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N,*N*-Bis(2-pyridiniomethyl)glycine diperchlorate

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In the crystal structure of the title compound, $C_{14}H_{17}N_3O_2^{2+}\cdot 2ClO_4^-$, at 180 K, O—H···O hydrogen-bonded dimers are formed across inversion centres.

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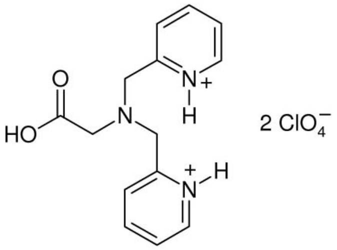
Key indicators

Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.009 Å
Disorder in solvent or counterion
R factor = 0.053
wR factor = 0.143
Data-to-parameter ratio = 7.5

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

Comment

The title compound, (I) (Fig. 1), was crystallized in an unsuccessful attempt to prepare an iron complex of *N*,*N*-bis(2-pyridylmethyl)glycine. In the crystal structure, the carboxylic acid groups form *syn*–*syn* dimers *via* O—H···O hydrogen bonds across centres of inversion (Fig. 2). The NH groups of the protonated pyridyl rings form N—H···O hydrogen bonds, bifurcated at the donor, to O atoms of the perchlorate anions.



(I)

Experimental

N,*N*-Bis(2-pyridylmethyl)glycine (bpgH; 0.0859 g, 0.334 mmol) in hot acetonitrile (2.75 ml) was added to $Fe(ClO_4)_3\cdot 9H_2O$ (0.0861 g, 0.167 mmol) in acetonitrile (0.25 ml). The orange precipitate was dissolved by addition of acetonitrile and water. $HClO_4$ (70%) in a large excess ($HClO_4$:bpgH *ca* 1000:1) was then added. After 9 d, colourless plate-like crystals precipitated in the yellow solution (yield *ca* 38%).

Crystal data

$C_{14}H_{17}N_3O_2^{2+}\cdot 2ClO_4^-$
$M_r = 458.21$
Monoclinic, $P2/c$
$a = 14.1367\ (12)$ Å
$b = 7.9021\ (8)$ Å
$c = 16.8034\ (16)$ Å
$\beta = 104.907\ (3)^\circ$
$V = 1813.9\ (3)$ Å^3
$Z = 4$

$D_x = 1.678$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 2492 reflections
$\theta = 2.5$–21.2°
$\mu = 0.42$ mm^{-1}
$T = 180\ (2)$ K
Plate, colourless
0.12 × 0.08 × 0.01 mm

Data collection

Bruker–Nonius X8APEX-II CCD diffractometer
Thin-slice ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.665$, $T_{\max} = 0.996$
15 019 measured reflections

2175 independent reflections
1542 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.068$
$\theta_{\max} = 22.0^\circ$
$h = -14 \rightarrow 14$
$k = -8 \rightarrow 8$
$l = -17 \rightarrow 17$

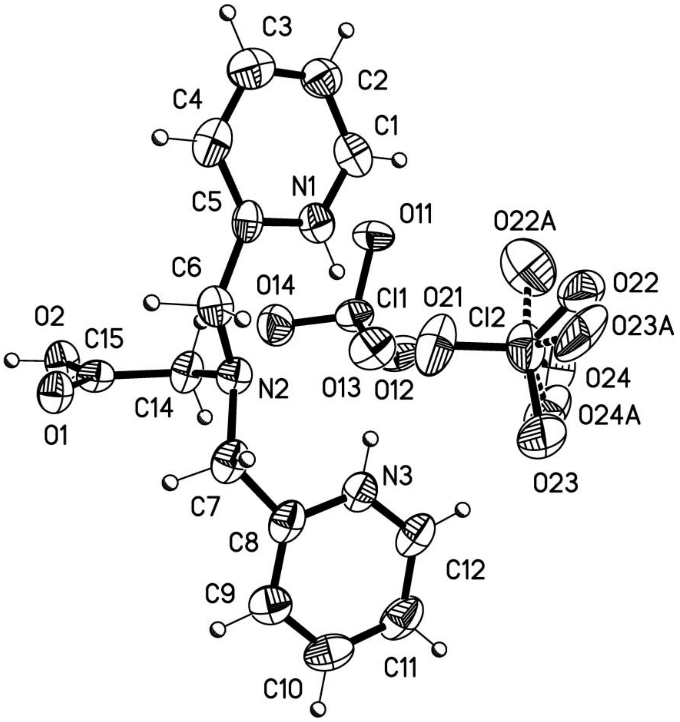


Figure 1
View of the title complex, showing displacement ellipsoids drawn at the 50% probability level for non-H atoms. H atoms are shown as spheres of arbitrary radii. Cl—O bonds in one component of the disordered perchlorate anion are shown as dashed lines.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.143$
$S = 1.07$
2175 reflections
290 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0784P)^2 + 1.4336P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.49$ e Å^{-3}
$\Delta\rho_{\min} = -0.40$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.85	1.82	2.669 (5)	173
N1—H1B···O13	0.88	2.29	3.052 (7)	145
N1—H1B···O21	0.88	2.32	2.877 (7)	121
N3—H3B···O13	0.88	2.48	3.017 (6)	120
N3—H3B···O21	0.88	2.11	2.889 (7)	147

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

Although the best available from the batch, the crystal was relatively small and diffraction was weak. The data were truncated at 0.95 Å resolution, and the structure is therefore of relatively low precision. The unusual ratio of apparent transmission factors suggests that the multi-scan correction is treating effects other than absorption by the crystal itself.

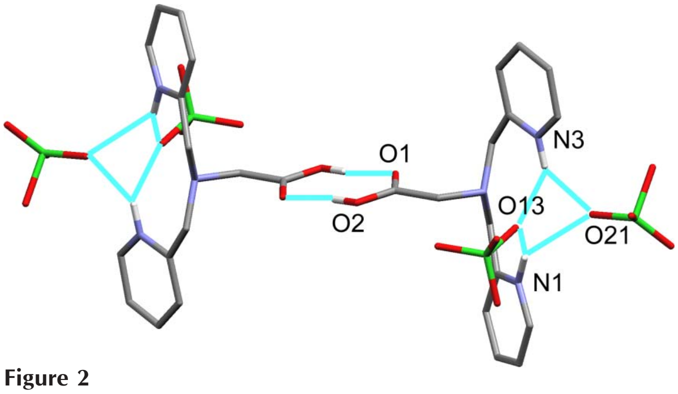


Figure 2
The hydrogen-bonded unit in (I). H atoms not involved in hydrogen bonding have been omitted and only one component of each disordered perchlorate anion is shown; O21, involved in hydrogen bonding, is common to both orientations.

H atoms bound to C atoms were positioned geometrically and allowed to ride during subsequent refinement, with C—H = 0.95 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) for the H atoms of the pyridyl rings, and C—H = 0.99 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) for the methylene groups. The H atoms bound to N atoms were evident in Fourier maps, but on account of the relatively low data-to-parameter ratio, they were placed in calculated positions in the final model and allowed to ride, with N—H = 0.88 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(N). The H atom of the carboxyl group was also located in a difference Fourier map, then moved along the O2—H2 bond vector to give O—H = 0.85 Å. It was subsequently allowed to ride on O2 with $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(O). One perchlorate anion was modelled as disordered over two orientations. The site-occupancy factors of the O atoms in the two orientations were refined initially with a single isotropic displacement parameter common to all. This gave refined site occupancies of 0.59 (1):0.41 (1), which were subsequently constrained to be 0.60:0.40. Sites Cl2 and O21 (involved in the N—H···O hydrogen bond) are common to both orientations and refined with full site occupancy. The anisotropic displacement parameters of the O atoms of the anion were restrained to be approximately isotropic.

Data collection: *APEX2* (Bruker–Nonius, 2004); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

Bruker–Nonius (2004). *APEX2*. Version 1.0-22. Bruker-Nonius BV, Delft, The Netherlands.
Bruker (2003). *SAINT*. Version 7.06a. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.

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