Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic vibrational parameters* ($\text{Å}^2 \times 10^4$) *for non-*H *atoms*

	x	y	z	U_{eq}*
C(1)	1198 (3)	1394 (8)	2736 (2)	749
N(2)	621 (3)	2341 (7)	2202 (2)	854
C(3)	793 (3)	1170 (9)	1775 (2)	802
C(4)	1513 (3)	−955 (9)	1861 (2)	741
C(4A)	2129 (3)	−1857 (7)	2435 (2)	643
N(5)	2813 (2)	−4221 (6)	2499 (1)	712
N(6)	3773 (2)	−4393 (6)	2907 (1)	665
C(6A)	4303 (3)	−2298 (6)	3339 (1)	535
C(7)	5403 (3)	−1854 (7)	3469 (1)	534
C(8)	5975 (3)	107 (7)	3896 (1)	559
C(9)	5510 (3)	1472 (7)	4221 (1)	556
C(10)	4439 (3)	888 (8)	4105 (2)	593
C(10A)	3829 (3)	−975 (7)	3667 (1)	564
C(11)	2644 (3)	−1618 (9)	3525 (2)	709
C(11A)	1973 (3)	−688 (7)	2899 (2)	628
C(12)	5949 (3)	−3395 (9)	3148 (2)	674
C(13)	6104 (3)	3517 (9)	4696 (2)	660
F(14)	1016 (2)	2627 (5)	3166 (1)	1000
F(15)	206 (2)	2126 (6)	1224 (1)	1244
F(16)	1616 (2)	−2078 (5)	1396 (1)	1010
O(17)	7131 (2)	4220 (5)	4688 (1)	705
C(18)	7700 (3)	6250 (9)	5059 (2)	678
O(19)	7414 (2)	7365 (6)	5390 (1)	1057
C(20)	8721 (5)	6942 (19)	4995 (3)	1129

$* \; U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}\mathbf{a}_i.\mathbf{a}_j a_i^* a_j^*.$

Table 2. *Selected bond lengths* (Å)

C(1)–N(2)	1·302 (5)	C(1)–C(11A)	1·383 (5)
C(1)–F(14)	1·337 (4)	N(2)–C(3)	1·312 (5)
C(3)–C(4)	1·369 (6)	C(3)–F(15)	1·336 (4)
C(4)–C(4A)	1·379 (5)	C(4)–F(16)	1·341 (4)
C(4A)–N(5)	1·435 (4)	C(4A)–C(11A)	1·383 (5)
N(5)–N(6)	1·263 (4)	N(6)–C(6A)	1·425 (4)
C(6A)–C(7)	1·402 (4)	C(6A)–C(10A)	1·396 (4)
C(7)–C(8)	1·385 (5)	C(7)–C(12)	1·500 (5)
C(8)–C(9)	1·390 (4)	C(9)–C(10)	1·385 (4)
C(9)–C(13)	1·483 (5)	C(10)–C(10A)	1·379 (5)
C(10A)–C(11)	1·524 (5)	C(11)–C(11A)	1·495 (5)
C(13)–O(17)	1·446 (4)	O(17)–C(18)	1·340 (4)
C(18)–O(19)	1·181 (4)	C(18)–C(20)	1·503 (6)

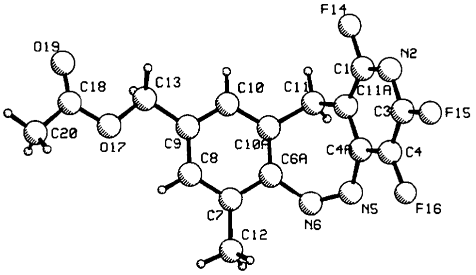

Fig. 1. Molecular view showing the atomic labelling produced using *PLUTO*78 (Motherwell & Clegg, 1978).

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12α-Hydroxy-3-oxooleanano-28,13-lactone

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Abstract. $C_{30}H_{46}O_4$, $M_r = 470·70$, triclinic, $P1$, $a = 7·155\ (3)$, $b = 14·802\ (5)$, $c = 6·744\ (3)$ Å, $\alpha = 99·97\ (3)$, $\beta = 114·04\ (3)$, $\gamma = 77·59\ (3)°$, $V = 634·1\ (9)$ Å^3, $Z = 1$, $D_x = 1·233$, D_m(flotation in aqueous $ZnCl_2$) = 1·23 (1) g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·742$ cm^{-1}, $F(000) = 258$, $T = 293$ K, $R = 0·040$ for 2629 unique observations. This molecule is a natural product isolated from a methanol-soluble fraction of dammar resin. Bond-distance arguments establish the carbonyl functionality at position 3, C(3)—O(3) = 1·201 (4) Å, and the hydroxyl group at position 12, C(12)—O(12) = 1·415 (3) Å. The hydroxyl

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substituent lies in an axial orientation toward the α face in a *transoid* orientation relative to the oxygen attached to position 13. Lactone formation between the 13-hydroxyl and the 28-acid functionalities has been confirmed. The lactone carbonyl oxygen, O(28), acts as an acceptor in an intermolecular hydrogen bond from the 12-hydroxyl. The associated metrical parameters are O(12)···O(28) = 2·875 (3), H(O12)···O(28) = 2·21 (1) Å with an angle at hydrogen of 156 (2)°.

Experimental. Natural product isolated from a methanol-soluble extract of dammar resin (Poehland, Carté, Francis, Hyland, Allaudeen & Troupe, 1987). Crystals grown by slow evaporation of a 50:50 $CHCl_3$:MeOH solution. The colorless prismatic crystal employed for data collection had approximate dimensions 0·70 × 0·40 × 0·20 mm and was mounted on a glass fiber with epoxy resin. Cell constants from a least-squares fit to the angular settings for 25 reflections with $30 \leq 2\theta(\text{Mo}) \leq 35°$ measured on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. No systematic absences. Intensity data were collected using a variable-speed ω–2θ scan to a maximum $(\sin\theta)/\lambda$ of 0·66 Å^{-1}, $-9 \leq h \leq 9$, $-19 \leq k \leq 19$, $0 \leq l \leq 8$. A total of 3301 observations were collected; symmetry-equivalent reflections were averaged (agreement factors: 2·1% on I, 1·9% on F_o) leaving 3045 unique observations of which 2629 were considered observed at the $3\sigma(I)$ level. Data were corrected for decay (±4·2% over 42 h of data collection) using the program *CHORT* of the Enraf–Nonius (1982) *SDP*, and for Lorentz–polarization effects, but not for absorption. The structure was solved by a combination of direct methods (*MULTAN*82: Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and difference Fourier syntheses. The coordinates of atom O(3) were fixed to define the origin. The structural model was refined by full-matrix least squares (on F). The function minimized was $\sum w(|F_o| - |F_c|)^2$ where the weights were defined as $w = 4F_o/\sigma^2(I)$ with $\sigma(I) = [\sigma(I)^2 + (pF_o)^2]^{1/2}$ and p, a small percentage value which dampens the weights of large intensities to prevent them from biasing the refinement, was assigned a value of 0·05. Non-H atoms were refined with anisotropic librational parameters; all H atoms were located from difference Fourier maps and were refined with isotropic temperature factors. There was no evidence for secondary extinction. The refinement converged (Δ/σ max. = 0·12) to values of the standard crystallographic residuals R = 3·96% and wR = 4·96%. The 'goodness-of-fit' (S) = 1·49. A final difference map was featureless with maximum positive and negative features of 0·247 and 0·250 e Å^{-3}, respectively. Refinement using 2940 observations with $I \geq 0{\cdot}01\sigma(I)$ gave R = 4·44% and wR = 5·10%. Values of the neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); H-atom scattering factors from Stewart, Davidson & Simpson (1965). Fractional atomic coordinates are contained in Table 1.* The atom numbering is given in Fig. 1.

Table 1. *Fractional atomic coordinates, equivalent isotropic thermal parameters and their e.s.d.'s for non*-H *atoms*

	x	y	z	B_{eq}*(Å^2)
O(3)	0·8925	1·3664	0·6485	9·38 (8)
O(12)	1·0760 (2)	0·8752 (1)	0·7524 (3)	3·30 (4)
O(13)	0·6325 (2)	0·8260 (1)	0·8264 (2)	2·06 (3)
O(28)	0·4269 (3)	0·7296 (1)	0·8117 (3)	3·11 (3)
C(1)	0·9735 (4)	1·1711 (2)	0·8997 (5)	3·32 (6)
C(2)	0·9882 (4)	1·2754 (2)	0·9382 (6)	4·54 (8)
C(3)	0·8359 (5)	1·3242 (2)	0·7452 (6)	4·75 (8)
C(4)	0·6090 (4)	1·3134 (2)	0·6696 (4)	3·08 (5)
C(5)	0·6045 (3)	1·2067 (2)	0·6489 (4)	2·25 (4)
C(6)	0·3857 (4)	1·1829 (2)	0·5662 (4)	2·65 (5)
C(7)	0·3873 (3)	1·0809 (2)	0·4754 (4)	2·49 (5)
C(8)	0·5370 (3)	1·0135 (1)	0·6443 (3)	1·83 (4)
C(9)	0·7528 (3)	1·0457 (1)	0·7647 (3)	1·89 (4)
C(10)	0·7543 (3)	1·1520 (2)	0·8469 (4)	2·25 (4)
C(11)	0·8910 (3)	0·9812 (2)	0·9427 (4)	2·48 (5)
C(12)	0·9319 (3)	0·8811 (1)	0·8503 (3)	2·13 (4)
C(13)	0·7353 (3)	0·8441 (1)	0·6872 (3)	1·70 (4)
C(14)	0·5778 (3)	0·9129 (1)	0·5236 (3)	1·79 (4)
C(15)	0·3737 (3)	0·8723 (2)	0·4061 (4)	2·43 (5)
C(16)	0·4034 (3)	0·7671 (2)	0·3429 (4)	2·44 (5)
C(17)	0·5704 (3)	0·7130 (1)	0·5312 (3)	2·00 (4)
C(18)	0·7803 (3)	0·7428 (1)	0·5903 (3)	1·88 (4)
C(19)	0·8649 (3)	0·7097 (2)	0·4116 (4)	2·38 (4)
C(20)	0·8845 (4)	0·6024 (2)	0·3643 (4)	2·72 (5)
C(21)	0·6757 (4)	0·5722 (2)	0·3107 (4)	2·96 (5)
C(22)	0·5824 (3)	0·6078 (2)	0·4825 (4)	2·65 (5)
C(23)	0·4861 (5)	1·3505 (2)	0·4479 (5)	4·14 (7)
C(24)	0·5196 (5)	1·3727 (2)	0·8306 (5)	3·97 (7)
C(25)	0·7065 (4)	1·1784 (2)	1·0537 (4)	3·25 (6)
C(26)	0·4311 (3)	1·0142 (2)	0·8016 (4)	2·65 (5)
C(27)	0·6684 (4)	0·9182 (2)	0·3530 (3)	2·68 (5)
C(28)	0·5311 (3)	0·7530 (2)	0·7350 (3)	2·16 (4)
C(29)	1·0545 (4)	0·5540 (2)	0·5562 (5)	3·49 (6)
C(30)	0·9419 (4)	0·5747 (2)	0·1645 (5)	4·01 (6)

* $B_{eq} = \frac{4}{3}(a^2B_{11} + b^2B_{22} + c^2B_{33} + abB_{12}\cos\gamma + acB_{13}\cos\beta + bcB_{23}\cos\alpha)$.

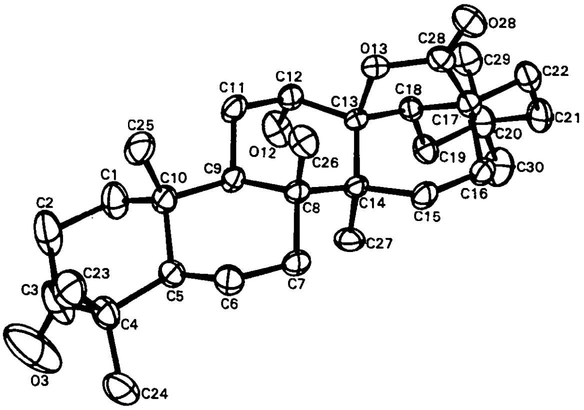

Fig. 1. *ORTEP*II diagram (Johnson, 1976) and atom-numbering scheme. Non-H ellipsoids at 50% probability level, H atoms as spheres of arbitrary size.

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* Lists of structure factors, anisotropic temperature factors, bond lengths and angles and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43781 (42 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. γ-Lactones of the oleanan-28-oic acid skeleton have been prepared synthetically by a number of routes leading to the 3-hydroxy-12-oxo, 3,12-dioxo and 3,12-dihydroxy species (Kitagawa, Kitazawa & Yosioka, 1968, 1972; Majumder & Chakraborty, 1979; Majumder & Bagchi, 1983). The 12-hydroxy-3-oxo compound reported here completes the tetrad, although this product was isolated from a natural source.

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Polymorphisme de la (−) Bromo-10 Méthoxycarbonyl-1 Nitro-16 Aspidospermidine-(2*R*,7*R*,16*S*,20*S*,21*S*): Structure d'une Nouvelle Variété

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Abstract. $C_{21}H_{26}BrN_3O_4$, $M_r = 464\cdot4$, monoclinic, $P2_1$, $a = 11\cdot370\ (2)$, $b = 10\cdot529\ (3)$, $c = 8\cdot536\ (4)$ Å, $\beta = 90\cdot66\ (2)°$, $V = 1021\cdot9$ Å^3, $Z = 2$, $D_x = 1\cdot509$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 2\cdot02$ mm^{-1}, $F(000) = 480$, $T = 294$ K, $R = 0\cdot053$ for 845 independent reflections $[I > \sigma(I)]$. The crystals of the previously described variety exhibit the orthorhombic $P2_12_12_1$ symmetry and have a density of 1·495 Mg m^{-3}. In the two forms the ring conformations are planar for *A* and *B*, chair for *D* and boat for *E*. For *C*, the conformation is sofa in the monoclinic form and approximately half-chair in the orthorhombic one. Homologous bond lengths and angles are not significantly different, except for the angles relative to atoms fixing substituent groups. The torsion-angle values indicate that the most important differences between the molecules of the two modifications consist of the relative orientations of the methoxycarbonyl, nitro and ethyl groups.

Partie expérimentale. Cristaux obtenus par évaporation d'une solution dans un mélange de $CHCl_3$ et C_2H_5OH. Cristal prismatique: 0,055 × 0,125 × 0,250 mm. Dimensions de la maille sur monocristal avec 25 réflexions telles que $2{,}63 \leq \theta \leq 11{,}73°$. Diffractomètre Enraf–Nonius CAD-4 $0{,}049 \leq (\sin\theta)/\lambda \leq 0{,}527$ Å^{-1}; $0 \leq h \leq 11$, $0 \leq k \leq 11$ et $-8 \leq l \leq 8$. Réflexions de contrôle des intensités: $00\bar{2}$, $2\bar{2}\bar{1}$ et $\bar{1}\bar{2}1$. Diminution de I au cours des mesures: 1,1%, $\sigma(I)/I_{\text{moyen}}$: 0,0036. 1327 réflexions indépendantes mesurées, 482 inobservées $[I < \sigma(I)]$. Corrections d'absorption (Coppens, Leiserowitz & Rabinovich, 1965). Valeurs extrêmes du coefficient de transmission: 0,895 et 0,779. Programme *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). H des CH_3: série de Fourier des ΔF; autres H: positions calculées. Affinement sur F, programme à matrice entière. Paramètres affinés: x, y, z de Br, O, N et C, β_{ij} de Br et B de O, N et C; O, N et C affinés en agitation isotrope

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