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3-[(3-Ethyl-1-methyl-4-nitro-1*H*-pyrazol-5-yl)-carbonyl]-1*H*-benzimidazole

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.136
Data-to-parameter ratio = 16.0

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

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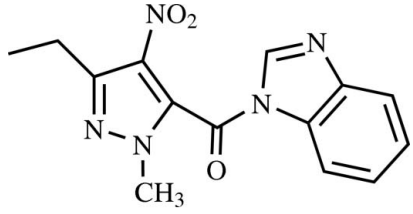
In the title compound, $C_{14}H_{13}N_5O_3$, the dihedral angle formed between the five-membered pyrazole and the benzimidazole ring system is 68.2 (2)°.

Comment

Derivatives of pyrazole have high potential for biological activity, and some of these have been widely used as pesticides and fungicides (Grenda *et al.*, 1965). As a continuation of our work on the structure–activity relationship of pyrazole derivatives, we have obtained a crystalline compound, (I), that was the product of the condensation reaction of 3-ethyl-1-methyl-4-nitropyrazole-5-carboxylic acid chloride and benzimidazole.



(I)

An inspection of the geometric parameters (Table 1) indicates that there is considerable delocalization of π-electron density over the pyrazole ring. However, this delocalization does not extend over the entire molecule; a dihedral angle of 68.7 (2)° is formed between the pyrazole and the central C9/C1/O1/N1 plane; this latter plane and the benzimidazole ring system are essentially coplanar, the dihedral angle between them being 4.2 (2)°.

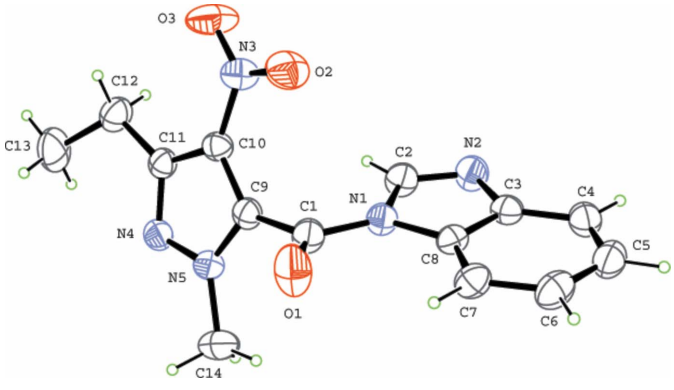


Figure 1
The structure of (I) with 40% probability displacement ellipsoids.

Experimental

3-Ethyl 1-methyl-4-nitropyrazole-5-carboxylic acid chloride (2.17 g, 10 mmol; Okada *et al.*, 1989) was added dropwise to a chloroform solution (30 ml) containing benzimidazole (1.18 g, 10 mmol), prepared according to the procedure of Wagner & Millett (1939), and triethylamine (1.2 g, 12 mmol). The mixture was stirred for 15 h, poured into ice–water and extracted with chloroform. The chloroform layer was washed with an aqueous solution of sodium carbonate, water and a saturated aqueous solution of sodium chloride. After drying over anhydrous sodium sulfate, the solution was concentrated under reduced pressure. The residue was then recrystallized from ethyl acetate to give colorless blocks (m.p. 414–416 K).

Crystal data

$C_{14}H_{13}N_5O_3$
M_r = 299.29
Monoclinic, $P2_1/n$
a = 10.442 (4) Å
b = 7.873 (4) Å
c = 17.380 (5) Å
β = 101.595 (15)°
V = 1399.7 (10) Å^3
Z = 4
D_x = 1.420 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (1) K
Block, colorless
0.42 × 0.38 × 0.30 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.959, T_{max} = 0.969
13220 measured reflections
3200 independent reflections
2413 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.025
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.136
S = 1.01
3200 reflections
200 parameters
H-atom parameters constrained
$w = 1/[0.0021F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—C1	1.1986 (15)	N2—C3	1.4028 (16)
O2—N3	1.2219 (17)	N3—C10	1.4264 (18)
O3—N3	1.2236 (17)	N4—N5	1.3570 (16)
N1—C1	1.3771 (16)	N4—C11	1.3232 (17)
N1—C2	1.3963 (15)	N5—C9	1.3386 (17)
N1—C8	1.4055 (17)	N5—C14	1.454 (2)
N2—C2	1.2851 (17)		

The H atoms were included in the final cycles of refinement in the riding model approximation, with C—H = 0.93, 0.96 and 0.97 Å for aromatic, methyl and methylene H atoms, respectively, and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *PROCESS-AUTO* (Rigaku/MSC, 2004); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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