along with the **U** tensors for the B and O atoms, were used as observations to *THMV*10. Full details of the thermal-motion descriptions for both TPHBRX and TPHBEN have been deposited.*

In both structures the **TLS** model fits the observed 'thermal' parameters adequately but not well. The quality of the structure determinations may be a problem. Non-rigidity of the B_3O_3 ring may be a complicating factor, as may other types of internal motion, such as bending at the B (or C) atom of the central ring or at the attached C atom. Several conclusions, however, can be drawn. First, the eigenvalues of the **T** tensor for TPHBRX are about 30% larger than they are for TPHBEN. Second, the torsional amplitudes about the B—C bonds in TPHBRX [35 (13), 22 (12), 40 (13)$^{\circ 2}$] are somewhat greater than the corresponding amplitudes for the C—C(Ph) bonds in TPHBEN [16 (10), 20 (11), 11 (10)$^{\circ 2}$], although the difference is only at the borderline of significance. It is interesting that the estimates of the torsional amplitudes derived for TPHBRX from the crystallographic model were considerably larger [73 (5), 64 (4), 86 (5)$^{\circ 2}$]. Large-amplitude motions result from wide, shallow potential-energy wells, which often have small central maxima. If there is a small maximum of this type, then as the temperature is lowered, it might become larger than kT. A phase transition could then be expected to occur. It seems likely (see *Experimental*) that such a transition takes place in TPHBRX crystals. We suggest that it is associated with torsional displacements about the B—C bonds.

Acknowledgement is made by CPB and RPM to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this research. KN thanks the office of Naval Research.

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* See previous footnote.

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Structure of a Bryonolic Acid Derivative, 3β-Hydroxy-*D:C*-friedoolean-8-en-29-oic Acid Methyl Ester

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(*Received* 23 *January* 1987; *accepted* 19 *March* 1987)

Abstract. $C_{31}H_{50}O_3$, $M_r = 470{\cdot}7$, trigonal, $P3_121$, $a = 18{\cdot}571$ (1), $c = 14{\cdot}018$ (2) Å, $V = 4187{\cdot}0$ (6) Å^3, $Z = 6$, $D_x = 1{\cdot}120$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ(Cu $K\alpha$) = 0·54 mm^{-1}, $F(000) = 1560$, $T = 295$ K, $R = 0{\cdot}044$ for 2639 reflections. The compound was derived from bryonolic acid isolated from *Luffa cylindrica* roots (Cucurbitaceae). Rings *A*, *B* and *C* are in the chair, half-chair and half-chair conformations, respectively. The *D*–*E* ring adopts a chair–chair form which is considerably distorted to relieve the repulsive

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interaction between Me(27) and C(29)OOMe, in contrast with that of the derivative substituted by $C(29)H_2OAc$, which takes the boat–boat conformation.

Introduction. ^{13}C NMR spectral study of the derivatives of bryonolic acid (3β-hydroxy-*D:C*-friedo-olean-8-en-29-oic acid) revealed that there are remarkable differences in the chemical shifts of the *D–E* ring carbons between the title compound (1) and *D:C*-friedoolean-8-en-3β,29-diol diacetate (2) (Kamisako, Suwa, Morimoto & Isoi, 1984). This appeared to be caused by the conformational change in the *D–E* ring moieties with different substituents at the 20α positions. It has been proposed that friedelin-type triterpenes possess rings of either chair–chair or boat–boat form but not a mixed form, *e.g.* chair–boat (Masaki, Niwa & Kikuchi, 1975). As the *D–E* ring of (2) was found to adopt the boat–boat form by X-ray study (Kamisako, Isoi, Nakai & Shiro, 1984), that of (1) was supposed to be chair–chair. Here we describe the structure determination of (1) which was undertaken to obtain detailed conformations to interpret the spectral data on the basis of the conformational dependency of the ^{13}C chemical shifts (Dalling & Grant, 1972).

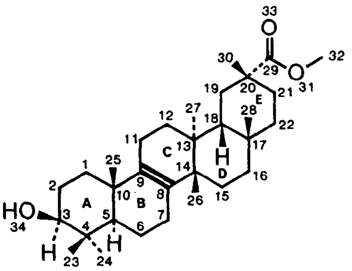

Table 1. *Atomic coordinates* (×10⁴) *and equivalent isotropic temperature factors* (Å² ×10²) *with e.s.d.'s in parentheses*

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

	x	*y*	*z*	$B_{\rm eq}$
C(1)	3206 (1)	5132 (1)	8382 (2)	423 (6)
C(2)	3133 (1)	5900 (1)	8594 (2)	468 (7)
C(3)	2527 (2)	5964 (1)	7920 (2)	457 (8)
C(4)	1652 (1)	5187 (1)	7932 (1)	411 (7)
C(5)	1758 (1)	4414 (1)	7786 (1)	326 (6)
C(6)	948 (1)	3585 (1)	7722 (2)	387 (6)
C(7)	1103 (1)	2934 (1)	7260 (1)	376 (6)
C(8)	1870 (1)	2937 (1)	7629 (1)	298 (5)
C(9)	2442 (1)	3568 (1)	8156 (1)	318 (5)
C(10)	2362 (1)	4321 (1)	8465 (1)	315 (5)
C(11)	3220 (1)	3575 (1)	8510 (1)	432 (7)
C(12)	3219 (1)	2759 (1)	8396 (1)	394 (7)
C(13)	2834 (1)	2332 (1)	7439 (1)	289 (5)
C(14)	1912 (1)	2147 (1)	7441 (1)	295 (5)
C(15)	1531 (1)	1741 (1)	6466 (1)	350 (6)
C(16)	1496 (1)	905 (1)	6349 (1)	382 (6)
C(17)	2302 (1)	880 (1)	6557 (1)	326 (5)
C(18)	2844 (1)	1499 (1)	7361 (1)	310 (5)
C(19)	3715 (1)	1569 (1)	7412 (1)	372 (6)
C(20)	4191 (1)	1621 (1)	6480 (2)	412 (7)
C(21)	3598 (1)	1026 (1)	5729 (2)	429 (7)
C(22)	2798 (1)	1050 (1)	5619 (1)	369 (6)
C(23)	1167 (2)	5154 (1)	8838 (2)	542 (9)
C(24)	1178 (2)	5262 (2)	7075 (2)	708 (13)
C(25)	2083 (2)	4171 (1)	9522 (1)	444 (8)
C(26)	1373 (1)	1541 (1)	8243 (1)	425 (7)
C(27)	3325 (1)	2953 (1)	6638 (1)	369 (6)
C(28)	2031 (1)	−11 (1)	6878 (2)	442 (7)
C(29)	4707 (1)	2513 (1)	6133 (2)	457 (7)
C(30)	4845 (2)	1365 (2)	6727 (2)	597 (11)
O(31)	4591 (1)	2617 (1)	5225 (1)	562 (6)
C(32)	5085 (2)	3451 (2)	4857 (3)	775 (13)
O(33)	5203 (1)	3075 (1)	6617 (2)	631 (7)
O(34)	2513 (1)	6718 (1)	8076 (1)	620 (7)

Experimental. Prismatic colorless crystals grown from ether solution. Crystal of dimensions 0·5 × 0·7 × 0·7 mm used, Rigaku AFC-5UD diffractometer, graphite-monochromatized Cu *K*α. Cell dimensions from 2θ angles for 20 reflections ($37 < 2\theta < 40°$). Intensities measured up to $2\theta = 140°$ in *h* −18/0, *k* −18/0 and *l* 0/17. Three standard reflections monitored every 100 measurements (±1% variation). 2769 unique reflections measured, 2699 intensities observed [$|F_o| > \sigma(F_o)$], no absorption corrections. Structure solved by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). H atoms located on a difference density map. Positional parameters of all atoms and anisotropic thermal parameters of non-H atoms refined by block-diagonal least squares. Temperature factor of each H atom equal to $B_{\rm eq}$ of the bonded atom. $R = 0{\cdot}044$, $wR = 0{\cdot}053$, $S = 1{\cdot}09$ for 2639 observed reflections ($w \neq 0$). No significant peaks in final difference map, highest peak 0·4 e Å⁻³. $\sum(w|\Delta F|^2)$ minimized, $w = [\sigma^2(F_o) + 0{\cdot}00194|F_o|^2]^{-1}$ for $w^{1/2}|F_c| \geq 1$ and $w^{1/2}|\Delta F| < 3$, $w = 0$ otherwise. Ratio of final shift to e.s.d. <1·0. Atomic scattering factors calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ ($i =$ 1–4) (*International Tables for X-ray Crystallography*, 1974). Calculations performed by FACOM M 340R computer at Shionogi Research Laboratories.

Discussion. Atomic coordinates and equivalent isotropic temperature factors of non-H atoms are listed in Table 1. Bond lengths and angles are given in Table 2.[*] A projection of the crystal structure drawn using the program *PLUTO* (Motherwell & Clegg, 1978) is presented in Fig. 1. The absolute configuration of the molecule was determined on the basis of the α-configuration of the C(27) methyl group. An intermolecular hydrogen bond of 2·931 Å is formed between O(33) and O(34).

The ring junctions *A/B*, *C/D* and *D/E* are respectively *trans*, *trans* and *cis*. The rings of *A*, *B* and *C* individually adopt the chair, half-chair and half-chair conformations like those in (2). The *D–E* ring is in the chair–chair form in contrast with that of (2) which takes the boat–boat one, being considerably distorted to

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[*] Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43903 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

C(1)–C(2)	1·528 (4)	C(13)–C(14)	1·569 (3)
C(1)–C(10)	1·542 (3)	C(13)–C(18)	1·560 (3)
C(2)–C(3)	1·519 (5)	C(13)–C(27)	1·540 (3)
C(3)–C(4)	1·542 (4)	C(14)–C(15)	1·550 (3)
C(3)–O(34)	1·430 (4)	C(14)–C(26)	1·551 (3)
C(4)–C(5)	1·557 (3)	C(15)–C(16)	1·530 (3)
C(4)–C(23)	1·540 (4)	C(16)–C(17)	1·548 (3)
C(4)–C(24)	1·536 (4)	C(17)–C(18)	1·565 (3)
C(5)–C(6)	1·525 (3)	C(17)–C(22)	1·545 (3)
C(5)–C(10)	1·545 (3)	C(17)–C(28)	1·537 (3)
C(6)–C(7)	1·520 (3)	C(18)–C(19)	1·558 (3)
C(7)–C(8)	1·513 (3)	C(19)–C(20)	1·553 (3)
C(8)–C(9)	1·343 (3)	C(20)–C(21)	1·525 (4)
C(8)–C(14)	1·530 (3)	C(20)–C(29)	1·520 (4)
C(9)–C(10)	1·540 (3)	C(20)–C(30)	1·549 (5)
C(9)–C(11)	1·522 (3)	C(21)–C(22)	1·516 (3)
C(10)–C(25)	1·548 (4)	C(29)–O(31)	1·321 (3)
C(11)–C(12)	1·523 (3)	C(29)–O(33)	1·199 (4)
C(12)–C(13)	1·540 (3)	O(31)–C(32)	1·444 (5)
C(2)–C(1)–C(10)	112·1 (2)	C(14)–C(13)–C(18)	109·6 (1)
C(1)–C(2)–C(3)	111·5 (2)	C(14)–C(13)–C(27)	109·8 (1)
C(2)–C(3)–C(4)	113·3 (3)	C(18)–C(13)–C(27)	113·3 (2)
C(2)–C(3)–O(34)	111·0 (3)	C(8)–C(14)–C(13)	111·3 (1)
C(4)–C(3)–O(34)	112·8 (3)	C(8)–C(14)–C(15)	112·9 (2)
C(3)–C(4)–C(5)	107·5 (2)	C(8)–C(14)–C(26)	104·3 (1)
C(3)–C(4)–C(23)	111·6 (2)	C(13)–C(14)–C(15)	106·7 (1)
C(3)–C(4)–C(24)	106·9 (2)	C(13)–C(14)–C(26)	113·4 (1)
C(5)–C(4)–C(23)	114·4 (2)	C(15)–C(14)–C(26)	108·3 (1)
C(5)–C(4)–C(24)	108·9 (2)	C(14)–C(15)–C(16)	111·4 (2)
C(23)–C(4)–C(24)	107·3 (2)	C(15)–C(16)–C(17)	117·3 (2)
C(4)–C(5)–C(6)	115·0 (2)	C(16)–C(17)–C(18)	113·0 (1)
C(4)–C(5)–C(10)	117·1 (2)	C(16)–C(17)–C(22)	108·7 (1)
C(6)–C(5)–C(10)	109·2 (2)	C(16)–C(17)–C(28)	106·6 (2)
C(5)–C(6)–C(7)	110·1 (2)	C(18)–C(17)–C(22)	111·4 (1)
C(6)–C(7)–C(8)	113·8 (2)	C(18)–C(17)–C(28)	108·9 (2)
C(7)–C(8)–C(9)	121·6 (2)	C(22)–C(17)–C(28)	108·1 (2)
C(7)–C(8)–C(14)	115·9 (2)	C(13)–C(18)–C(17)	117·2 (2)
C(9)–C(8)–C(14)	122·3 (2)	C(13)–C(18)–C(19)	116·1 (2)
C(8)–C(9)–C(10)	123·6 (2)	C(17)–C(18)–C(19)	110·7 (1)
C(8)–C(9)–C(11)	121·4 (2)	C(18)–C(19)–C(20)	120·1 (2)
C(10)–C(9)–C(11)	115·0 (2)	C(19)–C(20)–C(21)	110·7 (2)
C(1)–C(10)–C(5)	106·5 (2)	C(19)–C(20)–C(29)	111·5 (2)
C(1)–C(10)–C(9)	110·7 (2)	C(19)–C(20)–C(30)	107·6 (2)
C(1)–C(10)–C(25)	109·5 (2)	C(21)–C(20)–C(29)	113·7 (2)
C(5)–C(10)–C(9)	109·5 (1)	C(21)–C(20)–C(30)	109·0 (2)
C(29)–C(20)–C(30)	104·0 (2)	C(5)–C(10)–C(25)	114·4 (2)
C(20)–C(21)–C(22)	114·3 (2)	C(9)–C(10)–C(25)	106·3 (2)
C(17)–C(22)–C(21)	113·6 (2)	C(9)–C(11)–C(12)	115·4 (2)
C(20)–C(29)–O(31)	114·0 (2)	C(11)–C(12)–C(13)	111·9 (2)
C(20)–C(29)–O(33)	124·0 (3)	C(12)–C(13)–C(14)	106·6 (1)
O(31)–C(29)–O(33)	121·9 (3)	C(12)–C(13)–C(18)	109·8 (1)
C(29)–O(31)–C(32)	116·0 (2)	C(12)–C(13)–C(27)	107·5 (1)

relieve the repulsive interaction between the C(27) and C(29) substituents that occupy the axial positions of the *D–E* ring (Fig. 2). The torsion angle at the ring junction is 89·5 (2)° for C(22)–C(17)–C(18)–C(13) and −169·5 (1)° for C(16)–C(17)–C(18)–C(19). The plane of the C(29) methoxycarbonyl group approximately faces the C(27) methyl group, the contacts between the groups being 2·57 (1) Å for O(33)···H(C27), 2·61 (1) Å for C(29)···H(C27) and 2·90 (1) Å for O(31)···H′(C27). If the C(29) substituent were replaced by an acetoxymethyl group, which is the same as that of (2), the longest contact with the C(27) methyl group would be *ca* 2·1 Å between H(C27) and H(C29) for various relative arrangements resulting from the rotation of the C(29) group about the C(20)–C(29) bond. Because of such a short approach, the chair–chair form which is deformed as much as shown in Fig. 2 is unfavorable for the *D–E* ring. Thus, the ring of (2) seems to be stabilized in a more distorted chair–chair form or a stretched boat–boat form: in the latter form, the C(29) substituent occupying the pseudoequatorial position lies at a large distance from the C(27) methyl group. If only the interactions among ring substituents are considered, it is not obvious which of the two forms is more stable.

For maytenonic acid methyl ester (3) (Ramaiah, Devi, Frolow & Lavie, 1984) and epifriedelinol (4) (Laing, Burke-Laing, Bartho & Weeks, 1977), which are friedelane derivatives having *cis*-fused *D–E* rings similar to those of bryonolic acid derivatives, the conformations of the rings are chair–chair and boat–boat, respectively. The C(29) substituent is the methoxycarbonyl group for (3) and the methyl group for (4). Thus, the rings substituted at their 20α positions by sp^2-hybridized carbon atoms, in the cases of (1) and (3), adopt chair–chair forms and those substituted by sp^3-hybridized ones, (2) and (4), adopt boat–boat forms. As this holds also for other friedelin-type

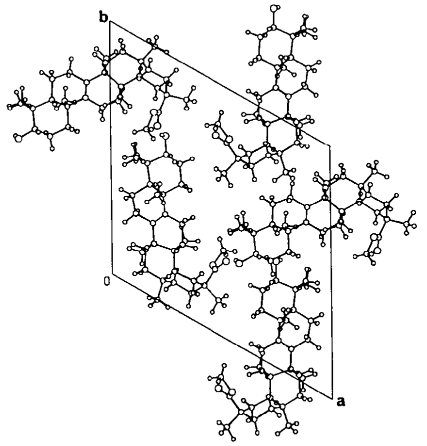

Fig. 1. [001] projection of the crystal structure bound between $z = \frac{1}{2}$ and 1.

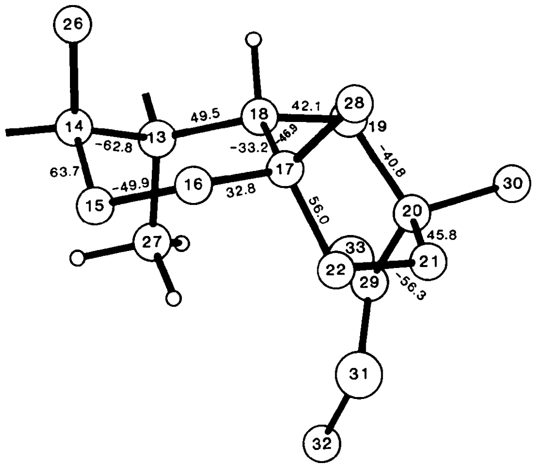

Fig. 2. Conformation of the *D–E* ring with the endocyclic torsion angles (°).

triterpenes lying in different crystal fields, we supposed that the conformation of the ring exhibited in the crystalline state might correspond to the more stable of the two forms. It was revealed that the chair–chair form exists predominantly in a solution of (1), and the two forms coexist in equilibrium in a solution of (2) (Kamisako, Suwa, Honda, Isoi, Nakai, Shiro & Machida, 1987). This suggests that the difference in the stabilities of the two forms is large in the case of (1) and small in the case of (2).

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Acta Cryst. (1987). C**43**, 1782–1784

Fluorene-9-spiro-1′-cyanocyclopropane*

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(*Received* 16 *February* 1987; *accepted* 21 *April* 1987)

Abstract. $C_{16}H_{11}N$, $M_r = 217{\cdot}27$, orthorhombic, *Pbcn*, $a = 17{\cdot}831\ (17)$, $b = 11{\cdot}246\ (3)$, $c = 11{\cdot}359\ (4)$ Å, $V = 2278\ (2)$ Å^3, $Z = 8$, $D_x = 1{\cdot}27$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}80$ cm^{-1}, $F(000) = 912$, $T = 291$ K, $R = 0{\cdot}045$ for 859 observed reflections. The cyclopropane bond lengths agree with those predicted following additivity of the bond-length asymmetries of the cyano and biphenyl substituents.

Introduction. The cyclopropane ring undergoes drastic geometrical changes under the influence of electron-withdrawing and -donating substituents. These changes have been discussed in detail by Allen (1980). We have already analysed the ring bond-length pattern and the orientation of the aromatic groups in a series of *trans* and *cis* 1-cyano-1,2-diphenylcyclopropanes (Tinant, Deblock, Declercq, Germain, Van Meerssche, Leroy & Weiler, 1982; Tinant, Wu, Declercq, Van Meerssche, Leroy & Weiler, 1987). Continuing our studies in this field, we report here the structure of the title compound. The unique feature of this compound is the rigidly maintained bisected conformation of the π system, in this case a fluorenyl group.

Experimental. 5 g of 9-diazafluorene, prepared in the usual way from commercially available fluorenone, *via* fluorenone hydrazone, were dissolved in cold benzene and 3 g of acrylonitrile were added. Escape of N_2 was observed during the reaction. After 24 h the solvent was removed. Single crystals in the form of colourless needles were grown by slow evaporation of a petroleum-ether solution of the crude precipitate. Approximate dimensions of the crystal chosen for X-ray measurements $0{\cdot}35 \times 0{\cdot}2 \times 0{\cdot}15$ mm. D_m not measured. Lattice parameters refined using 15 reflections in the range $5 \le 2\theta \le 25°$. Syntex $P2_1$, graphite-monochromatized Mo $K\alpha$ radiation. 1682 *hkl* independent reflections with $(\sin\theta)/\lambda \le 0{\cdot}561$ Å^{-1}; $0 \le h \le 20$, $0 \le k \le 12$, $0 \le l \le 12$; 859 with $I \ge 2{\cdot}5\sigma(I)$. Standard reflection (123) checked every 50 reflections: no

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* Spiro[cyclopropane-2,9′-fluorene]-1-carbonitrile.

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