

Two conformational polymorphs of a bioactive pyrazolo[3,4-d]pyrimidine

Sang Loon Tan ¹, Yee Seng Tan ¹, Jia Hui Ng ², Anton V. Dolzhenko ^{2,3}
and Edward R. T. Tiekink ^{1*}

¹ Research Centre for Crystalline Materials, School of Medical and Life Sciences, Sunway University, Bandar Sunway 47500, Selangor Darul Ehsan, Malaysia alant@sunway.edu.my (S.L.T.); yeesengt@sunway.edu.my (Y.S.T.)

² School of Pharmacy, Monash University Malaysia, Jalan Lagoon Selatan, Bandar Sunway, Selangor Darul Ehsan 47500, Malaysia
anton.dolzhenko@monash.edu (A.V.D.)

³ Curtin Medical School, Curtin Health Innovation Research Institute, Faculty of Health Sciences, Curtin University, GPO Box U1987 Perth, Western Australia 6845, Australia

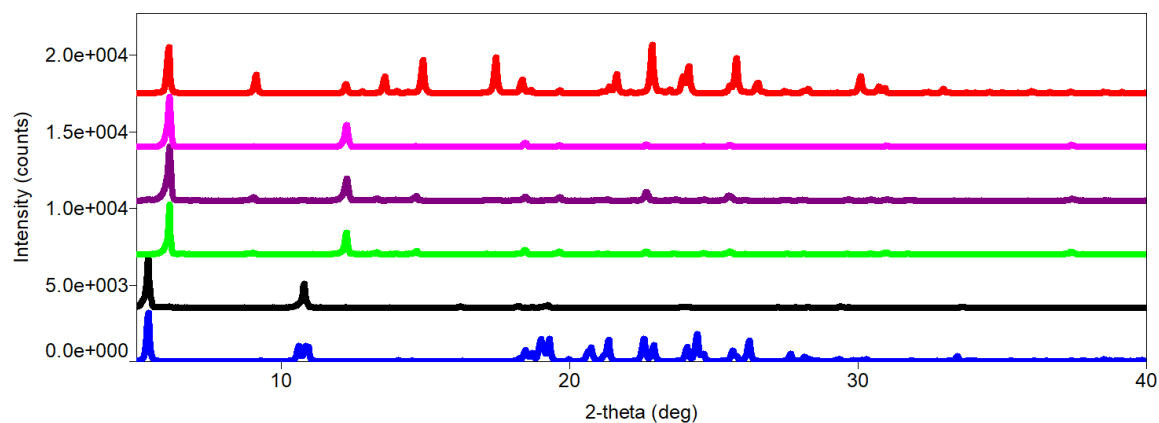
* Correspondence: edwardt@sunway.edu.my (E.R.T.T.); Tel.: +60-3-7491-7181

*** SUPPLEMENTARY MATERIALS ***

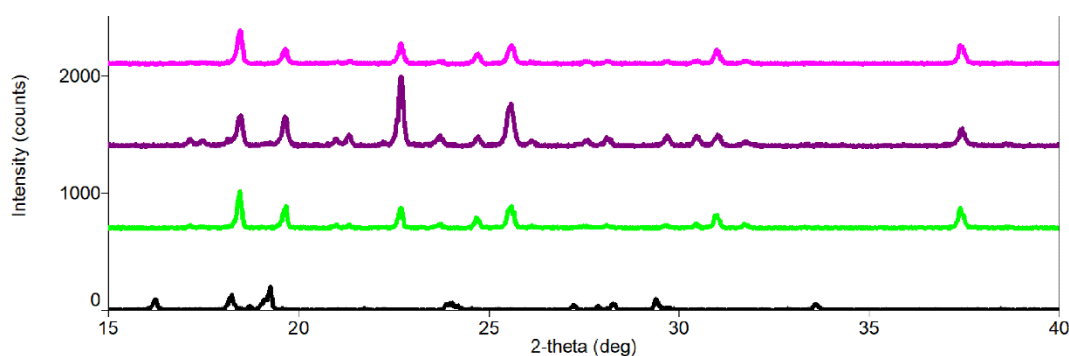
Figure S1. Powder X-ray diffraction (PXRD) patterns for the α - and β -polymorphs of **1**.

Figure S2. The DSC traces for the (a) α - and (b) β -polymorphs of **1**.

Table S1. Unit-cell data obtained from variable-temperature measurements on a single crystal of the β -form of **1**.

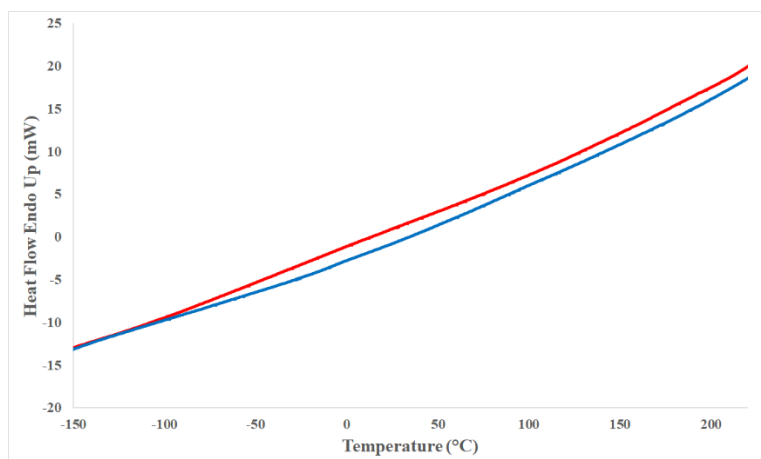


(a)

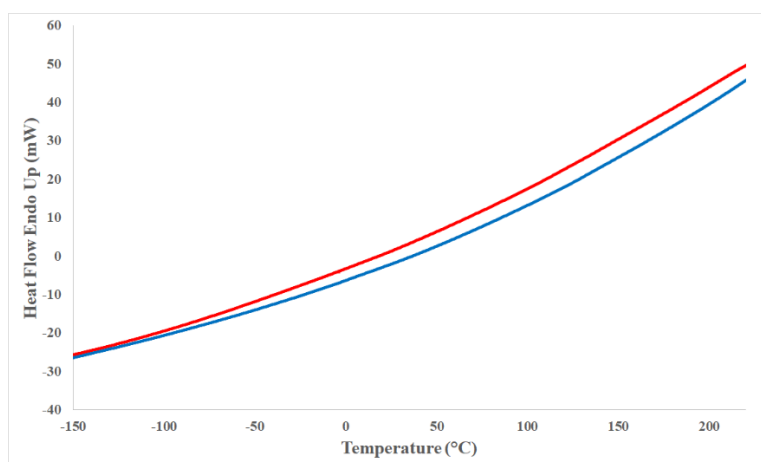


(b)

Figure S1. Powder X-ray diffraction (PXRD) patterns for the α - and β -polymorphs of **1**. (a) Simulated patterns: α -polymorph (blue trace) and β -polymorph (red). Experimental patterns: α -polymorph (black trace (from MeOH)) and β -polymorphs (green (from EtOH), purple (nPrOH) and pink (iPrOH)). (b) Experimental patterns: α -polymorph (black trace (from MeOH)) and β -polymorphs (green (from EtOH), purple (nPrOH) and pink (iPrOH)) plotted in the region 15 to 40° to negate the influence of the intense, low-angle peaks. The PXRD patterns were measured on a Rigaku SmartLab Powder X-ray diffractometer (Rigaku, Tokyo, Japan) using CuK α (λ = 1.5418 Å) radiation in the 2 θ range of 5 to 40° (step size = 0.01°).



(a)



(b)

Figure S2. The DSC traces for the (a) α - and (b) β -polymorphs of **1**. Each polymorph was heated to 220 °C from -150 °C followed by cooling. The red trace is the heating cycle and the blue trace, the cooling cycle.

Table S1. Unit-cell data obtained from variable-temperature measurements on a single crystal of the β -form of **1**. Experiments were performed on a Rigaku/Oxford Diffraction XtaLAB Synergy diffractometer (Dualflex, AtlasS2) fitted with CuK α radiation ($\lambda = 1.54184 \text{ \AA}$) fitted with an Oxford Cryosystem Cryostream 800.

Cooling cycle:

298 K

_cell_length_a	14.4148(4)
_cell_length_b	13.2814(4)
_cell_length_c	8.22721(19)
_cell_angle_alpha	90.0
_cell_angle_beta	91.535(2)
_cell_angle_gamma	90.0
_cell_volume	1574.52(8)
_cell_formula_units_Z	4.00
_cell_measurement_temperature	297.99(10)

200 K

_cell_length_a	14.42119(13)
_cell_length_b	13.14149(15)
_cell_length_c	8.17706(7)
_cell_angle_alpha	90.0
_cell_angle_beta	91.9418(7)
_cell_angle_gamma	90.0
_cell_volume	1548.79(3)
_cell_formula_units_Z	4.00
_cell_measurement_temperature	200.00(10)

100 K

_cell_length_a	14.43918(13)
_cell_length_b	13.01799(13)
_cell_length_c	8.12972(6)
_cell_angle_alpha	90.0
_cell_angle_beta	92.3447(8)
_cell_angle_gamma	90.0
_cell_volume	1526.86(2)
_cell_formula_units_Z	4.00
_cell_measurement_temperature	99.97(13)

Heating cycle:

200 K

_cell_length_a	14.42154(14)
_cell_length_b	13.13925(16)
_cell_length_c	8.17439(7)
_cell_angle_alpha	90.0
_cell_angle_beta	91.9377(8)
_cell_angle_gamma	90.0
_cell_volume	1548.07(3)
_cell_formula_units_Z	4.00
_cell_measurement_temperature	200.00(10)

298 K

_cell_length_a	14.4093(2)
_cell_length_b	13.2816(2)
_cell_length_c	8.22455(10)
_cell_angle_alpha	90.0
_cell_angle_beta	91.5168(11)
_cell_angle_gamma	90.0
_cell_volume	1573.45(4)
_cell_formula_units_Z	4.00
_cell_measurement_temperature	297.99(11)

350 K

_cell_length_a	14.4034(2)
_cell_length_b	13.3628(2)
_cell_length_c	8.25698(10)
_cell_angle_alpha	90.0
_cell_angle_beta	91.2756(13)
_cell_angle_gamma	90.0
_cell_volume	1588.82(4)
_cell_formula_units_Z	4.00
_cell_measurement_temperature	350.00(13)

373 K

_cell_length_a	14.3995(2)
_cell_length_b	13.4020(3)
_cell_length_c	8.27388(11)
_cell_angle_alpha	90.0
_cell_angle_beta	91.1560(13)
_cell_angle_gamma	90.0
_cell_volume	1596.39(5)
_cell_formula_units_Z	4.00
_cell_measurement_temperature	372.99(16)