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Dimethyl iminiodiacetate chloride

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.163
Data-to-parameter ratio = 14.8

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

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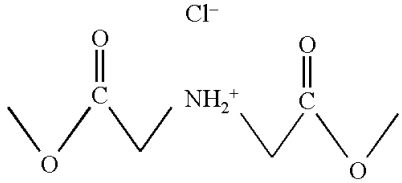
In the title compound, $C_6H_{12}NO_4^+\cdot Cl^-$, the cations and anions are joined together *via* strong hydrogen bonds, forming infinite chains running along the [100] direction. The cation has crystallographic mirror symmetry, and the anion also lies on a mirror plane.

Comment

The presence of two carboxylic groups in the iminodiacetic acid molecule accounts for its well known ion coordination ability, widely used in the preparation of modified polymers and resins for ion capture, for both analytical (Bashir & Paull, 2002; Grotti *et al.*, 2002; Moreno-Villoslada *et al.*, 2001) and industrial uses (Wang *et al.*, 2002; Sun *et al.*, 2002). Iminodiacetic acid derivatives are also used as solid phase supports for peptide synthesis (Khattab *et al.*, 2000) and as a base for chemical libraries in combinatorial chemistry (Goldberg *et al.*, 2002). They are of great importance in organic chemistry, for the synthesis of maleimides (Cheng *et al.*, 2001) and 3,4-disubstituted pyrroles (Friedman, 1964) through the Friedman method. For the preparation of β-octaphenylporphyrin, whose manganese complex is a good catalyst for the oxidation of hydrocarbons (Sobral & Rocha Gonsalves, 2001), we needed to synthesize several β,β-diphenylpyrroles, using some iminodiacetic derivatives as precursors. The title compound, (I), is among those precursors.



(I)

The iminodiacetic dimethyl ester molecules crystallize as positively charged ions, with the imino group protonated. A crystallographic mirror plane contains the NH_2 group. The non-H skeleton of the cation is approximately planar, with a maximum deviation from the least-squares plane of 0.059 (7) Å for O1. The molecule is extended in a *trans* conformation. The central C—N—C angle is 111.4 (3)°, a value in agreement with that found in the related compound iminodiacetic acid hydrofluoride monohydrate (Oskarsson, 1974). The iminodiacetic dimethyl ester cations are connected by N—H···Cl hydrogen bonds to the chloride anions (which also lie on mirror planes), forming infinite chains running along the direction [100]. Interactions between chains are van der Waals forces and weak C—H···O interactions.

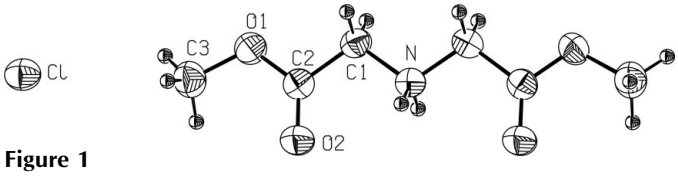


Figure 1
*ORTEP*II (Johnson, 1976) plot of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

The title compound was prepared according to the method described by Jongkees (1908), for the esterification of iminodiacetic acid with methanol and HCl, and crystallized directly from the reaction medium as its hydrochloride, in 80% yield.

Crystal data

$C_6H_{12}NO_4^+\cdot Cl^-$
M_r = 197.62
Orthorhombic, *Pnma*
a = 7.0228 (4) Å
b = 17.298 (9) Å
c = 7.6646 (5) Å
V = 931.1 (5) Å^3
Z = 4
D_x = 1.410 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 21.1–32.1°
μ = 3.51 mm^{-1}
T = 293 (2) K
Needle, colourless
0.49 × 0.08 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.706, T_{max} = 0.839
1837 measured reflections
950 independent reflections
787 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 72.4°
$h = 0 \rightarrow 8$
$k = -21 \rightarrow 21$
$l = -9 \rightarrow 0$
3 standard reflections
frequency: 180 min
intensity decay: 4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.163
S = 1.19
950 reflections
64 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0755P)^2 + 0.752P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0030 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H1···Cl^{i}	0.87 (5)	2.43 (5)	3.299 (3)	180 (4)
N—H2···Cl^{ii}	0.83 (5)	2.25 (5)	3.069 (3)	168 (5)

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

The imino H atoms were located in a difference Fourier map and refined with U_{iso}(H) = 1.2U_{eq}(N). The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å), with U_{iso}(H) = 1.5U_{eq}(C), but were allowed to rotate freely about the C—C bonds. Methylene H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C). Examination of the crystal structure with *PLATON* (Spek, 2002) showed that there are no solvent-accessible voids in the crystal structure.

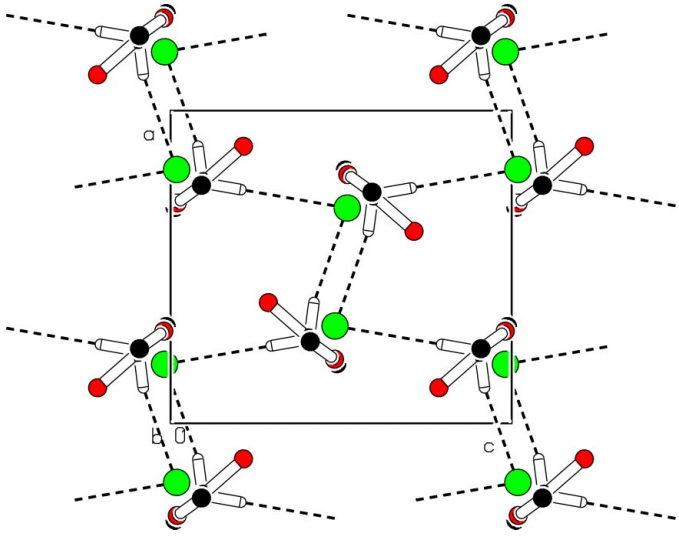


Figure 2
View of the packing, with the hydrogen bonding shown as dashed lines. H atoms not participating in the hydrogen bonding have been omitted for clarity.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *HELENA* (Spek, 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); software used to prepare material for publication: *SHELXL*97.

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