Structure p-1 (CCDC 1568403): $C_{147}H_{120}N_6O_{14}$

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

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

PLAT242_ALERT_2_B Low ’MainMol’ Ueq as Compared to Neighbors of C03H Check

PLAT242_ALERT_2_B Low ’MainMol’ Ueq as Compared to Neighbors of C04D Check

PLAT250_ALERT_2_B Large U3/U1 Ratio for Average U(i,j) Tensor .... 4.3 Note

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.

vrf<420_ALERT_2_B >;

PROBLEM: < D-H Without Acceptor O04J --H2BA Please Check.>

RESPONSE: < The solvent molecules are ethanol and the direction of H2BA is toward VOID(S).>;

vrf<910_ALERT_3_B >;

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): $C_{66}H_{62}N_3O_7$

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

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

PLAT220_ALERT_2_B Non-Solvent Resd 1 C Ueq(max)/Ueq(min) Range 9.4 Ratio

PLAT230_ALERT_2_B Hirshfeld Test Diff for O005 --C00X . 7.2 s.u.

PLAT230_ALERT_2_B Hirshfeld Test Diff for O006 --C00Y . 9.8 s.u.

PLAT230_ALERT_2_B Hirshfeld Test Diff for C00E --C015 . 10.6 s.u.

PLAT230_ALERT_2_B Hirshfeld Test Diff for C00O --C00Q . 9.0 s.u.

PLAT230_ALERT_2_B Hirshfeld Test Diff for C013 --C01G . 7.4 s.u.

PLAT230_ALERT_2_B Hirshfeld Test Diff for C013 --C01P . 7.8 s.u.

PLAT230_ALERT_2_B Hirshfeld Test Diff for C019 --C01B . 17.0 s.u.

PLAT230_ALERT_2_B Hirshfeld Test Diff for C019 --C01E . 9.5 s.u.

PLAT230_ALERT_2_B Hirshfeld Test Diff for C01C --C01M . 9.6 s.u.

PLAT234_ALERT_4_B Large Hirshfeld Difference C01T --C022 0.28 Ang.

PLAT234_ALERT_4_B Large Hirshfeld Difference C01U --C020 0.26 Ang.

PLAT340_ALERT_3_B Low Bond Precision on C-C Bonds ..... 0.01187 Ang.

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 co mpleted 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