Related literature. A fused pyrrolo-arabinofuranosyl-nucleoside has recently been reported (Girgis, Cottam, Larson & Robins, 1987*a*). Nucleoside and nucleotide structural parameters have been discussed by Altona & Sundaralingam (1972).

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9-Acetyloxymethyl-1,3,4-trifluoro-7-methyl-11*H*-pyrido[4,3-*c*]benzo[1,2]diazepine

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Abstract. $C_{16}H_{12}F_3N_3O_2$, $M_r = 335{\cdot}3$, monoclinic, $P2_1/c$, $a = 13{\cdot}591\,(4)$, $b = 4{\cdot}823\,(3)$, $c = 24{\cdot}930\,(4)$ Å, $\beta = 115{\cdot}60\,(2)°$, $V = 1473{\cdot}7$ Å^3, $Z = 4$, $D_x = 1{\cdot}51$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}084$ mm^{-1}, $F(000) = 688$, $T = 293$ K, $R = 0{\cdot}042$ for 1313 unique reflexions $[F > 3\sigma(F)]$. The ring skeleton is composed of a boat-shaped diazepine nucleus flanked by benzene and pyridine rings, whose planes intersect at an angle of 113·6 (6)°.

Experimental. The title compound was obtained in admixture with 1,3,4-trifluoro-7,9-dimethyl-11*H*-pyrido[4,3-*c*]benzo[1,2]diazepine by heating a solution of 2,3,5,6-tetrafluoro-4-(2,4,6-trimethylphenylazo)-pyridine in glacial acetic acid containing fused potassium acetate at 392 K for 4 h. The mixture was separated chromatographically followed by recrystallization from aqueous ethanol, m.p. 415 K. Crystal dimensions 0·2 × 0·2 × 0·1 mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, unit-cell dimensions from setting angles of 25 accurately centred reflexions ($4{\cdot}4 < \theta < 9{\cdot}1°$), ω–2θ scan mode used to measure 1905 reflexions with $I > 0$, ω-scan width of $0{\cdot}40° + 0{\cdot}35° \tan\theta$ and scan speed ranging from 0·6 to 5° min^{-1} according to the intensity gathered in a pre-scan, $-14 \le h \le 14$, $0 \le k \le 5$, $0 \le l \le 28$, $0 \le \theta \le 25°$, 1313 unique structure amplitudes with $F \ge 3\sigma(F)$, negligible drift in three intensity standards ($\bar{2}06$, $\bar{3}12$, 114) measured every 2 h, Lorentz and polarization corrections but absorption ignored, *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) used to solve the phase problem by direct methods, all non-H atoms found in a Fourier map, H atoms from difference Fourier map, full-matrix least-squares refinement based on *F* using *SHELX*76 (Sheldrick, 1976), final $R = 0{\cdot}042$, $wR = 0{\cdot}044$, $w = 1{\cdot}5022/[\sigma^2(F_o) + 0{\cdot}000272F_o^2]$, anisotropic thermal parameters for heavier atoms, isotropic for H. Maximum fluctuation in final difference map in range $-0{\cdot}15$ to $+0{\cdot}18$ e Å^{-3}, maximum LS shift-to-e.s.d. ratio 0·058 [*y*, H(20)].

Scattering factors from *International Tables for X-ray Crystallography* (1974), computations carried out on the joint CDC 7600/Amdahl 470 system of the University of Manchester Regional Computing Centre.

Fractional atomic coordinates and vibrational parameters are listed in Table 1,[*] selected bond lengths in Table 2. The molecule, including labelling scheme, is displayed in Fig. 1.

Related literature. Details of relevant structures can be found in Alty, Banks, Fishwick, Pritchard & Thompson (1984) and Pritchard (1987).

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[*] Lists of structure factors, anisotropic vibrational parameters, H-atom parameters, all bond lengths and angles, torsion angles and normalized least-squares-planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43690 (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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Table 1. *Fractional atomic coordinates* (×10⁴) *and equivalent isotropic vibrational parameters* (Å² ×10⁴) *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)

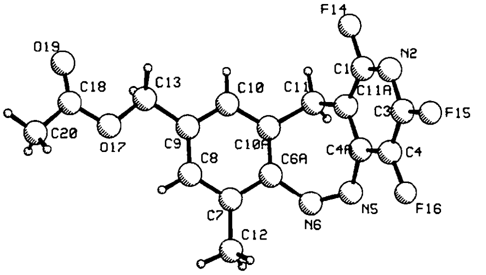

Fig. 1. Molecular view showing the atomic labelling produced using *PLUTO*78 (Motherwell & Clegg, 1978).

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

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Abstract. $C_{30}H_{46}O_4$, $M_r = 470{\cdot}70$, triclinic, $P1$, $a = 7{\cdot}155$ (3), $b = 14{\cdot}802$ (5), $c = 6{\cdot}744$ (3) Å, $\alpha = 99{\cdot}97$ (3), $\beta = 114{\cdot}04$ (3), $\gamma = 77{\cdot}59$ (3)°, $V = 634{\cdot}1$ (9) Å³, $Z = 1$, $D_x = 1{\cdot}233$, D_m(flotation in aqueous $ZnCl_2$) = 1·23 (1) g cm⁻³, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}742$ cm⁻¹, $F(000) = 258$, $T = 293$ K, $R = 0{\cdot}040$ 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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