# organic compounds

# 4-Acetamido-3-nitrophenyl acetate

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In the molecule of the title compound, $C_{10}H_{10}N_2O_5$, intramolecular C—H$\cdots$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$\cdots$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).
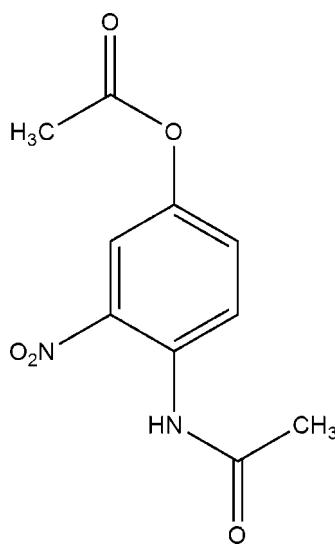


## Experimental

### Crystal data

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

### Data collection

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

### Refinement

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

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

| $D$—H$\cdots$A | $D$—H | H$\cdots$A | $D\cdots$A | $D$—H$\cdots$A |
|---|---|---|---|---|
| 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 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*.

## References

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Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
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# supporting information

## 4-Acetamido-3-nitrophenyl acetate

### Zhun Gu and Wei Cheng

### S1. 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.

### S2. 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.

### S3. 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.
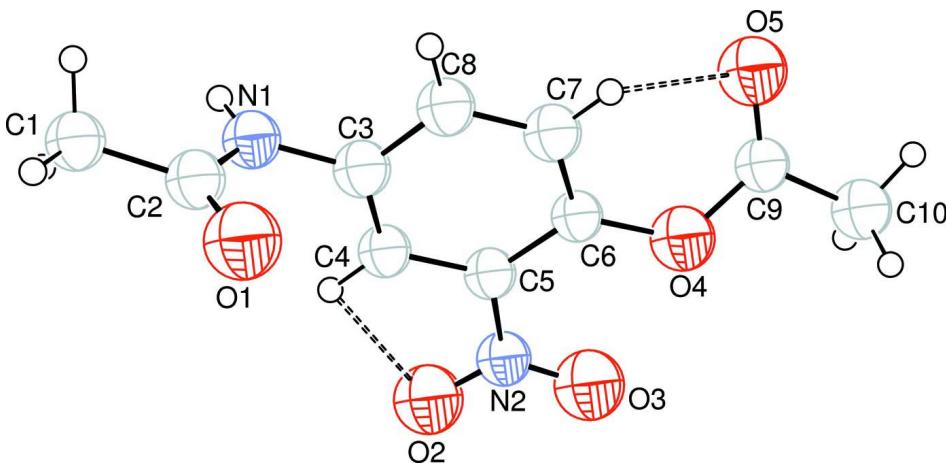
**Figure 1**

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$^3$ | $0.30 \times 0.10 \times 0.10$ mm |
| $Z = 8$ | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | 1992 independent reflections |
| Radiation source: fine-focus sealed tube | 1310 reflections with $I > 2\sigma(I)$ |
| Graphite monochromator | $R_{int} = 0.021$ |
| $\omega/2\theta$ scans | $\theta_{max} = 25.3°$, $\theta_{min} = 1.7°$ |
| Absorption correction: $\psi$ scan (North *et al.*, 1968) | $h = 0\rightarrow29$ |
| | $k = 0\rightarrow5$ |
| $T_{min} = 0.966$, $T_{max} = 0.988$ | $l = -23\rightarrow22$ |
| 2039 measured reflections | 3 standard reflections every 120 min |
| | intensity decay: 1% |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.064$ | |
| $wR(F^2) = 0.187$ | Hydrogen site location: inferred from neighbouring sites |
| $S = 1.00$ | |
| 1992 reflections | H-atom parameters constrained |
| 136 parameters | $w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 1.4P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 0 restraints | |

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

$\Delta\rho_{max} = 0.51$ e Å$^{-3}$

$\Delta\rho_{min} = -0.56$ e Å$^{-3}$

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 (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, º)

| | | | |
|---|---|---|---|
| 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, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4—H4*A*···O2 | 0.93 | 2.33 | 2.647 (4) | 100 |
| C7—H7*A*···O5 | 0.93 | 2.35 | 2.836 (4) | 113 |
| C10—H10*C*···O5[i] | 0.96 | 2.59 | 3.300 (4) | 130 |

Symmetry code: (i) *x*, *y*−1, *z*.