



The crystal structure of 5-(trifluoromethyl)picolinic acid monohydrate reveals a water-bridged hydrogen-bonding network

Naike Ye and Joseph M. Tanski*

Department of Chemistry, Vassar College, Poughkeepsie, NY 12604, USA. *Correspondence e-mail: jotanski@vassar.edu

Received 24 September 2020

Accepted 8 October 2020

Edited by G. Díaz de Delgado, Universidad de Los Andes, Venezuela

Keywords: crystal structure; hydrogen bonding; picolinic acid derivatives; trifluoromethyl group.

CCDC reference: 2036131

Supporting information: this article has supporting information at journals.iucr.org/e

The title compound [systematic name: 5-(trifluoromethyl)pyridine-2-carboxylic acid monohydrate], $C_7H_4F_3NO_2 \cdot H_2O$, is the acid hydrate of a pyridine with a carboxylic acid group and a trifluoromethyl substituent situated *para* to one another on the aromatic ring. The molecule forms a centrosymmetric water-bridged hydrogen-bonding dimer with graph-set notation $R_4^4(12)$. Hydrogen-bonding distances of 2.5219 (11) and 2.8213 (11) Å are observed between the donor carboxylic acid and the bridging water acceptor, and the donor water and carbonyl oxygen acceptor, respectively. The dimers are further linked into a two-dimensional sheet *via* two longer intermolecular hydrogen-bonding interactions between the second hydrogen atom on the bridging water molecule and both a pyridine nitrogen atom and carbonyl oxygen with distances of 3.1769 (11) and 2.8455 (11) Å, respectively. The trifluoromethyl groups extend out the faces of the sheet providing for $F \cdots F$ and $C-H \cdots F$ contacts between the sheets, completing the three-dimensional packing.

1. Chemical context

Picolinic acids, pyridine derivatives with a carboxylic acid substituent at the 2-position, are common bidentate chelating agents of metallic elements in the human body (Grant *et al.*, 2009). The title compound, the hydrate of 5-(trifluoromethyl)-2-pyridinecarboxylic acid (I), commonly known as 5-(trifluoromethyl)picolinic acid, is a derivative of picolinic acid with potent chelating abilities and biological activities (Li *et al.*, 2019). Its transition-metal complexes also exhibit outstanding photophysical and electrochemical properties that make them promising phosphorescent materials for OLEDs (Wei *et al.*, 2016). The compound may be synthesized from a range of synthetic routes, one of which relies on the carboxylation reaction of 2-bromo-5-(trifluoromethyl)pyridine with butyllithium (Cottet *et al.*, 2003).

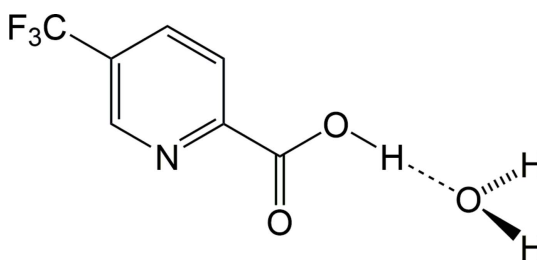
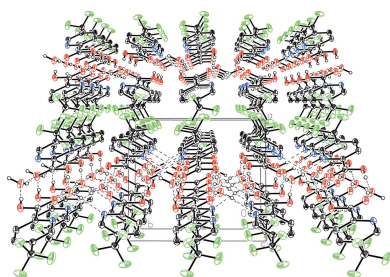


Table 1
 Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1—H1···O1W	0.92 (2)	1.60 (2)	2.5219 (11)	174 (2)
O1W—H2W···O2 ⁱ	0.808 (19)	2.038 (19)	2.8213 (11)	163.2 (17)
O1W—H1W···O2 ⁱⁱ	0.859 (19)	2.615 (18)	3.1769 (11)	124.1 (13)
O1W—H1W···N ⁱⁱ	0.859 (19)	2.008 (18)	2.8455 (11)	164.5 (16)

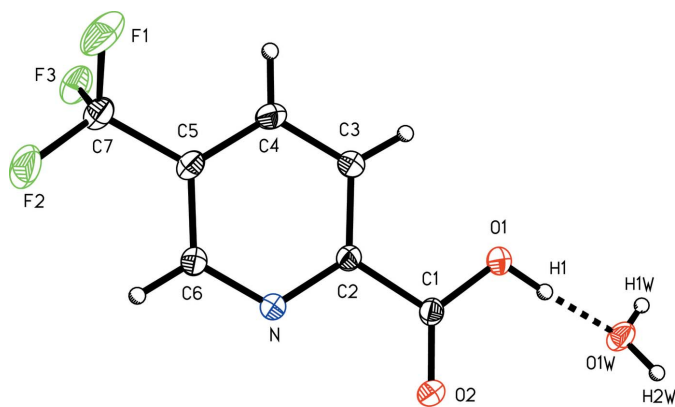
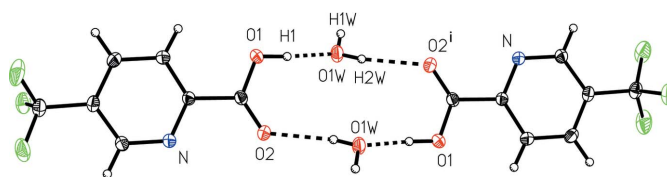
 Symmetry codes: (i) $-x + 1, -y + 1, -z + 2$; (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$.

2. Structural commentary

The structure of 5-(trifluoromethyl)picolinic acid (I) reveals that the crystalline material obtained from the supplier is a hydrate and confirms the position of the carboxylic acid group *ortho* to the pyridine nitrogen atom with trifluoromethyl substituent situated *para* to the acid group on the aromatic ring (Fig. 1). The two aromatic carbon–nitrogen bonds have bond lengths of N—C2 of 1.3397 (12) Å and N—C6 of 1.3387 (12) Å, shorter than the aromatic C—C bonds, which have an average bond length of 1.387 (3) Å, a wedge-type motif typical in pyridine structures (Montgomery *et al.*, 2015). The aromatic carboxylic acid substituent has a C1—C2 bond length of 1.5081 (13) Å, similar to that of the trifluoromethyl substituent C5—C7 of 1.5019 (13) Å, and the C—F bond lengths of the trifluoromethyl group have an average bond length of 1.335 (4) Å. The carboxylic acid group is co-planar with the aromatic pyridine ring, with least-squares planes at an angle of 1.8 (2)°.

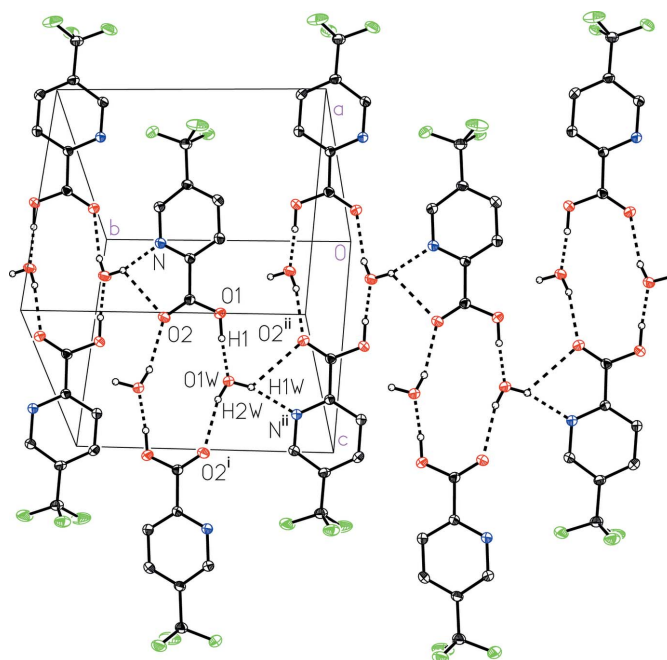
3. Supramolecular features

The structure of 5-(trifluoromethyl)picolinic acid (I) reported is a hydrate (Fig. 1) exhibiting a water-linked two-dimensional hydrogen-bonding network. Four different hydrogen-bonding interactions are observed between the picolinic acid and water molecule, which acts as both a hydrogen-bonding donor and acceptor with the carboxylic acid group and pyridine nitrogen atom (Table 1).


Figure 1
 A view of 5-(trifluoromethyl)picolinic acid (I) hydrate with the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

Figure 2
 A view of the intermolecular water-bridged hydrogen-bonding dimer in 5-(trifluoromethyl)picolinic acid (I) hydrate.

The molecular packing in the solid state can be characterized by the 5-(trifluoromethyl)picolinic acid (I) hydrate asymmetric unit first forming a centrosymmetric water-bridged dimer unit with graph-set notation $R_4^2(12)$ (Fig. 2). The carboxylic acid hydrogen atom and the water oxygen form the stronger hydrogen bond, with an O1···O1W distance of 2.5219 (11) Å characterizing the O1—H1···O1W hydrogen bond, while the water hydrogen atom H2W bonds to the carbonyl oxygen atom with an O1W···O2ⁱ distance of 2.8213 (11) Å characterizing the O1W—H2W···O2ⁱ hydrogen bond [symmetry code: (i) $-x + 1, -y + 1, -z + 2$].

The water molecules, specifically using the other water hydrogen atom H1W, further bridge the dimer units together to form a pleated strip or tape motif that propagates along the crystallographic [010] direction (Fig. 3). The the O1W···Nⁱⁱ and O1W···O2ⁱⁱ distances of 3.1769 (11) and 2.8455 (11) Å, respectively, characterize the O1W—H1W···Nⁱⁱ and O1W—H1W···O2ⁱⁱ hydrogen bonds [symmetry code: (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$]. The pleated nature of the strip exposes the H1W hydrogen atom of every other dimer to a pyridine nitrogen in the strip adjacent to it, forming a thick two-dimensional sheet


Figure 3
 A view of a pleated strip formed between the water-bridged hydrogen-bonding dimers in 5-(trifluoromethyl)picolinic acid (I) hydrate.

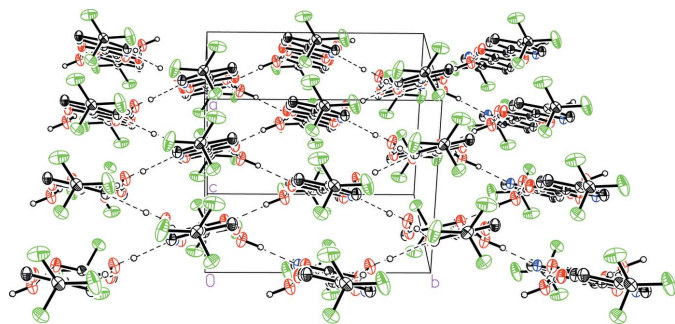


Figure 4
A view of the sheet hydrogen-bonding network in 5-(trifluoromethyl)picolinic acid (I) hydrate viewing the water-bridge hydrogen-bonding dimers end-on shows the hydrogen-bonding interactions between the pleated strips forming a two-dimensional sheet.

(Fig. 4). The sheet can be considered a bilayer with a hydrophilic core due to the presence of water molecules and strong hydrogen bonding in the center and the more hydrophobic trifluoromethylaromatic groups extending to the faces of the sheet (Fig. 5).

The sheets stack in the [100] direction (Fig. 6). The forces that guide the intermolecular interactions between neighboring sheets are van der Waals forces including $F \cdots F$ and $C-H \cdots F$ contacts. The shortest weak $C_{\text{aryl}}-H \cdots F$ interaction, $C4-H4A \cdots F2$ exhibits an $H \cdots F$ distance of 2.495 (1) Å. The most notable interaction is a dimeric $F \cdots F$ interaction between CF_3 groups on neighboring sheets with an $F1 \cdots F3$ distance of 3.077 (1) Å, which is ~ 0.15 Å longer than the sum of the van der Waals radii of fluorine (Bondi, 1964).

4. Database survey

Monocarboxylic derivatives of pyridine, pyridinecarboxylic acids, are also commonly known as picolinic acid, nicotinic acid, or isonicotinic acid when the carboxyl group resides at the 2-, 3-, or 4- position, respectively. The Cambridge Structural Database (Version 5.40, update of March 2020; Groom *et al.*, 2016) contains no isomers of trifluoromethyl-substituted pyridinecarboxylic acids. The crystal structure of the base of the title compound, picolinic acid (PICOLA02), was shown to be 1:1 co-crystals of its neutral and zwitterionic forms, where

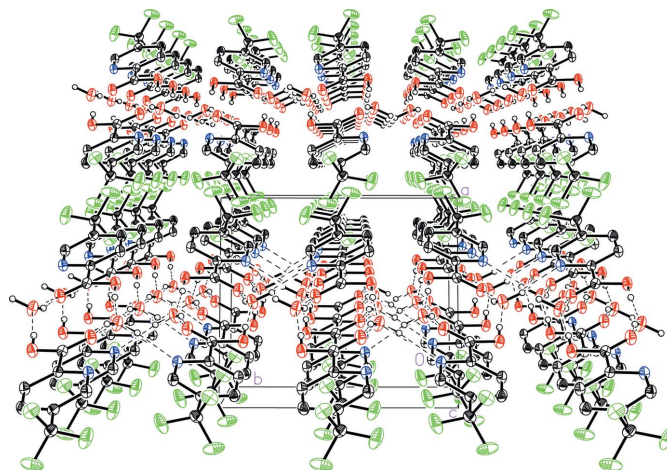


Figure 6
A view of the stacking of the two-dimensional sheets in 5-(trifluoromethyl)picolinic acid (I) hydrate showing the trifluoromethylaromatic interactions at the interfaces of the sheets.

the nitrogen atom can both be protonated and deprotonated (Hamazaki *et al.*, 1998). The interactions form a zigzag chain by $N-H \cdots N$ and $O-H \cdots O$ intermolecular hydrogen bonding. A zwitterionic hydrogen-bonding motif can be found in substituted picolinic acid derivatives as well, such as 3-thioxo-2-pyridinecarboxylic acid (MPYDCX01; Bourne & Taylor, 1983).

A related solvated picolinic acid crystal structure can be found in the crystal structure of picolinic acid peroxosolvate (ANINES) which, while zwitterionic, exhibits a solvate-linked hydrogen-bonding pattern (Medvedev *et al.*, 2013). In this structure, every hydrogen peroxide molecule links three picolinic acid molecules together with two hydrogen bonds between the H_2O_2 hydrogen atoms and two carboxylate groups, and an $N-H \cdots O$ hydrogen bond between the protonated pyridine nitrogen atom and one oxygen atom of the H_2O_2 molecule.

5-(Trifluoromethyl)-2-pyridinecarboxylic acid has been used as a monoanionic ligand in several metal complexes, including with Co^{II} (VOVZOY; Li *et al.*, 2019), Cr^{III} (QEGWOR; Chai *et al.*, 2017), Mn^{II} (ROKSIW; Wang *et al.*, 2019), and Ir^{III} [COKGAN and COKGIV (Sanner *et al.*, 2019);

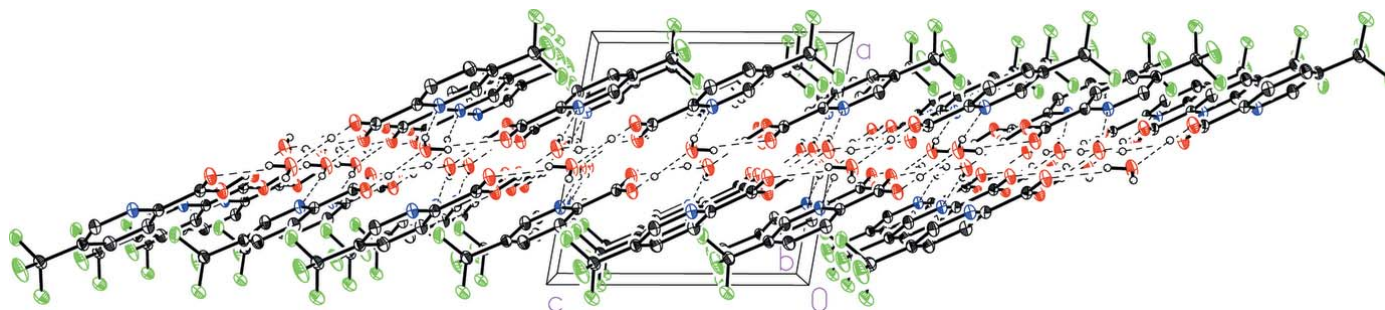


Figure 5
An edge-on view of the two-dimensional sheet formed between the water-bridged hydrogen-bonding dimers in 5-(trifluoromethyl)picolinic acid (I) hydrate.

GIZJOR (Hao *et al.*, 2019)]. While the Co^{II} and Mn^{II} complexes engage in intermolecular hydrogen bonding with metal-coordinated water molecules, the Cr^{III} complex contains a water of solvation that facilitates the formation of a hydrogen-bonding network. In a fashion reminiscent of 5-(trifluoromethyl)picolinic acid (I) hydrate itself, [Cr(5-(trifluoromethyl)picolinate)₂(H₂O)₂]NO₃·H₂O hydrogen bonds into thick two-dimensional sheets with the trifluoromethyl-aromatic groups extending to the faces of the sheets (Chai *et al.*, 2017).

5. Synthesis and crystallization

5-(Trifluoromethyl)-2-pyridinecarboxylic acid (I, 95%) was purchased from Aldrich Chemical Company, USA, and was used as received.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms on carbon atoms were included in calculated positions and refined using a riding model with C–H = 0.95 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ of the aryl C-atoms. The position of the carboxylic acid and water hydrogen atoms were found in the difference map and refined freely.

7. Analytical data

¹H NMR (Bruker Avance III HD 400 MHz, DMSO *d*₆): δ 3.70 (*br s*, OH), 8.21 (*d*, 1H, $C_{\text{aryl}}H$, $J_{\text{ortho}} = 8.0$ Hz), 8.37 (*dd*, 1H, $C_{\text{aryl}}H$, $J_{\text{ortho}} = 8.2$ Hz, $J_{\text{meta}} = 2.0$ Hz), 9.07 (*s*, 1H, $C_{\text{aryl}}H$). ¹³C NMR (¹³C{¹H}, 100.6 MHz, DMSO *d*₆): δ 123.23 (*q*, CF₃, $J_{\text{C-F}} = 272.8$ Hz), 124.73 (*s*, $C_{\text{aryl}}H$), 127.30 (*q*, $C_{\text{aryl}}\text{CF}_3$, $J_{\text{C-F}} = 32.7$ Hz), 135.12 (*q*, $C_{\text{aryl}}H$, $J_{\text{C-F}} = 3.5$ Hz), 146.24 (*q*, $C_{\text{aryl}}H$, $J_{\text{C-F}} = 3.8$ Hz), 151.98 (*s*, $C_{\text{aryl}}\text{COOH}$), 165.13 (*s*, COOH). ¹⁹F NMR (¹⁹F{¹H}, 376.5 MHz, DMSO *d*₆): δ −61.35 (*s*, 3F, CF₃). IR (Thermo Nicolet iS50, FT-IR, KBr pellet, cm^{−1}): 3469 (*s br*, O–H *str*), 3050 (*s*, $C_{\text{aryl}}\text{—H}$ *str*), 2849 (*w*), 2571 (*w*), 1961 (*m*), 1707 (*s*, C=O *str*), 1606 (*s*), 1582 (*s*), 1493 (*s*), 1440 (*s*), 1392 (*s*), 1328 (*m*), 1290 (*m*), 1251 (*s*), 1163 (*m*), 1126 (*s*), 1075 (*s*), 1023 (*s*), 948 (*s*), 878 (*m*), 864 (*s*), 806 (*s*), 760 (*s*), 704 (*s*), 643 (*s*), 524 (*s*). GC-MS (Agilent Technologies 7890A GC/5975C MS): $M^+ = 191$ amu, corresponding to the anhydrous form, 5-(trifluoromethyl)pyridine-2-carboxylic acid (I), whose calculated exact molecular ion mass is 191.02 amu.

Funding information

This work was supported by Vassar College. X-ray facilities were provided by the US National Science Foundation (grant Nos. 0521237 and 0911324 to JMT). We acknowledge the Salmon Fund and Olin College Fund of Vassar College for funding publication expenses.

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₇ H ₄ F ₃ NO ₂ ·H ₂ O
M_r	209.13
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	125
a, b, c (Å)	8.9213 (10), 10.0759 (12), 9.1010 (11)
β (°)	99.983 (2)
V (Å ³)	805.70 (16)
Z	4
Radiation type	Mo $K\alpha$
μ (mm ^{−1})	0.18
Crystal size (mm)	0.32 × 0.25 × 0.14
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (SADABS; Bruker, 2017)
$T_{\text{min}}, T_{\text{max}}$	0.89, 0.98
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19282, 2463, 2126
R_{int}	0.026
$(\sin \theta/\lambda)_{\text{max}}$ (Å ^{−1})	0.715
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.034, 0.103, 1.07
No. of reflections	2463
No. of parameters	139
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ^{−3})	0.50, −0.26

Computer programs: APEX2 and SAINT (Bruker, 2017), SHELXT2014/5 (Sheldrick, 2015a), SHELXL2017/1 (Sheldrick, 2015b), SHELXTL2014 (Sheldrick, 2008), OLEX2 (Dolomanov *et al.*, 2009), and Mercury (Macrae *et al.*, 2020).

References

- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
 Bourne, P. E. & Taylor, M. R. (1983). *Acta Cryst.* **C39**, 266–268.
 Bruker (2017). SAINT, SADABS and APEX2. Bruker AXS Inc., Madison, Wisconsin, USA.
 Chai, J., Liu, Y., Liu, B. & Yang, B. (2017). *J. Mol. Struct.* **1150**, 307–315.
 Cottet, F., Marull, M., Lefebvre, O. & Schlosser, M. (2003). *Eur. J. Org. Chem.* pp. 1559–1568.
 Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
 Grant, R. S., Coggan, S. E. & Smythe, G. A. (2009). *Int. J. Tryptophan Res.* **2**, 71–79.
 Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
 Hamazaki, H., Hosomi, H., Takeda, S., Kataoka, H. & Ohba, S. (1998). *Acta Cryst.* **C54**, IUC9800049.
 Hao, H., Liu, X., Ge, X., Zhao, Y., Tian, X., Ren, T., Wang, Y., Zhao, C. & Liu, Z. (2019). *J. Inorg. Biochem.* **192**, 52–61.
 Li, B., Wang, J., Song, H., Wu, H., Chen, X. & Ma, X. (2019). *J. Coord. Chem.* **72**, 2562–2573.
 Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.
 Medvedev, A. G., Mikhailov, A. A., Prikhodchenko, P. V., Tripol'skaya, T. A., Lev, O. & Churakov, A. V. (2013). *Russ. Chem. Bull.* **62**, 1871–1876.
 Montgomery, M. J., O'Connor, T. J. & Tanski, J. M. (2015). *Acta Cryst.* **E71**, 852–856.

Sanner, R. D., Cherepy, N. J., Martinez, H. P., Pham, H. Q. & Young, V. G. (2019). *Inorg. Chim. Acta*, **496**, 119040.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.

Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
Wang, J., Li, B., Wu, H., Tian, X. & Ma, X. (2019). *Jiegou Huaxue*. **38**, 1349–1355.
Wei, X., Wang, S. & Wei, D. (2016). *Huaxue Tongbao*, **79**, 947–951.

supporting information

Acta Cryst. (2020). E76, 1752-1756 [https://doi.org/10.1107/S2056989020013523]

The crystal structure of 5-(trifluoromethyl)picolinic acid monohydrate reveals a water-bridged hydrogen-bonding network

Naike Ye and Joseph M. Tanski

Computing details

Data collection: *APEX2* (Bruker, 2017); cell refinement: *SAINTE* (Bruker, 2017); data reduction: *SAINTE* (Bruker, 2017); program(s) used to solve structure: *SHELXT2014/5* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2017/1* (Sheldrick, 2015b); molecular graphics: *SHELXTL2014* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL2014* (Sheldrick, 2008), *OLEX2* (Dolomanov *et al.*, 2009), and *Mercury* (Macrae *et al.*, 2020).

5-(Trifluoromethyl)pyridine-2-carboxylic acid monohydrate

Crystal data

$C_7H_4F_3NO_2 \cdot H_2O$

$M_r = 209.13$

Monoclinic, $P2_1/c$

$a = 8.9213$ (10) Å

$b = 10.0759$ (12) Å

$c = 9.1010$ (11) Å

$\beta = 99.983$ (2)°

$V = 805.70$ (16) Å³

$Z = 4$

$F(000) = 424$

$D_x = 1.724$ Mg m⁻³

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

Cell parameters from 7786 reflections

$\theta = 3.0\text{--}30.2^\circ$

$\mu = 0.18$ mm⁻¹

$T = 125$ K

Block, colourless

$0.32 \times 0.25 \times 0.14$ mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.3333 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2017)

$T_{\min} = 0.89$, $T_{\max} = 0.98$

19282 measured reflections

2463 independent reflections

2126 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.026$

$\theta_{\max} = 30.5^\circ$, $\theta_{\min} = 2.3^\circ$

$h = -12 \rightarrow 12$

$k = -14 \rightarrow 14$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.103$

$S = 1.07$

2463 reflections

139 parameters

0 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2 + 0.2301P]$

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

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

$$\Delta\rho_{\max} = 0.50 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	1.02797 (8)	0.44945 (10)	0.19056 (8)	0.0387 (2)
F2	0.91945 (11)	0.63911 (8)	0.13936 (9)	0.0398 (2)
F3	0.80545 (8)	0.45876 (7)	0.06062 (7)	0.02763 (17)
O1	0.62864 (9)	0.36976 (7)	0.76271 (8)	0.02126 (17)
H1	0.572 (2)	0.365 (2)	0.838 (2)	0.056 (6)*
O2	0.57174 (9)	0.58725 (7)	0.74769 (8)	0.02259 (17)
N	0.69746 (10)	0.60686 (8)	0.49521 (9)	0.01815 (18)
C1	0.62637 (11)	0.48809 (9)	0.70248 (10)	0.01643 (18)
C2	0.70005 (11)	0.48977 (9)	0.56536 (10)	0.01547 (18)
C3	0.76396 (12)	0.37599 (9)	0.51689 (11)	0.01936 (19)
H3A	0.762813	0.29467	0.569519	0.023*
C4	0.82968 (12)	0.38369 (10)	0.38964 (11)	0.0205 (2)
H4A	0.87462	0.307748	0.353174	0.025*
C5	0.82825 (11)	0.50451 (10)	0.31708 (11)	0.01741 (19)
C6	0.76044 (12)	0.61375 (10)	0.37220 (11)	0.01917 (19)
H6A	0.758767	0.695976	0.32085	0.023*
C7	0.89493 (12)	0.51403 (10)	0.17683 (11)	0.0210 (2)
O1W	0.46520 (10)	0.34230 (8)	0.96069 (9)	0.02433 (18)
H2W	0.469 (2)	0.3734 (17)	1.043 (2)	0.040 (5)*
H1W	0.4241 (19)	0.2651 (19)	0.9607 (19)	0.043 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0238 (4)	0.0669 (6)	0.0280 (4)	0.0091 (3)	0.0118 (3)	-0.0011 (4)
F2	0.0630 (5)	0.0278 (4)	0.0368 (4)	-0.0179 (3)	0.0317 (4)	-0.0058 (3)
F3	0.0315 (4)	0.0352 (4)	0.0167 (3)	-0.0061 (3)	0.0055 (2)	-0.0055 (3)
O1	0.0303 (4)	0.0167 (3)	0.0190 (3)	0.0011 (3)	0.0104 (3)	0.0034 (3)
O2	0.0328 (4)	0.0185 (3)	0.0189 (3)	0.0026 (3)	0.0112 (3)	-0.0006 (3)
N	0.0240 (4)	0.0154 (4)	0.0161 (4)	0.0013 (3)	0.0066 (3)	0.0005 (3)
C1	0.0192 (4)	0.0165 (4)	0.0135 (4)	-0.0014 (3)	0.0027 (3)	0.0004 (3)
C2	0.0180 (4)	0.0150 (4)	0.0136 (4)	-0.0004 (3)	0.0031 (3)	-0.0006 (3)
C3	0.0252 (5)	0.0151 (4)	0.0188 (4)	0.0012 (3)	0.0066 (4)	0.0006 (3)
C4	0.0248 (5)	0.0174 (4)	0.0207 (4)	0.0015 (3)	0.0078 (4)	-0.0033 (3)
C5	0.0182 (4)	0.0197 (4)	0.0153 (4)	-0.0028 (3)	0.0056 (3)	-0.0031 (3)
C6	0.0252 (5)	0.0167 (4)	0.0168 (4)	-0.0006 (3)	0.0070 (3)	0.0006 (3)
C7	0.0218 (5)	0.0234 (5)	0.0194 (4)	-0.0044 (4)	0.0079 (4)	-0.0036 (4)

O1W 0.0376 (4) 0.0188 (3) 0.0198 (4) -0.0073 (3) 0.0140 (3) -0.0031 (3)

Geometric parameters (Å, °)

F1—C7	1.3402 (13)	C3—C4	1.3879 (13)
F2—C7	1.3335 (13)	C3—H3A	0.95
F3—C7	1.3317 (12)	C4—C5	1.3840 (14)
O1—C1	1.3109 (11)	C4—H4A	0.95
O1—H1	0.92 (2)	C5—C6	1.3908 (13)
O2—C1	1.2143 (12)	C5—C7	1.5019 (13)
N—C6	1.3387 (12)	C6—H6A	0.95
N—C2	1.3397 (12)	O1W—H2W	0.808 (19)
C1—C2	1.5081 (13)	O1W—H1W	0.859 (19)
C2—C3	1.3861 (13)		
C1—O1—H1	113.0 (13)	C4—C5—C6	119.52 (9)
C6—N—C2	117.98 (8)	C4—C5—C7	119.30 (9)
O2—C1—O1	125.78 (9)	C6—C5—C7	121.14 (9)
O2—C1—C2	121.95 (8)	N—C6—C5	122.17 (9)
O1—C1—C2	112.26 (8)	N—C6—H6A	118.9
N—C2—C3	123.42 (9)	C5—C6—H6A	118.9
N—C2—C1	115.48 (8)	F3—C7—F2	107.12 (9)
C3—C2—C1	121.09 (8)	F3—C7—F1	105.68 (8)
C2—C3—C4	118.41 (9)	F2—C7—F1	107.51 (9)
C2—C3—H3A	120.8	F3—C7—C5	112.15 (8)
C4—C3—H3A	120.8	F2—C7—C5	112.61 (8)
C5—C4—C3	118.49 (9)	F1—C7—C5	111.38 (9)
C5—C4—H4A	120.8	H2W—O1W—H1W	107.4 (16)
C3—C4—H4A	120.8		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1...O1W	0.92 (2)	1.60 (2)	2.5219 (11)	174 (2)
O1W—H2W...O2 ⁱ	0.808 (19)	2.038 (19)	2.8213 (11)	163.2 (17)
O1W—H1W...O2 ⁱⁱ	0.859 (19)	2.615 (18)	3.1769 (11)	124.1 (13)
O1W—H1W...N ⁱⁱ	0.859 (19)	2.008 (18)	2.8455 (11)	164.5 (16)

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $-x+1, y-1/2, -z+3/2$.