

10.1071/CH09606

© CSIRO 2010

Australian Journal of Chemistry 2010, 63(3), 458–462

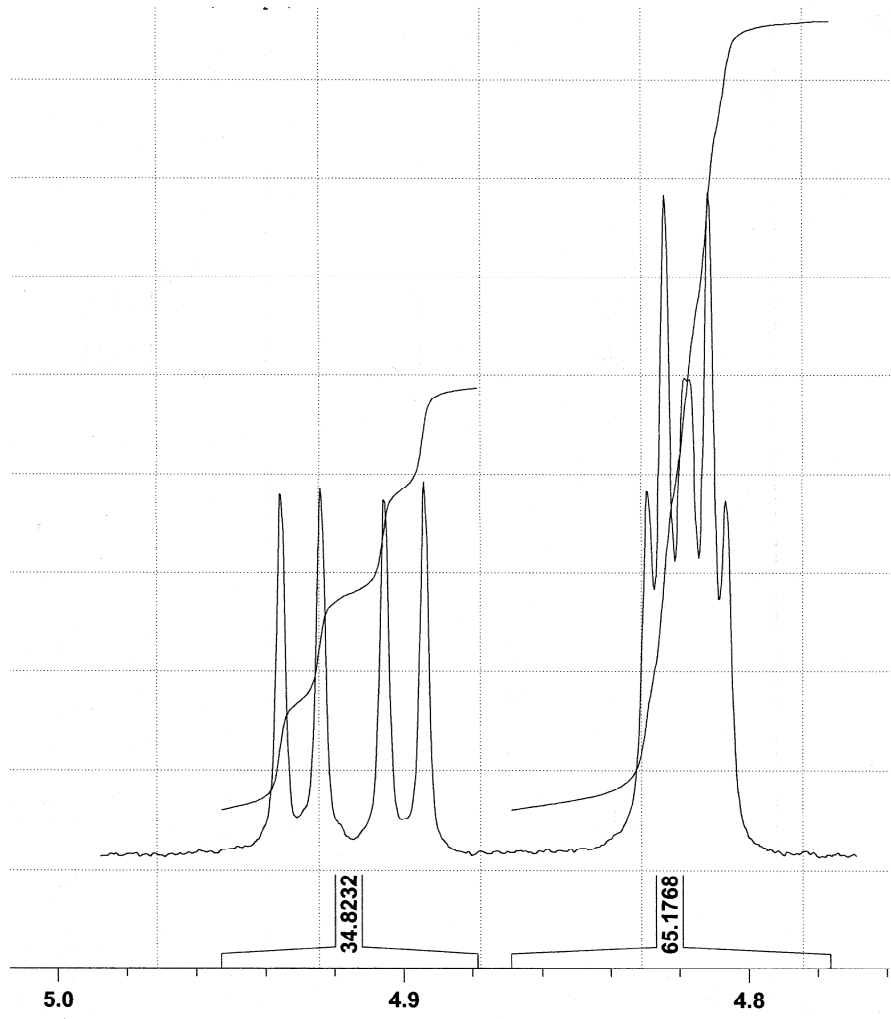
ACCESSORY PUBLICATION

## **On the Solid-State Isomerisation of Terpinolene Tetrabromide**

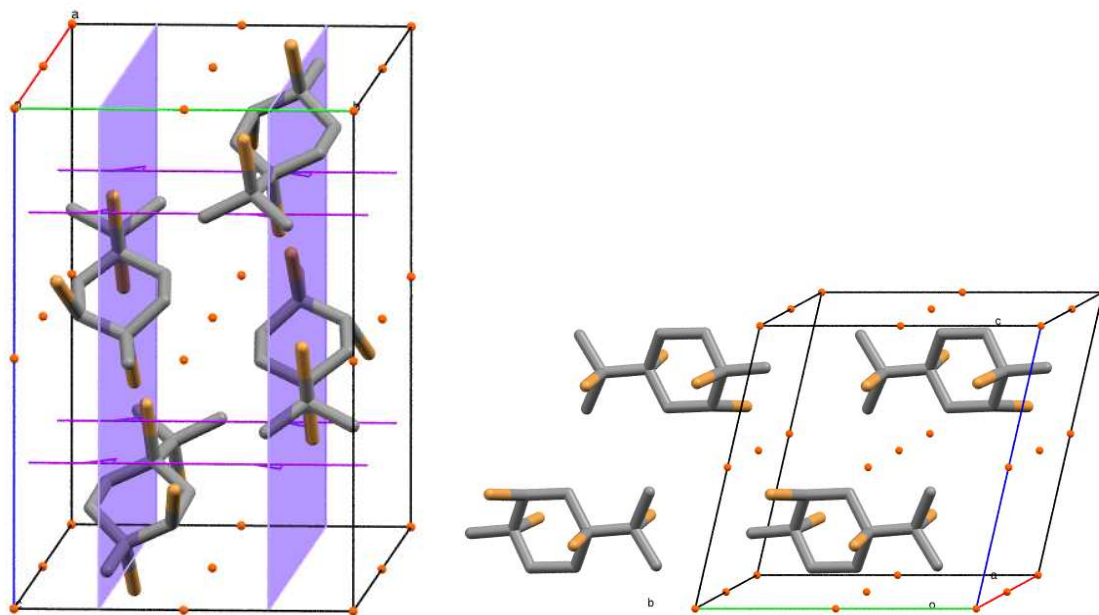
*Paul V. Bernhardt,<sup>A,B</sup> Raymond M. Carman,<sup>A</sup> and Tri T. Le<sup>A</sup>*

<sup>A</sup> School of Chemistry and Molecular Biosciences, University of Queensland, Brisbane QLD 4072, Australia.

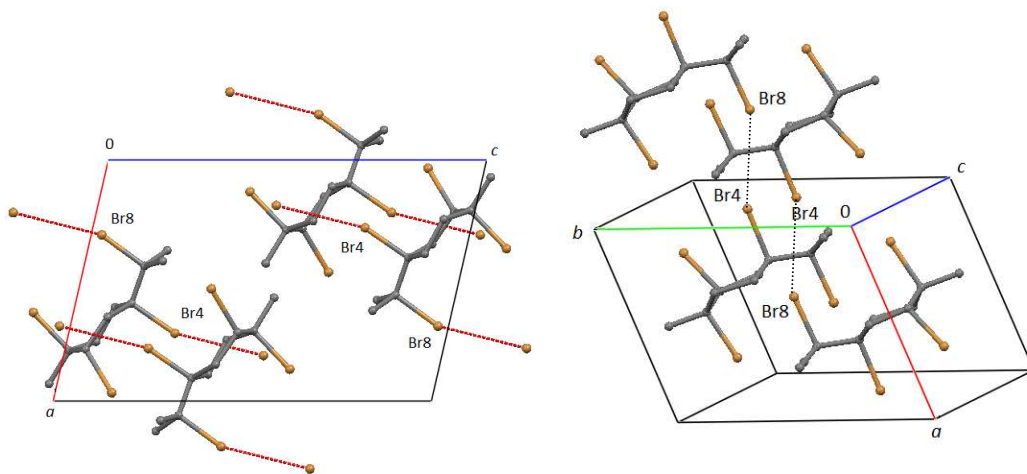
<sup>B</sup> Corresponding author. Email: [P.Bernhardt@uq.edu.au](mailto:P.Bernhardt@uq.edu.au)



**Figure S1.**  $^1\text{H}$  NMR spectrum (500 MHz,  $\text{CDCl}_3$ ) of the methine H2 proton region showing the relative proportions of compound **A** (65%,  $\delta 4.81$  (dt)) and compound **B** (35%,  $\delta 4.91$  (dd)).

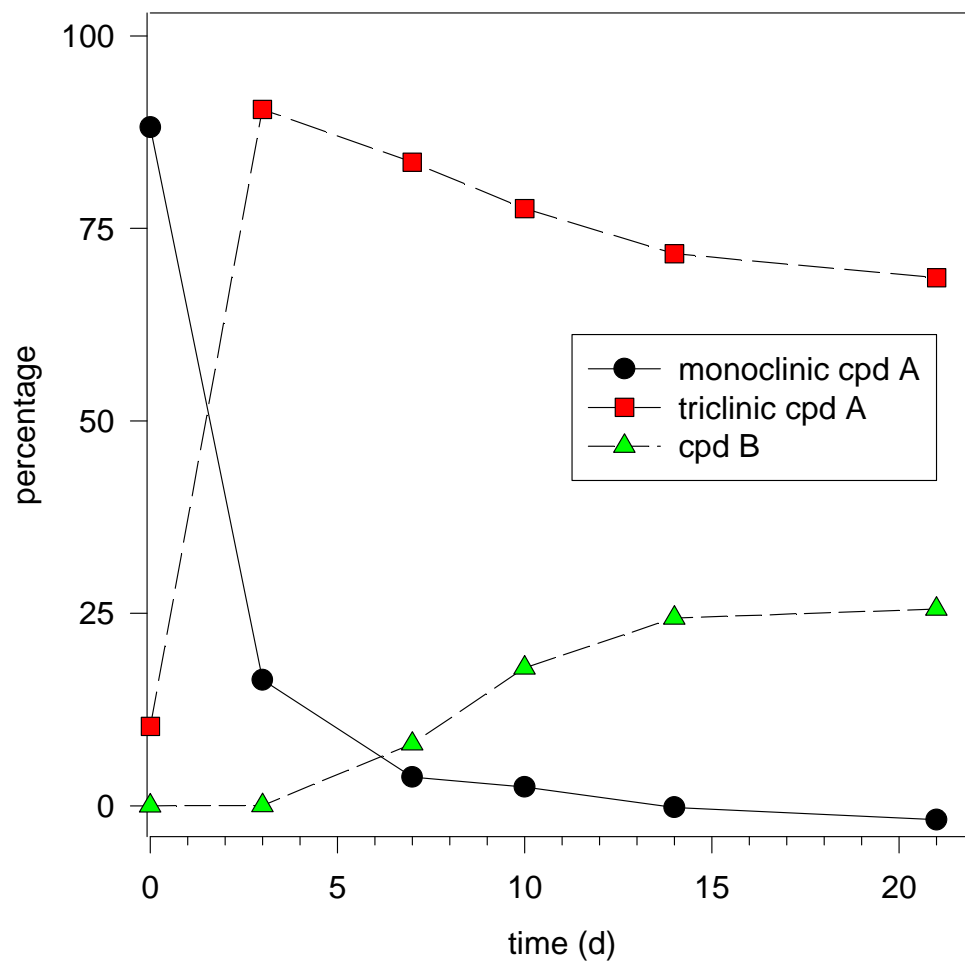


**Figure S2a.** Packing diagrams for monoclinic compound **A** (left) and triclinic compound **A** (right). The dots represent centres of inversion, the axes are 2-fold screw axes and the planes are *n* glide planes. The two lattices are not inter-convertible.



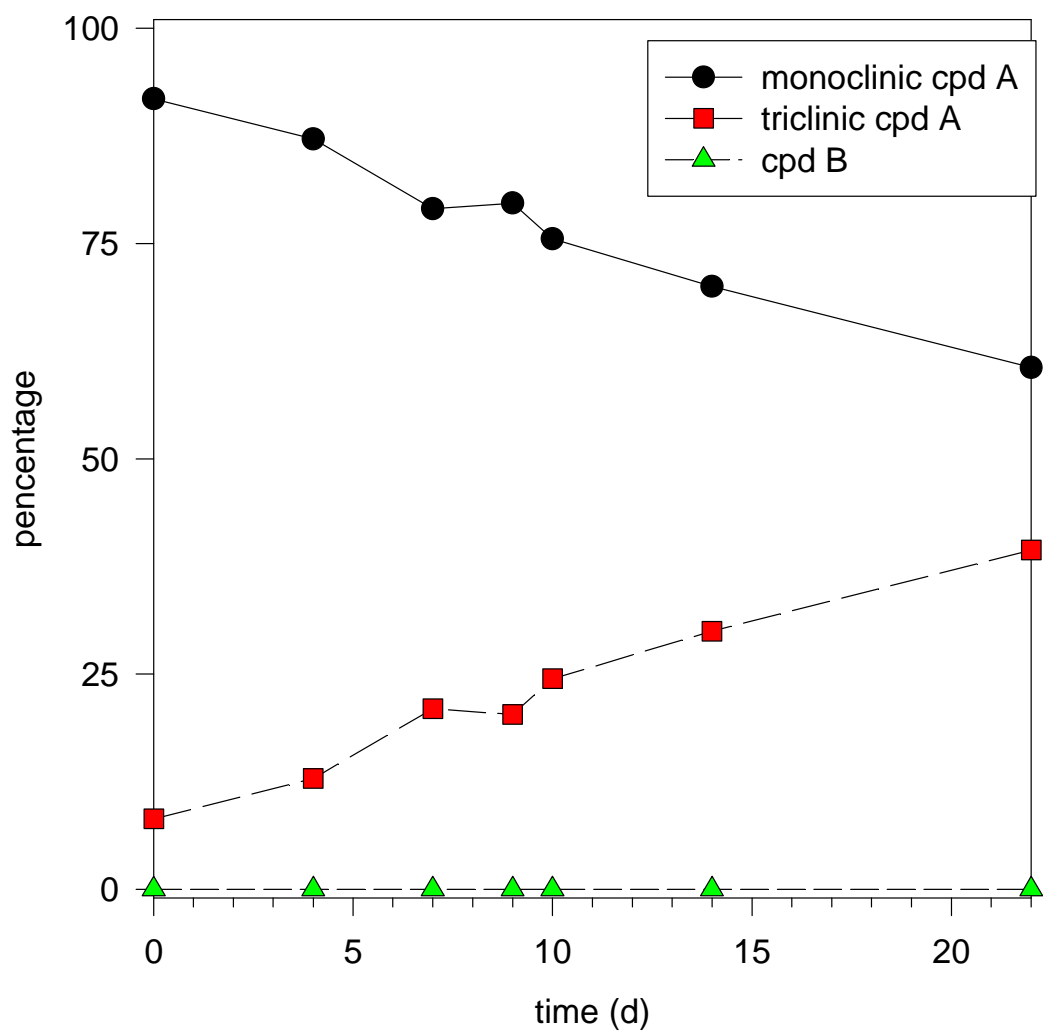
**Figure S2b.** Comparison of the Br...Br intermolecular contacts in monoclinic (left) and triclinic (right) structures of compound **A**. The close Br4...Br8 distances (3.600 Å, 0.100 Å less than the sum of the van der Waals radii) are apparent in the monoclinic structure. These Br...Br distances are significantly longer in the triclinic form (Br4...Br8 3.679 Å).

### 37 degrees

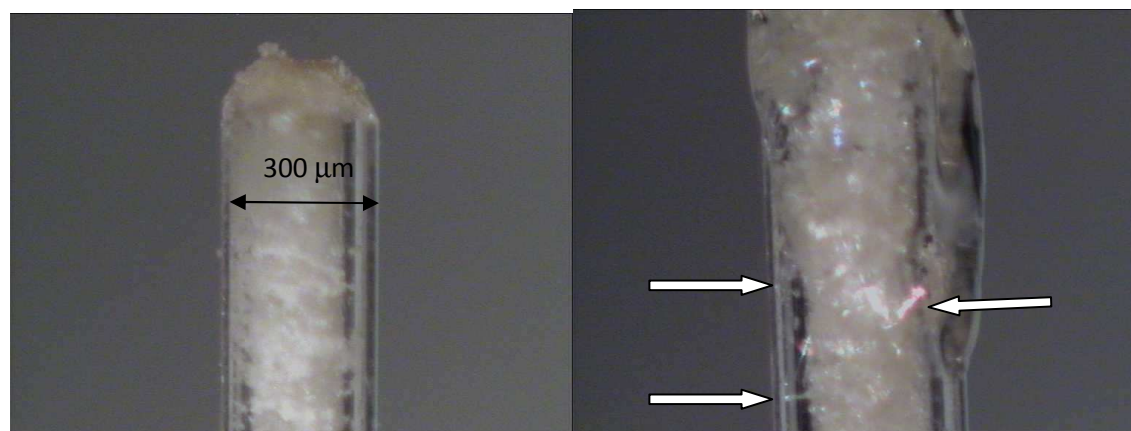


**Figure S3.** Speciation of the solid state reaction at 37°C as a function of time as determined from powder XRD.

## 25 degrees

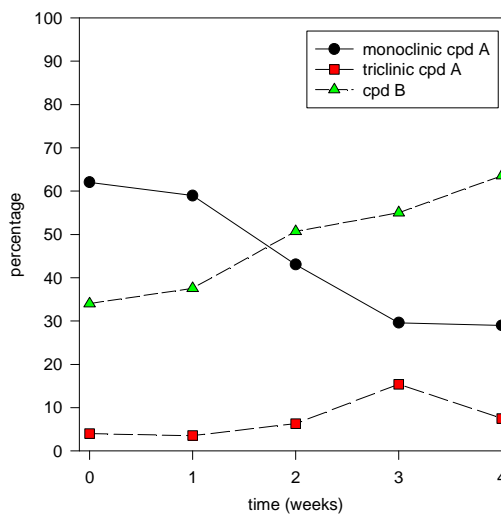
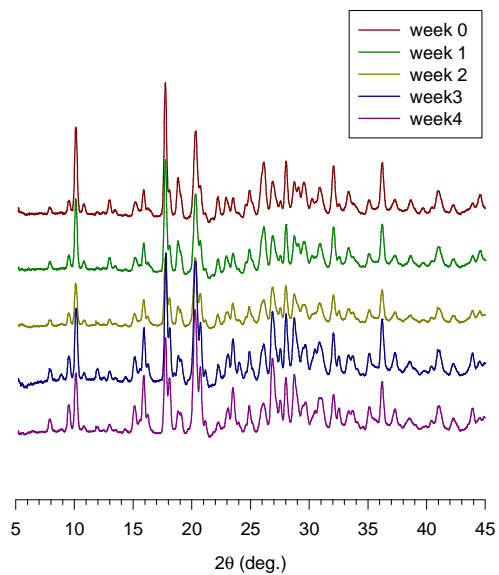


**Figure S4.** Speciation of the solid state reaction at 25°C as a function of time as determined from powder XRD.



**Figure S5.** Typical images of (left) the starting material ground into a fine powder (monoclinic compound **A**) and (right) the final product (compound **B**) both mounted in glass capillaries. Note the larger crystal size and greater luster of the sample on the right with some crystal needles highlighted by arrows.

Powder XRD					NMR Results
week	Monoclinic Cpd A	Triclinic Cpd A	Cpd B	Ratio A/B	
0	62	4	34	66/34	66/34
1	59	3	38	62/38	60/40
2	43	6	51	49/51	52/48
3	30	15	55	45/55	47/53
4	29	7	64	36/64	40/60



**Figure S6.** Comparison of powder XRD and NMR results for solid state reaction of an initially 2:1 mixture of compounds A and B at 1, 2, 3 and 4 weeks at 37 degrees.