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Structure of Methyl 8-Isopropyl-3,3a,8,8a-tetrahydroindeno[2,1-*c*]pyrazole-8a-carboxylate, $C_{15}H_{18}N_2O_2$

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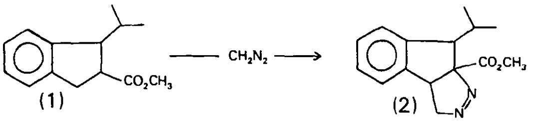
Abstract. $M_r = 258{\cdot}8$, triclinic, $P\bar{1}$, $a = 5{\cdot}764\,(4)$, $b = 15{\cdot}400\,(3)$, $c = 15{\cdot}810\,(4)$ Å, $\alpha = 97{\cdot}45\,(6)$, $\beta = 100{\cdot}39\,(5)$, $\gamma = 100{\cdot}82\,(6)°$, $V = 1375\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}25$, $D_m = 1{\cdot}27$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}078$ mm^{-1}, room temperature, $F(000) = 552$, $R_w = 0{\cdot}065$ for 1518 reflexions (6616 unique observations). There are two crystallographically independent molecules in the asymmetric unit. The isopropyl and methoxycarbonyl groups are *cis*.

Introduction. The cycloaddition reaction of diazomethane with compound (1) leads to the tricyclic compound (2). Knowledge of the stereochemistry of this compound was necessary to explain the stereospecificity of the reaction (Vebrel, Cetutti & Carrie, 1979; Vebrel & Carrie, 1983). An X-ray analysis was therefore necessary.



Experimental. Crystal (cubic form: 0·2 × 0·2 × 0·2 mm) grown from a saturated solution in ethanol. Unit cell: least-squares refinement of a set of 25 reflections. Automated Enraf–Nonius CAD-4 diffractometer, monochromatized Mo $K\alpha$ radiation, $\omega/2\theta$ scan mode. $\theta_{max} = 28°$. Range of hkl: $\bar{7}$, 7; $\overline{20}$, 20; 0, 21. Standard reflections (024, $\bar{1}01$, 040): ± 1%. 6853 reflections measured, 6616 unique [1518 with $I > \sigma(I)$], $R_{int} = 0{\cdot}024$. Structure solved by direct methods (Germain, Main & Woolfson, 1971) after computing normalized structure factors. All heavy atoms of the two independent molecules obtained with the best set. After refinements, a Fourier synthesis revealed all the H atoms (between 0·315 and 0·153 e Å^{-3}). The isotropic thermal parameters assigned to the H atoms were not refined. The last refinement (on F) with $1/w = \sigma^2/F = 1/4\,[(\sigma^2/I) + (0{\cdot}04)^2 I]$ lowered R_w to 0·065 and R to 0·069. $\Delta/\sigma = 0{\cdot}9$ for H atoms, 0·08 for non-H atoms. No correction for secondary extinction or absorption. All calculations performed on a Digital PDP 11/60 computer with the *SDP* package (Frenz, 1978).

Discussion. Atomic parameters are given in Table 1.* Bond distances and angles are in Fig. 1. These data agree with normal values, and differences between corresponding bond distances in molecules *A* and *B* are not statistically significant. The two pyrazoline rings have a classical conformation: the dihedral angles are respectively 25·5 and 24·6°.

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* Lists of structure factors, anisotropic thermal parameters for non-H atoms and atomic coordinates for H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38963 (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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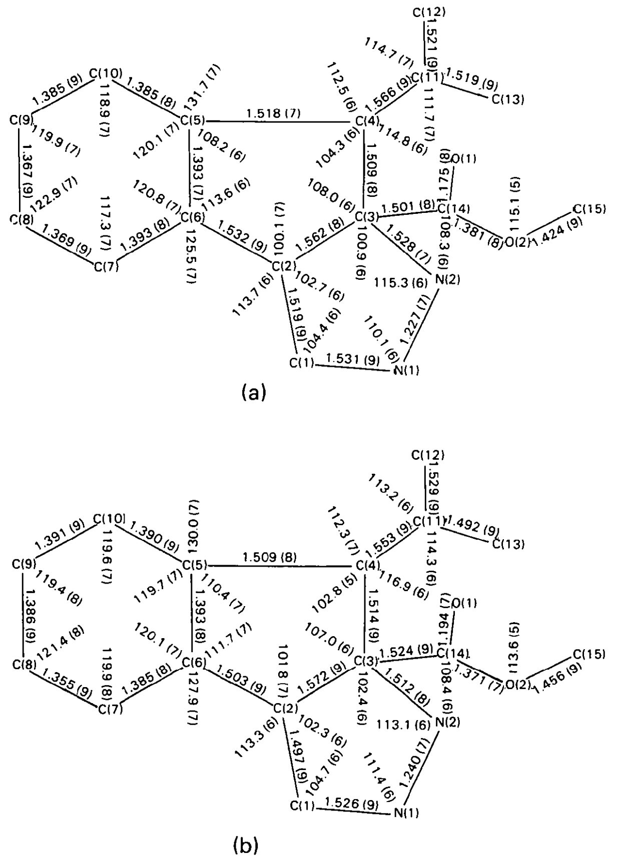

Fig. 1. Bond distances (Å) and angles (°) (*a*) for molecule *A* and (*b*) for molecule *B*. [C(4)–C(3)–C(14) 115·6 (6), 115·3 (6); C(14)–C(3)–N(2) 107·4 (5), 107·7 (5); C(3)–C(14)–O(1) 128·7 (7), 126·6 (7); O(1)–C(14)–O(2) 122·9 (7), 124·9 (6); C(12)–C(11)–C(13) 108·6 (7), 109·3 (7)° for *A*, *B* respectively.]

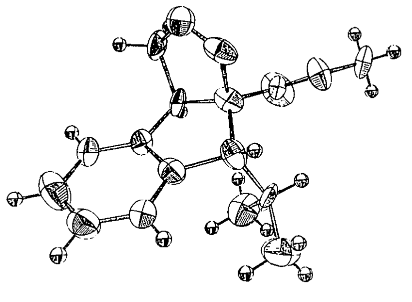

Fig. 2. A view of molecule *A*.

Table 1. *Atomic coordinates and B_{eq} values for molecules A and B*

$$B_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq} (Å^2)
O(1*A*)	0·4499 (10)	0·3850 (5)	0·8407 (4)	5·4 (2)
O(2*A*)	0·8172 (9)	0·3661 (4)	0·8744 (4)	4·1 (2)
N(1*A*)	0·455 (1)	0·4279 (5)	1·0810 (4)	3·9 (2)
N(2*A*)	0·610 (1)	0·3777 (5)	1·0395 (4)	3·7 (2)
C(1*A*)	0·210 (1)	0·4219 (6)	1·0621 (6)	3·9 (2)
C(2*A*)	0·248 (1)	0·3361 (6)	1·0214 (5)	3·7 (2)
C(3*A*)	0·520 (1)	0·3231 (5)	0·9835 (5)	2·5 (2)
C(4*A*)	0·623 (1)	0·2270 (6)	1·0018 (5)	2·9 (2)
C(5*A*)	0·456 (1)	0·1947 (6)	1·0799 (5)	3·1 (2)
C(6*A*)	0·242 (1)	0·2541 (6)	1·0867 (5)	2·7 (2)
C(7*A*)	0·052 (1)	0·2392 (6)	1·1511 (6)	3·5 (2)
C(8*A*)	0·083 (2)	0·1641 (6)	1·2066 (6)	4·5 (3)
C(9*A*)	0·295 (2)	0·1068 (6)	1·2036 (6)	4·9 (3)
C(10*A*)	0·485 (2)	0·1217 (6)	1·1399 (6)	4·1 (3)
C(11*A*)	0·658 (1)	0·1703 (6)	0·9252 (6)	3·8 (2)
C(12*A*)	0·419 (2)	0·1636 (7)	0·8971 (6)	5·8 (3)
C(13*A*)	0·785 (2)	0·0777 (8)	0·9443 (7)	6·3 (4)
C(14*A*)	0·577 (1)	0·3620 (6)	0·8922 (5)	3·7 (2)
C(15*A*)	0·902 (1)	0·3992 (7)	0·7887 (6)	5·0 (3)
O(1*B*)	−0·3282 (10)	0·1151 (5)	0·6587 (4)	5·3 (2)
O(2*B*)	0·0601 (9)	0·1333 (4)	0·6257 (3)	4·1 (2)
N(1*B*)	−0·037 (1)	0·0728 (5)	0·4191 (5)	4·2 (2)
N(2*B*)	0·032 (1)	0·1225 (4)	0·4602 (4)	3·5 (2)
C(1*B*)	−0·308 (1)	0·0807 (6)	0·4373 (6)	3·9 (2)
C(2*B*)	−0·394 (1)	0·1647 (6)	0·4784 (5)	3·2 (2)
C(3*B*)	−0·170 (1)	0·1761 (6)	0·5156 (6)	3·2 (2)
C(4*B*)	−0·149 (1)	0·2730 (6)	0·4984 (5)	3·1 (2)
C(5*B*)	−0·272 (1)	0·3035 (6)	0·4217 (5)	3·2 (2)
C(6*B*)	−0·422 (1)	0·2453 (6)	0·4146 (5)	3·4 (2)
C(7*B*)	−0·558 (1)	0·2617 (7)	0·3494 (6)	4·3 (3)
C(8*B*)	−0·544 (2)	0·3345 (7)	0·2924 (6)	4·6 (3)
C(9*B*)	−0·394 (2)	0·3930 (6)	0·2971 (5)	4·2 (3)
C(10*B*)	−0·257 (2)	0·3773 (6)	0·3622 (6)	4·5 (3)
C(11*B*)	−0·251 (1)	0·3308 (6)	0·5734 (6)	4·0 (3)
C(12*B*)	−0·520 (2)	0·3364 (7)	0·6012 (7)	6·0 (3)
C(13*B*)	−0·191 (2)	0·4217 (7)	0·5575 (6)	5·3 (3)
C(14*B*)	−0·164 (1)	0·1359 (6)	0·6079 (5)	3·6 (2)
C(15*B*)	0·087 (2)	0·0978 (7)	0·7132 (6)	4·7 (3)

The main result of this study is that the methoxycarbonyl and isopropyl groups are *cis*, as shown in Fig. 2. This geometry explains the Overhauser effect observed in ^{1}H NMR and elucidates the behaviour of compound (1) with diazomethane.

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