Acta Cryst. (1982). B38, 2692–2693

7-Isobutyl-5-methyl-2-phenylimidazo[5,1-*f*][1,2,4]triazin-4(3*H*)-one

BY PETER MURRAY-RUST AND JUDITH MURRAY-RUST

Department of Chemistry, University of Stirling, Stirling FK9 4LA, Scotland

AND ALEXANDER W. OXFORD

Glaxo Group Research Ltd, Ware, Hertfordshire SG12 0DJ, England

(*Received* 10 *December* 1981; *accepted* 18 *January* 1982)

Abstract. $C_{16}H_{18}N_4O$, M_r = 282·3, monoclinic, $P2_1/n$, a = 15·03 (5), b = 17·40 (5), c = 5·74 (4) Å, β = 93·81 (3)° from diffractometer measurements (Mo $K\bar{a}$ radiation), V = 1498·4 Å^3, Z = 4, D_c = 1·25 Mg m^{-3}, $F(000)$ = 600, μ = 0·047 mm^{-1}, approximate crystal dimensions 0·5 × 0·2 × 0·2 mm. R = 0·056 for 1131 observed reflections. The structure consists of hydrogen-bonded molecules about a centre of symmetry [N(3)···O′(1) = 2·86(1)Å].

Introduction. The title compound (IV) was recrystallized from methanol. Systematic absences (from precession photographs) $h0l$ $h + l$ odd and $0k0$ k odd indicated space group $P2_1/n$. Data were collected for $hk0$–5 with θ_{max} = 25° on a Stoe STADI-2 two-circle diffractometer (graphite-monochromated Mo $K\bar{a}$ radiation). This gave 2314 data of which 1131 unique reflexions with $I > 3\sigma(I)$ were used in subsequent calculations. The structure was solved by direct phasing methods with the *SHELX* 76 crystallographic programs (Sheldrick, 1976), which was used for all calculations. Complex neutral-atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Full-matrix least-squares refinement with unit weights (including isotropic H atoms) converged at R = 0·056 for 1131 observed reflexions ($R = \sum||F_o| - |F_c||/\sum|F_o|$). In the final cycle all shifts in parameters were less than their standard deviations. Positional parameters are given in

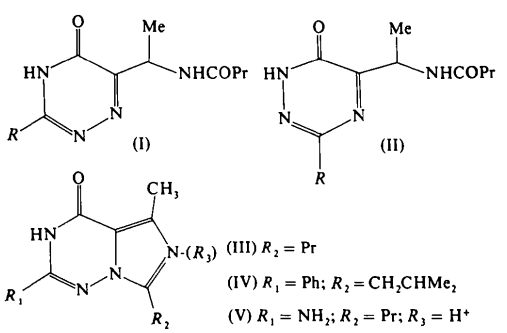

Table 1. *Fractional atomic coordinates and isotropic thermal parameters* (×10^4; *for* H ×10^3)

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 40 (×10^4). For H atoms, T = exp $(-8\pi^2U_{iso}\sin^2\theta/\lambda^2)$.

	x	y	z	U_{eq}/U_{iso} (Å^2)
C(41)	7945 (3)	1455 (3)	3484 (9)	365
C(42)	8001 (3)	961 (3)	1604 (10)	426
C(2)	8648 (3)	1504 (3)	5421 (9)	363
C(4*A*)	9967 (3)	1517 (3)	8938 (10)	376
C(43)	7336 (4)	949 (4)	−187 (11)	501
C(7)	8375 (3)	2568 (3)	10423 (10)	420
C(46)	7200 (4)	1929 (3)	3542 (13)	532
C(44)	6616 (4)	1430 (4)	−120 (12)	559
C(4)	9969 (3)	899 (3)	7327 (9)	415
C(45)	6546 (4)	1913 (4)	1714 (13)	643
C(5)	10442 (3)	1727 (3)	10952 (10)	426
C(10)	8756 (4)	3218 (3)	10743 (12)	462
C(11)	9111 (4)	3997 (4)	10041 (13)	607
C(14)	11244 (5)	1333 (5)	12126 (16)	607
C(13)	8482 (8)	4639 (5)	10815 (20)	858
N(12)	9211 (8)	4039 (6)	7448 (18)	938
N(1)	8624 (3)	2075 (2)	6825 (8)	370
N(7*A*)	9285 (3)	2063 (2)	8626 (7)	372
N(3)	9278 (3)	916 (3)	5613 (8)	416
N(6)	10076 (3)	2376 (3)	11851 (8)	475
O(1)	10521 (3)	370 (2)	7398 (7)	586
H(1)	815 (3)	310 (3)	970 (9)	57 (16)
H(2)	850 (3)	63 (3)	146 (9)	44 (15)
H(3)	871 (6)	510 (6)	1026 (16)	130 (38)
H(4)	711 (3)	223 (3)	493 (10)	44 (16)
H(5)	619 (3)	143 (3)	−139 (4)	39 (14)
H(6)	788 (4)	452 (4)	996 (13)	92 (25)
H(7)	972 (4)	409 (4)	1103 (11)	81 (19)
H(8)	862 (3)	326 (3)	1242 (10)	44 (15)
H(9)	849 (9)	384 (7)	667 (21)	213 (52)
H(10)	742 (4)	59 (3)	−147 (10)	62 (19)
H(11)	1187 (7)	133 (6)	1092 (20)	185 (44)
H(12)	930 (4)	49 (4)	452 (13)	97 (25)
H(13)	610 (4)	224 (3)	173 (11)	67 (20)
H(14)	836 (6)	456 (5)	1260 (20)	166 (43)
H(15)	940 (6)	448 (6)	686 (18)	148 (42)
H(16)	1152 (6)	162 (5)	1303 (17)	123 (36)
H(17)	1122 (5)	85 (5)	1188 (14)	104 (29)
H(18)	961 (7)	364 (6)	686 (17)	162 (43)

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Table 1 and bond distances and angles in Table 2.* The title compound is shown in Fig. 1, with atom numbering.

Discussion. The condensation of the amidrazone, $H_2NC(R)=NNH_2$, with an α-keto ester, MeOOCCOCH(Me)NHCOPr can lead to two possible products (I) and (II) (Domány, Nyitrai, Simig & Lempert, 1977; Charles, Latham, Hartley, Oxford & Scopes, 1980). In all cases examined the predominant component is the triazinone (I) which on cyclization gives the novel imidazo[5,1-*f*][1,2,4]triazinone (III).

We have examined the product when R = Ph and established that it is (IV) by crystallographic analysis. The crystal structure consists of hydrogen-bonded molecules [N(3)···O(1)(2 − *x*, −*y*, 1 − *z*) = 2·86 (1) Å] about a centre of symmetry. The phenyl ring is coplanar with the heterocyclic system.

The heterocyclic nucleus has previously been studied crystallographically (Riley, Heatley, Hillier, Murray-Rust & Murray-Rust, 1979) in (V) where the phenyl group is replaced by $-NH_2$ and N(6) is protonated. The bond lengths are very similar except for two regions of the molecule. In (IV) we find C(2)–N(1) = 1·282, C(2)–N(3) = 1·393 Å [1·337 (3), 1·375 (3) Å in (V)], a variation probably due to the lower electron-donating power of −Ph against $-NH_2$. Near the carbonyl, C(4)–O(1) = 1·237, C(4)–C(4*A*) = 1·419 Å [1·218 (3), 1·452 (3) Å in (V)]. In (IV) there is no hydrogen bonding to O(1) and this would lead to a shortening of the carbonyl bond and a consequential lengthening of the bonds attached to it. It is interesting that the protonation of N(6) in (V) causes no significant change in the C–N(6) bonds [1·334, 1·372 (6) Å in (IV); 1·337 (3), 1·368 (3) Å in (V)].

The side chain is nearly perpendicular to the heterocyclic part [N(6)–C(7)–C(10)–C(11) = 79 (1), N(7*A*)–C(7)–C(10)–C(11) = −103 (1)°], and the isopropyl group has a *trans* [C(7)–C(10)–C(11)–C(12) = −171 (1)°]-*gauche* [C(7)–C(10)–C(11)–C(13) = 66 (1)°] arrangement. The conformation of the *n*-propyl group in (V) is extremely similar, being perpendicular-*trans*.

One of us (JM-R) thanks Glaxo Group Research (Ware) Ltd for financial support.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36696 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(41)–C(42)	1·386 (7)	C(7)–N(6)	1·334 (6)
C(41)–C(2)	1·485 (7)	C(46)–C(45)	1·390 (8)
C(41)–C(46)	1·393 (7)	C(44)–C(45)	1·357 (9)
C(42)–C(43)	1·386 (8)	C(4)–N(3)	1·383 (6)
C(2)–N(1)	1·282 (6)	C(4)–O(1)	1·237 (6)
C(2)–N(3)	1·393 (6)	C(5)–C(14)	1·505 (8)
C(4*A*)–C(4)	1·419 (7)	C(5)–N(6)	1·372 (6)
C(4*A*)–C(5)	1·368 (7)	C(10)–C(11)	1·521 (8)
C(4*A*)–N(7*A*)	1·401 (6)	C(11)–C(13)	1·547 (10)
C(43)–C(44)	1·370 (8)	C(11)–C(12)	1·507 (11)
C(7)–C(10)	1·484 (7)	N(1)–N(7*A*)	1·385 (5)
C(7)–N(7*A*)	1·356 (6)		

C(2)–C(41)–C(42)	122·8 (5)	O(1)–C(4)–C(4*A*)	124·9 (5)
C(46)–C(41)–C(42)	118·4 (5)	O(1)–C(4)–N(3)	120·6 (5)
C(46)–C(41)–C(2)	118·7 (5)	C(44)–C(45)–C(46)	120·8 (7)
C(43)–C(42)–C(41)	120·6 (6)	C(14)–C(5)–C(4*A*)	127·9 (6)
N(1)–C(2)–C(41)	117·9 (5)	N(6)–C(5)–C(4*A*)	109·7 (4)
N(3)–C(2)–C(41)	117·5 (5)	N(6)–C(5)–C(14)	122·3 (5)
N(3)–C(2)–N(1)	124·5 (5)	C(11)–C(10)–C(7)	114·3 (5)
C(5)–C(4*A*)–C(4)	136·9 (5)	C(13)–C(11)–C(10)	109·6 (7)
N(7*A*)–C(4*A*)–C(4)	117·6 (4)	C(12)–C(11)–C(10)	111·3 (6)
N(7*A*)–C(4*A*)–C(5)	105·3 (4)	C(12)–C(11)–C(13)	110·6 (7)
C(44)–C(43)–C(42)	120·2 (6)	N(7*A*)–N(1)–C(2)	114·2 (4)
N(7*A*)–C(7)–C(10)	123·8 (5)	C(7)–N(7*A*)–C(4*A*)	107·9 (4)
N(6)–C(7)–C(10)	126·3 (5)	N(1)–N(7*A*)–C(4*A*)	126·0 (4)
N(6)–C(7)–N(7*A*)	109·9 (4)	N(1)–N(7*A*)–C(7)	126·1 (4)
C(45)–C(46)–C(41)	120·0 (7)	C(4)–N(3)–C(2)	123·1 (5)
C(45)–C(44)–C(43)	120·1 (6)	C(5)–N(6)–C(7)	107·2 (4)
N(3)–C(4)–C(4*A*)	114·5 (5)		

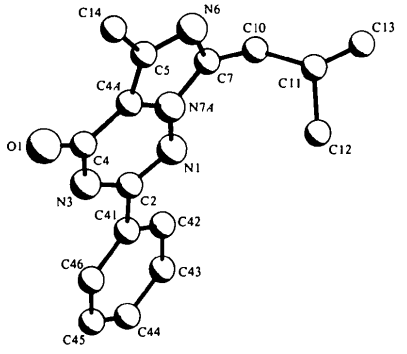

Fig. 1. The title compound (with H atoms omitted).

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