checkCIF/PLATON report

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

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 4b

```
Bond precision:  C-C = 0.0029 A          Wavelength=0.71073

Cell:           a=15.5497(7)    b=14.4945(7)         c=12.9298(6)
                alpha=90        beta=102.3006(6)     gamma=90
Temperature:    173 K

                Calculated                Reported
Volume          2847.3(2)                 2847.3(2)
Space group     P 21/c                    P 21/c
Hall group      -P 2ybc                   -P 2ybc
Moiety formula  C20 H29 N5, 2(C F3 O3 S)  C20 H29 N5, 2(C F3 O3 S)
Sum formula     C22 H29 F6 N5 O6 S2       C22 H29 F6 N5 O6 S2
Mr              637.64                    637.62
Dx,g cm-3       1.487                     1.487
Z               4                         4
Mu (mm-1)       0.272                     0.272
F000            1320.0                    1320.0
F000'           1321.86
h,k,lmax        19,18,16                  19,18,16
Nref            5837                      5831
Tmin,Tmax       0.880,0.989               0.883,0.989
Tmin'           0.880

Correction method= INTEGRATION

Data completeness= 0.999          Theta(max)= 26.400

R(reflections)= 0.0361( 4762)     wR2(reflections)= 0.0968( 5831)

S = 1.021                 Npar= 372
```

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

```
PLAT220_ALERT_2_C Large Non-Solvent    C     Ueq(max)/Ueq(min) ...        3.7 Ratio
PLAT242_ALERT_2_C Check Low       Ueq as Compared to Neighbors for        C15
PLAT242_ALERT_2_C Check Low       Ueq as Compared to Neighbors for        C18
```

Alert level G

```
PLAT005_ALERT_5_G No _iucr_refine_instructions_details in CIF ....          ?
PLAT432_ALERT_2_G Short Inter X...Y Contact  O5     ..  C6      ..       2.98 Ang.
PLAT432_ALERT_2_G Short Inter X...Y Contact  O5     ..  C4      ..       2.99 Ang.
```

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

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

Datablock: 5b

Bond precision: C-C = 0.0031 A Wavelength=0.71073

Cell: a=10.1647(5) b=10.4449(5) c=13.1034(6)
       alpha=76.4620(5) beta=72.6898(5) gamma=78.6303(5)
Temperature: 173 K

	Calculated	Reported
Volume	1279.03(11)	1279.03(11)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C22 H30 I2 N5 O2 Rh	C22 H30 I2 N5 O2 Rh
Sum formula	C22 H30 I2 N5 O2 Rh	C22 H30 I2 N5 O2 Rh
Mr	753.22	753.22
Dx,g cm-3	1.956	1.956
Z	2	2
Mu (mm-1)	3.109	3.109
F000	728.0	728.0
F000’	724.08	
h,k,lmax	13,13,17	13,13,17
Nref	5855	5814
Tmin,Tmax	0.449,0.856	0.487,0.855
Tmin’	0.415	

Correction method= NUMERICAL

Data completeness= 0.993 Theta(max)= 27.480

R(reflections)= 0.0172(5305) wR2(reflections)= 0.0436(5814)

S = 1.036 Npar= 292

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

PLAT411_ALERT_2_C Short Inter H...H Contact H5 .. H20 .. 2.14 Ang.

Alert level G

PLAT005_ALERT_5_G No _iucr_refine_instructions_details in CIF .... ?
PLAT232_ALERT_2_G Hirshfeld Test Diff (M-X) I2 -- Rh .. 12.7 su
PLAT232_ALERT_2_G Hirshfeld Test Diff (M-X) Rh -- O1 .. 5.0 su
PLAT232_ALERT_2_G Hirshfeld Test Diff (M-X) Rh -- C4 .. 5.6 su

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

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

Datablock: 7b

Bond precision: C-C = 0.0214 A Wavelength=0.71073

Cell: a=30.695(19) b=17.496(11) c=15.149(9)
alpha=90 beta=119.530(7) gamma=90
Temperature: 173 K

	Calculated	Reported
Volume	7079(8)	7079(7)
Space group	C 2/c	C2/c
Hall group	-C 2yc	-C 2yc
Moiety formula	C20 H28 I3 N5 Pd, C2 H3 N	C20 H28 I3 N5 Pd, 4(C2 H3 N)
Sum formula	C22 H31 I3 N6 Pd	C28 H40 I3 N9 Pd
Mr	866.63	989.79
Dx,g cm-3	1.626	1.857
Z	8	8
Mu (mm-1)	3.158	3.173
F000	3280.0	3808.0
F000’	3261.53	
h,k,lmax	38,21,18	38,21,18
Nref	7250	7143
Tmin,Tmax	0.664,0.752	0.252,0.757
Tmin’	0.148	

Correction method= NUMERICAL

Data completeness= 0.985 Theta(max)= 26.400

R(reflections)= 0.0734(4484) wR2(reflections)= 0.2186(7143)

S = 1.080 Npar= 288

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

CHEMW03_ALERT_2_A ALERT: The ratio of given/expected molecular weight as calculated from the _atom_site* data lies outside the range 0.90 <> 1.10

From the CIF: _cell_formula_units_Z 8
From the CIF: _chemical_formula_weight 989.79
TEST: Calculate formula weight from _atom_site_*

atom	mass	num	sum
C	12.01	22.00	264.24
H	1.01	31.00	31.25
N	14.01	6.00	84.04
Pd	106.42	1.00	106.42
I	126.90	3.00	380.71

Calculated formula weight 866.67

Author Response: Attempts to refine peaks of residual electron density as disordered or partial-occupancy solvent acetonitrile nitrogen or carbon atoms were unsuccessful. The data were corrected for disordered electron density through use of the SQUEEZE procedure (Sluis, P. van der; Spek, A. L. Acta Crystallogr. 1990, A46, 194-201) as implemented in PLATON (Spek, A. L. Acta Crystallogr. 1990, A46, C34; Spek, A. L. J. Appl. Cryst. 2003, 36, 7-13. PLATON - a multipurpose crystallographic tool. Utrecht University, Utrecht, The Netherlands). A total solvent-accessible void volume of 1470 cubic Angstroms with a total electron count of 519(consistent with 24 molecules of solvent acetonitrile, or 3 moleculesper formula unit of the palladium complex) was found in the unit cell.

```
PLAT043_ALERT_1_A Check Reported Molecular Weight ..............     989.79
```

Author Response: see response to CHEMW03.

Alert level B

```
PLAT342_ALERT_3_B Low Bond Precision on  C-C Bonds ..............     0.0214 Ang
```

Alert level C

```
PLAT041_ALERT_1_C Calc. and Reported SumFormula    Strings  Differ          ?
```

Author Response: see response to CHEMW03.

```
PLAT042_ALERT_1_C Calc. and Reported MoietyFormula Strings  Differ          ?
```

Author Response: see response to CHEMW03.

```
PLAT068_ALERT_1_C Reported F000 Differs from Calcd (or Missing)...          ?
```

Author Response: see response to CHEMW03.

```
PLAT234_ALERT_4_C Large Hirshfeld Difference N5     --  C9      ..       0.16 Ang.
PLAT234_ALERT_4_C Large Hirshfeld Difference C13    --  C14     ..       0.19 Ang.
PLAT242_ALERT_2_C Check Low       Ueq as Compared to Neighbors for        C18
PLAT244_ALERT_4_C Low   'Solvent' Ueq as Compared to Neighbors of         C1S
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor ....        2.2
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond  C9     -   C10    ...       1.37 Ang.
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond  C10    -   C11    ...       1.37 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:C28 H40 I3 N9 Pd1
           Atom count from the _atom_site data:  C22 H31 I3 N6 Pd1
```

Author Response: see response to CHEMW03.

```
CELLZ01_ALERT_1_G Difference between formula and atom_site contents detected.
```

Author Response: see response to CHEMW03.

```
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  C28 H40 I3 N9 Pd
           TEST: Compare cell contents of formula and atom_site data

           atom    Z*formula  cif sites diff
           C        224.00    176.00   48.00
           H        320.00    248.00   72.00
           I         24.00     24.00    0.00
           N         72.00     48.00   24.00
           Pd         8.00      8.00    0.00
```

Author Response: see response to CHEMW03.

```
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite          6
PLAT005_ALERT_5_G No _iucr_refine_instructions_details in CIF ....          ?
PLAT072_ALERT_2_G SHELXL First  Parameter in WGHT Unusually Large.       0.12
PLAT301_ALERT_3_G Note: Main Residue  Disorder ...................         10 Perc.
PLAT606_ALERT_4_G VERY LARGE Solvent Accessible VOID(S) in Structure        !
```

Author Response: see response to CHEMW03.

```
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #         15
            I3  -PD  -C1  -N1    175.00  4.00   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #         18
            I3  -PD  -C1  -N2     -7.00  5.00   1.555   1.555   1.555   1.555
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..........          3
PLAT860_ALERT_3_G Note: Number of Least-Squares Restraints .......          6
PLAT869_ALERT_4_G ALERTS Related to the use of SQUEEZE Suppressed           !
```

Author Response: see response to CHEMW03.

```
   2 ALERT level A = Most likely a serious problem - resolve or explain
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   6 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
   3 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
```

Datablock: 10b

```
Bond precision:  C-C = 0.0069 A          Wavelength=0.71073
```

Cell:	a=13.782(3)	b=15.694(3)	c=22.394(5)
	alpha=79.906(3)	beta=88.010(3)	gamma=86.666(3)
Temperature:	173 K		

	Calculated	Reported
Volume	4759.2(17)	4759.3(17)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	2(C38 H43 I2 N5 P Pd), 3(C2 H3 N), 2(I)	C38 H43 I2 N5 P Pd, 2(C2 H3 N), I
Sum formula	C82 H95 I6 N13 P2 Pd2	C42 H49 I3 N7 P Pd
Mr	2298.85	1169.95
Dx,g cm-3	1.604	1.633
Z	2	4
Mu (mm-1)	2.403	2.405
F000	2236.0	2280.0
F000’	2227.31	
h,k,lmax	17,20,29	17,20,29
Nref	22166	21838
Tmin,Tmax	0.450,0.866	0.393,0.877
Tmin’	0.312	

Correction method= NUMERICAL

Data completeness= 0.985 Theta(max)= 27.640

R(reflections)= 0.0351(16825) wR2(reflections)= 0.0924(21838)

S = 1.031 Npar= 951

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

PLAT220_ALERT_2_B Large Non-Solvent C Ueq(max)/Ueq(min) ... 4.4 Ratio

---

Alert level C

CHEMW03_ALERT_2_C The ratio of given/expected molecular weight as calculated from the _atom_site* data lies outside the range 0.99 <> 1.01

From the CIF: _cell_formula_units_Z 4

From the CIF: _chemical_formula_weight 1169.95

TEST: Calculate formula weight from _atom_site_*

atom	mass	num	sum
C	12.01	41.00	492.45
H	1.01	47.50	47.88
N	14.01	6.50	91.05

```
P         30.97    1.00   30.97
Pd       106.42    1.00  106.42
I        126.90    3.00  380.71
Calculated formula weight            1149.49
```

Author Response: Attempts to refine peaks of residual electron density as disordered or partial-occupancy solvent acetonitrile nitrogen or carbon atoms were unsuccessful. The data were corrected for disordered electron density through use of the SQUEEZE procedure (Sluis, P. van der; Spek, A. L. Acta Crystallogr. 1990, A46, 194-201) as implemented in PLATON (Spek, A. L. Acta Crystallogr. 1990, A46, C34; Spek, A. L. J. Appl. Cryst. 2003, 36, 7-13. PLATON - a multipurpose crystallographic tool. Utrecht University, Utrecht, The Netherlands). A total solvent-accessible void volume of 199 cubic Angstroms with a total electron count of 45(consistent with 2 molecule of solvent acetonitrile, or 0.5 molecule performula unit of the palladium complex) was found in the unit cell.

```
PLAT041_ALERT_1_C Calc. and Reported SumFormula    Strings  Differ          ?
```

Author Response: see response to CHEMW03.

```
PLAT042_ALERT_1_C Calc. and Reported MoietyFormula Strings  Differ          ?
```

Author Response: see response to CHEMW03.

```
PLAT043_ALERT_1_C Check Reported Molecular Weight ..............    1169.95
```

Author Response: see response to CHEMW03.

```
PLAT045_ALERT_1_C Calculated and Reported Z Differ by ............       0.50 Ratio
PLAT068_ALERT_1_C Reported F000 Differs from Calcd (or Missing)...          ?
```

Author Response: see response to CHEMW03.

```
PLAT220_ALERT_2_C Large Non-Solvent    C     Ueq(max)/Ueq(min) ...        3.5 Ratio
PLAT242_ALERT_2_C Check Low       Ueq as Compared to Neighbors for       C18A
PLAT242_ALERT_2_C Check Low       Ueq as Compared to Neighbors for       C15B
PLAT244_ALERT_4_C Low   'Solvent' Ueq as Compared to Neighbors of         C1S
PLAT244_ALERT_4_C Low   'Solvent' Ueq as Compared to Neighbors of         C3S
```

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:C42 H49 I3 N7 P1 Pd1
           Atom count from the _atom_site data:  C41 H47.5 I3 N6.5 P1 Pd1
```

Author Response: see response to CHEMW03.

```
CELLZ01_ALERT_1_G Difference between formula and atom_site contents detected.
```

Author Response: see response to CHEMW03.

```
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  C42 H49 I3 N7 P Pd
           TEST: Compare cell contents of formula and atom_site data

           atom    Z*formula  cif sites diff
           C        168.00    164.00    4.00
           H        196.00    190.00    6.00
           I         12.00     12.00    0.00
           N         28.00     26.00    2.00
           P          4.00      4.00    0.00
           Pd         4.00      4.00    0.00
```

Author Response: see response to CHEMW03.

```
PLAT005_ALERT_5_G No _iucr_refine_instructions_details in CIF ....          ?
PLAT154_ALERT_1_G The su's on the Cell Angles are Equal .........    0.00300 Deg.
PLAT232_ALERT_2_G Hirshfeld Test Diff (M-X)  I1A    --  PdA     ..        9.0 su
PLAT232_ALERT_2_G Hirshfeld Test Diff (M-X)  I2A    --  PdA     ..        8.5 su
PLAT232_ALERT_2_G Hirshfeld Test Diff (M-X)  I2B    --  PdB     ..        7.5 su
PLAT244_ALERT_4_G Low   'Solvent' Ueq as Compared to Neighbors of         C7S
PLAT302_ALERT_4_G Note: Anion/Solvent Disorder ...................        27 Perc.
PLAT380_ALERT_4_G Check Incorrectly? Oriented X(sp2)-Methyl Moiety        C8B
PLAT432_ALERT_2_G Short Inter X...Y Contact  N3S    ..  C8S     ..       3.00 Ang.
PLAT605_ALERT_4_G Structure Contains Solvent Accessible VOIDS of .        199 A**3
```

Author Response: see response to CHEMW03.

```
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #          3
            I1A -PDA -P1A -C21A  176.50  0.30   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #          6
            I1A -PDA -P1A -C31A  -60.80  0.40   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #          9
            I1A -PDA -P1A -C41A   58.30  0.40   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #         23
            I2A -PDA -C1A -N1A  -165.20  1.20   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #         26
            I2A -PDA -C1A -N2A     9.50  1.70   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #        115
            I1B -PDB -P1B -C21B  149.90  0.30   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #        118
            I1B -PDB -P1B -C31B  -87.10  0.30   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #        121
            I1B -PDB -P1B -C41B   32.40  0.30   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #        135
            I2B -PDB -C1B -N1B  -162.30  0.80   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #        138
            I2B -PDB -C1B -N2B    11.60  1.40   1.555   1.555   1.555   1.555
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..........         36
PLAT869_ALERT_4_G ALERTS Related to the use of SQUEEZE Suppressed           !
```

Author Response: see response to CHEMW03.

```
  0 ALERT level A = Most likely a serious problem - resolve or explain
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  8 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
  0 ALERT type 3 Indicator that the structure quality may be low
 18 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

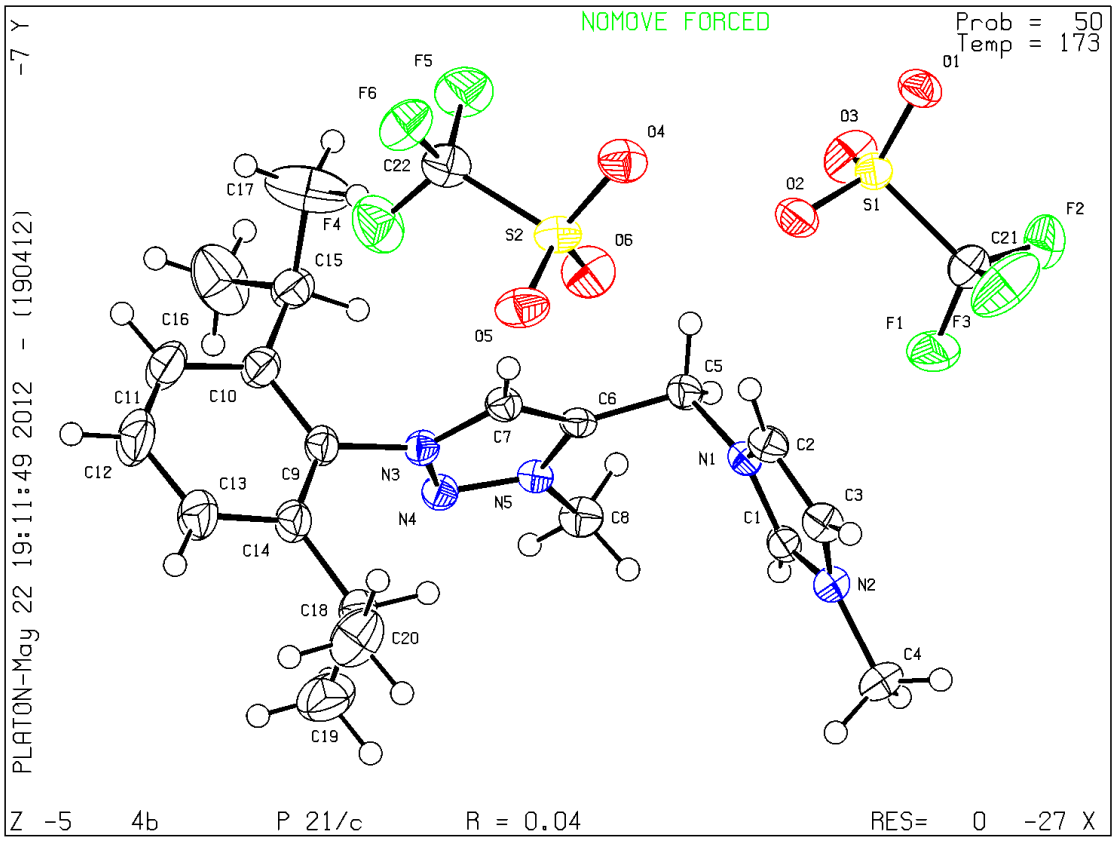
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section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

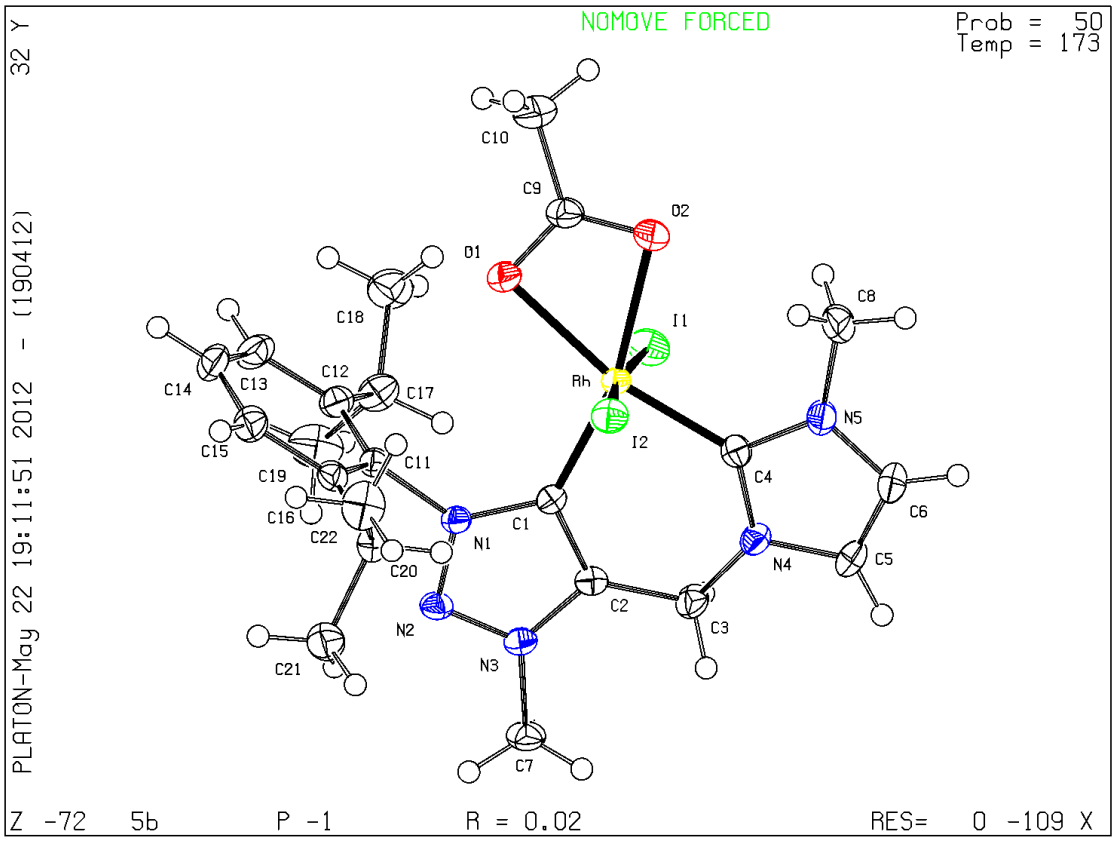
Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 19/04/2012; check.def file version of 14/04/2012

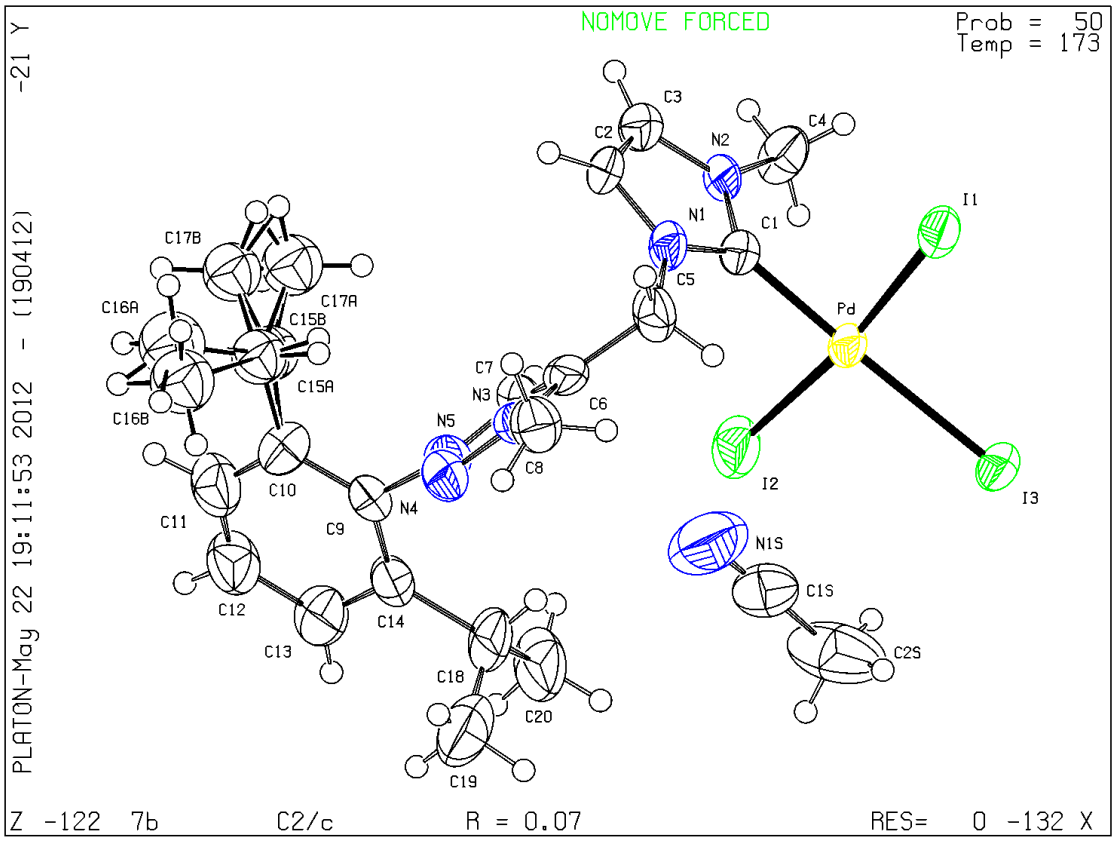
Datablock 4b - ellipsoid plot



Datablock 5b - ellipsoid plot



Datablock 7b - ellipsoid plot



Datablock 10b - ellipsoid plot