organic compounds

1-(2-Methyl-5-nitro-1*H*-imidazol-1-yl)-propan-2-yl acetate

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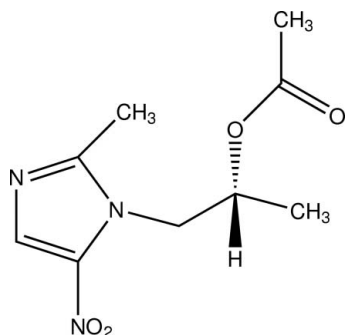Correspondence e-mail: dr.sammer.yousuf@gmail.com

In the title compound, $C_9H_{13}N_3O_4$, an ester of the anti-infection drug secnidazole, the dihedral angle between the nitroimidazole mean plane (r.m.s. deviation = 0.028 Å) and the pendant acetate group is 43.17 (11)°. In the crystal, inversion dimers linked by pairs of C—H⋯O interactions generate $R_2^2(10)$ loops and further C—H⋯O hydrogen bonds link the dimers into [100] chains. Weak aromatic π–π stacking interactions with a centroid–centroid distance of 3.7623 (11) Å are also observed.

Related literature

For background to the antibacterial properties of nitroimidazole and secnidazole-like compounds, see: Mital (2009); Edwards (1993); Crozet *et al.* (2009). For the crystal structures of related compounds, see: Yousuf *et al.* (2013); Tao *et al.* (2008);Zeb *et al.* (2012).

Experimental

Crystal data

$C_9H_{13}N_3O_4$	V = 1131.58 (16) Å³
M_r = 227.22	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 6.1771 (5) Å	μ = 0.11 mm⁻¹
b = 8.9928 (7) Å	T = 273 K
c = 20.3736 (16) Å	0.45 × 0.27 × 0.06 mm
β = 90.978 (2)°	

Data collection

Bruker SMART APEX CCD diffractometer	6541 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	2042 independent reflections
T_{min} = 0.954, T_{max} = 0.994	1567 reflections with $I > 2\sigma(I)$
	R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043	145 parameters
$wR(F^2)$ = 0.119	H-atom parameters constrained
S = 1.02	$\Delta\rho_{max}$ = 0.20 e Å⁻³
2042 reflections	$\Delta\rho_{min}$ = −0.12 e Å⁻³

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C4—H4A⋯O1[i]	0.93	2.45	3.369 (2)	168
C6—H6A⋯O4[ii]	0.97	2.53	3.460 (2)	161

Symmetry codes: (i) $-x + 2, -y + 1, -z$; (ii) $x - 1, y, z$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

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supporting information

1-(2-Methyl-5-nitro-1*H*-imidazol-1-yl)propan-2-yl acetate

Hafiz Abdullah Shahid, Ejaz Hussain, Sajid Jahangir and Sammer Yousuf

S1. Comment

The title compound (I) is an ester derivative of well known 5-nitroimidazole drug i.e secnidazole. The worthwhile use of nitroimidazole derivatives is in the treatment of diseases caused by protozoa and anaerobic bacteria (Mital, 2009). Members of nitroimidazole drugs are pronounced in thier wide-range activities and in addition during their use the rate of resistance in anaerobes is still very low (Edwards, 1993). Antiprotozoal and bactericidal properties of nitroimidazoles are associated with their aromatic nitro group. The Secnidazole like chemotherapeutic agents inhibit the growth of both anaerobic bacteria and some anaerobic protozoa (Crozet *et al.* 2009).

The structure of the title compound (I) is similar to our previously reported compound 1-(2-Methyl-5-nitro-1*H*-imidazol-1-yl)acetone with the difference that acetone moiety is replaced by propyl acetate group (C6—C10/O3,O4) (Yousuf *et al.* 2013);. It also exhibits bond lengths and angles that are of normal range (Yousuf *et al.* 2013); A three dimensional consolidated architecture is formed by the non-covalent interactions of molecules in the crystal *via* C4–H4A···O1 [2.45 Å], and C6– H6A···O4 [2.53 Å] hydrogen bonding with $R_2^2(10)$ ring motifs. Possible weak pi-pi interactions (*Cg*1···*Cg*1) with minimum centroid-centroid distance of 3.7623 (11) Å are also observed.

S2. Experimental

The title compound was synthesized by adding acetic anhydride (1.2 ml, 12.70 mmol)to a hot (70 °C) stired solution of secnidazole (2 g m, 10.8 mmol) in pyridine (2 ml) and toluene (10 ml). The reaction mixture was further processed to refluxed for 5 hrs, cooled, treated with water and then organic phase was evaporated to obtain solid product which was recrystallized from chloroform and toluene solution to yield greenish plates in 81% yield. Melting point 346–348 K. ^{1}H NMR (300 MHz, DMSO-d6): δ 8.006 (s, 1 H, imidazole H), 5.162–5.089 (m, 1 H, CH), 4.573–4.322 (m, 2 H, CH$_2$), 3.300 (s, 3 H, CH$_3$), 1.856 (s, 3 H CH$_3$), 1.265–1.244 (d, J=6.3 Hz, 3 H, CH$_3$). ^{13}C NMR (75 MHz, DMSO-d6): δ 169.35 (C=O), 151.52 (N=C), 138.40 (C—NO$_2$), 133.01 (N—CH), 68.64 (O—CH), 49.31 (N—CH$_2$), 20.44 (CH$_3$), 17.11 (CH$_3$), 13.93 (CH$_3$). IR (neat, cm^{-1}): 3434, 3122, 2994, 1732, 1532, 1368, 1140, 1080.

S3. Refinement

The hydrogen atoms are positioned at their calculated positions geometrically with C—H = 0.9300 Å, 0.9600 Å, 0.9700 Å, 0.9800 Å for aromatic, methyl, methylen, and methin H respectively. These are constrained to ride on their parent atoms during subsequent refinement with U_{iso}(H) = 1.2U_{eq}(C) for methyl, and U_{iso}(H) = 1.5$_{eq}$(C) for rest of the H atoms.
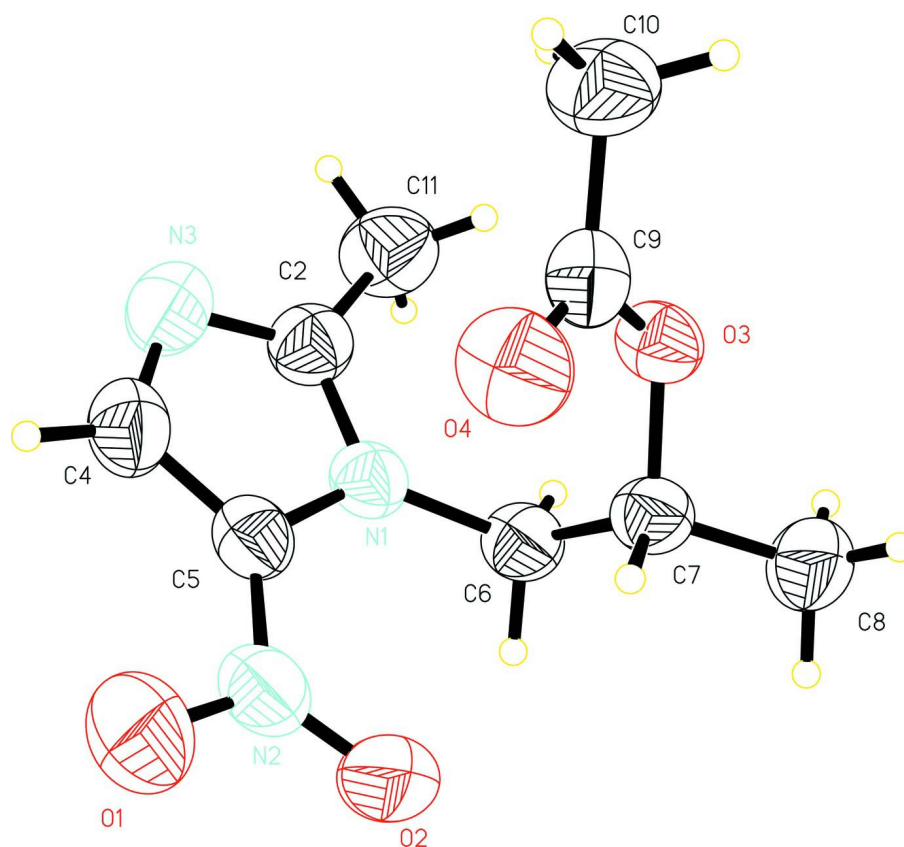
Figure 1

Fig:1 The molecular structure of title compound I, showing displacement ellipsoids drawn at 50% probability level.
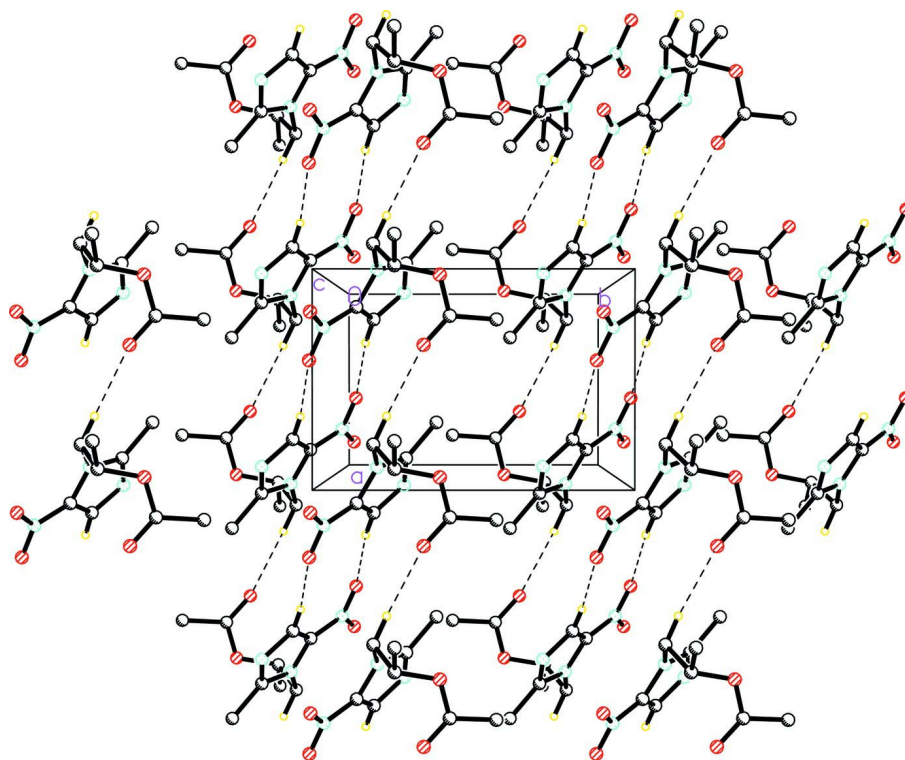
Figure 2

Fig: 2 Crystal packing diagram, showing intermolecular hydrogen bonding as dashed lines.

1-(2-Methyl-5-nitro-1*H*-imidazol-1-yl)propan-2-yl acetate

Crystal data

$C_9H_{13}N_3O_4$	$F(000) = 480$
$M_r = 227.22$	$D_x = 1.334$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 6.1771$ (5) Å	Cell parameters from 1751 reflections
$b = 8.9928$ (7) Å	$\theta = 2.5$–22.3°
$c = 20.3736$ (16) Å	$\mu = 0.11$ mm^{-1}
$\beta = 90.978$ (2)°	$T = 273$ K
$V = 1131.58$ (16) Å^3	Plate, colourless
$Z = 4$	$0.45 \times 0.27 \times 0.06$ mm

Data collection

Bruker SMART APEX CCD diffractometer	6541 measured reflections
	2042 independent reflections
Radiation source: fine-focus sealed tube	1567 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.025$
ω scan	$\theta_{max} = 25.5°$, $\theta_{min} = 2.0°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$h = -7 \rightarrow 7$
	$k = -10 \rightarrow 10$
$T_{min} = 0.954$, $T_{max} = 0.994$	$l = -23 \rightarrow 24$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.043$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.119$	
$S = 1.02$	H-atom parameters constrained
2042 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.1628P]$ where $P = (F_o^2 + 2F_c^2)/3$
145 parameters	
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.20$ e Å^{-3}
	$\Delta\rho_{min} = -0.12$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.9023 (2)	0.57210 (19)	0.08857 (8)	0.0881 (5)
O2	0.6626 (3)	0.55701 (17)	0.16347 (8)	0.0834 (5)
O3	0.48247 (17)	0.11443 (13)	0.18968 (6)	0.0498 (3)
O4	0.8273 (2)	0.17014 (17)	0.16857 (8)	0.0744 (5)
N2	0.7361 (3)	0.51890 (17)	0.11093 (9)	0.0604 (5)
N3	0.5484 (3)	0.2475 (2)	−0.00606 (8)	0.0658 (5)
N1	0.4442 (2)	0.33433 (15)	0.09081 (7)	0.0476 (4)
C5	0.6300 (3)	0.40876 (19)	0.07274 (9)	0.0497 (5)
C4	0.6890 (3)	0.3540 (2)	0.01383 (10)	0.0599 (5)
H4A	0.8088	0.3851	−0.0095	0.072*
C2	0.4036 (3)	0.2373 (2)	0.04109 (10)	0.0552 (5)
C11	0.2199 (3)	0.1309 (3)	0.03950 (12)	0.0744 (6)
H11A	0.2237	0.0738	−0.0003	0.112*
H11B	0.0859	0.1848	0.0412	0.112*
H11C	0.2312	0.0654	0.0766	0.112*
C6	0.3239 (3)	0.3431 (2)	0.15215 (9)	0.0517 (5)
H6A	0.1826	0.2979	0.1455	0.062*
H6B	0.3018	0.4468	0.1633	0.062*
C7	0.4387 (3)	0.26641 (19)	0.20886 (9)	0.0489 (5)
H7A	0.5748	0.3178	0.2193	0.059*
C8	0.2980 (4)	0.2624 (2)	0.26847 (10)	0.0674 (6)
H8A	0.3741	0.2134	0.3038	0.101*
H8B	0.1670	0.2092	0.2583	0.101*
H8C	0.2633	0.3622	0.2814	0.101*
C9	0.6837 (3)	0.0809 (2)	0.17030 (9)	0.0514 (5)

C10	0.7003 (3)	−0.0780 (2)	0.15128 (12)	0.0731 (6)
H10A	0.8452	−0.0989	0.1377	0.110*
H10B	0.6007	−0.0982	0.1157	0.110*
H10C	0.6658	−0.1395	0.1882	0.110*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0725 (10)	0.0951 (12)	0.0972 (12)	−0.0330 (9)	0.0186 (9)	−0.0015 (10)
O2	0.1073 (12)	0.0620 (9)	0.0817 (11)	−0.0251 (8)	0.0283 (10)	−0.0176 (8)
O3	0.0430 (6)	0.0450 (7)	0.0615 (8)	−0.0013 (5)	0.0057 (6)	0.0015 (6)
O4	0.0452 (7)	0.0801 (10)	0.0982 (12)	−0.0049 (7)	0.0122 (7)	0.0021 (9)
N2	0.0624 (10)	0.0496 (9)	0.0693 (12)	−0.0064 (8)	0.0080 (9)	0.0057 (8)
N3	0.0700 (11)	0.0718 (11)	0.0558 (11)	0.0022 (9)	0.0071 (9)	−0.0034 (9)
N1	0.0473 (8)	0.0420 (7)	0.0536 (9)	0.0043 (6)	0.0056 (7)	0.0033 (7)
C5	0.0488 (9)	0.0452 (10)	0.0551 (12)	0.0015 (8)	0.0040 (9)	0.0065 (8)
C4	0.0565 (11)	0.0639 (12)	0.0595 (13)	0.0037 (10)	0.0109 (10)	0.0096 (10)
C2	0.0551 (11)	0.0536 (11)	0.0570 (12)	0.0043 (8)	0.0002 (9)	−0.0004 (9)
C11	0.0676 (13)	0.0759 (14)	0.0794 (16)	−0.0104 (11)	−0.0022 (12)	−0.0129 (12)
C6	0.0461 (9)	0.0476 (10)	0.0618 (12)	0.0042 (8)	0.0119 (9)	−0.0012 (9)
C7	0.0482 (9)	0.0444 (9)	0.0544 (11)	−0.0031 (8)	0.0073 (8)	−0.0037 (8)
C8	0.0717 (13)	0.0701 (13)	0.0609 (13)	−0.0068 (10)	0.0190 (11)	−0.0045 (10)
C9	0.0445 (9)	0.0612 (11)	0.0486 (11)	0.0040 (9)	0.0015 (8)	0.0070 (9)
C10	0.0706 (13)	0.0668 (13)	0.0820 (16)	0.0185 (11)	0.0043 (12)	−0.0038 (12)

Geometric parameters (Å, º)

O1—N2	1.2276 (19)	C11—H11B	0.9600
O2—N2	1.219 (2)	C11—H11C	0.9600
O3—C9	1.3447 (19)	C6—C7	1.512 (3)
O3—C7	1.448 (2)	C6—H6A	0.9700
O4—C9	1.197 (2)	C6—H6B	0.9700
N2—C5	1.414 (2)	C7—C8	1.506 (2)
N3—C2	1.327 (2)	C7—H7A	0.9800
N3—C4	1.351 (3)	C8—H8A	0.9600
N1—C2	1.357 (2)	C8—H8B	0.9600
N1—C5	1.384 (2)	C8—H8C	0.9600
N1—C6	1.467 (2)	C9—C10	1.485 (3)
C5—C4	1.353 (3)	C10—H10A	0.9600
C4—H4A	0.9300	C10—H10B	0.9600
C2—C11	1.484 (3)	C10—H10C	0.9600
C11—H11A	0.9600		
C9—O3—C7	117.93 (13)	C7—C6—H6A	109.0
O2—N2—O1	122.85 (18)	N1—C6—H6B	109.0
O2—N2—C5	120.29 (15)	C7—C6—H6B	109.0
O1—N2—C5	116.86 (17)	H6A—C6—H6B	107.8
C2—N3—C4	105.66 (17)	O3—C7—C8	107.95 (14)

C2—N1—C5	104.86 (14)	O3—C7—C6	108.15 (14)
C2—N1—C6	125.45 (14)	C8—C7—C6	110.95 (15)
C5—N1—C6	129.44 (15)	O3—C7—H7A	109.9
C4—C5—N1	107.28 (17)	C8—C7—H7A	109.9
C4—C5—N2	127.87 (17)	C6—C7—H7A	109.9
N1—C5—N2	124.84 (16)	C7—C8—H8A	109.5
N3—C4—C5	110.04 (17)	C7—C8—H8B	109.5
N3—C4—H4A	125.0	H8A—C8—H8B	109.5
C5—C4—H4A	125.0	C7—C8—H8C	109.5
N3—C2—N1	112.15 (17)	H8A—C8—H8C	109.5
N3—C2—C11	123.62 (19)	H8B—C8—H8C	109.5
N1—C2—C11	124.23 (17)	O4—C9—O3	123.20 (17)
C2—C11—H11A	109.5	O4—C9—C10	125.65 (18)
C2—C11—H11B	109.5	O3—C9—C10	111.14 (16)
H11A—C11—H11B	109.5	C9—C10—H10A	109.5
C2—C11—H11C	109.5	C9—C10—H10B	109.5
H11A—C11—H11C	109.5	H10A—C10—H10B	109.5
H11B—C11—H11C	109.5	C9—C10—H10C	109.5
N1—C6—C7	112.86 (13)	H10A—C10—H10C	109.5
N1—C6—H6A	109.0	H10B—C10—H10C	109.5
C2—N1—C5—C4	0.4 (2)	C5—N1—C2—N3	−0.6 (2)
C6—N1—C5—C4	174.87 (16)	C6—N1—C2—N3	−175.27 (16)
C2—N1—C5—N2	−178.36 (17)	C5—N1—C2—C11	179.00 (18)
C6—N1—C5—N2	−3.9 (3)	C6—N1—C2—C11	4.3 (3)
O2—N2—C5—C4	179.99 (19)	C2—N1—C6—C7	100.0 (2)
O1—N2—C5—C4	−0.1 (3)	C5—N1—C6—C7	−73.4 (2)
O2—N2—C5—N1	−1.5 (3)	C9—O3—C7—C8	−139.19 (17)
O1—N2—C5—N1	178.41 (17)	C9—O3—C7—C6	100.71 (17)
C2—N3—C4—C5	−0.1 (2)	N1—C6—C7—O3	−55.09 (18)
N1—C5—C4—N3	−0.2 (2)	N1—C6—C7—C8	−173.30 (14)
N2—C5—C4—N3	178.55 (18)	C7—O3—C9—O4	0.4 (3)
C4—N3—C2—N1	0.4 (2)	C7—O3—C9—C10	−178.99 (16)
C4—N3—C2—C11	−179.12 (19)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···O1[i]	0.93	2.45	3.369 (2)	168
C6—H6A···O4[ii]	0.97	2.53	3.460 (2)	161

Symmetry codes: (i) −x+2, −y+1, −z; (ii) x−1, y, z.