(10)	24(3)	40(9)	45(8)	1(5)	1(4)	21(7)
C(4)	4770(5)	−4701(12)	−47(13)	11(2)	54(12)	115(13)	5(5)	4(5)	−16(11)
C(5)	4836(6)	−5922(13)	1(10)	15(3)	71(14)	61(9)	11(5)	0(4)	− 4(10)
C(6)	5367(6)	−6260(9)	605(10)	20(3)	9(7)	65(9)	0(4)	13(4)	13(7)
C(6a)	5618(6)	−5339(11)	1268(8)	27(3)	49(10)	26(6)	3(5)	−1(4)	−25(7)
C(7)	6099(6)	−5732(10)	1909(10)	18(3)	31(8)	68(9)	4(4)	17(4)	27(7)
C(8)	6650(6)	−5426(12)	1864(9)	22(3)	84(12)	28(7)	6(5)	14(4)	−3(8)
C(9)	7155(6)	−5770(15)	2520(13)	18(3)	112(18)	103(13)	−16(6)	12(5)	−17(13)
C(10)	7233(6)	−5007(12)	3385(10)	31(4)	56(12)	43(8)	−4(6)	10(5)	11(9)
C(11)	6734(5)	−5178(10)	4132(10)	17(2)	25(9)	66(8)	11(4)	−7(4)	−22(7)
C(12)	6418(6)	−4187(13)	4502(12)	23(3)	56(13)	77(11)	−8(6)	11(5)	23(10)
C(13)	6094(6)	−3745(11)	3644(10)	19(3)	62(11)	52(8)	13(5)	3(4)	−19(8)
C(14)	5595(5)	−4150(10)	3263(8)	15(2)	41(9)	43(7)	11(4)	17(3)	6(7)
C(15)	5295(5)	−3785(10)	2386(10)	18(3)	48(9)	53(8)	−7(4)	−3(4)	6(8)
C(15a)	5082(6)	−4729(10)	1737(9)	23(3)	35(9)	39(7)	−2(4)	3(4)	−27(7)
C(16)	3664(6)	−3595(12)	1662(14)	16(3)	72(13)	102(14)	−10(5)	4(6)	−11(11)
C(17)	3076(8)	−3850(13)	2016(16)	25(5)	63(12)	142(17)	−27(7)	17(9)	−32(13)
C(18)	2899(9)	−3798(15)	2939(21)	48(5)	110(16)	161(25)	−5(8)	24(11)	−43(19)
C(19)	2338(12)	−4049(17)	3241(18)	51(9)	137(17)	164(21)	−47(11)	31(12)	−21(16)
C(20)	1912(8)	−4249(23)	2597(22)	36(5)	219(34)	166(32)	−19(11)	40(11)	−108(30)
C(21)	2067(10)	−4360(23)	1643(20)	32(6)	166(33)	155(26)	−5(12)	−15(10)	−51(25)
C(22)	2628(7)	−4074(20)	1325(17)	24(4)	209(26)	117(20)	−4(8)	−3(7)	−64(20)
C(23)	4327(7)	−4302(14)	−776(12)	25(4)	91(15)	77(11)	5(7)	−2(6)	4(11)
C(24)	4477(8)	−6620(15)	−379(14)	24(4)	97(16)	104(14)	16(8)	−10(7)	29(14)
C(25)	7830(7)	−5245(16)	3881(12)	29(4)	125(18)	65(10)	2(7)	−15(5)	30(11)
C(26)	6832(7)	−3358(12)	4950(10)	31(4)	51(10)	62(8)	−7(6)	−23(5)	19(9)
C(27)	6033(9)	−7614(17)	38(15)	38(5)	109(19)	106(14)	−3(9)	2(7)	16(14)
C(28)	6499(11)	−7787(19)	−622(21)	55(8)	93(22)	175(28)	−30(11)	29(13)	42(21)
C(29)	6105(7)	−5154(13)	5899(10)	30(4)	61(12)	42(8)	−8(6)	−14(5)	2(9)
C(30)	5599(6)	−5859(13)	6222(9)	17(3)	82(13)	36(6)	6(5)	6(4)	−9(8)
C(31)	5614(7)	−6336(14)	7083(9)	30(4)	110(17)	31(7)	11(7)	3(5)	23(9)
C(32)	5168(9)	−7065(14)	7398(13)	47(6)	69(15)	58(11)	6(8)	14(7)	−2(12)
C(33)	4711(7)	−7268(16)	6778(14)	19(3)	75(18)	104(14)	6(6)	4(6)	−6(14)
C(34)	4689(8)	−6810(16)	5901(16)	38(5)	121(16)	109(18)	15(8)	−20(8)	−60(16)
C(35)	5131(7)	−6034(17)	5637(12)	22(3)	115(20)	64(11)	−1(7)	0(5)	−32(13)
O(1′)	3141(9)	−6813(16)	2690(15)	93(8)	198(22)	217(19)	74(12)	85(11)	67(20)
C(1′)	2839(11)	−7096(22)	3450(19)	63(7)	139(29)	90(21)	27(13)	−17(11)	−12(22)
C(2′)	2257(11)	−7353(31)	3386(29)	43(7)	216(53)	328(44)	46(17)	43(15)	68(42)
C(3′)	3174(16)	−6815(41)	4183(27)	79(13)	568(80)	189(37)	−59(29)	−14(18)	111(50)

* In the form: $T = \exp\{-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)\}$.

Structure Analysis.—The position of the bromine atom was determined from three Harker sections of the three-dimensional Patterson synthesis as $x = 0{\cdot}075$, $y = 0{\cdot}180$, $z = 0{\cdot}220$. A first set of structure factors was calculated for 2318 reflexions, and the related three-dimensional Fourier synthesis was summed with phases based on the bromine atom alone. The initial R was 0·46, and the positions of 19 atoms were assigned from this electron-density distribution. The other atom positions were successively derived within seven subsequent Fourier refinements, and R was reduced to 0·35. The locations of 40 hydrogen atoms (except those of isopropyl alcohol) have been determined from a difference-Fourier synthesis.

At this stage, several cycles of least-squares refinement were carried out with the use of IBM 1620 programmes.[5] The final block-diagonal refinement with anisotropic temperature factors reduced R to 0·098 for the 2318 reflexions.


(I)


(II) R = H
(III) R = $-OC\cdot C_6H_4\cdot Br$-*p*

All observed intensities were included in the refinement with equal weight and those of magnitude less than twice the background count (the unobserved reflexions) were excluded.[6] B-Values of all hydrogen atoms were fixed at 1·923 Å^2.

Observed and calculated structure amplitudes are listed in Supplementary publication No. SUP 20314 (6 pp., 1 microfiche).* The atomic scattering factors were taken from ref. 7. The final positional and thermal parameters with their estimated standard deviations are given in Tables 1 and 2.

The bond distances and valency angles are given in Table 3, together with their estimated standard deviations computed from the least-squares residuals. The molecular packing arrangement along the b axis is illustrated in Figure 1. All intermolecular distances were calculated and the most significant contacts ($\leqslant$3·60 Å) are given in Table 4.

Absolute Configuration.—In the final stage of the analysis the absolute configuration of the molecule was determined by the anomalous dispersion method.[8] The differences between $I_0(hkl)$ and $I_0(\bar{h}\bar{k}\bar{l})$ were measured visually from rotation photographs about the b axis, taken with Cr-K_α radiation (λ = 2·2909 Å). Structure factors were calculated for all the hkl and $\bar{h}\bar{k}\bar{l}$ reflexions, with a scattering factor for the bromine atom of the form: $f_{Br} = f_{Br} + \Delta f'_{Br} + i\Delta f''_{Br}$, when $\Delta f'_{Br} = -0{\cdot}6$ and $\Delta f''_{Br} = 2{\cdot}7$. The

TABLE 2

Final fractional hydrogen atomic co-ordinates ($\times 10^3$) with standard deviations in parentheses, and isotropic temperature factors

Atom	x	y	z	B/Å^2
H(O7)	544(4)	−246(8)	171(7)	1·923
H(N2)	382(4)	−587(8)	233(7)	1·923
H(C3)	381(4)	−490(8)	74(7)	1·923
H(C3a)	462(4)	−344(8)	97(7)	1·923
H(C4)	518(4)	−448(8)	−23(7)	1·923
H(C6)	521(4)	−679(9)	105(7)	1·923
H(C6a)	578(4)	−472(8)	86(7)	1·923
H(C7)	600(4)	−631(7)	234(7)	1·923
H(C8)	674(4)	−486(8)	151(7)	1·923
H(C9)a	701(4)	−647(8)	280(7)	1·923
H(C9)b	745(4)	−576(8)	212(8)	1·923
H(C10)	722(4)	−418(8)	324(7)	1·923
H(C13)	630(4)	−319(9)	338(7)	1·923
H(C14)	542(4)	−467(8)	359(7)	1·923
H(C15)	497(4)	−347(8)	267(7)	1·923
H(C16)a	394(4)	−320(8)	214(7)	1·923
H(C16)b	360(4)	−298(8)	101(7)	1·923
H(C18)	323(4)	−363(8)	337(7)	1·923
H(C19)	226(4)	−403(8)	388(7)	1·923
H(C20)	157(4)	−431(8)	275(7)	1·923
H(C21)	178(4)	−447(8)	123(7)	1·923
H(C22)	272(4)	−415(9)	72(7)	1·923
H(C23)a	440(4)	−475(8)	−134(7)	1·923
H(C23)b	387(4)	−448(8)	−55(7)	1·923
H(C23)c	427(4)	−349(8)	−81(7)	1·923
H(C24)a	412(4)	−658(8)	−69(7)	1·923
H(C24)b	453(4)	−740(8)	−27(7)	1·923
H(C25)a	806(4)	−543(8)	335(7)	1·923
H(C25)b	781(4)	−590(8)	426(7)	1·923
H(C25)c	797(4)	−456(8)	432(7)	1·923
H(C26)a	715(4)	−364(8)	531(7)	1·923
H(C26)b	667(4)	−281(8)	535(7)	1·923
H(C26)c	696(4)	−273(8)	452(7)	1·923
H(C28)a	691(4)	−826(9)	−43(7)	1·923
H(C28)b	642(4)	−823(9)	−106(7)	1·923
H(C28)c	662(4)	−706(8)	−95(7)	1·923
H(C31)	595(4)	−617(8)	751(7)	1·923
H(C32)	516(4)	−723(8)	804(7)	1·923
H(C34)	436(4)	−696(8)	547(7)	1·923
H(C35)	512(4)	−573(8)	496(7)	1·923

TABLE 3

Interatomic distances (Å) and angles (°), with standard deviations in parentheses

(*a*) Bond distances

(i) Skeleton of the molecule			
C(1)–C(15a)	1·557(18)	C(6a)–C(15a)	1·573(20)
C(3)–C(3a)	1·555(21)	C(8)–C(9)	1·527(21)
C(3)–C(16)	1·547(22)	C(9)–C(10)	1·540(24)
C(3a)–C(4)	1·562(23)	C(10)–C(11)	1·553(20)
C(3a)–C(15a)	1·541(20)	C(10)–C(25)	1·548(23)
C(4)–C(5)	1·510(22)	C(11)–C(12)	1·505(21)
C(4)–C(23)	1·512(24)	C(12)–C(13)	1·508(21)
C(5)–C(6)	1·528(20)	C(12)–C(26)	1·519(22)
C(6)–C(6a)	1·570(18)	C(14)–C(15)	1·470(18)
C(6a)–C(7)	1·491(19)	C(15)–C(15a)	1·549(19)
N(2)–C(1)	1·379(16)	N(2)–C(3)	1·414(19)
O(2)–C(6)	1·418(18)	O(5)–C(12)	1·452(18)
O(7)–C(15)	1·399(16)		
C(5)–C(24)	1·298(24)	C(7)–C(8)	1·305(20)
C(13)–C(14)	1·345(19)		
O(1)–C(1)	1·190(16)	O(4)–C(11)	1·177(17)
(ii) *p*-Bromobenzoate group			
Br–C(33)	1·866(18)	C(30)–C(35)	1·356(22)
O(5)–C(29)	1·313(18)	C(31)–C(32)	1·421(26)
O(6)–C(29)	1·190(19)	C(32)–C(33)	1·374(27)
C(29)–C(30)	1·507(21)	C(33)–C(34)	1·348(30)
C(30)–C(31)	1·338(20)	C(34)–C(35)	1·432(27)

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* For details of Supplementary Publications see Notice to Authors No. 7 in *J. Chem. Soc.* (*A*), 1970, Issue No. 20 (items less than 10 pp. will be supplied as full size copies).

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[7] 'International Tables for *X*-Ray Crystallography,' vol. 3, Kynoch Press, Birmingham, 1962, pp. 201—207.

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TABLE 3 (*Continued*)

(iii) Phenyl group

C(16)–C(17)	1·458(25)	C(19)–C(20)	1·343(37)
C(17)–C(18)	1·350(36)	C(20)–C(21)	1·384(41)
C(17)–C(22)	1·426(30)	C(21)–C(22)	1·394(30)
C(18)–C(19)	1·376(35)		

(iv) *O*-Acetyl group

O(2)–C(27)	1·361(24)	C(27)–C(28)	1·419(35)
O(3)–C(27)	1·205(28)		

(v) Isopropyl group

O(1′)–C(1′)	1·310(34)	C(1′)–C(3′)	1·320(46)
C(1′)–C(2′)	1·361(37)		

(*b*) Valency angles

C(6)–O(2)–C(27)	117·8(12)	C(12)–C(13)–C(14)	126·2(13)
C(12)–O(5)–C(29)	119·6(11)	C(13)–C(14)–C(15)	127·1(12)
C(1)–N(2)–C(3)	115·8(11)	O(7)–C(15)–C(14)	111·6(10)
O(1)–C(1)–N(2)	125·5(12)	O(7)–C(15)–C(15a)	108·7(11)
O(1)–C(1)–C(15a)	127·2(11)	C(14)–C(15)–C(15a)	113·8(10)
N(2)–C(1)–C(15a)	107·1(10)	C(1)–C(15a)–C(3a)	104·0(11)
N(2)–C(3)–C(3a)	105·3(11)	C(1)–C(15a)–C(6a)	110·4(10)
N(2)–C(3)–C(16)	113·9(13)	C(1)–C(15a)–C(15)	109·2(10)
C(3a)–C(3)–C(16)	111·2(12)	C(3a)–C(15a)–C(6a)	112·5(10)
C(3)–C(3a)–C(4)	112·3(11)	C(3a)–C(15a)–C(15)	109·2(10)
C(3)–C(3a)–C(15a)	105·6(11)	C(6a)–C(15a)–C(15)	111·0(11)
C(4)–C(3a)–C(15a)	113·2(11)	C(3)–C(16)–C(17)	113·7(13)
C(3a)–C(4)–C(5)	107·7(13)	C(16)–C(17)–C(18)	125·7(19)
C(3a)–C(4)–C(23)	113·3(12)	C(16)–C(17)–C(22)	117·6(19)
C(5)–C(4)–C(23)	114·8(13)	C(18)–C(17)–C(22)	116·3(18)
C(4)–C(5)–C(6)	112·0(12)	C(17)–C(18)–C(19)	123·8(23)
C(4)–C(5)–C(24)	125·1(14)	C(18)–C(19)–C(20)	120·0(25)
C(6)–C(5)–C(24)	122·7(15)	C(19)–C(20)–C(21)	118·6(21)
O(2)–C(6)–C(5)	108·8(11)	C(20)–C(21)–C(22)	120·9(23)
O(2)–C(6)–C(6a)	106·6(11)	C(17)–C(22)–C(21)	118·8(22)
C(5)–C(6)–C(6a)	114·5(11)	O(2)–C(27)–O(3)	119·7(18)
C(6)–C(6a)–C(7)	112·6(11)	O(2)–C(27)–C(28)	110·6(18)
C(6)–C(6a)–C(15a)	108·0(11)	O(3)–C(27)–C(28)	129·6(21)
C(7)–C(6a)–C(15a)	118·0(10)	O(5)–C(29)–O(6)	125·5(14)
C(6a)–C(7)–C(8)	125·2(12)	O(5)–C(29)–C(30)	111·0(12)
C(7)–C(8)–C(9)	127·5(13)	O(6)–C(29)–C(30)	123·3(14)
C(8)–C(9)–C(10)	112·8(13)	C(29)–C(30)–C(31)	120·0(13)
C(9)–C(10)–C(11)	111·1(12)	C(29)–C(30)–C(35)	120·5(13)
C(9)–C(10)–C(25)	109·6(13)	C(31)–C(30)–C(35)	119·3(15)
C(11)–C(10)–C(25)	108·1(12)	C(30)–C(31)–C(32)	122·4(15)
O(4)–C(11)–C(10)	120·2(12)	C(31)–C(32)–C(33)	117·1(16)
O(4)–C(11)–C(12)	121·8(13)	Br–C(33)–C(32)	116·0(14)
C(10)–C(11)–C(12)	117·9(11)	Br–C(33)–C(34)	122·3(14)
O(5)–C(12)–C(11)	106·4(12)	C(32)–C(33)–C(34)	121·5(17)
O(5)–C(12)–C(13)	105·0(11)	C(33)–C(34)–C(35)	119·1(18)
O(5)–C(12)–C(26)	114·2(12)	C(30)–C(35)–C(34)	119·9(17)
C(11)–C(12)–C(13)	104·5(12)	O(1′)–C(1′)–C(2′)	120·9(28)
C(11)–C(12)–C(26)	112·9(12)	O(1′)–C(1′)–C(3′)	104·9(26)
C(13)–C(12)–C(26)	112·7(13)	C(2′)–C(1′)–C(3′)	132·0(31)

(*c*) Bond distances associated with hydrogen positions

O(7)–H(O7)	0·92(10)	C(21)–H(C21)	0·87(10)
N(2)–H(N2)	0·98(10)	C(22)–H(C22)	0·87(11)
C(3)–H(C3)	1·02(10)	C(23)–H(C23)a	0·99(10)
C(3a)–H(C3a)	1·02(10)	C(23)–H(C23)b	1·08(10)
C(4)–H(C4)	1·00(10)	C(23)–H(C23)c	0·99(10)
C(6)–H(C6)	0·96(10)	C(24)–H(C24)a	0·90(10)
C(6a)–H(C6a)	1·01(10)	C(24)–H(C24)b	0·98(11)
C(7)–H(C7)	0·96(10)	C(25)–H(C25)a	0·93(10)
C(8)–H(C8)	0·86(10)	C(25)–H(C25)b	0·97(11)
C(9)–H(C9)a	1·00(10)	C(25)–H(C25)c	1·08(10)
C(9)–H(C9)b	0·86(10)	C(26)–H(C26)a	0·95(10)
C(10)–H(C10)	1·02(10)	C(26)–H(C26)b	0·94(10)
C(13)–H(C13)	0·90(10)	C(26)–H(C26)c	1·01(10)
C(14)–H(C14)	0·88(10)	C(28)–H(C28)a	1·13(10)
C(15)–H(C15)	0·91(10)	C(28)–H(C28)b	0·84(11)
C(16)–H(Cl6)a	1·04(10)	C(28)–H(C28)c	1·03(11)
C(16)–H(C16)b	1·18(11)	C(31)–H(C31)	1·00(10)
C(18)–H(C18)	1·00(10)	C(32)–H(C32)	0·92(10)
C(19)–H(C19)	0·91(11)	C(34)–H(C34)	0·96(10)
C(20)–H(C20)	0·80(10)	C(35)–H(C35)	1·00(10)

results (see Table 5) unanimously indicated that the parameters of Table 1 represent the right-hand co-ordinate system.

TABLE 4

Intermolecular distances ($\leqslant$3·60 Å)

Br · · · O(6$^{\mathrm{IV}}$)	3·511	O(7) · · · C(1$^{\mathrm{V}}$)	3·439
Br · · · C(29$^{\mathrm{IV}}$)	3·582	O(7) · · · O(1′$^{\mathrm{V}}$)	3·220
O(1) · · · C(15$^{\mathrm{III}}$)	3·247	O(7) · · · C(3′$^{\mathrm{V}}$)	3·406
O(4) · · · C(16$^{\mathrm{III}}$)	3·478	C(13) · · · O(1′$^{\mathrm{V}}$)	3·482
O(6) · · · C(9$^{\mathrm{VI}}$)	3·535	C(23) · · · C(33$^{\mathrm{V}}$)	3·600
O(6) · · · C(2′$^{\mathrm{II}}$)	3·494	C(24) · · · C(32$^{\mathrm{I}}$)	3·519
N(2)–H · · · O(1′)	2·971	O(1) · · · H–O(7$^{\mathrm{III}}$)	2·655

The roman numerals as superscripts refer to the following equivalent position relative to the reference molecule at x, y, z:

I	$x, y, z - 1$	IV	$1 - x, -\frac{1}{2} + y, \frac{3}{2} - z$
II	$\frac{1}{2} + x, -\frac{3}{2} - y, 1 - z$	V	$1 - x, \frac{1}{2} + y, \frac{1}{2} - z$
III	$1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$	VI	$\frac{3}{2} - x, -1 - y, \frac{1}{2} + z$

TABLE 5

Determination of the absolute configuration
(Cr-K_{α} radiation)

h	k	l	$\lvert F_c(hkl)\rvert^2$	$\lvert F_c(h\bar{k}l)\rvert^2$	$I_0(hkl) \gtrless I_0(h\bar{k}l)$
5	1	3	8082	7157	>
7	1	1	7938	9604	<
8	1	4	900	428	>
9	1	4	1339	985	>
10	1	1	605	432	>
12	1	1	1128	967	>

DISCUSSION

The present *X*-ray analysis has established the complete structure, including the absolute configuration, of isozygosporin A *p*-bromobenzoate. The absolute configuration of the molecule, (*E*,*E*)-(3*S*,3a*R*,4*S*,6*S*,6a*R*,10*S*,12*R*,15*R*,15a*R*)-6-acetoxy-3-benzyl-12-(*p*-bromobenzoyloxy)-3,3a,4,5,6,6a,9,10,12,15-decahydro-15-hydroxy-4,10,12-trimethyl-5-methylene-1*H*-cycloundeca[*d*]isoindole-1,11(2*H*)-dione, is illustrated[9] in Figure 2.

The skeleton of the molecule consists of a five-membered ring A, a six-membered ring B, and an eleven-membered ring C. For the most part the corresponding bond distances are not significantly different from expected values.[10] The mean values of the bond distances for the various types of bonds are calculated to be 1·534 for C–C single bonds, 1·396 for C–N single bonds, 1·426 for C–O single bonds, 1·316 for C=C double bonds, and 1·183 Å for C=O double bonds in the skeleton of the molecule. Two or three differences which appear to be significant in the terms of the estimated standard deviations are more likely to indicate a slight underestimation of errors.

The deviations from the best plane through the various atom groups are in Table 6. Ring A is a slightly distorted envelope and ring B has a distorted boat conformation with atoms [C(5), C(6), C(3a), C(15a)] in the plane. In order to obtain detailed information on the molecular shape and conformational features, the displacements from the plane through

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[9] C. K. Johnson, 1965, ORTEP, A Fortran thermal-ellipsoid plot program, Oak Ridge National Laboratory, Tennessee.

[10] L. E. Sutton, *Chem. Soc., Special Publ.*, 1965, No. 18.

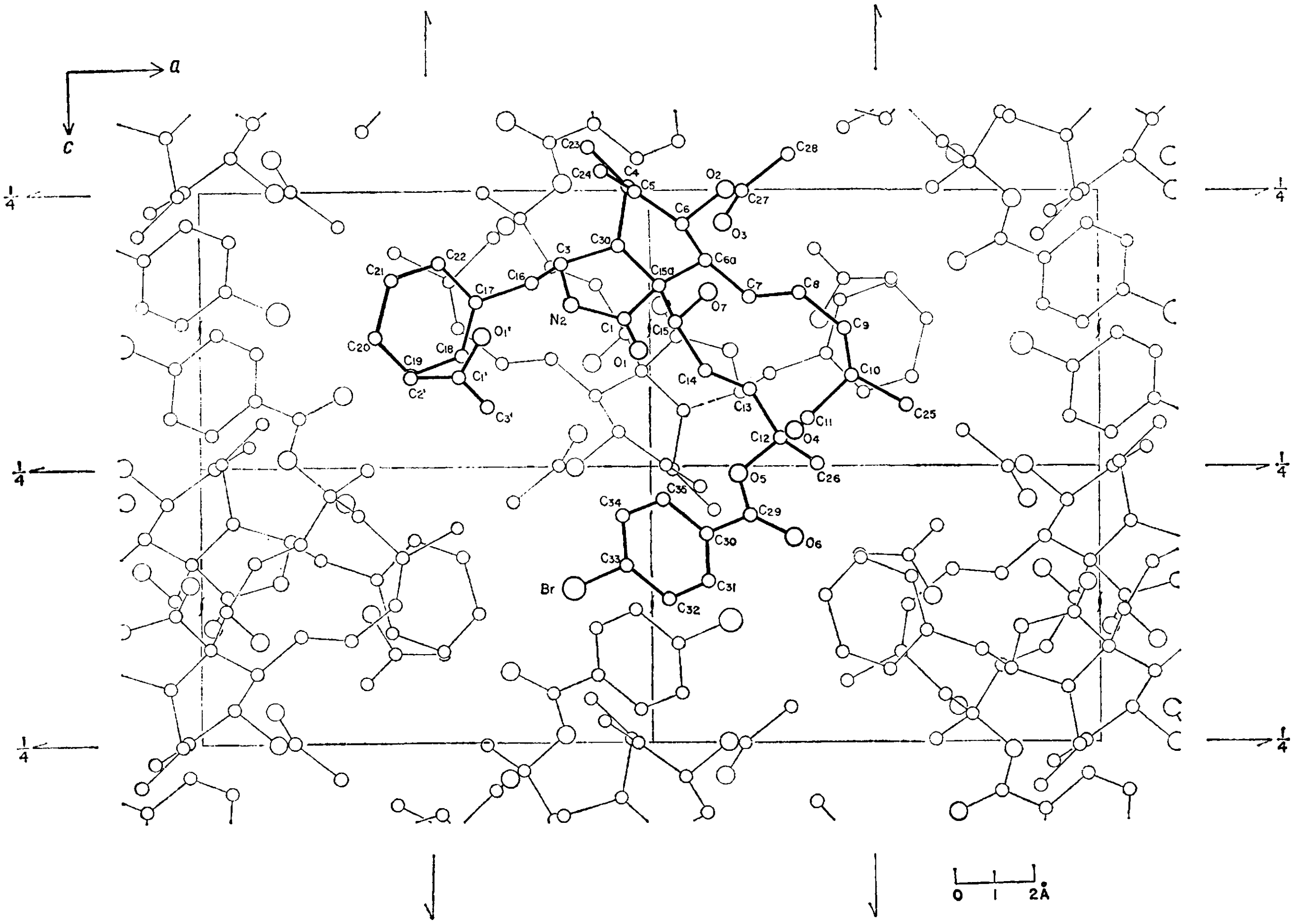

FIGURE 1 The view of the structure looking down the *b* axis (*b* axis down)

TABLE 6

Equations of planes, and in square brackets displacements of the atoms (Å), from the mean planes: X, Y, and Z are orthogonal co-ordinates (Å)

Plane (I):		
C(30)—(35)	$0{\cdot}5127X - 0{\cdot}7602Y - 0{\cdot}3987Z = 8{\cdot}5078$	[C(30) −0·01, C(31) 0·00, C(32) 0·00, C(33), 0·00, C(34) −0·02, C(35) 0·03, Br 0·01, O(5) 0·15, O(6) −0·42, C(29) −0·12]
Plane (II):		
C(17)—(22)	$0{\cdot}2322X - 0{\cdot}9683Y + 0{\cdot}0919Z = 6{\cdot}4438$	[C(17) −0·02, C(18) 0·01, C(19) −0·02, C(20) 0·04, C(21) −0·04, C(22) 0·03, C(16) 0·01]
Plane (III):		
C(1), N(2), C(15a), and O(1)	$0{\cdot}0769X - 0{\cdot}6826Y - 0{\cdot}7266Z = 3{\cdot}0920$	[C(1) 0·00, N(2) 0·00, C(15a) 0·00, O(1) 0·00]
Plane (IV):		
C(4)—(6), and C(24)	$0{\cdot}5627X - 0{\cdot}0047Y - 0{\cdot}8266Z = 6{\cdot}1226$	[C(4) 0·00, C(5) 0·01, C(6) 0·00, C(24) 0·00]
Plane (V):		
C(10)—(12), and O(4)	$0{\cdot}6605X + 0{\cdot}0523Y + 0{\cdot}7489Z = 14{\cdot}0643$	[C(10) 0·00, C(11) 0·01, C(12) 0·00, O(4) 0·00]
Plane (VI):		
O(2), O(3), C(27), and C(28)	$0{\cdot}6626X + 0{\cdot}2758Y + 0{\cdot}6962Z = 6{\cdot}5205$	[O(2) 0·00, O(3) 0·00, C(27) 0·00, C(28) 0·00]

C(4), C(6a), and C(15) in the skeleton of the molecule were calculated, and are shown in Figure 3. The γ-lactam ring A is *cis*-fused to the six-membered ring B, and the junction between ring B and ring C is *trans*.

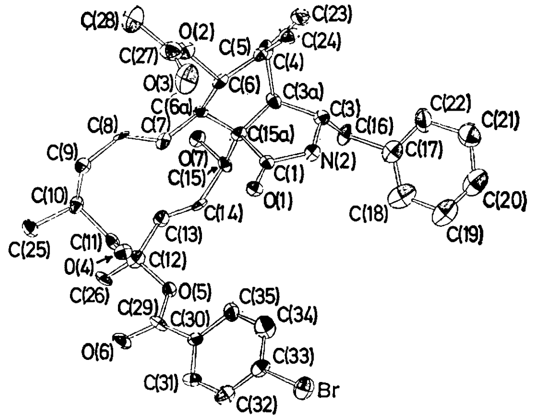

FIGURE 2 A perspective view of the molecule

The mean values of the angles are calculated to be 107·7° for ring A and 111·3°[11] for the ring B. The

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[11] J. B. Lambert, R. E. Carhart, P. W. R. Corfield, and J. H. Enemark, *Chem. Comm.*, 1968, 999.

various torsion angles in ring C were calculated and are listed in Table 7. The mean value of the angles at the C(7)=C(8) and C(13)=C(14) double bonds is 126·5°, while the other mean C–C–C bond angle in ring C is 112·7°.

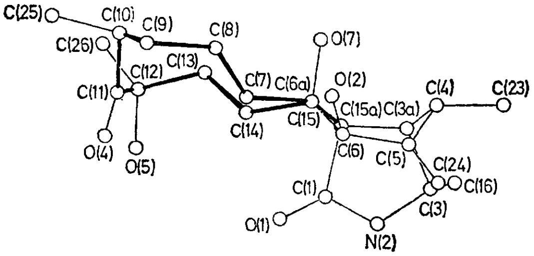

FIGURE 3 Displacements from the plane through C(4), C(6a), and C(15) in the skeleton of the molecule

The *O*-acetyl side-chain and the isopropyl alcohol molecule are planar. Two oxygen atoms at C(11) and C(12) are on the same side, and the other oxygen atom at C(15) is on the opposite side of ring C. Two methyl groups at C(10), and C(12) are both *gauche* to O(14) and the group of atoms C(1), C(3a), C(6a), C(15), and C(15a) is tetrahedral (109·3°). The hydrogen atoms at the C(7)=C(8) double bond are *trans* and the hydrogen atoms at the other C(13)=C(14) double bond in ring C are also *trans*.

TABLE 7

The torsion angles (°) in the eleven-membered ring C

System	Angles
C(6a)–C(7)–C(8)–C(9)	177·7
C(7)–C(8)–C(9)–C(10)	−90·9
C(8)–C(9)–C(10)–C(11)	73·2
C(9)–C(10)–C(11)–C(12)	−130·8
C(10)–C(11)–C(12)–C(13)	66·3
C(11)–C(12)–C(13)–C(14)	76·4
C(12)–C(13)–C(14)–C(15)	−174·9
C(13)–C(14)–C(15)–C(15a)	136·2
C(14)–C(15)–C(15a)–C(6a)	−69·4
C(15)–C(15a)–C(6a)–C(7)	52·0
C(15a)–C(6a)–C(7)–C(8)	−119·0

The most significant intermolecular distances are N(2)–H $\cdots$ O(1′) 2·971 Å, and O(1) $\cdots$ H–O(7^{III}) 2·655 Å, and these represent typical hydrogen bonds. The packing is efficient with several contacts approaching the sum of the van der Waals radii; the shortest contact, excluding hydrogen atoms, is 3·224 Å for O(1) $\cdots$ C(15^{III}).

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