Crystal and Molecular Structure of a New Cyclopropane Triterpene: Passifloric Acid Methyl Ester [Methyl (22*R*,24*S*)-22,31-Epoxy-1α,3β,24,31-tetrahydroxy-24-methyl-9,19-cyclo-9β-lanostan-28-oate]

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The crystal structure of the title compound, a new cyclopropane triterpene extracted from *Passiflora edulis*, has been determined by *X*-ray diffraction from diffractometer data and refined by least-squares to *R* 0.047 for 3 078 reflections. Crystals are monoclinic, space group $P2_1$, $a = 8.161(4)$, $b = 25.472(8)$, $c = 7.697(5)$ Å, $\beta = 101.4(1)°$, $Z = 2$. The ring fusions A–B and C–D are *trans*, while B–C is *cis*, ring C being in the boat conformation.

RECENTLY Bombardelli *et al.*[1] have isolated from *Passiflora edulis* Sims collected in India a new cyclopropane triterpene glycoside, named by them passiflorine. The structure of this compound (I) has been determined by chemical and spectroscopic data. In order to confirm the structure and to determine the conformation of the molecule an *X*-ray analysis on the methyl ester of passifloric acid has been carried out and the results are reported here.

EXPERIMENTAL

The compound was recrystallized from methanol as monoclinic colourless prisms. A crystal with dimensions *ca.* 0.3 × 0.6 × 0.3 mm was used for intensity measurements. Space group and initial unit-cell parameters were obtained from oscillation and Weissenberg photographs. Accurate unit-cell parameters were obtained by a least-squares treatment of $(\theta,\chi,\phi)_{hkl}$ values of 16 reflections measured on a Siemens single-crystal diffractometer.

Crystal Data.—$C_{32}H_{52}O_7$, $M = 548.4$. Monoclinic, $a = 8.161(4)$, $b = 25.472(8)$, $c = 7.697(5)$ Å, $\beta = 101.4(1)°$, $U = 1\,568.5$ Å^3, $Z = 2$, $D_m = 1.16$ g cm^{-3}. Cu-K_α radiation, $\lambda = 1.541\,8$ Å, $\mu(\text{Cu-}K_\alpha) = 6.51$ cm^{-1}. Space group $P2_1$ from systematic absences.

The diffraction data were collected automatically by use of Cu-K_α radiation. The moving-counter moving-crystal scan and the so-called ' five-points '[2] technique were used for intensity and background measurement. Of 3 260 independent reflections measured ($\theta_{max.}$ 70°), 182 having intensities $<2[\sigma^2(I) + (0.01 \times I)^2]^{\frac{1}{2}}$ [where *I* is the relative intensity and $\sigma^2(I)$ its variance] were considered unobserved and not used in the refinement. No corrections for absorption were applied.

Structure Analysis and Refinement.—The structure amplitudes were put on an absolute scale by the method of Wilson[3] (B 4.6 Å^2) and normalized structure amplitudes $|E_{hkl}|$ were then obtained by using the overall temperature parameter. 361 Reflections with $E \geqslant 1.47$ were used for phase determination. The structure was solved by the multisolution and phase permutation technique through the programme MULTAN.[4] An *E* map computed by use of the most consistent set of phases revealed the whole structure. A structure-factor calculation based on the co-ordinates derived from the *E* map for all atoms gave *R* 0.29. Refinement was carried out by cycles of block-diagonal least-squares, first with isotropic and then with

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[1] E. Bombardelli, A. Bonati, B. Gabetta, E. M. Martinelli, G. Mustich, and B. Danieli, *Phytochemistry*, 1975, **13**, 2661.

[2] W. Hoppe, *Acta Cryst.*, 1969, **A25**, 67.

[3] A. J. Wilson, *Nature*, 1942, **150**, 151.

[4] P. Main, M. M. Woolfson, and G. Germain, MULTAN, A Computer programme for the Automatic solution of Crystal Structures, 1971, University of York.

anisotropic thermal parameters, when R was reduced to 0.088. A difference-Fourier synthesis revealed 67 residual peaks >0.3 eÅ^{-3}, of which 52 were interpreted as being due to hydrogen atoms. Further least-squares cycles were computed, hydrogen atoms being included with isotropic thermal parameters, and led to a final R of 0.047. No attempts were made to determine the absolute configuration by the Bijovet technique, since the crystal shape was very irregular and it was therefore impossible to make an accurate correction for absorption effects. Atomic scattering factors of ref. 5 were used for oxygen and carbon, and of ref. 6 for hydrogen. Measured and calculated structure factors and anisotropic thermal parameters are listed in Supplementary Publication SUP 21925 (16 pp., 1 microfiche).* Positional parameters together with their standard deviations are given in Table 1.

TABLE 1

(*a*) Fractional co-ordinates ($\times 10^4$) with estimated standard deviations in parentheses, for non-hydrogen atoms

	x	*y*	*z*
O(1)	8 708(3)	8 595(1)	1 233(4)
O(2)	13 680(4)	9 012(1)	3 608(4)
O(3)	15 397(3)	8 571(2)	−311(5)
O(4)	13 141(4)	9 088(1)	−876(4)
O(5)	−65(4)	5 005(1)	−4 678(5)
O(6)	4 224(3)	4 790(1)	−2 709(4)
O(7)	1 752(4)	4 629(1)	−1 762(4)
C(1)	9 773(5)	8 212(2)	2 232(6)
C(2)	11 224(5)	8 513(2)	3 400(6)
C(3)	12 374(5)	8 782(2)	2 345(5)
C(4)	13 074(4)	8 385(2)	1 137(5)
C(5)	11 524(4)	8 125(1)	−98(5)
C(6)	11 927(5)	7 797(2)	−1 615(6)
C(7)	10 298(6)	7 576(2)	−2 728(6)
C(8)	9 588(6)	7 164(2)	−1 662(5)
C(9)	9 362(4)	7 371(1)	185(5)
C(10)	10 434(4)	7 836(1)	998(5)
C(11)	7 621(5)	7 334(2)	612(6)
C(12)	6 620(6)	6 826(2)	15(6)
C(13)	7 327(5)	6 503(2)	−1 354(6)
C(14)	7 994(5)	6 889(2)	−2 660(5)
C(15)	8 302(6)	6 507(2)	−4 120(6)
C(16)	6 907(6)	6 080(2)	−4 266(6)
C(17)	6 056(5)	6 159(2)	−2 651(6)
C(18)	8 740(6)	6 145(2)	−352(7)
C(19)	10 852(5)	7 285(2)	1 699(5)
C(20)	5 509(6)	5 636(2)	−1 902(7)
C(21)	4 697(8)	5 706(2)	−313(7)
C(22)	4 403(5)	5 328(2)	−3 388(6)
C(23)	2 590(6)	5 504(2)	−4 044(7)
C(24)	1 681(5)	4 986(2)	−4 659(6)
C(25)	1 907(6)	4 833(2)	−6 521(7)
C(26)	1 289(8)	4 230(3)	−6 967(8)
C(27)	881(9)	5 203(3)	−7 899(8)
C(28)	2 565(5)	4 611(2)	−3 206(6)
C(29)	14 279(5)	7 986(2)	2 226(6)
C(30)	14 037(5)	8 685(2)	−74(5)
C(31)	13 905(7)	9 397(2)	−2 100(7)
C(32)	6 640(5)	7 281(2)	−3 539(7)

(*b*) Fractional co-ordinates ($\times 10^3$) and isotropic thermal parameters (in 10^{-1} Å^2) for hydrogen atoms

TABLE 1 (*Continued*)

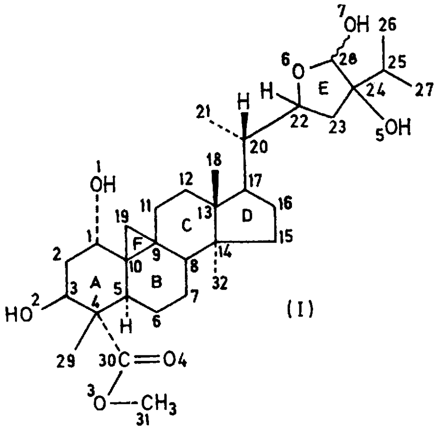
	x	*y*	*z*	*B*
H[O(1)]	768(5)	846(2)	66(5)	57(10)
H[O(2)]	1 424(5)	924(2)	303(5)	63(11)
H[O(5)]	−43(5)	506(2)	−345(6)	67(11)
H[O(7)]	143(5)	428(2)	−230(6)	80(12)
H[C(1)]	912(4)	803(1)	298(5)	45(8)
H[C(2)]	1 187(4)	829(1)	434(5)	49(9)
H[C(2)]	1 065(5)	875(1)	401(5)	49(9)
H[C(3)]	1 171(4)	908(2)	161(5)	48(10)
H[C(5)]	1 064(5)	840(1)	−77(5)	50(9)
H[C(6)]	1 278(5)	748(1)	−112(5)	51(9)
H[C(6)]	1 247(5)	799(2)	−225(5)	53(11)
H[C(7)]	935(5)	787(2)	−327(6)	56(10)
H[C(7)]	1 058(4)	739(1)	−382(5)	48(10)
H[C(8)]	1 050(4)	689(1)	−139(5)	43(9)
H[C(11)]	666(5)	767(2)	−4(6)	59(9)
H[C(11)]	772(4)	741(1)	191(5)	45(8)
H[C(12)]	635(5)	691(1)	−60(5)	53(10)
H[C(12)]	650(5)	663(2)	97(5)	56(12)
H[C(15)]	951(5)	628(2)	−362(5)	58(11)
H[C(15)]	835(5)	667(2)	−514(5)	53(10)
H[C(16)]	756(5)	567(2)	−413(5)	57(10)
H[C(16)]	604(5)	611(2)	−548(5)	50(9)
H[C(17)]	497(5)	638(1)	−313(5)	51(9)
H[C(18)]	963(5)	635(2)	57(6)	58(11)
H[C(18)]	845(5)	588(2)	34(5)	55(12)
H[C(18)]	931(5)	596(2)	−120(6)	55(12)
H[C(19)]	1 183(4)	708(1)	134(5)	45(9)
H[C(19)]	1 058(5)	718(2)	298(5)	59(11)
H[C(20)]	654(5)	540(1)	−138(5)	53(10)
H[C(21)]	389(5)	551(2)	−19(5)	89(12)
H[C(21)]	397(5)	604(2)	−77(5)	62(11)
H[C(21)]	516(5)	589(2)	87(6)	88(13)
H[C(22)]	505(5)	524(1)	−445(5)	53(11)
H[C(23)]	263(5)	577(2)	−528(5)	59(10)
H[C(23)]	228(5)	572(2)	−295(5)	58(10)
H[C(25)]	320(5)	473(2)	−664(5)	55(11)
H[C(26)]	164(5)	415(3)	−821(6)	97(13)
H[C(26)]	3(6)	420(3)	−707(6)	88(12)
H[C(26)]	187(7)	398(3)	−601(6)	79(12)
H[C(27)]	99(5)	514(2)	−901(6)	100(10)
H[C(27)]	187(5)	550(2)	−789(5)	91(12)
H[C(27)]	−46(5)	513(2)	−791(5)	95(11)
H[C(28)]	259(4)	426(1)	−369(5)	51(9)
H[C(29)]	1 367(5)	776(2)	311(6)	63(10)
H[C(29)]	1 437(5)	765(2)	162(6)	57(9)
H[C(29)]	1 526(5)	817(2)	295(7)	55(10)
H[C(31)]	1 309(5)	965(2)	−313(6)	68(11)
H[C(31)]	1 428(6)	917(2)	−299(5)	81(10)
H[C(31)]	1 496(6)	952(2)	−158(6)	92(11)
H[C(32)]	551(5)	715(2)	−398(5)	61(10)
H[C(32)]	721(6)	753(2)	−447(6)	73(11)
H[C(32)]	645(7)	751(2)	−278(5)	81(12)

Temperature factors are of the form $\exp(-B\sin^2\theta/\lambda^2)$.

All calculations were carried out on a CDC Cyber 76 of Centro di Calcolo Interuniversitario dell'Italia Nord-Orientale, Casalecchio, Bologna.

MOLECULAR GEOMETRY AND DISCUSSION

The atom labelling system used in the analysis for the non-hydrogen skeleton is shown in (I) and a projection



(I)

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* For details see Notice to Authors No. 7 in *J.C.S. Perkin II*, 1975, Index issue.

[5] D. T. Cromer and J. B. Mann, *Acta Cryst.*, 1968, **A24**, 321.

[6] R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, 1965, **42**, 3175.

of the molecule on (100) is given in the Figure. The present *X*-ray analysis has established the structure of passifloric acid to be in accord with that obtained by chemical and spectroscopic investigation. The absolute configuration at the carbon atoms C(22) and C(24) is assumed to be as determined chemically, so the stereochemistry of the remaining twelve chiral carbon atoms can be assigned. The structure possesses a 24-methylcycloartane skeleton. Bond lengths and angles (Table 2) are not significantly different from expected values, with the exception of severe distortions observed in the bond angles of rings B and C due to the presence of the

TABLE 2

Bond distances (Å), and bond and torsion angles (°), with estimated standard deviations in parentheses

(*a*) Bond distances

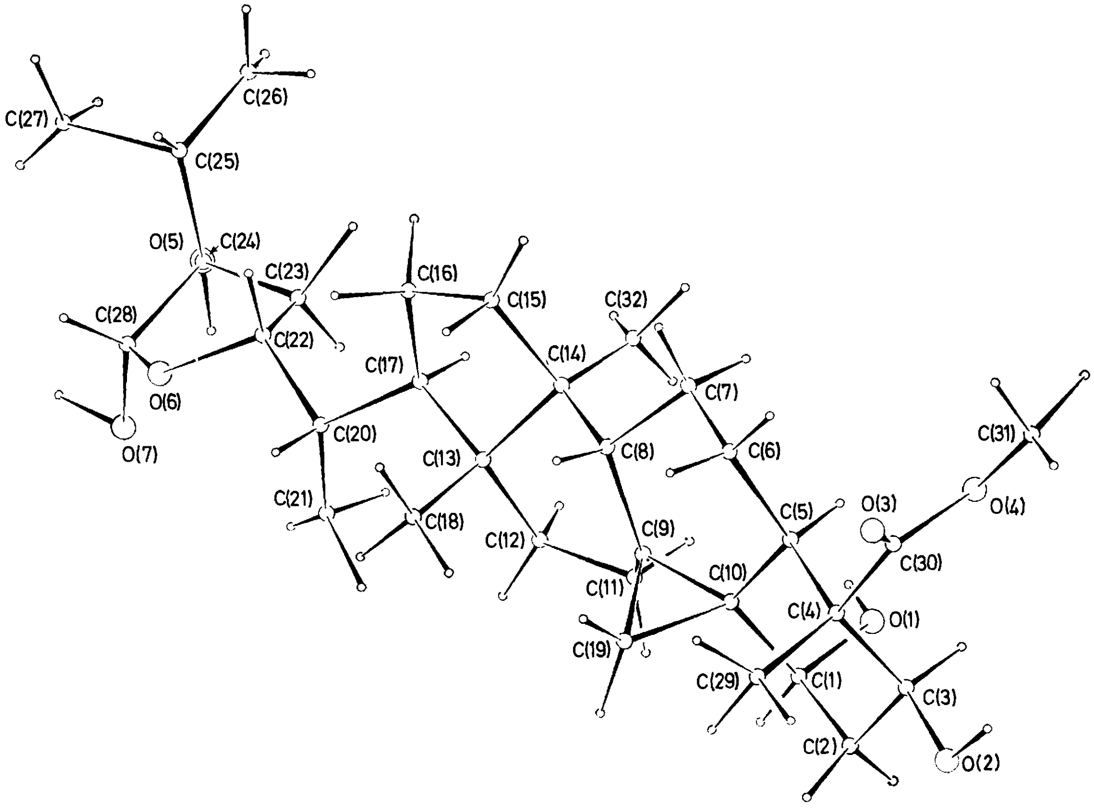
O(1)–C(1)	1.426(5)	C(4)–C(30)	1.536(6)	C(13)–C(18)	1.551(6)
O(2)–C(3)	1.419(4)	C(5)–C(6)	1.524(6)	C(14)–C(15)	1.544(6)
O(3)–C(30)	1.196(5)	C(5)–C(10)	1.530(5)	C(14)–C(32)	1.543(6)
O(4)–C(30)	1.338(5)	C(6)–C(7)	1.539(6)	C(15)–C(16)	1.562(7)
O(4)–C(31)	1.459(6)	C(7)–C(8)	1.516(6)	C(16)–C(17)	1.552(7)
O(5)–C(24)	1.423(5)	C(8)–C(9)	1.561(5)	C(17)–C(20)	1.552(7)
O(6)–C(22)	1.484(5)	C(8)–C(14)	1.542(5)	C(20)–C(21)	1.512(8)
O(6)–C(28)	1.408(4)	C(9)–C(10)	1.531(4)	C(20)–C(22)	1.527(6)
O(7)–C(28)	1.403(6)	C(9)–C(11)	1.523(5)	C(22)–C(23)	1.533(6)
C(1)–C(2)	1.542(6)	C(9)–C(19)	1.525(5)	C(23)–C(24)	1.542(6)
C(1)–C(10)	1.521(6)	C(10)–C(19)	1.518(5)	C(24)–C(25)	1.531(7)
C(2)–C(3)	1.520(6)	C(11)–C(12)	1.551(6)	C(24)–C(28)	1.538(6)
C(3)–C(4)	1.557(6)	C(12)–C(13)	1.536(7)	C(25)–C(26)	1.632(9)
C(4)–C(5)	1.571(4)	C(13)–C(14)	1.578(6)	C(25)–C(27)	1.537(8)
C(4)–C(29)	1.542(6)	C(13)–C(17)	1.559(6)		

(*b*) Bond angles

C(30)–O(4)–C(31)	116.6(7)	C(10)–C(9)–C(11)	116.2(5)	C(13)–C(17)–C(20)	116.6(7)
C(22)–O(6)–C(28)	111.1(6)	C(10)–C(9)–C(19)	59.6(2)	C(16)–C(17)–C(20)	113.2(7)
O(1)–C(1)–C(2)	106.9(5)	C(11)–C(9)–C(19)	117.6(6)	C(9)–C(19)–C(10)	60.4(2)
O(1)–C(1)–C(10)	110.3(5)	C(1)–C(10)–C(5)	111.0(6)	C(17)–C(20)–C(21)	113.8(8)
C(2)–C(1)–C(10)	110.6(6)	C(1)–C(10)–C(9)	119.9(6)	C(17)–C(20)–C(22)	109.5(6)
C(1)–C(2)–C(3)	113.4(6)	C(1)–C(10)–C(19)	116.3(6)	C(21)–C(20)–C(22)	112.2(7)
O(2)–C(3)–C(2)	106.2(5)	C(5)–C(10)–C(9)	119.6(5)	O(6)–C(22)–C(20)	107.2(6)
O(2)–C(3)–C(4)	111.3(6)	C(5)–C(10)–C(19)	121.8(6)	O(6)–C(22)–C(23)	103.3(5)
C(2)–C(3)–C(4)	111.4(6)	C(9)–C(10)–C(19)	60.0(2)	C(20)–C(22)–C(23)	119.1(7)
C(3)–C(4)–C(5)	106.8(5)	C(9)–C(11)–C(12)	116.6(6)	C(22)–C(23)–C(24)	103.0(6)
C(3)–C(4)–C(29)	111.9(6)	C(11)–C(12)–C(13)	113.8(7)	O(5)–C(24)–C(23)	113.2(6)
C(3)–C(4)–C(30)	109.4(6)	C(12)–C(13)–C(14)	109.1(6)	O(5)–C(24)–C(25)	107.7(7)
C(5)–C(4)–C(29)	113.7(5)	C(12)–C(13)–C(17)	116.6(7)	O(5)–C(24)–C(28)	110.7(6)
C(5)–C(4)–C(30)	106.9(5)	C(12)–C(13)–C(18)	108.5(7)	C(23)–C(24)–C(25)	111.4(7)
C(29)–C(4)–C(30)	108.1(6)	C(14)–C(13)–C(17)	102.2(6)	C(23)–C(24)–C(28)	100.4(6)
C(4)–C(5)–C(6)	115.2(6)	C(14)–C(13)–C(18)	111.5(6)	C(25)–C(24)–C(28)	113.4(7)
C(4)–C(5)–C(10)	110.8(5)	C(17)–C(13)–C(18)	108.9(6)	C(24)–C(25)–C(26)	110.3(8)
C(6)–C(5)–C(10)	113.3(6)	C(8)–C(14)–C(13)	109.2(6)	C(24)–C(25)–C(27)	109.9(7)
C(5)–C(6)–C(7)	109.5(6)	C(8)–C(14)–C(15)	114.0(6)	C(26)–C(25)–C(27)	109.2(9)
C(6)–C(7)–C(8)	109.4(6)	C(8)–C(14)–C(32)	112.6(6)	O(6)–C(28)–O(7)	110.7(5)
C(7)–C(8)–C(9)	112.7(6)	C(13)–C(14)–C(15)	101.4(6)	O(6)–C(28)–C(24)	106.5(5)
C(7)–C(8)–C(14)	114.8(7)	C(13)–C(14)–C(32)	112.0(6)	O(7)–C(28)–C(24)	109.2(6)
C(9)–C(8)–C(14)	111.5(5)	C(15)–C(14)–C(32)	107.1(6)	O(3)–C(30)–O(4)	123.7(7)
C(8)–C(9)–C(10)	118.2(5)	C(14)–C(15)–C(16)	105.7(6)	O(3)–C(30)–C(4)	125.0(7)
C(8)–C(9)–C(11)	117.3(6)	C(15)–C(16)–C(17)	106.7(6)	O(4)–C(30)–C(4)	111.3(6)
C(8)–C(9)–C(19)	115.3(6)	C(13)–C(17)–C(16)	103.8(6)		

(*c*) Torsion angles defining the conformation of the rings. The angle A–B–C–D is defined as positive if, when viewed along the B–C bond, atom A has to be rotated clockwise to eclipse atom D. The standard deviation of the torsion angles is *ca.* 0.7°

C(10)–C(1)–C(2)–C(3)	−52.6	C(7)–C(8)–C(9)–C(10)	23.5	C(13)–C(14)–C(8)–C(9)	−46.2
C(1)–C(2)–C(3)–C(4)	55.4	C(7)–C(8)–C(9)–C(19)	91.1	C(13)–C(14)–C(15)–C(16)	−34.3
C(2)–C(3)–C(4)–C(5)	−57.4	C(7)–C(8)–C(9)–C(11)	−123.7	C(14)–C(15)–C(16)–C(17)	11.1
C(3)–C(4)–C(5)–C(6)	−169.3	C(8)–C(9)–C(10)–C(5)	−7.4	C(15)–C(16)–C(17)–C(20)	144.5
C(3)–C(4)–C(5)–C(10)	60.5	C(8)–C(9)–C(19)–C(10)	−109.2	C(15)–C(16)–C(17)–C(13)	17.1
C(4)–C(5)–C(10)–C(1)	−60.6	C(8)–C(9)–C(11)–C(12)	−40.3	C(16)–C(17)–C(13)–C(14)	38.2
C(4)–C(5)–C(10)–C(9)	153.1	C(9)–C(11)–C(12)–C(13)	15.9	C(16)–C(17)–C(13)–C(12)	157.1
C(4)–C(5)–C(10)–C(19)	82.0	C(11)–C(12)–C(13)–C(14)	37.0	C(16)–C(17)–C(13)–C(18)	−80.0
C(6)–C(5)–C(10)–C(19)	−49.2	C(11)–C(12)–C(13)–C(17)	152.1	O(6)–C(22)–C(23)–C(24)	31.4
C(6)–C(5)–C(10)–C(9)	21.9	C(11)–C(12)–C(13)–C(18)	−84.7	C(22)–C(23)–C(24)–C(28)	−38.9
C(10)–C(5)–C(6)–C(7)	−52.0	C(12)–C(13)–C(14)–C(8)	−70.5	C(23)–C(24)–C(28)–O(6)	33.1
C(5)–C(6)–C(7)–C(8)	69.6	C(12)–C(13)–C(14)–C(15)	168.9	C(24)–C(28)–O(6)–C(22)	14.2
C(6)–C(7)–C(8)–C(9)	−53.8	C(18)–C(13)–C(14)–C(32)	174.8	C(28)–O(6)–C(22)–C(23)	−11.0
C(6)–C(7)–C(8)–C(14)	177.0	C(13)–C(14)–C(8)–C(7)	−175.9		



Projection of the molecule on (100)

TABLE 3

(*a*) Distances (Å) of ring atoms from their least-squares planes

Plane (*A*): C(1)—(5), C(10)

C(1) 0.199, C(2) −0.221, C(3) 0.234, C(4) −0.263, C(5) 0.289, C(10) −0.238

Plane (*B*): C(5)—(10)

C(5) 0.086, C(6) −0.358, C(7) 0.391, C(8) −0.100, C(9) −0.013, C(10) −0.007

Plane (*C*): C(8), C(9), C(11)—(14)

C(8) −0.080, C(9) −0.259, C(11) 0.253, C(12) 0.008, C(13) −0.370, C(14) 0.449

Plane (*D*): C(13)—(17)

C(13) −0.291, C(14) 0.261, C(15) −0.136, C(16) −0.009, C(17) 0.176

Plane (*E*): O(6), C(22)—(24), C(28)

O(6) 0.005, C(22) 0.141, C(23) −0.233, C(24) 0.239, C(28) −0.152

Plane (*F*): C(9), C(10), C(19)

Interplanar angles (°): (*A*)–(*B*) 163.2, (*B*)–(*C*) 167.5, (*C*)–(*D*) 169.2, (*D*)–(*E*) 83.6, (*B*)–(*F*) 72.1

(*b*) Equations of least-squares of planes in the form $lX + mY + nZ = p$, where X, Y, Z are orthogonal co-ordinates in Å. The transformation matrix from crystallographic to orthogonal axes is:

$$\| 1\ 0\ \cos\beta \ /\ 0\ 1\ 0\ /\ 0\ 0\ \sin\beta \|$$

Plane	$10^4 l$	$10^4 m$	$10^4 n$	$10^4 p$
(*A*)	−4 348	6 892	−5 796	99 224
(*B*)	−6 501	6 551	−3 849	73 318
(*C*)	−5 062	6 651	−5 490	88 164
(*D*)	−6 563	6 027	−4 538	66 791
(*E*)	4 718	−2 972	−8 301	−1 129
(*F*)	7 390	−1 259	−6 618	31 687

cyclopropane ring. The ring fusions A–B and C–D are *trans*, while B–C is *cis*. The deviations of atoms from the best planes through the rings are reported in Table 3 and the torsion angles within each ring are given in Table 2. Ring A adopts a slightly deformed chair conformation. The conformation of ring B can be described as deformed skew-boat with the torsion angle around C(9)–C(10) constrained to be close to 0° (−7.4°) by the fusion with the cyclopropane ring. The deformation of ring C is even greater and its conformation can be best described as a severely distorted boat form. The five-membered rings E and D adopt a half-chair conformation.

The molecules are packed by a three-dimensional network of hydrogen bonds. The highly oxygenated ends of the molecules are joined by $O(1) \cdots O(7^{I})$ 2.703(4) Å $\{O(1) \cdots H[O(7)]–O(7)$ 131.3° $(I = 1 - x, \frac{1}{2} + y, -z)\}$ hydrogen bonds, which link the molecules around the screw axes. The helicoidal arrangements are held together by $O(1) \cdots O(3^{II})$ 2.729(3) Å $\{O(1)–H[O(1)] \cdots O(3)$ 149.0° $(II = x - 1, y, z)\}$ interactions. All other contacts are consistent with van der Waals interactions.

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