Title: Data to support study of Di-Iron(II) [2+2] Helicates of Bis-(Dipyrazolylpyridine) Ligands – the Influence of the Ligand Linker Group on Spin State Properties

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Publication Year: 2022

Description: A diiron(II) complex has been crystallised in three different helicate conformations, which differ in the torsions of the butane-1,4-diyl ligand linker groups. The crystals exhibit a range of spin state properties, including stepwise spin-crossover of the two iron atoms. A related ligand with a rigid pyrid-2,6-diyl spacer forms more a distorted, high-spin diiron(II) helicate structure.

Cite as: Kulmaczewski, Rafal, Armstrong, Isaac T., Catchpole, Pip, Ratcliffe, Emily S. J., Vasili, Hari Babu, Warriner, Stuart L., Cespedes, Oscar and Halcrow, Malcolm A. (2022). Data to support study of Di-Iron(II) [2+2] Helicates of Bis-(Dipyrazolylpyridine) Ligands – the Influence of the Ligand Linker Group on Spin State Properties. University of Leeds. [Dataset] https://doi.org/10.5518/1205.

Related publication: Kulmaczewski, Rafal, Armstrong, Isaac T., Catchpole, Pip, Ratcliffe, Emily S. J., Vasili, Hari Babu, Warriner, Stuart L., Cespedes, Oscar and Halcrow, Malcolm A. (2022). Di-Iron(II) [2+2] Helicates of Bis-(Dipyrazolylpyridine) Ligands – the Influence of the Ligand Linker Group on Spin State Properties. *Chemistry – a European Journal*, doi: 10.1002/chem.202202578.

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2. TERMS OF USE

3. PROJECT AND FUNDING INFORMATION

Title: Understanding and Engineering Function in Switchable Molecular Crystals
Dates: 2013-2018
Funding organisation: EPSRC
Grant no.: EP/K012576/1

Title: Metallo-Supramolecular and Functional Complexes
Dates: 2016-2018
Funding organisation: Diamond Light Source
Grant no.: MT-15059.

4. CONTENTS

The dataset contains data for this study:

Elemental microanalyses (*microanalysis.zip*).

Electrospray mass spectra of the new ligands and complexes, including simulations of iron complex peaks containing overlapping monoanion and dianion components (*ESMS.zip*).

^{1}H and ^{13}C NMR spectra of the new ligands, and paramagnetic ^{1}H NMR spectra of the complexes (raw and processed data – *NMR*.zip).

X-ray powder diffraction data (measured and simulated – *XRPD*.zip).

Solid state magnetic susceptibility measurements (raw and processed data – *SQUID*.zip).

Density functional calculations (*SPARTAN* files – *DFT*.zip).

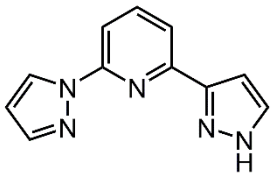
X-ray Crystallographic data (*crystal*.zip):

- Structure of α-1,3-bpp (CCDC 2169630).
- Structure of β-1,3-bpp (preliminary structure solution – no CCDC number).
- Structure of **1[BF$_4$]$_4$**·*n*Me$_2$CO at 250 K (CCDC 2169631).
- Structure of **1[BF$_4$]$_4$**·*n*Me$_2$CO at 100 K (CCDC 2169632).
- Structure of **1[BF$_4$]$_4$**·2MeCN·Et$_2$O at 125 K (CCDC 2169633).
- Structure of **1[BF$_4$]$_2$**·*m*MeNO$_2$ at 125 K (CCDC 2169634).
- Structure of **1[BF$_4$]$_2$**·2MeNO$_2$ at 125 K (CCDC 2169635).
- Structure of **2[BF$_4$]$_4$**·MeNO$_2$·Et$_2$O at 120 K (preliminary structure solution – no CCDC number).
- Structure of **2[ClO$_4$]$_2$** at 120 K (CCDC 2169636).
- Structure of **2[ClO$_4$]$_4$**·3MeNO$_2$·0.75H$_2$O at 120 K (CCDC 2169637).

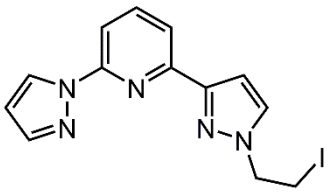
5. METHODS

Full details are provided in the related publication, listed above.

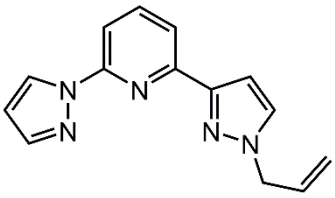
Ligands and other organic compounds referred to in this dataset



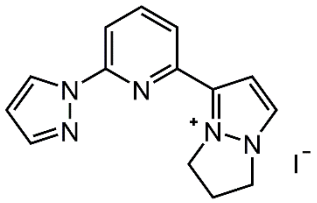
1,3-bpp
2-(Pyrazol-1-yl)-6-(1*H*-pyrazol-3-yl)pyridine
$C_{11}H_9N_5$



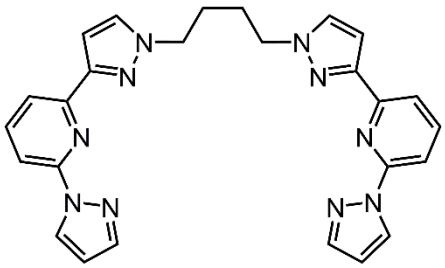
1-(3-{2-[Pyrazol-1-yl]pyrid-6-yl}pyrazol-1-yl)-2-iodoethane
$C_{13}H_{12}IN_5$



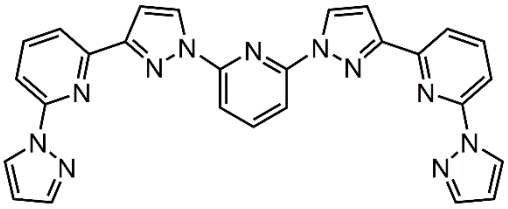
3-(3-{2-[Pyrazol-1-yl]pyrid-6-yl}pyrazol-1-yl)prop-1-ene
$C_{14}H_{13}N_5$



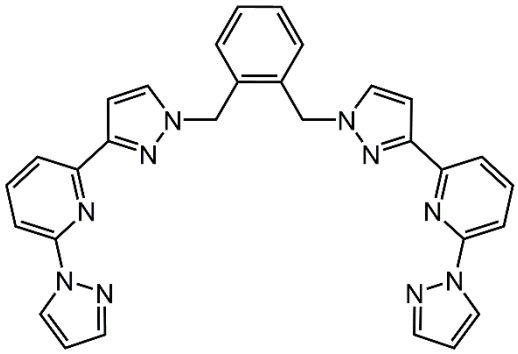
5-{2-[pyrazol-1-yl]pyrid-6-yl}-2,3-dihydro-pyrazolo[1,2-a]pyrazolium iodide
$C_{14}H_{14}IN_5$



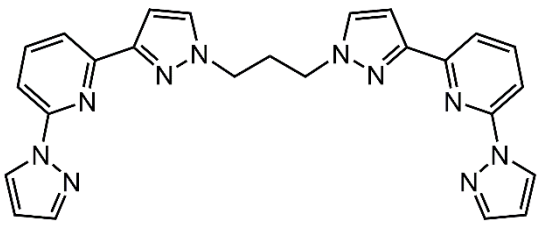
L^1
1,4-*Bis*(3-{2-[pyrazol-1-yl]pyrid-6-yl}pyrazol-1-yl)-butane
$C_{26}H_{24}N_{10}$



L^2
2,6-*Bis*(3-{2-[pyrazol-1-yl]pyrid-6-yl}pyrazol-1-yl)-pyridine
$C_{27}H_{19}N_{11}$

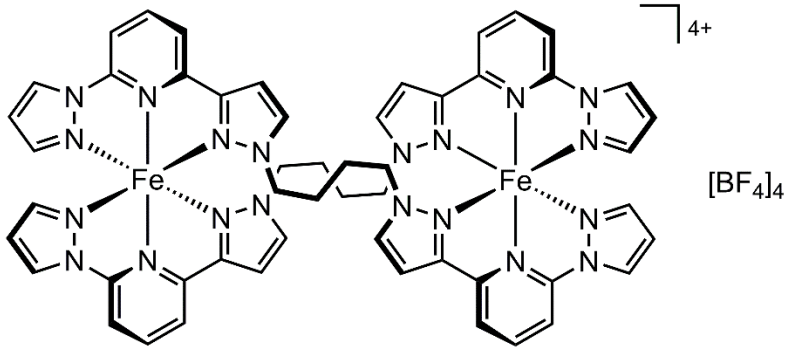


L^3
1,2-*Bis*(3-{2-[pyrazol-1-yl]pyrid-6-yl}pyrazol-1-ylmethyl)-benzene
$C_{30}H_{24}N_{10}$



L^4
1,3-*Bis*(3-{2-[pyrazol-1-yl]pyrid-6-yl}pyrazol-1-yl)-propane
$C_{25}H_{22}N_{10}$

Metal complexes referred to in this dataset

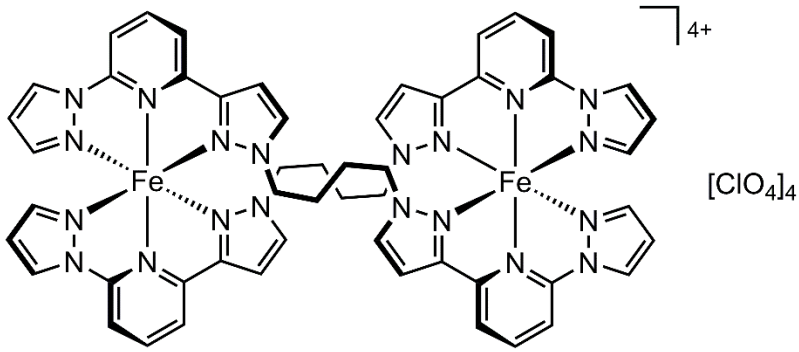


1[BF$_4$]$_4$

[Fe$_2$(μ-*L*^{1})$_2$][BF$_4$]$_4$

Bis[1,4-*Bis*(3-{2-[pyrazol-1-yl]pyrid-6-yl}pyrazol-1-yl)butane]diiron(II) tetra[tetrafluoroborate]

$C_{52}H_{48}B_4F_{16}Fe_2N_{20}$

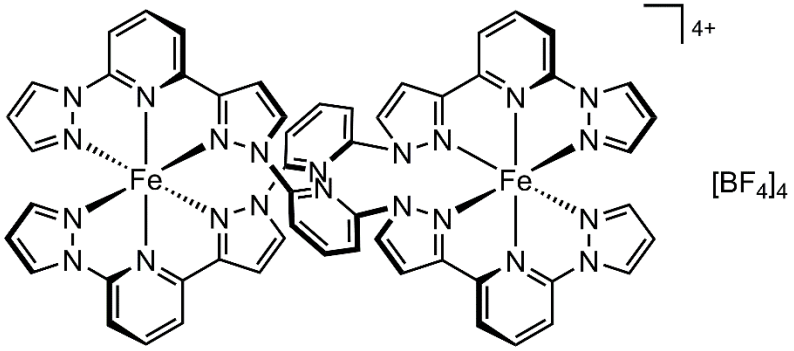


1[ClO$_4$]$_4$

[Fe$_2$(μ-*L*^{1})$_2$][ClO$_4$]$_4$

Bis[1,4-*Bis*(3-{2-[pyrazol-1-yl]pyrid-6-yl}pyrazol-1-yl)butane]diiron(II) tetraperchlorate

$C_{52}H_{48}Cl_4Fe_2N_{20}O_{16}$



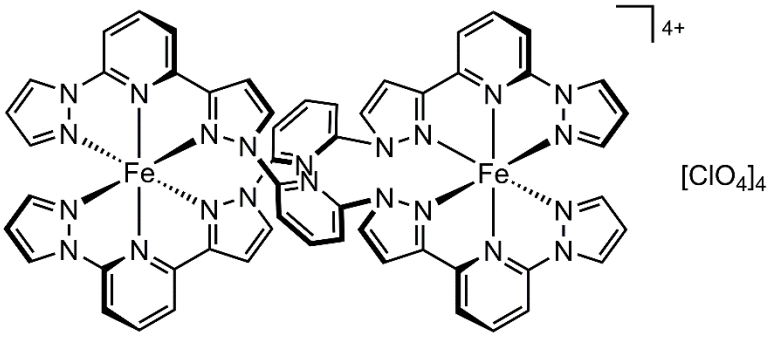
2[BF$_4$]$_4$

[Fe$_2$(μ-*L*^{2})$_2$][BF$_4$]$_4$

Bis[2,6-*Bis*(3-{2-[pyrazol-1-yl]pyrid-6-yl}pyrazol-1-yl)pyridine]diiron(II) tetra[tetrafluoroborate]

$C_{54}H_{38}B_4F_{16}Fe_2N_{22}$

Metal complexes referred to in this dataset (continued)

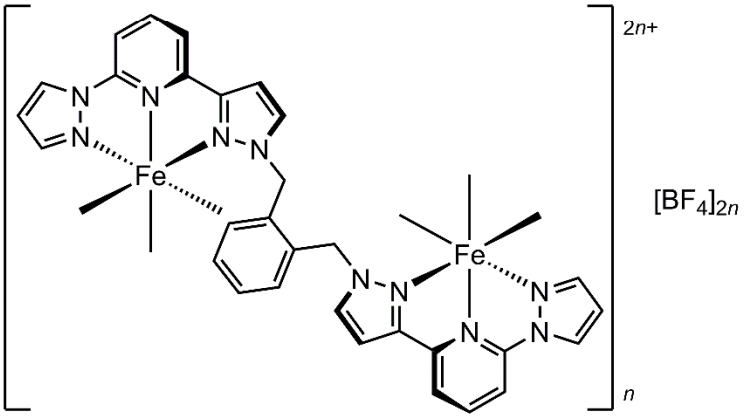


2[ClO$_4$]$_4$

[Fe$_2$(μ-L^2)$_2$][ClO$_4$]$_4$

Bis[2,6-*Bis*(3-{2-[pyrazol-1-yl]pyrid-6-yl}pyrazol-1-yl)pyridine]diiron(II) tetraperchlorate

$C_{54}H_{38}Cl_4Fe_2N_{22}O_{16}$

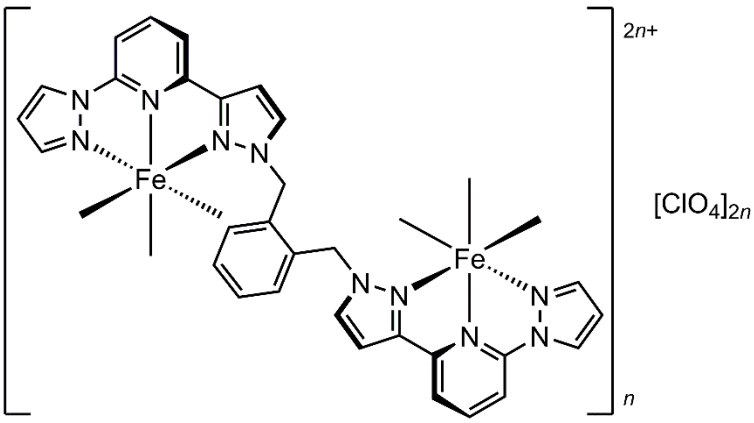


3[BF$_4$]$_2$

{[Fe(L^3)][BF$_4$]$_2$}$_n$

[1,2-*Bis*(3-{2-[pyrazol-1-yl]pyrid-6-yl}pyrazol-1-ylmethyl)benzene]iron(II) di[tetrafluoroborate]

{$C_{30}H_{24}B_2F_8FeN_{10}$}$_n$



3[ClO$_4$]$_2$

{[Fe(L^3)][ClO$_4$]$_2$}$_n$

[1,2-*Bis*(3-{2-[pyrazol-1-yl]pyrid-6-yl}pyrazol-1-ylmethyl)benzene]iron(II) diperchlorate

{$C_{30}H_{24}Cl_2FeN_{10}O_8$}$_n$