checkCIF/PLATON report

Structure factors have been supplied for datablock(s) shelx

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: shelx

```
Bond precision:   C-C = 0.0062 A           Wavelength=1.54184

Cell:              a=21.6107(1)      b=21.6107(1)      c=12.3823(1)
                   alpha=90          beta=90           gamma=90
Temperature:       100 K

                 Calculated                  Reported
Volume           5782.81(7)                  5782.81(6)
Space group      P 41 21 2                   P 41 21 2
Hall group       P 4abw 2nw                  P 4abw 2nw
Moiety formula   C25 H37 N3 O8 [+ solvent]   C25 H37 N3 O8 [+ solvent]
Sum formula      C25 H37 N3 O8 [+ solvent]   C25 H37 N3 O8 [+ solvent]
Mr               507.58                      539.63
Dx,g cm-3        1.166                       1.240
Z                8                           8
Mu (mm-1)        0.723                       0.777
F000             2176.0                      2328.6
F000'            2183.18
h,k,lmax         26,26,14                    26,18,14
Nref             5272[ 3014]                 5263
Tmin,Tmax        0.869,0.890                 0.878,0.897
Tmin'            0.869

Correction method= # Reported T Limits: Tmin=0.878 Tmax=0.897
AbsCorr = MULTI-SCAN

Data completeness= 1.75/1.00        Theta(max)= 68.050

                                                    wR2(reflections)=
R(reflections)= 0.0786( 5116)                       0.2362( 5263)
S = 1.034                  Npar= 330
```

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

```
ABSMU01_ALERT_1_B  The ratio of given/expected absorption coefficient lies
                   outside the range 0.95 <> 1.05
                Calculated value of mu =     0.723
                Value of mu given      =     0.777
CHEMW01_ALERT_1_B  The ratio of given/expected molecular weight as calculated
                from the _chemical_formula_sum lies outside
                the range 0.95 <> 1.05
                Calculated formula weight =   507.5826
                Formula weight given      =   539.6300
DIFMN02_ALERT_2_B  The minimum difference density is < -0.1*ZMAX*1.00
                _refine_diff_density_min given =     -1.276
                Test value =     -0.800
PLAT097_ALERT_2_B Large Reported Max.  (Positive) Residual Density       1.19 eA-3
PLAT098_ALERT_2_B Large Reported Min.  (Negative) Residual Density      -1.28 eA-3
PLAT230_ALERT_2_B Hirshfeld Test Diff for    O1       --C11      .       13.5 s.u.
```

Alert level C

```
CHEMW01_ALERT_1_C  The difference between the given and expected weight for
                compound is greater 1 mass unit. Check that all hydrogen
                atoms have been taken into account.
DIFMN03_ALERT_1_C  The minimum difference density is < -0.1*ZMAX*0.75
                The relevant atom site should be identified.
DIFMX02_ALERT_1_C  The maximum difference density is > 0.1*ZMAX*0.75
                The relevant atom site should be identified.
PLAT031_ALERT_4_C Refined Extinction Parameter Within Range of ...      2.500 Sigma
PLAT218_ALERT_3_C Constrained U(i,j) Components(s) for C12                6 Check
PLAT220_ALERT_2_C NonSolvent   Resd 1  C   Ueq(max)/Ueq(min) Range        4.1 Ratio
PLAT222_ALERT_3_C NonSolvent Resd 1  H   Uiso(max)/Uiso(min) Range        4.9 Ratio
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of        C18 Check
PLAT340_ALERT_3_C Low Bond Precision on  C-C Bonds ...............    0.00616 Ang.
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) .          7 Check
PLAT921_ALERT_1_C R1  in the CIF and FCF Differ by ...............    -0.0013 Check
PLAT922_ALERT_1_C wR2 in the CIF and FCF Differ by ...............    -0.0013 Check
PLAT923_ALERT_1_C S    Values in the CIF and FCF Differ by .......     -0.026 Check
```

Alert level G

```
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    8
           From the CIF: _chemical_formula_sum  C25 H37 N3 O8 [+ solvent]
           TEST: Compare cell contents of formula and atom_site data

           atom    Z*formula  cif sites diff
           C        200.00    200.00    0.00
           H        296.00    296.00    0.00
           N         24.00     24.00    0.00
```

```
           O         64.00     64.00    0.00
           [+         8.00      0.00    8.00
           solve      8.00      0.00    8.00
PLAT051_ALERT_1_G Mu(calc) and Mu(cif) Ratio Differs from 1.0 by .       7.01 %
PLAT072_ALERT_2_G SHELXL First  Parameter in WGHT  Unusually Large       0.16 Report
PLAT073_ALERT_1_G H-atoms ref, but _hydrogen_treatment Reported as     constr Check
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT  Unusually Large       7.00 Why ?
PLAT143_ALERT_4_G s.u. on c - Axis Small or Missing ..............    0.00010 Ang.
PLAT398_ALERT_2_G Deviating  C-O-C   Angle From 120 for O2       .      109.5 Degree
PLAT605_ALERT_4_G Largest Solvent Accessible VOID in the Structure         79 A**3
PLAT769_ALERT_4_G CIF Embedded Explicitly Supplied Scattering Data     Please Note
PLAT791_ALERT_4_G Model has Chirality at C16        (Sohncke SpGr)         S Verify
PLAT868_ALERT_4_G ALERTS Due to the Use of _smtbx_masks Suppressed          ! Info
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary .     Please Do !
PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still        91% Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600          2 Note
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ...........     12.401 Note
             Predicted wR2: Based on SigI**2  1.91 or SHELX Weight 22.42
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          0 Info
```

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

  13 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
  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
   7 ALERT type 4 Improvement, methodology, query or suggestion
   1 ALERT type 5 Informative message, check
```

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_ABSMU01_shelx
;
PROBLEM: The ratio of given/expected absorption coefficient lies
RESPONSE: ...
;
_vrf_CHEMW01_shelx
;
PROBLEM: The ratio of given/expected molecular weight as calculated
RESPONSE: ...
;
_vrf_DIFMN02_shelx
;
PROBLEM: The minimum difference density is < -0.1*ZMAX*1.00
RESPONSE: ...
;
_vrf_DIFMN03_shelx
;
PROBLEM: The minimum difference density is < -0.1*ZMAX*0.75
RESPONSE: ...
```

```
;
_vrf_DIFMX02_shelx
;
PROBLEM: The maximum difference density is > 0.1*ZMAX*0.75
RESPONSE: ...
;
_vrf_PLAT097_shelx
;
PROBLEM: Large Reported Max.  (Positive) Residual Density       1.19 eA-3
RESPONSE: ...
;
_vrf_PLAT098_shelx
;
PROBLEM: Large Reported Min.  (Negative) Residual Density      -1.28 eA-3
RESPONSE: ...
;
_vrf_PLAT230_shelx
;
PROBLEM: Hirshfeld Test Diff for    O1       --C11      .       13.5 s.u.
RESPONSE: ...
;
_vrf_PLAT031_shelx
;
PROBLEM: Refined Extinction Parameter Within Range of ...      2.500 Sigma
RESPONSE: ...
;
_vrf_PLAT218_shelx
;
PROBLEM: Constrained U(i,j) Components(s) for C12                 6 Check
RESPONSE: ...
;
_vrf_PLAT220_shelx
;
PROBLEM: NonSolvent   Resd 1  C   Ueq(max)/Ueq(min) Range        4.1 Ratio
RESPONSE: ...
;
_vrf_PLAT222_shelx
;
PROBLEM: NonSolvent Resd 1  H   Uiso(max)/Uiso(min) Range        4.9 Ratio
RESPONSE: ...
;
_vrf_PLAT242_shelx
;
PROBLEM: Low    'MainMol' Ueq as Compared to Neighbors of        C18 Check
RESPONSE: ...
;
_vrf_PLAT340_shelx
;
PROBLEM: Low Bond Precision on  C-C Bonds ..............    0.00616 Ang.
RESPONSE: ...
;
_vrf_PLAT918_shelx
;
PROBLEM: Reflection(s) with I(obs) much Smaller I(calc) .          7 Check
RESPONSE: ...
;
_vrf_PLAT921_shelx
```

```
;
PROBLEM: R1  in the CIF and FCF Differ by ..............    -0.0013 Check
RESPONSE: ...
;
_vrf_PLAT922_shelx
;
PROBLEM: wR2 in the CIF and FCF Differ by ..............    -0.0013 Check
RESPONSE: ...
;
_vrf_PLAT923_shelx
;
PROBLEM: S    Values in the CIF and FCF Differ by .......     -0.026 Check
RESPONSE: ...
;
# end Validation Reply Form
```

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 22/08/2024; check.def file version of 21/08/2024

Datablock shelx - ellipsoid plot