Supplementary Information

Compound 1

Figure S1. MALDI TOF/TOF-MS spectrum of 1.
Figure S2. $^1$H NMR spectra of 1.
SpinWorks 3: L. Bärisic 2603 50 mM
Figure S3. $^{13}$C NMR spectra of 1.
SpinWorks 3: L. Barisic 2603 50 mM
SpinWorks 3: L. Barisic 2603 50 mM
**Figure S4.** $^1$H NMR titration of compound 1 with DMSO in CDCl$_3$. 

SpinWorks 3: L. Barisic 2603 CDC13
SpinWorks 3: L. Barisic 2603 CDCl3 + 10 uL DMSO
SpinWorks 3: L. Barisic 2603 CDCl3 + 40 uL DMSO
SpinWorks 3: L. Barisic 2603 CDCl3 + 50 uL DMSO
SpinWorks 3: L. Barisic 2603 CDCl3 + 70 uL DMSO
SpinWorks 3: L. Barisic 2603 CDCl3 + 110 uL DMSO
SpinWorks 3: L. Barisic 2603 CDCl3 + 430 uL DMSO
SpinWorks 3: L. Barisic 2603 CDCl3 + 630 uL DMSO
Figure S5. Variable-temperature $^1$H NMR spectra of 1.
Compound 2

Figure S6. MALDI TOF/TOF-MS spectrum of 2.
Figure S7. $^1$H NMR spectra of 2.
SpinWorks 3: L. Barisic 2604_CDCl3
Figure S8. $^{13}$C NMR spectra of 2.

SpinWorks 3: L. Barisic 2604_CDCl3
SpinWorks 3: L. Barisic 2604_CDCl3
Figure S9. $^1$H NMR titration of compound 2 with DMSO in CDCl$_3$. 

SpinWorks 3: L. Barisic 2604 25 mM CDCl$_3$
SpinWorks 3: L. Barisic 2604 25 mM CDCl3 + 20uL DMSO
SpinWorks 3: L. Barisic 2604 25 mM CDCl3 + 30uL DMSO
SpinWorks 3: L. Barisic 2604 25 mM CDCl3 + 90 uL DMSO
SpinWorks 3: L. Barisic 2604 25 mM CDCl3 + 130 uL DMSO
SpinWorks 3: L. Barisic 2604 25 mM CDCl3 + 180 uL DMSO
SpinWorks 3: L. Barisic 2604 25 mM CDCl3 + 630 uL DMSO
Figure S10. Variable-temperature $^1$H NMR spectra of 2.
Compound 3

Figure S11. MALDI TOF/TOF-MS spectrum of 3.
Figure S12. $^1$H NMR spectra of 3.
SpinWorks 3: L. Barisic 2601 -45°C
SpinWorks 3: L. Barisic 2601 -45 ºC
Figure S13. $^{13}$C NMR spectra of 3.
SpinWorks 3: L. Barisic 2601_CDCl3 -45 ◦C
SpinWorks 3: L. Barisic 2601 CDCI3 -45 °C
Figure S14. $^1$H NMR titration of compound 3 with DMSO in CDCl$_3$. 

SpinWorks 3: L. Barisic 2601 25 mM
SpinWorks 3: L. Barisic 2601 25 mM + 10 uL DMSO
SpinWorks 3: L. Barisic 2601 25 mM + 50 uL DMSO
SpinWorks 3: L. Barisic 2601 25 mM + 130 uL DMSO
SpinWorks 3: L. Barisic 2601 25 mM + 280 uL DMSO
SpinWorks 3: L. Barisic 2601 25 mM + 430 uL DMSO
SpinWorks 3: L. Barisic 2601 25 mM + 630 uL DMSO
Figure S15. Variable-temperature $^1\text{H}$ NMR spectra of 3.
**Compound 4**

**Figure S16.** MALDI TOF/TOF-MS spectrum of 4.
Figure S17. $^1$H NMR spectra of 4.

SpinWorks 3: L. Barisic 2602 50 mM
SpinWorks 3: L. Barisic 2602 50 mM
SpinWorks 3: L. Barisic 2602 50 mM
SpinWorks 3: L. Barisic 2602 50 mM
Figure S18. $^{13}$C NMR spectra of 4.

SpinWorks 3: L. Barisic 2602 50 mM
Figure S19. $^1$H NMR titration of compound 1 with DMSO in CDCl$_3$. 

SpinWorks 3: L. Barisic 2602 CDCl3
SpinWorks 3: L. Barisic 2602 CDCl3 + 30 uL DMSO
SpinWorks 3: L. Barisic 2602 CDCl3 + 50 uL DMSO

PPM

8.4  8.0  7.6  7.2  6.8  6.4  6.0  5.6  5.2  4.8  4.4  4.0  3.6  3.2  2.8  2.4  2.0  1.6  1.2  0.8  0.4  0.0
SpinWorks 3: L. Barisic 2602 CDCl3 + 70 uL DMSO
SpinWorks 3: L. Barisic 2602 CDCl₃ + 110 μL DMSO
SpinWorks 3: L. Barisic 2602 CDCl3 + 130 uL DMSO
SpinWorks 3: L. Barisic 2602 CDCl3 + 280 uL DMSO
SpinWorks 3: L. Barisic 2602 CDCL3 + 430 uL DMSO
SpinWorks 3: L. Barisic 2602 CDCl$_3$ + 630 uL DMSO
Figure S20. Variable-temperature $^1$H NMR spectra of 4.
SpinWorks 3: L. Barisic 2602 -15 C
**Figure S21.** CheckCIF_PLATON report of compound 3.

**checkCIF/PLATON report**

Structure factors have been supplied for datablock(s) 1

**THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.**

No syntax errors found.  CIF dictionary  Interpreting this report

**Datablock: I**

<table>
<thead>
<tr>
<th>Bond precision:</th>
<th>C-C = 0.0140 Å</th>
<th>Wavelength=1.54179</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>a=10.951(5)</td>
<td>b=8.571(5)</td>
<td>c=11.944(5)</td>
</tr>
<tr>
<td>alpha=90</td>
<td>beta=107.315(5)</td>
<td>gamma=90</td>
</tr>
<tr>
<td>Temperature:</td>
<td>293 K</td>
<td></td>
</tr>
<tr>
<td>Volume:</td>
<td>Calculated</td>
<td>Reported</td>
</tr>
<tr>
<td>1070.3(9)</td>
<td>1079.3(9)</td>
<td></td>
</tr>
<tr>
<td>Space group:</td>
<td>P 21</td>
<td>P 1 2 1 1</td>
</tr>
<tr>
<td>Hall group:</td>
<td>P 2yb</td>
<td>7</td>
</tr>
<tr>
<td>Molecular formula</td>
<td>C22 H28 Fe N2 O5</td>
<td>C22 H28 Fe1 N2 O5</td>
</tr>
<tr>
<td>Mr</td>
<td>456.31</td>
<td>456.31</td>
</tr>
<tr>
<td>D, g cm-3</td>
<td>1.416</td>
<td>1.416</td>
</tr>
<tr>
<td>Z</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Mu (mm-1)</td>
<td>5.954</td>
<td>5.954</td>
</tr>
<tr>
<td>F000</td>
<td>480.0</td>
<td>480.0</td>
</tr>
<tr>
<td>F000’</td>
<td>479.11</td>
<td></td>
</tr>
<tr>
<td>h,k,lmax</td>
<td>13,10,15</td>
<td>13,10,14</td>
</tr>
<tr>
<td>Nref</td>
<td>4462[2385]</td>
<td>3439</td>
</tr>
<tr>
<td>Tmin, T’max</td>
<td>0.579, 0.551</td>
<td>0.291, 1.000</td>
</tr>
<tr>
<td>Tmin’</td>
<td>0.525</td>
<td></td>
</tr>
</tbody>
</table>

Correction method= MULTI-SCAN

Data completeness= 1.44/0.77  Theta(max) = 75.860

R(reflections)= 0.1024(3108)  wR2(reflections)= 0.2806(3430)

S = 1.268  Npar= Npar = 271

The following ALERTS were generated. Each ALERT has the format test-name_ALERT_alert-type_alert-level. Click on the hyperlinks for more details of the test.
Alert level C

ABSTY02_ALERT_1_C An _exptl_absorp_correction_type has been given without a literature citation. This should be contained in the _exptl_absorp_process_details field.

Absorption correction given as multi-scan

RFACCD1_ALERT_3_C The value of the R factor is > 0.10
R factor given 0.102

RFACR01_ALERT_3_C The value of the weighted R factor is > 0.25
Weighted R factor given 0.281

PLAT084_ALERT_3_C High wR2 Value (i.e. > 0.25) 0.28 Why?
PLAT090_ALERT_3_C Poor Data / Parameter Ratio (E_max > 18) 0.68 Note
PLAT126_ALERT_4_C No _symmetry_space_group_name_Hall Given Please Do!
PLAT147_ALERT_3_C su on Symmetry Constrained Cell Angle(s) Please Check
PLAT242_ALERT_3_C Low Ueq as Compared to Neighbors for C12 Check
PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds 0.0140 Ang.
PLAT913_ALERT_3_C Low Friedel Pair Coverage Please Do!

Alert level G

PLAT005_ALERT_3_G No _lucl_refine_instructions_details in the CIF
PLAT007_ALERT_3_G Number of Unrefined Donor-H Atoms 1 Why?
PLAT072_ALERT_2_G SHELXL First Parameter in WGT Unusually Large 0.20 Why?
PLAT083_ALERT_1_G No su's on H-positions, refinement reported as mixed
PLAT199_ALERT_1_G Reported _cell_measurement_temperature (K) 293 Check
PLAT200_ALERT_1_G Reported _diffnr_ambient_temperature (K) 293 Check
PLAT791_ALERT_3_G The Model has Chirality at C7 Please Do!
PLAT912_ALERT_4_G Missing & of FCF Reflections Above STR/Law 0.600 31 Note

0 ALERT level A - Most likely a serious problem - resolve or explain
10 ALERT level C - Check. Ensure it is not caused by an omission or oversight
0 ALERT level G - General information/check it is not something unexpected

5 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
2 ALERT type 2 Indicator that the structure model may be wrong or deficient
6 ALERT type 3 Indicator that the structure quality may be low
3 ALERT type 4 Improvement, methodology, query or suggestion
2 ALERT type 5 Informative message, check
Publication of your CIF

You should attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the nature of your study may justify the reported deviations from journal submission requirements and the more serious of these should be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. *checkCIF* was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

If you wish to submit your CIF for publication in *Acta Crystallographica* Section C or E, you should upload your CIF via the web. If your CIF is to form part of a submission to another IUCr journal, you will be asked, either during electronic submission or by the Co-editor handling your paper, to upload your CIF via our web site.

---

**PLATON version of 05/02/2014; check.def file version of 05/02/2014**

Datacheck 1 - ellipsoid plot

![Graphical representation of molecule with atom labels and bond lines.](image-url)