checkCIF/PLATON report

Structure factors have been supplied for datablock(s) rebe18

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: rebe18

```
Bond precision:   C-C = 0.0060 A              Wavelength=1.54178

Cell:             a=17.7361(4)      b=17.7361(4)       c=17.7361(4)
                  alpha=90          beta=90            gamma=90
Temperature:      100 K

                  Calculated                Reported
Volume            5579.2(4)                 5579.2(4)
Space group       P a -3                    P a -3
Hall group        -P 2ac 2ab                -P 2ac 2ab
Moiety formula    C18 H15 Br3 Ni P, C8 H20 N C18 H15 Br3 Ni P, C8 H20 N
Sum formula       C26 H35 Br3 N Ni P        C26 H35 Br3 N Ni P
Mr                690.90                    690.96
Dx,g cm-3         1.645                     1.645
Z                 8                         8
Mu (mm-1)         6.678                     6.678
F000              2767.9                    2768.0
F000'             2733.87
h,k,lmax          21,21,21                  20,21,21
Nref              1705                      1703
Tmin,Tmax         0.471,0.766               0.560,0.766
Tmin'             0.427

Correction method= # Reported T Limits: Tmin=0.560 Tmax=0.766
AbsCorr = MULTI-SCAN

Data completeness= 0.999          Theta(max)= 68.148

R(reflections)= 0.0398( 1475)     wR2(reflections)= 0.1030( 1703)

S = 1.061                  Npar= 159
```

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

PLAT244_ALERT_4_C Low   'Solvent' Ueq as Compared to Neighbors of   N1 Check

Author Response: Central nitrogen atom of a tetraethylammonium cation strongly disordered around a threefold axis.

PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor ....   2.2 Note

Author Response: No obivous preferred orientation of ADPs visible in packing diagrams. Eventually related with higher disorder perpendicular to the 3-fold axis.

⚪ Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite   12 Note

Author Response: The [NEt4]+ cation was found situated on a three-fold axis and was - as to be expected - highly disordered. SADI and RIGU restraints were applied to bond distances and angles in the [NEt4]+ cation. Increased ADPs and high residual charges also indicated a disorder of the bromine atoms. Most likely, a tilting of the NiBr3 moiety relativ to the Ni-P bond, so that P-Ni-Br angles become unequal. Two positions were refined for Br with SADI restraints on the Ni-Br distance, but no further restraints. Occupation factors refined to 0.54:0.46. Refinement of three bromine positions further lowered R1 and wR2 values, but yielded two practically superimposed bromine atoms with slightly different ADPs. We restricted the refinement thus to two bromine positions.

PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ...   9 Report
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large   10.84 Why ?
PLAT176_ALERT_4_G The CIF-Embedded .res File Contains SADI Records   4 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records   1 Report
PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records   1 Report
PLAT232_ALERT_2_G Hirshfeld Test Diff (M-X) Br1   --Ni1   .   8.8 s.u.
PLAT232_ALERT_2_G Hirshfeld Test Diff (M-X) Br2   --Ni1   .   8.5 s.u.
PLAT300_ALERT_4_G Atom Site Occupancy of C11   Constrained at 0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of C12   Constrained at 0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of C21   Constrained at 0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of C22   Constrained at 0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of C31   Constrained at 0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of C32   Constrained at 0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of C41   Constrained at 0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of C42   Constrained at 0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H11A   Constrained at 0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H11B   Constrained at 0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H12A   Constrained at 0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H12B   Constrained at 0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H12C   Constrained at 0.3333 Check

```
PLAT300_ALERT_4_G Atom Site Occupancy of H21A        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H21B        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H22A        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H22B        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H22C        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H31A        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H31B        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H32A        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H32B        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H32C        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H41A        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H41B        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H42A        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H42B        Constrained at     0.3333 Check
PLAT300_ALERT_4_G Atom Site Occupancy of H42C        Constrained at     0.3333 Check
PLAT301_ALERT_3_G Main Residue  Disorder ..............(Resd  1  )        13% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  2  )       89% Note
PLAT789_ALERT_4_G Atoms with Negative _atom_site_disorder_group  #        30 Check
PLAT811_ALERT_5_G No ADDSYM Analysis: Too Many Excluded Atoms ....         ! Info
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............       121 Note
PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still       67% Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600         1 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.         4 Info
```

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

   0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   7 ALERT type 2 Indicator that the structure model may be wrong or deficient
   3 ALERT type 3 Indicator that the structure quality may be low
  35 ALERT type 4 Improvement, methodology, query or suggestion
   1 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 16/07/2020; check.def file version of 12/07/2020