Chemistry of Heterocyclic Compounds **2022**, *58*(6/7), 368–370

Characterization and structure analysis of triclinic γ-cyclodextrin hydrate methanol solvate

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SUPPLEMENTARY INFORMATION

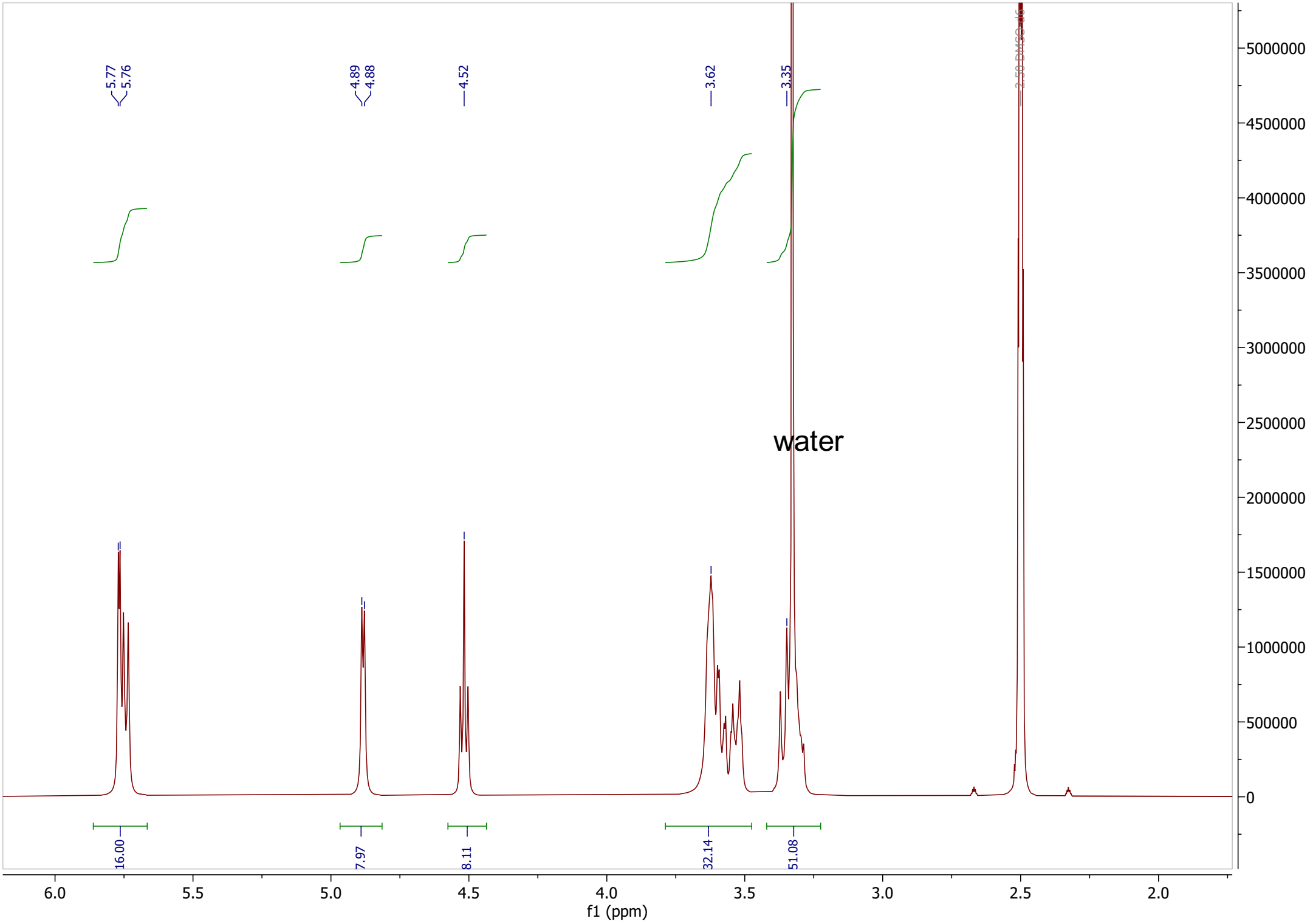
5.77
5.76
4.89
4.88
4.52
3.62
3.35
water
16.00
7.97
8.11
32.14
51.08
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
f1 (ppm)

Table S2. Crystal data and structure refinement of new γ -CD hydrate

Parameter	γ -CD hydrate
Radiation	Cu Kα (λ = 1.54184 Å)
Moiety chemical formula	$2(C_{48}H_{80}O_{40})\cdot 16(H_2O)\cdot CH_4O$
Crystal system	Triclinic
Space group	P1
a (Å)	15.12930(10)
b (Å)	16.60258(11)
c (Å)	17.28163(10)
α (°)	89.8670(5)
β (°)	83.1246(5)
γ (°)	82.2938(6)
Volume (Å^3)	4270.32(5)
Z	1
ρ_{calc} (cm^3)	1.133
μ (mm^{-1})	0.897
F(000)	1849
Crystal size (mm)	0.11 x 0.04 x 0.03
Temperature (K)	140
2Θ max. for data collection (°)	161
Reflections collected	185 443
Independent reflections	34 537
Goodness-of-fit on F^2	0.967
Final *R* indexes [$I > 2\sigma(I)$]	0.0712
Final *R* indexes [all data]	0.0743
Flack's *x* parameter	0.1(2)

Table S1. Full list of the hydrogen bond connection in the structure

Atom1	Atom2	Length, Å
O88	O84	2.828
O83	O80	2.646
O59	O64	2.713
O71	O67	2.851
O60	O87	2.744
O72	O75	2.823
O68	O63	2.795
O88	O158	2.754
O83	O155	2.834
O80	O151	2.433
O59	O164	2.785
O71	O170	2.764
O60	O163	2.431
O72	O174	2.799
O67	O169	2.899
O84	O157	2.936
O79	O152	2.786
O68	O165	2.697
O75	O173	2.695
O71	O157	2.675
O72	O155	3.026
O59	O152	2.465
O64	O147	2.963
O70	O168	2.655
O86	O154	2.716
O82	O150	2.927
O58	O160	2.824
O66	O5	2.881
O74	O172	2.769
O70	O175	2.782
O82	O176	2.808
O67	O177	2.808

O58	O179	2.876
O11	O181	3.013
O86	O181	2.74
O66	O186	2.716
O87	O187	2.715
O83	O1P	2.87
O80	O1	2.619
O27	O2S	2.976
O78	O2S	2.837
O20	O1N	2.842
O20	O1O	3.03
O82	O1N	2.827
O82	O1O	2.82
O60	O180	2.703
O60	O4	2.69
O76	O4Q	2.366
O64	O4Q	2.082
O63	O4Q	2.129
O152	O147	2.68
O155	O157	2.834
O170	O174	2.861
O156	O151	2.734
O158	O163	2.605
O169	O165	2.808
O148	O173	2.876
O172	O175	2.736
O154	O176	2.818
O158	O177	2.795
O150	O179	3.003
O162	O179	3.008
O89	O181	3.002
O168	O181	2.758
O160	O184	2.759
O169	O1Q	2.701
O156	O1P	3.04
O163	O1M	2.674
O115	O1S	2.939
O146	O2S	2.821
O150	O1N	2.732
O151	O180	2.703
O151	O4	2.717
O147	O4Q	2.101
O148	O4Q	2.188

O166	O4Q	2.752
O146	O4P	2.936
O175	O176	2.764
O175	O1R	2.782
O176	O2	2.797
O177	O1Q	2.696
O177	O8Q	2.943
O179	O1N	2.737
O181	O184	2.735
O181	O186	2.91
O184	O1Q	2.734
O186	O187	2.919
O187	O7Q	2.867
O1P	O1R	2.561
O1S	O8Q	3.019
O2S	O4P	2.266
O180	O8Q	2.721
O180	O7Q	2.718
O4	O7Q	2.455

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) kkb15

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: kkb15

Bond precision: C-C = 0.0073 A Wavelength=1.54184

Cell: a=15.12936(10) b=16.60258(11) c=17.28163(10)
alpha=89.8970(5) beta=83.1246(5) gamma=82.2938(6)
Temperature: 140 K

	Calculated	Reported
Volume	4270.32(5)	4270.32(5)
Space group	P 1	P 1
Hall group	P 1	P 1
Moiety formula	2(C48 H80 O40), C O, O2, 14(O) [+ solvent]	2(C48 H80 O40), 16(H2 O), C H4 O
Sum formula	C97 H160 O97 [+ solvent]	C97 H196 O97
Mr	2878.26	2912.97
Dx,g cm-3	1.119	1.133
Z	1	1
Mu (mm-1)	0.897	0.897
F000	1518.0	1849.0
F000'	1524.39	
h,k,lmax	19,21,22	19,21,21
Nref	37468[18734]	34537
Tmin,Tmax	0.958,0.973	
Tmin'	0.906	

Correction method= Not given

Data completeness= 1.84/0.92 Theta(max)= 80.456

R(reflections)= 0.0712(32052) wR2(reflections)= 0.2150(34537)

S = 0.967 Npar= 1743

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

PLAT312_ALERT_2_A Strange C-O-H Geometry (C-O < 1.25 Ang) ..... O146 Check

Author Response: The bond length is underestimated due to the libration effect of considerable thermal motions of the atoms (see W.R.Busing & H.A.Levy, Acta Cryst Perhaps there is occured also disorder.

PLAT415_ALERT_2_A Short Inter D-H..H-X H ..H8FA . 1.53 Ang.
x,y,-1+z = 1_554 Check

Author Response: This H...H distance is close to the sum of Van der Waals radii and can be tolerated for this complicated structure.

PLAT417_ALERT_2_A Short Inter D-H..H-D Hd ..Hr . 1.33 Ang.
1+x,y,z = 1_655 Check

Author Response: This H...H distance is close to the sum of Van der Waals radii and can be tolerated for this complicated structure.

PLAT430_ALERT_2_A Short Inter D...A Contact O2S ..O4P . 2.27 Ang.
x,y,z = 1_555 Check

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

PLAT430_ALERT_2_A Short Inter D...A Contact O4 ..O7Q . 2.46 Ang.
x,y,z = 1_555 Check

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

Alert level B

PLAT097_ALERT_2_B Large Reported Max. (Positive) Residual Density 0.84 eA-3

Author Response: Considering that the solvents are disordered, this value of residual d

PLAT201_ALERT_2_B Isotropic non-H Atoms in Main Residue(s) .... 6 Report
O222 C61 O5 O146 C5Q etc.

Author Response: The value of the goodness-of-fit is < 1, i.e. further increase in the number of refined parameters will not be entirely correc Furthermore, the refinement of these atoms in anisotropic approximations leads t

PLAT306_ALERT_2_B Isolated Oxygen Atom (H-atoms Missing ?) .... O2 Check

Author Response: There are 20 isolated oxygen atoms belonging to the water molecules. Unfortunately the difference synthesis was not able to reveal reliably positions of the related hydrogen atoms.

PLAT306_ALERT_2_B Isolated Oxygen Atom (H-atoms Missing ?) .... O175 Check

Author Response: There are 20 isolated oxygen atoms belonging to the water molecules. Unfortunately the difference synthesis was not able to reveal reliably positions of the related hydrogen atoms.

PLAT306_ALERT_2_B Isolated Oxygen Atom (H-atoms Missing ?) .... O176 Check

Author Response: There are 20 isolated oxygen atoms belonging to the water molecules. Unfortunately the difference synthesis was not able to reveal reliably positions of the related hydrogen atoms.

PLAT306_ALERT_2_B Isolated Oxygen Atom (H-atoms Missing ?) .... O177 Check

Author Response: There are 20 isolated oxygen atoms belonging to the water molecules. Unfortunately the difference synthesis was not able to reveal reliably positions of the related hydrogen atoms.

PLAT306_ALERT_2_B Isolated Oxygen Atom (H-atoms Missing ?) .... O179 Check

Author Response: There are 20 isolated oxygen atoms belonging to the water molecules. Unfortunately the difference synthesis was not able to reveal reliably positions of the related hydrogen atoms.

PLAT306_ALERT_2_B Isolated Oxygen Atom (H-atoms Missing ?) .... O181 Check

Author Response: There are 20 isolated oxygen atoms belonging to the water molecules. Unfortunately the difference synthesis was not able to reveal reliably positions of the related hydrogen atoms.

```
PLAT306_ALERT_2_B Isolated Oxygen Atom (H-atoms Missing ?) .......       O184 Check
```

Author Response: There are 20 isolated oxygen atoms belonging to the water molecules. Unfortunately the difference synthesis was not able to reveal reliably positions of the related hydrogen atoms.

```
PLAT306_ALERT_2_B Isolated Oxygen Atom (H-atoms Missing ?) .......       O186 Check
```

Author Response: There are 20 isolated oxygen atoms belonging to the water molecules. Unfortunately the difference synthesis was not able to reveal reliably positions of the related hydrogen atoms.

```
PLAT306_ALERT_2_B Isolated Oxygen Atom (H-atoms Missing ?) .......       O187 Check
```

Author Response: There are 20 isolated oxygen atoms belonging to the water molecules. Unfortunately the difference synthesis was not able to reveal reliably positions of the related hydrogen atoms.

```
PLAT416_ALERT_2_B Short Intra D-H..H-D       Hu       ..H5GA     .       1.57 Ang.
                                                      x,y,z  =      1_555 Check
```

Author Response: It is not possible to perform a refinement for hydrogen atoms. Therefore, the geometric parameters for H atoms may not be realistic and should

```
PLAT417_ALERT_2_B Short Inter D-H..H-D       Hb       ..Hc       .       2.06 Ang.
                                                      x,y,z  =      1_555 Check
```

Author Response: This H...H distance is close to the sum of Van der Waals radii and can be tolerated for this complicated structure.

```
PLAT417_ALERT_2_B Short Inter D-H..H-D       H4AA     ..H5AA     .       2.06 Ang.
                                                      x,y,z  =      1_555 Check
```

Author Response: This H...H distance is close to the sum of Van der Waals radii and can be tolerated for this complicated structure.

```
PLAT420_ALERT_2_B D-H Bond Without Acceptor  O88      --H        .     Please Check
```

Author Response: Unfortunately the difference synthesis was not able to reveal reliably positions for all hydrogen atoms. Therefore their orientation may be not entirely accurate.

PLAT420_ALERT_2_B D-H Bond Without Acceptor O79 --Hw . Please Check

Author Response: Unfortunately the difference synthesis was not able to reveal reliably positions for all hydrogen atoms. Therefore their orientation may be not entirely accurate.

PLAT420_ALERT_2_B D-H Bond Without Acceptor O154 --Hy . Please Check

Author Response: Unfortunately the difference synthesis was not able to reveal reliably positions for all hydrogen atoms. Therefore their orientation may be not entirely accurate.

PLAT420_ALERT_2_B D-H Bond Without Acceptor O150 --H3AA . Please Check

Author Response: Unfortunately the difference synthesis was not able to reveal reliably positions for all hydrogen atoms. Therefore their orientation may be not entirely accurate.

PLAT420_ALERT_2_B D-H Bond Without Acceptor O75 --H5AA . Please Check

Author Response: Unfortunately the difference synthesis was not able to reveal reliably positions for all hydrogen atoms. Therefore their orientation may be not entirely accurate.

PLAT420_ALERT_2_B D-H Bond Without Acceptor O172 --H6AA . Please Check

Author Response: Unfortunately the difference synthesis was not able to reveal reliably positions for all hydrogen atoms. Therefore their orientation may be not entirely accurate.

PLAT420_ALERT_2_B D-H Bond Without Acceptor O76 --H6BA . Please Check

Author Response: Unfortunately the difference synthesis was not able to reveal reliably positions for all hydrogen atoms. Therefore their orientation may be not entirely accurate.

PLAT420_ALERT_2_B D-H Bond Without Acceptor O222 --H2NA . Please Check

Author Response: Unfortunately the difference synthesis was not able to reveal reliably positions for all hydrogen atoms. Therefore their orientation may be not entirely accurate.

```
PLAT430_ALERT_2_B Short Inter D...A Contact  O1N      ..O179     .       2.74 Ang.
                                                     x,y,z  =      1_555 Check
```

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

```
PLAT430_ALERT_2_B Short Inter D...A Contact  O1N      ..O20      .       2.84 Ang.
                                                     x,y,z  =      1_555 Check
```

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

```
PLAT430_ALERT_2_B Short Inter D...A Contact  O1P      ..O1R      .       2.56 Ang.
                                                     x,y,z  =      1_555 Check
```

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

```
PLAT430_ALERT_2_B Short Inter D...A Contact  O1Q      ..O177     .       2.70 Ang.
                                                     x,y,z  =      1_555 Check
```

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

```
PLAT430_ALERT_2_B Short Inter D...A Contact  O1Q      ..O184     .       2.73 Ang.
                                                     x,y,z  =      1_555 Check
```

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

```
PLAT430_ALERT_2_B Short Inter D...A Contact  O1R      ..O175     .       2.78 Ang.
                                              1+x,y,-1+z  =      1_654 Check
```

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

```
PLAT430_ALERT_2_B Short Inter D...A Contact  O2       ..O176     .       2.80 Ang.
                                               x,y,-1+z  =      1_554 Check
```

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

```
PLAT430_ALERT_2_B Short Inter D...A Contact  O7Q      ..O180     .       2.72 Ang.
                                               x,y,z  =      1_555 Check
```

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

```
PLAT430_ALERT_2_B Short Inter D...A Contact  O8Q      ..O180     .       2.72 Ang.
                                               x,y,z  =      1_555 Check
```

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

```
PLAT430_ALERT_2_B Short Inter D...A Contact  O175     ..O176     .       2.76 Ang.
                                               x,y,z  =      1_555 Check
```

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

```
PLAT430_ALERT_2_B Short Inter D...A Contact  O181     ..O184     .       2.73 Ang.
                                              x,y,1+z  =      1_556 Check
```

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

```
PLAT934_ALERT_3_B Number of (Iobs-Icalc)/Sigma(W) > 10 Outliers ..          3 Check
```

Author Response: Removal of reflections for which (Iobs-Icalc)/Sigma(W) > 10 practicall the values of the refinement parameters.

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.
DIFMX02_ALERT_1_C  The maximum difference density is > 0.1*ZMAX*0.75
           The relevant atom site should be identified.
PLAT213_ALERT_2_C Atom O75             has ADP max/min Ratio .....        3.6 prolat
PLAT213_ALERT_2_C Atom O166            has ADP max/min Ratio .....        3.8 prolat
PLAT220_ALERT_2_C NonSolvent   Resd 1  O   Ueq(max)/Ueq(min) Range        4.4 Ratio
PLAT220_ALERT_2_C NonSolvent   Resd 2  C   Ueq(max)/Ueq(min) Range        3.2 Ratio
PLAT220_ALERT_2_C NonSolvent   Resd 2  O   Ueq(max)/Ueq(min) Range        4.5 Ratio
PLAT222_ALERT_3_C NonSolvent Resd 1  H   Uiso(max)/Uiso(min) Range        5.4 Ratio
PLAT222_ALERT_3_C NonSolvent Resd 2  H   Uiso(max)/Uiso(min) Range        5.9 Ratio
PLAT230_ALERT_2_C Hirshfeld Test Diff for    O150     --C149     .        7.0 s.u.
PLAT230_ALERT_2_C Hirshfeld Test Diff for    O162     --C161     .        5.5 s.u.
PLAT234_ALERT_4_C Large Hirshfeld Difference O166     --C105     .       0.16 Ang.
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of        C24 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of        C57 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of       C106 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of       C149 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including        O2      0.108 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including      O179      0.125 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including      O184      0.112 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including      O186      0.104 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including      O187      0.155 Check
PLAT309_ALERT_2_C Single Bonded Oxygen (C-O > 1.3 Ang) ...........         O1 Check
PLAT340_ALERT_3_C Low Bond Precision on  C-C Bonds ..............    0.00731 Ang.
```

Author Response: This is a consequence of high level of disorder in the structure, high percentage of solvent area volume and crystal twinning that infleunced the quality of diffraction data.

```
PLAT410_ALERT_2_C Short Intra H...H Contact  H6GA     ..H8HA     .       1.99 Ang.
                                               x,y,z  =      1_555 Check
PLAT411_ALERT_2_C Short Inter H...H Contact  H8HA     ..H5LA     .       2.11 Ang.
                                             x,-1+y,z  =      1_545 Check
PLAT411_ALERT_2_C Short Inter H...H Contact  H8KA     ..H2LA     .       2.08 Ang.
                                              x,1+y,z  =      1_565 Check
PLAT416_ALERT_2_C Short Intra D-H..H-D       Ha       ..H0AA     .       1.92 Ang.
                                               x,y,z  =      1_555 Check
```

Author Response: It is not possible to perform a refinement for hydrogen atoms. Therefore, the geometric parameters for H atoms may not be realistic and should

```
PLAT417_ALERT_2_C Short Inter D-H..H-D       Hh       ..Hi       .       2.14 Ang.
                                                    x,y,z  =      1_555 Check
```

Author Response: This H...H distance is close to the sum of Van der Waals radii and can be tolerated for this complicated structure.

```
PLAT430_ALERT_2_C Short Inter D...A Contact  O7Q      ..O187     .       2.87 Ang.
                                                  x,-1+y,z  =      1_545 Check
```

Author Response: All mentioned distances represent hydrogen bonds of O-H...O type and their lengths are close to standard value. The problem arose because the corresponding hydrogen atoms were not included in the calculations since the difference synthesis was not able to reveal reliably their positions.

```
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600         14 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) .          8 Check
PLAT975_ALERT_2_C Check Calcd Resid. Dens.  1.08Ang From O181    .       0.76 eA-3
PLAT975_ALERT_2_C Check Calcd Resid. Dens.  1.01Ang From O146    .       0.68 eA-3
PLAT975_ALERT_2_C Check Calcd Resid. Dens.  0.67Ang From O1P     .       0.65 eA-3
PLAT975_ALERT_2_C Check Calcd Resid. Dens.  0.82Ang From O1P     .       0.61 eA-3
PLAT975_ALERT_2_C Check Calcd Resid. Dens.  0.80Ang From O76     .       0.56 eA-3
PLAT975_ALERT_2_C Check Calcd Resid. Dens.  0.78Ang From O1S     .       0.52 eA-3
PLAT975_ALERT_2_C Check Calcd Resid. Dens.  0.41Ang From O1Q     .       0.48 eA-3
PLAT975_ALERT_2_C Check Calcd Resid. Dens.  0.89Ang From O1S     .       0.42 eA-3
```

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:C97 H196 O97
            Atom count from the _atom_site data:  C97 H160 O97
CELLZ01_ALERT_1_G Difference between formula and atom_site contents detected.
CELLZ01_ALERT_1_G WARNING: H atoms missing from atom site list. Is this intentional?
           From the CIF: _cell_formula_units_Z    1
           From the CIF: _chemical_formula_sum  C97 H196 O97
           TEST: Compare cell contents of formula and atom_site data

           atom    Z*formula  cif sites diff
           C         97.00     97.00    0.00
           H        196.00    160.00   36.00
           O         97.00     97.00    0.00
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms .............         48 Report
PLAT041_ALERT_1_G Calc. and Reported SumFormula    Strings  Differ     Please Check
PLAT042_ALERT_1_G Calc. and Reported MoietyFormula Strings  Differ     Please Check
PLAT072_ALERT_2_G SHELXL First  Parameter in WGHT  Unusually Large       0.20 Report
PLAT300_ALERT_4_G Atom Site Occupancy of O1         Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of C6Q        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O1M        Constrained at        0.5 Check
```

```
PLAT300_ALERT_4_G Atom Site Occupancy of C183       Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O1N        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O1O        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O4         Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O180       Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O1P        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O1Q        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O1R        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O1S        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O2S        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O4P        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O4Q        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O7N        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O7Q        Constrained at        0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of O8Q        Constrained at        0.5 Check
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  3  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  4  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  5  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  6  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  7  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  8  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd  9  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 10  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 12  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 13  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 14  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 15  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 16  )       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 17  )       100% Note
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd  7  )       0.50 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd  8  )       0.50 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd  9  )       0.50 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd 10  )       0.50 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd 12  )       0.50 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd 13  )       0.50 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd 14  )       0.50 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd 15  )       0.50 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd 16  )       0.50 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd 17  )       0.50 Check
PLAT309_ALERT_2_G Single Bonded Oxygen (C-O > 1.3 Ang) .............      O1M Check
PLAT311_ALERT_2_G Isolated Disordered Oxygen Atom (No H's ?) .....        O1P Check
PLAT311_ALERT_2_G Isolated Disordered Oxygen Atom (No H's ?) .....        O1Q Check
PLAT311_ALERT_2_G Isolated Disordered Oxygen Atom (No H's ?) .....        O1R Check
PLAT311_ALERT_2_G Isolated Disordered Oxygen Atom (No H's ?) .....        O1S Check
PLAT311_ALERT_2_G Isolated Disordered Oxygen Atom (No H's ?) .....        O2S Check
PLAT311_ALERT_2_G Isolated Disordered Oxygen Atom (No H's ?) .....        O4P Check
PLAT311_ALERT_2_G Isolated Disordered Oxygen Atom (No H's ?) .....        O4Q Check
PLAT311_ALERT_2_G Isolated Disordered Oxygen Atom (No H's ?) .....        O7N Check
PLAT311_ALERT_2_G Isolated Disordered Oxygen Atom (No H's ?) .....        O7Q Check
PLAT311_ALERT_2_G Isolated Disordered Oxygen Atom (No H's ?) .....        O8Q Check
PLAT315_ALERT_2_G Singly Bonded Carbon Detected (H-atoms Missing).        C6Q Check
```

Author Response: Singly Bonded Carbon atoms C8, C8ZZ, C11, C14 and C183 belong to methanol molecules. In the absence of any hydrogen atoms the program does not allow to generate other hydrogen atoms geometrically.

```
PLAT315_ALERT_2_G Singly Bonded Carbon Detected (H-atoms Missing).       C183 Check
```

Author Response: Singly Bonded Carbon atoms C8, C8ZZ, C11, C14 and C183 belong to methanol molecules. In the absence of any hydrogen atoms the program does not allow to generate other hydrogen atoms geometrically.

```
PLAT432_ALERT_2_G Short Inter X...Y Contact  O4Q      ..C140     .       2.88 Ang.
                                                   x,1+y,z  =      1_565 Check
PLAT432_ALERT_2_G Short Inter X...Y Contact  O4Q      ..C45      .       2.90 Ang.
                                                     x,y,z  =      1_555 Check
PLAT432_ALERT_2_G Short Inter X...Y Contact  O4Q      ..C44      .       2.90 Ang.
                                                     x,y,z  =      1_555 Check
PLAT432_ALERT_2_G Short Inter X...Y Contact  O4Q      ..C139     .       2.93 Ang.
                                                   x,1+y,z  =      1_565 Check
PLAT606_ALERT_4_G Solvent Accessible VOID(S) in Structure ........          ! Info
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..........        159 Note
PLAT721_ALERT_1_G Bond    Calc     0.84000, Rep     0.89210 Dev...       0.05 Ang.
             O76      -H6BA            1_555   1_555 ........     #  154 Check
PLAT721_ALERT_1_G Bond    Calc     0.84000, Rep     0.86600 Dev...       0.03 Ang.
             O166     -H9FA            1_555   1_555 ........     #  243 Check
PLAT722_ALERT_1_G Angle   Calc      109.00, Rep      110.30 Dev...       1.30 Degree
             C24      -O75     -H5AA       1_555  1_555  1_555     #   63 Check
PLAT722_ALERT_1_G Angle   Calc      110.00, Rep      114.40 Dev...       4.40 Degree
             C23      -O76     -H6BA       1_555  1_555  1_555     #   96 Check
PLAT722_ALERT_1_G Angle   Calc      109.00, Rep      112.40 Dev...       3.40 Degree
             C105     -O166    -H9FA       1_555  1_555  1_555     #  332 Check
PLAT722_ALERT_1_G Angle   Calc      110.00, Rep      108.80 Dev...       1.20 Degree
             O74      -C73     -H1MA       1_555  1_555  1_555     #  626 Check
PLAT722_ALERT_1_G Angle   Calc      110.00, Rep      111.30 Dev...       1.30 Degree
             O5       -C5Q     -H9MA       1_555  1_555  1_555     #  648 Check
PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd.  #         12 Note
             O
PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd.  #         18 Note
             O
PLAT791_ALERT_4_G Model has Chirality at C1          (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C2          (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C4          (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C5          (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C6          (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C7          (Sohnke SpGr)          R Verify
PLAT791_ALERT_4_G Model has Chirality at C9          (Sohnke SpGr)          R Verify
PLAT791_ALERT_4_G Model has Chirality at C10         (Sohnke SpGr)          R Verify
PLAT791_ALERT_4_G Model has Chirality at C12         (Sohnke SpGr)          R Verify
PLAT791_ALERT_4_G Model has Chirality at C13         (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C15         (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C16         (Sohnke SpGr)          R Verify
PLAT791_ALERT_4_G Model has Chirality at C17         (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C18         (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C19         (Sohnke SpGr)          R Verify
PLAT791_ALERT_4_G Model has Chirality at C22         (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C23         (Sohnke SpGr)          R Verify
PLAT791_ALERT_4_G Model has Chirality at C24         (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C25         (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C26         (Sohnke SpGr)          R Verify
PLAT791_ALERT_4_G Model has Chirality at C29         (Sohnke SpGr)          S Verify
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PLAT791_ALERT_4_G Model has Chirality at C30 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C31 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C32 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C33 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C36 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C37 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C38 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C39 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C40 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C43 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C44 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C45 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C46 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C47 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C50 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C51 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C52 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C53 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C54 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C90 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C91 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C93 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C94 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C96 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C97 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C98 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C99 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C100 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C101 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C104 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C105 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C106 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C107 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C108 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C110 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C111 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C112 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C113 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C114 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C118 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C119 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C120 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C121 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C122 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C124 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C125 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C126 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C127 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C128 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C131 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C132 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C133 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C134 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C135 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C138 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C139 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C140 (Sohnke SpGr) S Verify

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PLAT791_ALERT_4_G Model has Chirality at C141        (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C142        (Sohnke SpGr)          R Verify
PLAT802_ALERT_4_G CIF Input Record(s) with more than 80 Characters          7 Info
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary .     Please Do !
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        341 Note
PLAT913_ALERT_3_G Missing # of Very Strong Reflections in FCF ....          1 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          0 Info
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   5 ALERT level A = Most likely a serious problem - resolve or explain
  34 ALERT level B = A potentially serious problem, consider carefully
  39 ALERT level C = Check. Ensure it is not caused by an omission or oversight
 162 ALERT level G = General information/check it is not something unexpected

  14 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
  89 ALERT type 2 Indicator that the structure model may be wrong or deficient
   7 ALERT type 3 Indicator that the structure quality may be low
 129 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 18/05/2022; check.def file version of 17/05/2022

Datablock kkb15 - ellipsoid plot