checkCIF/PLATON report

Structure factors have been supplied for datablock(s) I

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: I

Bond precision: C-C = 0.0631 A Wavelength=1.54180

Cell: a=14.7299(3) b=21.2699(5) c=25.8105(5)
alpha=111.285(2) beta=92.8643(17) gamma=93.898(2)
Temperature: 100 K

	Calculated	Reported
Volume	7493.2(3)	7493.2(3)
Space group	P -1	P -1
Hall group	-P 1	?
Moiety formula	C72 H153 Au30 S19	C72 H162 Au30 S19
Sum formula	C72 H153 Au30 S19	C72 H162 Au30 S19
Mr	7537.14	7498.29
Dx,g cm-3	3.341	3.323
Z	2	2
Mu (mm-1)	56.264	56.255
F000	6518.0	6488.0
F000'	6282.27	
h,k,lmax	18,26,32	18,26,32
Nref	30223	29386
Tmin,Tmax	0.000,0.185	0.065,1.000
Tmin'	0.000	

Correction method= MULTI-SCAN

Data completeness= 0.972 Theta(max)= 73.574

R(reflections)= 0.0921(21922) wR2(reflections)= wR= 0.1025(19357)

S = 1.081 Npar= 719

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

PLAT075_ALERT_1_A Occupancy 4. greater than 1.0 for C70

Author Response: The site labelled C70 is the annular description of the 4 atoms of the t-Bu group disordered about the gold and sulfur atoms modelled at 50% occupancy as described in the refinment explanation

PLAT201_ALERT_2_A Isotropic non-H Atoms in Main Residue(s) ..... 69 Report

Author Response: see refinement explanation all carbon atoms of tBu groups are modelled isotropically as their contribution to the scattering is limited and affected by disorder.

PLAT310_ALERT_2_A H70 Deleted (Close to C70) Dist ..... 0.010 Ang.

Author Response: this site is an annulus as described in refinement details it is occupied by a total of 9 hydrogen atoms it is not to be deleted, it is a part of the model and correctly reported relative to the coincident centres of the annuli describing the disorder of the tBu groups attached to the disordered sulfur sites

PLAT360_ALERT_2_A Short C(sp3)-C(sp3) Bond C5 - C6 ... 1.23 Ang.

Author Response: an unrestrained isotropic refinement has deliberately been employed this group is subject to significant disorder for which no further modelling was attempted

PLAT602_ALERT_2_A VERY LARGE Solvent Accessible VOID(S) in Structure ! Info

Author Response: The void abuts the highly disordered t-butyl groups and is extremely diffuse. It occupies around 25% of the cell volume and the associated electron denisty is consistent with disordered solvent inclusion. The potential for the presence of counter ions in the void cannot be excluded on th basis of the crystallographic findings - the structure is reported assuming that the formulation is correct based on other experimental findings.

Alert level B

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DIFMN02_ALERT_2_B  The minimum difference density is < -0.1*ZMAX*1.00
           _refine_diff_density_min given =     -9.810
           Test value =     -7.900
DIFMX01_ALERT_2_B  The maximum difference density is > 0.1*ZMAX*1.00
           _refine_diff_density_max given =     10.870
           Test value =      7.900
PLAT097_ALERT_2_B Large Reported Max.  (Positive) Residual Density      10.87 eA-3
PLAT098_ALERT_2_B Large Reported Min.  (Negative) Residual Density      -9.81 eA-3
PLAT220_ALERT_2_B Large Non-Solvent  C     Ueq(max)/Ueq(min) Range       10.0 Ratio
PLAT242_ALERT_2_B Low       Ueq as Compared to Neighbors for .....         C5 Check
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Author Response: This is unremarkable for the tertiary carbon of a tBu group in a \ structure where significant disorder of the numerous tBu groups is observed

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PLAT342_ALERT_3_B Low Bond Precision on  C-C Bonds ..............     0.0631 Ang.
PLAT390_ALERT_3_B Deviating Methyl C6      X-C-H Bond Angle ......         92 Degree
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Author Response: This reflects the choice not to invoke restraints and introduce a more complicated model where the data is limited by the dominance of the \ Au30S19 core and by twinning. Significant disorder of the numerous tBu groups is evident \ and this group lies in a region of the structure where lower steric pressure is evident by \ observation of van der Waals surfaces and noting that the atoms are part of one of the \ conformationally less rigid StBu units which only bridges two gold atoms. Unmodelled disorder

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PLAT390_ALERT_3_B Deviating Methyl C7      X-C-H Bond Angle ......        100 Degree
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Author Response: This reflects the choice not to invoke restraints and introduce a more complicated model where the data is limited by the dominance of the \ Au30S19 core and by twinning. Significant disorder of the numerous tBu groups is evident \ and this group lies in a region of the structure where lower steric pressure is evident by \ observation of van der Waals surfaces and noting that the atoms are part of one of the \ conformationally less rigid StBu units which only bridges two gold atoms. Unmodelled disorder

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PLAT390_ALERT_3_B Deviating Methyl C7      X-C-H Bond Angle ......        119 Degree
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Author Response: This reflects the choice not to invoke restraints and introduce a more complicated model where the data is limited by the dominance of the \ Au30S19 core and by twinning. Significant disorder of the numerous tBu groups is evident \ and this group lies in a region of the structure where lower steric pressure is evident by \ observation of van der Waals surfaces and noting that the atoms are part of one of the \ conformationally less rigid StBu units which only bridges two gold atoms. Unmodelled disorder

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.

Author Response: This is an artefact of the modelling ot the disordered tBu group \ using a special shape which is not not readily interpreted by the checkcif routine

DIFMN03_ALERT_1_C The minimum difference density is < -0.1*ZMAX*0.75 The relevant atom site should be identified.

Author Response: This is within the metal core and is attributed to unmodelled \ relatively small twin components. The unmodelled twinning is well below the 10% minor component which is modelled

DIFMX02_ALERT_1_C The maximum difference density is > 0.1*ZMAX*0.75 The relevant atom site should be identified.

Author Response: This is within the metal core and is attributed to unmodelled \ relatively small twin components. The unmodelled twinning is well below the 10% minor component which is modelled

PLAT041_ALERT_1_C Calc. and Reported SumFormula Strings Differ Please Check

Author Response: This is an artefact of the modelling ot the disordered tBu group \ using a special shape which is not not readily interpreted by the checkcif routine

PLAT043_ALERT_1_C Calculated and Reported Mol. Weight Differ by .. 38.85 Check

Author Response: This is an artefact of the modelling ot the disordered tBu group using a special shape which is not not readily interpreted by the checkcif routine

PLAT068_ALERT_1_C Reported F000 Differs from Calcd (or Missing)... Please Check

Author Response: This is an artefact of the modelling ot the disordered tBu group using a special shape which is not not readily interpreted by the checkcif routine

PLAT222_ALERT_3_C Large Non-Solvent H Uiso(max)/Uiso(min) .. 6.4 Ratio

Author Response: There is significant disorder associated with the tBu groups which make a limited contribution to the scattering from the molecule which is dominated by the Au30S19 core- see refinement description

PLAT241_ALERT_2_C High Ueq as Compared to Neighbors for ..... Au3 Check

Author Response: This site is part of a staple unit at the opposite end of the molecule from the sulfide. There is potentially some element of conformational flexibility for the gold in this structure element. There is little to choose between a 2-site disorder model and a slightly larger displacement ellipsoid and the \ analyst has elected to retain a single site model

PLAT242_ALERT_2_C Low Ueq as Compared to Neighbors for ..... C17 Check

Author Response: This is unremarkable for the tertiary carbon of a tBu group in a \ structure where significant disorder of the numerous tBu groups is observed

PLAT242_ALERT_2_C Low Ueq as Compared to Neighbors for ..... C21 Check

Author Response: This is unremarkable for the tertiary carbon of a tBu group in a \ structure where significant disorder of the numerous tBu groups is observed

PLAT242_ALERT_2_C Low Ueq as Compared to Neighbors for ..... C25 Check

Author Response: This is unremarkable for the tertiary carbon of a tBu group in a \ structure where significant disorder of the numerous tBu groups is observed

PLAT242_ALERT_2_C Low Ueq as Compared to Neighbors for ..... C29 Check

Author Response: This is unremarkable for the tertiary carbon of a tBu group in a \ structure where significant disorder of the numerous tBu groups is observed

PLAT242_ALERT_2_C Low Ueq as Compared to Neighbors for ..... C33 Check

Author Response: This is unremarkable for the tertiary carbon of a tBu group in a \ structure where significant disorder of the numerous tBu groups is observed

PLAT242_ALERT_2_C Low Ueq as Compared to Neighbors for ..... C37 Check

Author Response: This is unremarkable for the tertiary carbon of a tBu group in a \ structure where significant disorder of the numerous tBu groups is observed

PLAT242_ALERT_2_C Low Ueq as Compared to Neighbors for ..... C45 Check

Author Response: This is unremarkable for the tertiary carbon of a tBu group in a \ structure where significant disorder of the numerous tBu groups is observed

PLAT242_ALERT_2_C Low Ueq as Compared to Neighbors for ..... C53 Check

Author Response: This is unremarkable for the tertiary carbon of a tBu group in a \ structure where significant disorder of the numerous tBu groups is observed

PLAT242_ALERT_2_C Low Ueq as Compared to Neighbors for ..... C57 Check

Author Response: This is unremarkable for the tertiary carbon of a tBu group in a \ structure where significant disorder of the numerous tBu groups is observed

PLAT242_ALERT_2_C Low Ueq as Compared to Neighbors for ..... C61 Check

Author Response: This is unremarkable for the tertiary carbon of a tBu group in a \ structure where significant disorder of the numerous tBu groups is observed

PLAT360_ALERT_2_C Short C(sp3)-C(sp3) Bond C1 - C3 ... 1.38 Ang.

Author Response: an unrestrained isotropic refinement has deliberately been employed this group is subject to significant disorder for which no further modelling was attempted

PLAT360_ALERT_2_C Short C(sp3)-C(sp3) Bond C17 - C19 ... 1.43 Ang.

Author Response: an unrestrained isotropic refinement has deliberately been employed this group is subject to significant disorder for which no further modelling was attempted

PLAT360_ALERT_2_C Short C(sp3)-C(sp3) Bond C17 - C20 ... 1.41 Ang.

Author Response: an unrestrained isotropic refinement has deliberately been employed this group is subject to significant disorder for which no further modelling was attempted

PLAT360_ALERT_2_C Short C(sp3)-C(sp3) Bond C33 - C34 ... 1.37 Ang.

Author Response: an unrestrained isotropic refinement has deliberately been employed this group is subject to significant disorder for which no further modelling was attempted

PLAT360_ALERT_2_C Short C(sp3)-C(sp3) Bond C53 - C55 ... 1.38 Ang.

Author Response: an unrestrained isotropic refinement has deliberately been employed this group is subject to significant disorder for which no further modelling was attempted

PLAT360_ALERT_2_C Short C(sp3)-C(sp3) Bond C61 - C62 ... 1.43 Ang.

Author Response: an unrestrained isotropic refinement has deliberately been employed this group is subject to significant disorder for which no further modelling was attempted

PLAT361_ALERT_2_C Long C(sp3)-C(sp3) Bond C41 - C44 ... 1.67 Ang.

Author Response: This reflects the choice not to invoke restraints and introduce a more complicated model where the data is limited by the dominance of the \ Au30S19 core and by twinning. Significant disorder of the numerous tBu groups is evident \ and this group lies in a region of the structure where lower steric pressure is evident by \ observation of van der Waals surfaces and noting that the atoms are part of one of the \ conformationally less rigid StBu units which only bridges two gold atoms. Unmodelled \ disorder

PLAT390_ALERT_3_C Deviating Methyl C2 X-C-H Bond Angle ..... 116 Degree

Author Response: This reflects the choice not to invoke restraints and introduce a more complicated model where the data is limited by the dominance of the \ Au30S19 core and by twinning. Significant disorder of the numerous tBu groups is evident \ and this group lies in a region of the structure where lower steric pressure is evident by \ observation of van der Waals surfaces and noting that the atoms are part of one of the \ conformationally less rigid StBu units which only bridges two gold atoms. Unmodelled disorder

PLAT390_ALERT_3_C Deviating Methyl C6 X-C-H Bond Angle ..... 118 Degree

Author Response: This reflects the choice not to invoke restraints and introduce a more complicated model where the data is limited by the dominance of the \ Au30S19 core and by twinning. Significant disorder of the numerous tBu groups is evident \ and this group lies in a region of the structure where lower steric pressure is evident by \ observation of van der Waals surfaces and noting that the atoms are part of one of the \ conformationally less rigid StBu units which only bridges two gold atoms. Unmodelled disorder

PLAT390_ALERT_3_C Deviating Methyl C59 X-C-H Bond Angle ..... 116 Degree

Author Response: This reflects the choice not to invoke restraints and introduce a more complicated model where the data is limited by the dominance of the \ Au30S19 core and by twinning. Significant disorder of the numerous tBu groups is evident \ and this group lies in a region of the structure where lower steric pressure is evident by \ observation of van der Waals surfaces and noting that the atoms are part of one of the \ conformationally less rigid StBu units which only bridges two gold atoms. Unmodelled disorder

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PLAT711_ALERT_1_C BOND    Unknown or Inconsistent Label .....        H70
                    S181   H70
```

Author Response: H70 is the model element which contains the 9 H atoms of the tBu \ included as an annulus in the region where disorder of a gold and sulfur atom has been \ modelled

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PLAT712_ALERT_1_C ANGLE   Unknown or Inconsistent Label .....        H70
                    AU23   S181   H70
```

Author Response: H70 is the model element which contains the 9 H atoms of the tBu \ included as an annulus in the region where disorder of a gold and sulfur atom has been \ modelled

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PLAT712_ALERT_1_C ANGLE   Unknown or Inconsistent Label .....        H70
                    AU41   S181   H70
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Author Response: H70 is the model element which contains the 9 H atoms of the tBu \ included as an annulus in the region where disorder of a gold and sulfur atom has been \ modelled

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PLAT712_ALERT_1_C ANGLE   Unknown or Inconsistent Label .....        H70
                    AU40   S181   H70
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Author Response: H70 is the model element which contains the 9 H atoms of the tBu \ included as an annulus in the region where disorder of a gold and sulfur atom has been \ modelled

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PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L=  0.600         16 Report
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Author Response: In the context of the complexity of this structure this is a small \ number

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PLAT913_ALERT_3_C Missing # of Very Strong Reflections in FCF ....          3 Note
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Author Response: A small number of strong reflections remain affected by unmodelled \ twinning and the weighting scheme employed has zero weighted them in a manner which is prope

PLAT920_ALERT_1_C Theta(Max) in CIF and FCF Differ by ....   0.16 Degree

Author Response: In Cu Ka radiation this difference is trivial

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Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite   6 Note
PLAT005_ALERT_5_G No _iucr_refine_instructions_details in the CIF   Please Do !
PLAT042_ALERT_1_G Calc. and Reported MoietyFormula Strings Differ   Please Check
PLAT301_ALERT_3_G Main Residue Disorder ..... Percentage =   2 Note
PLAT764_ALERT_4_G Overcomplete CIF Bond List Detected (Rep/Expd) .   1.21 Ratio
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... #   738 Check
             AU40 -S180 -AU41   1.555   1.555   1.555   13.99 Deg.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... #   744 Check
             AU41 -S181 -AU40   1.555   1.555   1.555   13.64 Deg.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... #   1159 Check
             S180 -C70 -S181   1.555   1.555   1.555   22.60 Deg.
PLAT808_ALERT_5_G No Parseable SHELXL Style Weighting Scheme Found   Please Check
PLAT860_ALERT_3_G Number of Least-Squares Restraints .....   6 Note
PLAT910_ALERT_3_G Missing # of FCF Reflections Below Th(Min) .....   4 Report
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600   905 Note
PLAT929_ALERT_5_G No Weight Pars,Obs and Calc R1,wR2,S not checked   ! Info
PLAT960_ALERT_3_G Number of Intensities with I <   - 2*sig(I) ...   77 Check

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

  13 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
  29 ALERT type 2 Indicator that the structure model may be wrong or deficient
  14 ALERT type 3 Indicator that the structure quality may be low
   5 ALERT type 4 Improvement, methodology, query or suggestion
   3 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 20/08/2014; check.def file version of 18/08/2014

Datablock I - ellipsoid plot