Supplementary data

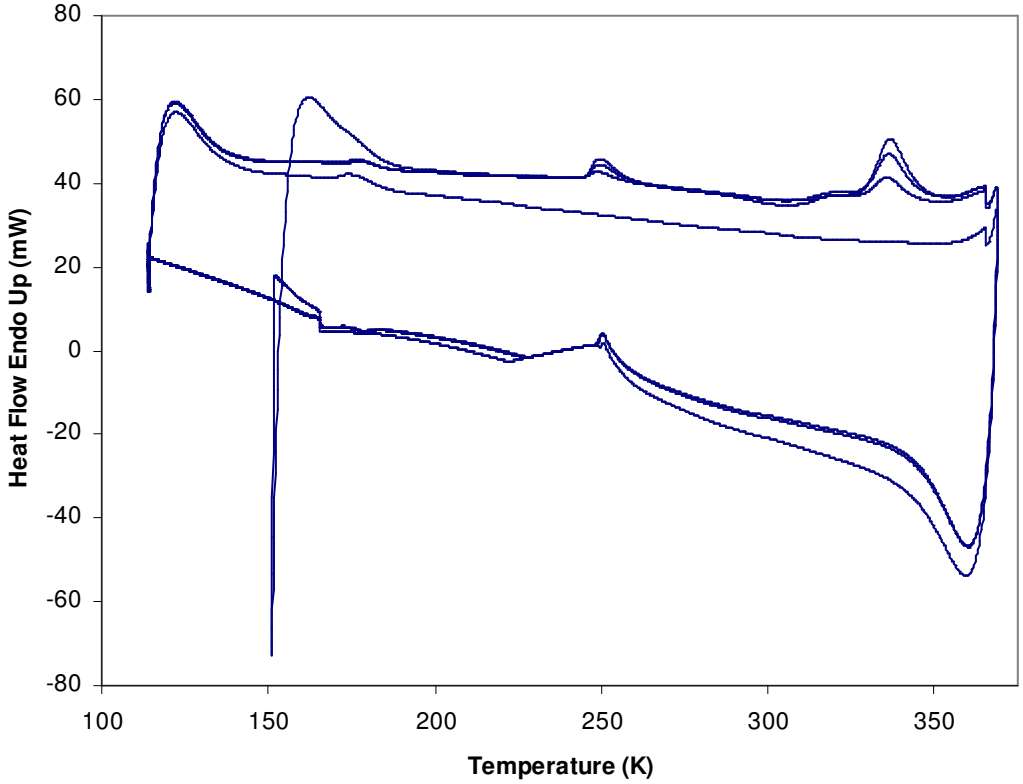


Figure S1. DSC trace of sublimed $SnMe_3OH$

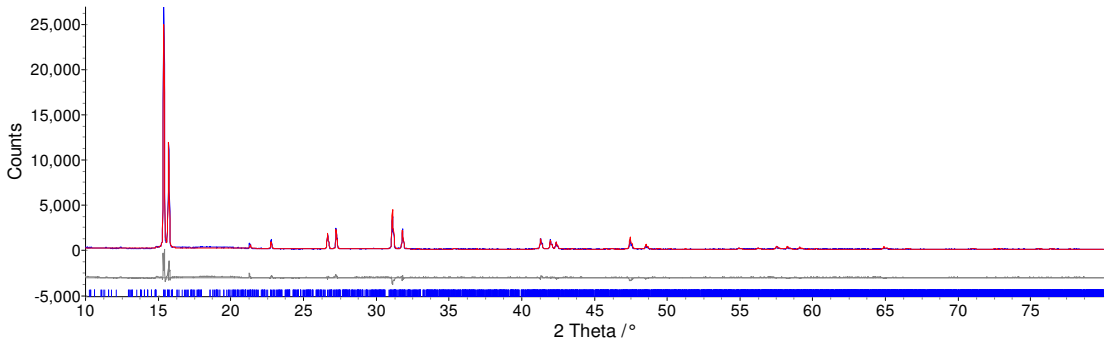


Figure S2. Rietveld fit of 'as received' trimethyltin hydroxide. The R_{wp} for this refinement was 14.8 %. The figure shows experimental data in blue, calculated pattern in red and difference plot in grey. The predicted reflections arising from this structure are shown in blue at the bottom of the plot.

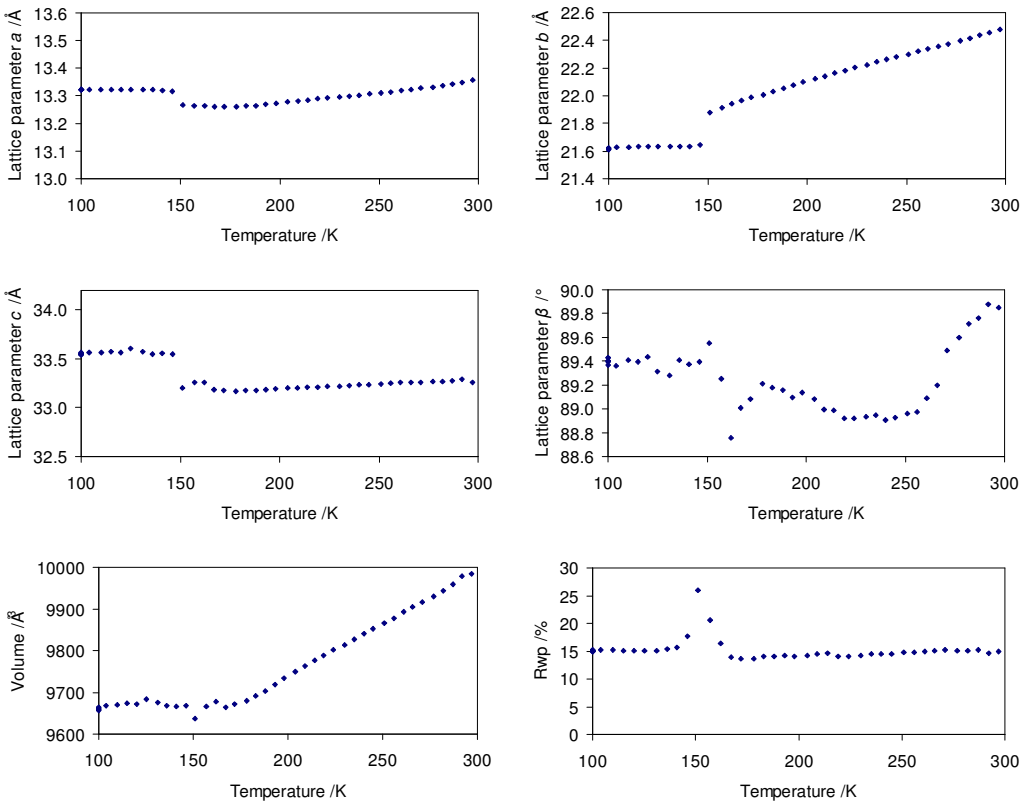


Figure S3. Refined lattice parameters, volume and R_{wp} values from each of the sequential Rietveld refinements on the data obtained while cooling an 'as received' sample of trimethyltin hydroxide from 300 to 100 K. Lattice parameters are shown on an identical % change scale for ease of comparison. A clear first order phase transition is noted at 151 K. The R_{wp} rises at this transition temperature due to the peaks being "locked in" following the transition and hence the structural model not being able to accurately fit all the data.

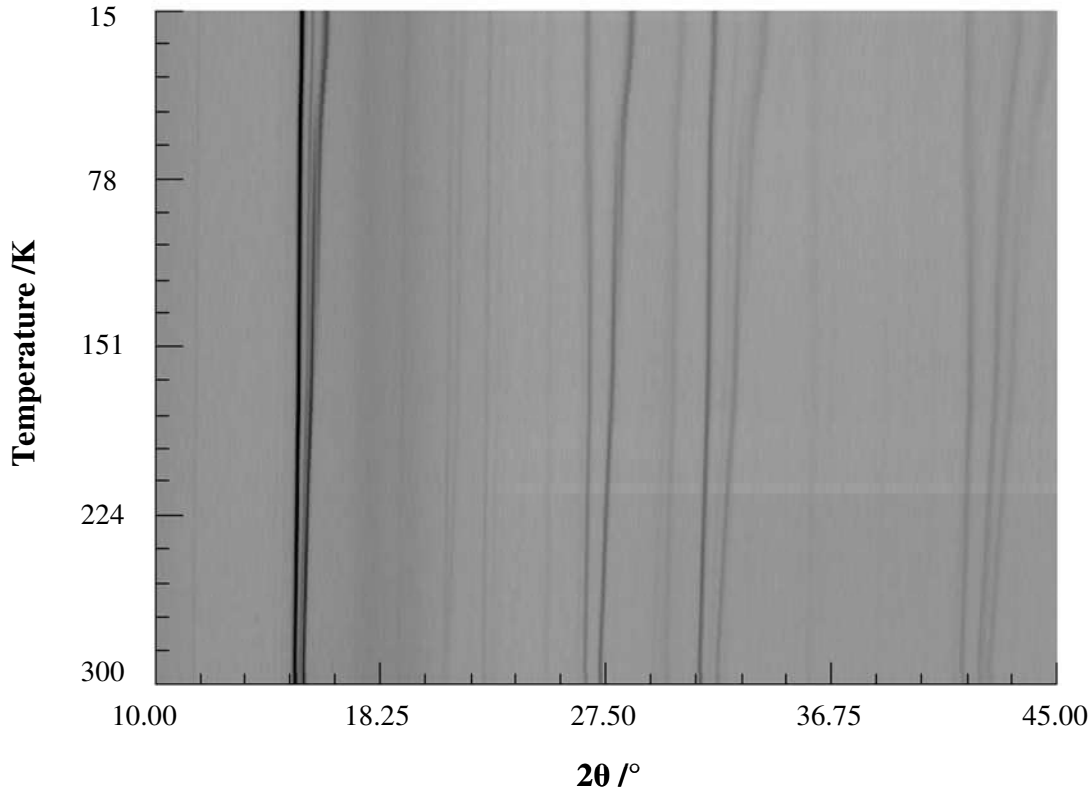


Figure S4. Two dimensional film representation of powder diffraction data recorded while cooling a sample of the vacuum-sublimed trimethyltin hydroxide from 300 to 15 K in ~5 K steps. The sharp first order phase transition beginning at *ca.* 141 K corresponding to the change from structure **1** to structure **2** is clearly evident.

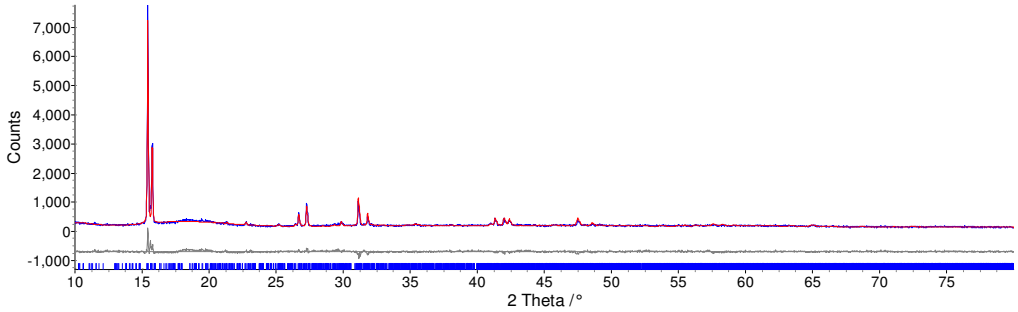


Figure S5. Rietveld fit of sublimed trimethyltin hydroxide. The R_{wp} for this refinement was 10.02 %. The figure shows experimental data in blue, calculated pattern in red and difference plot in grey. The predicted reflections arising from this structure are shown in blue at the bottom of the plot.

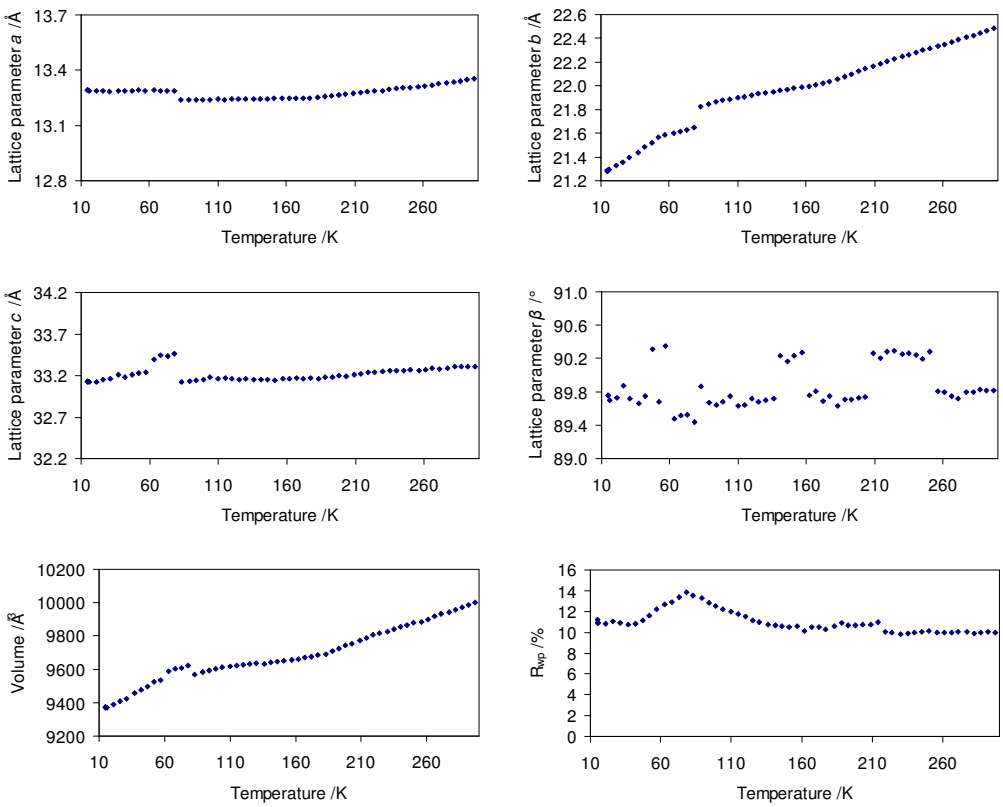
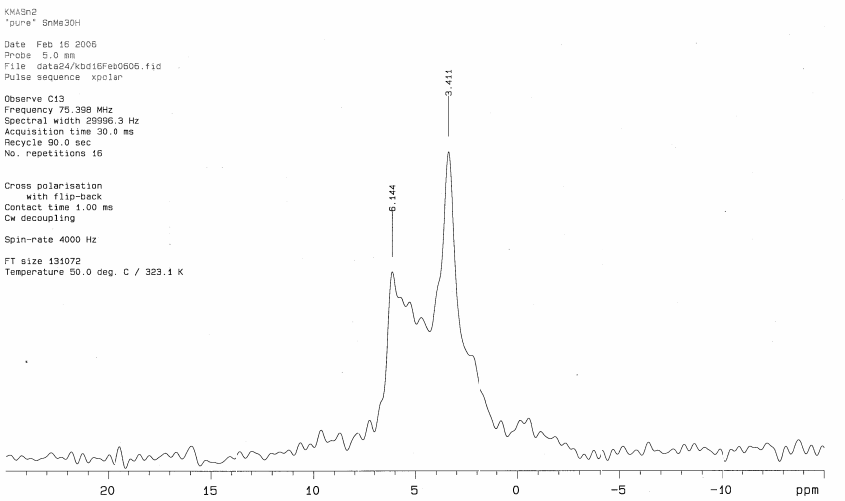
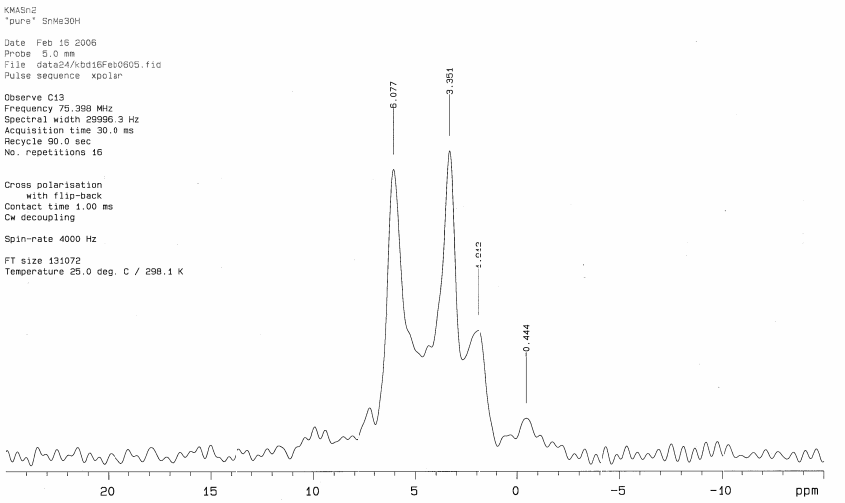


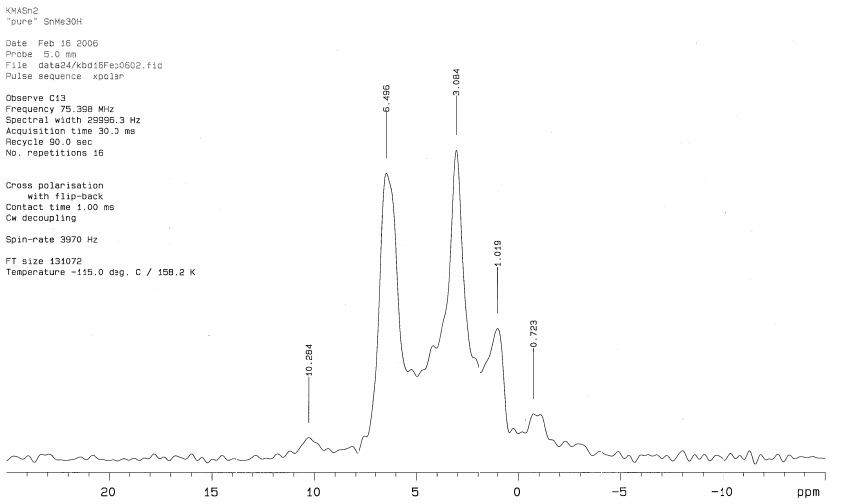
Figure S6. Refined lattice parameters, volume and R_{wp} values from sequential Rietveld refinements on the data obtained while cooling a sublimed sample of trimethyltin hydroxide from 300 to 12 K. Lattice parameters are shown on an identical A clear first order phase transition is noted at 78 K. More subtle phase transitions are seen at 245 and 177 K in these data. The R_{wp} rises at the 78 K transition due to the peaks being "locked in" following the transition and hence the structural model does not accurately fit all the data.


KMASn2
"pure" SnMe3OH
Date Feb 16 2006
Probe 5.0 mm
File data24/kbd16Feb0605.fid
Pulse sequence xpolar
Observe C13
Frequency 75.398 MHz
Spectral width 29996.3 Hz
Acquisition time 30.0 ms
Recycle 90.0 sec
No. repetitions 16
Cross polarisation
with flip-back
Contact time 1.00 ms
Cw decoupling
Spin-rate 4000 Hz
FT size 131072
Temperature 50.0 deg. C / 323.1 K
6.144
3.411
20
15
10
5
0
-5
-10
ppm

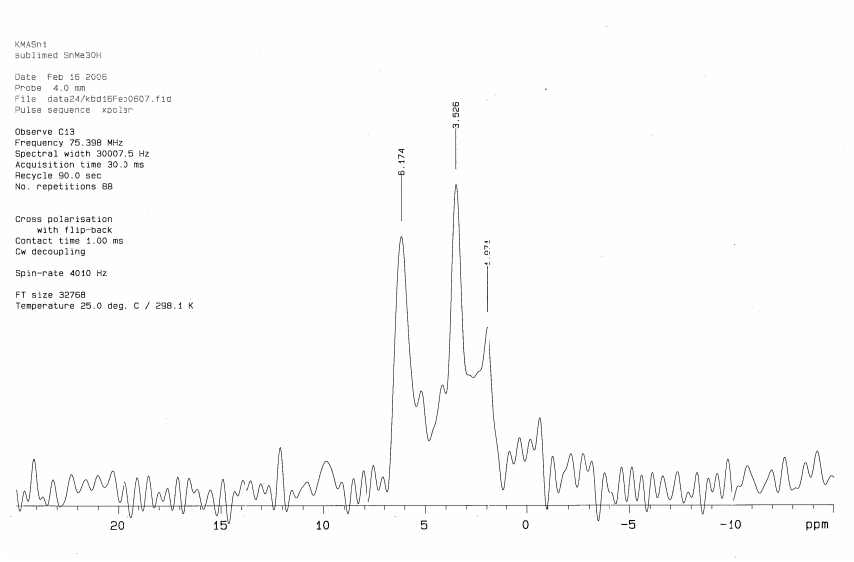
(a)


KMASn2
"pure" SnMe3OH
Date Feb 16 2006
Probe 5.0 mm
File data24/kbd16Feb0605.fid
Pulse sequence xpolar
Observe C13
Frequency 75.398 MHz
Spectral width 29996.3 Hz
Acquisition time 30.0 ms
Recycle 90.0 sec
No. repetitions 16
Cross polarisation
with flip-back
Contact time 1.00 ms
Cw decoupling
Spin-rate 4000 Hz
FT size 131072
Temperature 25.0 deg. C / 298.1 K
6.077
3.351
1.212
0.444
20
15
10
5
0
-5
-10
ppm

(b)


KMASn2
"pure" SnMe3OH
Date Feb 16 2006
Probe 5.0 mm
File data24/kbd15Feb0602.fid
Pulse sequence xpolar
Observe C13
Frequency 75.398 MHz
Spectral width 29996.3 Hz
Acquisition time 30.0 ms
Recycle 90.0 sec
No. repetitions 16
Cross polarisation
with flip-back
Contact time 1.00 ms
Cw decoupling
Spin-rate 3970 Hz
FT size 131072
Temperature -115.0 deg. C / 158.2 K
10.284
6.496
3.084
1.019
0.723
20
15
10
5
0
-5
-10
ppm

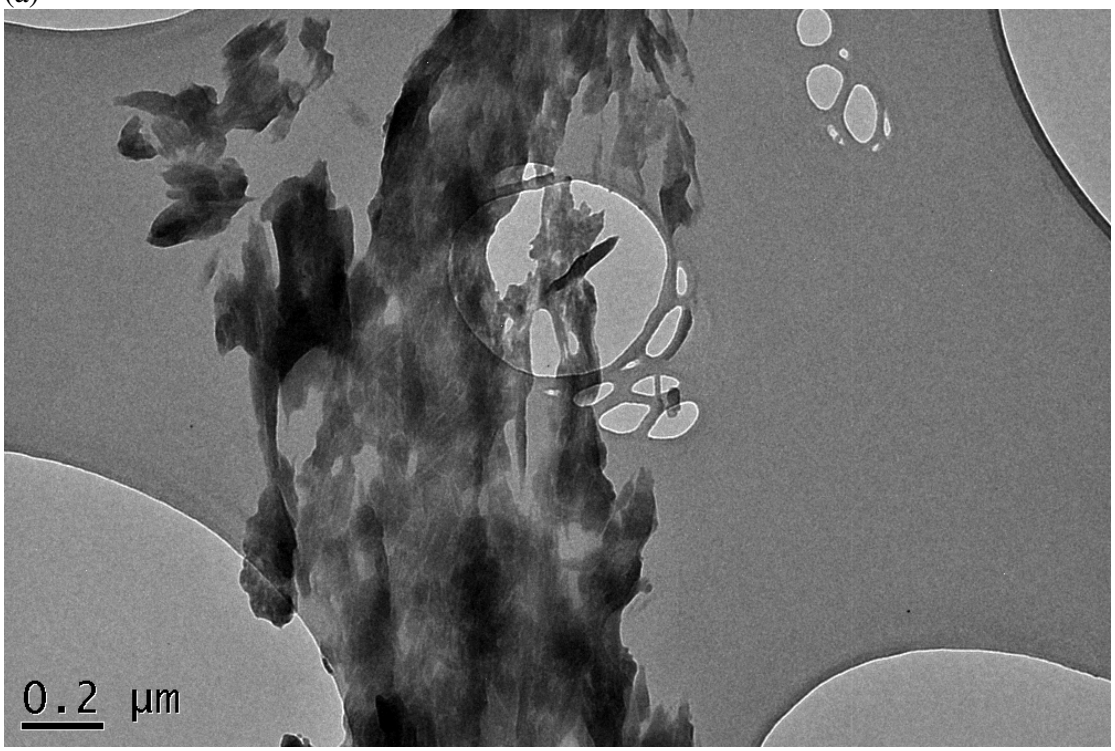
(c)



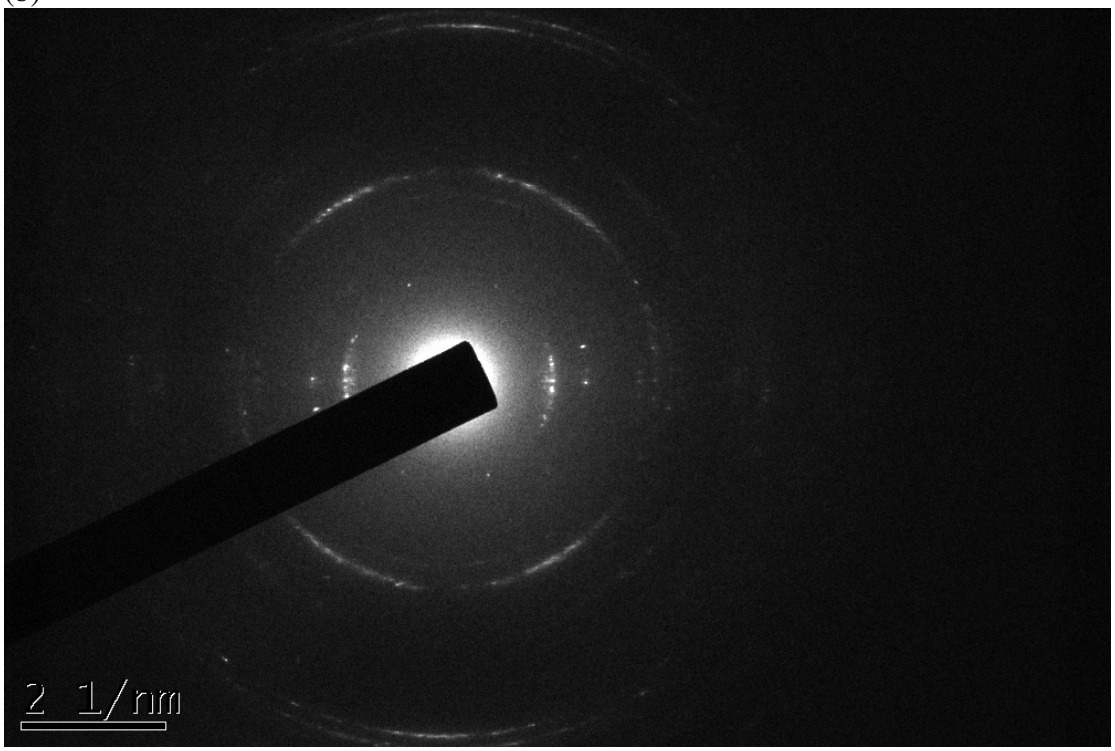
(d)

Figure S7 CP-MAS ^{13}C NMR spectra for 'as received' Me_3SnOH at (a) 50 ^{o}C, (b) 25 ^{o}C, (c) –115 ^{o}C (d) sublimed Me_3SnOH, 25 ^{o}C.

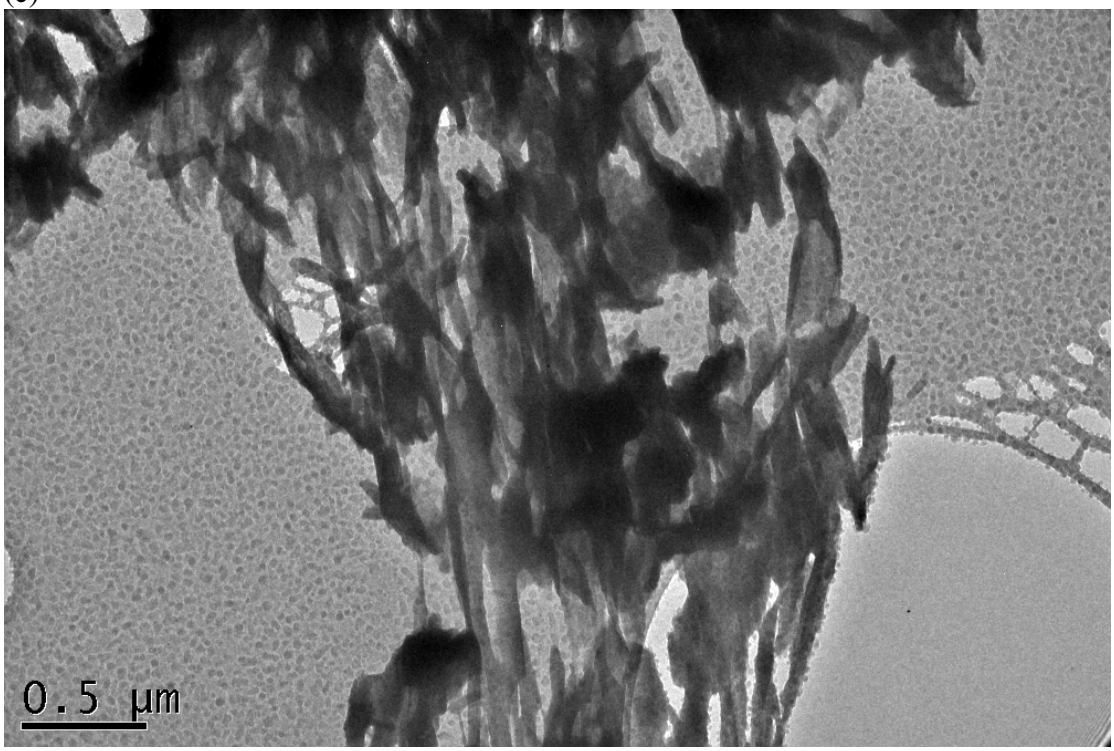
(a)



(b)



(c)



(d)

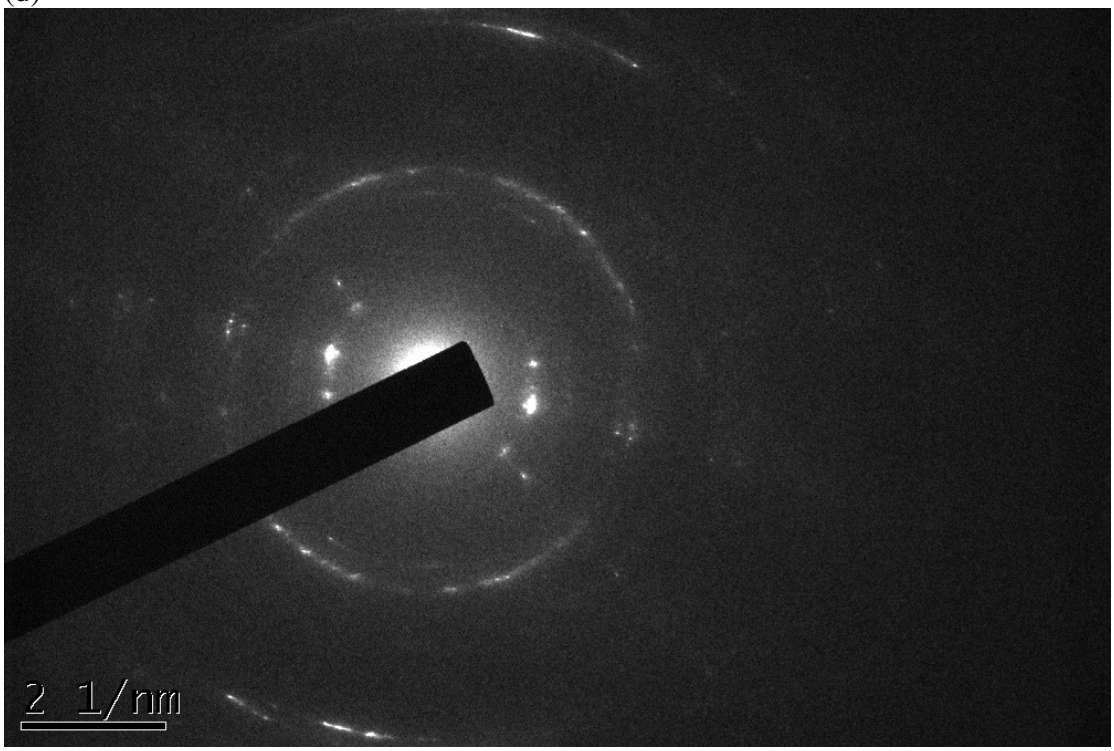


Figure S8 (a) and (b) TEM image and electron diffraction pattern of 'as received' $SnMe_3OH$ at -70 ^oC (c) and (d) TEM image and electron diffraction pattern of the same sample at -150 ^oC (granular features on the holey carbon grid are ice crystallites).