checkCIF/PLATON report

Structure factors have been supplied for datablock(s) cage3

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: cage3

```
Bond precision:    C-C = 0.0104 A              Wavelength=0.68890

Cell:              a=20.9197(2)       b=14.8105(2)        c=21.4828(2)
                   alpha=90           beta=90             gamma=90
Temperature:       230 K

                   Calculated                 Reported
Volume             6656.04(13)                6656.04(13)
Space group        P n a 21                   P n a 21
Hall group         P 2c -2n                   P 2c -2n
Moiety formula     C69 H57 N8, 3(F6 P)        ?
Sum formula        C69 H57 F18 N8 P3          C69 H63 F18 N8 P3
Mr                 1433.14                    1439.18
Dx,g cm-3          1.430                      1.436
Z                  4                          4
Mu (mm-1)          0.172                      0.135
F000               2936.0                     2960.0
F000'              2938.85
h,k,lmax           31,22,31                   31,22,31
Nref               22952[ 11745]              20389
Tmin,Tmax          0.981,0.987                0.968,1.000
Tmin'              0.976

Correction method= # Reported T Limits: Tmin=0.968 Tmax=1.000
AbsCorr = EMPIRICAL

Data completeness= 1.74/0.89        Theta(max)= 30.836

                                                 wR2(reflections)=
R(reflections)= 0.1627( 9779)                    0.4433( 20389)

S = 1.475                      Npar= 806
```

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 A**

PLAT051_ALERT_1_A Mu(calc) and Mu(CIF) Ratio Differs from 1.0 by .   27.50 %

Author Response: data collected with synchrotron radiation

PLAT234_ALERT_4_A Large Hirshfeld Difference C7   --C1   .   0.32 Ang.

Author Response: unresolved disorder

🟠 **Alert level B**

PLAT082_ALERT_2_B High R1 Value ....   0.16 Report

Author Response: pure organic cage. Thermal and positional disorder detected

PLAT084_ALERT_3_B High wR2 Value (i.e. > 0.25) ....   0.44 Report

Author Response: pure organic cage. Thermal and positional disorder detected

PLAT094_ALERT_2_B Ratio of Maximum / Minimum Residual Density ....   4.37 Report

Author Response: unresolved disorder and unassigned diffuse electron density

PLAT097_ALERT_2_B Large Reported Max. (Positive) Residual Density   2.64 eA-3

Author Response: unresolved disorder and unassigned diffuse electron density

PLAT201_ALERT_2_B Isotropic non-H Atoms in Main Residue(s) ....   1 Report
              C1A

Author Response: positional disorder, maximum of restrains needed

PLAT213_ALERT_2_B Atom C7   has ADP max/min Ratio .....   4.5 prolat

Author Response: thermal disorder

```
PLAT220_ALERT_2_B NonSolvent   Resd 1  C   Ueq(max)/Ueq(min) Range      7.6 Ratio
PLAT230_ALERT_2_B Hirshfeld Test Diff for    N5       --C63      .      13.3 s.u.
```

Author Response: dynamical disorder

```
PLAT230_ALERT_2_B Hirshfeld Test Diff for    C50       --C51      .       7.6 s.u.
```

Author Response: dynamical disorder

```
PLAT234_ALERT_4_B Large Hirshfeld Difference C2       --C19      .       0.28 Ang.
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_B High   'MainMol' Ueq as Compared to Neighbors of       C7 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_B High   'MainMol' Ueq as Compared to Neighbors of       C19 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_B High   'MainMol' Ueq as Compared to Neighbors of       C37 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_B High   'MainMol' Ueq as Compared to Neighbors of       C65 Check
```

Author Response: unresolved disorder

```
PLAT340_ALERT_3_B Low Bond Precision on  C-C Bonds ..............  0.01035 Ang.
```

Author Response: To improve the quality of the refinement, we measured 1(PF6)3 at synchrotron and at low temperature, with the intention to reduce the severe thermal disorder observed for sample at room temperature, but, unfortunately, without successful results. The best model has been obtained when the positional disorder of the carbon atoms was defined. Unfortunately, around one of the two highly disordered rings there is also a lot of thermal disorder. But we are confident the crystal is reliable as the quality of data is very high with a resolution up to 2theta of 60 degrees, measured with synchrotron light.

🟡 **Alert level C**

```
DIFMX02_ALERT_1_C  The maximum difference density is > 0.1*ZMAX*0.75
            The relevant atom site should be identified.
PLAT026_ALERT_3_C Ratio Observed / Unique Reflections (too) Low ..      48% Check
PLAT041_ALERT_1_C Calc. and Reported SumFormula    Strings  Differ   Please Check
PLAT043_ALERT_1_C Calculated and Reported Mol. Weight Differ by ..    6.04 Check
PLAT068_ALERT_1_C Reported F000 Differs from Calcd (or Missing)...   Please Check
PLAT213_ALERT_2_C Atom C8            has ADP max/min Ratio .....       3.9 prolat
```

Author Response: thermal disorder

```
PLAT213_ALERT_2_C Atom C19           has ADP max/min Ratio .....       3.1 prolat
```

Author Response: thermal disorder

```
PLAT222_ALERT_3_C NonSolvent Resd 1  H   Uiso(max)/Uiso(min) Range     6.6 Ratio
PLAT230_ALERT_2_C Hirshfeld Test Diff for    C15      --C16     .       5.5 s.u.
```

Author Response: dynamical disorder

```
PLAT230_ALERT_2_C Hirshfeld Test Diff for    C23      --C24     .       5.8 s.u.
```

Author Response: dynamical disorder

```
PLAT230_ALERT_2_C Hirshfeld Test Diff for    C58      --C59     .       6.2 s.u.
```

Author Response: dynamical disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference N3       --C43     .      0.17 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference N3       --C62     .      0.17 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference N4       --C48     .      0.21 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference C9       --C64     .      0.17 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference C17      --C65     .      0.17 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference C22      --C23     .      0.19 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference C27      --C28     .      0.17 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference C35      --C67     .      0.17 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference C38      --C39     .      0.16 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference C40      --C41     .      0.16 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference C44      --C45     .      0.19 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference C49      --C50     .      0.19 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference C51      --C52     .      0.19 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference C52      --C53     .      0.19 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference C55      --C56     .      0.18 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference C59      --C60     .      0.19 Ang.
```

Author Response: unresolved disorder

```
PLAT234_ALERT_4_C Large Hirshfeld Difference P4       --F9      .      0.23 Ang.
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of      C8 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of      C13 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of      C22 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of      C27 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of      C30 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of      C31 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of      C53 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of       C56 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of       C63 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of       C66 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of       C67 Check
```

Author Response: unresolved disorder

```
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of       C69 Check
```

Author Response: unresolved disorder

```
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of          N3 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of          N5 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of          N6 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         C10 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         C12 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         C17 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         C20 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         C21 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         C23 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         C35 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         C41 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         C55 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including       P1    0.133 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including       P3    0.197 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including       P4    0.174 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......    3.584 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600      40 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) .      13 Check
PLAT971_ALERT_2_C Check Calcd Resid. Dens.  1.34Ang From C53        2.16 eA-3
PLAT971_ALERT_2_C Check Calcd Resid. Dens.  0.11Ang From P3        1.64 eA-3
```

● 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:C69 H63 F18 N8 P3
          Atom count from the _atom_site data:  C69 H57 F18 N8 P3
ABSMU01_ALERT_1_G  Calculation of _exptl_absorpt_correction_mu
             not performed for this radiation type.
CELLZ01_ALERT_1_G Difference between formula and atom_site contents detected.
```

```
CELLZ01_ALERT_1_G WARNING: H atoms missing from atom site list. Is this intentional?
          From the CIF: _cell_formula_units_Z    4
          From the CIF: _chemical_formula_sum  C69 H63 F18 N8 P3
          TEST: Compare cell contents of formula and atom_site data

          atom    Z*formula  cif sites diff
          C        276.00    276.00    0.00
          H        252.00    228.00   24.00
          F         72.00     72.00    0.00
          N         32.00     32.00    0.00
          P         12.00     12.00    0.00
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite        10 Note
PLAT072_ALERT_2_G SHELXL First  Parameter in WGHT  Unusually Large      0.20 Report
PLAT092_ALERT_4_G Check: Wavelength Given is not Cu,Ga,Mo,Ag,In Ka   0.68890 Ang.
PLAT111_ALERT_2_G ADDSYM Detects New (Pseudo) Centre of Symmetry .        84 %Fit
PLAT113_ALERT_2_G ADDSYM Suggests Possible Pseudo/New  Space Group    Pnna Check
PLAT143_ALERT_4_G s.u. on c - Axis Small or Missing ..............   0.00020 Ang.
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records         2 Report
PLAT230_ALERT_2_G Hirshfeld Test Diff for    C4        --C37      .      7.9 s.u.
```

Author Response: dynamical disorder

```
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       P3 Check
PLAT244_ALERT_4_G Low    'Solvent' Ueq as Compared to Neighbors of       P4 Check
PLAT301_ALERT_3_G Main Residue  Disorder ..............(Resd  1  )       8% Note
PLAT432_ALERT_2_G Short Inter X...Y Contact  F12      ..C62      .     2.82 Ang.
                                             x,1+y,z  =    1_565 Check
PLAT432_ALERT_2_G Short Inter X...Y Contact  F17      ..C63      .     2.91 Ang.
                                             x,-1+y,z =    1_545 Check
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels .........        6 Note
PLAT773_ALERT_2_G Check long C-C Bond in CIF: C13      --C3A         1.77 Ang.
PLAT773_ALERT_2_G Check long C-C Bond in CIF: C31      --C6A         1.76 Ang.
PLAT773_ALERT_2_G Check long C-C Bond in CIF: C37      --C4A         1.74 Ang.
PLAT792_ALERT_1_G Model has Chirality at C19       (Polar  SPGR)        R Verify
PLAT802_ALERT_4_G CIF Input Record(s) with more than 80 Characters        1 Info
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............       10 Note
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary .   Please Do !
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600      432 Note
PLAT913_ALERT_3_G Missing # of Very Strong Reflections in FCF ....        2 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........      3.9 Low
PLAT954_ALERT_1_G Reported (CIF) and Actual (FCF) Kmax Differ by .        1 Units
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.        0 Info
```

```
  2 ALERT level A = Most likely a serious problem - resolve or explain
 15 ALERT level B = A potentially serious problem, consider carefully
 60 ALERT level C = Check. Ensure it is not caused by an omission or oversight
 31 ALERT level G = General information/check it is not something unexpected


 11 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 58 ALERT type 2 Indicator that the structure model may be wrong or deficient
 11 ALERT type 3 Indicator that the structure quality may be low
 28 ALERT type 4 Improvement, methodology, query or suggestion
  0 ALERT type 5 Informative message, check
```

It is advisable to 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 purpose of your study may justify the reported deviations and the more serious of these should normally 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.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 18/05/2022; check.def file version of 17/05/2022

Datablock cage3 - ellipsoid plot

NOMOVE FORCED

Prob = 50
Temp = 230

56 Y

PLATON-Jul 28 15:52:11 2022 - (180522)

Z 81   cage3   P n a 21   R = 0.16   RES= O -31 X