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4-Acetamido-3-nitrophenyl acetate

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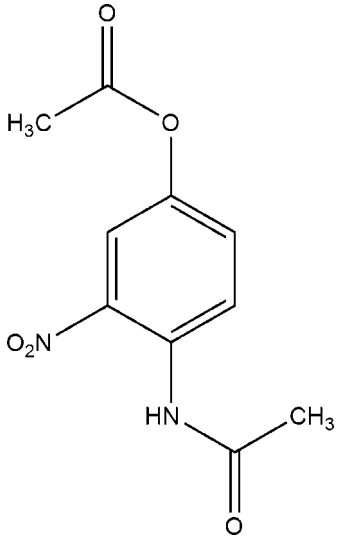
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.064; wR factor = 0.187; data-to-parameter ratio = 14.6.

In the molecule of the title compound, $C_{10}H_{10}N_2O_5$, intramolecular C—H···O interactions result in the formation of a five- and a six-membered ring. The five-membered ring is planar and is oriented at a dihedral angle of 0.34 (3)° with respect to the plane of the aromatic ring, while the six-membered ring has a twist conformation. In the crystal structure, intermolecular C—H···O interactions link the molecules into chains.

Related literature

For a related structure, see: Gu (2007). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{10}H_{10}N_2O_5$
M_r = 238.20
Monoclinic, $C2/c$
a = 24.859 (5) Å
b = 4.7060 (9) Å
c = 19.773 (4) Å
β = 108.67 (3)°
V = 2191.4 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 K
0.30 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.966, T_{max} = 0.988
2039 measured reflections
1992 independent reflections
1310 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.187
S = 1.00
1992 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···O2	0.93	2.33	2.647 (4)	100
C7—H7A···O5	0.93	2.35	2.836 (4)	113
C10—H10C···O5^{i}	0.96	2.59	3.300 (4)	130

Symmetry code: (i) $x, y-1, z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2677).

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supplementary materials

Acta Cryst. (2009). E**65**, o1194 [**doi:10.1107/S1600536809015797**]

4-Acetamido-3-nitrophenyl acetate

Z. Gu and W. Cheng

Comment

The title compound is an important medical intermediate used to synthesize 3,4-diaminophenol, which is the main raw material of luxabendazole (Gu, 2007). We report herein the crystal structure of the title compound, which is of interest to us in the field.

In the molecule of the title compound (Fig 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Ring A (C3-C8) is, of course, planar. Intramolecular C-H···O interactions (Table 1) result in the formations of five- and six-membered rings: B (O2/N2/C4/C5/H4A) and C (O4/O5/C5-C7/C9/H7A). Ring B is planar and it is oriented with respect to ring A at a dihedral angle of 0.34 (3)°, while ring C has a twisted conformation.

In the crystal structure, intermolecular C-H···O interactions (Table 1) link the molecules into chains, in which they may be effective in the stabilization of the structure.

Experimental

The title compound was prepared by the reaction of 4-aminophenol, fuming nitric acid and acetic anhydride (Gu, 2007). Crystals suitable for X-ray analysis were obtained by dissolving the title compound (0.2 g) in ethanol (25 ml) and evaporating the solvent slowly at room temperature for about 2 d.

Refinement

H atoms were positioned geometrically, with N-H = 0.86 Å (for NH) and C-H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C,N)$, where x = 1.5 for methyl H and x = 1.2 for all other H atoms.

Figures

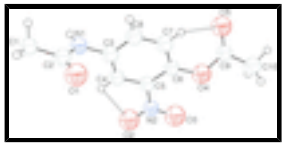

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Hydrogen bonds are shown as dashed lines.

4-Acetamido-3-nitrophenyl acetate

Crystal data

$C_{10}H_{10}N_2O_5$	$F_{000} = 992$
$M_r = 238.20$	$D_x = 1.444$ Mg m^{-3}

Monoclinic, $C2/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 25 reflections
$a = 24.859$ (5) Å	$\theta = 9$–13°
$b = 4.7060$ (9) Å	$\mu = 0.12$ mm^{-1}
$c = 19.773$ (4) Å	$T = 298$ K
$\beta = 108.67$ (3)°	Needle, colorless
$V = 2191.4$ (8) Å^3	0.30 × 0.10 × 0.10 mm
$Z = 8$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.021$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.3^\circ$
Monochromator: graphite	$\theta_{min} = 1.7^\circ$
$T = 298$ K	$h = 0 \rightarrow 29$
$\omega/2\theta$ scans	$k = 0 \rightarrow 5$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = -23 \rightarrow 22$
$T_{min} = 0.966$, $T_{max} = 0.988$	3 standard reflections
2039 measured reflections	every 120 min
1992 independent reflections	intensity decay: 1%
1310 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.064$	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 1.4P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.187$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.00$	$\Delta\rho_{max} = 0.51$ e Å^{-3}
1992 reflections	$\Delta\rho_{min} = -0.56$ e Å^{-3}
136 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008)
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.091 (8)
Secondary atom site location: difference Fourier map	

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}
N1	0.07647 (10)	0.5724 (5)	0.10146 (12)	0.0515 (4)
H1A	0.0516	0.7061	0.0897	0.062*
N2	0.06554 (11)	−0.0326 (5)	−0.09742 (14)	0.0514 (7)
O1	0.12640 (12)	0.2734 (6)	0.18301 (13)	0.0844 (9)
O2	0.02074 (10)	−0.1112 (6)	−0.09213 (14)	0.0857 (9)
O3	0.08434 (11)	−0.1300 (6)	−0.14164 (15)	0.0872 (9)
O4	0.17417 (10)	0.2065 (5)	−0.10210 (12)	0.0688 (7)
O5	0.21944 (11)	0.6153 (5)	−0.11162 (14)	0.0770 (8)
C1	0.06037 (13)	0.5863 (7)	0.21107 (15)	0.0515 (4)
H1B	0.0723	0.5004	0.2576	0.077*
H1C	0.0204	0.5560	0.1888	0.077*
H1D	0.0680	0.7866	0.2156	0.077*
C2	0.09173 (14)	0.4575 (7)	0.16704 (16)	0.0515 (4)
C3	0.10194 (13)	0.4694 (7)	0.05257 (16)	0.0515 (4)
C4	0.07426 (12)	0.2714 (6)	0.00398 (15)	0.0457 (7)
H4A	0.0399	0.1961	0.0048	0.055*
C5	0.09830 (12)	0.1834 (6)	−0.04697 (15)	0.0441 (7)
C6	0.14971 (11)	0.2930 (6)	−0.05065 (15)	0.0427 (7)
C7	0.17661 (12)	0.4918 (6)	0.00190 (17)	0.0512 (8)
H7A	0.2116	0.5645	0.0029	0.061*
C8	0.15321 (13)	0.5825 (6)	0.05171 (16)	0.0514 (8)
H8A	0.1716	0.7196	0.0850	0.062*
C9	0.20627 (12)	0.3744 (6)	−0.13076 (16)	0.0484 (7)
C10	0.22342 (15)	0.2336 (7)	−0.18863 (18)	0.0607 (9)
H10A	0.2458	0.3629	−0.2061	0.091*
H10B	0.1901	0.1808	−0.2270	0.091*
H10C	0.2454	0.0667	−0.1699	0.091*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0594 (9)	0.0497 (9)	0.0486 (8)	0.0021 (7)	0.0217 (7)	0.0021 (7)
N2	0.0494 (14)	0.0471 (15)	0.0587 (16)	−0.0106 (12)	0.0185 (12)	−0.0027 (13)
O1	0.0895 (18)	0.093 (2)	0.0743 (17)	0.0254 (16)	0.0320 (14)	0.0246 (15)
O2	0.0725 (16)	0.093 (2)	0.100 (2)	−0.0430 (15)	0.0402 (15)	−0.0344 (16)
O3	0.0887 (18)	0.095 (2)	0.0935 (19)	−0.0414 (16)	0.0506 (16)	−0.0460 (16)
O4	0.0795 (16)	0.0577 (14)	0.0814 (17)	−0.0073 (12)	0.0426 (14)	−0.0024 (12)
O5	0.109 (2)	0.0442 (14)	0.0964 (19)	−0.0279 (13)	0.0595 (16)	−0.0069 (13)
C1	0.0594 (9)	0.0497 (9)	0.0486 (8)	0.0021 (7)	0.0217 (7)	0.0021 (7)
C2	0.0594 (9)	0.0497 (9)	0.0486 (8)	0.0021 (7)	0.0217 (7)	0.0021 (7)
C3	0.0594 (9)	0.0497 (9)	0.0486 (8)	0.0021 (7)	0.0217 (7)	0.0021 (7)
C4	0.0444 (15)	0.0400 (16)	0.0561 (17)	−0.0079 (13)	0.0207 (13)	−0.0007 (14)

C5	0.0483 (15)	0.0337 (14)	0.0490 (16)	−0.0059 (13)	0.0139 (13)	−0.0002 (13)
C6	0.0463 (15)	0.0332 (14)	0.0508 (16)	−0.0008 (13)	0.0184 (13)	0.0066 (13)
C7	0.0479 (16)	0.0421 (16)	0.0637 (19)	−0.0107 (13)	0.0181 (15)	−0.0017 (15)
C8	0.0601 (18)	0.0422 (17)	0.0498 (17)	−0.0059 (15)	0.0146 (14)	−0.0020 (14)
C9	0.0521 (17)	0.0392 (17)	0.0585 (18)	−0.0001 (14)	0.0239 (14)	0.0058 (14)
C10	0.076 (2)	0.0516 (19)	0.068 (2)	−0.0007 (17)	0.0421 (18)	0.0031 (17)

Geometric parameters (Å, °)

O1—C2	1.191 (4)	C3—C4	1.357 (4)
O4—C9	1.368 (3)	C3—C8	1.386 (4)
O4—C6	1.402 (3)	C4—C5	1.389 (4)
O5—C9	1.207 (3)	C4—H4A	0.9300
N1—C2	1.343 (4)	C5—C6	1.402 (4)
N1—C3	1.401 (4)	C6—C7	1.399 (4)
N1—H1A	0.8600	C7—C8	1.363 (4)
N2—O3	1.206 (3)	C7—H7A	0.9300
N2—O2	1.209 (3)	C8—H8A	0.9300
N2—C5	1.474 (4)	C9—C10	1.497 (4)
C1—C2	1.473 (4)	C10—H10A	0.9600
C1—H1B	0.9600	C10—H10B	0.9600
C1—H1C	0.9600	C10—H10C	0.9600
C1—H1D	0.9600		
C9—O4—C6	125.4 (2)	C4—C5—C6	122.6 (3)
C2—N1—C3	118.5 (3)	C4—C5—N2	115.1 (2)
C2—N1—H1A	120.8	C6—C5—N2	122.3 (3)
C3—N1—H1A	120.8	C7—C6—C5	115.7 (3)
O2—N2—C5	118.5 (3)	C7—C6—O4	121.2 (2)
O3—N2—O2	121.8 (3)	C5—C6—O4	123.0 (3)
O3—N2—C5	119.6 (2)	C8—C7—C6	122.2 (3)
C2—C1—H1B	109.5	C8—C7—H7A	118.9
C2—C1—H1C	109.5	C6—C7—H7A	118.9
H1B—C1—H1C	109.5	C7—C8—C3	119.7 (3)
C2—C1—H1D	109.5	C7—C8—H8A	120.2
H1B—C1—H1D	109.5	C3—C8—H8A	120.2
H1C—C1—H1D	109.5	O5—C9—O4	123.2 (3)
O1—C2—N1	120.4 (3)	O5—C9—C10	122.7 (3)
O1—C2—C1	128.2 (3)	O4—C9—C10	114.1 (3)
N1—C2—C1	111.5 (3)	C9—C10—H10A	109.5
C4—C3—C8	121.0 (3)	C9—C10—H10B	109.5
C4—C3—N1	119.2 (3)	H10A—C10—H10B	109.5
C8—C3—N1	119.7 (3)	C9—C10—H10C	109.5
C3—C4—C5	118.7 (3)	H10A—C10—H10C	109.5
C3—C4—H4A	120.7	H10B—C10—H10C	109.5
C5—C4—H4A	120.7		
C3—N1—C2—O1	0.4 (5)	N2—C5—C6—C7	−178.6 (3)
C3—N1—C2—C1	−179.1 (3)	C4—C5—C6—O4	−179.7 (3)
C2—N1—C3—C4	97.3 (4)	N2—C5—C6—O4	−0.2 (4)
C2—N1—C3—C8	−86.5 (4)	C9—O4—C6—C7	−33.5 (4)

C8—C3—C4—C5	0.0 (5)	C9—O4—C6—C5	148.2 (3)
N1—C3—C4—C5	176.2 (3)	C5—C6—C7—C8	−2.7 (4)
C3—C4—C5—C6	−0.7 (5)	O4—C6—C7—C8	178.9 (3)
C3—C4—C5—N2	179.8 (3)	C6—C7—C8—C3	2.2 (5)
O3—N2—C5—C4	−177.0 (3)	C4—C3—C8—C7	−0.7 (5)
O2—N2—C5—C4	1.6 (4)	N1—C3—C8—C7	−176.9 (3)
O3—N2—C5—C6	3.5 (4)	C6—O4—C9—O5	3.9 (5)
O2—N2—C5—C6	−178.0 (3)	C6—O4—C9—C10	−175.7 (3)
C4—C5—C6—C7	1.9 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4A···O2	0.93	2.33	2.647 (4)	100
C7—H7A···O5	0.93	2.35	2.836 (4)	113
C10—H10C···O5^{i}	0.96	2.59	3.300 (4)	130

Symmetry codes: (i) *x*, *y*−1, *z*.

Fig. 1