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## Structure Reports

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# Pyrimidine-4-carboxylic acid

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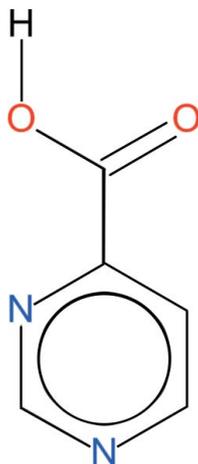
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 Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.048;  $wR$  factor = 0.124; data-to-parameter ratio = 9.4.

The crystal structure of the title compound,  $\text{C}_5\text{H}_4\text{N}_2\text{O}_2$ , is built of acid molecules located on a mirror plane. They form sheets stacked along the  $b$ -axis direction. The molecules interact *via*  $\text{O}-\text{H}\cdots\text{N}$  hydrogen bonds, forming [001] chains, and weak van der Waals interactions.

## Related literature

For the structure of a Li complex with pyrimidine-4-carboxylate and aqua ligands, see: Starosta & Leciejewicz (2012).



## Experimental

### Crystal data

$\text{C}_5\text{H}_4\text{N}_2\text{O}_2$	$V = 264.41$ (9) Å <sup>3</sup>
$M_r = 124.10$	$Z = 2$
Monoclinic, $P2_1/m$	Mo $K\alpha$ radiation
$a = 6.0080$ (12) Å	$\mu = 0.12$ mm <sup>-1</sup>
$b = 6.3519$ (13) Å	$T = 293$ K
$c = 7.4834$ (15) Å	$0.17 \times 0.16 \times 0.06$ mm
$\beta = 112.20$ (3)°	

### Data collection

Kuma KM-4 four-circle diffractometer	545 independent reflections
Absorption correction: analytical ( <i>CrysAlis RED</i> ; Oxford Diffraction, 2008)	349 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.973$ , $T_{\max} = 0.994$	$R_{\text{int}} = 0.129$
1981 measured reflections	3 standard reflections every 200 reflections
	intensity decay: 0.9%

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.124$	$\Delta\rho_{\text{max}} = 0.15$ e Å <sup>-3</sup>
$S = 1.00$	$\Delta\rho_{\text{min}} = -0.34$ e Å <sup>-3</sup>
545 reflections	
58 parameters	

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}^i$	1.04 (4)	1.62 (4)	2.660 (3)	179 (3)

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

Data collection: *KM-4 Software* (Kuma, 1996); cell refinement: *KM-4 Software*; data reduction: *DATAPROC* (Kuma, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6903).

## References

- Kuma (1996). *KM-4 Software*. Kuma Diffraction Ltd, Wrocław, Poland.  
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 Oxford Diffraction (2008). *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Starosta, W. & Leciejewicz, J. (2012). *Acta Cryst.* **E68**, m1065–m1066.

## supporting information

*Acta Cryst.* (2013). E69, o885 [doi:10.1107/S1600536813012610]

## Pyrimidine-4-carboxylic acid

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### S1. Comment

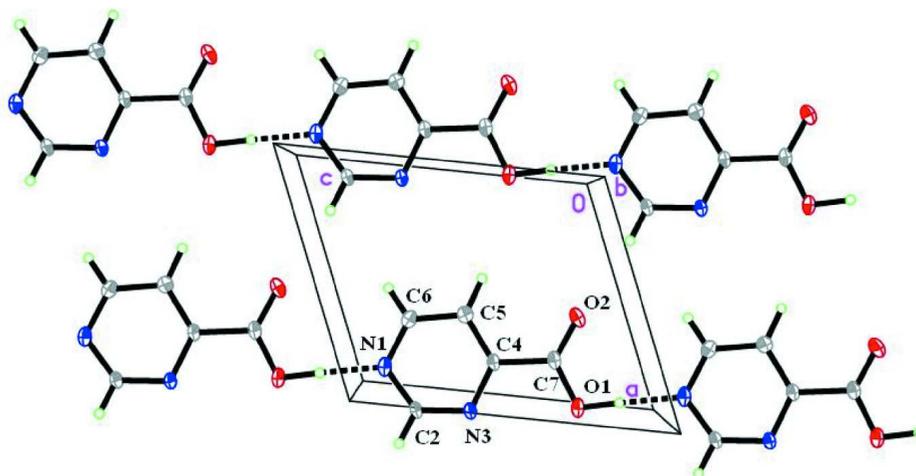
The monoclinic structure of pyrimidine-4-carboxylic acid  $C_5H_4N_2O_2$  is composed of molecular sheets stacked along [010] crystal direction (Fig.1). Within a sheet, hetero-ring and carboxylate group atoms are coplanar. Acid molecules interact *via* hydrogen bonds of 2.658 (3) Å in which protonated carboxylate O atoms are as donors and hetero-ring N atoms in adjacent acid molecules act as acceptors. The C—C and C—N bond distances and bond angles within the acid molecule do not differ from those reported earlier in the structure of the Li complex with the title acid (Starosta & Leciejewicz, 2012). The sheets are held together by van der Waals interactions as indicated by the distance between adjacent sheets which is 3.171 (1) Å.

### S2. Experimental

75 ml of a hot (*ca* 350 K) aqueous solution containing 31.6 mmol of potassium permanganate was added dropwise during 3 h to 8 ml of stirred aqueous solution containing 21.3 mmol of 4-methylpyrimidine and 5 mmol of NaOH. After stirring for half an hour longer, 1 ml of methanol was added to decompose the excess of potassium permanganate. The hot solution was filtered and the solid washed twice with 5 ml of water. Then, the filtrate and the washings were concentrated to *ca* 15 ml and acidified to pH 2–3 with concentrated HCl. After cooling to room temperature the precipitate containing 10.5 mmol of crude pyrimidine-4-carboxylic acid was recrystallized from a mixture of water and methanol taken in 20:1 ratio to give 1.1 g. (8.9 mmol) of colourless crystal blocks of the title acid (yield 42%, m.p. 508–509 K).

### S3. Refinement

The hydrogen atom attached to the carboxylic group was located in a difference map and refined isotropically, while the three H atoms attached to pyrimidine C atoms were located at a calculated positions and treated as riding on the parent atoms with C—H=0.93 Å.

**Figure 1**

Molecules of the title compound with atom labelling scheme and 50% probability displacement ellipsoids viewed along the *b* axis.

### Pyrimidine-4-carboxylic acid

#### Crystal data

$C_5H_4N_2O_2$

$M_r = 124.10$

Monoclinic,  $P2_1/m$

Hall symbol:  $-P\ 2y\ b$

$a = 6.0080$  (12) Å

$b = 6.3519$  (13) Å

$c = 7.4834$  (15) Å

$\beta = 112.20$  (3)°

$V = 264.41$  (9) Å<sup>3</sup>

$Z = 2$

$F(000) = 128$

$D_x = 1.559$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 25 reflections

$\theta = 6\text{--}15^\circ$

$\mu = 0.12$  mm<sup>-1</sup>

$T = 293$  K

Plate, colourless

$0.17 \times 0.16 \times 0.06$  mm

#### Data collection

Kuma KM-4 four-circle  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator  
profile data from  $\omega/2\theta$  scan

Absorption correction: analytical  
(*CrysAlis RED*; Oxford Diffraction, 2008)

$T_{\min} = 0.973$ ,  $T_{\max} = 0.994$

1981 measured reflections

545 independent reflections

349 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.129$

$\theta_{\max} = 25.7^\circ$ ,  $\theta_{\min} = 2.9^\circ$

$h = -7 \rightarrow 7$

$k = -7 \rightarrow 7$

$l = -9 \rightarrow 9$

3 standard reflections every 200 reflections

intensity decay: 0.9%

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.124$

$S = 1.00$

545 reflections

58 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H atoms treated by a mixture of independent  
and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0689P)^2]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.15 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.34 \text{ e } \text{\AA}^{-3}$$

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	1.0036 (4)	0.2500	0.2735 (3)	0.0416 (6)
C4	0.8398 (5)	0.2500	0.5171 (4)	0.0308 (7)
N1	0.9072 (4)	0.2500	0.8961 (3)	0.0397 (7)
N3	1.0647 (4)	0.2500	0.6476 (3)	0.0371 (7)
O2	0.6049 (4)	0.2500	0.1835 (3)	0.0649 (8)
C7	0.8036 (5)	0.2500	0.3063 (4)	0.0364 (7)
C5	0.6416 (5)	0.2500	0.5672 (4)	0.0403 (8)
H5	0.4860	0.2500	0.4747	0.048*
C6	0.6868 (5)	0.2500	0.7631 (4)	0.0448 (9)
H6	0.5573	0.2500	0.8024	0.054*
C2	1.0847 (5)	0.2500	0.8311 (4)	0.0401 (8)
H4	1.2401	0.2500	0.9240	0.048*
H1	0.967 (6)	0.2500	0.126 (5)	0.064 (11)*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0403 (11)	0.0677 (13)	0.0196 (9)	0.000	0.0145 (8)	0.000
C4	0.0308 (14)	0.0389 (14)	0.0225 (13)	0.000	0.0097 (11)	0.000
N1	0.0401 (13)	0.0606 (16)	0.0208 (11)	0.000	0.0142 (11)	0.000
N3	0.0276 (11)	0.0651 (16)	0.0173 (12)	0.000	0.0071 (9)	0.000
O2	0.0364 (12)	0.130 (2)	0.0206 (10)	0.000	0.0020 (9)	0.000
C7	0.0366 (15)	0.0507 (16)	0.0217 (13)	0.000	0.0109 (12)	0.000
C5	0.0287 (14)	0.0613 (19)	0.0293 (15)	0.000	0.0093 (13)	0.000
C6	0.0325 (14)	0.072 (2)	0.0342 (15)	0.000	0.0179 (12)	0.000
C2	0.0304 (13)	0.0718 (19)	0.0160 (12)	0.000	0.0062 (11)	0.000

*Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )*

O1—C7	1.314 (4)	N3—C2	1.332 (3)
O1—H1	1.04 (4)	O2—C7	1.200 (3)
C4—N3	1.335 (3)	C5—C6	1.386 (4)

C4—C5	1.376 (4)	C5—H5	0.9300
C4—C7	1.509 (3)	C6—H6	0.9300
N1—C6	1.322 (4)	C2—H4	0.9300
N1—C2	1.329 (4)		
C7—O1—H1	110.9 (19)	C4—C5—C6	116.4 (3)
N3—C4—C5	122.8 (2)	C4—C5—H5	121.8
N3—C4—C7	118.1 (2)	C6—C5—H5	121.8
C5—C4—C7	119.1 (3)	N1—C6—C5	122.4 (3)
C6—N1—C2	116.0 (2)	N1—C6—H6	118.8
C2—N3—C4	115.2 (2)	C5—C6—H6	118.8
O2—C7—O1	124.9 (2)	N1—C2—N3	127.3 (2)
O2—C7—C4	120.6 (3)	N1—C2—H4	116.4
O1—C7—C4	114.5 (2)	N3—C2—H4	116.4

*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>
O1—H1 $\cdots$ N1 <sup>i</sup>	1.04 (4)	1.62 (4)	2.660 (3)	179 (3)

Symmetry code: (i) *x*, *y*, *z*−1.