Electronic Supporting Information

Effect of Solvent Polarity in Mechanochemistry: Preparation of Conglomerate vs. Racemate

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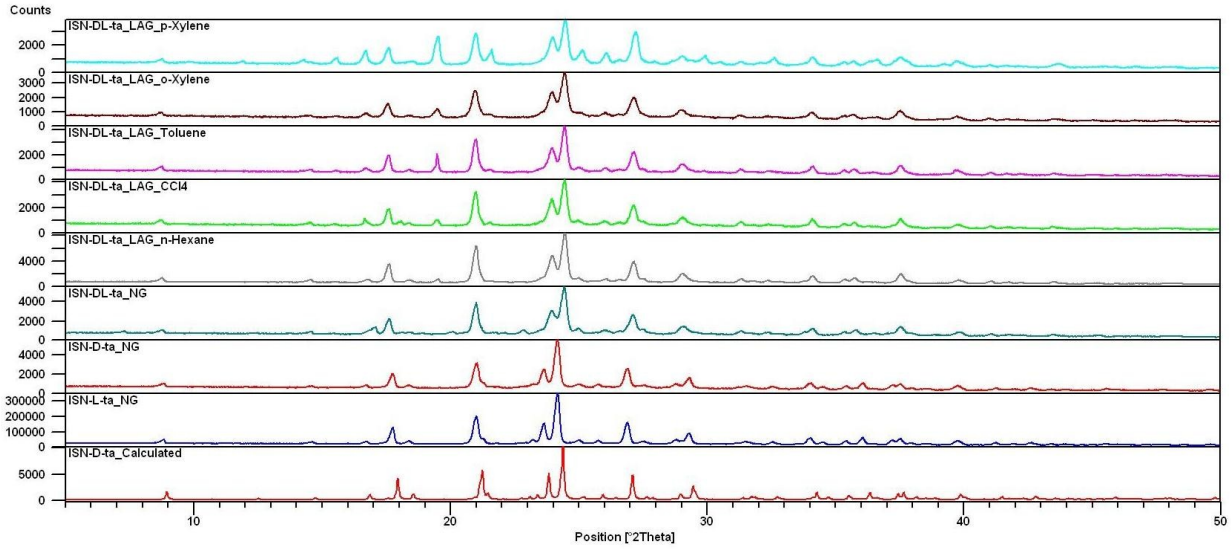


Figure S1. PXRD patterns of equimolar mixture of ISN and DL-ta obtained using LAG with various non-polar liquids compared with samples obtained from enantiomeric ISN-D-ta/ L-ta LAG as well as calculated powder pattern of chiral salt ISN•D-ta.

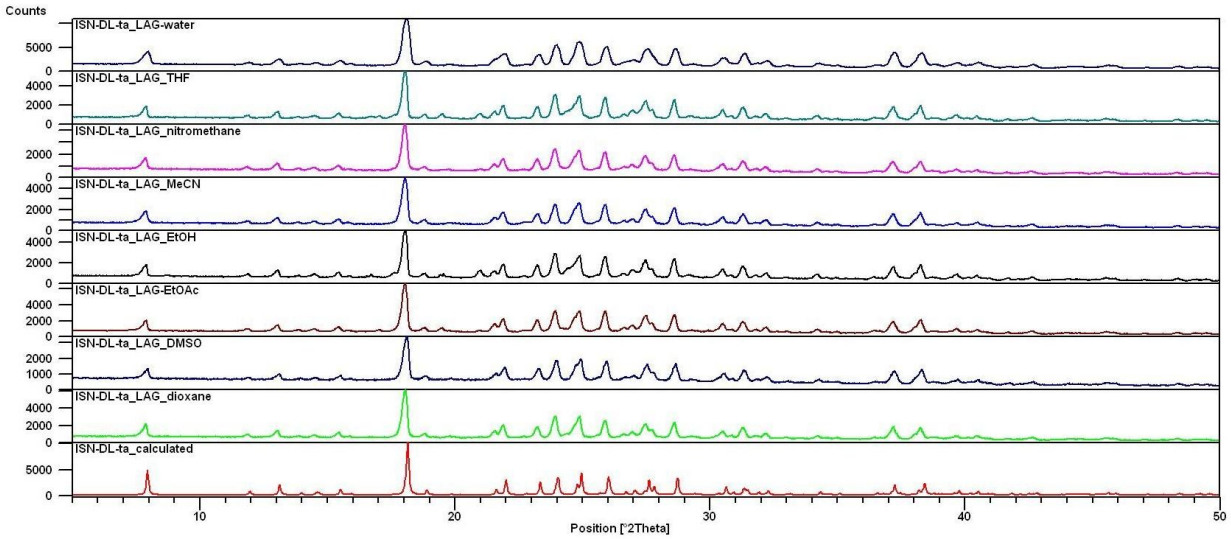


Figure S2. PXRD patterns of equimolar mixture of ISN and DL-ta obtained using LAG with various polar liquids compared with calculated powder pattern of racemic ISN•DL-ta salt.

ATR FT-IR spectra of the respective conglomerate and racemate of ISN and DL-ta showed characteristic shift of stretching frequency 1720-1680 cm^{-1} correspond to carboxylic acid group (COOH) of tartaric acid to lower wavenumber corresponding to carboxylate group (COO^{-}) confirms formation of salt structure for both the multicomponent solids.

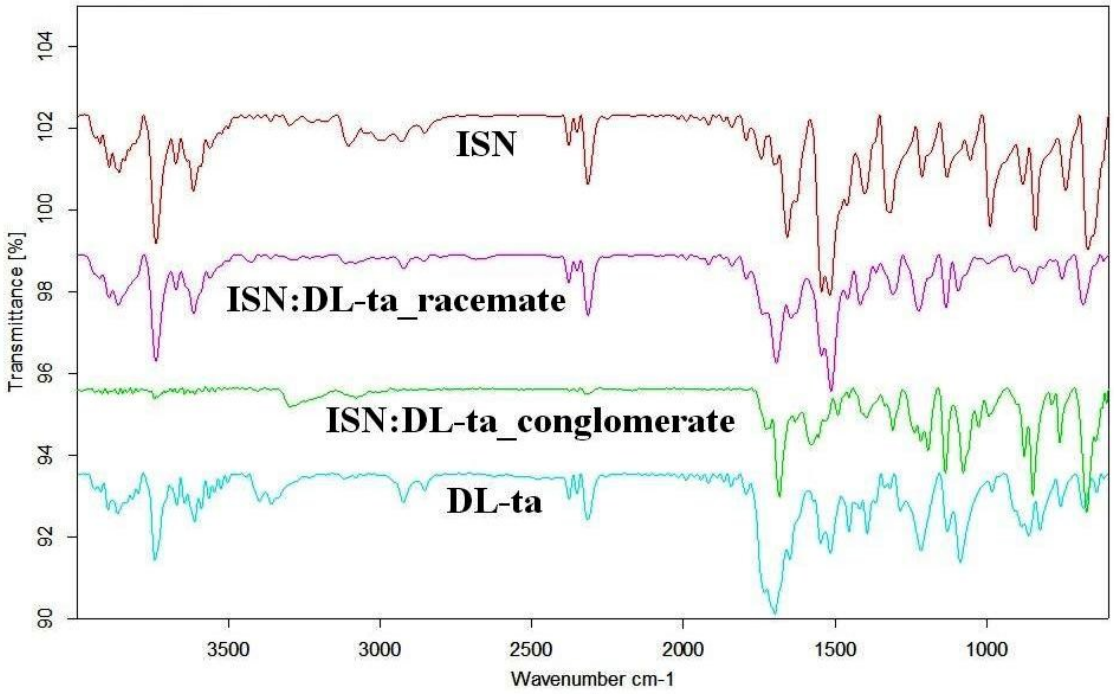
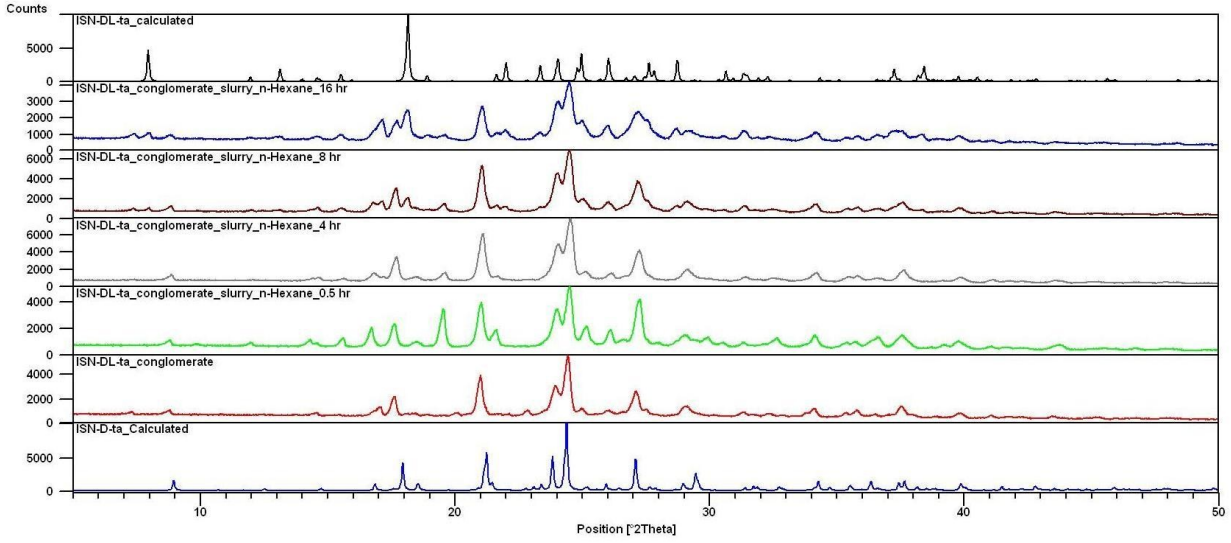
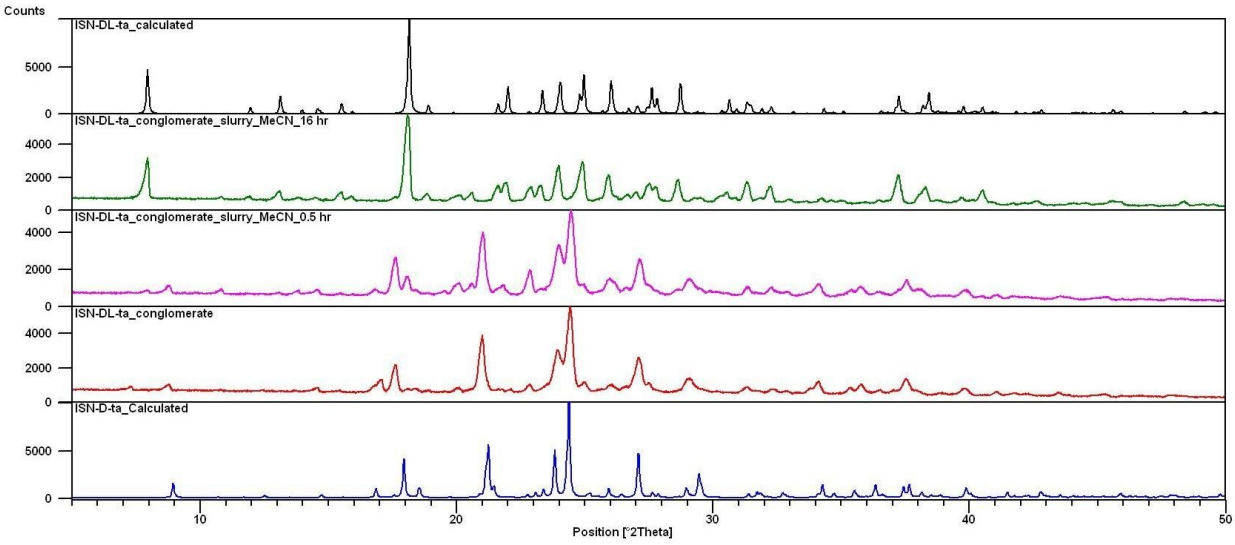


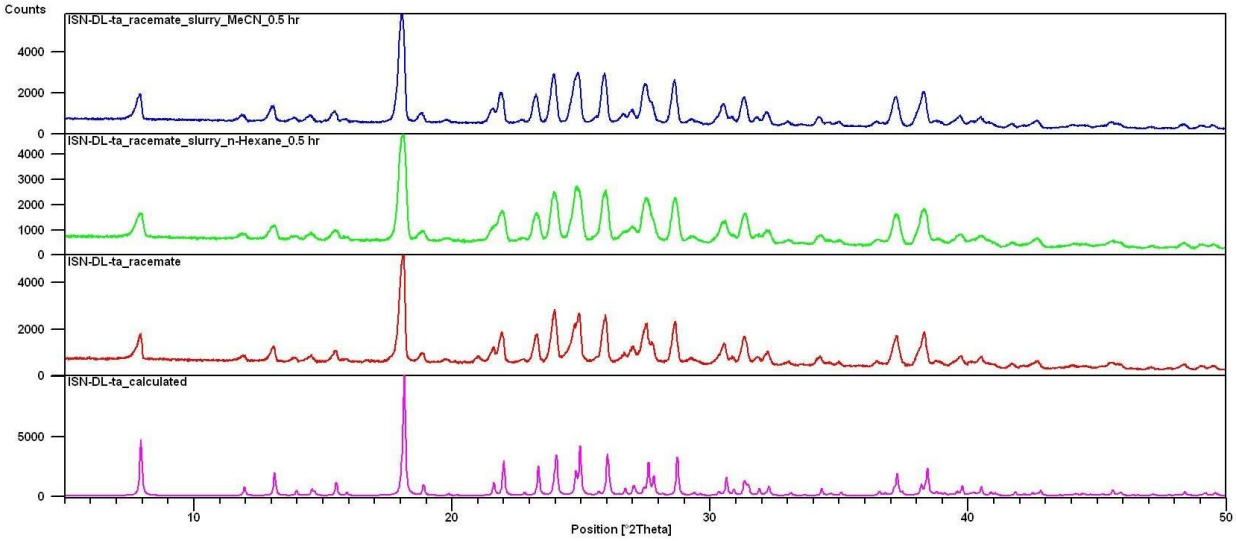
Figure S3. ATR FT-IR spectra of conglomerate (ISN•D-ta/ ISN•L-ta) powder samples obtained from NG and racemic salt (ISN•DL-ta) obtained from LAG with MeCN as liquid compared with respective starting materials ISN and DL-ta.



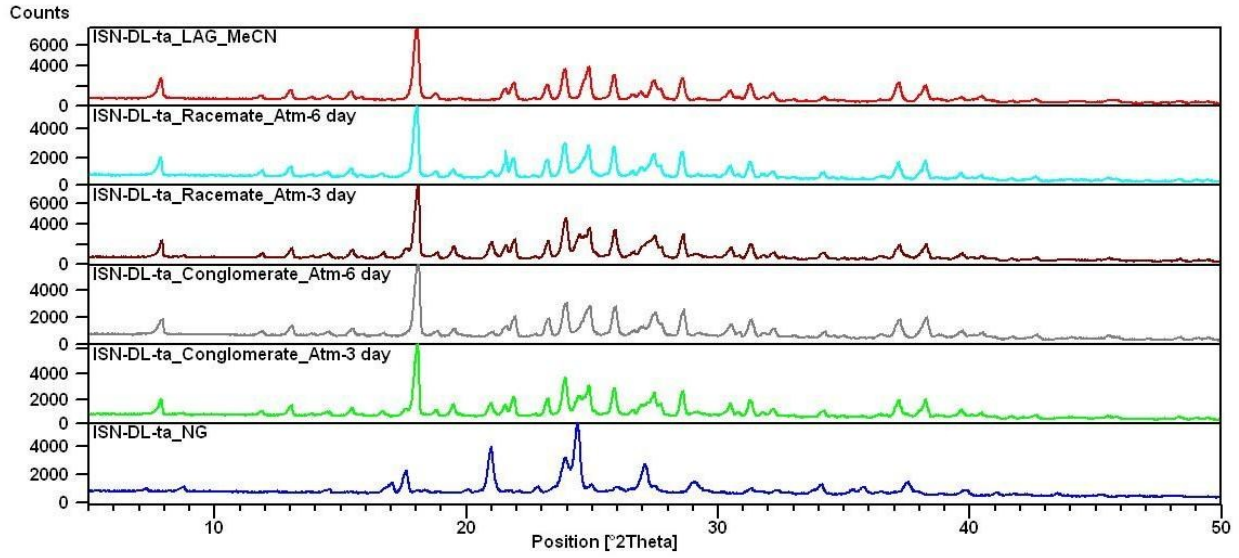
(a)


Counts
ISN-DL-ta_calculated
ISN-DL-ta_conglomerate_slurry_MeCN_16 hr
ISN-DL-ta_conglomerate_slurry_MeCN_0.5 hr
ISN-DL-ta_conglomerate
ISN-D-ta_Calculated
Position [°2Theta]

(b)


Counts
ISN-DL-ta_racemate_slurry_MeCN_0.5 hr
ISN-DL-ta_racemate_slurry_n-Hexane_0.5 hr
ISN-DL-ta_racemate
ISN-DL-ta_calculated
Position [°2Theta]

(c)



(d)

Figure S4. PXRD patterns of powder samples of respective conglomerate (ISN•D-ta/ ISN•L-ta) slurried in (a) hexane (non-polar solvent) and (b) MeCN (polar solvent); (c) racemic salt (ISN•DL-ta) slurried in hexane and MeCN for half an hour; (d) PXRD pattern of powder sample of conglomerate under atmospheric humidity for 3 days converts the sample to its racemic mixture, on the other hand racemate is stable under atmospheric humidity.

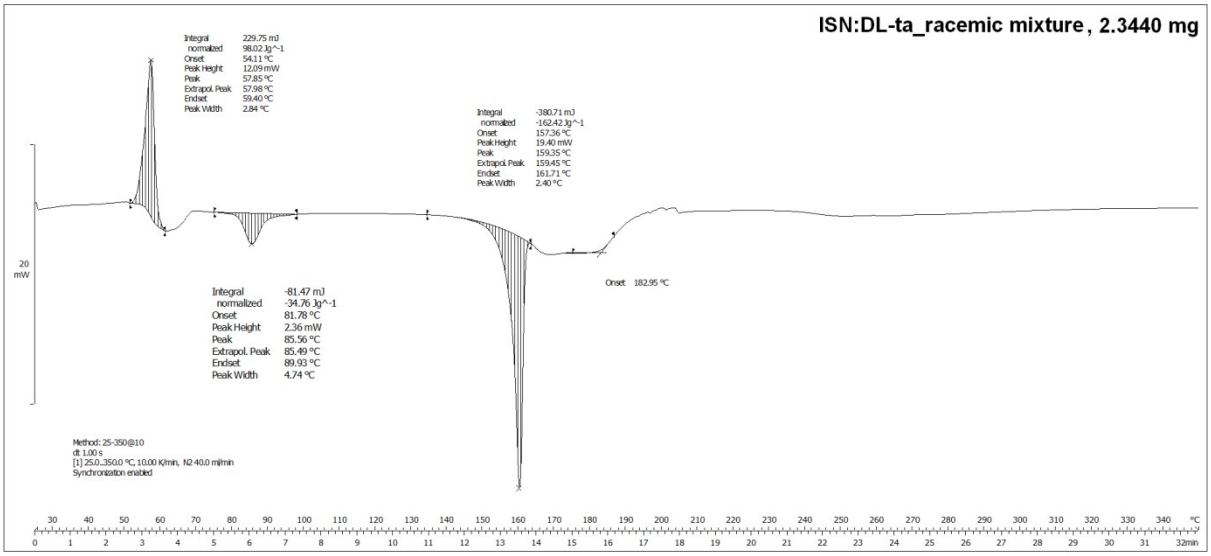
Characterisation of the powder samples using DSC thermogram:

(a) (ISN•DL-ta) Racemic salt

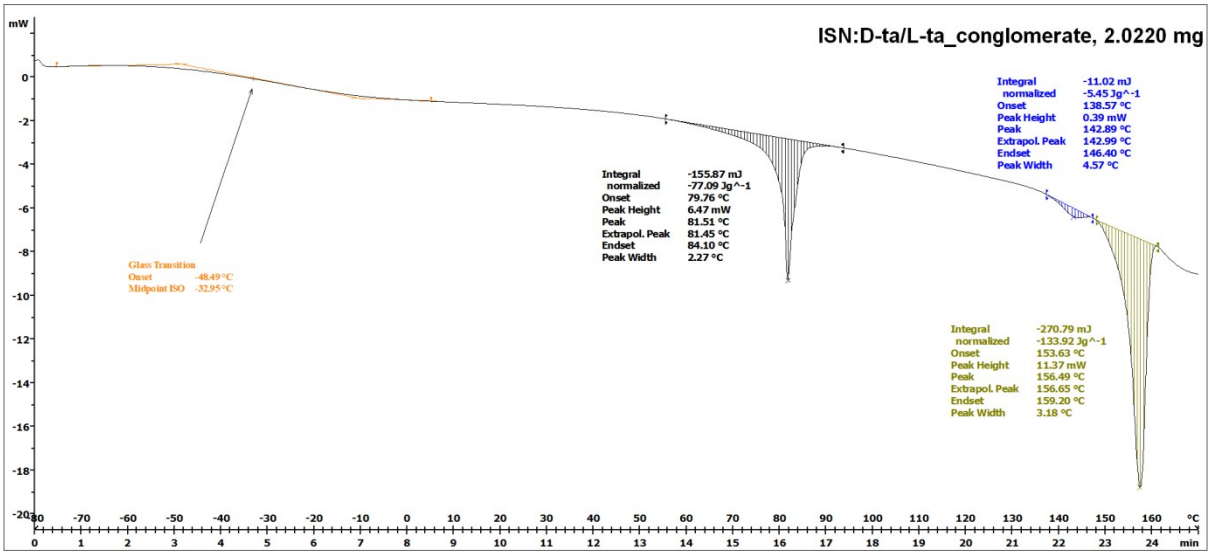
The exothermic peak observed at 57.8 °C may be due to crystallization of the salt due to formation of a glassy phase prior to salt formation (not observed in the DSC thermogram). The endothermic peak at 85 °C is due to the loss of solvent acetonitrile (boiling point 82 °C) present in the surface of the powder sample prepared using LAG. The endotherm at 159 °C is the melting point of the racemic salt (ISN•DL-ta).

(b) (ISN•D-ta/ ISN•L-ta) Conglomerate salt

Glass transition is observed at –48 °C for the conglomerate salt. The endothermic peak at 81 °C is due to the loss of solvent carbon tetrachloride (boiling point 77 °C) present in the surface of the powder sample prepared using LAG. The endotherm at 143 °C may be due to polymorphic phase transformation (not to the racemic form as the melting point do not coincide) followed by melting at 156 °C.



(a)



(b)

Figure S5. DSC endotherm of (a) racemic salt (ISN•DL-ta) and (b) conglomerate (ISN•D-ta/ ISN•L-ta) powder prepared using mechanochemical grinding.

Table S1. Crystallographic parameter of racematic salt (ISN•DL-ta) and enantiomeric (ISN•D-ta) salt.

Crystal data	ISN•DL-ta	ISN•D-ta	ISN•D-ta

Chemical formula	$C_{10}H_{13}N_3O_7$	$C_{20}H_{26}N_6O_{14}$	$C_{20}H_{26}N_6O_{14}$
M_r	287.23	574.47	574.47
Crystal system	Monoclinic	Orthorhombic	Orthorhombic
space group	$P2_1/n$	$P2_12_12_1$	$P2_12_12_1$
Temperature (K)	296	296	200
a (Å)	12.409 (2)	7.8825(5)	7.8533(2)
b (Å)	7.1753 (14)	15.1865(9)	15.1576(4)
c (Å)	14.653 (3)	19.8838(11)	19.7612(5)
β (°)	113.098 (10)	90	90
V (Å^3)	1200.1 (4)	2380.2(2)	2352.33(11)
Z	4	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
Density	1.590	1.441	1.622
μ (mm^{-1})	0.14	0.130	0.139
Diffractometer	Bruker *APEX*-II	Bruker *APEX*-II	Bruker *APEX*-II
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15957, 2930, 1893	11003, 4831, 4522	8495, 4924, 4467
$R[F^2 > 2\sigma(F^2)]$, ,	0.056	0.086	0.063
$wR(F^2)$	0.166	0.245	0.178
S	1.04	1.680	1.04
No. of reflections	2930	4831	4924
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.32	1.39, −0.82	1.87, −0.64

CCDC No.	1916551	1916550	1916549