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3,4-Dihydro-2,5-dimethyl-7-propylimidazo[5,1-*f*][1,2,4]triazine

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Abstract. $C_{10}H_{16}N_4$, $M_r = 192{\cdot}2$, monoclinic, $P2_1/c$, $a = 7{\cdot}90\ (3)$, $b = 11{\cdot}83\ (4)$, $c = 14{\cdot}23\ (8)$ Å, $\beta = 56{\cdot}75\ (5)°$ from diffractometer measurements (Mo $K\bar{\alpha}$ radiation), $V = 1112{\cdot}2$ Å^3, $Z = 4$, $D_c = 1{\cdot}15$ Mg m^{-3}, $F(000) = 416$, $\mu = 0{\cdot}042$ mm^{-1}, approximate crystal dimensions $0{\cdot}8 \times 0{\cdot}4 \times 0{\cdot}2$ mm. $R = 0{\cdot}068$ for 1171 observed reflexions. The structure consists of hydrogen-bonded molecules [N(3)···N′(6) = 2·94 (1) Å].

Introduction. The title compound (II) was recrystallized from ethyl acetate. Systematic absences indicated space group $P2_1/c$. Data were collected for $hk0$–12 with $\theta_{max} = 25°$ on a Stoe STADI-2 two-circle diffractometer (graphite-monochromated Mo $K\bar{\alpha}$ radiation). This gave 1694 data of which 1171 unique reflexions with $I > 3\sigma(I)$ were used in subsequent calculations. Lorentz and polarization corrections (but none for extinction or absorption) were applied, and the data scaled by a Wilson plot. The structure was solved by direct phasing methods with the *SHELX* 76 system of programs (Sheldrick, 1976), which was used for all calculations. Complex neutral-atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Weighted full-matrix least-squares refinement [including constrained isotropic H atoms except H(3)] converged at $R = 0{\cdot}068$ for 1171 observed reflexions ($R = \sum ||F_o| - |F_c|| / \sum |F_o|$); $R_w = 0{\cdot}075$ $\{R_w = \sum (||F_o| - |F_c||.w^{1/2}) / \sum (|F_o|.w^{1/2})$, $w = 5{\cdot}58/[\sigma^2(F_o) + 2{\cdot}2 \times 10^{-4} F_o^2]\}$. A difference map and high temperature factors for the atoms concerned suggest that there is disorder in the H atoms of the two methyl substituents and in the propyl group [C(13), C(14) and attached H atoms]. In the final cycle all shifts in parameters were less than their standard deviations. Positional parameters are given in Table 1 and bond distances and angles in Table 2.* A view of the molecule is shown in Fig. 1.

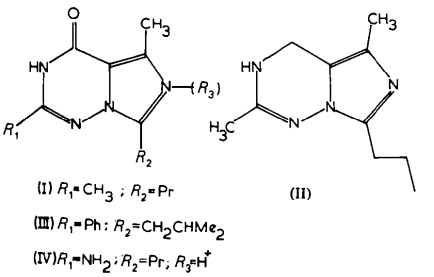

Table 1. *Fractional atomic coordinates* [$\times 10^4$; *for* H(3) $\times 10^3$] *and thermal parameters* (U_{eq} $\times 10^4$; $U_{iso} \times 10^3$)

The expression for U_{eq} is $U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{12}\cos\gamma + 2U_{13}\cos\beta + 2U_{23}\cos\alpha)$. E.s.d.'s for U_{eq} are about 25 (Å$^2 \times 10^4$). For H atoms, the expression for the temperature factor is $\exp(-8\pi^2 U_{iso} \sin^2\theta/\lambda^2)$.

	x	*y*	*z*	U_{eq}/U_{iso} (Å^2)
C(2)	2736 (6)	3404 (3)	4116 (3)	413
C(4)	5479 (6)	2194 (4)	2684 (3)	490
C(4*a*)	4922 (5)	2726 (3)	1942 (3)	393
C(5)	5478 (6)	2586 (4)	867 (3)	494
C(7)	3294 (6)	3935 (3)	1529 (3)	442
C(11)	6975 (9)	1757 (6)	−4 (4)	867
C(12)	1828 (7)	4847 (4)	1683 (4)	565
C(13)	179 (13)	4442 (7)	1598 (10)	1197
C(14)	−1253 (13)	5371 (7)	1719 (10)	1236
N(1)	2447 (5)	3998 (3)	3459 (3)	465
N(3)	3955 (5)	2505 (3)	3836 (3)	479
N(6)	4463 (5)	3338 (3)	607 (3)	503
N(7*a*)	3538 (4)	3585 (3)	2356 (2)	385
H(3)	414 (7)	216 (4)	441 (4)	98 (16)
H(4*A*)	6945	2495	2462	93 (14)
H(4*B*)	5523	1286	2594	88 (14)
H(10*A*)	1910	3173	5808	172 (25)
H(10*B*)	−68	3668	5686	278 (44)
H(10*C*)	1890	4592	5435	211 (34)
H(11*A*)	7608	1243	358	166 (25)
H(11*B*)	8174	2215	−717	229 (40)
H(11*C*)	6211	1222	−273	273 (49)
H(12*A*)	2652	5483	1045	172 (26)
H(12*B*)	1188	5219	2504	114 (17)
H(13*A*)	817	4047	787	240 (44)
H(13*B*)	−677	3825	2252	308 (63)
H(14*A*)	−2436	5013	1647	178 (28)
H(14*B*)	−423	5992	1064	162 (33)
H(14*C*)	−1917	5770	2530	220 (39)

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36743 (9 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 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(2)–C(10)	1·520 (5)	C(7)–C(12)	1·508 (5)
C(2)–N(1)	1·286 (5)	C(7)–N(6)	1·321 (4)
C(2)–N(3)	1·340 (5)	C(7)–N(7*a*)	1·357 (5)
C(4)–C(4*a*)	1·488 (5)	C(4*a*)–N(7*a*)	1·367 (4)
C(4)–N(3)	1·451 (5)	C(12)–C(13)	1·454 (7)
C(5)–C(4*a*)	1·350 (5)	C(13)–C(14)	1·514 (8)
C(5)–C(11)	1·512 (6)	N(1)–N(7*a*)	1·400 (4)
C(5)–N(6)	1·377 (5)	N(3)–H(3)	0·99 (6)

N(1)–C(2)–C(10)	116·9 (4)	N(7*a*)–C(4*a*)–C(4)	119·1 (3)
N(3)–C(2)–C(10)	116·1 (4)	N(7*a*)–C(4*a*)–C(5)	105·4 (3)
N(3)–C(2)–N(1)	127·0 (4)	C(13)–C(12)–C(7)	113·8 (4)
N(3)–C(4)–C(4*a*)	108·0 (3)	C(14)–C(13)–C(12)	113·5 (6)
C(11)–C(5)–C(4*a*)	129·0 (4)	N(7*a*)–N(1)–C(2)	112·2 (3)
N(6)–C(5)–C(4*a*)	110·4 (3)	C(4)–N(3)–C(2)	123·6 (4)
N(6)–C(5)–C(11)	120·5 (4)	C(7)–N(6)–C(5)	105·8 (3)
N(6)–C(7)–C(12)	126·1 (4)	C(4*a*)–N(7*a*)–C(7)	108·3 (3)
N(7*a*)–C(7)–C(12)	123·7 (3)	N(1)–N(7*a*)–C(7)	124·9 (3)
N(7*a*)–C(7)–N(6)	110·1 (3)	N(1)–N(7*a*)–C(4*a*)	126·7 (3)
C(5)–C(4*a*)–C(4)	135·5 (3)		

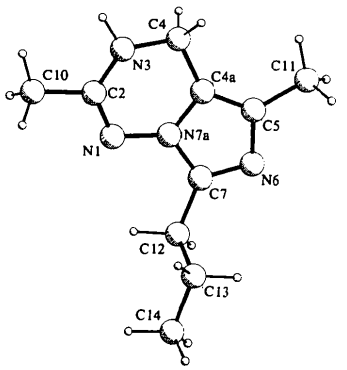

Fig. 1. The principal conformation of the title compound in the crystal.

Discussion. The imidazo[5,1-*f*][1,2,4]triazinone (I) was prepared as a member of a series of biologically active compounds (Charles, Latham, Hartley, Oxford & Scopes, 1980). In order to establish unequivocally the structure of this compound the lactam group was reduced to give the dihydro compound (II) from which crystals were obtained that were suitable for an X-ray crystallographic study.

The structure analysis of (II) shows it to consist of hydrogen-bonded [N(3)···N(6)(x, $\frac{1}{2} - y$, $\frac{1}{2} + z$) = 2·94 (1) Å] molecules, where the propyl group has a perpendicular [N(6)–C(7)–C(12)–C(13) = 63·7 (7), N(7*a*)–C(7)–C(12)–C(13) = −113·7 (7)°]–*trans*-[C(7)–C(12)–C(13)–C(14) = −178·2 (7)°] conformation, very similar to that found in the closely related molecules (III) (Murray-Rust, Murray-Rust & Oxford, 1982) and (IV) (Riley, Heatley, Hillier, Murray-Rust & Murray-Rust, 1979). Inspection of the final difference map suggests that although this is not the only conformation in the crystal it is the most important. Clearly, however, the side chain in the imidazotriazinones can show some flexibility.

The heterocyclic system, being partially reduced, cannot be directly compared with (III) and (IV) but the five-membered ring has virtually identical geometry to that in (III) and (IV).

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