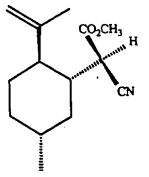
$wR = 0.061$; all non-H atoms anisotropic, H atoms were included using a riding model [C—H 0.96 Å, $U(H) = 0.08$ Å^2, except for: $U(H) = 0.12$ Å^2 for the methyl H of C(9) and C(14); $U(H) = 0.15$ Å^2 for the methyl H of C(5) and the olefinic H of C(4)]. 155 parameters were refined, $S = 1.63$, weighting scheme $w^{-1} = \sigma^2(F) + 0.0005F^2$ which led to a featureless analysis of variance in terms of $\sin\theta$ and F_o, max. $\Delta/\sigma = 0.001$, max. and min. height in final $\Delta\rho$ map 0.16 and -0.16 e Å^{-3} respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).



Atomic parameters are given in Table 1, selected bond distances and angles in Table 2.* Fig. 1 shows a thermal-ellipsoid plot with atom numbering.

* 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 52059 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The synthesis of the title compound *via* an intramolecular ene reaction has been published (Tietze & Beifuss, 1986). Experimental details of the synthesis and spectroscopic data of the compound will be published (Tietze, Beifuss & Ruther, 1989). For intramolecular ene reactions, see Tietze & Beifuss (1988).

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Structure of a 3,4-Dihydro-2*H*-pyran Derivative

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Abstract. (2*RS*,3*RS*,4*RS*)-(±)-3-Acetoxy-2-ethoxy-4-methoxycarbonylamino-3,4-dihydro-2*H*-pyran-5-carboxylic acid methyl ester, $C_{13}H_{19}NO_8$, $M_r = 317.30$, monoclinic, $P2_1/n$, $a = 11.839$ (2), $b = 8.767$ (1), $c = 15.293$ (4) Å, $\beta = 98.84$ (2)°, $V = 1568.36$ Å^3, $Z = 4$, $D_x = 1.344$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.11$ mm^{-1}, $F(000) = 672$, $T = 298$ K, $R = 0.060$ for 1906 observed reflections. The structure was investigated to determine the relative configuration, which could not be established unambiguously by NMR. The dihydro-2*H*-pyran ring adopts a half-chair conformation.

Experimental. (I): The crystal size was 0.4 × 0.4 × 0.7 mm. The intensity data were collected with a Stoe–Siemens four-circle diffractometer using monochromated Mo $K\alpha$ radiation and a profile-fitting mode involving variable scan width and speed (Clegg, 1981). 4316 reflections were measured, $2\theta_{max} = 50°$, $h -14 \rightarrow 14$, $k 0 \rightarrow 10$, $l -7 \rightarrow 18$. Three check

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$)

	x	y	z	U_{eq}
O(6)	5807 (2)	778 (2)	1427 (1)	57 (1)
O(9)	4654 (2)	−3334 (2)	2182 (1)	57 (1)
O(1)	3363 (2)	22 (3)	4197 (2)	63 (1)
O(8)	3233 (2)	−4107 (2)	2872 (2)	64 (1)
O(3)	5682 (2)	1971 (2)	3332 (1)	51 (1)
N(5)	4202 (2)	373 (3)	2039 (2)	43 (1)
O(7)	4096 (2)	1708 (2)	804 (1)	59 (1)
C(4)	4757 (2)	−393 (3)	2834 (2)	41 (1)
O(2)	4257 (2)	2234 (3)	4624 (2)	66 (1)
C(3)	5072 (2)	691 (3)	3609 (2)	44 (1)
C(4*a*)	4795 (3)	936 (3)	1429 (2)	42 (1)
C(5)	3984 (2)	−1600 (3)	3118 (2)	41 (1)
C(5*a*)	3899 (2)	−3126 (3)	2729 (2)	45 (1)
O(4)	7311 (2)	643 (3)	3657 (2)	75 (1)
C(6)	3379 (3)	−1293 (4)	3768 (2)	53 (1)
C(3*a*)	6823 (3)	1750 (4)	3345 (2)	56 (1)
C(2)	4001 (3)	1304 (4)	3911 (2)	53 (1)
C(3*b*)	7347 (3)	3024 (4)	2914 (3)	81 (1)
C(2*a*)	3267 (4)	3144 (6)	4799 (3)	112 (2)
C(5*b*)	4644 (3)	−4812 (4)	1761 (3)	76 (1)
C(2*b*)	3533 (5)	4127 (9)	5478 (4)	195 (3)
C(4*b*)	4625 (3)	2337 (5)	100 (2)	77 (2)

Table 2. *Bond lengths* (Å) *and angles* (°)

O(6)—C(4*a*)	1·206 (4)	O(9)—C(5*a*)	1·328 (4)
O(9)—C(5*b*)	1·446 (4)	O(1)—C(6)	1·328 (4)
O(1)—C(2)	1·459 (4)	O(8)—C(5*a*)	1·209 (4)
O(3)—C(3)	1·433 (4)	O(3)—C(3*a*)	1·362 (4)
N(5)—C(4)	1·454 (3)	N(5)—C(4*a*)	1·345 (4)
O(7)—C(4*a*)	1·346 (3)	O(7)—C(4*b*)	1·437 (4)
C(4)—C(3)	1·521 (4)	C(4)—C(5)	1·506 (4)
O(2)—C(2)	1·358 (4)	O(2)—C(2*a*)	1·476 (5)
C(3)—C(2)	1·513 (4)	C(5)—C(5*a*)	1·461 (4)
C(5)—C(6)	1·339 (4)	O(4)—C(3*a*)	1·191 (4)
C(3*a*)—C(3*b*)	1·480 (5)	C(2*a*)—C(2*b*)	1·349 (8)
C(5*a*)—O(9)—C(5*b*)	116·3 (2)	C(6)—O(1)—C(2)	118·2 (3)
C(3)—O(3)—C(3*a*)	115·3 (2)	C(4)—N(5)—C(4*a*)	122·1 (2)
C(4*a*)—O(7)—C(4*b*)	115·8 (2)	N(5)—C(4)—C(3)	112·8 (2)
N(5)—C(4)—C(5)	110·4 (2)	C(3)—C(4)—C(5)	107·5 (2)
C(2)—O(2)—C(2*a*)	112·7 (3)	O(3)—C(3)—C(4)	109·5 (2)
O(3)—C(3)—C(2)	107·2 (2)	C(4)—C(3)—C(2)	110·1 (2)
O(6)—C(4*a*)—N(5)	125·8 (3)	O(6)—C(4*a*)—O(7)	123·9 (3)
N(5)—C(4*a*)—O(7)	110·3 (2)	C(4)—C(5)—C(5*a*)	122·2 (3)
C(4)—C(5)—C(6)	119·4 (3)	C(5*a*)—C(5)—C(6)	118·4 (3)
O(9)—C(5*a*)—O(8)	122·5 (3)	O(9)—C(5*a*)—C(5)	111·8 (3)
O(8)—C(5*a*)—C(5)	125·7 (3)	O(1)—C(6)—C(5)	126·3 (3)
O(3)—C(3*a*)—O(4)	122·7 (3)	O(3)—C(3*a*)—C(3*b*)	111·7 (3)
O(4)—C(3*a*)—C(3*b*)	125·6 (3)	O(1)—C(2)—O(2)	106·5 (3)
O(1)—C(2)—C(3)	108·3 (2)	O(2)—C(2)—C(3)	111·4 (2)
O(2)—C(2*a*)—C(2*b*)	112·8 (4)		

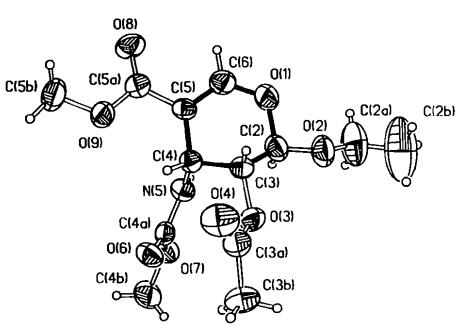
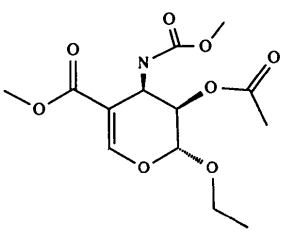


Fig. 1. Atom-numbering scheme and 50% probability thermal-motion ellipsoids for the title compound.

reflections showed no significant intensity change. 2755 unique reflections measured ($R_{int} = 0·022$), of which 1906 with $F > 4\sigma(F)$ were used for all calculations (*SHELXS*86, Sheldrick, 1985; *SHELX*76, Sheldrick, 1976). Cell constants refined from $\pm 2\theta$ values of 40 reflections in the range 20–25°. Absorption and extinction corrections were not necessary. The structure was solved by direct methods. Refinement on F converged to $R = 0·060$, $wR = 0·071$; all non-H atoms anisotropic. H atoms were included using a riding model [C—H 0·96 Å, $U(H) = 0·08$ Å^2, except for methyl protons $U(H) = 0·10$ Å^2, with $U(H) = 0·25$ Å^2 for the methyl protons of C(2*b*)]. 199 parameters were refined, giving $S = 2·00$, using a weighting scheme $w^{-1} = \sigma^2(F) + 0·0005F^2$ which led to a featureless analysis of variance in terms of $\sin\theta$ and F_o, $\Delta/\sigma \leq 0·001$, max. and min. heights in final $\Delta\rho$ map 0·24 and −0·32 e Å^{-3} respectively. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The large thermal motion of the terminal methyl group C(2*b*) probably contributed to the relatively high R index.

Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 shows a thermal-ellipsoid plot with atom numbering.



(I)

Related literature. For the preparation of the compound see Hartfiel (1987). For the preparation of some related compounds see Tietze, Voss, Harms & Sheldrick (1985) and Voss (1987).

We thank the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support.

* Lists of structure factors, hydrogen coordinates and anisotropic vibrational parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52076 (12 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 (3*R*,6*R*,7*R*,11*S*)-7-Acetoxymethyl-5,8,10-trioxa-1-azatricyclo[4.3.2.$0^{3,11}$]undecan-4-one

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Abstract. $C_{10}H_{13}NO_6$, $M_r = 243{\cdot}2$, monoclinic, $P2_1$, $a = 5{\cdot}558\ (2)$, $b = 8{\cdot}119\ (4)$, $c = 11{\cdot}947\ (4)$ Å, $\beta = 98{\cdot}19\ (2)°$, $V = 534\ (2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}514$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1{\cdot}185$ cm^{-1}, $F(000) = 256$, $T = 298$ K, final $R = 0{\cdot}046$ for 920 observed reflections. The two five-membered rings adopt different conformations: envelope and half-chair. The seven-membered ring shows the distorted chair conformation. The shortest non-bonded contact is 3·29 Å for non-H atoms.

Experimental. Crystal of approximate dimensions 0·28 × 0·28 × 0·30 mm; intensities measured at 298 K on an Enraf–Nonius CAD-4 four-circle diffractometer (Mo $K\alpha$ radiation, graphite monochromator). Lattice parameters determined by least squares from 20 reflections ($12 \le 2\theta \le 28°$). Total of 1174 reflections up to $\theta = 26°$ ($0 \le h \le 6$, $0 \le k \le 10$, $-14 \le l \le 14$) measured in the ω–2θ scan mode, 920 reflections considered as observed [$F_o > 1{\cdot}5\sigma(F_o)$]. Three reference reflections monitored every hour showed no significant variation in intensity. No absorption or secondary-extinction correction. Structure solved by *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least squares with the *SDP* system (B. A. Frenz & Associates Inc., 1985). Weights of each reflection in refinement (on F) calculated from $w = 1/\sigma^2(F_o)$, $\sigma(F_o)$ being the e.s.d., based on counting statistics, of the observed structure factor. Scattering factors taken from *International Tables for X-ray Crystallography* (1974). The total number of parameters refined was 153: one scale factor, position parameters and anisotropic thermal parameters for non-H atoms; no attempt was made to refine the positions [calculated at d(C—H) = 0·95 Å] or isotropic thermal parameters ($B = 5{\cdot}0$ Å^2) of the H atoms. Refinement resulted in final values of $R = 0{\cdot}046$, $wR = 0{\cdot}049$ and $S = 3{\cdot}18$; in the last cycle $(\Delta/\sigma)_{max} = 0{\cdot}02$. Final max. and min. $\Delta\rho$ were 0·21 and $-0{\cdot}28$ e Å^{-3}, respectively. All calculations performed on a MicroPDP11/73 computer. The final

Table 1. *Fractional atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2) *for non-*H *atoms*

E.s.d.'s are in parentheses.

$B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	z	B_{eq}
C(1)	6743 (7)	9188 (6)	3037 (3)	2·85 (8)
O(2)	8225 (5)	7740*	2927 (2)	3·35 (6)
C(3)	8373 (8)	7438 (6)	1829 (4)	3·57 (9)
O(3)	9739 (7)	6432 (5)	1555 (3)	6·46 (9)
C(4)	6521 (8)	8443 (6)	1072 (3)	3·62 (9)
C(5)	7581 (8)	9748 (7)	362 (3)	4·1 (1)
N(6)	7212 (6)	11351 (5)	881 (3)	3·56 (7)
C(7)	9187 (8)	11937 (6)	1696 (3)	3·70 (9)
O(8)	10055 (4)	10827 (4)	2582 (2)	2·71 (5)
C(9)	8402 (7)	10615 (5)	3399 (3)	2·48 (7)
C(10)	5205 (7)	9435 (6)	1868 (3)	3·34 (9)
O(11)	5115 (5)	11094 (4)	1457 (2)	3·73 (6)
C(12)	10061 (7)	10343 (6)	4516 (3)	2·89 (8)
O(12)	8813 (5)	9580 (4)	5371 (2)	2·83 (5)
C(13)	7069 (7)	10491 (5)	5764 (3)	2·83 (8)
O(13)	6515 (5)	11839 (4)	5423 (2)	3·66 (6)
C(14)	5975 (8)	9573 (7)	6648 (3)	3·74 (9)

* Origin-defining coordinate.

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