Structure p-1 (CCDC 1568403): C$_{147}$H$_{120}$N$_6$O$_{14}$

# start Validation Reply Form

_vrf_<990_ALERT_1_B>

PROBLEM: <Deprecated .res/.hkl Input Style SQUEEZE Job ... ! Note.>

RESPONSE: <Due to the crystals had large voids with badly disordered solvent molecules, the crystallographic refinement was completed with the solvent contribution subtracted from the data using SQUEEZE from the PLATON package of crystallographic software. Although we try our best to solve this problem, the problem remains because of the different solvents were used and we can’t use the latest edition PLATON to exactly calculate the quantity of solvent molecule squeezed.>

_vrf_<026_ALERT_3_B>

029_ALERT_3_B
220_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
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230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
241_ALERT_2_B
242_ALERT_2_B
242_ALERT_2_B
242_ALERT_2_B
242_ALERT_2_B
250_ALERT_2_B
420_ALERT_2_B
420_ALERT_2_B>
PROBLEM: < Ratio Observed / Unique Reflections (too) Low.. 37% Check
diffrn_measured_fraction_theta_full value Low. 0.957 Why?
Non-Solvent Resd 1 C Ueq(max)/Ueq(min) Range 8.1 Ratio
Hirshfeld Test Diff for C133 --C137_a . 7.9 s.u.
Hirshfeld Test Diff for C134 --C135 . 7.4 s.u.
Hirshfeld Test Diff for C136 --C137 . 9.3 s.u.
Hirshfeld Test Diff for C137 --C133_a . 7.9 s.u.
Hirshfeld Test Diff for C140 --C141 . 10.3 s.u.
Hirshfeld Test Diff for C143 --C144 . 8.0 s.u.
High 'MainMol' Ueq as Compared to Neighbors of C140 Check
Low 'MainMol' Ueq as Compared to Neighbors of C121 Check
Low 'MainMol' Ueq as Compared to Neighbors of C141 Check
Low 'MainMol' Ueq as Compared to Neighbors of C143 Check
Low 'MainMol' Ueq as Compared to Neighbors of C146 Check
Large U3/U1 Ratio for Average U(i,j) Tensor .... 4.5 Note
D-H Without Acceptor O13 --H13 Please Check
D-H Without Acceptor O14 --H14 Please Check >

RESPONSE: < After the dogged refinements, some alerts (level B) have also been remarkably improved but cannot be settled satisfactorily (see the new alerts), which have been reasonably explained as following:

PLAT026_ALERT_3_B Ratio Observed / Unique Reflections (too) Low .. 37% Check
PLAT029_ALERT_3_B diffrn_measured_fraction_theta_full value Low . 0.957 Why?
PLAT220_ALERT_2_B Non-Solvent Resd 1 C Ueq(max)/Ueq(min) Range 8.0 Ratio
PLAT230_ALERT_2_B Hirshfeld Test Diff for C3 --C6 . 7.9 s.u.
PLAT230_ALERT_2_B Hirshfeld Test Diff for C03W --C048 . 7.6 s.u.
PLAT230_ALERT_2_B Hirshfeld Test Diff for C04D --C04E . 9.7 s.u.
PLAT230_ALERT_2_B Hirshfeld Test Diff for C04I --C04M . 9.2 s.u.
PLAT241_ALERT_2_B High 'MainMol' Ueq as Compared to Neighbors of C3 Check
Response: Again, due to the large voids with highly volatile solvent molecules, the crystals were unstable and thus their diffraction data are too bad to settle the alerts satisfactorily. However, these alerts do not affect the main results in this manuscript.

_PROBLEM:_ D-H Without Acceptor O04J --H2BA Please Check.

_RESPONSE:_ The solvent molecules are ethanol and the direction of H2BA is toward VOID(S).

_PROBLEM:_ Missing # of FCF Reflection(s) Below Theta(Min). 13 Note.

_RESPONSE:_ This alert may be caused by several diffraction data sheltered by the beam stop.

# end Validation Reply Form
Structure p21c (CCDC 1568405): C_{66}H_{62}N_{3}O_{7}

# start Validation Reply Form

_vrf_<080_ALERT_2_A
084_ALERT_3_A
213_ALERT_2_A
375_ALERT_2_A
082_ALERT_2_B
097_ALERT_2_B
213_ALERT_2_B
220_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
230_ALERT_2_B
234_ALERT_4_B
241_ALERT_2_B
242_ALERT_2_B
242_ALERT_2_B
340_ALERT_3_B
420_ALERT_2_B
601_ALERT_2_B>

PROBLEM: < Maximum Shift/Error ............................ 0.79 Why?

High wR2 Value (i.e. > 0.25) .................. 0.53 Report
Atom C27 has ADP max/min Ratio ..... 5.8 oblate
Strange C-O-H Geometry (C-O > 1.45 Ang) ..... O7 Check
High R1 Value .............................. 0.20 Report
Large Reported Max. (Positive) Residual Density 1.29 eA-3
Atom C11 has ADP max/min Ratio ..... 4.5 prolat
Non-Solvent Resd 1 C Ueq(max)/Ueq(min) Range 7.6 Ratio
Hirshfeld Test Diff for C1 --C8 . 10.9 s.u.
Hirshfeld Test Diff for C3 --C4 . 11.1 s.u.
Hirshfeld Test Diff for C14 --C18 . 7.4 s.u.
Hirshfeld Test Diff for C26 --C27 . 10.8 s.u.
Hirshfeld Test Diff for C31 --C32 . 8.6 s.u.
Hirshfeld Test Diff for C39 --C40 . 16.0 s.u.
Hirshfeld Test Diff for C39 --C44 . 7.7 s.u.
Hirshfeld Test Diff for C62 --C63 . 7.5 s.u.
Large Hirshfeld Difference C54 --C62 0.28 Ang.
High 'MainMol' Ueq as Compared to Neighbors of C62 Check
Low 'MainMol' Ueq as Compared to Neighbors of C10 Check
Low 'MainMol' Ueq as Compared to Neighbors of C59 Check
Low Bond Precision on C-C Bonds ............... 0.01233 Ang.
D-H Without Acceptor O7 --H7 Please Check
Structure Contains Solvent Accessible VOIDS of . 135 Ang**3>

RESPONSE: < After the dogged refinements, most of the alerts been settled satisfactorily or at least remarkably improved (see the updated CIFs, which have been re-submitted to the CCDC). For those cannot be settled satisfactorily (see the new alerts), they have been reasonably explained as following:

PLAT084_ALERT_3_A High wR2 Value (i.e. > 0.25) ................. 0.51 Report
PLAT082_ALERT_2_B High R1 Value .............................. 0.18 Report
PLAT201_ALERT_2_B Isotropic non-H Atoms in Main Residue(s) ....... 1 Report
Response: Similarly, due to the large voids with highly volatile solvent molecules, the crystals were unstable and thus their diffraction data are too bad to settle the alerts satisfactorily. However, these alerts do not affect the main results in this manuscript.>

_vrf_<910_ALERT_3_B>;

PROBLEM: <Missing # of FCF Reflection(s) Below Theta(Min). 29 Note.>

RESPONSE: <Similarly, alert may be caused by several diffraction data sheltered by the beam stop.>

_vrf_<990_ALERT_1_B>;

PROBLEM: <Deprecated .res/.hkl Input Style SQUEEZE Job ... ! Note.>

RESPONSE: <Due to the crystals had large voids with badly disordered solvent molecules, the crystallographic refinement was completed with the solvent contribution subtracted from the data using SQUEEZE from the PLATON package of crystallographic software. Although we try our best to solve this problem, the problem remains because of the different solvents were used and we can’t use the latest edition PLATON to exactly calculate the quantity of solvent molecule squeezed.>

# end Validation Reply Form