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4-Methylacetophenone [(2-methyl-4-nitro-1*H*-imidazol-1-yl)acetyl]hydrazone

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.065
wR factor = 0.256
Data-to-parameter ratio = 14.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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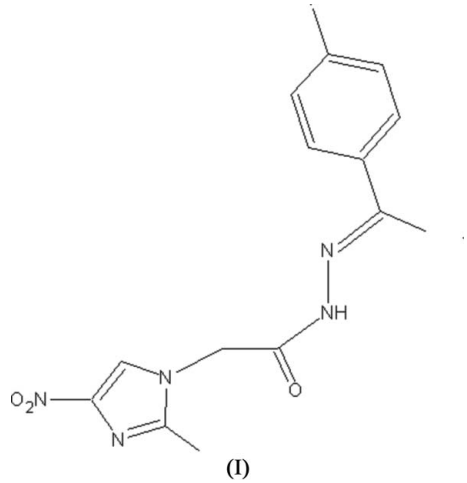
In the title molecule, $C_{15}H_{17}N_5O_3$, the nitro group lies in the plane of the imidazole group, which makes a dihedral angle of 59.9 (2)° with the benzene ring. Intermolecular N—H···O hydrogen bonds link the molecules into centrosymmetric dimers. The crystal packing is further stabilized by weak C—H···O and C—H···N interactions.

Comment

The imidazole nucleus appears in a number of naturally occurring products, among which the most important are the amino acid histidine and the base purine. Imidazole derivatives exhibit a broad spectrum of pharmacological activities, such as anticonvulsant (Verma *et al.*, 1974), anti-Parkinsonian (Naithani *et al.*, 1989) and monoamine oxidase inhibitory (Harfenist *et al.*, 1978) activities. To study the correlation between the effect of acethydrazone group substitution at position 1 of the imidazole ring and its geometry, the crystal structure of the title compound, (I), has been determined.



(I)

In (I) (Fig. 1), all bond lengths and angles (Table 1) are normal. The imidazole group is essentially planar and forms a dihedral angle of 59.9 (2)° with the benzene ring. The nitro group lies in the plane of the imidazole group. In the crystal structure (Fig. 2), intermolecular N—H···O hydrogen bonds (Table 2) link the molecules into centrosymmetric dimers. The packing is further stabilized by weak C—H···O and C—H···N interactions (Table 2).

Experimental

A mixture of 2-methyl-4-nitro-1-imidazoacetohydrazide (19.9 g, 0.1 mol), *p*-methylacetophenone (13.4 g, 0.1 mol) and glacial acetic acid (50 ml) was refluxed for 1 h. The reaction mixture was cooled to

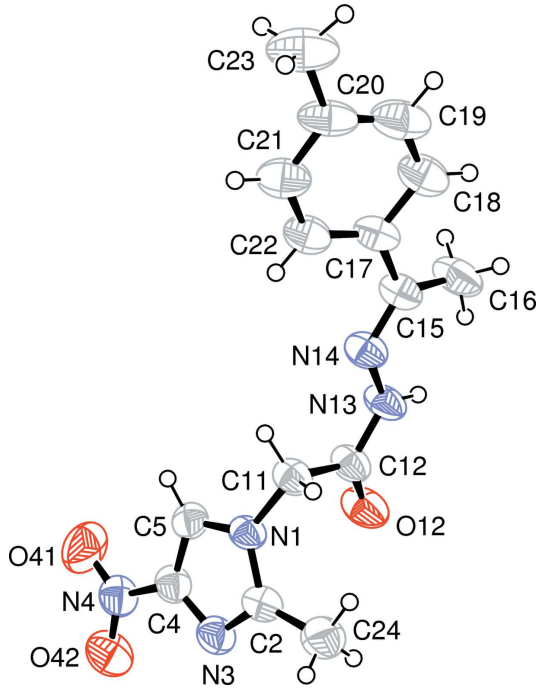


Figure 1
View of (I) showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

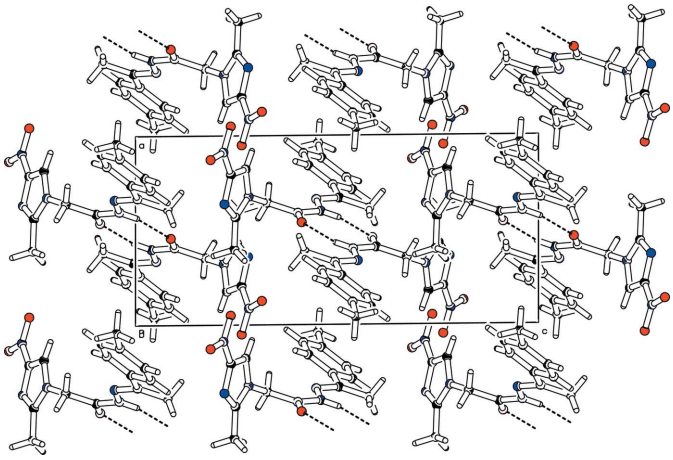


Figure 2
The molecular packing of (I), viewed down the *b* axis, showing the hydrogen-bonded (dashed lines) centrosymmetric dimers.

room temperature and poured into ice-cold water. The solid mass which separated was filtered off, dried and recrystallized from a mixture of ethanol and dimethylformamide (1:1). The yield of the isolated product was 58%.

Crystal data

$C_{15}H_{17}N_5O_3$
M_r = 315.34
Monoclinic, $P2_1/c$
a = 8.423 (2) Å
b = 10.577 (4) Å
c = 17.97 (3) Å
β = 89.46 (8)°
V = 1601 (3) Å^3
Z = 4
D_x = 1.308 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 15–25°
μ = 0.78 mm^{-1}
T = 298 (2) K
Needle, colourless
0.3 × 0.1 × 0.1 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.799, T_{max} = 0.926
3111 measured reflections
2902 independent reflections
2512 reflections with $I > 2\sigma(I)$
R_{int} = 0.066
θ_{max} = 68.0°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 12$
$l = -21 \rightarrow 21$
2 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.256
S = 1.14
2902 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1295P)^2 + 1.2895P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O12—C12	1.245 (4)	N3—C2	1.319 (5)
O41—N4	1.222 (5)	N3—C4	1.345 (5)
O42—N4	1.220 (5)	N4—C4	1.434 (5)
N1—C2	1.369 (5)	N13—N14	1.387 (5)
N1—C5	1.344 (5)	N13—C12	1.342 (5)
N1—C11	1.453 (5)	N14—C15	1.284 (5)
C2—N1—C5	108.4 (3)	N3—C2—C24	126.6 (3)
C2—N1—C11	126.8 (3)	N3—C4—C5	112.3 (3)
C5—N1—C11	124.0 (3)	N4—C4—C5	124.9 (3)
C2—N3—C4	104.9 (3)	N3—C4—N4	122.8 (3)
O41—N4—O42	123.4 (4)	N1—C5—C4	104.3 (3)
O41—N4—C4	117.7 (3)	N1—C11—C12	108.7 (3)
O42—N4—C4	118.9 (3)	O12—C12—N13	120.7 (3)
N14—N13—C12	119.2 (3)	N13—C12—C11	118.3 (3)
N13—N14—C15	116.7 (3)	O12—C12—C11	121.0 (3)
N1—C2—C24	123.3 (3)	N14—C15—C17	114.3 (3)
N1—C2—N3	110.2 (3)	N14—C15—C16	125.9 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N13—H13···O12^{i}	0.86	2.03	2.858 (6)	162
C5—H5···O42^{ii}	0.93	2.54	3.411 (7)	156
C11—H11*A*···N3^{iii}	0.97	2.45	3.397 (7)	165
C11—H11*B*···O41^{ii}	0.97	2.41	3.249 (7)	145
C24—H24*A*···O42^{iii}	0.96	2.58	3.318 (8)	134

Symmetry codes: (i) $-x+1, -y+2, -z$; (ii) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

The H atoms were positioned geometrically and allowed to ride on their parent atoms, with N—H = 0.86 Å, C—H = 0.93–0.97 Å and U_{iso} = 1.2–1.5U_{eq}(parent atom).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Farrugia, 1999); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2004); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

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References

Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2004). *SIR2004*. University of Bari, Italy.

Enraf–Nonius (1989). *CAD-4 Software*. Version 5. Enraf–Nonius, Delft, The Netherlands.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Harfenist, M., Soroko, E. F. & Mekenzie, G. M. (1978). *J. Med. Chem.* **21**, 405–409.

Naithani, P. K., Srivastava, V. K., Barthwal, J. P., Saxena, S. K., Gupta, T. K. & Shanker, K. (1989). *Indian J. Chem. Sect B*, **28**, 299–302.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Verma, M., Chaturvedi, A. K., Chowdhari, A. & Parmar, S. S. (1974). *J. Pharm. Sci.* **63**, 1740–1747.