Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Matrinium tetrachloroferrate(III)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.054
wR factor = 0.123
Data-to-parameter ratio = 18.8

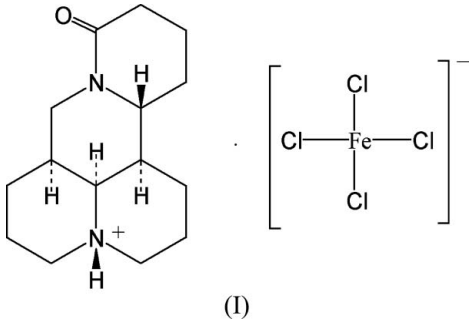
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 11 October 2005
Accepted 27 October 2005
Online 5 November 2005

The title compound, $(C_{15}H_{25}N_2O)[FeCl_4]$, consists of one matrinium cation {matrine is (7a*S*,13a*R*,13b*R*,13c*S*)-dodecahydro-1*H*,5*H*,8*H*-dipyrido[2,1-*f*:3′,2′,1′-*ij*][1,6]-naphthyridin-10-one} and one $[FeCl_4]^-$ anion. One ring of the matrinium cation has a half-chair conformation, whereas the others have chair conformations. Chiral chains of the title compound are formed by C—H···O and N—H···O hydrogen bonds.

Comment

Matrine is one of the most important active ingredients of the traditional Chinese herbal medicine Ligusticum Wallichii Franchat (Ku Shen). It possesses anti-inflammatory properties (Tan *et al.*, 1985), an anti-arrhythmic effect (Zhang *et al.*, 1990), a significant inhibitive effect on proliferation cells and an inductive effect on differentiation in K-562 cells (Zhang *et al.*, 2001); it also has a protective effect on lipopolysacchride-reduced liver injury (Lin *et al.*, 1997), and on restraint and water immersion stress ulcers in mice (Yamazaki *et al.*, 1984). The title compound, (I), has been synthesized, and its structure (Table 1) is discussed here.



(I)

The asymmetric unit of (I) comprises one $[FeCl_4]^-$ anion and one matrinium cation (Fig. 1). The *D* ring (containing atom C15) of the matrinium cation has a half-chair conformation, whereas the other rings adopt chair forms. The *A* (containing C2) and *C* (containing C17), and *B* (containing C10) and *C* rings have a *cis*-type linkage, whereas *A* and *B* have a *trans*-type linkage. The chiral C5(*S*), C6(*S*), C7(*R*) and C11(*R*) atoms have the same absolute configurations as those reported previously (Ibragimov *et al.*, 1978; Zhang *et al.*, 2003).

The NH group is engaged in a hydrogen bond with the O atom of a symmetry-related molecule. The H1*N*···O1 distance (Table 1), which is shorter than the H···O distances observed in other compounds [1.96 Å (de Figueiredo *et al.*, 2002) and 2.131–2.142 Å (Morzyk-Ociepa *et al.*, 2004)], indicates a strong hydrogen bond. C—H···O hydrogen bonds can play important roles in determining molecular packing (Braga *et al.*, 1999;

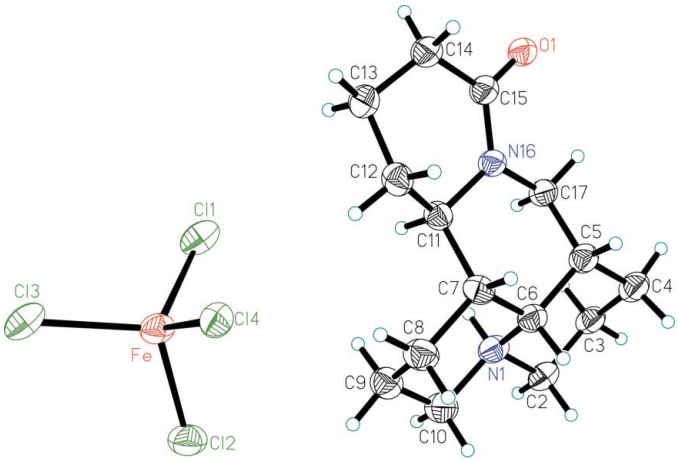


Figure 1
The asymmetric unit of (I), with atom labels, showing 35% probability displacement ellipsoids.

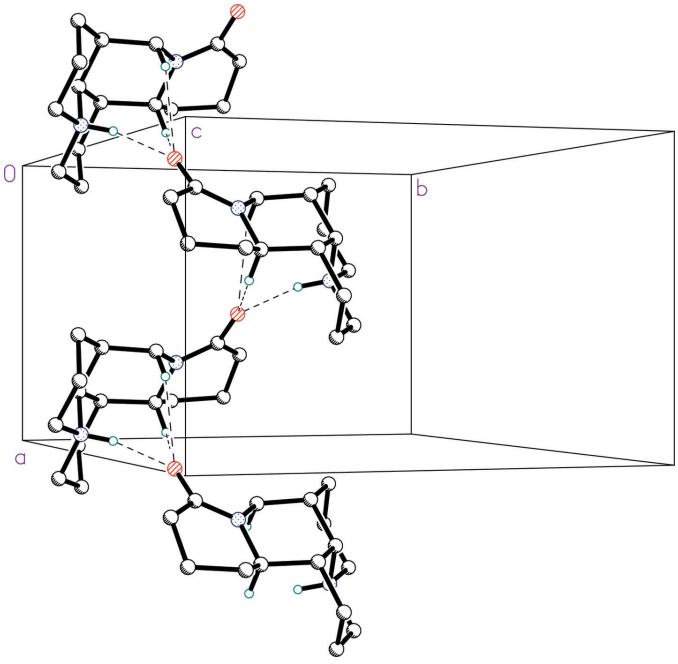


Figure 2
Part of the packing of (I), showing the chiral chain running along the *a* axis. Hydrogen bonds are depicted as dashed lines. H atoms not involved in these interactions have been omitted.

Janiak & Scharmann, 2003; Chang *et al.*, 2005; Zhu *et al.*, 2005). In the crystal structure of (I), beside the strong N—H···O hydrogen bond, there are also two weak C—H···O hydrogen interactions, resulting in a triple O acceptor (Table 1 and Fig. 2). The matrinium cations are linked with one another *via* these N—H···O and C—H···O interactions to form a chiral chain running along the *a* axis (Fig. 2).

Experimental

The caulis of Ligusticum Wallichii Franchat from Qinling Mountain (20.05 kg) was extracted with 70% aqueous EthOH at room temperature. After evaporation of EthOH, the concentrate was acidified to pH 4 with dilute HCl and extracted with CH_2Cl_2. Then, the CH_2Cl_2 extract was chromatographed on a silica gel column to give matrine (1.27 g), which was comfirmed by ^{1}H NMR analysis. Matrine, aqueous HCl and $FeCl_3$ in a molar ratio of 1:1:1 were mixed and dissolved in sufficient water by heating to 353 K, when a clear solution resulted. Crystals of (I) were formed by gradual evaporation of excess water over a period of one week at 293 K.

Crystal data

$(C_{15}H_{25}N_2O)[FeCl_4]$
M_r = 447.02
Orthorhombic, $P2_12_12_1$
a = 8.4871 (8) Å
b = 12.9037 (12) Å
c = 18.2256 (17) Å
V = 1996.0 (3) Å^3
Z = 4
D_x = 1.488 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5069 reflections
θ = 2.6–26.6°
μ = 1.30 mm^{-1}
T = 293 (2) K
Block, orange
0.33 × 0.25 × 0.2 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.69, T_{max} = 0.77
10747 measured reflections
3921 independent reflections
3402 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 26.0°
$h = -10 \rightarrow 8$
$k = -15 \rightarrow 15$
$l = -17 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.123
S = 1.11
3921 reflections
209 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.046P)^2 + 2.4474P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Absolute structure: Flack (1983), 1671 Friedel pairs
Flack parameter: 0.06 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1^{i}	0.91	1.86	2.748 (6)	166
C11—H11···O1^{i}	0.98	2.45	3.241 (6)	138
C17—H17*B*···O1^{i}	0.97	2.54	3.316 (7)	137

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$.

All H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.91 (N—H), 0.97 (methylene) and 0.98 Å (methine), with U_{iso}(H) values of 1.2 times U_{eq} of the parent atoms.

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

We are indebted to the National Natural Science Foundation of China (No. 30500339) for financial support.

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