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Structure of Methyl 2-Cyano-2-(2-isopropenyl-5-methylcyclohexyl)acetate

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Abstract. Methyl (2*RS*,1′*RS*,2′*RS*,5′*RS*)-2-cyano-2-(2′-isopropenyl-5′-methylcyclohexyl)acetate, $C_{14}H_{21}NO_2$, $M_r = 235{\cdot}33$, monoclinic, $P2_1/c$, $a = 15{\cdot}631\ (2)$, $b = 11{\cdot}021\ (1)$, $c = 8{\cdot}332\ (1)$ Å, $\beta = 92{\cdot}99\ (1)°$, $V = 1433{\cdot}4$ Å^3, $Z = 4$, $D_x = 1{\cdot}09$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}07$ mm^{-1}, $F(000) = 512$, $T = 298$ K, $R = 0{\cdot}053$ for 1575 observed reflections. The structure was investigated to determine the relative configuration, which could not be established unambiguously by NMR. The cyclohexane ring adopts a chair conformation. There are two short intermolecular distances N(1)···H(2*a*) [2·65 Å (x, $-0{\cdot}5-y$, $0{\cdot}5+z$)] and H(1*aa*)···O(1) [2·47 Å ($-x$, $0{\cdot}5+y$, $0{\cdot}5-z$)].

Experimental. Crystal size 0·2 × 0·6 × 0·4 mm. Stoe–Siemens four-circle diffractometer, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 3684 reflections measured, $2\theta_{\max}$ 50°, $\pm h + k + l$, three check reflections with no significant intensity change. 2433 unique reflections ($R_{\rm int} = 0{\cdot}021$), of which 1575 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 36 reflections in the range 20–25°. Absorption correction was not necessary. Extinction correction was performed yielding a secondary-extinction coefficient x of 0·011 (1), where $F_c^* = F_c(1 + 0{\cdot}002xF_c^2/\sin 2\theta)^{-0{\cdot}25}$. Structure solution by direct methods. Refinement on F to $R = 0{\cdot}053$,

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

	x	y	z	$U_{\rm eq}$*
C(1)	2606 (1)	50 (2)	4381 (2)	52 (1)
C(2)	1689 (1)	−399 (2)	4572 (3)	70 (1)
C(3)	1202 (2)	−632 (3)	2992 (3)	90 (1)
C(4)	920 (2)	−1736 (3)	2620 (4)	129 (2)
C(5)	1041 (2)	404 (4)	1901 (4)	135 (2)
C(6)	1221 (2)	476 (3)	5647 (3)	97 (1)
C(7)	1691 (2)	661 (3)	7255 (3)	88 (1)
C(8)	2593 (2)	1112 (2)	7093 (3)	67 (1)
C(9)	3075 (2)	1259 (2)	8710 (3)	91 (1)
C(10)	3064 (2)	264 (2)	6012 (2)	58 (1)
C(11)	3121 (1)	−823 (2)	3340 (3)	52 (1)
C(12)	3182 (2)	−2035 (2)	4066 (3)	66 (1)
C(13)	4013 (1)	−368 (2)	3060 (3)	55 (1)
C(14)	4787 (2)	1274 (3)	2061 (4)	89 (1)
N(1)	3207 (2)	−2962 (2)	4652 (3)	101 (1)
O(1)	4661 (1)	−900 (2)	3387 (3)	88 (1)
O(2)	3974 (1)	719 (1)	2398 (2)	67 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

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

C(1)—C(2)	1·532 (3)	C(1)—C(10)	1·520 (3)
C(1)—C(11)	1·548 (3)	C(2)—C(3)	1·508 (3)
C(2)—C(6)	1·528 (4)	C(3)—C(4)	1·326 (5)
C(3)—C(5)	1·473 (5)	C(6)—C(7)	1·508 (3)
C(7)—C(8)	1·508 (4)	C(8)—C(9)	1·518 (3)
C(8)—C(10)	1·514 (3)	C(11)—C(12)	1·467 (3)
C(11)—C(13)	1·511 (3)	C(12)—N(1)	1·132 (3)
C(13)—O(1)	1·190 (3)	C(13)—O(2)	1·319 (3)
C(14)—O(2)	1·452 (3)		
C(2)—C(1)—C(10)	110·9 (2)	C(2)—C(1)—C(11)	112·0 (2)
C(10)—C(1)—C(11)	111·3 (2)	C(1)—C(2)—C(3)	113·3 (2)
C(1)—C(2)—C(6)	109·7 (2)	C(3)—C(2)—C(6)	112·6 (2)
C(2)—C(3)—C(4)	120·5 (3)	C(2) —C(3)—C(5)	118·0 (3)
C(4)—C(3)—C(5)	121·5 (3)	C(2)—C(6)—C(7)	112·3 (2)
C(6)—C(7)—C(8)	112·3 (2)	C(7)—C(8)—C(9)	112·3 (2)
C(7)—C(8)—C(10)	109·6 (2)	C(9)—C(8)—C(10)	111·1 (2)
C(1)—C(10)—C(8)	114·0 (2)	C(1)—C(11)—C(12)	111·1 (2)
C(1)—C(11)—C(13)	113·0 (2)	C(12)—C(11)—C(13)	109·0 (2)
C(11)—C(12)—N(1)	177·9 (3)	C(11)—C(13)—O(1)	125·6 (2)
C(11)—C(13)—O(2)	110·1 (2)	O(1)—C(13)—O(2)	124·3 (2)
C(13)—O(2)—C(14)	116·2 (2)		

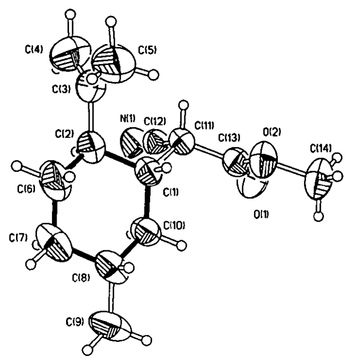

Fig. 1. A thermal-ellipsoid plot of the molecule showing the atom numbering.

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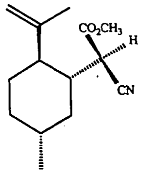
wR = 0·061; all non-H atoms anisotropic, H atoms were included using a riding model [C—H 0·96 Å, U(H) = 0·08 Å^2, except for: U(H) = 0·12 Å^2 for the methyl H of C(9) and C(14); U(H) = 0·15 Å^2 for the methyl H of C(5) and the olefinic H of C(4)]. 155 parameters were refined, S = 1·63, weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}0005F^2$ which led to a featureless analysis of variance in terms of sinθ and F_o, max. Δ/σ = 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.

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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* 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.

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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, β = 98·84 (2)°, V = 1568·36 Å^3, Z = 4, D_x = 1·344 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 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→14, k0→10, l −7→18. Three check

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