

4-Acetamido-3-nitrophenyl acetate

Zhun Gu^a and Wei Cheng^{b*}

^aDepartment of Chemical Engineering, Chien-shiung Institute of Technology, Suzhou 215411, People's Republic of China, and ^bDepartment of Applied Chemistry, College of Science, Nanjing University of Technology, Nanjing 210009, People's Republic of China

Correspondence e-mail: larry_18@163.com

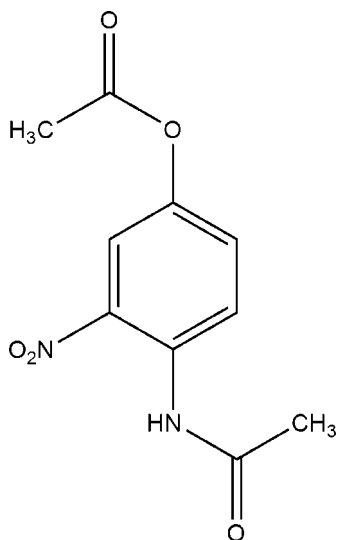
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.064; wR factor = 0.187; data-to-parameter ratio = 14.6.

In the molecule of the title compound, $\text{C}_{10}\text{H}_{10}\text{N}_2\text{O}_5$, intramolecular $\text{C}-\text{H}\cdots\text{O}$ 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 $0.34(3)^\circ$ with respect to the plane of the aromatic ring, while the six-membered ring has a twist conformation. In the crystal structure, intermolecular $\text{C}-\text{H}\cdots\text{O}$ 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

$\text{C}_{10}\text{H}_{10}\text{N}_2\text{O}_5$
 $M_r = 238.20$
 Monoclinic, $C2/c$
 $a = 24.859(5)$ Å
 $b = 4.7060(9)$ Å
 $c = 19.773(4)$ Å
 $\beta = 108.67(3)^\circ$
 $V = 2191.4(8)$ Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.12$ mm⁻¹
 $T = 298$ K
 $0.30 \times 0.10 \times 0.10$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 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, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{C4}-\text{H4A}\cdots\text{O2}$ | 0.93 | 2.33 | 2.647 (4) | 100 |
| $\text{C7}-\text{H7A}\cdots\text{O5}$ | 0.93 | 2.35 | 2.836 (4) | 113 |
| $\text{C10}-\text{H10C}\cdots\text{O5}^i$ | 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

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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 \cdots 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) $^\circ$, while ring C has a twisted conformation.

In the crystal structure, intermolecular C-H \cdots 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 $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C,N})$, where $x = 1.5$ for methyl H and $x = 1.2$ for all other H atoms.

Figures

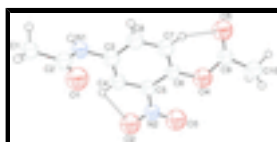


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Hydrogen bonds are shown as dashed lines.

4-Acetamido-3-nitrophenyl acetate

Crystal data

$\text{C}_{10}\text{H}_{10}\text{N}_2\text{O}_5$

$M_r = 238.20$

$F_{000} = 992$

$D_x = 1.444 \text{ Mg m}^{-3}$

supplementary materials

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 24.859\ (5)\ \text{\AA}$

$b = 4.7060\ (9)\ \text{\AA}$

$c = 19.773\ (4)\ \text{\AA}$

$\beta = 108.67\ (3)^\circ$

$V = 2191.4\ (8)\ \text{\AA}^3$

$Z = 8$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 25 reflections

$\theta = 9\text{--}13^\circ$

$\mu = 0.12\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Needle, colorless

$0.30 \times 0.10 \times 0.10\ \text{mm}$

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298\ \text{K}$

$\omega/2\theta$ scans

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 $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 25.3^\circ$

$\theta_{\min} = 1.7^\circ$

$h = 0 \rightarrow 29$

$k = 0 \rightarrow 5$

$l = -23 \rightarrow 22$

3 standard reflections

every 120 min

intensity decay: 1%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.064$

$wR(F^2) = 0.187$

$S = 1.00$

1992 reflections

136 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 1.4P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.51\ \text{e}\ \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.56\ \text{e}\ \text{\AA}^{-3}$

Extinction correction: SHELXL97 (Sheldrick, 2008)

Extinction coefficient: 0.091 (8)

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|-------------|---------------|----------------------------------|
| 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 (\AA^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) |

supplementary materials

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| 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, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C4—H4A \cdots O2 | 0.93 | 2.33 | 2.647 (4) | 100 |
| C7—H7A \cdots O5 | 0.93 | 2.35 | 2.836 (4) | 113 |
| C10—H10C \cdots O5 ⁱ | 0.96 | 2.59 | 3.300 (4) | 130 |

Symmetry codes: (i) *x*, *y*-1, *z*.

Fig. 1

