Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

3-Hydroxypyridinium 3-(carboxymethoxy)phenoxyacetate

Shan Gao,[a] Ji-Wei Liu,[a] Li-Hua Huo,[a] Hui Zhao[a] and Seik Weng Ng[b]*

[a]College of Chemistry and Chemical Technology, Heilongjiang University, Harbin 150080, People's Republic of China, and [b]Department of Chemistry, University of Malaya, Kuala Lumpur 50603, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Received 3 September 2004
Accepted 17 September 2004
Online 25 September 2004

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.057
wR factor = 0.129
Data-to-parameter ratio = 15.3

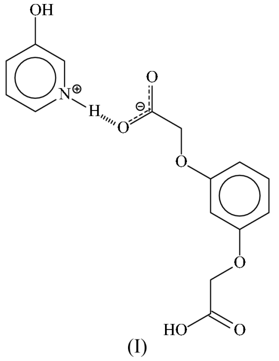
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The crystal structure of the title compound, $C_5H_6NO^+\cdot C_{10}H_9O_6^-$, consists of pairs of 3-hydroxypyridinium cations and pairs of 3-(carboxymethoxy)phenoxyacetate anions that are connected *via* hydrogen bonding into linear ribbons, which are approximately planar. The asymmetric unit consists of one cation and one anion, each located in a general position.

Comment

Pyridine is not commonly used to form pyridinium carboxylates and examples of such salts are rare (Palmore & McBride-Wieser, 1997). The reaction of pyridine with 1,3-phenylenedi(oxyacetic acid) under hydrothermal conditions leads to the formation of the 3-hydroxypyridinium cation, which is formed *in situ* by oxidation of the pyridine at its 3-position.

In the crystal structure of the title salt, (I), anions are linked by O—H···O hydrogen bonding into pairs, which are located around a center of inversion. These pairs are connected into ribbons by two 3-hydroxypyridinium cations, which are also located around a center of inversion, *via* N—H···O and O—H···O hydrogen bonding (Table 2 and Fig. 1).



(I)

A search of the Cambridge Structural Database (Version 5.25; Allen, 2002) yielded a number of 3-hydroxypyridinium carboxylates (Aakeroy & Nieuwenhuyen, 1994; Byriel *et al.*, 1992; Fukunaga *et al.*, 2003; Lynch *et al.*, 1992, 1999; Tafeenko *et al.*, 1990). In these compounds, as well as in the title compound, the cations form two hydrogen bonds.

Experimental

The title compound was prepared by the hydrothermal reaction of pyridine (3 ml) and 1,3-phenylenedi(oxyacetic acid) (4.52 g, 20 mmol) in water. The mixture was sealed in a 15 ml Teflon-lined stainless steel bomb. The bomb was heated at 403 K for 72 h and then cooled slowly to room temperature. Colorless crystals were obtained when the filtered solution was stored for a few days. Analysis calculated for $C_{15}H_{15}NO_7$: C 56.08, H 4.71, N 4.36%; found: C 56.20, H 4.65, N 4.29%.

Crystal data

$C_5H_6NO^+ \cdot C_{10}H_9O_6^-$
$M_r = 321.28$
Monoclinic, $P2_1/n$
$a = 15.960$ (2) Å
$b = 4.8404$ (4) Å
$c = 20.313$ (3) Å
$\beta = 110.58$ (4)°
$V = 1469.1$ (3) Å^3
$Z = 4$

$D_x = 1.453$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6106 reflections
$\theta = 4.4$–27.5°
$\mu = 0.12$ mm^{-1}
$T = 295$ (2) K
Prism, colorless
0.38 × 0.25 × 0.19 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.790$, $T_{\max} = 0.978$
13 092 measured reflections

3367 independent reflections
2121 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.063$
$\theta_{\max} = 27.5°$
$h = -20 \rightarrow 20$
$k = -6 \rightarrow 5$
$l = -26 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.129$
$S = 1.02$
3367 reflections
220 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.1723P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.20$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.334 (3)	C2—C3	1.385 (3)
O2—C6	1.257 (3)	C3—C4	1.372 (3)
O3—C6	1.250 (2)	C4—C5	1.369 (4)
O4—C8	1.374 (2)	C6—C7	1.514 (3)
O4—C7	1.416 (2)	C8—C9	1.391 (3)
O5—C10	1.371 (2)	C8—C13	1.382 (3)
O5—C14	1.410 (2)	C9—C10	1.384 (3)
O6—C15	1.196 (2)	C10—C11	1.385 (3)
O7—C15	1.318 (3)	C11—C12	1.370 (3)
N1—C1	1.327 (3)	C12—C13	1.391 (3)
N1—C5	1.331 (3)	C14—C15	1.506 (3)
C1—C2	1.383 (3)		
C7—O4—C8	118.7 (2)	O4—C8—C13	124.4 (2)
C10—O5—C14	116.7 (2)	O4—C8—C9	114.0 (2)
C1—N1—C5	123.4 (2)	C9—C8—C13	121.6 (2)
N1—C1—C2	119.7 (2)	C8—C9—C10	118.8 (2)
O1—C2—C1	122.7 (2)	O5—C10—C9	124.4 (2)
O1—C2—C3	119.1 (2)	O5—C10—C11	115.2 (2)
C1—C2—C3	118.2 (2)	C9—C10—C11	120.4 (2)
C2—C3—C4	120.0 (2)	C10—C11—C12	119.7 (2)
C3—C4—C5	120.0 (2)	C11—C12—C13	121.4 (2)
N1—C5—C4	118.8 (2)	C8—C13—C12	118.0 (2)
O3—C6—O2	124.0 (2)	O5—C14—C15	109.6 (2)
O3—C6—C7	116.8 (2)	O6—C15—O7	125.8 (2)
O2—C6—C7	119.2 (2)	O6—C15—C14	125.8 (2)
O4—C7—C6	108.5 (2)	O7—C15—C14	108.4 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O2	0.87 (1)	1.77 (1)	2.638 (3)	173 (3)
O1—H1O···O3$^{\rm i}$	0.86 (1)	1.72 (1)	2.569 (2)	172 (3)
O7—H7O···O2$^{\rm ii}$	0.85 (1)	1.82 (1)	2.663 (2)	170 (3)

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

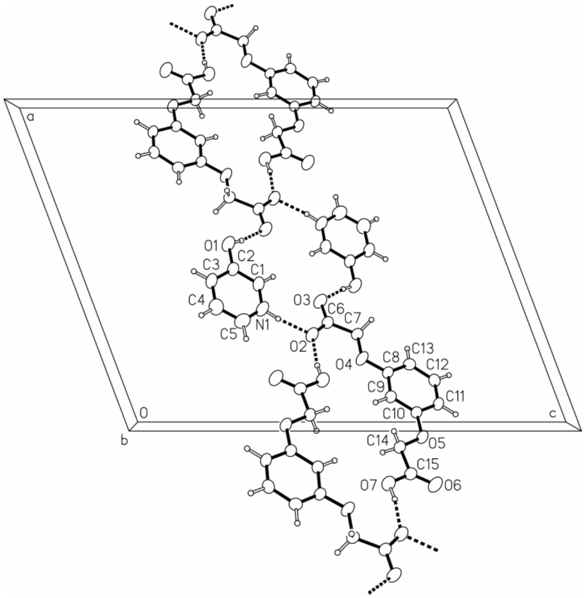


Figure 1
*ORTEP*II (Johnson, 1976) plot with the atom labeling, showing the ribbon structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii and hydrogen bonding is indicated by dotted lines.

Carbon-bound H atoms were placed in calculated positions [aromatic C—H = 0.93 Å and aliphatic C—H = 0.97 Å, and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C)] and were refined isotropically using a riding model. The N—H, hydroxyl and carboxyl H atoms were located in a difference map and refined with individual isotropic displacement parameters and with a distance restraint of 0.85 (1) Å for N—H and O—H.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976).

We thank the National Natural Science Foundation of China (No. 20101003), the Educational Committee Foundation of Heilongjiang Province, Heilongjiang University and the University of Malaya for supporting this study.

References

Aakeroy, C. B. & Nieuwenhuyen, M. (1994). *J. Am. Chem. Soc.* **116**, 10983–10991.
Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Byriel, K. A., Kennard, C. H. L., Lynch, D. E., Smith, G. & Thompson, J. G. (1992). *Aust. J. Chem.* **45**, 969–981.
Fukunaga, T., Kashino, S. & Ishida, H. (2003). *Acta Cryst.* E**59**, o420–o422.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Lynch, D. E., Lad, J., Smith, G. & Parsons, S. (1999). *Cryst. Eng.* **2**, 65–77.
Lynch, D. E., Smith, G., Byriel, K. A. & Kennard, C. H. L. (1992). *Acta Cryst.* C**48**, 533–536.
Palmore, G. T. R. & McBride-Wieser, M. T. (1997). *Acta Cryst.* C**53**, 1904–1907.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Rigaku/MSC Inc., 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Tafeenko, V. A., Bespalov, B. P. & Gakel, V. R. (1990). *Zh. Strukt. Khim.* **31**, 165–167.

Acta Cryst. (2004). E**60**, o1854–o1855 Shan Gao *et al.* • $C_5H_6NO^+ \cdot C_{10}H_9O_6^-$ **o1855**