X-ray Structure Analysis Online

Crystal Structure of 2-(2-Methyl-4-nitro-1*H*-imidazol-1-yl)-*N*′-[(1*E*)-1-phenylethylidene]acetohydrazide

N. Vinutha,*1 Priya V. Frank,*2 Balakrishna Kalluraya,*3 Sumati Anthal,*4 Rajnikant,*4 Vivek K. Gupta,*4† and D. Revannasiddaiah*1

*1 *Department of Studies in Physics, University of Mysore, Manasagangotri, Mysore-570 006, India*
*2 *Department of Chemistry, Canara Engineering College, Mangalore-574 219, India*
*3 *Department of Studies in Chemistry, Mangalore University, Mangalagangotri, Mangalore-574 199, India*
*4 *Post-Graduate Department of Physics & Electronics, University of Jammu, Jammu Tawi-180 006, India*

The title compound, 2-(2-methyl-4-nitro-1*H*-imidazol-1-yl)-*N*′-[(1*E*)-1-phenylethylidene]acetohydrazide, $C_{14}H_{15}N_5O_3$, crystallizes in the monoclinic space group $P2_1/n$ with unit-cell parameters: a = 10.8689(4), b = 10.5644(3), c = 12.9608(4) Å, β = 94.606(3)°, Z = 4. In the molecule, the mean plane of the imidazole ring forms a dihedral angle of 64.46(7)° with the benzene ring. The crystal structure was refined to a final R-value of 0.0474 for 2112 observed reflections. In the crystal, N–H···O intermolecular hydrogen bonds link the molecules into dimmers.

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In view of the obvious importance of naturally occurring imidazoles in biological systems, a vast number of synthetic imidazoles have been prepared as potential pharmacological agents. Imidazole derivatives possess a broad spectrum of pharmacological activities, such as ulcerinhibiting analgesics and anti-inflammatory drugs,[1] for the treatment of asthma, nasal allergy or hives,[2] hypertension and congestive heart failure.[3] Crystal studies on imidazole derivatives have also been described in the literature.[4] Prompted by these observations and in continuation of our research work in the field of biologically active imidazole derivatives, here in we report on the crystal structure of the above mentioned imidazole derivative.

X-ray intensity data of 18720 reflections (of which 2908 unique) were collected at 293(2)K on a *X'calibur* CCD area-detector diffractometer equipped with graphite-monochromated Mo K_α radiation (λ = 0.71073 Å). The crystal used for data collection had dimensions of 0.30 × 0.20 × 0.10 mm. The intensities were measured by the ω scan mode for θ ranges of 3.70 to 26.00°; 2112 reflections were treated as observed ($I > 2\sigma(I)$). Data were corrected for Lorentz and polarization factors. The structure was solved by direct methods using SHELXS97.[5] All non-hydrogen atoms of the molecule were located in the best E-map. A full-matrix least-squares refinement was carried out using SHELXL97.[5] All of the hydrogen atoms were geometrically fixed and allowed to ride on the corresponding non-H atoms with C–H = 0.93 - 0.97 Å, and U_{iso} = 1.5U_{eq} of the attached C atom for methyl H atoms and 1.2U_{eq} for other H atoms. The final refinement cycles converged to R = 0.0474 and wR (F^2) = 0.1148 for the observed 2112 reflections. The residual electron densities ranged from –0.164 to 0.174 eÅ^{-3}. The atomic scattering factors were taken from International Tables for X-ray Crystallography (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4). The crystallographic data are

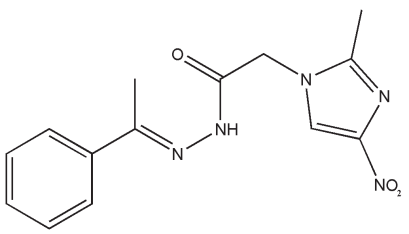

Fig. 1 Structure of 2-(2-methyl-4-nitro-1*H*-imidazol-1-yl)-*N*′-[(1*E*)-1-phenylethylidene]acetohydrazide.

Table 1 Crystal and experimental data

Chemical formula: $C_{14}H_{15}N_5O_3$	
Formula weight: 301.31	
T = 293 K	
Crystal system: monoclinic	Space group: $P2_1/n$
a = 10.8689(4)Å	
b = 10.5644(3)Å	β = 94.606(3)°
c = 12.9608(4)Å	
V = 1483.40(8)Å^3	Z = 4
D_x = 1.349 g/cm^3	D_m (floatation) = 1.354 g/cm^3
Radiation: Mo K_α (λ = 0.71073 Å)	
μ(Mo K_α) = 0.099 mm^{-1}	F(0 0 0) = 632
Crystal size = 0.30 × 0.20 × 0.10 mm	
No. of reflections collected = 18720	
No. of independent reflections = 2908	
θ range for data collection: 3.70 to 26.00°	
Data/Restraints/parameter = 2908/0/201	
Goodness-of-fit on F^2 = 1.014	
R indices [$I > 2\sigma(I)$]: R1 = 0.0474, wR2 = 0.1148	
R indices (all data): R1 = 0.0691, wR2 = 0.1274	
$(\Delta/\sigma)_{max}$ = 0.002 for tors H15A	
$(\Delta\rho)_{max}$ = 0.174 eÅ^{-3}	$(\Delta\rho)_{min}$ = –0.164 eÅ^{-3}
Measurement: *X'calibur system — Oxford diffraction make, U.K.*	
Programs system: SHELXL-97, CRYSALIS RED	
Structure determination: SHELXS-97	
CCDC deposition number: 915023	

† To whom correspondence should be addressed.
E-mail: vivek_gupta2k2@hotmail.com

Table 2 Selected bond lengths (Å), bond angles (°) and torsion angles (°) for non-hydrogen atoms (e.s.d.'s are given in parentheses)

Bond lengths			
N13 - C14	1.280(2)	N13 - N12	1.375(2)
N12 - C11	1.337(2)	N1 - C5	1.352(2)
N1 - C2	1.371(2)	N1 - C10	1.457(2)
O11 - C11	1.219(2)	N3 - C2	1.315(2)
N3 - C4	1.354(2)	C14 - C16	1.481(2)
N7 - O8	1.220(2)	N7 - O9	1.230(2)

Bond angles			
C14 - N13 - N12	118.37(14)	C11 - N12 - N13	119.39(14)
C5 - N1 - C2	107.67(16)	C5 - N1 - C10	125.16(17)
C2 - N1 - C10	126.77(17)	C2 - N3 - C4	104.26(16)
N13 - C14 - C16	114.97(15)	N13 - C14 - C15	125.05(17)
O11 - C11 - N12	122.78(16)	O11 - C11 - C10	121.03(16)
N12 - C11 - C10	116.18(15)	N1 - C5 - C4	104.49(17)
C5 - C4 - N3	112.61(17)	C5 - C4 - N7	125.51(19)
N3 - C4 - N7	121.87(18)	N3 - C2 - N1	110.96(17)
N3 - C2 - C6	125.48(19)	N1 - C2 - C6	123.55(18)
O8 - N7 - O9	123.9(2)	O8 - N7 - C4	119.3(2)
O9 - N7 - C4	116.8(2)	N1 - C10 - C11	109.61(14)

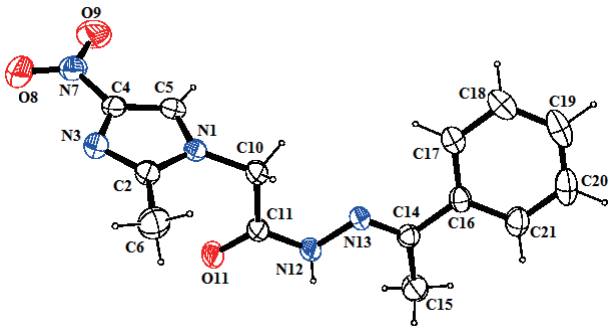

Fig. 2 *ORTEP* view of the molecule showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as small spheres of arbitrary radii.

summarized in Table 1. Selected bond lengths, bond angles and torsion angles are given in Table 2. An ORTEP view of the title compound with atomic labeling is shown in Fig. 2.[6] The geometry of the molecule was calculated using the WinGX[7] and PARST software.[8]

The bond lengths and bond angles are comparable to the expected values.[9] The geometric parameters of the imidazole ring are comparable to that normally found in imidazole and related molecules.[10] The benzene (C16–C21) and imidazole (N1/C2/N3/C4/C5) rings make a dihedral angle of 64.46(7)° with each other. The –C10–C11(=O11)–N12–N13=C14– bridge is nearly planar [maximum deviation = 0.023(2)Å for atom N13], and forms dihedral angles of 74.87(8) and 19.86(7)° with the imidazole and benzene rings, respectively. The nitro (O8/O9/N7) group is coplanar with the imidazole ring, as indicated by the torsion angles O8–N7–C4–N3 [–4.3(3)°] and O9–N7–C4–C5 [–3.7(3)°]. The double-bond C11–O11 is confirmed by its respective distance of 1.219(2)Å. The length of the double-bond C11–O11 is larger than the standard value for the carbonyl group, 1.192 Å, and lengthening of the C11–O11 double bond is due to a strong intermolecular hydrogen bond, N12–H12···O11.

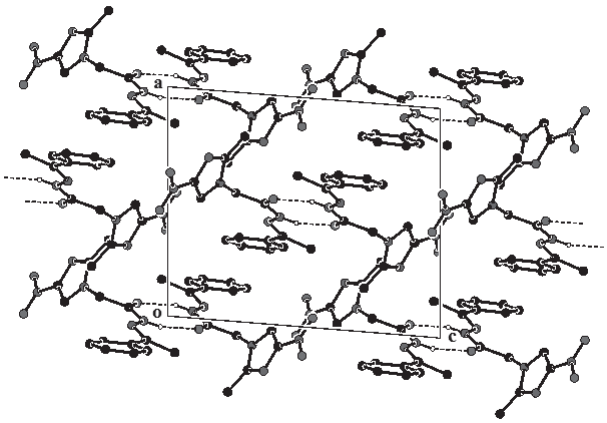

Fig. 3 Molecular packing of the title compound down the *b*-axis.

Table 3 Hydrogen-bonding geometry (e.s.d.'s are given in parentheses)

D-H···A	D-H(Å)	H···A(Å)	D···A(Å)	D-H···A(°)
N12-H12···O11^{i}	0.86	2.02	2.847(2)	160

Symmetry code: (i) –*x*, –*y*+1, –*z*.

A packing view of the molecules in the unit cell viewed down the *b*-axis is shown in Fig. 3. The packing of the title compound involves intermolecular hydrogen bonds. In the crystal, two molecules are connected by N–H···O hydrogen bonds around an inversion center (Table 3).

Acknowledgements

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