included in the refinement in a riding model with fixed isotropic *U*'s. Final full-matrix least-squares refinement (*SHELXTL-Plus*) converged to $R = 0\cdot046$, $wR = 0\cdot048$, $S = 2\cdot21$ for 235 parameters; $w = 1/[\sigma^2(F) + 0\cdot00018F^2]$, $\sum w(|F_o| - |F_c|)^2$ minimized; $(\Delta/\sigma)_{\max} = 0\cdot003$, $\Delta\rho_{\max,\,\min} = 0\cdot32$, $-0\cdot26$ e Å^{-3} in final difference Fourier map. Atomic scattering factors for C, H, O and N were those stored in *SHELXTL-Plus* which were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1,* bond lengths, angles and selected torsion angles are given in Table 2. The identification of the atoms and the configuration are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1.

Related literature. Recently we observed (Khanapure, Crenshaw, Reddy & Biehl, 1988) that the reactions of arynes generated from 2-bromoanisoles and lithium diisopropylamide with lithioarylnitriles led to the formation of polysubstituted benzenes *via* a tandem addition rearrangement involving an initial addition (Siriwardane, Khanapure, Chu & Biehl, 1988*b*), a formation of a benzocyclobutenimine intermediate (Siriwardane, Reddy, Chu & Biehl, 1988) and ring opening of the cyclobutenimine (Siriwardane, Khanapure, Chu & Biehl, 1988*a*). The diastereoselective formation (Crenshaw *et al.*, 1988) of (1) in the electrophilic quenching using benzaldehyde is consistent with Cram's rule (Cram & Wilson, 1963).

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* Anisotropic temperature factors, bond lengths, bond angles, torsion angles, H parameters and lists of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51827 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Bryonolic Acid Derivative, Bis(3*β*-acetoxy-*D*:*C*-friedoolean-8-en-29-oic) Anhydride

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Abstract. $C_{64}H_{98}O_7$, $M_r = 979\cdot47$, monoclinic, $C2$, $a = 13\cdot574\,(1)$, $b = 6\cdot403\,(1)$, $c = 32\cdot504\,(2)$ Å, $\beta = 98\cdot57\,(1)°$, $V = 2793\cdot4\,(5)$ Å^3, $Z = 2$, $D_x = 1\cdot164$ Mg m^{-3}, $\lambda(\text{Cu }K\alpha) = 1\cdot54178$ Å, $\mu(\text{Cu }K\alpha) = 0\cdot58$ mm^{-1}, $F(000) = 1076$, $T = 295$ K, $R = 0\cdot062$ for 2268 observed reflections $[F_o > 3\sigma(F_o)]$. The molecule lies on a twofold rotational axis. The ring junctions *A*/*B*, *C*/*D* and *D*/*E* are *trans*, *trans* and *cis*, respectively. The conformations of rings *A*–*E* are chair, half-chair, half-chair, boat and boat, respectively.

Experimental. Colorless plates obtained from methanol. Crystal of dimensions 0·25 × 0·15 × 0·05 mm. Rigaku AFC-5 diffractometer, graphite-monochromatized Cu *Kα* radiation. Cell dimensions determined from 2θ angles for 25 reflections in the range $30 < 2\theta < 45°$. Intensities measured up to $2\theta = 140°$ in h −16/16, k 0/7 and l 0/39, ω–2θ scans, ω-scan width $(1\cdot0 + 0\cdot2\tan\theta)°$. 2834 unique reflections measured, 2268 intensities observed $[F_o \leq 3\sigma(F_o)$ and two very strong reflections rejected], no absorption corrections. Three standard reflections moni-

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tored every 100 measurements showed no significant change.

Structures solved by *MULTAN*84 (Main, Germain & Woolfson, 1984). H atoms located on a difference density map. Positional and thermal parameters refined by block-diagonal least squares, isotropic for H and anisotropic for other atoms. $\sum(w|\Delta F|^2)$ minimized, $w = 1/[\sigma^2(F_o) + 0·0018|F_o|^2]$, $w = 0$ for 47 reflections with $w^{1/2}|\Delta F| > 3$. Final $R = 0·062$, $wR = 0·068$, $S = 1·1110$. Highest and lowest peaks in final difference map 0·1 and $-0·1$ e Å^{-3}. Max. Δ/σ in the

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
C(1)	0·6273 (3)	0·3954	0·8305 (1)	4·3 (1)
C(2)	0·6621 (3)	0·4243 (8)	0·8776 (1)	4·7 (1)
C(3)	0·5851 (3)	0·5510 (7)	0·8957 (1)	4·3 (1)
C(4)	0·4808 (3)	0·4565 (7)	0·8903 (1)	4·2 (1)
C(5)	0·4506 (3)	0·4089 (7)	0·8431 (1)	3·8 (1)
C(6)	0·3476 (3)	0·3165 (8)	0·8316 (1)	4·8 (1)
C(7)	0·3132 (3)	0·3470 (9)	0·7857 (1)	5·0 (1)
C(8)	0·3910 (3)	0·2937 (7)	0·7585 (1)	3·8 (1)
C(9)	0·4876 (3)	0·2749 (7)	0·7744 (1)	3·6 (1)
C(10)	0·5267 (3)	0·2848 (7)	0·8210 (1)	3·8 (1)
C(11)	0·5653 (3)	0·2281 (9)	0·7471 (1)	4·5 (1)
C(12)	0·5253 (3)	0·1560 (8)	0·7025 (1)	4·0 (1)
C(13)	0·4308 (3)	0·2751 (7)	0·6836 (1)	3·6 (1)
C(14)	0·3504 (2)	0·2391 (7)	0·7125 (1)	3·8 (1)
C(15)	0·2567 (3)	0·3687 (9)	0·6959 (1)	4·9 (1)
C(16)	0·2393 (3)	0·4146 (9)	0·6493 (1)	5·3 (1)
C(17)	0·2810 (3)	0·2547 (8)	0·6208 (1)	4·3 (1)
C(18)	0·3918 (3)	0·1897 (6)	0·6394 (1)	3·6 (1)
C(19)	0·4632 (3)	0·2416 (7)	0·6086 (1)	4·1 (1)
C(20)	0·4323 (3)	0·1549 (7)	0·5643 (1)	4·1 (1)
C(21)	0·3238 (3)	0·2194 (10)	0·5472 (1)	5·6 (1)
C(22)	0·2799 (3)	0·3560 (9)	0·5783 (1)	5·2 (1)
C(23)	0·4746 (4)	0·2693 (9)	0·9191 (1)	5·5 (1)
C(24)	0·4085 (4)	0·6253 (9)	0·9020 (1)	5·5 (1)
C(25)	0·5404 (3)	0·0548 (7)	0·8355 (1)	4·8 (1)
C(26)	0·3238 (3)	0·0043 (7)	0·7159 (1)	5·0 (1)
C(27)	0·4583 (3)	0·5090 (7)	0·6832 (1)	4·4 (1)
C(28)	0·2127 (3)	0·0659 (10)	0·6155 (2)	6·2 (2)
C(29)	0·4955 (3)	0·2605 (9)	0·5364 (1)	5·0 (1)
C(30)	0·4470 (4)	−0·0810 (9)	0·5630 (1)	5·7 (1)
C(31)	0·6647 (3)	0·7587 (9)	0·9535 (1)	5·1 (1)
C(32)	0·6825 (5)	0·7727 (12)	0·9998 (2)	7·9 (2)
O(33)	0·6168 (2)	0·5827 (5)	0·9405 (1)	4·9 (1)
O(34)	0·6903 (3)	0·8847 (7)	0·9302 (1)	7·2 (1)
O(35)	0·5394 (3)	0·4202 (9)	0·5420 (1)	9·1 (2)
O(36)	0·5000	0·1524 (8)	0·5000	5·7 (1)

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·545 (7)	C(13)—C(18)	1·555 (6)
C(1)—C(10)	1·528 (6)	C(13)—C(27)	1·544 (6)
C(2)—C(3)	1·510 (7)	C(14)—C(15)	1·547 (7)
C(3)—C(4)	1·526 (6)	C(14)—C(26)	1·554 (6)
C(3)—O(33)	1·470 (6)	C(15)—C(16)	1·526 (8)
C(4)—C(5)	1·558 (6)	C(16)—C(17)	1·544 (8)
C(4)—C(23)	1·531 (7)	C(17)—C(18)	1·591 (7)
C(4)—C(24)	1·545 (7)	C(17)—C(22)	1·524 (8)
C(5)—C(6)	1·513 (7)	C(17)—C(28)	1·518 (8)
C(5)—C(10)	1·561 (6)	C(18)—C(19)	1·530 (6)
C(6)—C(7)	1·508 (8)	C(19)—C(20)	1·542 (6)
C(7)—C(8)	1·514 (7)	C(20)—C(21)	1·551 (8)
C(8)—C(9)	1·341 (6)	C(20)—C(29)	1·499 (7)
C(8)—C(14)	1·554 (6)	C(20)—C(30)	1·525 (7)
C(9)—C(10)	1·530 (6)	C(21)—C(22)	1·524 (9)
C(9)—C(11)	1·506 (7)	C(29)—O(35)	1·184 (8)
C(10)—C(25)	1·549 (6)	C(29)—O(36)	1·380 (8)
C(11)—C(12)	1·541 (8)	C(31)—C(32)	1·491 (10)
C(12)—C(13)	1·539 (7)	C(31)—O(33)	1·338 (7)
C(13)—C(14)	1·560 (6)	C(31)—O(34)	1·193 (7)

C(2)—C(1)—C(10)	112·8 (4)	C(18)—C(13)—C(27)	112·5 (4)
C(1)—C(2)—C(3)	108·9 (4)	C(8)—C(14)—C(13)	111·5 (4)
C(2)—C(3)—C(4)	115·2 (4)	C(8)—C(14)—C(15)	111·7 (4)
C(2)—C(3)—O(33)	110·0 (4)	C(8)—C(14)—C(26)	101·7 (3)
C(4)—C(3)—O(33)	107·4 (3)	C(13)—C(14)—C(15)	108·9 (4)
C(3)—C(4)—C(5)	107·1 (4)	C(13)—C(14)—C(26)	112·1 (4)
C(3)—C(4)—C(23)	112·0 (4)	C(15)—C(14)—C(26)	110·9 (4)
C(3)—C(4)—C(24)	107·9 (4)	C(14)—C(15)—C(16)	116·6 (5)
C(5)—C(4)—C(23)	114·7 (4)	C(15)—C(16)—C(17)	116·8 (5)
C(5)—C(4)—C(24)	107·2 (4)	C(16)—C(17)—C(18)	110·9 (4)
C(23)—C(4)—C(24)	107·6 (4)	C(16)—C(17)—C(22)	107·9 (4)
C(4)—C(5)—C(6)	114·7 (4)	C(16)—C(17)—C(28)	108·9 (5)
C(4)—C(5)—C(10)	117·2 (4)	C(18)—C(17)—C(22)	109·7 (4)
C(6)—C(5)—C(10)	109·7 (4)	C(18)—C(17)—C(28)	111·2 (4)
C(5)—C(6)—C(7)	109·4 (4)	C(22)—C(17)—C(28)	108·2 (5)
C(6)—C(7)—C(8)	114·1 (4)	C(13)—C(18)—C(17)	115·6 (4)
C(7)—C(8)—C(9)	121·5 (4)	C(13)—C(18)—C(19)	111·5 (3)
C(7)—C(8)—C(14)	115·7 (4)	C(17)—C(18)—C(19)	110·5 (4)
C(9)—C(8)—C(14)	122·5 (4)	C(18)—C(19)—C(20)	115·0 (4)
C(8)—C(9)—C(10)	123·6 (4)	C(19)—C(20)—C(21)	110·5 (4)
C(8)—C(9)—C(11)	121·4 (4)	C(19)—C(20)—C(29)	107·6 (4)
C(10)—C(9)—C(11)	114·9 (4)	C(19)—C(20)—C(30)	111·3 (4)
C(1)—C(10)—C(5)	107·7 (3)	C(21)—C(20)—C(29)	105·2 (4)
C(1)—C(10)—C(9)	112·8 (4)	C(21)—C(20)—C(30)	112·0 (4)
C(1)—C(10)—C(25)	108·4 (4)	C(29)—C(20)—C(30)	109·9 (4)
C(5)—C(10)—C(9)	108·7 (4)	C(20)—C(21)—C(22)	111·1 (5)
C(5)—C(10)—C(25)	113·7 (4)	C(17)—C(22)—C(21)	113·9 (5)
C(9)—C(10)—C(25)	105·7 (4)	C(20)—C(29)—O(35)	128·1 (6)
C(9)—C(11)—C(12)	115·7 (4)	C(20)—C(29)—O(36)	113·3 (5)
C(11)—C(12)—C(13)	112·5 (4)	O(35)—C(29)—O(36)	118.7 (6)
C(12)—C(13)—C(14)	107·6 (4)	C(32)—C(31)—O(33)	111·6 (5)
C(12)—C(13)—C(18)	110·1 (4)	C(32)—C(31)—O(34)	125·5 (6)
C(12)—C(13)—C(27)	107·2 (4)	O(33)—C(31)—O(34)	122·9 (5)
C(14)—C(13)—C(18)	109·4 (3)	C(3)—O(33)—C(31)	119·1 (4)
C(14)—C(13)—C(27)	109·9 (4)	C(29)—O(36)—C(29′)	119·8 (5)

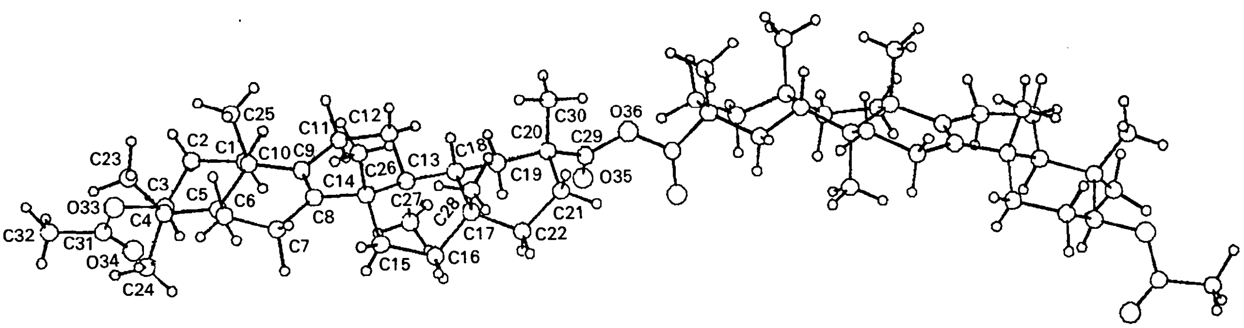

Fig. 1. Perspective view with the atom-numbering system.

final cycle 0·2. Atomic scattering factors calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)]+c$ $(i = 1,\ldots,4)$ (*International Tables for X-ray Crystallography*, 1974). Calculations performed on FACOM M-730 computer at Shionogi Research Laboratories. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1. Bond distances and angles are listed in Table 2.* A perspective view of the molecule with the atom-numbering system and a stereoview of the crystal packing drawn using the program *PLUTO* (Motherwell & Clegg, 1978) are presented in Figs. 1 and 2, respectively.

Related literature. The structure of the title compound reported here has been discussed in Kamisako, Suwa, Honda, Isoi, Nakai, Shiro & Machida (1987).

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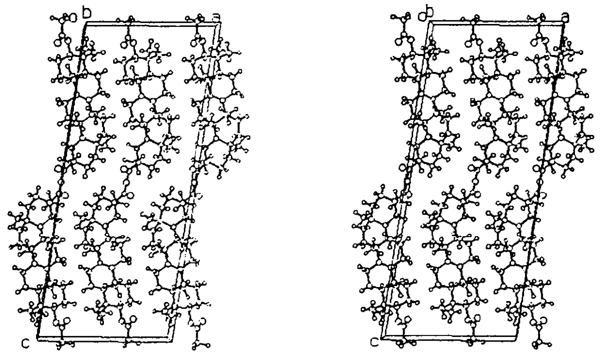

Fig. 2. A stereoview of the unit-cell packing.

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* Lists of structure factors, anisotropic temperature factors of the non-H atoms and atomic coordinates of the H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51858 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 2,3-Dihydro-6-(3-nitrobenzylidene)-imidazo[2,1-*b*]thiazol-5(6*H*)-one

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Abstract. $C_{12}H_9N_2O_3S$, $M_r = 261{\cdot}29$, monoclinic, $P2_1/n$, $a = 5{\cdot}3014\,(4)$, $b = 20{\cdot}8420\,(2)$, $c = 10{\cdot}8287\,(3)$ Å, $\beta = 98{\cdot}08\,(8)^\circ$, $V = 1184{\cdot}6\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}47$ g cm^{-3}, $\lambda(\mathrm{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}27$ mm^{-1}, $F(000) = 540$, final $R = 0{\cdot}068$ for 1129 observed reflections $[I > 2\sigma(I)]$. The two fused five-membered rings forming the main part of the molecule are coplanar. The benzene ring is at an angle of 8·1 (7)° to this plane.

Experimental. The title compound (I) crystallized from glacial acetic acid in the form of a very thin yellow needle, monoclinic form. Crystal dimensions 0·2 × 0·15 × 0·4 mm. Preliminary data obtained from oscillation and Weissenberg photographs (Cu $K\alpha$ radiation); CAD-4 diffractometer. Final lattice parameters from least-squares refinement of 25 reflections $(4 < \theta < 12^\circ)$; no absorption correction applied; $\theta < 25^\circ$ (h −6/6, k 0/24, l 0/9); ω–2θ scan technique; Mo $K\alpha$ radiation at room temperature; intensity of three ($\bar{2}7\bar{3}$, $7\bar{1}2$, $6\bar{1}5$) standard reflections

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