Supplementary Material (ESI) for Chemical Communications

Electronic Supplementary Information

Dynamic ligation at the first amine-coordinated iron hydrogenase active site mimic

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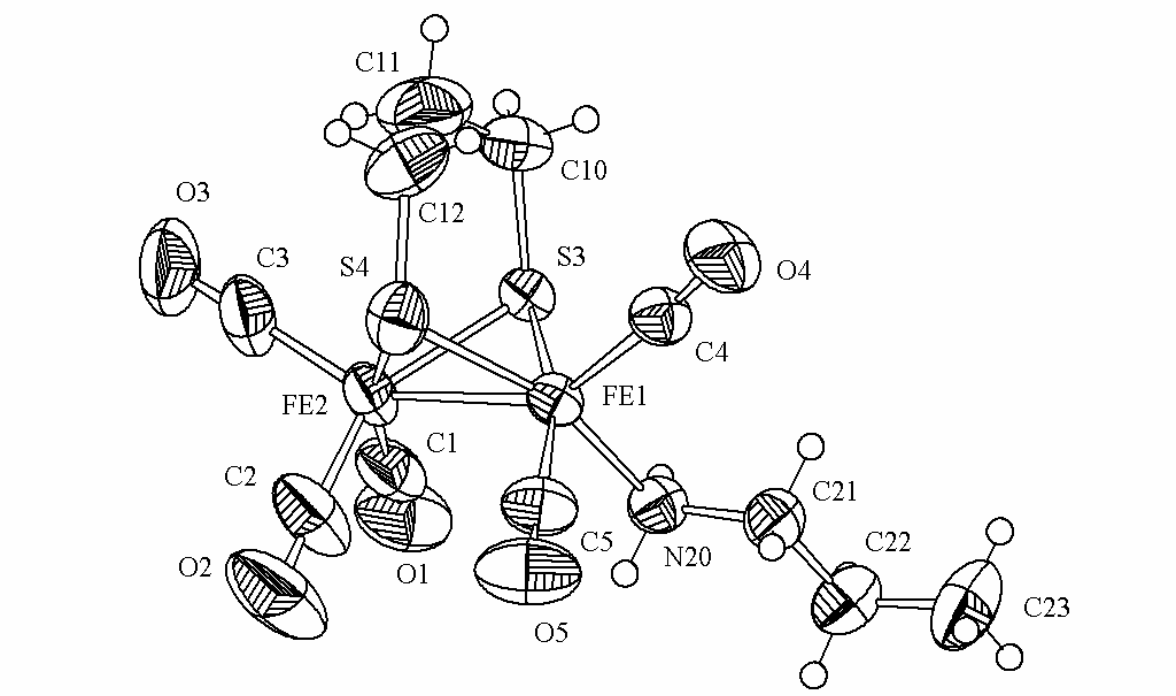
X-ray analysis of 1:



Displacement ellipsoids draw at 50% probability level. Hydrogen atoms shown as small circles draw with arbitrary radii.

Single crystal X-ray diffraction data was recorded with an Oxford Diffraction Excalibur diffractometer equipped with a Sapphire-III CCD detector, using Mo-radiation source

(λ= 0.71073Å). Data processing by the diffractometer software [1]. The structure was solved by conventional direct methods using SHELXS97 [2] giving electron density maps where most of the non-hydrogen atoms could be resolved. The rest of the non-hydrogen atoms were located from difference electron density maps and the structure model was refined with full matrix least square calculations on F^2 using the program SHELXL97-2 [3]. All non-hydrogen atoms were refined with anisotropic displacement parameters and the hydrogen's, which were placed at geometrically calculated positions and let to ride on the atoms they were bonded to, were given isotropic displacement parameters calculated as $\xi \cdot U_{eq.}$ for the non-hydrogen atoms with ξ = 1.5 for methyl hydrogen's ($-CH_3$) and ξ=1.2 for methylenic ($-CH_2-$) hydrogen's. The picture is made with the program Diamond [4].

Selected crystal data are given in Table 1.

References

1) Oxford diffraction, CrysAlis Pro, version 1.71.229.9
2) Sheldrick, G.M. (1990). Acta Cryst. A46, 467-473
3) Sheldrick, G.M. (1997). Computer program for the refinement of crystal structures, Göttingen, Germany
4) DIAMOND - Visual Crystal Structure Information System, CRYSTAL IMPACT, Postfach 1251, D-53002 Bonn, Germany

Table 1. Selected crystal data for **1**.

code	Ls95
Empirical formula	$C_{11}\ H_{15}\ Fe_2\ N_5\ O_5\ S_2$
Temperature, K	293(2)
Fw	417.06
Crystal system	Monoclinic
Space group	P 2_1/a
A, Å	11.279(1)
B, Å	13.631(1)
C, Å	12.135(1)
α, °	90
β, °	113.86(1)
γ, °	90
V, $Å^3$	1706.4(3)
Z	4
ρ_{calc}, g cm^{-3}	1.623
μ (MoKα), (mm^{-1})	1.960

N(meas), N(uniq), R(int)	28673, 5528, 0.0619
N(obs), N(par), S (GoF)	3597, 190, 1.06
R1, wR2 both with ($I > 2\sigma(I)$)	0.0493, 0.1320
R1, wR2 (all data)	0.0776, 0.1455
$\Delta\rho_{min}$, $\Delta\rho_{max}$ ($e/Å^3$)	-0.635 – 0.781

Complete cif file output from SHELXL97

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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc.
and is
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 factors based on ALL data will be even larger.
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C5 Fe1 Fe2 C1 -101.23(15) . . . . ?
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S3 Fe1 Fe2 C1 72.83(11) . . . . ?
C4 Fe1 Fe2 C3 -15.6(3) . . . . ?
C5 Fe1 Fe2 C3 119.1(2) . . . . ?
N20 Fe1 Fe2 C3 -145.3(2) . . . . ?
S4 Fe1 Fe2 C3 42.5(2) . . . . ?
S3 Fe1 Fe2 C3 -66.8(2) . . . . ?
C4 Fe1 Fe2 S3 51.25(15) . . . . ?
C5 Fe1 Fe2 S3 -174.06(10) . . . . ?
N20 Fe1 Fe2 S3 -78.44(7) . . . . ?
S4 Fe1 Fe2 S3 109.32(4) . . . . ?
C4 Fe1 Fe2 S4 -58.07(15) . . . . ?
C5 Fe1 Fe2 S4 76.62(10) . . . . ?
N20 Fe1 Fe2 S4 172.24(7) . . . . ?
S3 Fe1 Fe2 S4 -109.32(4) . . . . ?
C4 Fe1 S3 C10 -45.35(16) . . . . ?
C5 Fe1 S3 C10 126.0(4) . . . . ?
N20 Fe1 S3 C10 -144.59(14) . . . . ?
S4 Fe1 S3 C10 55.39(13) . . . . ?
Fe2 Fe1 S3 C10 107.46(13) . . . . ?
C4 Fe1 S3 Fe2 -152.81(10) . . . . ?
C5 Fe1 S3 Fe2 18.6(3) . . . . ?
N20 Fe1 S3 Fe2 107.95(6) . . . . ?
S4 Fe1 S3 Fe2 -52.07(3) . . . . ?
C2 Fe2 S3 C10 -130.9(3) . . . . ?
C1 Fe2 S3 C10 139.30(17) . . . . ?
C3 Fe2 S3 C10 44.05(18) . . . . ?
S4 Fe2 S3 C10 -55.20(12) . . . . ?
Fe1 Fe2 S3 C10 -105.99(12) . . . . ?
C2 Fe2 S3 Fe1 -24.9(3) . . . . ?
C1 Fe2 S3 Fe1 -114.71(12) . . . . ?
C3 Fe2 S3 Fe1 150.05(14) . . . . ?
S4 Fe2 S3 Fe1 50.79(3) . . . . ?
C4 Fe1 S4 C12 43.43(17) . . . . ?
C5 Fe1 S4 C12 142.01(18) . . . . ?
N20 Fe1 S4 C12 -128.1(2) . . . . ?
S3 Fe1 S4 C12 -55.06(15) . . . . ?
Fe2 Fe1 S4 C12 -106.53(16) . . . . ?
C4 Fe1 S4 Fe2 149.96(10) . . . . ?
C5 Fe1 S4 Fe2 -111.46(10) . . . . ?
N20 Fe1 S4 Fe2 -21.5(2) . . . . ?
S3 Fe1 S4 Fe2 51.47(2) . . . . ?
C2 Fe2 S4 C12 -156.7(2) . . . . ?
C1 Fe2 S4 C12 112.8(4) . . . . ?
C3 Fe2 S4 C12 -53.44(18) . . . . ?
S3 Fe2 S4 C12 54.54(15) . . . . ?
Fe1 Fe2 S4 C12 105.86(15) . . . . ?
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C2 Fe2 S4 Fe1 97.44(17) . . . . ?
C1 Fe2 S4 Fe1 7.0(3) . . . . ?
C3 Fe2 S4 Fe1 -159.30(11) . . . . ?
S3 Fe2 S4 Fe1 -51.31(3) . . . . ?
Fe1 S3 C10 C11 -60.4(4) . . . . ?
Fe2 S3 C10 C11 15.3(4) . . . . ?
S3 C10 C11 C12 51.4(6) . . . . ?
C10 C11 C12 S4 -51.6(6) . . . . ?
Fe1 S4 C12 C11 60.5(4) . . . . ?
Fe2 S4 C12 C11 -14.7(4) . . . . ?
C2 Fe2 C1 O1 150(9) . . . . ?
C3 Fe2 C1 O1 46(9) . . . . ?
S3 Fe2 C1 O1 -63(9) . . . . ?
S4 Fe2 C1 O1 -120(9) . . . . ?
Fe1 Fe2 C1 O1 -114(9) . . . . ?
C1 Fe2 C2 O2 38(10) . . . . ?
C3 Fe2 C2 O2 135(10) . . . . ?
S3 Fe2 C2 O2 -50(10) . . . . ?
S4 Fe2 C2 O2 -125(10) . . . . ?
Fe1 Fe2 C2 O2 -70(10) . . . . ?
C2 Fe2 C3 O3 -79(16) . . . . ?
C1 Fe2 C3 O3 15(16) . . . . ?
S3 Fe2 C3 O3 104(16) . . . . ?
S4 Fe2 C3 O3 -169(16) . . . . ?
Fe1 Fe2 C3 O3 157(16) . . . . ?
C5 Fe1 C4 O4 -117(3) . . . . ?
N20 Fe1 C4 O4 150(3) . . . . ?
S4 Fe1 C4 O4 -27(3) . . . . ?
S3 Fe1 C4 O4 60(3) . . . . ?
Fe2 Fe1 C4 O4 20(3) . . . . ?
C4 Fe1 C5 O5 71(11) . . . . ?
N20 Fe1 C5 O5 171(11) . . . . ?
S4 Fe1 C5 O5 -30(11) . . . . ?
S3 Fe1 C5 O5 -100(10) . . . . ?
Fe2 Fe1 C5 O5 -84(11) . . . . ?
C4 Fe1 N20 C21 23.5(2) . . . . ?
C5 Fe1 N20 C21 -75.5(2) . . . . ?
S4 Fe1 N20 C21 -164.97(17) . . . . ?
S3 Fe1 N20 C21 122.6(2) . . . . ?
Fe2 Fe1 N20 C21 176.68(19) . . . . ?
Fe1 N20 C21 C22 178.2(2) . . . . ?
N20 C21 C22 C23 177.0(3) . . . . ?

_diffrn_measured_fraction_theta_max    0.857
_diffrn_reflns_theta_full              33.07
_diffrn_measured_fraction_theta_full   0.857
_refine_diff_density_max    0.781
_refine_diff_density_min   -0.635
_refine_diff_density_rms    0.087
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Supplementary Material (ESI) for Chemical Communications