checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

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: 1581

```
Bond precision:  C-C = 0.0014 A          Wavelength=0.71073

Cell:          a=9.6854(2)       b=9.9691(2)      c=10.4536(2)
               alpha=87.793(2)   beta=63.452(2)   gamma=85.496(2)
Temperature:   100 K
```

	Calculated	Reported
Volume	900.13(3)	900.12(3)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C22 H20 N2 O4	C22 H20 N2 O4
Sum formula	C22 H20 N2 O4	C22 H20 N2 O4
Mr	376.40	376.40
Dx,g cm-3	1.389	1.389
Z	2	2
Mu (mm-1)	0.097	0.097
F000	396.0	396.0
F000’	396.19	
h,k,lmax	13,14,14	13,14,14
Nref	5251	5208
Tmin,Tmax	0.960,0.971	0.890,0.960
Tmin’	0.958	

```
Correction method= MULTI-SCAN

Data completeness= 0.992          Theta(max)= 30.000

R(reflections)= 0.0406( 4614)     wR2(reflections)= 0.1179( 5208)

S = 1.018                  Npar= 257
```

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

```
PLAT414_ALERT_2_C Short Intra D-H..H-X       H1N    ..  H5      ..       1.97 Ang.
PLAT414_ALERT_2_C Short Intra D-H..H-X       H2N    ..  H18     ..       1.95 Ang.
```

Alert level G

```
PLAT005_ALERT_5_G No _iucr_refine_instructions_details  in the CIF     Please Do !
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..............          4 Report
PLAT154_ALERT_1_G The su's on the Cell Angles are Equal ..........    0.00200 Degree
PLAT899_ALERT_4_G SHELXL97   is Deprecated and Succeeded by SHELXL       2014 Note
```

```
   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
   4 ALERT level G = General information/check it is not something unexpected

   1 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
   0 ALERT type 3 Indicator that the structure quality may be low
   1 ALERT type 4 Improvement, methodology, query or suggestion
   2 ALERT type 5 Informative message, check
```

Datablock: 1580

Bond precision: C-C = 0.0018 A Wavelength=0.71073

Cell: a=27.3312(9) b=12.3272(4) c=17.8035(6)
       alpha=90 beta=125.968(2) gamma=90
Temperature: 100 K

	Calculated	Reported
Volume	4854.7(3)	4854.7(3)
Space group	C 2/c	C 2/c
Hall group	-C 2yc	-C 2yc
Moiety formula	C30 H24 N2 O4	C30 H24 N2 O4
Sum formula	C30 H24 N2 O4	C30 H24 N2 O4
Mr	476.51	476.51
Dx,g cm-3	1.304	1.304
Z	8	8
Mu (mm-1)	0.087	0.087
F000	2000.0	2000.0
F000'	2000.92	
h,k,lmax	38,17,25	37,17,25
Nref	7094	7071
Tmin,Tmax	0.969,0.983	0.850,0.970
Tmin'	0.966	

Correction method= MULTI-SCAN

Data completeness= 0.997 Theta(max)= 30.000

R(reflections)= 0.0427(5654) wR2(reflections)= 0.1237(7071)

S = 1.037 Npar= 329

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

PLAT414_ALERT_2_C Short Intra D-H..H-X H1N .. H5 .. 1.90 Ang.
PLAT414_ALERT_2_C Short Intra D-H..H-X H2N .. H18 .. 1.91 Ang.

Alert level G

PLAT005_ALERT_5_G No _iucr_refine_instructions_details in the CIF Please Do !
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..... 4 Report
PLAT128_ALERT_4_G Alternate Setting for Input Space Group C2/c I2/a Note
PLAT899_ALERT_4_G SHELXL97 is Deprecated and Succeeded by SHELXL 2014 Note

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

Datablock: 1598

Bond precision: C-C = 0.0070 A Wavelength=0.71073

Cell: a=12.0549(6) b=12.4484(6) c=12.7005(6)
alpha=65.574(3) beta=63.112(3) gamma=85.976(3)
Temperature: 100 K

	Calculated	Reported
Volume	1531.60(14)	1531.60(13)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C56 H52 Co4 N10 O10, 2(O)	C56 H52 Co4 N10 O10, 2(H2 O), 2(C2 H3 N)
Sum formula	C56 H52 Co4 N10 O12	C60 H62 Co4 N12 O12
Mr	1292.80	1378.94
Dx,g cm-3	1.402	1.495
Z	1	1
Mu (mm-1)	1.130	1.136
F000	662.0	710.0
F000'	663.63	
h,k,lmax	14,14,15	13,14,15
Nref	5399	5127
Tmin,Tmax	0.761,0.834	0.660,0.840
Tmin'	0.680	

Correction method= MULTI-SCAN

Data completeness= 0.950 Theta(max)= 25.000

R(reflections)= 0.0542(3398) wR2(reflections)= 0.1575(5127)

S = 1.090 Npar= 375

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 1
From the CIF: _chemical_formula_weight 1378.94
TEST: Calculate formula weight from _atom_site_*

atom	mass	num	sum
C	12.01	56.00	672.62
H	1.01	52.00	52.42
N	14.01	10.00	140.07
O	16.00	12.00	191.99
Co	58.93	4.00	235.73

Calculated formula weight 1292.82

Author Response: The compound was crystallized during electrosynthesis of the tetranuclear Co(ii)/Co(iii) complex in an acetonitrile solution in the presence of moisture. The solid state compound is a solvate of H2O and CH3CN. The acetonitrile solvent molecule was found near the centre of inversion at x,y,z=0,1/2,0 and was disordered. This acetonitrile was squeezed with program PLATON prior to final refinement. The molecule is situated in a single centrosymmetric cavity with a solvent accessible volume of ca. 200 A3 and contains apparently two acetonitrile molecules per cavity and per one tetranuclear Co(ii)/Co(iii) complex. The theoretical electron count of these two molecules is 44, whereas PLATON calculated an electron count of 48 e from the diffraction data. Chemical formula and derived quantities are given for (C60 H62 Co4 N10 O10), 2(H2 O), 2(C2 H3 N), which is also the unit cell content. The difference between atom count of the crystal structure model and the given chemical composition leads to various other checkCIF/PLATON alerts which are not separately outlined. These alerts are CHEMW03, PLAT041, PLAT042, PLAT068, FORMU01, CELLZ01. The region where the acetonitrile solvent molecules are located can be easily recognized in a packing diagram viewed along [100].

```
PLAT029_ALERT_3_B _diffrn_measured_fraction_theta_full Low .......      0.950 Note
PLAT306_ALERT_2_B Isolated Oxygen Atom (H-atoms Missing ?) .......        O6W Check
```

Author Response: O6w is the oxygen of a water molecule the hydrogen atoms of which could not be located. It resides in a pocket of the Co complex between the aetonitrile ligands N4-C25-C26H3 and N5-C27-C28H3 and the phenyl ring C15 - C20. The distances from O6w to C25, C27, and the ring centroid C15 through C20 are 3.18, 3.17 and 3.33 A, respectively. The contact O6w...O2 = 2.68 A to the keto oxygen atom O2 represents probably a O-H...O hydrogen bond.

```
PLAT420_ALERT_2_B D-H Without Acceptor       O5H    -   H5O    ...     Please Check
```

Author Response: This is an OH group with a pyramidal coordination by three Co atoms and located in a cavity of a tetranuclear Co complex. This OH group may have hydrogen bond interactions to a disordered acetonitrile solvent molecule that was squeezed with program PLATON. See comments on other alerts for further information.

```
PLAT430_ALERT_2_B Short Inter D...A Contact  O2     ..  O6W     ..       2.68 Ang.
```

Author Response: O6w is the oxygen of a water molecule the hydrogen atoms of which could not be located. It resides in a pocket of the Co complex between the aetonitrile ligands N4-C25-C26H3 and N5-C27-C28H3 and the phenyl ring C15 - C20. The distances from O6w to C25, C27, and the ring centroid C15 through C20 are 3.18, 3.17 and 3.33 A, respectively. The contact O6w...O2 = 2.68 A to the keto oxygen atom O2 represents probably a O-H...O hydrogen bond.

Alert level C

```
PLAT041_ALERT_1_C Calc. and Reported SumFormula    Strings  Differ     Please Check
PLAT068_ALERT_1_C Reported F000 Differs from Calcd (or Missing)...     Please Check
PLAT341_ALERT_3_C Low Bond Precision on  C-C Bonds ..............     0.0070 Ang.
```

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:C60 H62 Co4 N12 O12
           Atom count from the _atom_site data:  C56 H52 Co4 N10 O12
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    1
           From the CIF: _chemical_formula_sum  C60 H62 Co4 N12 O12
           TEST: Compare cell contents of formula and atom_site data

           atom    Z*formula  cif sites diff
           C         60.00     56.00    4.00
           H         62.00     52.00   10.00
           Co         4.00      4.00    0.00
           N         12.00     10.00    2.00
           O         12.00     12.00    0.00
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite         40 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ...         40 Report
PLAT005_ALERT_5_G No _iucr_refine_instructions_details  in the CIF     Please Do !
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..............          1 Report
PLAT042_ALERT_1_G Calc. and Reported MoietyFormula Strings  Differ     Please Check
PLAT154_ALERT_1_G The su's on the Cell Angles are Equal ..........    0.00300 Degree
PLAT605_ALERT_4_G Structure Contains Solvent Accessible VOIDS of .        201 A**3
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Author Response: Acetonitrile solvent squeezed. See CHEMW03 and PLAT869 for further information

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PLAT860_ALERT_3_G Number of Least-Squares Restraints .............        150 Note
```

Author Response: Soft bond distance restraints (sigma 0.003 - 0.005 A) were used to emphasize the 2/m pseudosymmetry of the tetranuclear Co complex (but not of the crystal structure, which is cleanly triclinic P-1). Moreover a DELU 0.005 0.005 restraint was applied to the Uij.

```
PLAT869_ALERT_4_G ALERTS Related to the use of SQUEEZE Suppressed           ! Info
```

Author Response: The compound was crystallized during electrosynthesis of the tetranuclear Co(ii)/Co(iii) complex in an acetonitrile solution in the presence of moisture. The solid state compound is a solvate of H2O and CH3CN. The acetonitrile solvent molecule was found near the centre of inversion at x,y,z=0,1/2,0 and was disordered. This acetonitrile was squeezed with program PLATON prior to final refinement. The molecule is situated in a single centrosymmetric cavity with a solvent accessible volume of ca. 200 A3 and contains apparently two acetonitrile molecules per cavity and per one tetranuclear Co(ii)/Co(iii) complex. The theoretical electron count of these two molecules is 44, whereas PLATON calculated an electron count of 48 e from the diffraction data. Chemical formula and derived quantities are given for (C60 H62 Co4 N10 O10), 2(H2 O), 2(C2 H3 N), which is also the unit cell content. The difference between atom count of the crystal structure model and the given chemical composition leads to various other checkCIF/PLATON alerts which are not separately outlined. These alerts are CHEMW03, PLAT041, PLAT042, PLAT068, FORMU01, CELLZ01. The region where the acetonitrile solvent molecules are located can be easily recognized in a packing diagram viewed along [100].

```
PLAT899_ALERT_4_G SHELXL97   is Deprecated and Succeeded by SHELXL       2014 Note
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```

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

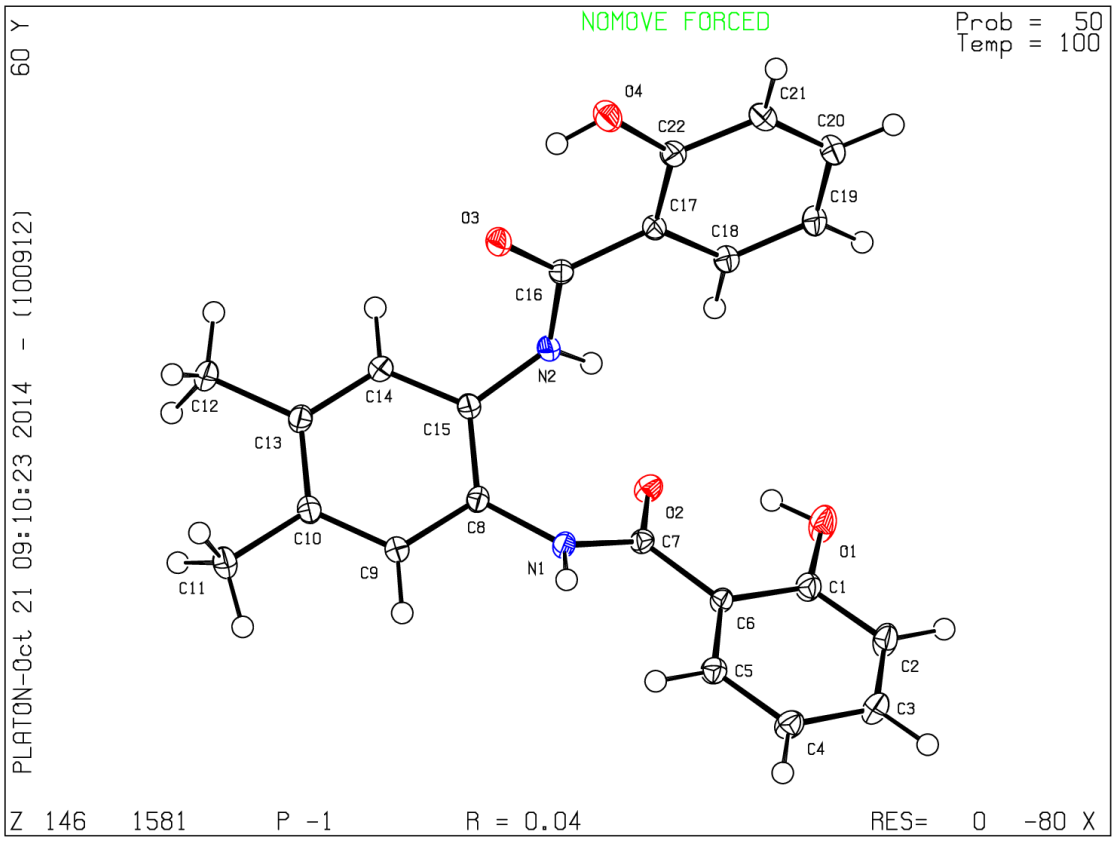
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, 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

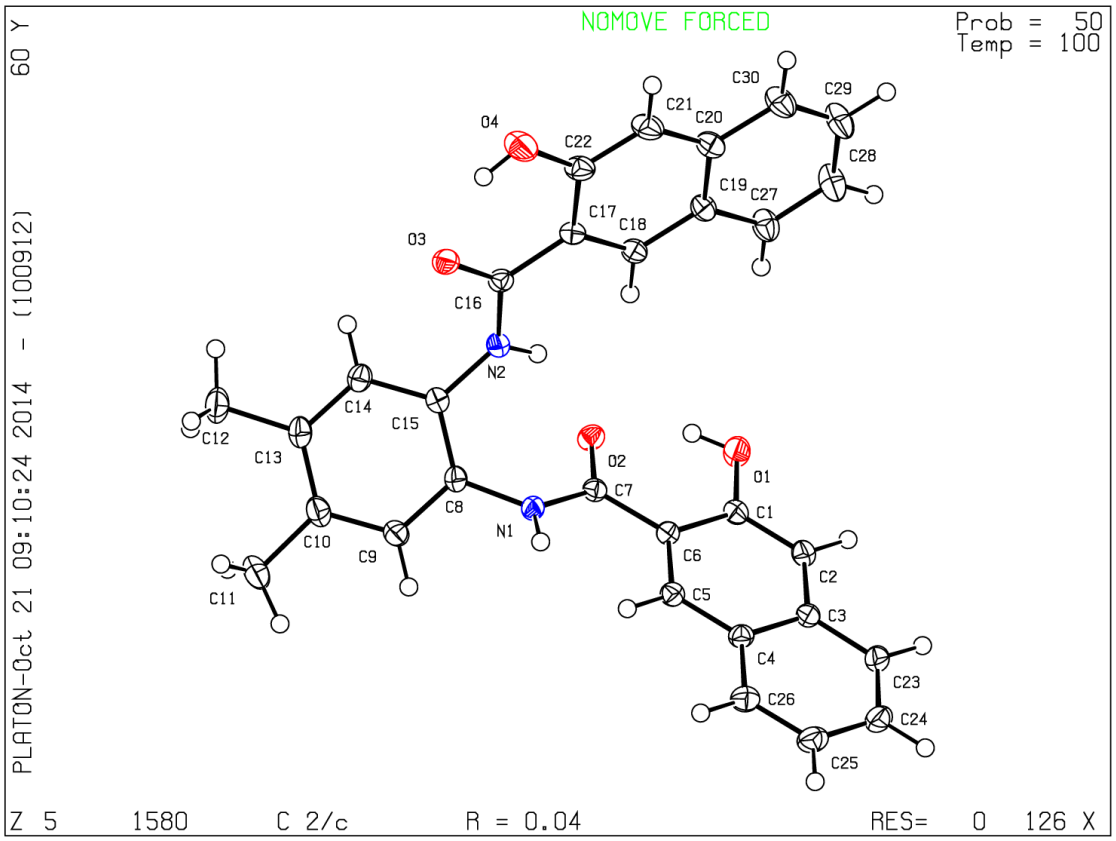
Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 20/08/2014; check.def file version of 18/08/2014

Datablock 1581 - ellipsoid plot



Datablock 1580 - ellipsoid plot



Datablock 1598 - ellipsoid plot