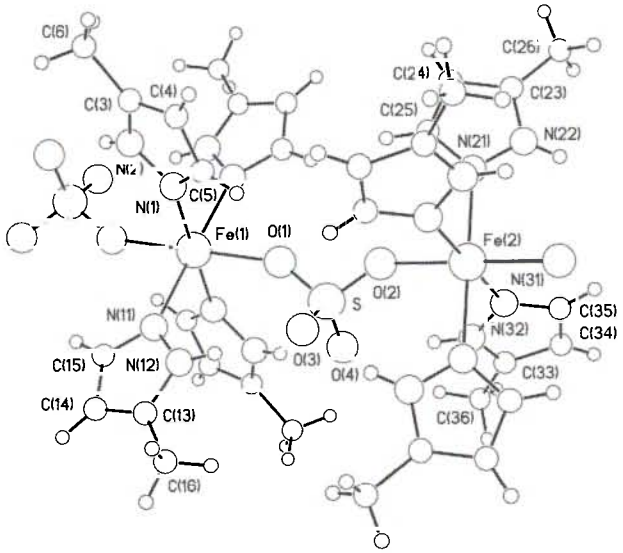
Crystal structure of tetrakis(3-methylpyrazole)iron(II) sulphate, $C_{16}H_{24}FeN_8O_4S$

H. Dittmar*[I], K. Böhn[II] and N. Walker[II]

[I] BASF AG, ADP/V M505, D-67056 Ludwigshafen, Germany
[II] BASF AG, ZHF/W A30, D-67056 Ludwigshafen, Germany

Received August 26, 1999, CCDC-No. 1267/278



Abstract

$C_{16}H_{24}FeN_8O_4S$, triclinic, $P\bar{1}$ (No. 2), $a = 9.101(2)$ Å, $b = 10.162(2)$ Å, $c = 13.066(2)$ Å, $\alpha = 97.91(1)°$, $\beta = 109.97(1)°$, $\gamma = 100.68(1)°$, $V = 1089.4$ Å^3, $Z = 2$, $R_{gt}(F) = 0.044$, $wR(F^2) = 0.103$, $T = 203$ K.

Source of material

36.5 g (NH_4) $FeSO_4$ were dissolved in 100 g H_2O, and 31 g 3-methylpyrazole were added under stirring. The precipitate was filtered, washed with 50 ml H_2O and with 100 ml acetone and dried for two hours at 323 K. The melting point of the salt is 508 K.

Discussion

The product exists as a non-ionic organometallic polymer of stoichiometric $Fe(II)L_4SO_4$, were L = 3-methylpyrazole. Four L ligands are coordinated to each Fe in a square-planar configuration, and these are linked together by sulphate groups which bind axially and complete the octahedral coordination of each Fe. The crystallographic asymmetric unit contains two "half" FeL_4 groups, with the Fe atoms sitting on crystallographic inversion centres, and a complete sulphate group.

Table 1. Data collection and handling.

Crystal:	white parallelepiped, size 0.08 × 0.10 × 0.20 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	67.98 cm^{-1}
Diffractometer, scan mode:	Siemens P4, θ/2θ
$2\theta_{max}$:	113.28°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2893, 2893
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2065
$N(param)_{refined}$:	295
Programs:	SHELXS-86 [1], SHELXL-93 [2], SHELXTL [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	2*i*	0.0293(5)	-0.1861(4)	-0.2090(3)	0.08(2)
H(4)	2*i*	0.4593(6)	-0.2199(5)	-0.0402(4)	0.06(2)
H(5)	2*i*	0.3744(6)	-0.0621(5)	0.0795(4)	0.03(1)
H(6A)	2*i*	0.314(2)	-0.359(3)	-0.259(1)	0.10(1)
H(6B)	2*i*	0.191(5)	-0.282(1)	-0.3281(7)	0.10(1)
H(6C)	2*i*	0.127(3)	-0.410(2)	-0.284(2)	0.10(1)
H(12)	2*i*	0.3157(5)	0.2677(4)	0.1312(3)	0.07(2)
H(14)	2*i*	0.1538(7)	0.4864(5)	-0.0836(4)	0.09(2)
H(15)	2*i*	-0.0254(7)	0.2512(5)	-0.1445(5)	0.06(2)
H(16A)	2*i*	0.526(1)	0.515(2)	0.110(2)	0.10(1)
H(16B)	2*i*	0.455(3)	0.519(3)	0.2045(5)	0.10(1)
H(16C)	2*i*	0.416(2)	0.6163(5)	0.119(2)	0.10(1)
H(22)	2*i*	0.4312(4)	-0.2820(3)	0.5894(3)	0.02(1)
H(24)	2*i*	0.1954(6)	-0.5074(5)	0.2900(4)	0.04(1)
H(25)	2*i*	0.2975(6)	-0.2678(5)	0.2802(4)	0.04(1)
H(26A)	2*i*	0.210(5)	-0.539(3)	0.556(4)	0.13(2)
H(26B)	2*i*	0.390(1)	-0.547(3)	0.578(3)	0.13(2)
H(26C)	2*i*	0.247(6)	-0.6375(6)	0.4685(8)	0.13(2)
H(32)	2*i*	0.1935(4)	0.1266(3)	0.4833(3)	0.02(1)
H(34)	2*i*	0.2076(6)	0.0806(5)	0.7772(4)	0.06(2)
H(35)	2*i*	0.4076(6)	-0.0228(5)	0.7319(4)	0.04(1)
H(36A)	2*i*	-0.026(3)	0.200(3)	0.5198(6)	0.09(1)
H(36B)	2*i*	0.065(1)	0.299(1)	0.639(3)	0.09(1)
H(36C)	2*i*	-0.061(2)	0.160(2)	0.624(3)	0.09(1)

* Correspondence author

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe(1)	1*a*	0	0	0	0.0263(6)	0.0256(6)	0.0219(5)	0.0080(5)	0.0096(4)	0.0032(4)
Fe(2)	1*f*	1/2	0	1/2	0.0229(6)	0.0258(6)	0.0238(5)	0.0077(5)	0.0095(5)	0.0067(4)
S	2*i*	0.2568(1)	0.0984(1)	0.27701(8)	0.0268(7)	0.0312(6)	0.0216(6)	0.0081(5)	0.0064(5)	0.0062(5)
O(1)	2*i*	0.1483(4)	0.0142(3)	0.1665(2)	0.035(2)	0.032(2)	0.022(2)	0.005(2)	0.008(1)	0.001(1)
O(2)	2*i*	0.3250(4)	0.0066(3)	0.3484(2)	0.031(2)	0.035(2)	0.036(2)	0.011(2)	0.013(2)	0.014(1)
O(3)	2*i*	0.3918(4)	0.1931(3)	0.2655(2)	0.030(2)	0.043(2)	0.030(2)	–0.002(2)	0.004(2)	0.013(2)
O(4)	2*i*	0.1675(4)	0.1749(3)	0.3267(2)	0.040(2)	0.043(2)	0.034(2)	0.021(2)	0.013(2)	0.005(2)
N(1)	2*i*	0.1641(5)	–0.0936(4)	–0.0520(3)	0.034(3)	0.033(2)	0.025(2)	0.009(2)	0.013(2)	0.003(2)
N(2)	2*i*	0.1212(5)	–0.1751(4)	–0.1543(3)	0.031(2)	0.038(2)	0.027(2)	0.014(2)	0.011(2)	0.005(2)
C(3)	2*i*	0.2361(6)	–0.2365(5)	–0.1616(4)	0.035(3)	0.034(3)	0.039(3)	0.012(2)	0.021(2)	0.012(2)
C(4)	2*i*	0.3606(6)	–0.1946(5)	–0.0610(4)	0.037(3)	0.051(3)	0.051(3)	0.028(3)	0.022(3)	0.015(3)
C(5)	2*i*	0.3118(6)	–0.1066(5)	0.0048(4)	0.029(3)	0.055(3)	0.035(3)	0.016(3)	0.009(2)	0.005(2)
C(6)	2*i*	0.2149(7)	–0.3302(5)	–0.2675(4)	0.052(4)	0.053(3)	0.049(3)	0.019(3)	0.028(3)	0.004(3)
N(11)	2*i*	0.1188(4)	0.2070(4)	–0.0059(3)	0.031(2)	0.029(2)	0.029(2)	0.003(2)	0.007(2)	0.009(2)
N(12)	2*i*	0.2553(5)	0.2919(4)	0.0730(3)	0.039(3)	0.028(2)	0.030(2)	0.007(2)	0.013(2)	0.011(2)
C(13)	2*i*	0.2875(6)	0.4178(5)	0.0517(4)	0.045(3)	0.033(3)	0.045(3)	0.009(3)	0.027(3)	0.015(2)
C(14)	2*i*	0.1669(7)	0.4143(5)	–0.0460(4)	0.048(4)	0.047(3)	0.056(3)	0.012(3)	0.012(3)	0.031(3)
C(15)	2*i*	0.0673(7)	0.2827(5)	–0.0786(5)	0.042(3)	0.047(3)	0.048(3)	0.006(3)	0.009(3)	0.023(3)
C(16)	2*i*	0.4341(7)	0.5267(5)	0.1279(4)	0.058(4)	0.040(3)	0.047(3)	–0.005(3)	0.018(3)	0.005(3)
N(21)	2*i*	0.3945(5)	–0.2211(3)	0.4483(3)	0.036(2)	0.025(2)	0.027(2)	0.004(2)	0.012(2)	0.004(2)
N(22)	2*i*	0.3880(4)	–0.3072(3)	0.5170(3)	0.037(3)	0.028(2)	0.029(2)	0.000(2)	0.016(2)	0.001(2)
C(23)	2*i*	0.3076(6)	–0.4365(5)	0.4615(4)	0.049(3)	0.028(3)	0.049(3)	0.004(3)	0.029(3)	0.001(2)
C(24)	2*i*	0.2566(6)	–0.4335(5)	0.3508(4)	0.046(3)	0.034(3)	0.032(3)	–0.005(3)	0.014(3)	–0.009(2)
C(25)	2*i*	0.3137(6)	–0.2996(5)	0.3465(4)	0.045(3)	0.040(3)	0.032(3)	0.012(3)	0.013(2)	0.007(2)
C(26)	2*i*	0.2869(8)	–0.5499(5)	0.5213(5)	0.094(5)	0.023(3)	0.067(4)	–0.001(3)	0.047(4)	0.003(3)
N(31)	2*i*	0.3380(4)	0.0361(4)	0.5842(3)	0.028(2)	0.032(2)	0.032(2)	0.014(2)	0.011(2)	0.010(2)
N(32)	2*i*	0.2210(4)	0.1037(3)	0.5478(3)	0.031(2)	0.027(2)	0.031(2)	0.009(2)	0.017(2)	0.009(2)
C(33)	2*i*	0.1523(5)	0.1315(4)	0.6217(4)	0.028(3)	0.028(3)	0.046(3)	0.007(2)	0.020(2)	0.004(2)
C(34)	2*i*	0.2281(6)	0.0783(5)	0.7113(4)	0.050(4)	0.050(3)	0.037(3)	0.016(3)	0.028(3)	0.012(2)
C(35)	2*i*	0.3390(6)	0.0213(5)	0.6851(4)	0.041(3)	0.044(3)	0.030(3)	0.010(3)	0.014(2)	0.016(2)
C(36)	2*i*	0.0210(6)	0.2037(5)	0.5992(4)	0.043(3)	0.037(3)	0.063(4)	0.011(3)	0.032(3)	0.011(3)

References

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