# checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 1_AsF6_120K_SQUEEZE, 1_PF6_120K_SQUEEZE

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: 1_PF6_120K_SQUEEZE

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Bond precision: C-C = 0.0092 A Wavelength=0.71070

Cell: a=19.049(6) b=19.056(5) c=20.600(9)
alpha=90 beta=115.832(3) gamma=90
Temperature: 120 K

| | Calculated | Reported |
|---|---|---|
| Volume | 6731(4) | 6731(4) |
| Space group | C 2/c | C 2/c |
| Hall group | -C 2yc | -C 2yc |
| Moiety formula | C46 H52 Fe2 N20, 4(F6 P) [+ solvent] | C46 H52 Fe2 N20 2+, (P F6 1-)4, (H2 O)1.5, (C2 H3 N)1.5 |
| Sum formula | C46 H52 F24 Fe2 N20 P4 [+ solvent] | C49 H59.50 F24 Fe2 N21.50 O1.50 P4 |
| Mr | 1576.66 | 1665.26 |
| Dx,g cm-3 | 1.556 | 1.643 |
| Z | 4 | 4 |
| Mu (mm-1) | 0.642 | 0.649 |
| F000 | 3184.0 | 3376.0 |
| F000’ | 3190.41 | |
| h,k,lmax | 24,24,26 | 24,24,26 |
| Nref | 7714 | 7550 |
| Tmin,Tmax | 0.897,0.937 | 0.900,0.962 |
| Tmin’ | 0.878 | |

Correction method= # Reported T Limits: Tmin=0.900 Tmax=0.962
AbsCorr = NUMERICAL

Data completeness= 0.979 Theta(max)= 27.469

R(reflections)= 0.0879( 5906) wR2(reflections)= 0.2286( 7550)

S = 1.131 Npar= 544

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 B

CHEMW03_ALERT_2_B WARNING: The ratio of given/expected molecular weight as calculated from the _atom_site* data lies outside the range 0.95 <> 1.05

From the CIF: _cell_formula_units_Z 4

From the CIF: _chemical_formula_weight 1665.26

TEST: Calculate formula weight from _atom_site_*

| atom | mass | num | sum |
|---|---|---|---|
| C | 12.01 | 46.00 | 552.51 |
| H | 1.01 | 52.00 | 52.42 |
| N | 14.01 | 20.00 | 280.14 |
| F | 19.00 | 24.00 | 455.95 |
| P | 30.97 | 4.00 | 123.90 |
| Fe | 55.85 | 2.00 | 111.69 |
| O | 16.00 | 0.00 | 0.00 |

Calculated formula weight 1576.60

## Alert level C

| | | |
|---|---|---|
| PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds ..... | 0.00922 | Ang. |
| PLAT906_ALERT_3_C Large K value in the Analysis of Variance ..... | 9.243 | Check |
| PLAT906_ALERT_3_C Large K value in the Analysis of Variance ..... | 2.778 | Check |
| PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min) | 9 | Note |
| PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.600 | 19 | Report |
| PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density. | 0 | Note |

## Alert level G

FORMU01_ALERT_2_G There is a discrepancy between the atom counts in the _chemical_formula_sum and the formula from the _atom_site* data.
Atom count from _chemical_formula_sum:C49 H59.5 F24 Fe2 N21.5 O1.5 P
Atom count from the _atom_site data: C46 H52 F24 Fe2 N20 P4

CELLZ01_ALERT_1_G Difference between formula and atom_site contents detected.

CELLZ01_ALERT_1_G ALERT: Large difference may be due to a symmetry error - see SYMMG tests

From the CIF: _cell_formula_units_Z 4

From the CIF: _chemical_formula_sum C49 H59.50 F24 Fe2 N21.50 O1.50

TEST: Compare cell contents of formula and atom_site data

| atom | Z*formula | cif sites | diff |
|---|---|---|---|
| C | 196.00 | 184.00 | 12.00 |
| H | 238.00 | 208.00 | 30.00 |
| F | 96.00 | 96.00 | 0.00 |
| Fe | 8.00 | 8.00 | 0.00 |
| N | 86.00 | 80.00 | 6.00 |
| O | 6.00 | 0.00 | 6.00 |
| P | 16.00 | 16.00 | 0.00 |

| | | |
|---|---|---|
| PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... | 26 | Report |
| PLAT014_ALERT_1_G N.O.K. _shelx_fab_checksum found in CIF ..... | Please | Check |
| PLAT041_ALERT_1_G Calc. and Reported SumFormula Strings Differ | Please | Check |
| PLAT042_ALERT_1_G Calc. and Reported MoietyFormula Strings Differ | Please | Check |
| PLAT044_ALERT_1_G Calculated and Reported Density Dx Differ by .. | 0.0871 | Check |
| PLAT068_ALERT_1_G Reported F000 Differs from Calcd (or Missing)... | Please | Check |
| PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large | 27.41 | Why ? |
| PLAT176_ALERT_4_G The CIF-Embedded .res File Contains SADI Records | 6 | Report |

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PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records          1 Report
PLAT244_ALERT_4_G Low    'Solvent' Ueq as Compared to Neighbors of         P1 Check
PLAT244_ALERT_4_G Low    'Solvent' Ueq as Compared to Neighbors of         P2 Check
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  2)..         86 % Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  3)..         86 % Note
PLAT432_ALERT_2_G Short Inter X...Y Contact  F3A    ..  C12     ..       2.87 Ang.
PLAT432_ALERT_2_G Short Inter X...Y Contact  F3A    ..  C13     ..       2.89 Ang.
PLAT432_ALERT_2_G Short Inter X...Y Contact  F1B    ..  C22     ..       2.96 Ang.
PLAT605_ALERT_4_G Largest Solvent Accessible VOID in the Structure        223 A**3
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............       252 Note
PLAT869_ALERT_4_G ALERTS Related to the use of SQUEEZE Suppressed           ! Info
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        136 Note
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   0 ALERT level A = Most likely a serious problem - resolve or explain
   1 ALERT level B = A potentially serious problem, consider carefully
   6 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  23 ALERT level G = General information/check it is not something unexpected

   7 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   8 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
   9 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
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# Datablock: 1_AsF6_120K_SQUEEZE

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Bond precision:  C-C = 0.0062 A  Wavelength=0.71070

| | | | |
|---|---|---|---|
| Cell: | a=19.410(3) | b=19.034(3) | c=20.772(5) |
| | alpha=90 | beta=115.723(2) | gamma=90 |
| Temperature: | 120 K | | |

| | Calculated | Reported |
|---|---|---|
| Volume | 6914(2) | 6914(2) |
| Space group | C 2/c | C 2/c |
| Hall group | -C 2yc | -C 2yc |
| Moiety formula | C46 H52 Fe2 N20, 4(As F6) [+ solvent] | C46 H52 Fe2 N20 2+, (As F6 1-)4, (H2 O)3, (C2 H3 N) |
| Sum formula | C46 H52 As4 F24 Fe2 N20 [+ solvent] | C48 H61 As4 F24 Fe2 N21 O3 |
| Mr | 1752.46 | 1847.55 |
| Dx,g cm-3 | 1.684 | 1.775 |
| Z | 4 | 4 |
| Mu (mm-1) | 2.433 | 2.441 |
| F000 | 3472.0 | 3680.0 |
| F000' | 3477.53 | |
| h,k,lmax | 25,24,26 | 25,24,26 |
| Nref | 7946 | 7923 |
| Tmin,Tmax | 0.568,0.845 | 0.601,0.921 |
| Tmin' | 0.557 | |

Correction method= # Reported T Limits: Tmin=0.601 Tmax=0.921
AbsCorr = NUMERICAL

Data completeness= 0.997 Theta(max)= 27.489

R(reflections)= 0.0520( 6650) wR2(reflections)= 0.1236( 7923)

S = 1.113 Npar= 633

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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.

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## Alert level B

CHEMW03_ALERT_2_B WARNING: The ratio of given/expected molecular weight as calculated from the _atom_site* data lies outside the range 0.95 <> 1.05

From the CIF: _cell_formula_units_Z 4
From the CIF: _chemical_formula_weight 1847.55
TEST: Calculate formula weight from _atom_site_*

| atom | mass | num | sum |
|---|---|---|---|
| C | 12.01 | 46.00 | 552.51 |
| H | 1.01 | 52.00 | 52.42 |
| N | 14.01 | 20.00 | 280.14 |
| O | 16.00 | 0.00 | 0.00 |
| F | 19.00 | 24.00 | 455.95 |
| Fe | 55.85 | 2.00 | 111.69 |
| As | 74.92 | 4.00 | 299.69 |

Calculated formula weight 1752.40

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## Alert level C

PLAT230_ALERT_2_C Hirshfeld Test Diff for C3 -- C4 .. 5.2 s.u.
PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00622 Ang.
PLAT906_ALERT_3_C Large K value in the Analysis of Variance ..... 4.482 Check
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min) 10 Note
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.600 11 Report

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## Alert level G

FORMU01_ALERT_2_G There is a discrepancy between the atom counts in the _chemical_formula_sum and the formula from the _atom_site* data.
Atom count from _chemical_formula_sum:C48 H61 As4 F24 Fe2 N21 O3
Atom count from the _atom_site data: C46 H52 As4 F24 Fe2 N20

CELLZ01_ALERT_1_G Difference between formula and atom_site contents detected.
CELLZ01_ALERT_1_G ALERT: Large difference may be due to a symmetry error - see SYMMG tests

From the CIF: _cell_formula_units_Z 4
From the CIF: _chemical_formula_sum C48 H61 As4 F24 Fe2 N21 O3
TEST: Compare cell contents of formula and atom_site data

| atom | Z*formula | cif sites | diff |
|---|---|---|---|
| C | 192.00 | 184.00 | 8.00 |
| H | 244.00 | 208.00 | 36.00 |
| As | 16.00 | 16.00 | 0.00 |
| F | 96.00 | 96.00 | 0.00 |
| Fe | 8.00 | 8.00 | 0.00 |

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           N         84.00     80.00    4.00
           O         12.00      0.00   12.00
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ...         37 Report
PLAT014_ALERT_1_G N.O.K. _shelx_fab_checksum found in CIF ........     Please Check
PLAT041_ALERT_1_G Calc. and Reported SumFormula    Strings  Differ     Please Check
PLAT042_ALERT_1_G Calc. and Reported MoietyFormula Strings  Differ     Please Check
PLAT044_ALERT_1_G Calculated and Reported Density Dx  Differ by ..     0.0914 Check
PLAT068_ALERT_1_G Reported F000 Differs from Calcd (or Missing)...     Please Check
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT  Unusually Large      16.34 Why ?
PLAT171_ALERT_4_G The CIF-Embedded .res File Contains EADP Records          2 Report
PLAT176_ALERT_4_G The CIF-Embedded .res File Contains SADI Records          6 Report
PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records          1 Report
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  2)..         86 % Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  3)..         86 % Note
PLAT432_ALERT_2_G Short Inter X...Y Contact  F2C    ..  C22     ..       2.94 Ang.
PLAT432_ALERT_2_G Short Inter X...Y Contact  F3B    ..  C12     ..       2.90 Ang.
PLAT432_ALERT_2_G Short Inter X...Y Contact  F3B    ..  C13     ..       2.93 Ang.
PLAT432_ALERT_2_G Short Inter X...Y Contact  F4B    ..  C8      ..       2.94 Ang.
PLAT605_ALERT_4_G Largest Solvent Accessible VOID in the Structure        229 A**3
PLAT811_ALERT_5_G No ADDSYM Analysis: Too Many Excluded Atoms ....          ! Info
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............        272 Note
PLAT869_ALERT_4_G ALERTS Related to the use of SQUEEZE Suppressed           ! Info
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600          4 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          1 Note
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0 **ALERT level A** = Most likely a serious problem - resolve or explain
1 **ALERT level B** = A potentially serious problem, consider carefully
5 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
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10 ALERT type 2 Indicator that the structure model may be wrong or deficient
5 ALERT type 3 Indicator that the structure quality may be low
8 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

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# checkCIF publication errors

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**Alert level A**

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PUBL012_ALERT_1_A  _publ_section_abstract is missing.
           Abstract of paper in English.
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1 **ALERT level A** = Data missing that is essential or data in wrong format
0 **ALERT level G** = General alerts. Data that may be required is missing

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**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 level A alerts remain, which you believe to be justified deviations, and you intend to submit this CIF for publication in a journal, you should additionally insert an explanation in your CIF using the Validation Reply Form (VRF) below. This will allow your explanation to be considered as part of the review process.

**Validation response form**

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```
# start Validation Reply Form
_vrf_PUBL012_GLOBAL
;
PROBLEM: _publ_section_abstract is missing.
RESPONSE: ...
;
# end Validation Reply Form
```

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 you wish to submit your CIF for publication in IUCrData 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.

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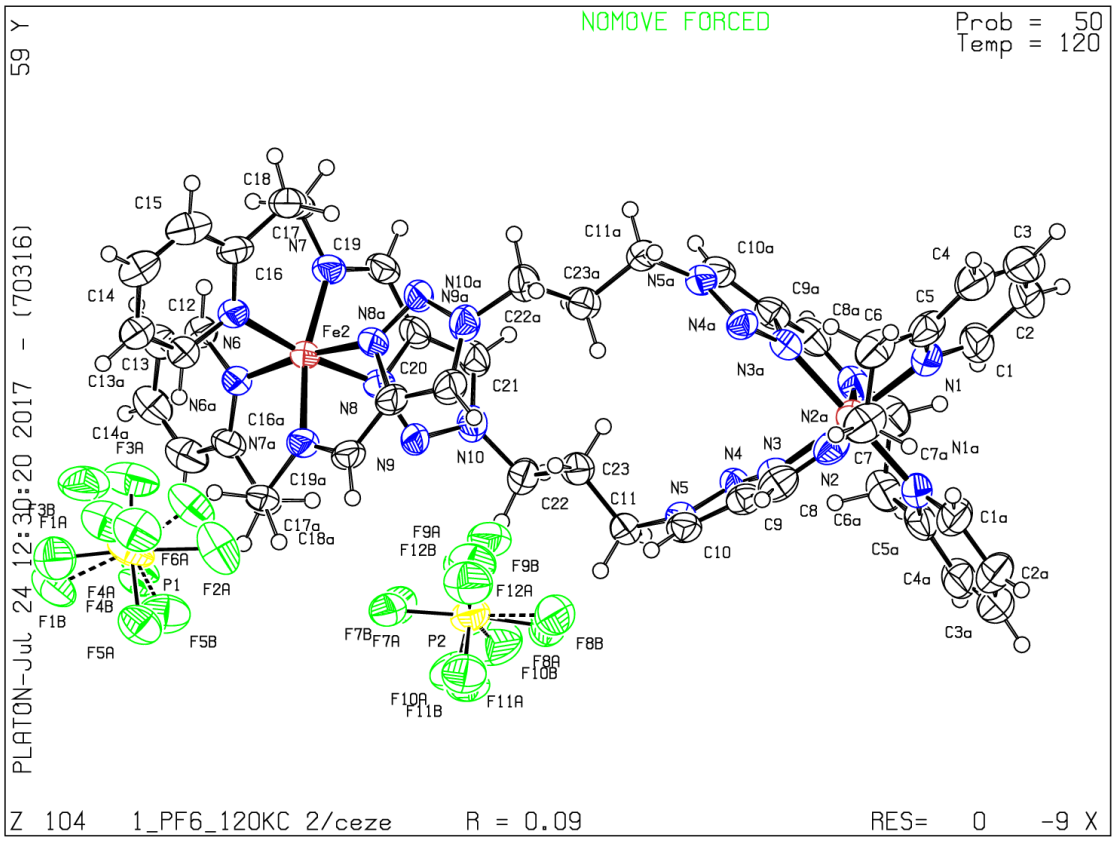
**PLATON version of 27/03/2017; check.def file version of 24/03/2017**

**Datablock 1_PF6_120K_SQUEEZE** - ellipsoid plot



**Datablock 1_AsF6_120K_SQUEEZE** - ellipsoid plot