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Bis(1,8-diazabicyclo[5.4.0]undec-7-en-8-ium) *trans*-Dichlorotetra(pyridine-*N*)-iron(II) Dichloride

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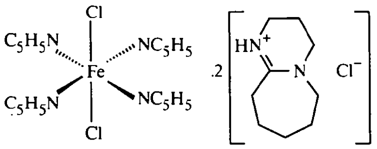
Abstract

The title compound, $(C_9H_{17}N_2)_2[FeCl_2(C_5H_5N)_4]Cl_2$, consists of well separated $[FeCl_2(NC_5H_5)_4]$ complex molecules, 1,8-diazabicyclo[5.4.0]undec-7-en-8-ium ($HDBU^+$) cations and Cl^- anions. The Fe atom, located at an inversion center, is octahedrally coordinated, with the two chloro ligands arranged *trans*. The Fe—Cl distance is 2.4067 (8) Å. The N atoms of the four pyridine ligands constitute the equatorial plane, with Fe—N distances in the range 2.273 (3)–2.279 (3) Å.

Comment

The $[FeCl_2(NC_5H_5)_4]$ complex was formed as a crystalline by-product in an attempt to synthesize an iron(III) complex of the tetradentate ligand glyoxal-bis(2-mercaptoanil). Mercapto groups are well known as reducing agents for trivalent iron compounds (Hagen *et al.*, 1981). 1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU) was used as an auxiliary base.



(I)

The structure of $[FeCl_2(NC_5H_5)_4].2[(HDBU)Cl]$, (I), is shown in Fig. 1, together with the atomic numbering scheme. In the structure, the Fe atom occupies the inversion center at (0,0,0). It is octahedrally coordinated

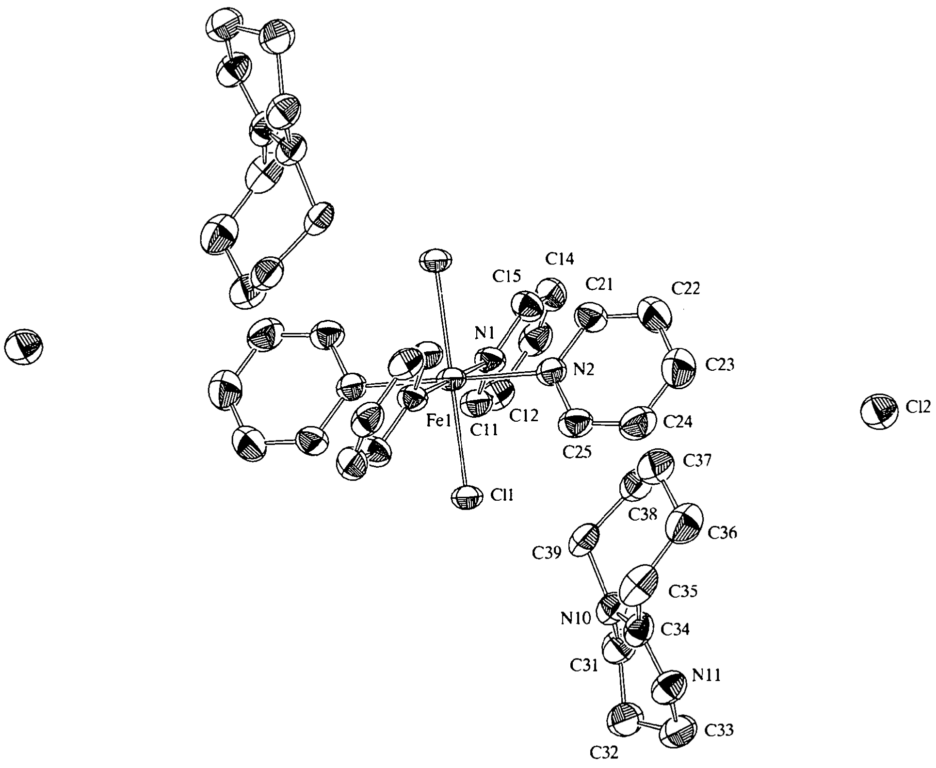


Fig. 1. *ZORTEP* plot (Zsolnai, 1994) of $[FeCl_2(NC_5H_5)_4].2[(HDBU)Cl]$ with the atomic numbering scheme and 50% probability ellipsoids.

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by two Cl atoms, in a *trans* arrangement, and the four N atoms of the pyridine ligands, which form the equatorial plane; the Fe—Cl distances are 2.4067 (8) Å, while the Fe—N distances are in the range 2.273 (3)–2.279 (3) Å. A slight distortion of the octahedral coordination is indicated by the angle N1—Fe—N2 of 95.23 (11)°.

A comparison of the Fe—N and Fe—Cl distances with the corresponding values in other structurally characterized iron(II)- and iron(III)–chloro–pyridine complexes (Hoser *et al.*, 1983; Januszczyk *et al.*, 1991) confirms the divalent character of the iron center, as the Fe—N and the Fe—Cl distances in trivalent iron complexes are approximately 0.1 Å shorter.

The H atom at the secondary N atom of the DBU molecule was located in the Fourier map thus confirming the HDBU$^+$ cation.

Experimental

The black product of the reaction of iron(III) chloride, glyoxal–bis(2-mercaptoanil) (Bayer & Breitmaier, 1968) and 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) in methanol was dissolved in pyridine and covered with a layer of hexane. This resulted in a black powder which was [glyoxal–bis(2-mercaptoanil)]iron(II) and yellow block-shaped crystals of [FeCl$_2$(NC$_5$H$_5$)$_4$].2[(HDBU)Cl].

Crystal data

$(C_9H_{17}N_2)_2[FeCl_2(C_5H_5N)_4]Cl_2$
$M_r = 820.54$
Triclinic
$P\bar{1}$
$a = 7.2582\,(14)$ Å
$b = 9.585\,(2)$ Å
$c = 15.470\,(3)$ Å
$\alpha = 93.626\,(12)°$
$\beta = 101.703\,(11)°$
$\gamma = 101.479\,(10)°$
$V = 1026.7\,(3)$ Å^3
$Z = 1$
$D_x = 1.327$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 15.5$–$26.9°$
$\mu = 5.629$ mm^{-1}
$T = 223\,(2)$ K
Block
0.2 × 0.2 × 0.1 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.454$, $T_{\max} = 0.570$
4384 measured reflections
3497 independent reflections

3140 reflections with $F > 2\sigma(F)$
$R_{\rm int} = 0.129$
$\theta_{\max} = 64.84°$
$h = -1 \rightarrow 8$
$k = -11 \rightarrow 10$
$l = -18 \rightarrow 18$
3 standard reflections every 300 reflections intensity decay: 0.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.154$
$S = 1.128$
3497 reflections
237 parameters
H atoms calculated at idealized positions
$w = 1/[\sigma^2(F_o^2) + (0.0819P)^2 + 0.9531P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.824$ e Å^{-3}
$\Delta\rho_{\min} = -1.138$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.033 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Fe1—Cl1	2.4067 (8)	C11—C12	1.374 (5)
Fe1—Cl1^{i}	2.4067 (8)	C12—C13	1.386 (5)
Fe1—N1	2.279 (3)	C13—C14	1.375 (6)
Fe1—N1^{i}	2.279 (3)	C14—C15	1.375 (5)
Fe1—N2	2.273 (3)	C21—C22	1.377 (6)
Fe1—N2^{i}	2.273 (3)	C22—C23	1.369 (7)
N1—C11	1.338 (5)	C23—C24	1.378 (6)
N1—C15	1.345 (4)	C24—C25	1.367 (6)
N2—C21	1.344 (4)	C31—C32	1.494 (6)
N2—C25	1.344 (5)	C32—C33	1.495 (7)
N10—C34	1.322 (5)	C34—C35	1.495 (6)
N10—C31	1.472 (5)	C35—C36	1.528 (7)
N10—C39	1.478 (5)	C36—C37	1.512 (7)
N11—C34	1.314 (5)	C37—C38	1.517 (7)
N11—C33	1.466 (6)	C38—C39	1.517 (6)
Cl1—Fe1—Cl1^{i}	180.0	N2—C21—C22	123.0 (4)
Cl1—Fe1—N1	89.81 (7)	C21—C22—C23	119.5 (4)
Cl1—Fe1—N1^{i}	90.19 (7)	C22—C23—C24	118.3 (4)
Cl1^{i}—Fe1—N1	90.19 (7)	C23—C24—C25	119.1 (4)
Cl1—Fe1—N2	89.75 (7)	C24—C25—N2	123.6 (3)
Cl1—Fe1—N2^{i}	90.25 (7)	C25—N2—C21	116.4 (3)
Cl1^{i}—Fe1—N2	90.25 (7)	N10—C31—C32	109.9 (3)
N1—Fe1—N1^{i}	180.0	N10—C34—C35	120.3 (3)
N1—Fe1—N2	95.23 (11)	N10—C39—C38	112.7 (3)
N1—Fe1—N2^{i}	84.77 (11)	N10—C34—N11	121.7 (4)
N1^{i}—Fe1—N2	84.77 (11)	N11—C33—C32	110.0 (3)
N2—Fe1—N2^{i}	180.0	N11—C34—C35	117.8 (4)
Fe1—N1—C11	120.6 (2)	C31—N10—C34	121.3 (3)
Fe1—N1—C15	122.8 (2)	C31—N10—C39	116.6 (3)
Fe1—N2—C21	122.4 (2)	C34—N10—C39	121.0 (3)
Fe1—N2—C25	121.1 (2)	C33—N11—C34	122.9 (4)
N1—C11—C12	124.0 (3)	C31—C32—C33	110.8 (4)
C11—C12—C13	118.5 (4)	C34—C35—C36	112.1 (3)
C12—C13—C14	118.4 (4)	C35—C36—C37	114.8 (4)
C13—C14—C15	119.4 (4)	C36—C37—C38	114.1 (4)
C14—C15—N1	123.1 (3)	C37—C38—C39	113.9 (3)
C15—N1—C11	116.6 (3)		

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

H atoms were calculated at ideal positions with common isotropic displacement parameters ($U_{\rm iso} = 0.08$ Å^2).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *SET*4 (de Boer & Duisenberg, 1984) in *CAD*-4 *Software*. Data reduction: *SDP* (Enraf–Nonius, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1994). Software used to prepare material for publication: *SHELXL*93. Computations were performed on a DEC VAX Station 3100, a CONVEX C3860 and a Pentium 120 PC.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV1020). Services for accessing these data are described at the back of the journal.

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Dichloro(μ-[18]aneN_2S_4)dipalladium(II) Bis(triiodide)

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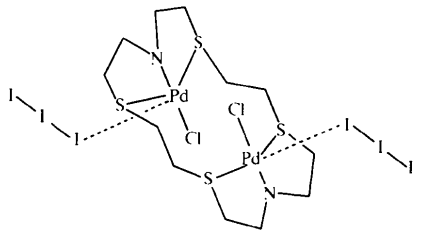
Abstract

In the title compound, μ-(1,4,10,13-tetrathia-7,16-diazacyclooctadecane)-S^4,N^7,S^{10}:S^1,N^{16},S^{13}-bis[chloropalladium(II)] bis(triiodide), $[Pd_2Cl_2(C_{12}H_{26}N_2S_4)](I_3)_2$, the I_3^- anions are linked together through I···I contacts of 4.0236 (6) Å to form infinite sinusoidal chains. These chains are linked by dinuclear palladium complex units *via* Pd···I contacts of 3.5429 (6) Å to form undulating sheets.

Comment

On the grounds that large cations tend to stabilize large anions, we have been interested in ascertaining whether metal–thioether macrocycle cations could be used as templating agents for the self-assembly of extended polyiodide arrays (Blake *et al.*, 1995, 1996; Blake, Devillanova *et al.*, 1998). In this context, numerous polyiodide networks have been synthesized, featuring spirals, belts, ribbons, chains, sheets and cages as their motifs (Blake, Devillanova *et al.*, 1998; Blake, Gould *et al.*, 1998). In some cases, the basic polyiodide units (I_3^-, I_5^-, I_7^-, I_8^{2-}, I_9^- and I_{12}^{2-}), which act as the building blocks for the construction of these extended multidimensional anionic networks, are more isolated from each other and no I···I interactions below *ca* 4.2 Å are observed in the lattice. Nevertheless, S···I and metal···I contacts frequently lead to interesting patterns in the organization of metal cations and polyiodide anions (Blake, Li *et al.*, 1998; Blake, Lippolis *et al.*, 1998), and the title compound, (I), is one example of this.



(I)

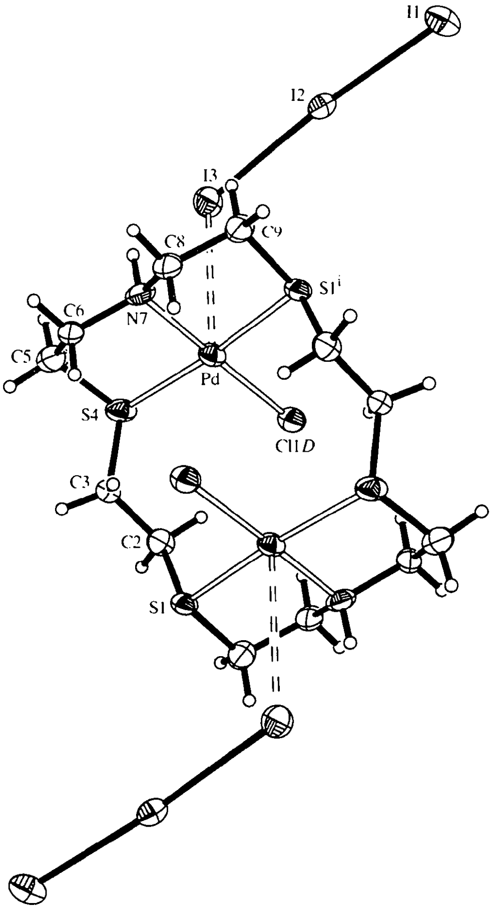


Fig. 1. A view of the title compound showing the atom-numbering system. The dication lies across a crystallographic inversion centre which also relates the two triiodides. Displacement ellipsoids enclose 50% probability surfaces and H atoms are shown as spheres of arbitrary radii. Only one of the four components (Cl1*D*) of the disordered chloride is shown. Double dashed lines indicate Pd···I distances of 3.5429 (6) Å. [Symmetry code: (i) 2 − *x*, 1 − *y*, 1 − *z*.]

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