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Supporting information for article:

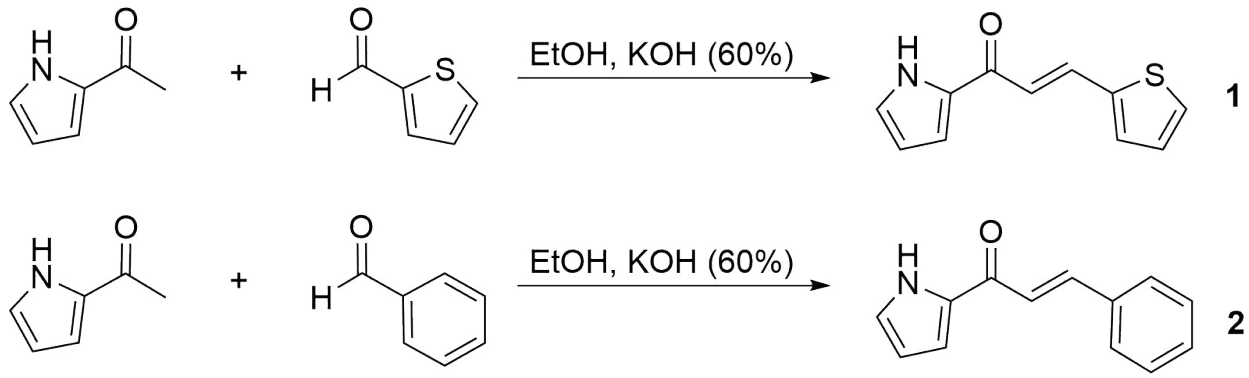
The solubility and stability of heterocyclic chalcones compared with *trans*-chalcone

Stephen G. Sweeting, Charlie L. Hall, Jason Potticary, Natalie E. Pridmore, Stephen D. Warren, Matthew E. Cremeens, Gemma D. D'Ambruoso, Masaomi Matsumoto and Simon R. Hall

Contents of supporting information

8- Single crystal x-ray diffraction data collected for (*E*)-1-(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)-prop-2-en-1-one.

1- Detailed synthesis of both heterocyclic chalcones



2-acetylpyrrole and 2-thiophenaldehyde were commercially available from TCI and used as obtained. Benzaldehyde was used as obtained from Aldrich. NMR was obtained on a Bruker Advance III HD 400 MHz.

(*E*)-1-(1*H*-pyrrol-2-yl)-3-(2-thienyl)prop-2-en-1-one

0.327 g (3.0 mmol) of 2-acetylpyrrole was added to a centrifuge tube containing 1.5 mL of 95% ethanol. 0.28 mL (0.34 g, 3.0 mmol) of 2-thiophenaldehyde was added to the centrifuge tube. After thorough mixing 0.20 mL of 60% wt/wt KOH (aq) was added to the centrifuge tube. The reaction was allowed to proceed for ~30 minutes with periodic thorough mixing. ~6 mL of ice water was added to the centrifuge tube and mixed followed be the addition of ~6 mL of CH_2Cl_2. The tube was thoroughly shaken, and the aqueous layer removed. The organic layer was extracted twice more with ice water. The tube was left uncapped and the CH_2Cl_2 slowly evaporated over several days to give a solid that was recrystallized out of a minimal amount of hot methanol. 0.140g, 23% yield. ^{1}H NMR $CDCl_3$ (**Figure S1**) TMS ref. δ 6.35 (m, 1H), 7.08 (m, 2H), 7.14 (d/m, 2H, J=16 Hz), 7.335 (d, 1H, J=3.5 Hz), 7.395 (d, 1H, J=5 Hz), 7.95 (d, 1H, J=16 Hz), 10.18 (s br, 1H). ^{13}C NMR, $CDCl_3$ (**Figure S2 and S3**) ref. δ 111.08, 116.55, 121.12, 125.72, 128.36, 128.42, 131.55, 133.20, 134.85, 140.62, 178.62.

(*E*)-3-phenyl-1-(1*H*-pyrrol-2-yl)prop-2-en-1-one

0.327 g (3.0 mmol) of 2-acetylpyrrole was added to a centrifuge tube containing 1.5 mL of 95% ethanol. 0.31 mL (0.34 g, 3.0 mmol) of benzaldehyde was added to the centrifuge tube. After thorough mixing 0.20 mL of 60% wt/wt KOH (aq) was added to the centrifuge tube. The reaction was allowed to proceed for ~30 minutes with periodic thorough mixing. ~6 mL of ice water was added to the centrifuge tube and mixed followed be the addition of ~6 mL of CH_2Cl_2. The tube was thoroughly shaken, and the aqueous layer removed. The organic layer was extracted twice more with ice water. The tube was left uncapped and the CH_2Cl_2 slowly evaporated over several days to give a solid that was recrystallized out of a minimal amount of hot methanol. 0.130g, 22% yield. ^{1}H NMR $CDCl_3$ (**Figure S4**) TMS ref. δ 6.36 (m, 1H), 7.09 (s br, 1H), 7.13 (s br, 1H), 7.335 (d/m, 4H, J=16 Hz), 7.64 (m, 2H), 7.84 (d, 1H, J=16 Hz), 9.85 (s br, 1H). ^{13}C NMR, $CDCl_3$ (**Figure S5 and S6**) ref. δ 111.15, 116.42, 122.06, 125.50, 128.46, 129.04, 130.36, 133.30, 135.15, 142.42, 178.99

2- Solubility data

Table S1- Solubility data for both heterocyclic chalcones and *trans*-chalcone, with solvent properties.

Solvent	Nature of solvent	Relative polarity [a]	Solubility limit of *trans*-chalcone / mg ml^{-1}	Solubility limit of **1** / mg ml^{-1}	Solubility limit of **2** / mg ml^{-1}
n-Hexane	Non-polar, Aliphatic	0.009	44.0 ± 3.6	Insoluble	Insoluble
Toluene	Non-polar, Aromatic	0.099	630 ± 6.2	Insoluble	14.0 ± 7.1
THF	Polar, aprotic, cyclic	0.207	1010 ± 8.8	213 ± 3.9	105 ± 7.3
Ethyl acetate	Polar, aprotic	0.228	855 ± 7.7	49.5 ± 3.6	49.0 ± 7.1
Chloroform	Polar, aprotic	0.259	730 ± 6.8	65.5 ± 3.6	51.0 ± 7.1
Acetone	Polar, aprotic	0.355	1050 ± 9.1	101 ± 3.6	82.0 ± 7.2
Ethanol	Polar, protic	0.654	175 ± 3.8	9.00 ± 3.5	11.0 ± 7.1
Methanol	Polar, protic	0.762	130 ± 3.7	14.0 ± 3.5	10.5 ± 7.1

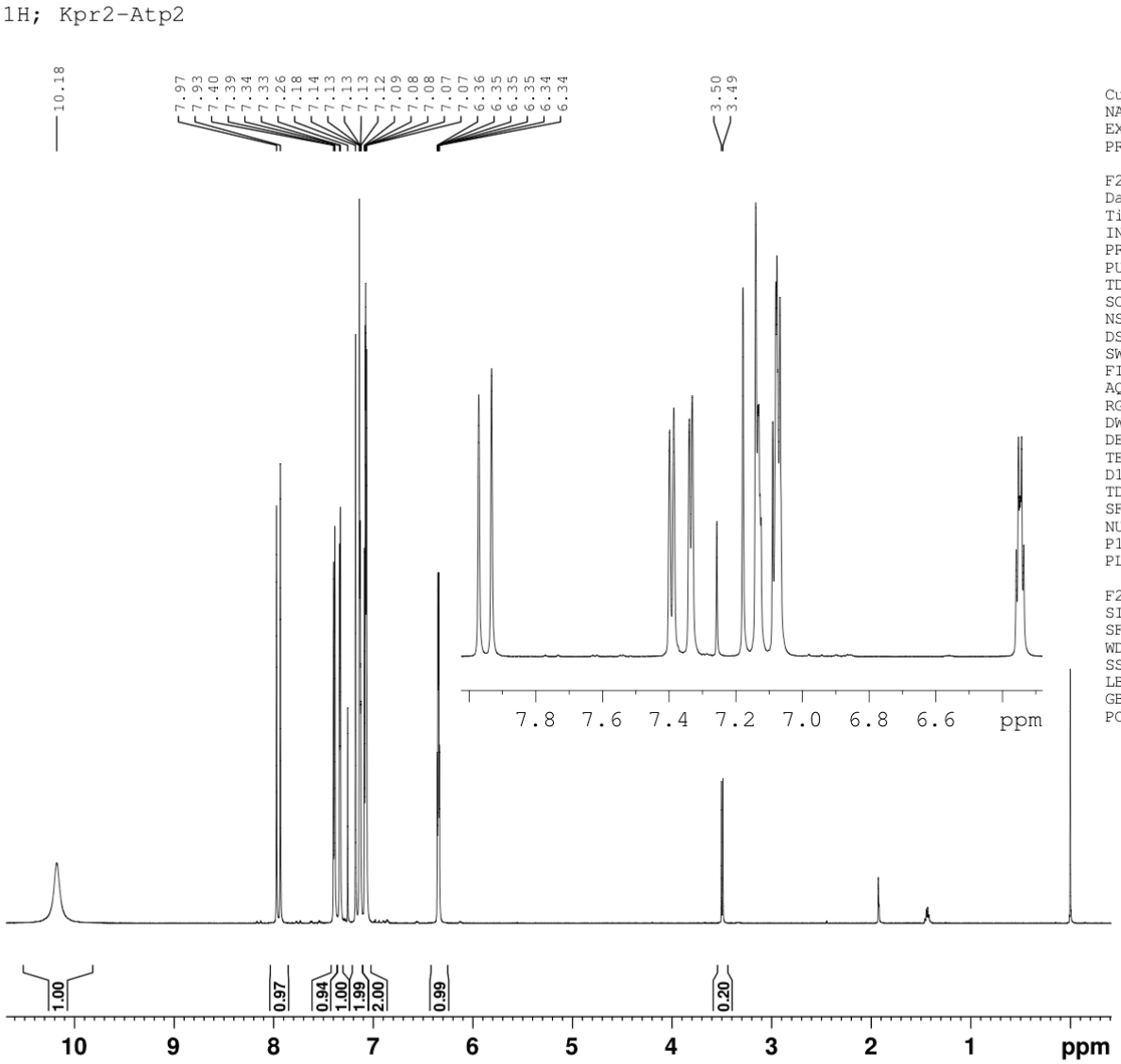
[a] The values for relative polarity are normalised from measurements of solvent shifts of absorption (Reichardt and Welton, 2011).

The solvents were chosen to cover a range of different potential interactions that could occur between the chalcones and the solvents. These solvents are also common in most laboratories and are so most likely to be used in the synthesis of more complex molecules where a chalcone is used as a reactant. Values were found by forming saturated solutions of each solvent and chalcone combination, with a volume of either 0.2 cm^3 for *trans*-chalcone and **1**, or 0.05 cm^3 of **2**. Volumes were measured using a Gilson pipette. Different volumes were used due to material availability. Once the saturated solution was formed, the solutions were decanted into pre-weighed test tubes to remove any undissolved chalcone from the solutions. The saturated solutions were left until all the solvent was evaporated. The mass of dissolved chalcone was then recorded and converted into a concentration. Uncertainties for the saturated solution concentrations (δC) was calculated using the formula:

$$\delta(C) = C\sqrt{\left(\frac{\sqrt{2(\delta m)^2}}{m}\right)^2 + \left(\frac{\delta V}{V}\right)^2}$$

Where C is the concentration of the saturated solution, δm is the resolution of the balance being used, m is the mass of the dissolved chalcone, δV is the uncertainty in the Gilson pipette and V is the volume of solution that was used to form the saturated solution.

3- Nuclear Magnetic Resonance (NMR) data



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DS                      2
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TE                  294.3 K
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TD0                     1
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NUC1                   1H
P1                  14.00 usec
PLW1          11.72999954 W

F2 - Processing parameters
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Figure S1- ^{1}H NMR of (*E*)-1-(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one.

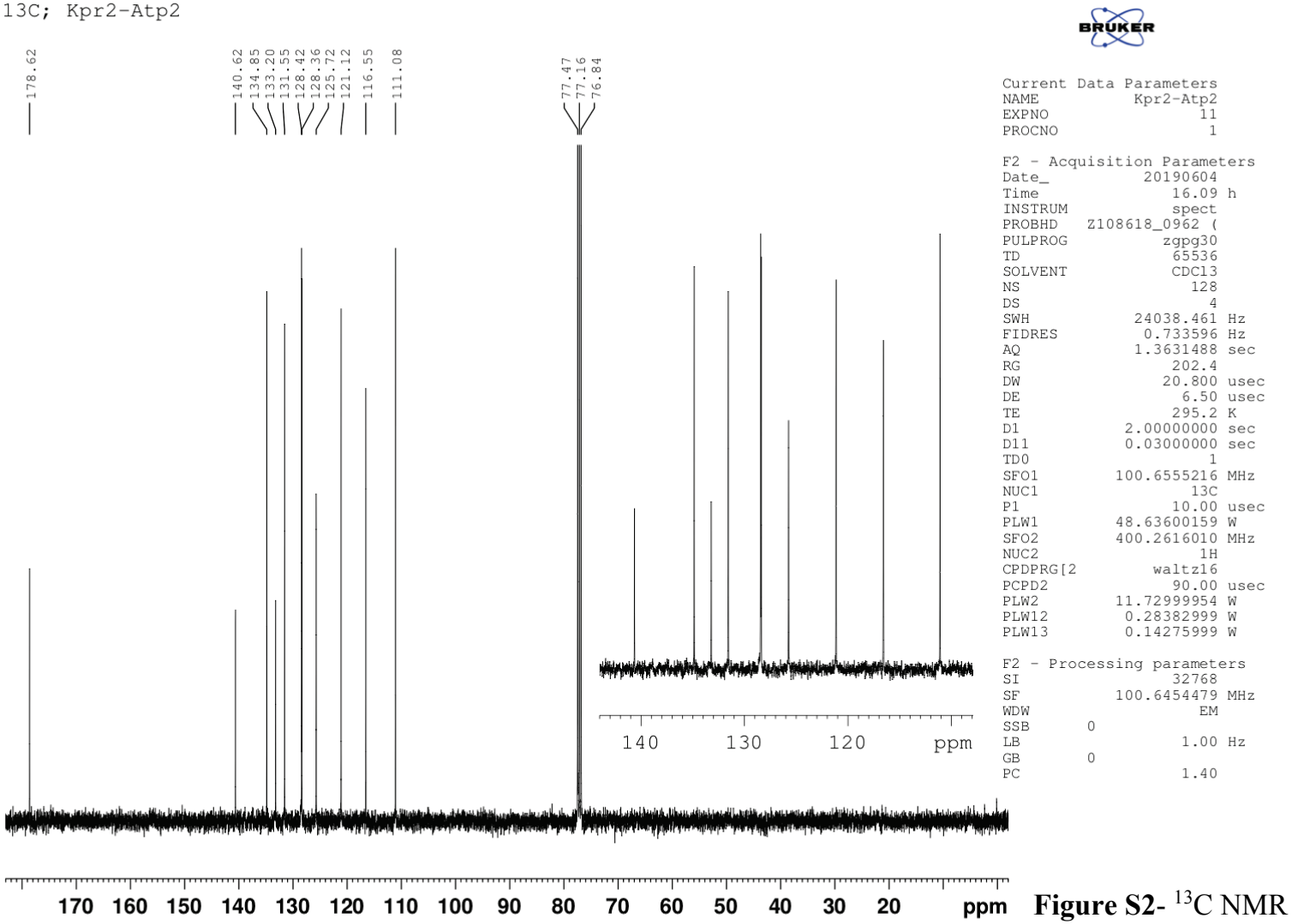
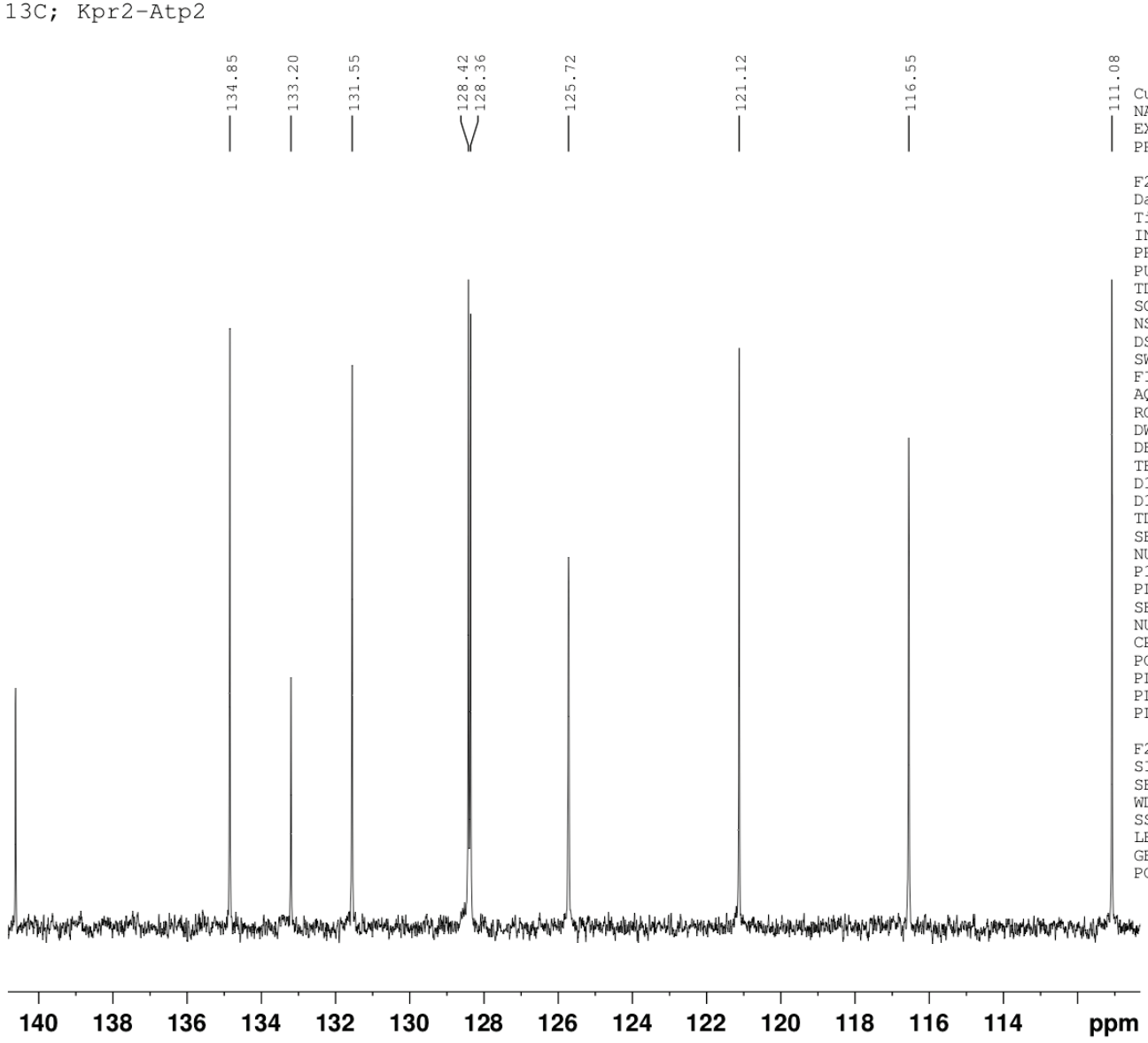


Figure S2- ^{13}C NMR of (*E*)-1-(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one.

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SOLVENT          CDCl3
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DS                   4
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FIDRES        0.733596 Hz
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RG               202.4
DW              20.800 usec
DE               6.50 usec
TE              295.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1
SFO1        100.6555216 MHz
NUC1               13C
P1               10.00 usec
PLW1        48.63600159 W
SFO2        400.2616010 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2            90.00 usec
PLW2        11.72999954 W
PLW12        0.28382999 W
PLW13        0.14275999 W

F2 - Processing parameters
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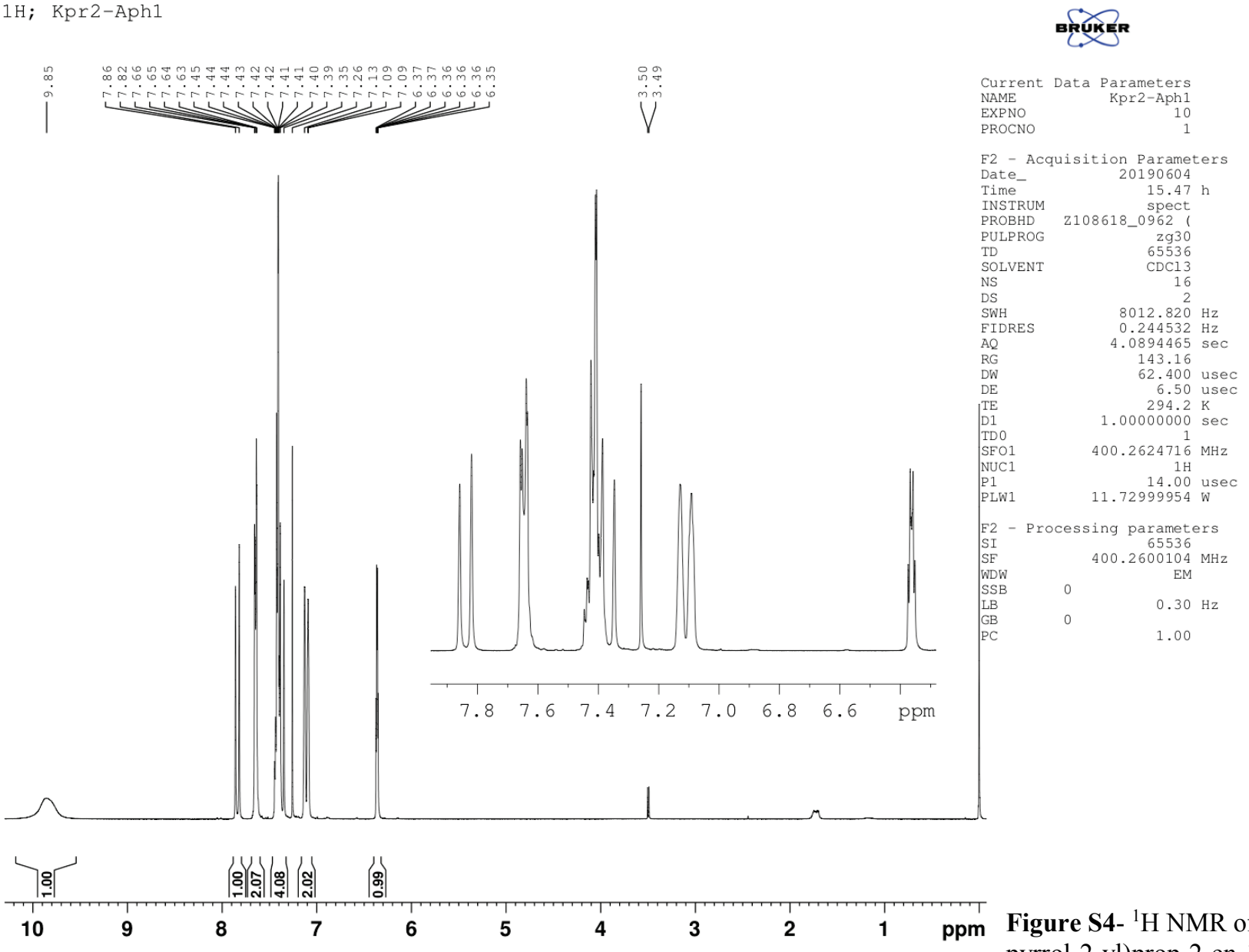
Figure S3- ^{13}C NMR of (*E*)-1-(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one in the range 110 – 142 ppm.

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DS                     2
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FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 143.16
DW                 62.400 usec
DE                  6.50 usec
TE                 294.2 K
D1            1.00000000 sec
TD0                    1
SFO1          400.2624716 MHz
NUC1                  1H
P1                 14.00 usec
PLW1          11.72999954 W

F2 - Processing parameters
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WDW                   EM
SSB       0
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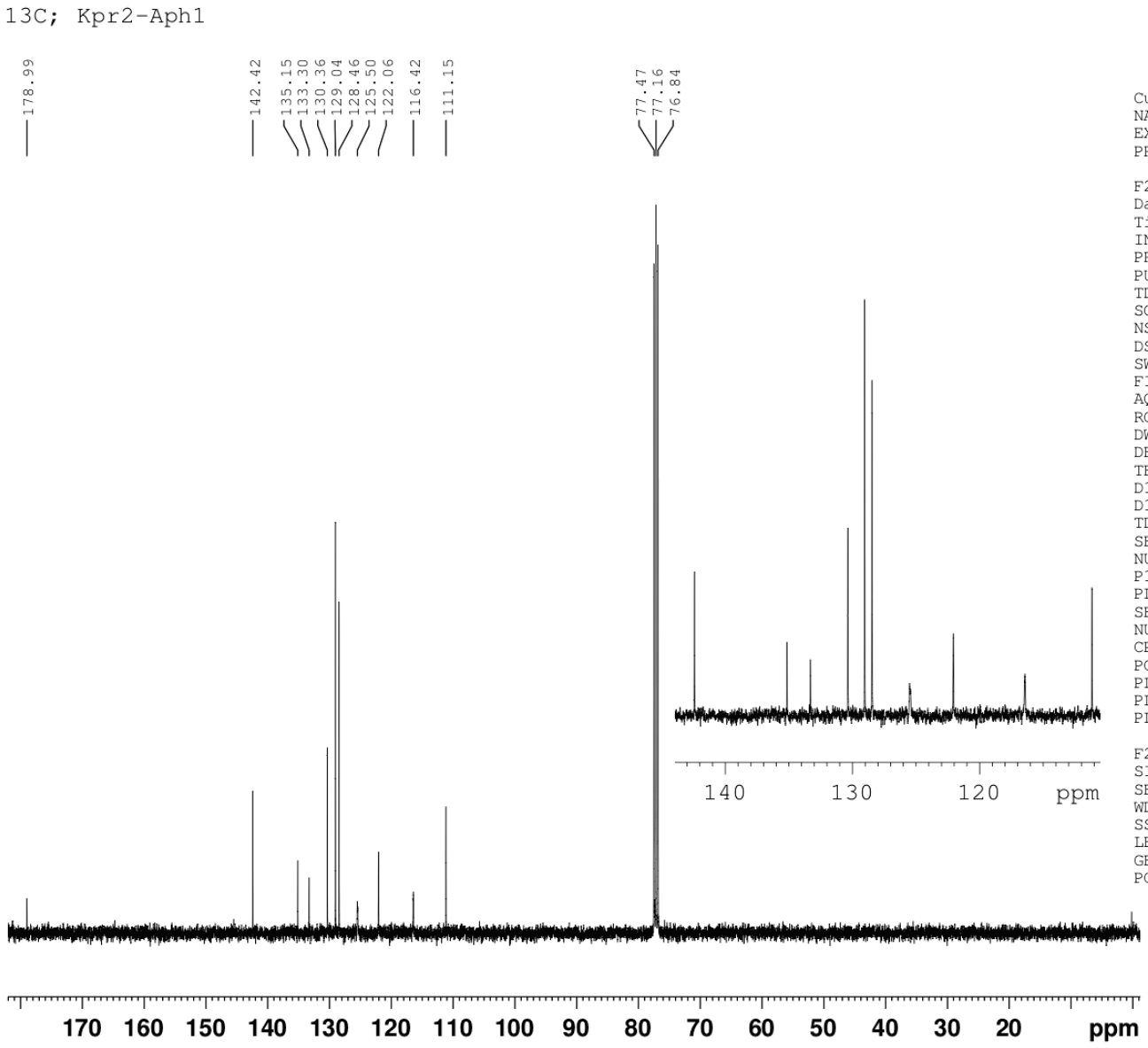
Figure S4- ^{1}H NMR of (*E*)-3-phenyl-1-(1*H*-pyrrol-2-yl)prop-2-en-1-one.



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NS                         128
DS                           4
SWH                  24038.461 Hz
FIDRES                0.733596 Hz
AQ                    1.3631488 sec
RG                       202.4
DW                      20.800 usec
DE                       6.50 usec
TE                       295.1 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          1
SFO1                100.6555216 MHz
NUC1                        13C
P1                      10.00 usec
PLW1                48.63600159 W
SFO2                400.2616010 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                   90.00 usec
PLW2                11.72999954 W
PLW12                0.28382999 W
PLW13                0.14275999 W

F2 - Processing parameters
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WDW                         EM
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LB                       1.00 Hz
GB        0
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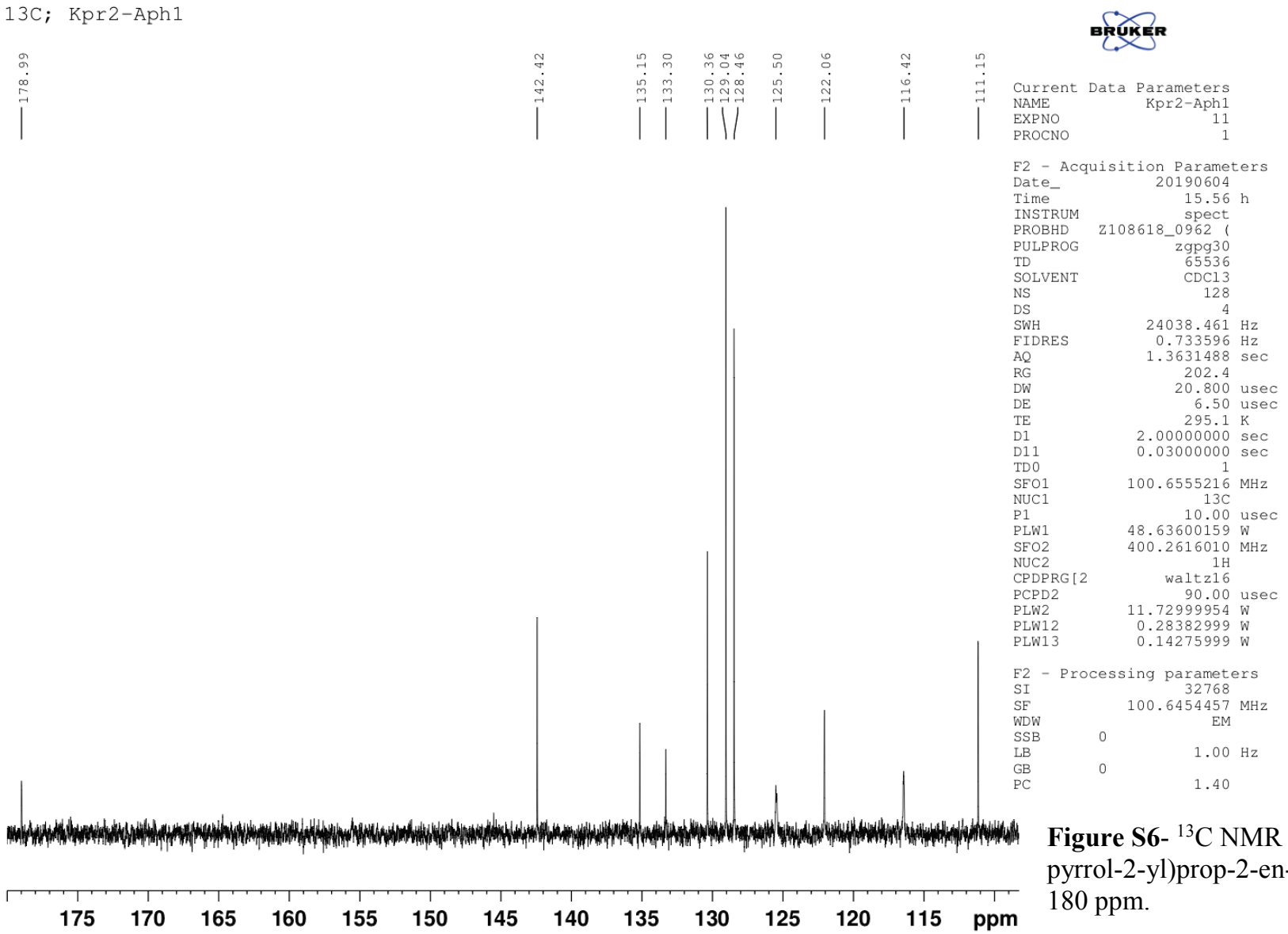
Figure S5- ^{13}C NMR of (*E*)-3-phenyl-1-(1*H*-pyrrol-2-yl)prop-2-en-1-one.

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PROCNO                1

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TD                65536
SOLVENT           CDCl3
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DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG                202.4
DW               20.800 usec
DE                6.50 usec
TE                295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
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SFO1        100.6555216 MHz
NUC1                13C
P1                10.00 usec
PLW1        48.63600159 W
SFO2        400.2616010 MHz
NUC2                 1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        11.72999954 W
PLW12        0.28382999 W
PLW13        0.14275999 W

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Figure S6- ^{13}C NMR of (*E*)-3-phenyl-1-(1*H*-pyrrol-2-yl)prop-2-en-1-one in the range 108 – 180 ppm.

4- Crystal growth results via slow evaporation

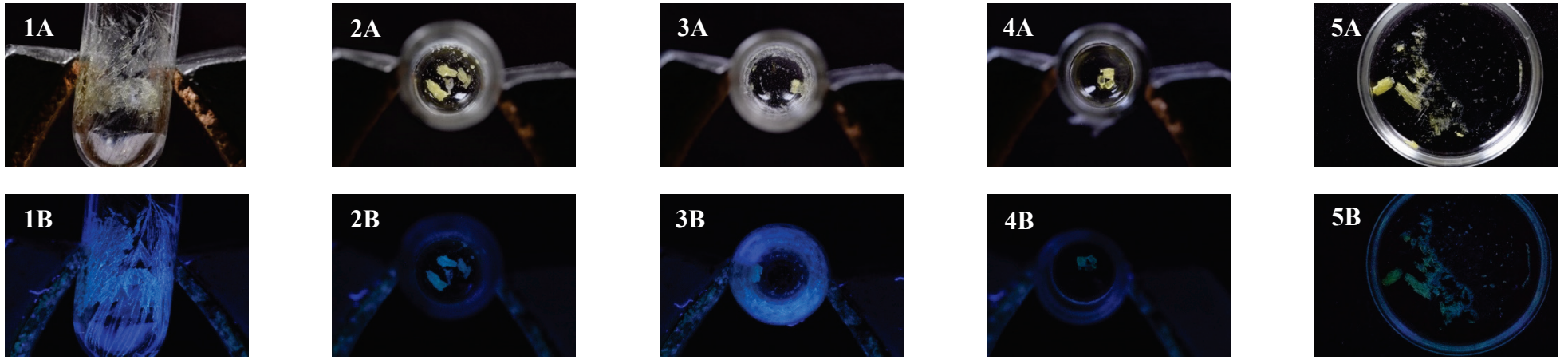

Figure S7- Images of (*E*)-1-(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one crystals that were grown by the slow evaporation process from a number of different solvents. All the solutions used had a concentration of 0.05 mol dm^{-3}. 1: Ethyl acetate; 2: THF; 3: Chloroform; 4: Methanol, 5: Acetone. Row A are pictures taken under visible light and row B are pictures taken under a UV lamp. Camera settings for row B – ISO: 1600, Shutter speed: 30 seconds, aperture: 40.0.

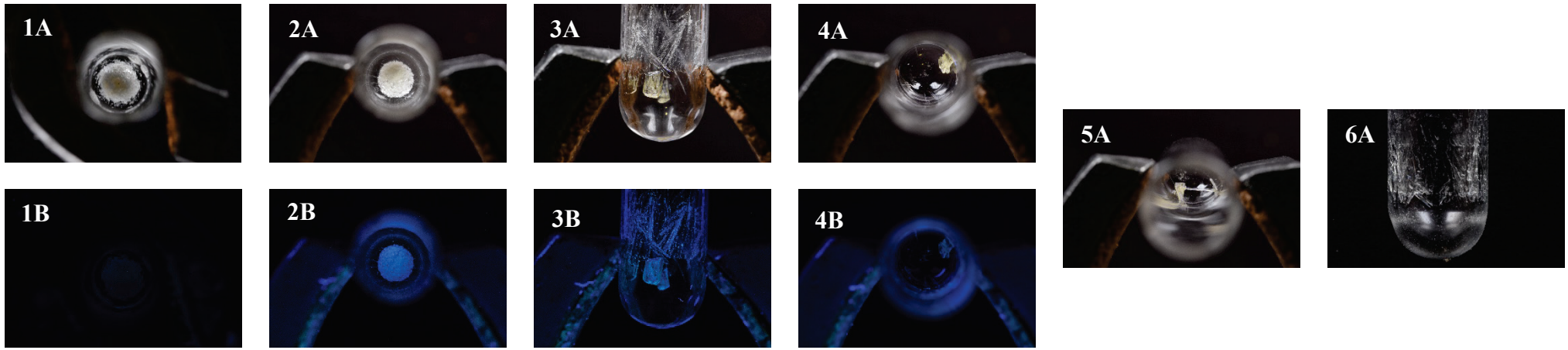

Figure S8- Images of (*E*)-3-phenyl-1-(1*H*-pyrrol-2-yl)prop-2-en-1-one crystals that were grown by the slow evaporation process from a number of different solvents. All the solutions used had a concentration of 0.05 mol dm^{-3}. 1: Ethyl acetate; 2: THF; 3: Methanol; 4: Acetone; 5: Chloroform; 6: Toluene. Row A are pictures taken under visible light and row B are pictures taken under a UV lamp. Camera settings for row B – ISO: 1600, Shutter speed: 30 seconds, aperture: 40.0.

5- Molecular electrostatic potential (MEP) plots

Molecular electrostatic potentials for the chalcones were plotted in GaussView 5.0.8 after optimisation of the structures using the B3LYP functional and 6-31G basis set (Frisch *et al.,* 2009)

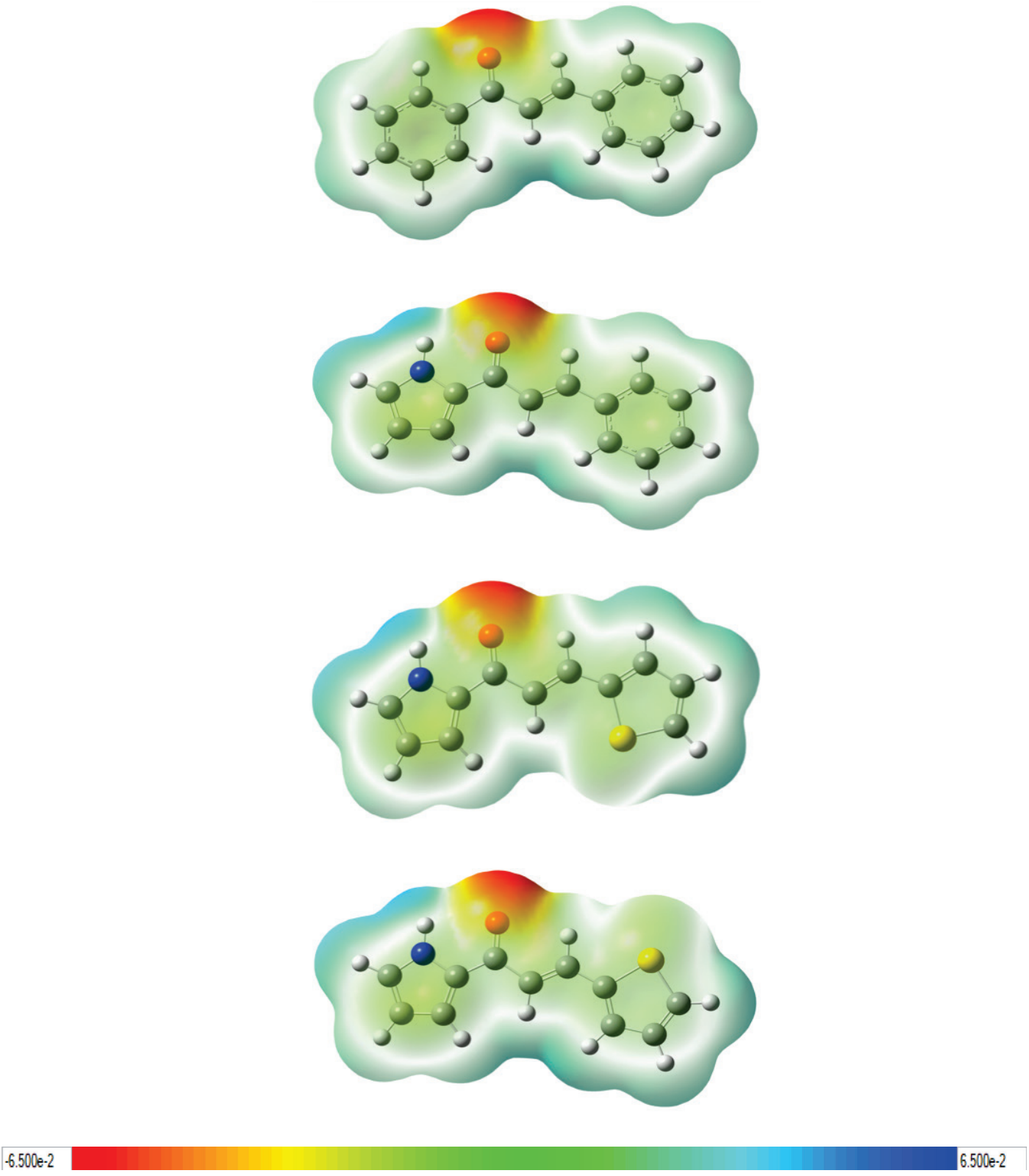

Figure S9: The MEP's for the three chalcones being considered in the range -6.5 x 10^{-2} to 6.5 x 10^{-2} with an isovalue, MO = 0.020000, density = 0.000400. Top to Bottom: *Trans*-chalcone; (*E*)-3-phenyl-1-(1*H*-pyrrol-2-yl)prop-2-en-1-one; (*E*)-1-(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one (major component); (*E*)-1-(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one (minor component).

6- Differential Scanning Calorimetry

The DSC experiments involved cooling and heating the heterocyclic chalcones in hermetic aluminium pans between 200 °C to 0 °C at a rate of 10 °C min $^{-1}$, using a TA instruments DSC 25 instrument under a dry nitrogen purge. *Trans*-chalcone was heated on a cycle between 100 °C to -50 °C at a rate of 10 °C min $^{-1}$.

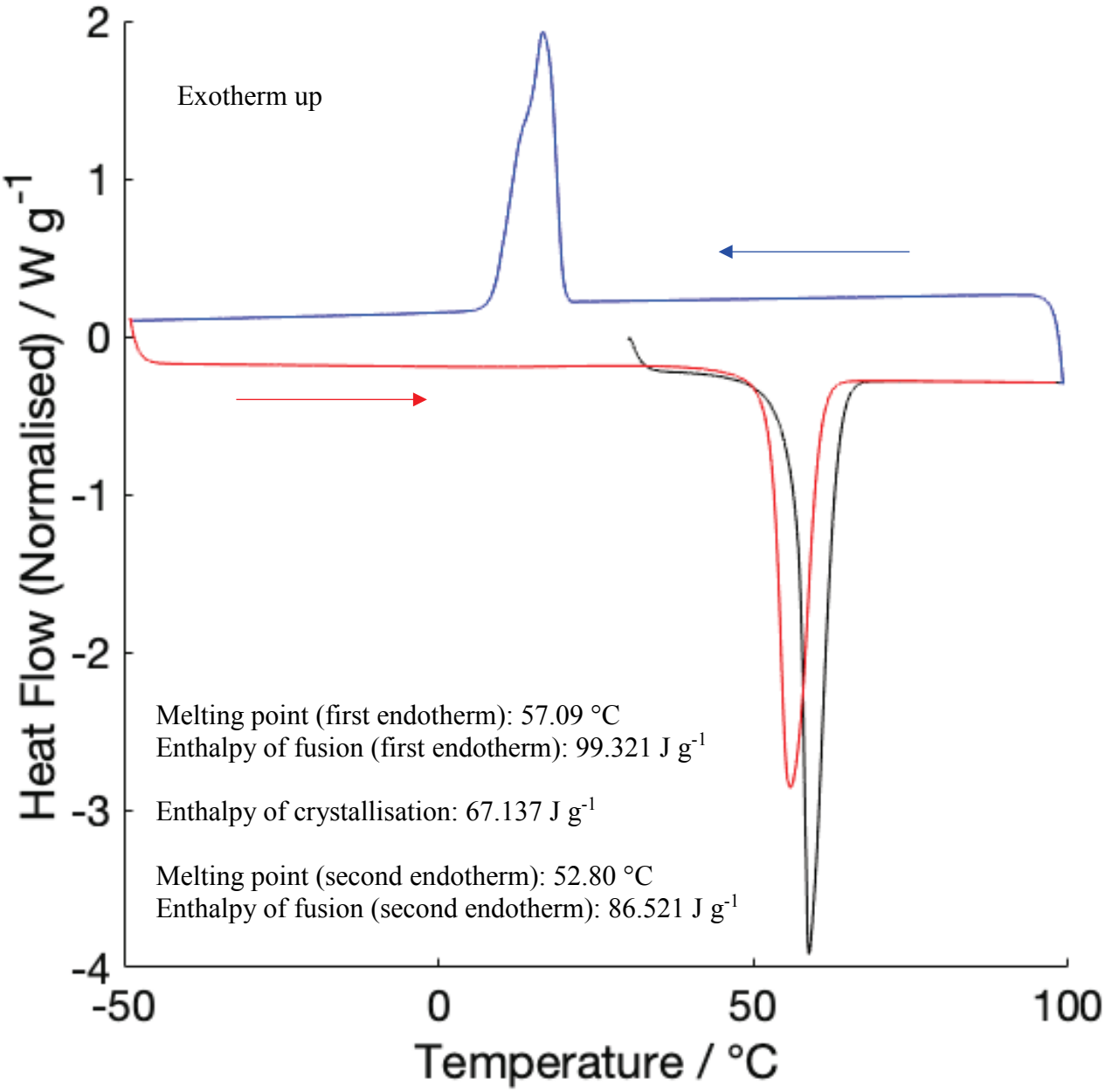

Figure S10- DSC heating and cooling cycle for *trans*-chalcone. Black line: Heating on cycle 1; Blue line: Cooling; Red line: Heating on cycle 2. Enthalpy of fusion for both endotherms and the enthalpy of crystallisation are provided, along with the melting points which were found. The mass of chalcone used was 10.1 mg (48 µmol).

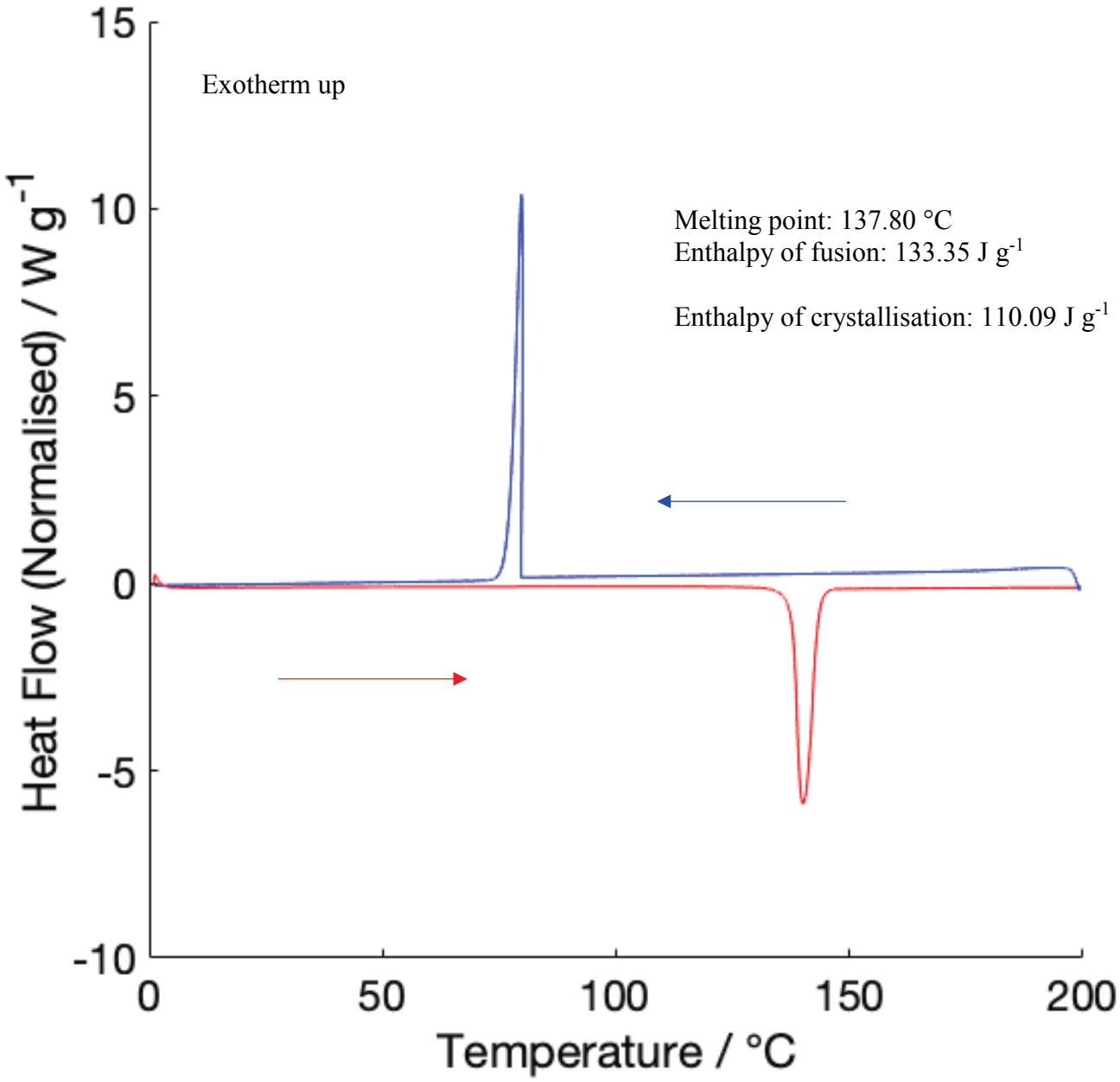

Figure S11- DSC heating and cooling cycle for (*E*)-3-phenyl-1-(1*H*-pyrrol-2-yl)prop-2-en-1-one. Blue line: Cooling; Red line: Heating. Enthalpy of fusion and crystallisation are provided along with the melting point. The mass of chalcone used was 2.4 mg (12 µmol).

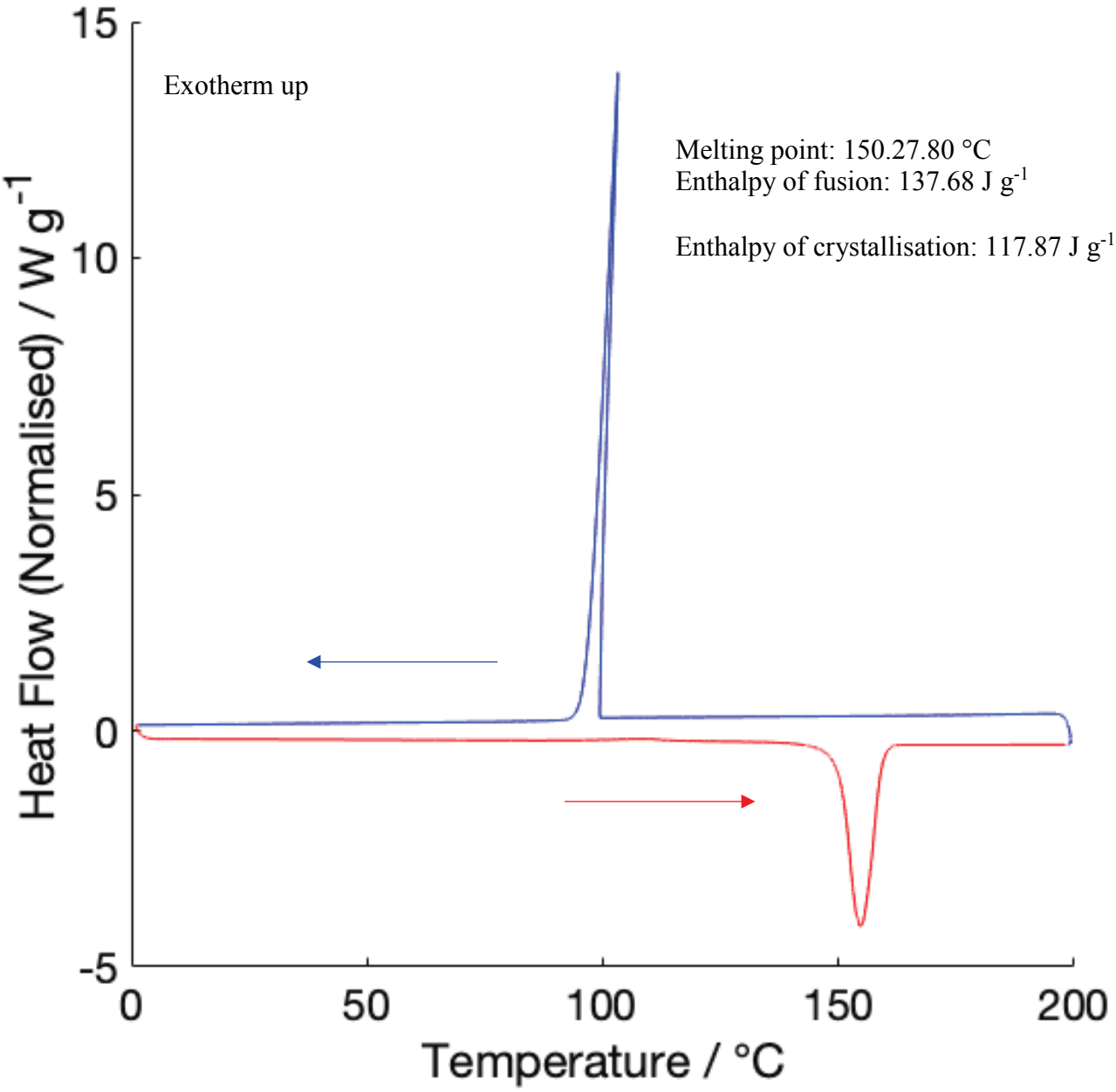

Figure S12- DSC heating and cooling cycle for (*E*)-1-(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one. Blue line: Cooling; Red line: Heating. Enthalpy of fusion and crystallisation are provided along with the melting point. The mass of chalcone used was 7.6 mg (37 µmol)

7- Powder X-ray diffraction data

Powder X-ray diffraction data was collected on a Bruker D8 Advance with Cu-K α radiation (λ = 1.540600 Å) and a PSD LynxEye detector, in Bragg-Brentano geometry. Data was collected over a 2θ range of 5-50° with a 0.013° step size and 1 second per step. The sample stage was not rotated. The simulated powder patterns obtained from the single crystal structures, using Mercury, matched the diffractograms collected and were used to assign the indices (Macrae *et al.,* 2006).

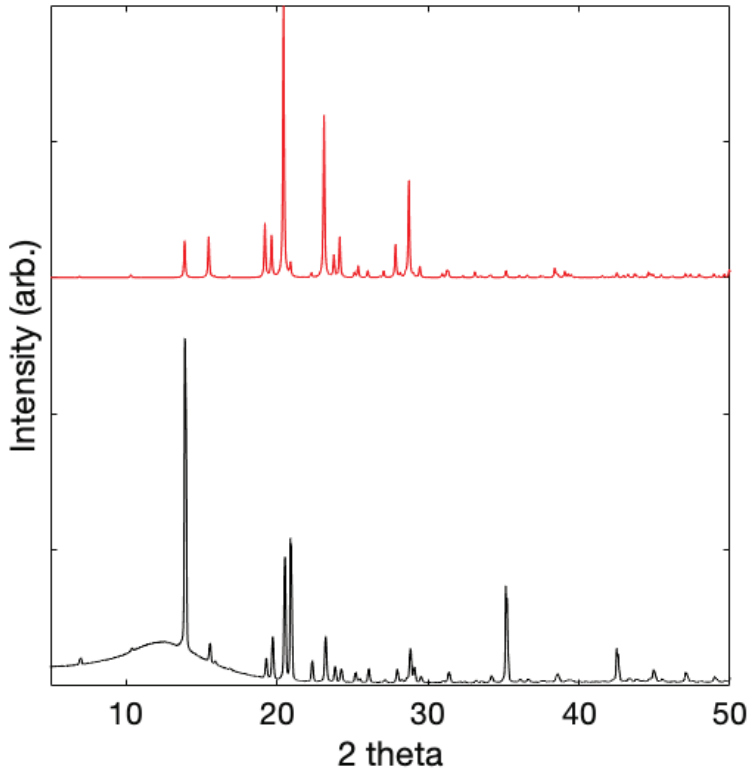

Figure S13- Comparison of the simulated powder X-ray pattern (red line) of *trans*-chalcone Form I to experimental data (black line).

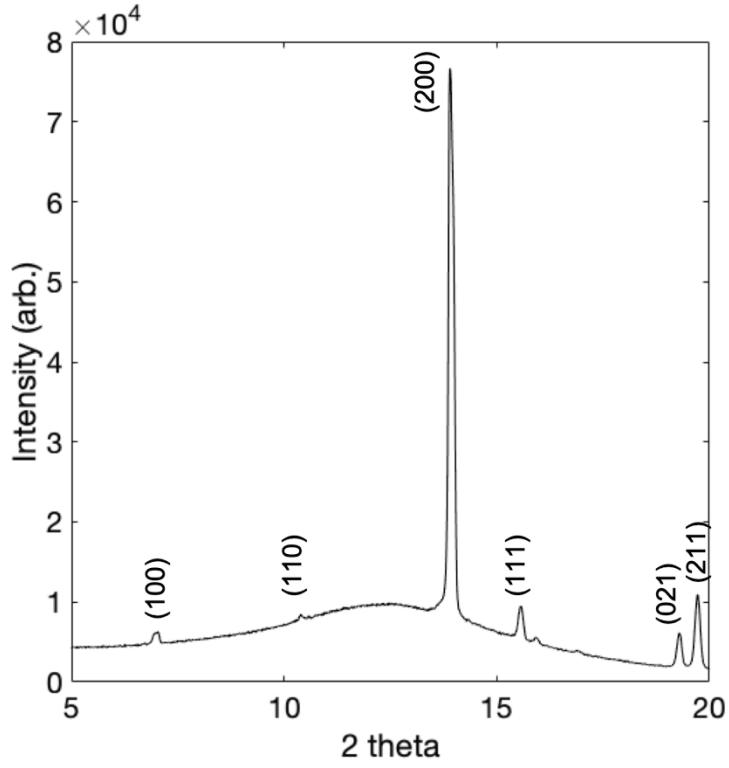

Figure S14- Powder X-ray diffraction data for *trans*-chalcone Form I in the range 5° 2θ to 20° 2θ with reflections indexed.

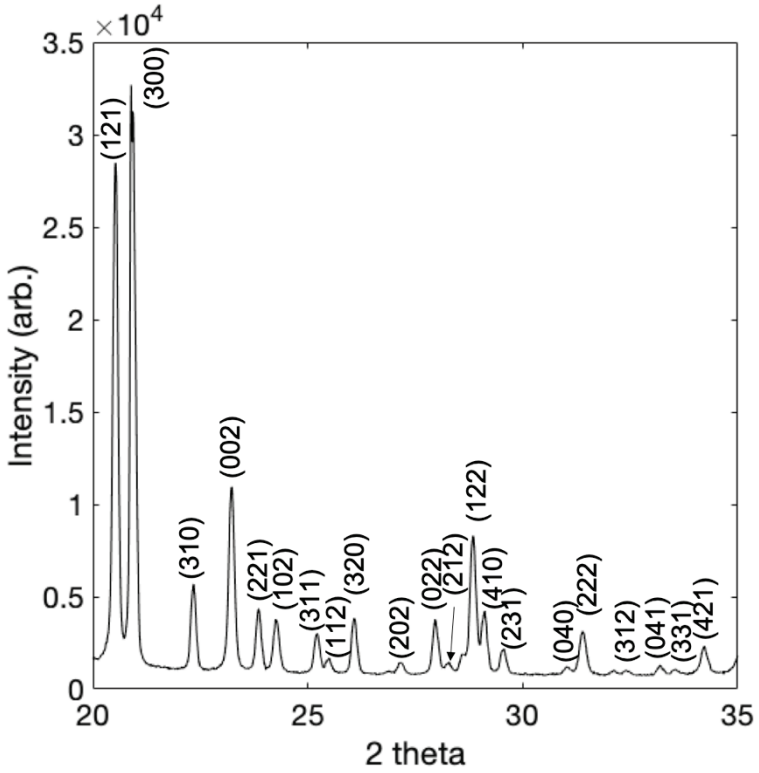

Figure S15- Powder X-ray diffraction data for *trans*-chalcone Form I in the range 20° 2θ to 35° 2θ with reflections indexed.

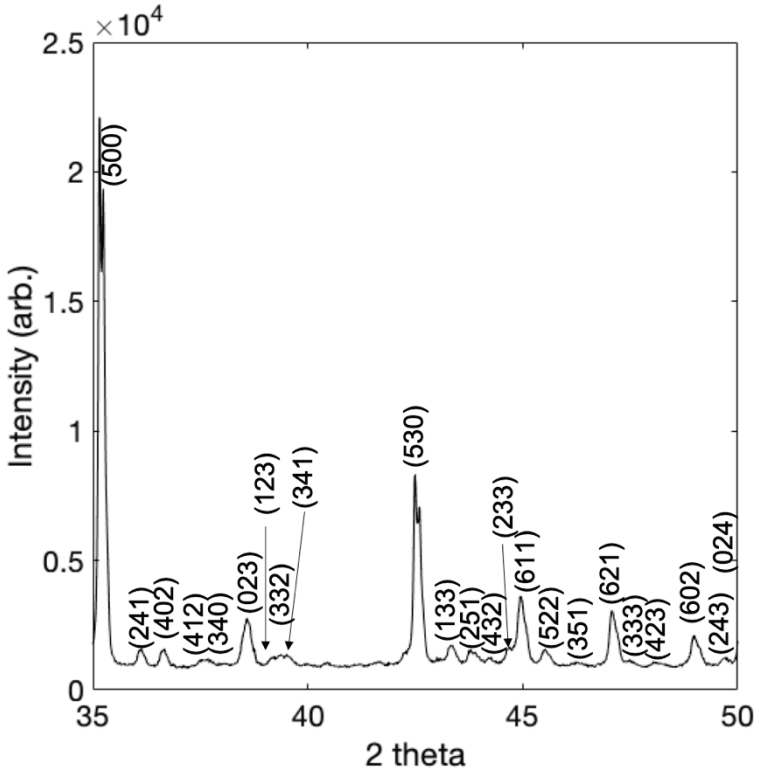

Figure S16- Powder X-ray diffraction data for *trans*-chalcone Form I in the range 35° 2θ to 50° 2θ with reflections indexed.

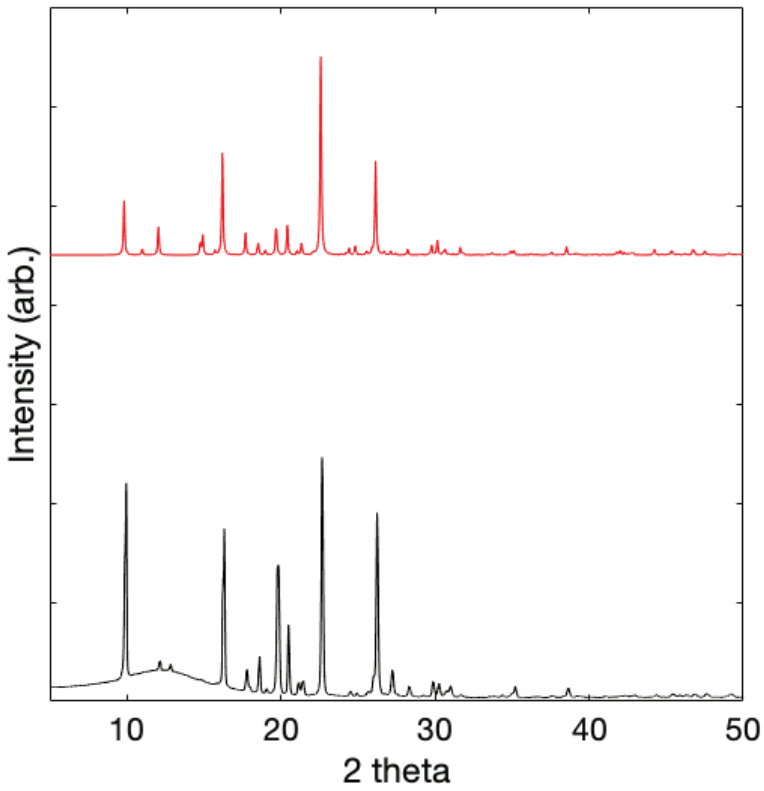

Figure S17- Comparison of the simulated powder X-ray pattern (red line) of *trans*-chalcone Form II to experimental data (black line).

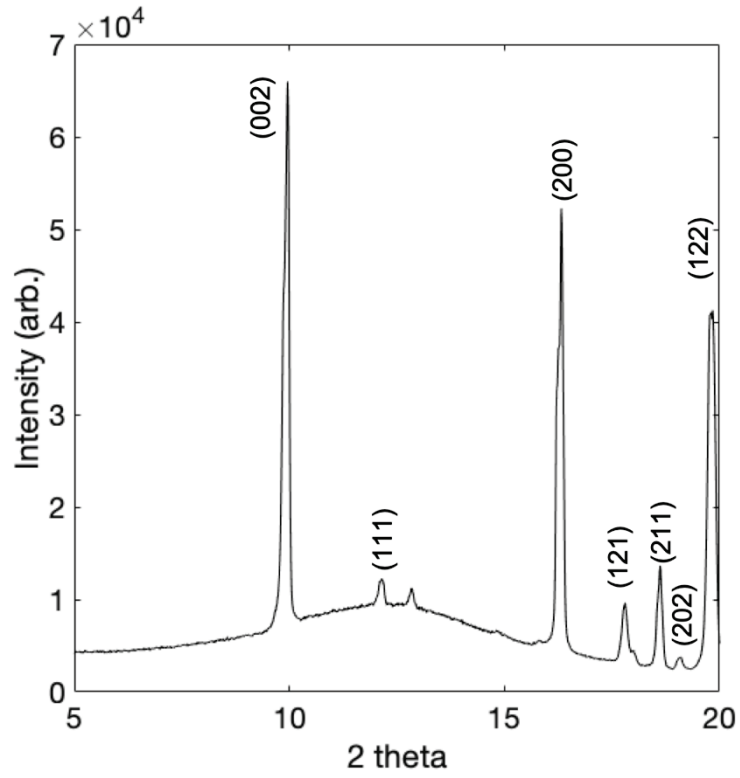

Figure S18- Powder X-ray diffraction data for *trans*-chalcone Form II in the range 5° 2θ to 20° 2θ with reflections indexed.

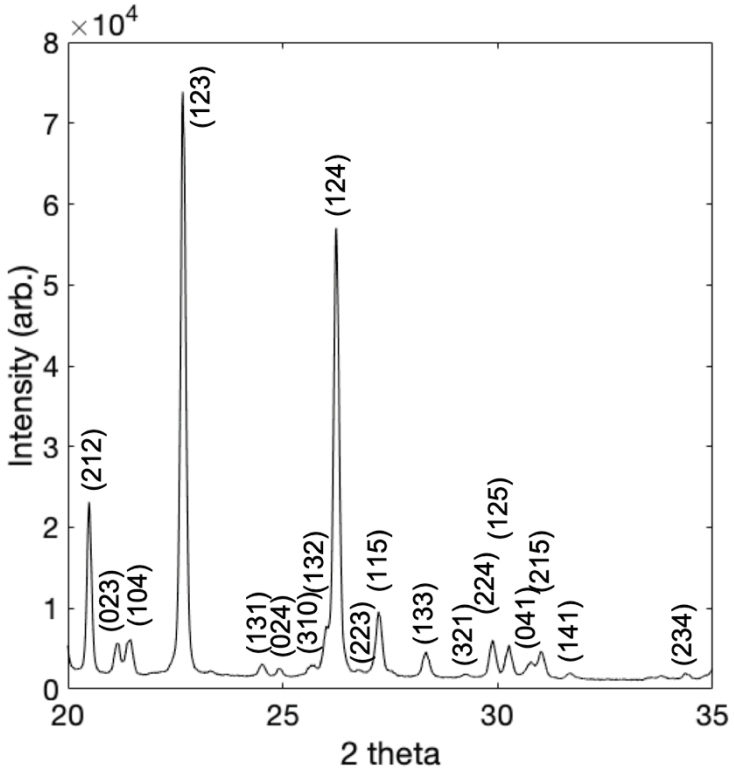

Figure S19- Powder X-ray diffraction data for *trans*-chalcone Form II in the range 20° 2θ to 35° 2θ with reflections indexed.

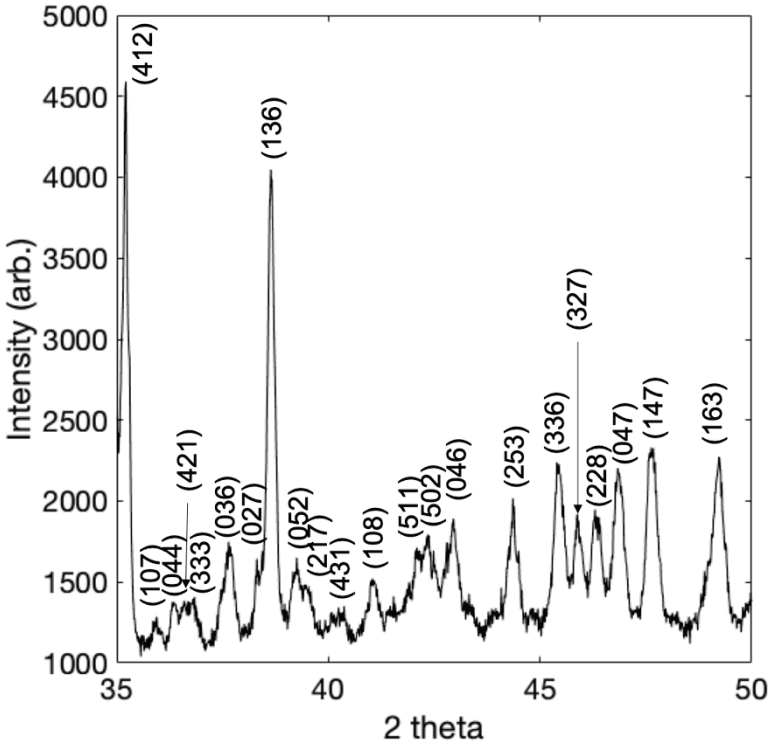

Figure S20- Powder X-ray diffraction data for *trans*-chalcone Form II in the range 35° 2θ to 50° 2θ with reflections indexed.

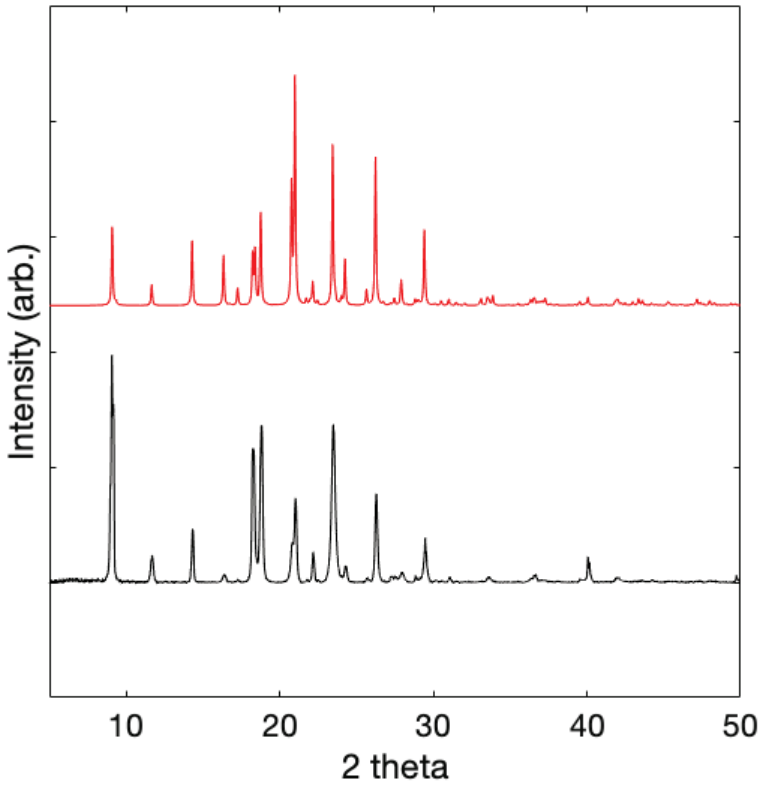

Figure S21- Comparison of the simulated powder X-ray pattern (red line) of (*E*)-3-phenyl-1-(1*H*-pyrrol-2-yl)prop-2-en-1-one to experimental data (black line).

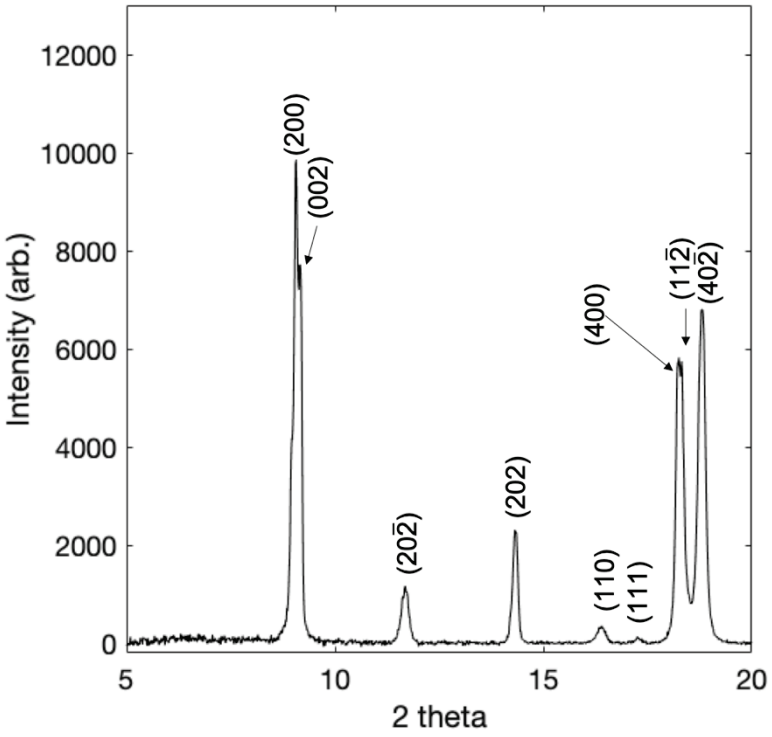

Figure S22- Powder X-ray diffraction data for (*E*)-3-phenyl-1-(1*H*-pyrrol-2-yl)prop-2-en-1-one in the range 5° 2θ to 20° 2θ with reflections indexed.

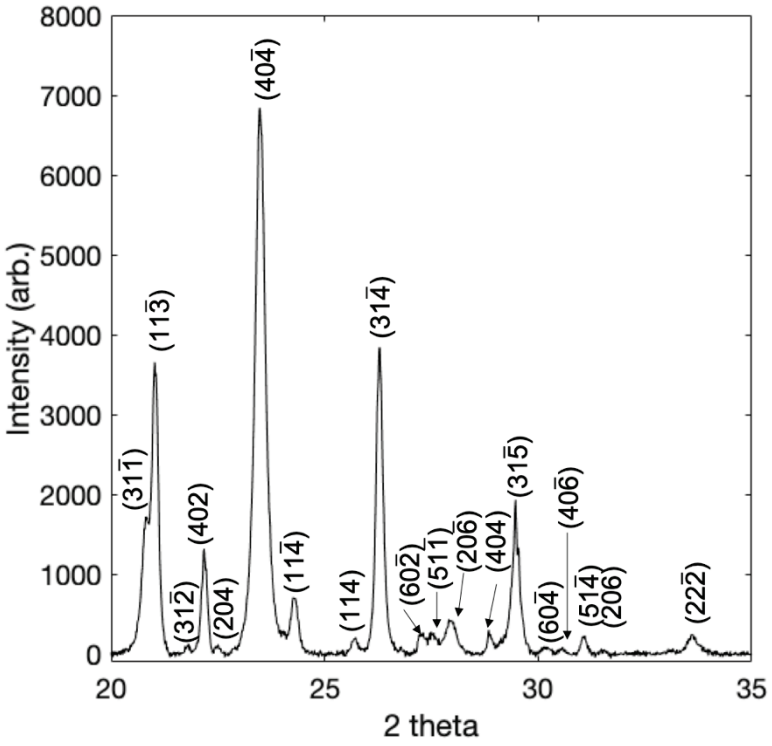

Figure S23- Powder X-ray diffraction data for (*E*)-3-phenyl-1-(1*H*-pyrrol-2-yl)prop-2-en-1-one in the range 20° 2θ to 35° 2θ with reflections indexed.

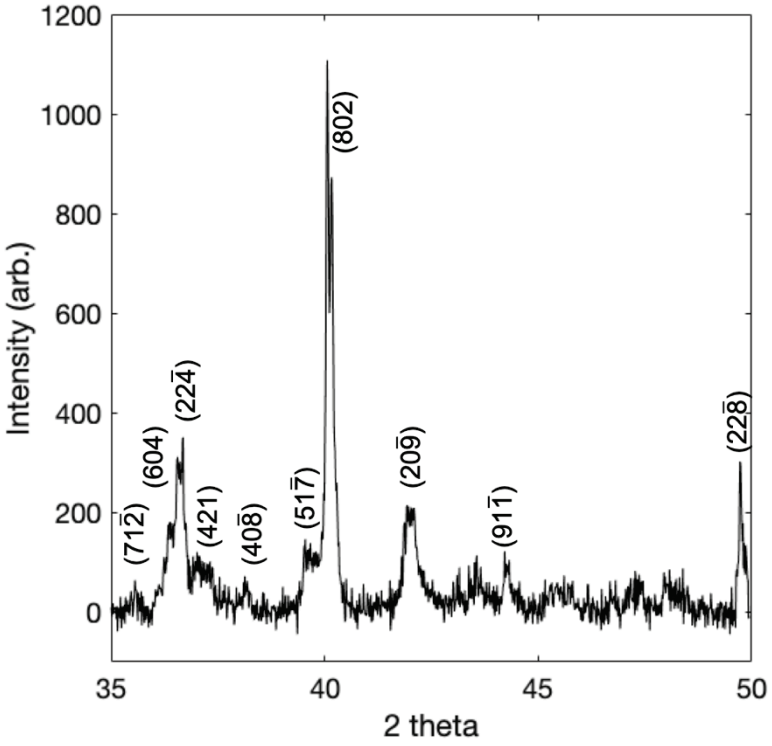

Figure S24- Powder X-ray diffraction data for (*E*)-3-phenyl-1-(1*H*-pyrrol-2-yl)prop-2-en-1-one in the range 35° 2θ to 50° 2θ with reflections indexed.

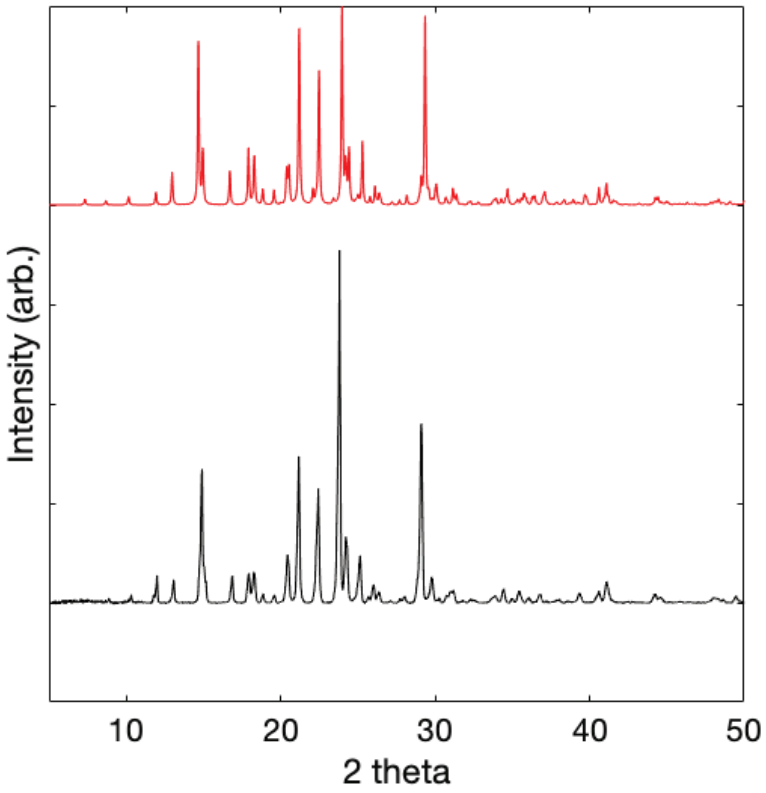

Figure S25- Comparison of the simulated powder X-ray pattern (red line) of (*E*)-1-(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one to experimental data (black line).

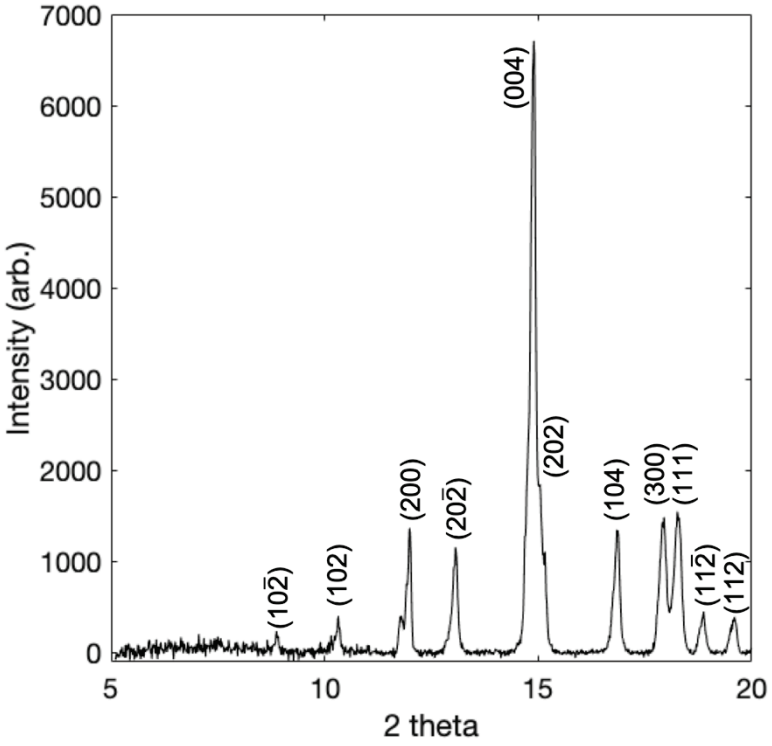

Figure S26- Powder X-ray diffraction data for (*E*)-1(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one in the range 5° 2θ to 20° 2θ with reflections indexed.

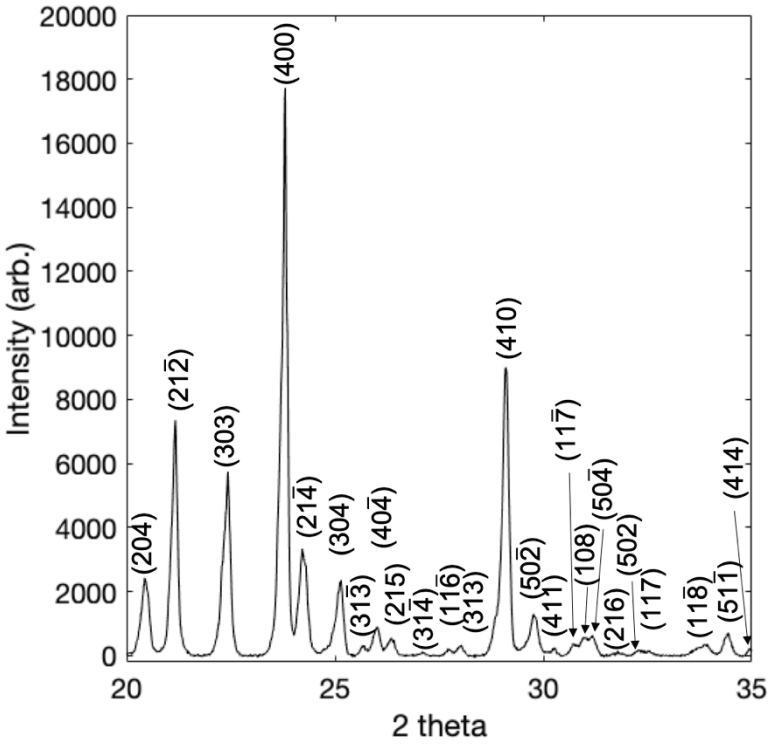

Figure S27- Powder X-ray diffraction data for (*E*)-1(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one in the range 20° 2θ to 35° 2θ with reflections indexed.

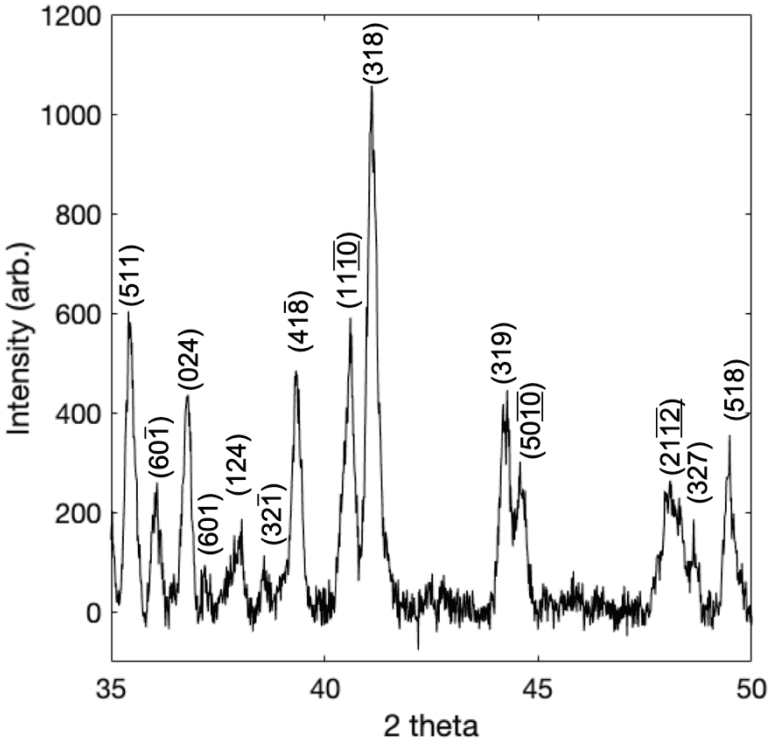

Figure S28- Powder X-ray diffraction data for (*E*)-1(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one in the range 35° 2θ to 50° 2θ with reflections indexed.

8- Single crystal X-ray diffraction data

Single crystals of $C_{11}H_9NOS$ [(*E*)-1-(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one] were grown by slow evaporation from THF at a concentration of 0.05 mol dm^{-3}.

Single crystal X-ray diffraction experiments were carried out at 100(2) K on a Bruker APEX II CCD diffractometer using Mo-K α radiation (λ = 0.71073 Å). Intensities were integrated and absorption corrections were based on equivalent reflections using SADABS (*SAINT+*; Bruker, 2007; *SADABS*; Bruker, 2001). The structure was solved using Superflip and refined against F^2 in SHELXL using Olex2 (Palatinus and Chapuis, 2007; Palatinus *et al.,* 2012; Sheldrick, 2008; Sheldrick, 2015; Dolomanov *et al.,* 2009). Crystal structure and refinement data are given in Table S2.

The structure was refined as a 2-component non-merohedral twin. Hydrogen atoms on nitrogen atoms were found by difference map after all other atoms had been refined. All other hydrogen atoms were placed geometrically and refined using a riding model. Both molecules, in the asymmetric unit, contain a disordered thiophene group which has been modelled in two positions with a refined occupancy ratio of 0.922:0.078(2) in molecule 1 and 0.884:0.116(2) in molecule 2. Equivalent atom distances in the major and minor components (e.g. C1-C2 and C1A-C2A) have been restrained to be the same. The ADPs of atoms in close proximity have also been restrained to be the same. Crystal figures were produced on Mercury 4.1.3 (Macrae *et al.,* 2006).

CCDC 1952662 contains the supplementary crystallographic data for this paper.

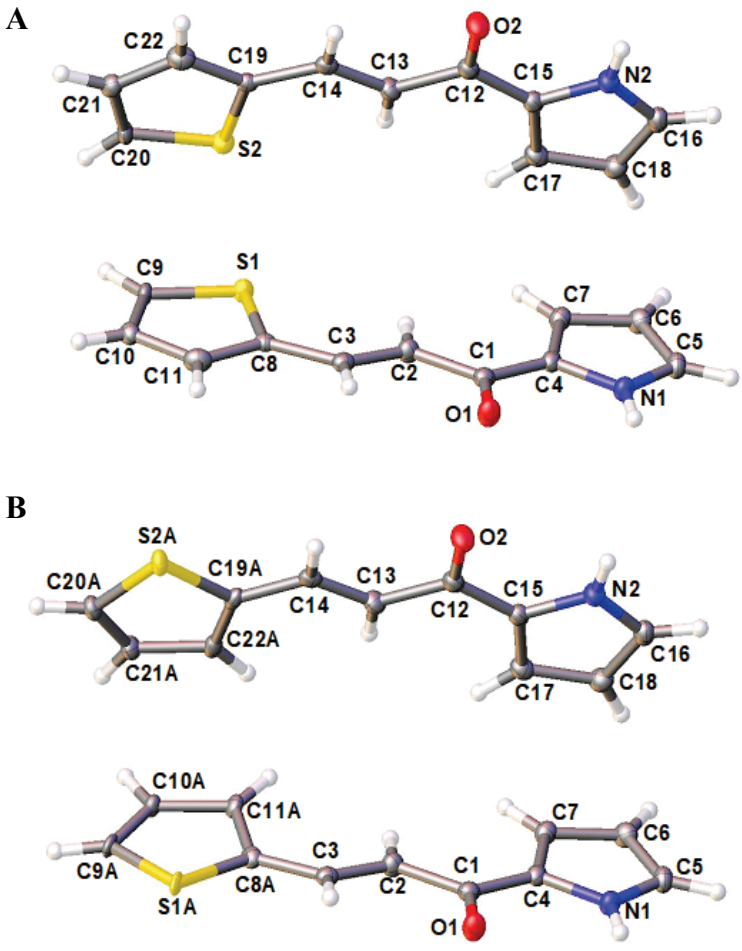

Figure S29- Diagrams of (E)-1(1H-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one showing the two disordered components separately. A: Major occupancy arrangement; B: Minor occupancy arrangement.

Table S2- Crystal data and structure refinement relating to (*E*)-1-(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one.

Empirical formula	$C_{11}H_9NOS$
Formula weight / g mol^{-1}	203.25
Temperature / K	99.96
Crystal system	Monoclinic
Space Group	$P2_1/c$
Hall Group	-*P* 2ybc
a / Å	15.0288(5)
b / Å	5.3086(2)
c / Å	24.4440(8)
α / °	90
β / °	99.133(2)
γ / °	90
Volume / Å^3	1925.46(12)
Z, Z'	8, 2
ρ_{calc} / g cm^{-3}	1.402
μ / mm^{-1}	0.298
F_{000}	848.0
Crystal size / mm^3	0.534 x 0.284 x 0.257
Radiation	Mo Kα (λ = 0.71073 Å)
2θ range for data collection	2.744 to 55.122
Index ranges	-19 ≤ h ≤ 19 0 ≤ k ≤ 6 0 ≤ l ≤ 31
Reflections collected	6848
Independent reflections	6848 [R_{sigma} = 0.0295]
Data/restraints/ parameters	6848/312/354
Goodness-of-fit on F^2	1.031
Final R indexes [I>=2σ (I)]	R_1 = 0.0365, wR_2 = 0.0856
Final R indexes [all data]	R_1 = 0.0505, wR_2 = 0.0923
Largets diff. peak/hole / e Å^{-3}	0.30 / -0.27

Table S3- Hydrogen bonds for (*E*)-1-(1*H*-pyrrol-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one.

D	H	A	d(D-H) / Å	d(H-A) / Å	d(D-A) / Å	D-H-A / °
N1	H1B	O1[1]	0.83(2)	2.05(2)	2.841(2)	160(2)
N2	H2B	O2[2]	0.84(2)	2.03(2)	2.832(2)	158(2)

[1]2-X, 2-Y, 1-Z; [2]1-X, 3-Y, 1-Z

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