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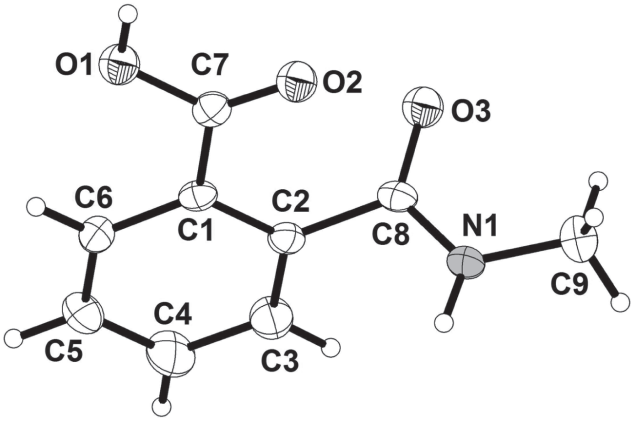
# A single crystal study on 2-(methylcarbamoyl) benzoic acid, $C_9H_9NO_3$

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## Abstract

$C_9H_9NO_3$, monoclinic, $P2_1/c$ (no. 14), $a = 8.7201(8)$ Å, $b = 8.6453(9)$ Å, $c = 11.8083(9)$ Å, $\beta = 100.830(3)°$, $V = 874.35(14)$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.0489$, $wR_{ref}(F^2) = 0.1142$, $T = 296(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

## Source of materials

The title compound, can be synthesized according to the literature [5]. In a typical reaction 0.5 g of the title compound was dissolved in 8 mL methanol. Evaporation at room temperature yielded colorless crystals of 2-(methylcarbamoyl)benzoic acid.

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

| | |
|---|---|
| Crystal: | Colourless block |
| Size: | 0.28 × 0.21 × 0.17 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 1.0 cm$^{-1}$ |
| Diffractometer, scan mode: | PHOTON100 CMOS, $\omega$ scans |
| $2\theta_{max}$, completeness: | 50.8°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 7023, 1603, 0.072 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 1127 |
| $N(param)_{refined}$: | 125 |
| Programs: | CAD-4 [1, 2], SHELX [3, 4] |

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$).

| Atom | x | y | z | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.7659(2) | 0.9801(2) | 0.65405(17) | 0.0178(5) |
| C2 | 0.7244(2) | 0.9605(2) | 0.76231(17) | 0.0188(5) |
| C3 | 0.8129(3) | 0.8626(3) | 0.84236(19) | 0.0292(6) |
| H3 | 0.7840 | 0.8476 | 0.9152 | 0.035* |
| C4 | 0.9419(3) | 0.7869(3) | 0.81791(19) | 0.0348(6) |
| H4 | 1.0025 | 0.7225 | 0.8745 | 0.042* |
| C5 | 0.9830(3) | 0.8044(3) | 0.71166(19) | 0.0299(6) |
| H5 | 1.0710 | 0.7511 | 0.6943 | 0.036* |
| C6 | 0.8954(2) | 0.9003(3) | 0.63019(19) | 0.0233(5) |
| H6 | 0.9238 | 0.9119 | 0.5569 | 0.028* |
| C7 | 0.6744(2) | 1.0860(2) | 0.56710(17) | 0.0194(5) |
| C8 | 0.5960(2) | 1.0504(2) | 0.80081(16) | 0.0175(5) |
| C9 | 0.3470(3) | 1.0341(3) | 0.8655(2) | 0.0357(6) |
| H9A | 0.3798 | 1.1326 | 0.9036 | 0.054* |
| H9B | 0.3171 | 0.9621 | 0.9217 | 0.054* |
| H9C | 0.2576 | 1.0516 | 0.8030 | 0.054* |
| N1 | 0.4755(2) | 0.9687(2) | 0.81858(15) | 0.0230(5) |
| H1B | 0.467(3) | 0.871(3) | 0.7987(19) | 0.028* |
| O1 | 0.74698(18) | 1.1135(2) | 0.47935(13) | 0.0296(4) |
| H1A | 0.692(3) | 1.175(3) | 0.428(2) | 0.036* |
| O2 | 0.55076(17) | 1.14259(18) | 0.57593(11) | 0.0247(4) |
| O3 | 0.61125(16) | 1.19134(17) | 0.82207(11) | 0.0238(4) |

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## Experimental details

All hydrogen atoms were identified in difference Fourier syntheses and either placed on calculated positions with the help of the SHELXL program (AFIX 43 or 137 option

for alkyl and aromatic hydrogens) [3] or freely refined with $U_{iso}(H) = 1.2U_{eq}(N,O)$.

## Comment

The compound we report here is a very important impurity in the production of milnacipran hydrochloride. Milnacipran hydrochloride is a serotonin-norepinephrine reuptake inhibitor (SNRI) used in the clinical treatment of fibromyalgia that was first approved for the treatment of major depressive episodes in France in December 1996. The bond lengths and angles in the crystal structure are within normal ranges [6]. The carboxyl (C7/O1/O2) and acylamide (C8/O3/N1/C9) groups are oriented at 11.3(3)° and 70.6(4)° with respect to the benzene ring (C1/C2/C3/C4/C5/C6) to which they are bonded. In the crystal of the title compound there are O—H···O and N—H···O intermolecular bydrogen bonds, but no π-π stacking interactions, as the centroid to centroid distance between the two nearest rings is larger than 4 Å.

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