# Crystal structure of *N*-(2,4-dimethyl-6-nitrophenyl)acetamide, $C_{10}H_{12}N_2O_3$

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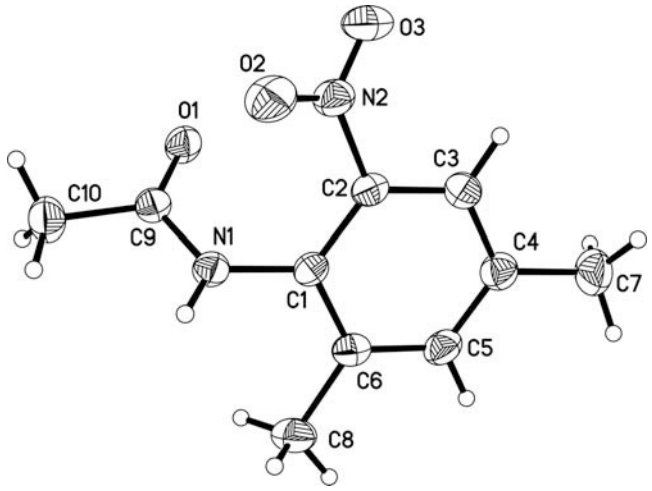


**Abstract**

$C_{10}H_{12}N_2O_3$, tetragonal, $P4_3$ (no. 78), $a$ = 8.4653(8) Å, $c$ = 14.904(1) Å, $V$ = 1068.0 Å$^3$, $Z$ = 4, $R_{gt}(F)$ = 0.031, $wR_{ref}(F^2)$ = 0.085, $T$ = 298 K.

**Source of material**

A solution of 2,4-dimethylacetanilide (24.2 g, 0.2 mol), acetic acid (23 mL) and acetic anhydride (19 mL) was refluxed for 1 h and cooled to 35 °C. Then, the mixed solution of concentrated sulfuric acid (35 mL) and concentrated nitric acid (17 mL) was slowly dropped into it after concentrated sulfuric acids (40 mL) was added. The mixture was reacted for 1 h and cooled to the room tempreture, and was added to the cooled water and colorless precipitate appeared. The precipitate was filtered and washed with cooled water until the pH value of the filtrate is 7. The precipitate was recrystallized from ethanol to afford the title compound (38.5 g) in good yield (92.5 %, mp. 172 - 174 °C).

**Experimental details**

H atoms were positioned geometrically, with $d$(N—H) = 0.86 Å and $d$(C—H) = 0.93 (aromatic), 0.96 Å (methyl), respectively, and constrained to ride on their parent atoms, with $U_{iso}$(H) = 1.2 $U_{eq}$(N) and $U_{iso}$(H) = $x$ $U_{eq}$(C), where $x$ = 1.2 for aromatic H and $x$ = 1.5 for methyl H atoms. The Flack parameter –10(10) has no meaning for the absolute structure.

**Discussion**

2,4-dimethyl-6-nitroacetanide act as a very important intermediate of dyes and pigments [1-3]. It is of practical significance for research and develop 2,4-dimethyl-6-nitroacetanide because of difficult synthesis process, higher costs and low yield, but in great demand [4].

The crystal structure of the title compound is built up by only the $C_{10}H_{12}N_2O_3$ molecules. All bond lengths and angles are in normal ranges. The dihedral angle between the phenyl ring and the nitryl plane is 34.61(1)° (O2–N2–O3), the torsion angle of C1–N1–C9–C10 is 177.2(2)°, the phenyl ring is nearly planar and the mean deviation of the atoms from the least-square plane is 0.009 Å. The substituent groups are slightly twisted out of benzene mean plane, atoms N1, C7 deviate from phenyl ring by 0.102 Å and 0.049 Å, whereas N2, C8 by 0.099 Å and 0.033 Å on the opposite side, respectively. There are intermolecular hydrogen bonds in the complex: N1–H1···O1 ($x$+1½,–$y$–½, $z$–1), C3–H3···O1 ($x$+1½,½–$y$,$z$–1), C10–H10B···O1 ($x$+1½,$y$–½,$z$–1), C10–H10C···O3 ($x$ +1½,–$y$–½,$z$–1) and C5–H5···O2 ($x$,$y$–1,$z$). These hydrogen bonds play an very important role in the formation, stability and crystallization of the title compound.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | colorless block, size 0.40 × 0.47 × 0.49 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.97 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker SMART CCD, $\varphi/\omega$ |
| $2\theta_{max}$: | 50° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 4816, 985 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 859 |
| $N(param)_{refined}$: | 137 |
| Programs: | SHELXS-97, SHELXL-97, SHELXTL [6] |

**Table 2.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1) | 4*a* | 0.9681 | 0.2433 | 0.3733 | 0.053 |
| H(3) | 4*a* | 0.3804 | 0.3670 | 0.3869 | 0.060 |
| H(5) | 4*a* | 0.6929 | 0.7191 | 0.3896 | 0.063 |
| H(7A) | 4*a* | 0.4259 | 0.7783 | 0.4268 | 0.114 |
| H(7B) | 4*a* | 0.3078 | 0.6393 | 0.4450 | 0.114 |
| H(7C) | 4*a* | 0.3324 | 0.7020 | 0.3470 | 0.114 |
| H(8A) | 4*a* | 0.9630 | 0.6813 | 0.3638 | 0.095 |
| H(8B) | 4*a* | 1.0102 | 0.5306 | 0.3088 | 0.095 |
| H(8C) | 4*a* | 1.0214 | 0.5293 | 0.4138 | 0.095 |
| H(10A) | 4*a* | 1.0087 | –0.0571 | 0.2477 | 0.095 |
| H(10B) | 4*a* | 1.1030 | 0.0660 | 0.3052 | 0.095 |
| H(10C) | 4*a* | 1.0892 | 0.0880 | 0.2010 | 0.095 |

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**Table 3.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| N(1) | 4*a* | 0.8872(3) | 0.2443(3) | 0.3384(2) | 0.045(1) | 0.046(1) | 0.042(1) | 0.002(1) | –0.008(1) | –0.005(1) |
| N(2) | 4*a* | 0.5637(3) | 0.1314(3) | 0.3662(2) | 0.060(2) | 0.044(1) | 0.057(2) | –0.009(1) | 0.008(1) | –0.001(1) |
| O(1) | 4*a* | 0.7755(2) | 0.1387(2) | 0.2151(1) | 0.054(1) | 0.058(1) | 0.046(1) | 0.0046(9) | –0.0078(9) | –0.0138(9) |
| O(2) | 4*a* | 0.6554(3) | 0.0359(2) | 0.3985(2) | 0.083(2) | 0.043(1) | 0.089(2) | –0.002(1) | –0.001(1) | 0.015(1) |
| O(3) | 4*a* | 0.4357(3) | 0.0951(3) | 0.3350(2) | 0.071(2) | 0.058(1) | 0.113(2) | –0.021(1) | –0.006(2) | –0.011(1) |
| C(1) | 4*a* | 0.7614(3) | 0.3477(3) | 0.3593(2) | 0.046(1) | 0.042(1) | 0.031(1) | –0.001(1) | –0.002(1) | –0.004(1) |
| C(2) | 4*a* | 0.6055(3) | 0.2996(3) | 0.3682(2) | 0.051(2) | 0.039(1) | 0.038(1) | –0.007(1) | 0.002(1) | –0.002(1) |
| C(3) | 4*a* | 0.4836(3) | 0.4038(3) | 0.3824(2) | 0.049(2) | 0.048(2) | 0.053(2) | –0.005(1) | 0.006(1) | 0.000(1) |
| C(4) | 4*a* | 0.5149(4) | 0.5644(3) | 0.3898(2) | 0.057(2) | 0.045(2) | 0.058(2) | –0.000(1) | 0.003(1) | 0.000(1) |
| C(5) | 4*a* | 0.6703(3) | 0.6121(3) | 0.3836(2) | 0.064(2) | 0.035(1) | 0.059(2) | –0.004(1) | 0.003(2) | –0.005(1) |
| C(6) | 4*a* | 0.7942(3) | 0.5090(3) | 0.3689(2) | 0.051(2) | 0.044(1) | 0.044(2) | –0.008(1) | –0.002(1) | –0.000(1) |
| C(7) | 4*a* | 0.3833(4) | 0.6816(4) | 0.4034(3) | 0.068(2) | 0.060(2) | 0.100(3) | 0.013(2) | 0.014(2) | –0.004(2) |
| C(8) | 4*a* | 0.9626(4) | 0.5679(4) | 0.3633(3) | 0.059(2) | 0.057(2) | 0.074(2) | –0.015(1) | –0.007(2) | –0.003(2) |
| C(9) | 4*a* | 0.8873(3) | 0.1474(3) | 0.2669(2) | 0.046(2) | 0.043(1) | 0.040(1) | –0.001(1) | –0.001(1) | –0.000(1) |
| C(10) | 4*a* | 1.0354(4) | 0.0525(4) | 0.2540(2) | 0.055(2) | 0.074(2) | 0.061(2) | 0.014(2) | –0.002(2) | –0.013(2) |

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