

Supporting Information

The Crystal Landscape of Primary Aromatic

Thioamides

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[†] Department of Chemistry, Analytical and Biological Chemistry Research Facility, Synthesis and Solid State Pharmaceutical Centre, University College Cork, Cork, Ireland

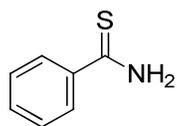
[§] Department of Chemistry and School of Pharmacy, Analytical and Biological Chemistry Research Facility, Synthesis and Solid State Pharmaceutical Centre, University College Cork, Cork, Ireland

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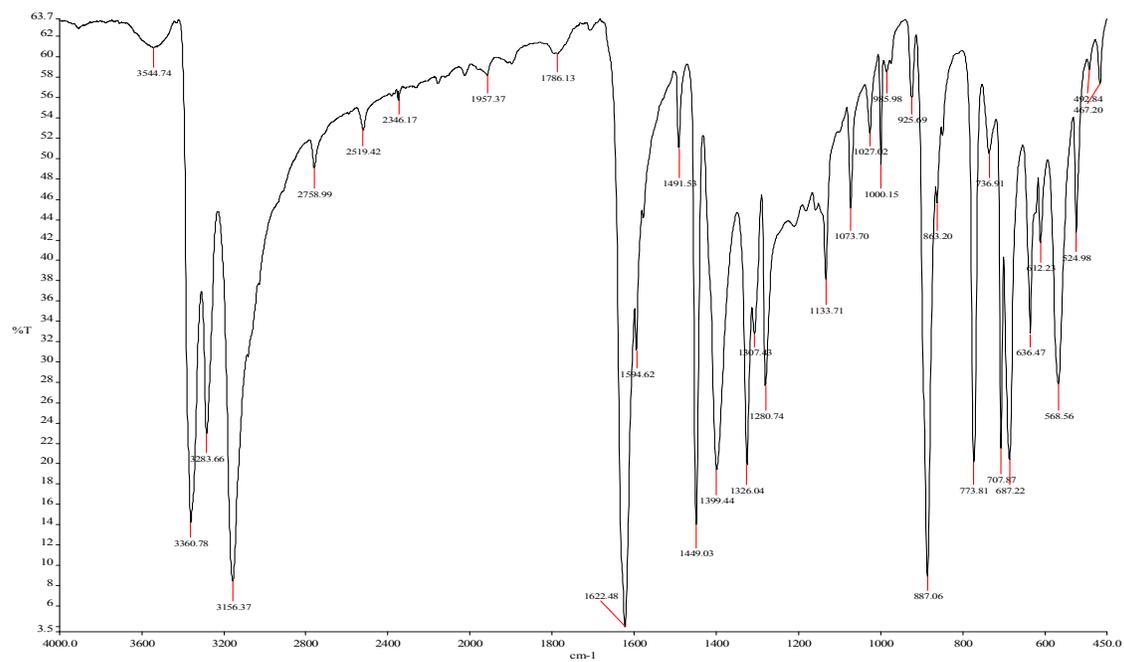
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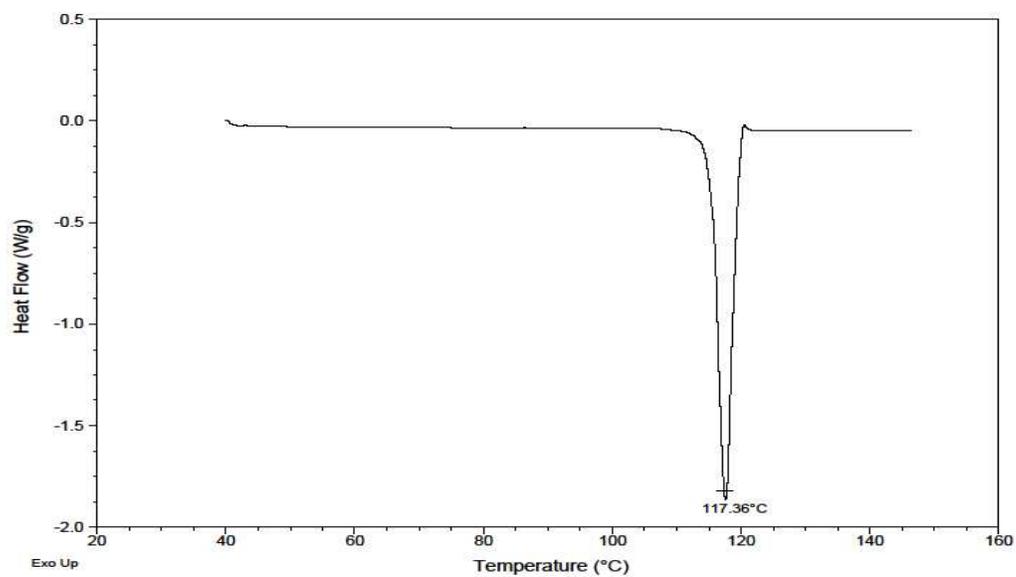
Analytical Data and Figures for Thiobenzamide, 1



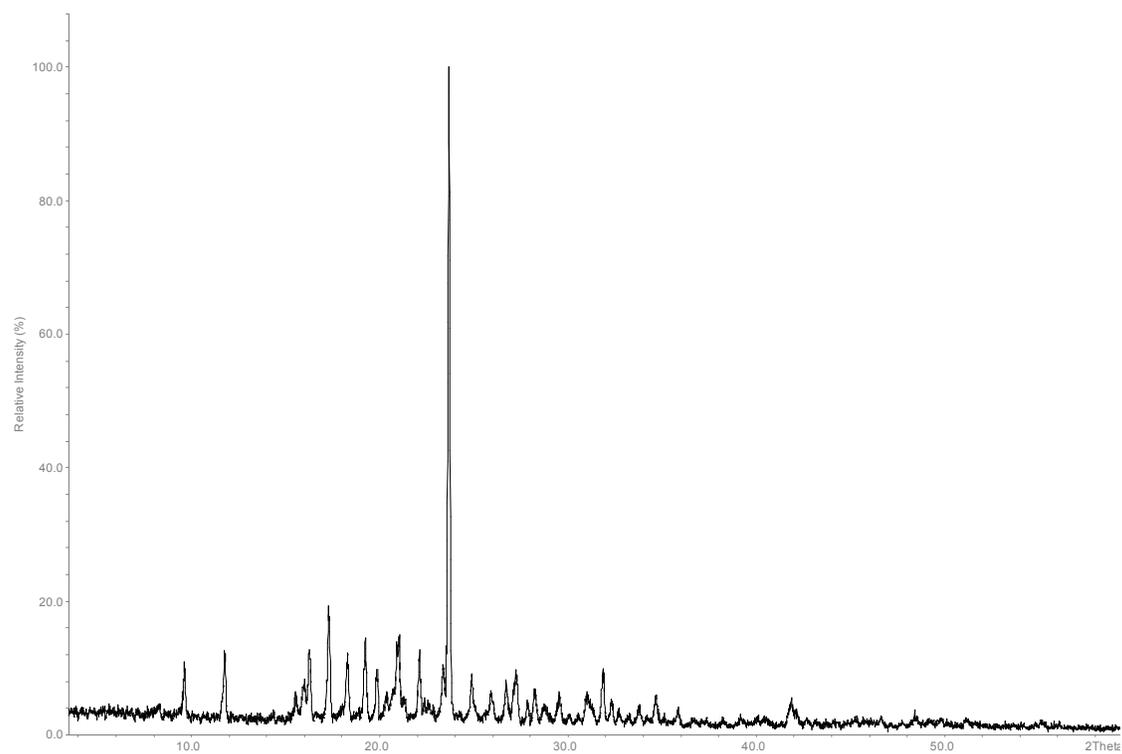
IR spectra for 1.



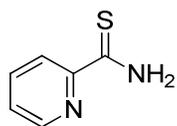
DSC of 1 obtained by solution crystallization (onset temperature 116 °C)



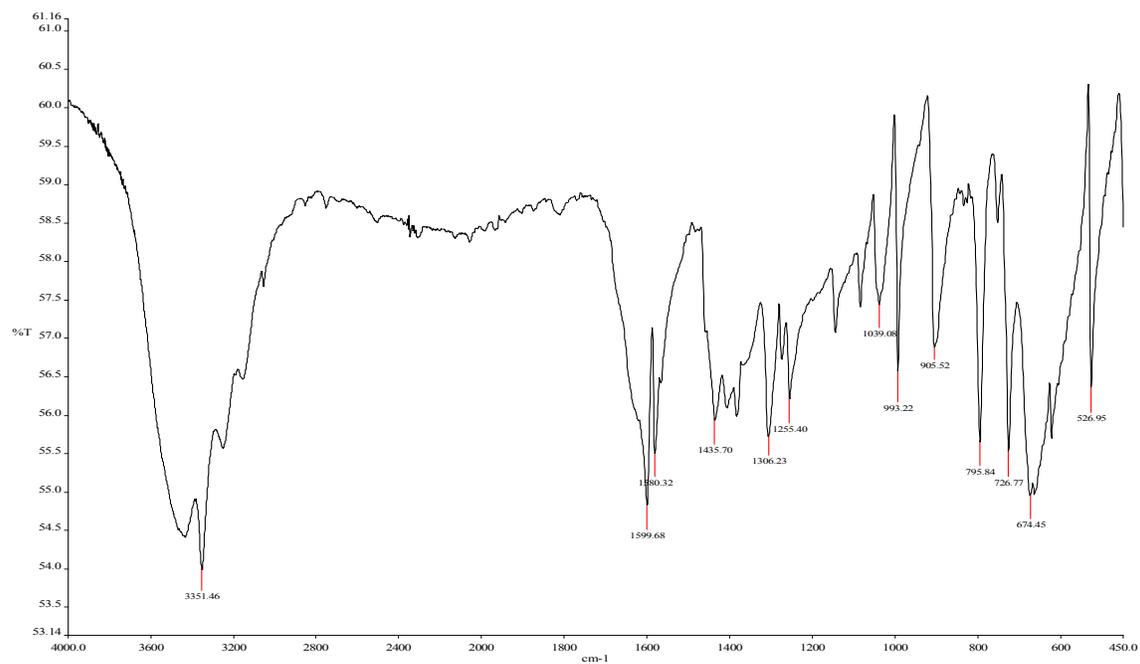
PXRD pattern of **1** obtained from solution crystallization (black)



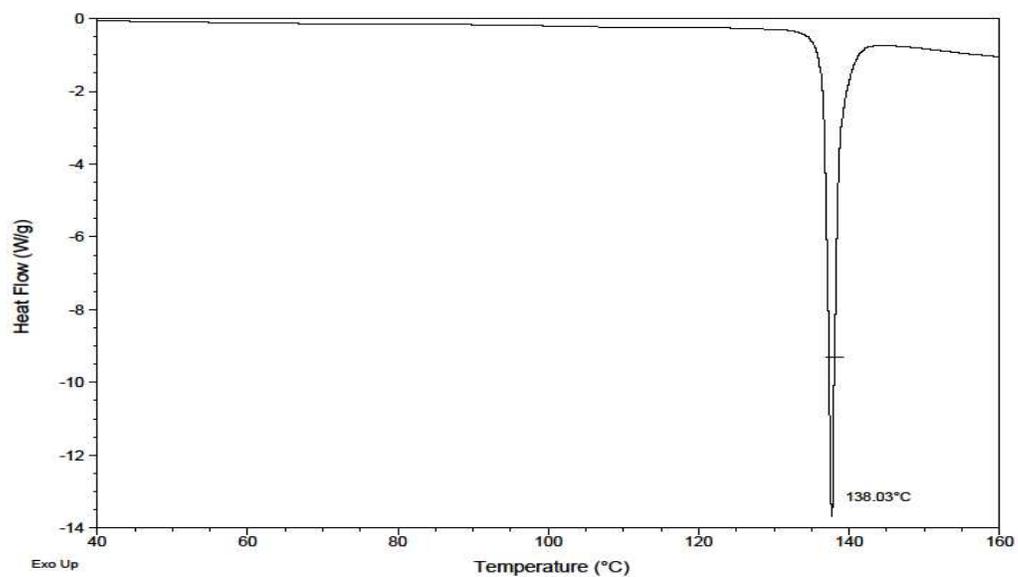
Analytical Data and Figures for Pyridine-2-carbothioamide, 2



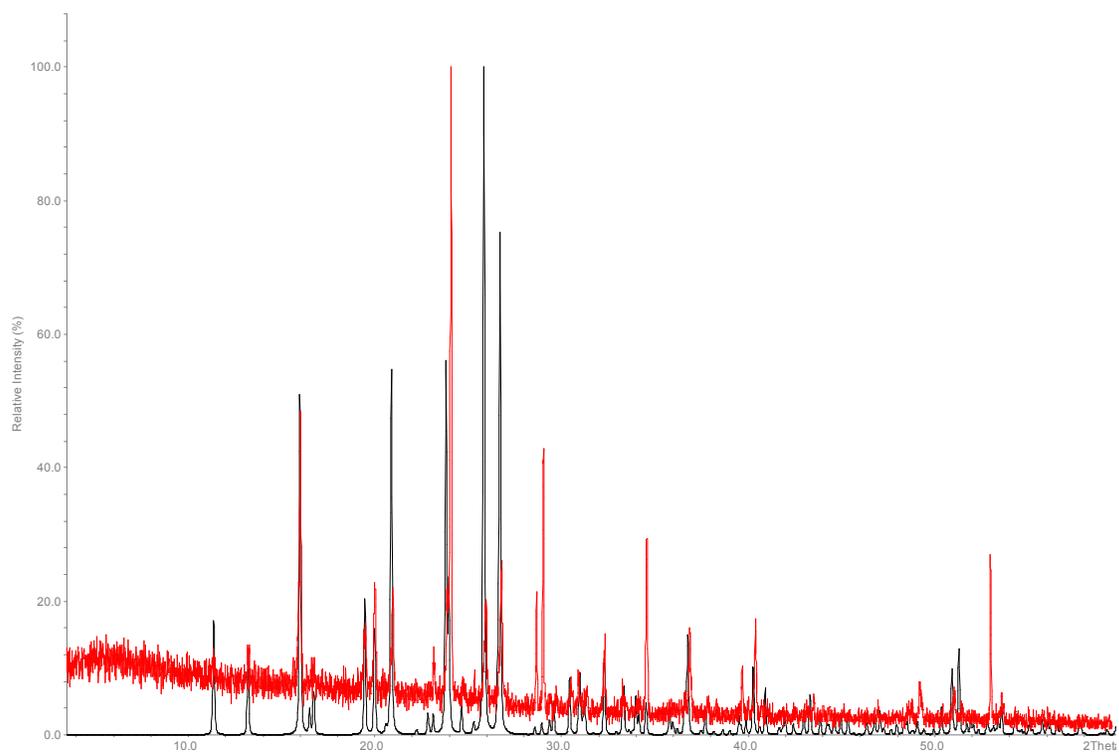
IR spectra for 2.



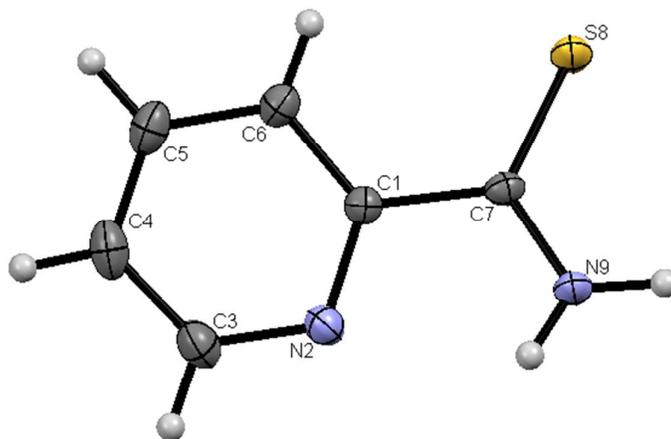
DSC of 2 obtained by solution crystallization (onset temperature 136 °C)



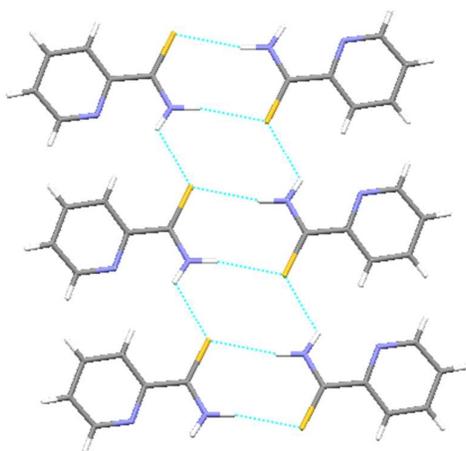
PXRD pattern of **2** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



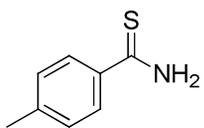
ORTEP plot of **2** (ellipsoids shown at 30% probability).



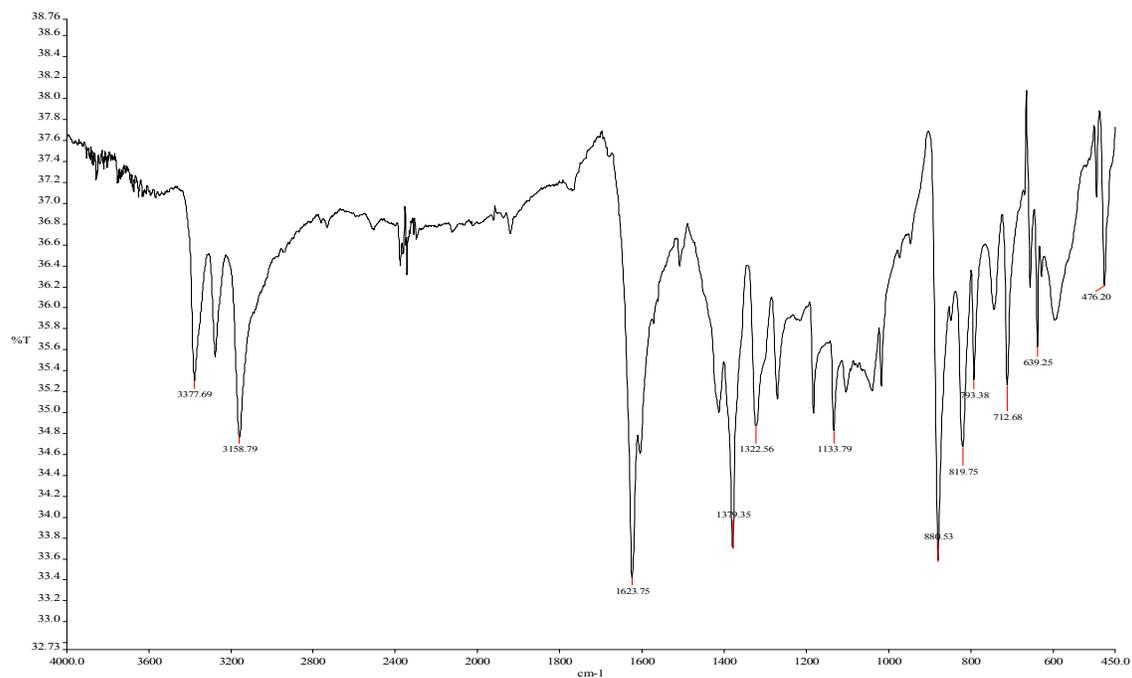
Packing Diagram for 2



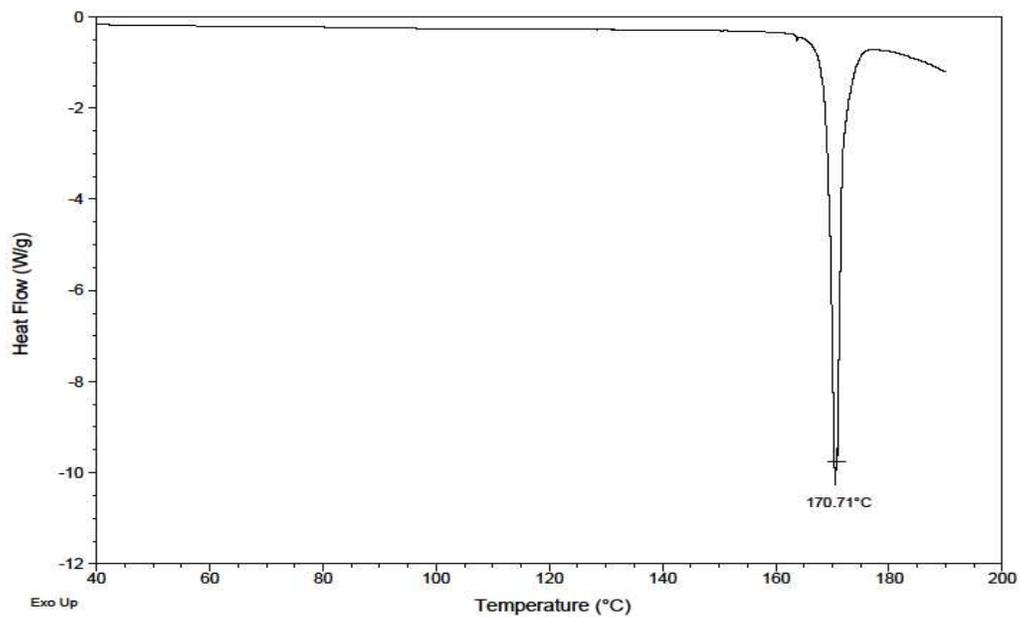
Analytical Data and Figures for 4-Methylthiobenzamide, **3**



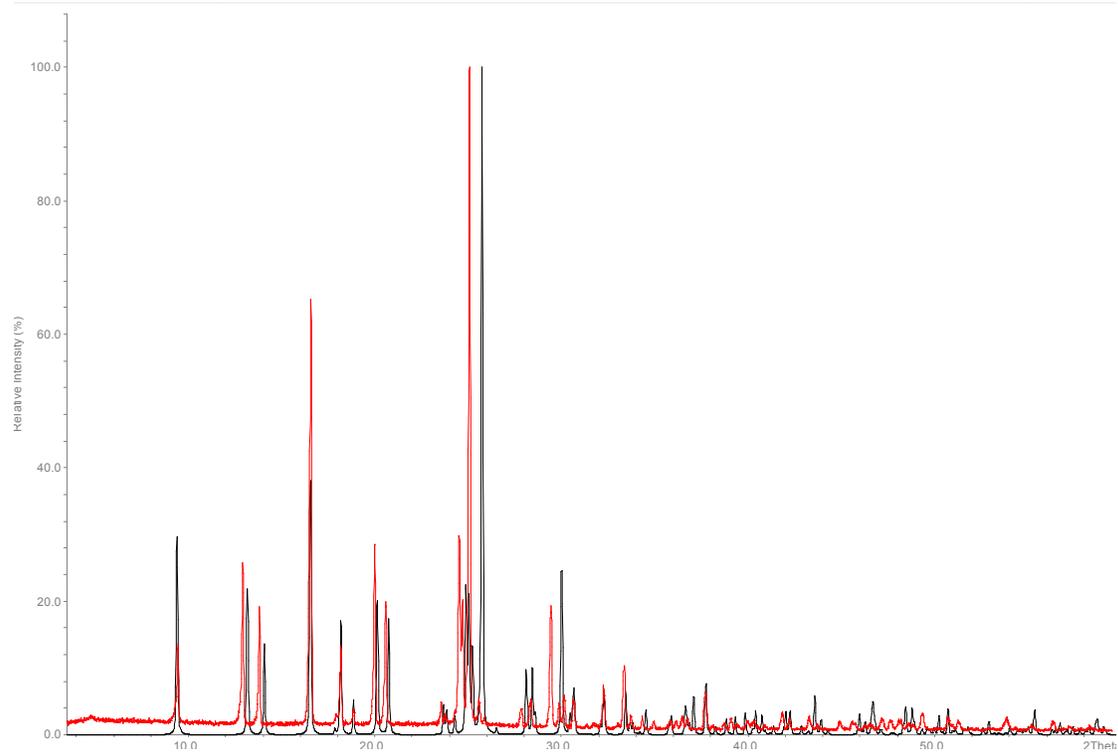
IR spectra for **3**.



DSC of **3** obtained by solution crystallization (onset temperature 168 °C)



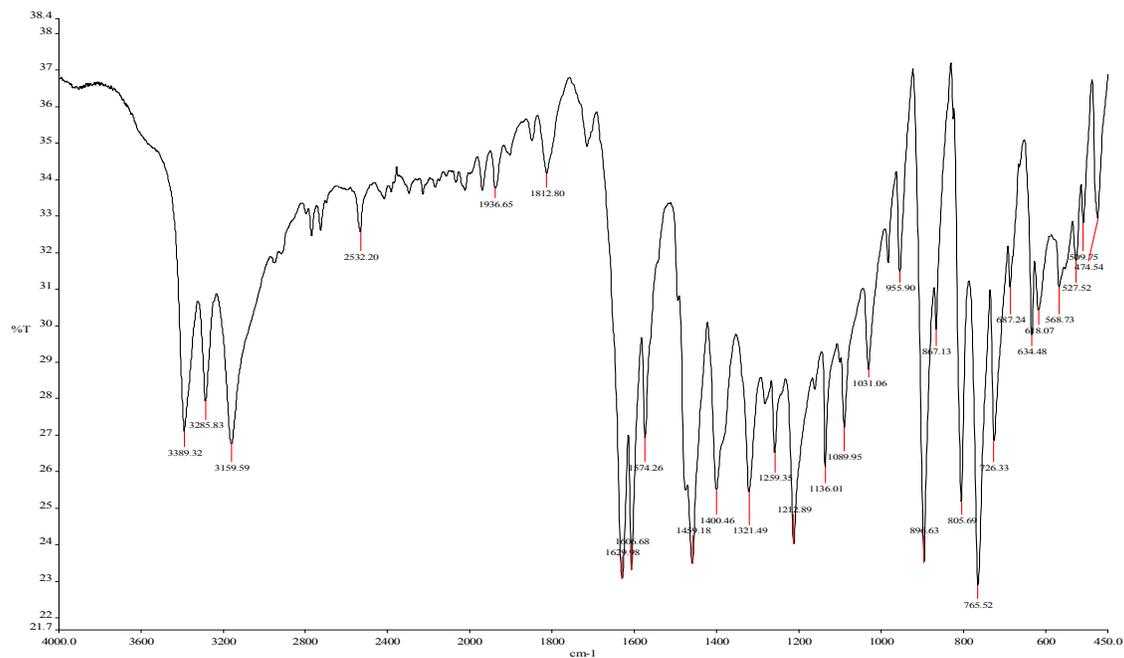
PXRD pattern of **3** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



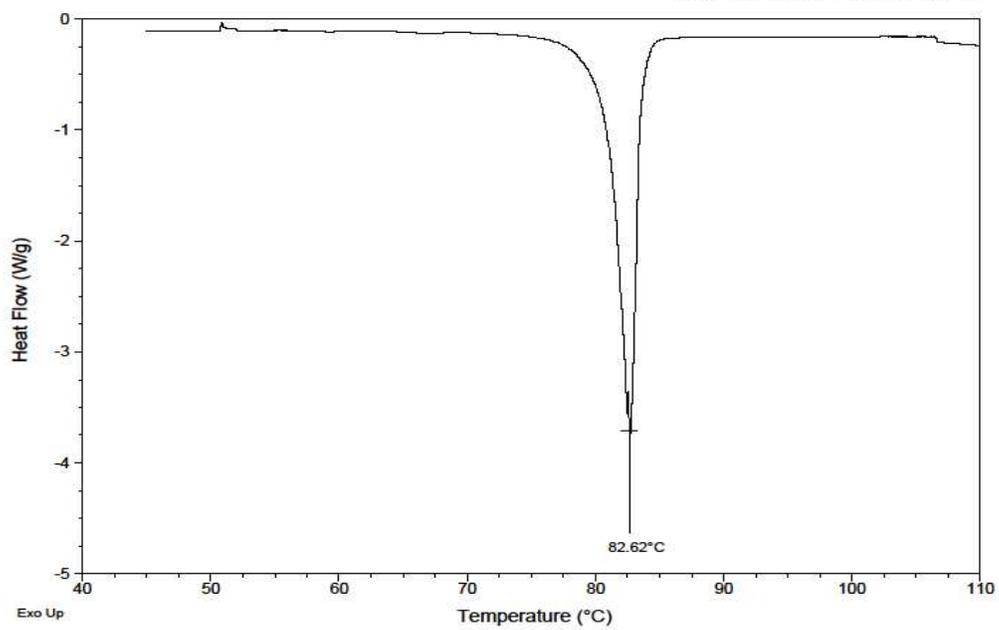
Analytical Data and Figures for 2-Fluorothiobenzamide, 4



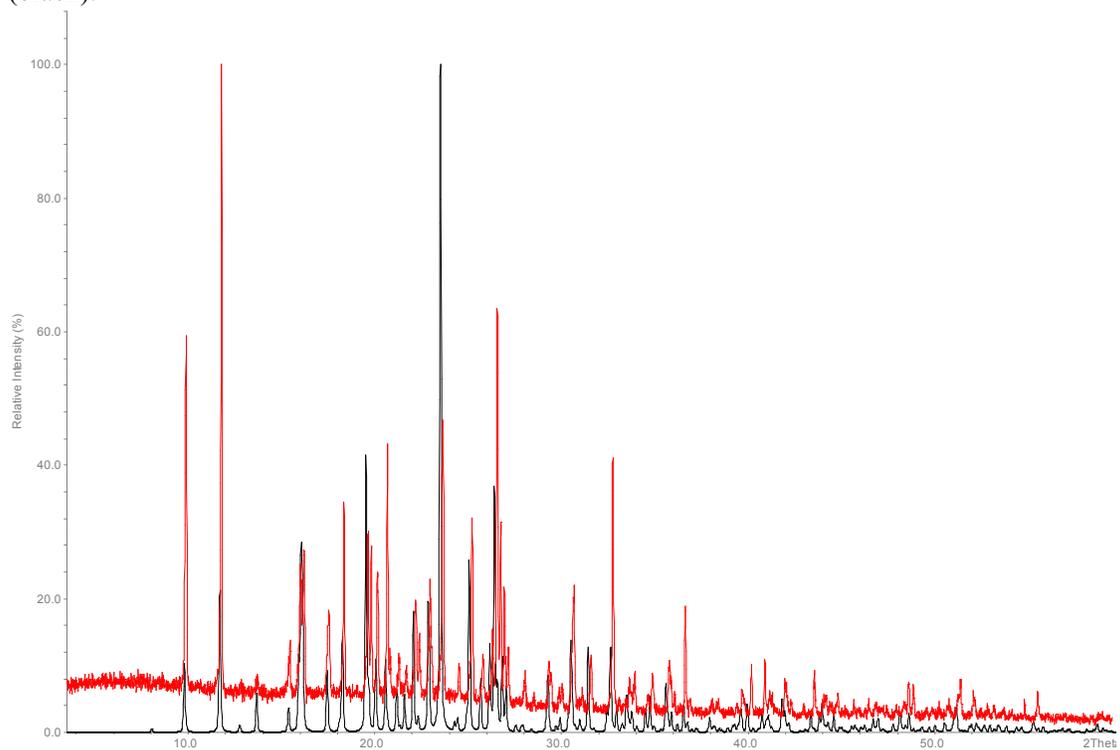
IR spectra for 4.



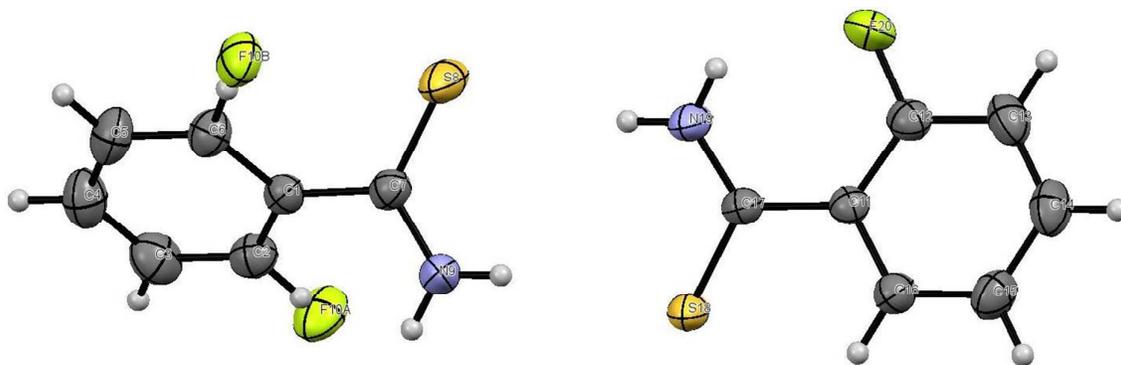
DSC of 4 obtained by solution crystallization (onset temperature 81 °C)



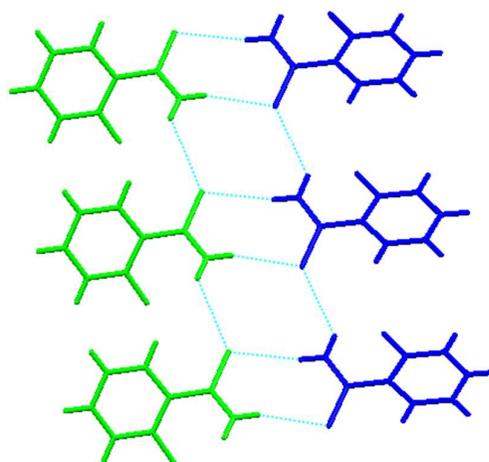
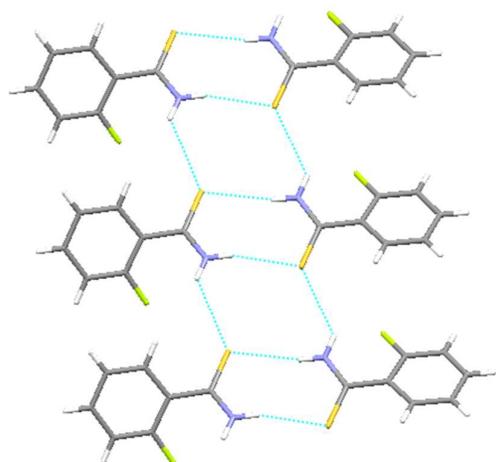
PXRD pattern of **4** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



ORTEP plot of **4** (ellipsoids shown at 30% probability).



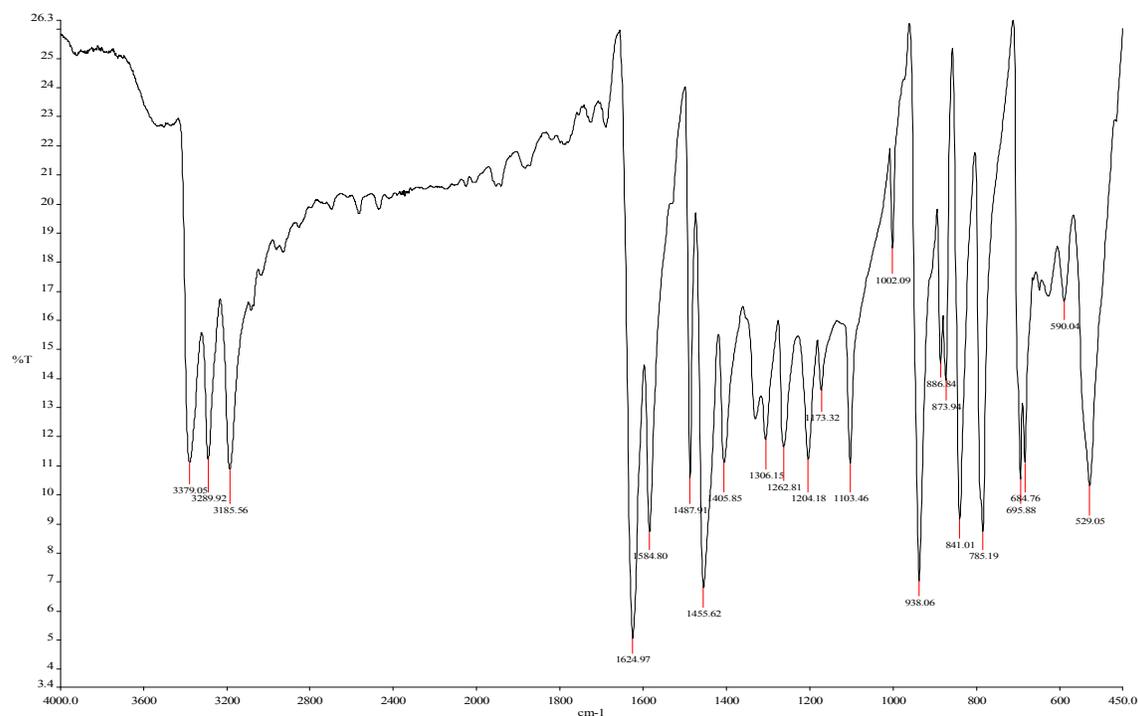
Packing Diagram for 4



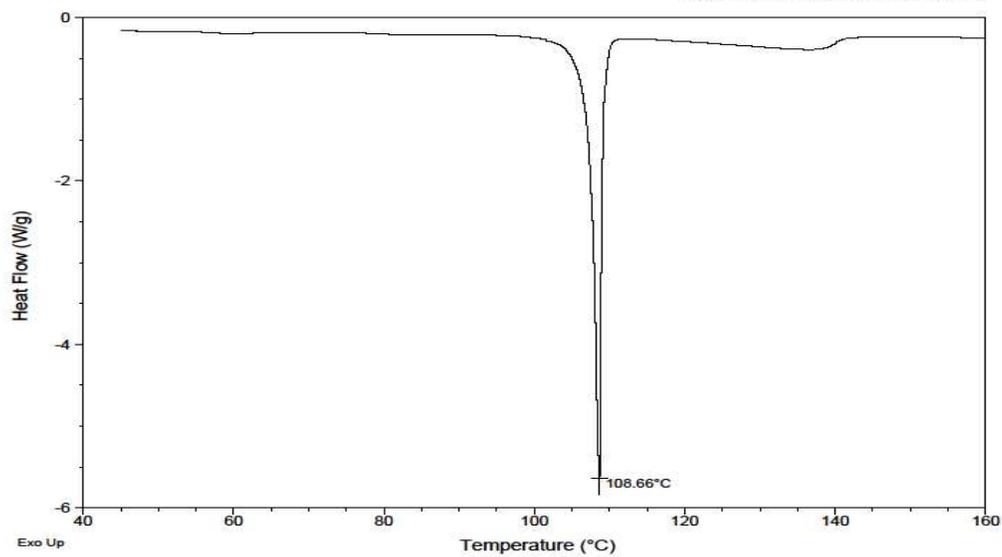
Analytical Data and Figures for 3-Fluorothiobenzamide, **5**



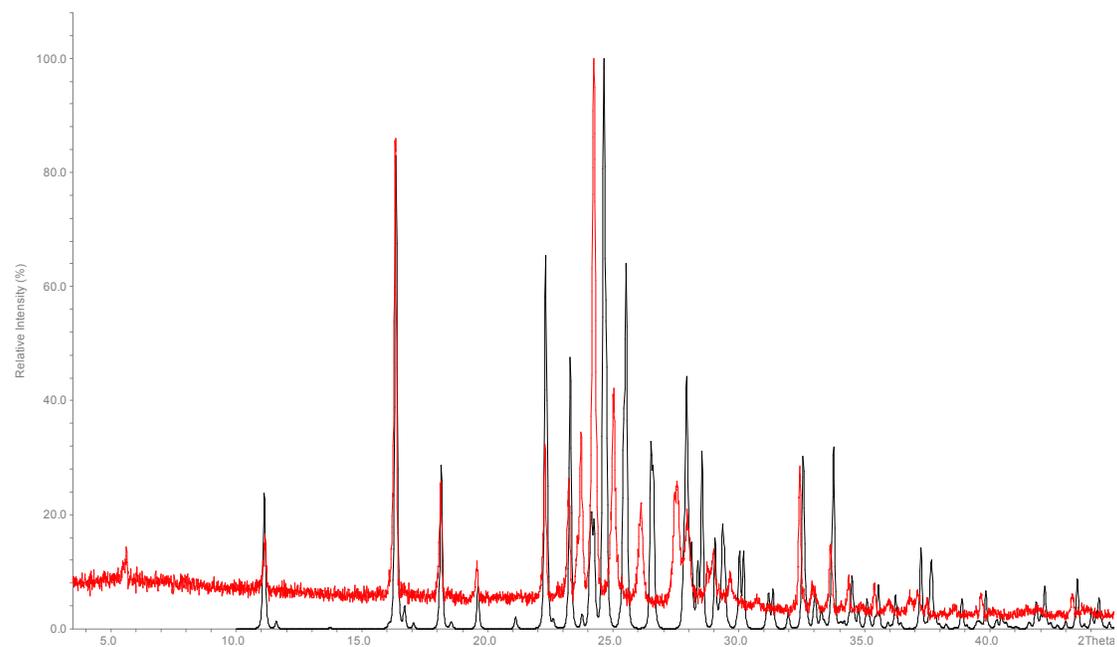
IR spectra for **5**.



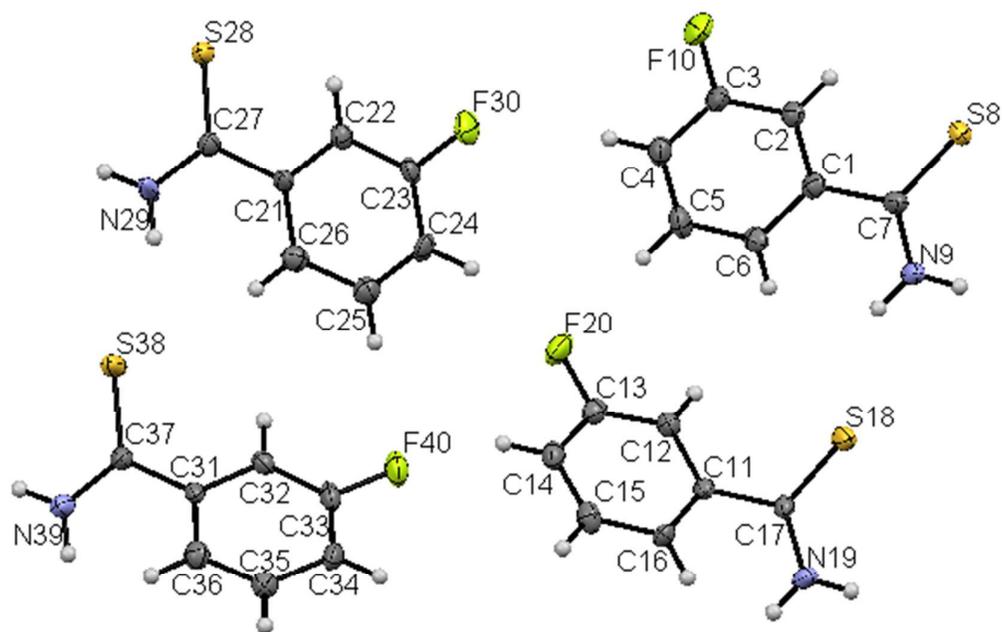
DSC of **5** obtained by solution crystallization (onset temperature 107 °C)



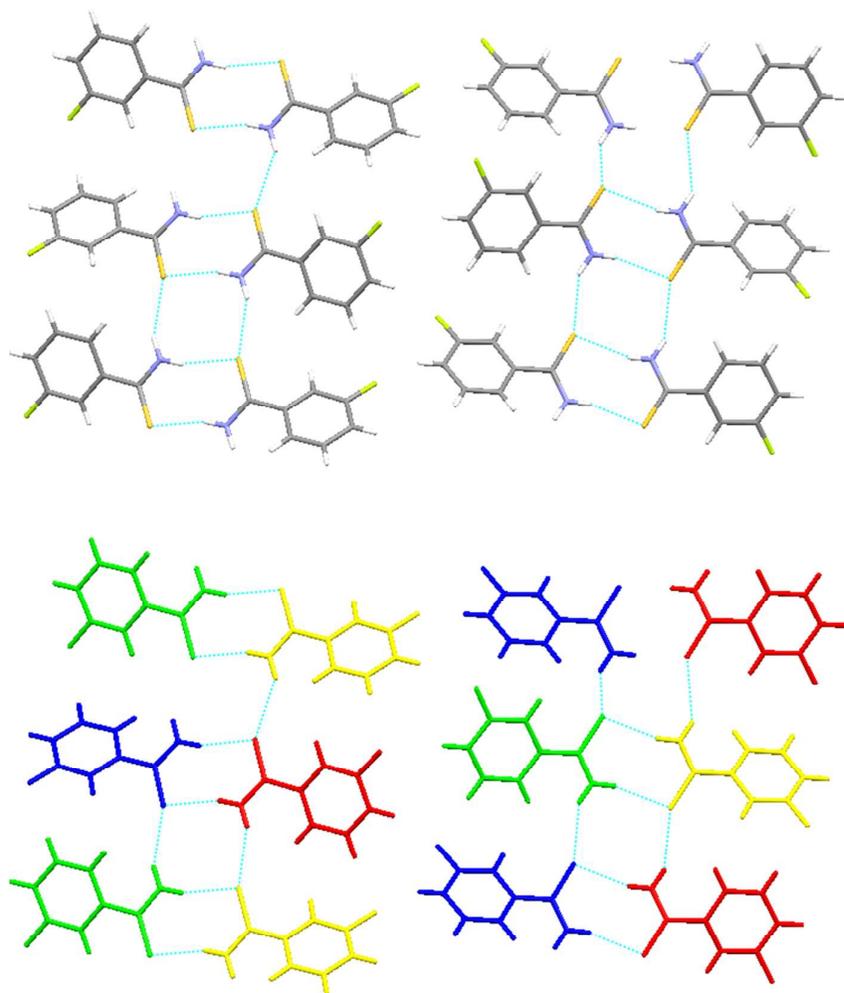
PXRD pattern of **5** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



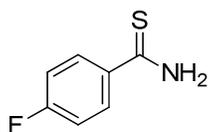
ORTEP plot of **5** (ellipsoids shown at 50% probability).



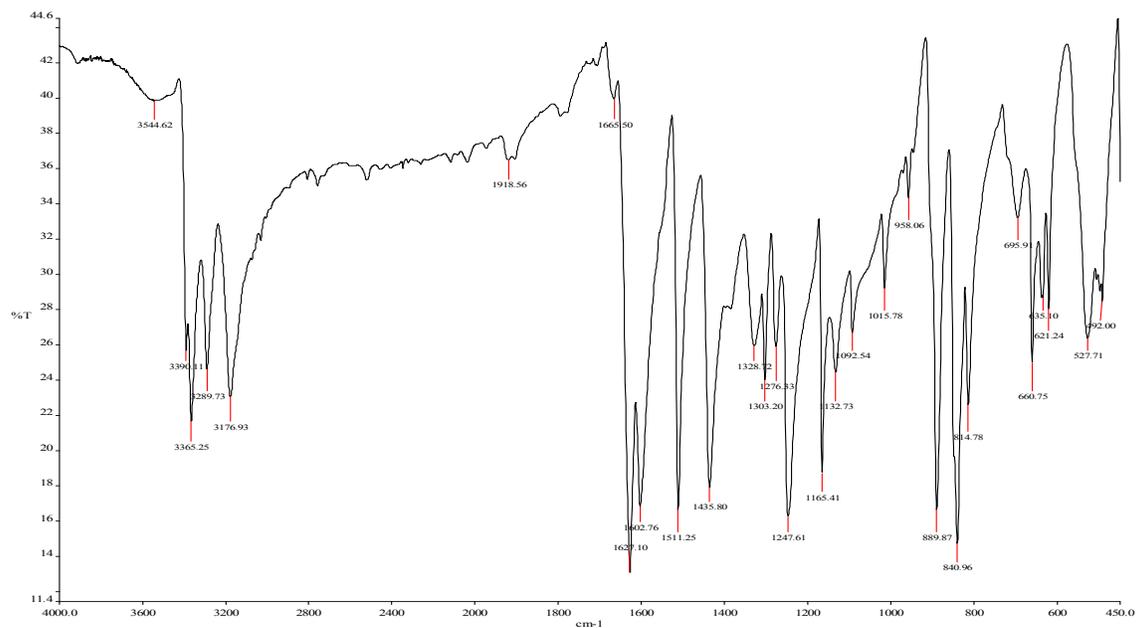
Packing Diagram for 5



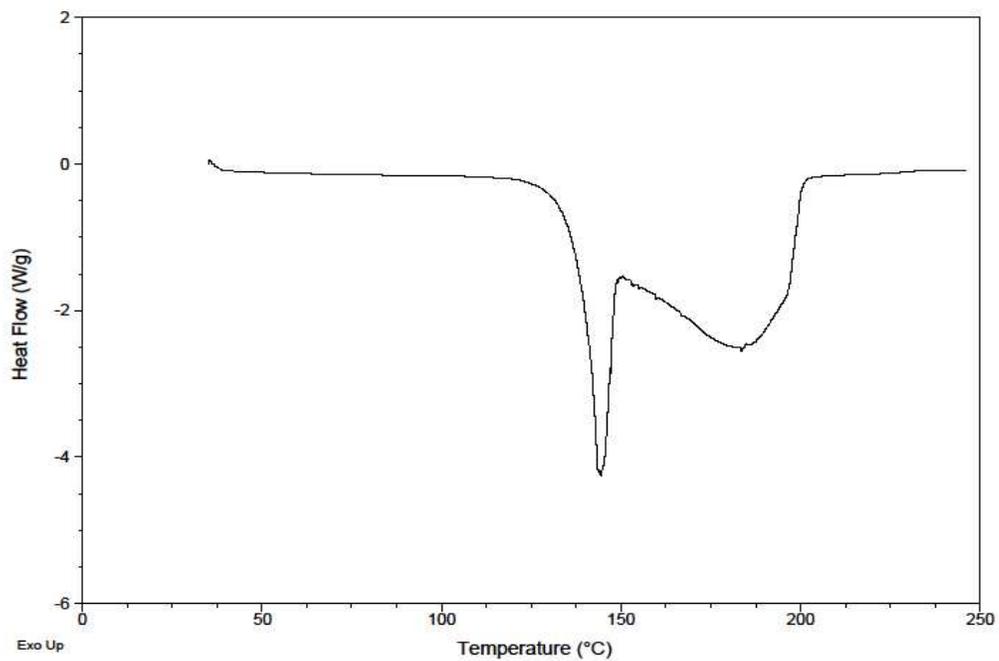
Analytical Data and Figures for 4-Fluorothiobenzamide, 6



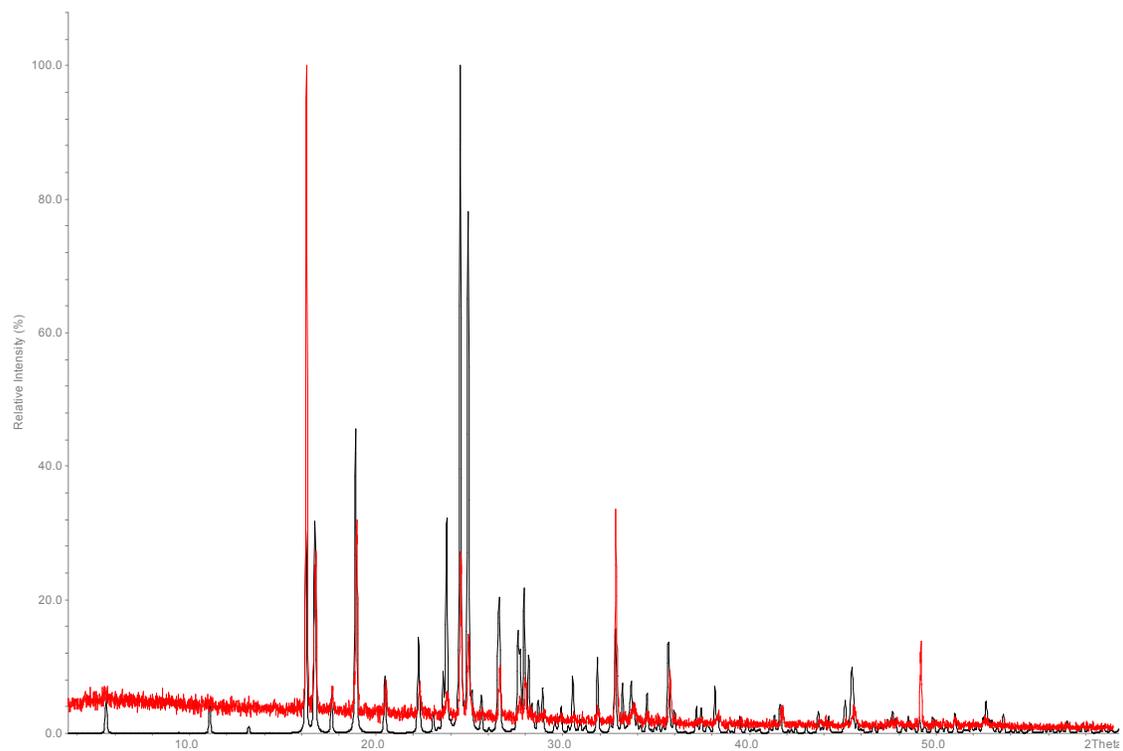
IR spectra for 6.



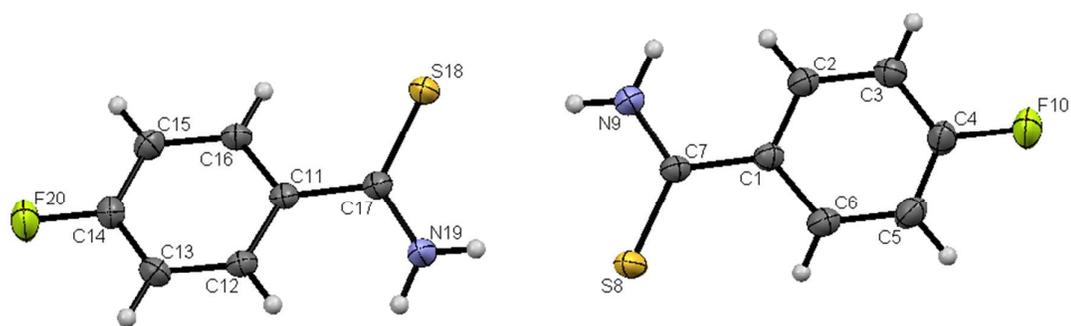
DSC of 6 obtained by solution crystallization (onset temperature 139 °C)



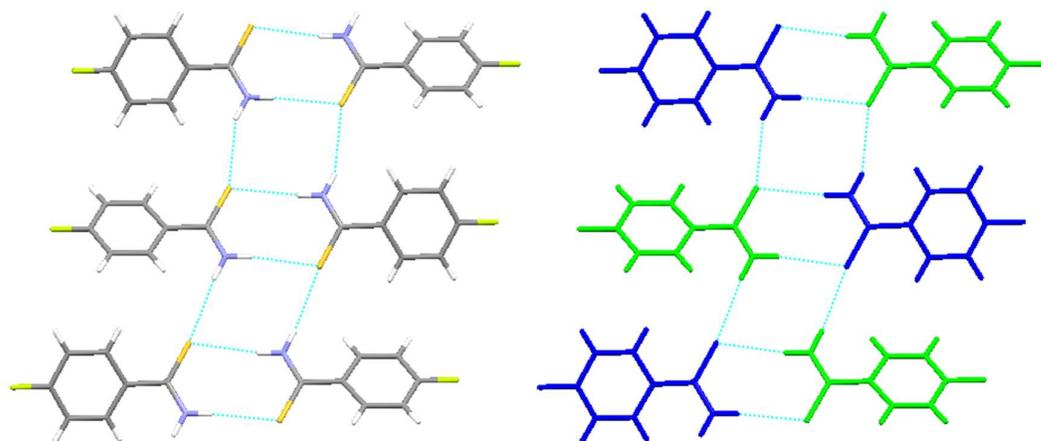
PXRD pattern of **6** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



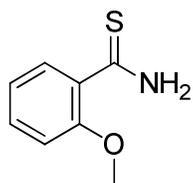
ORTEP plot of **6** (ellipsoids shown at 30% probability).



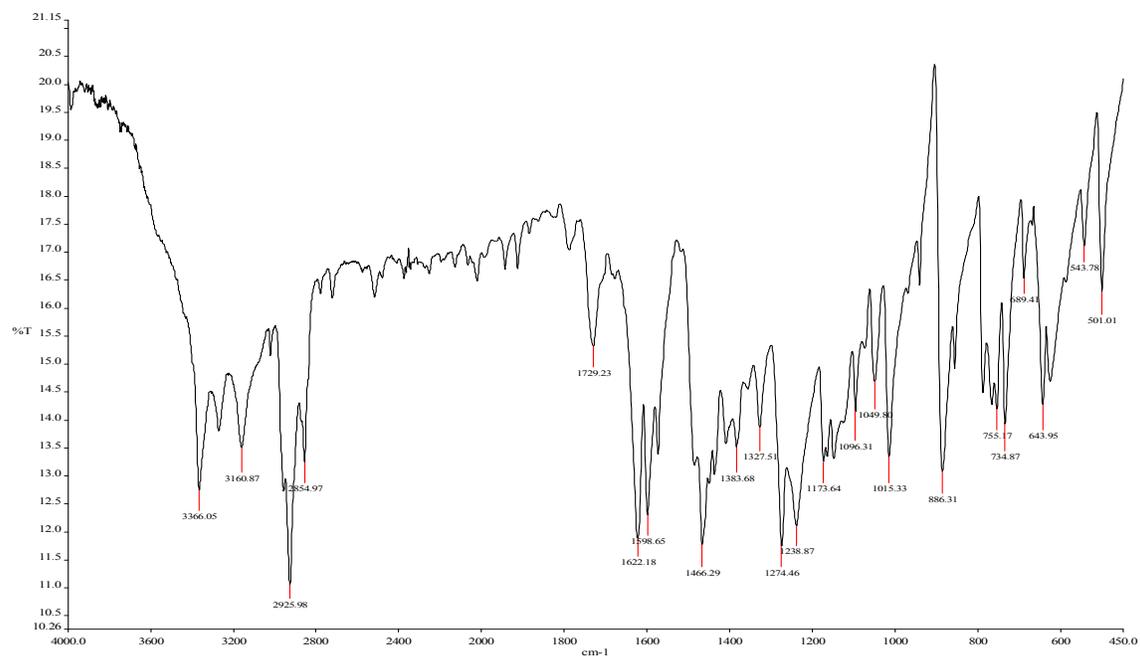
Packing Diagram for 6



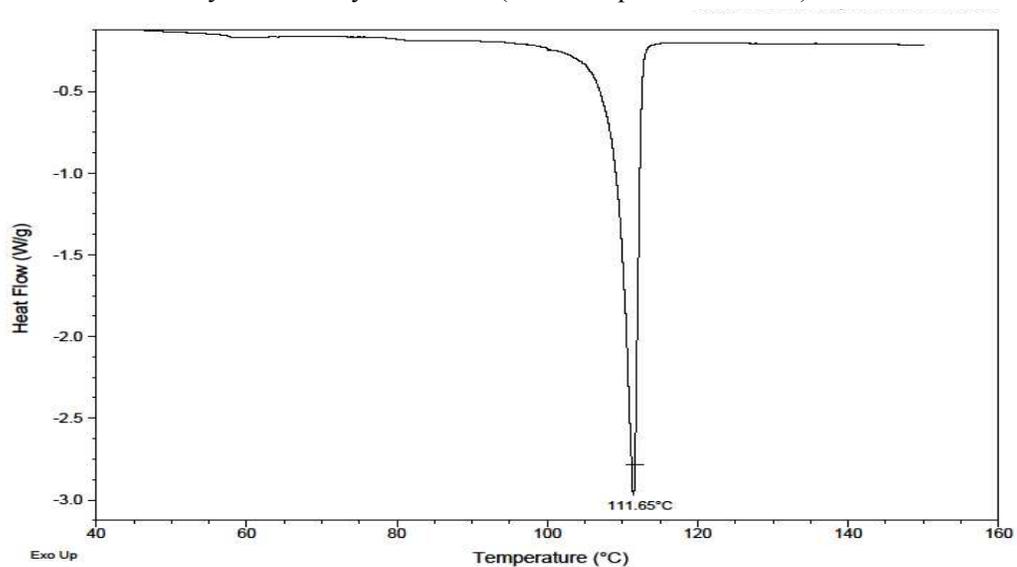
Analytical Data and Figures for 2-Methoxythiobenzamide, 7



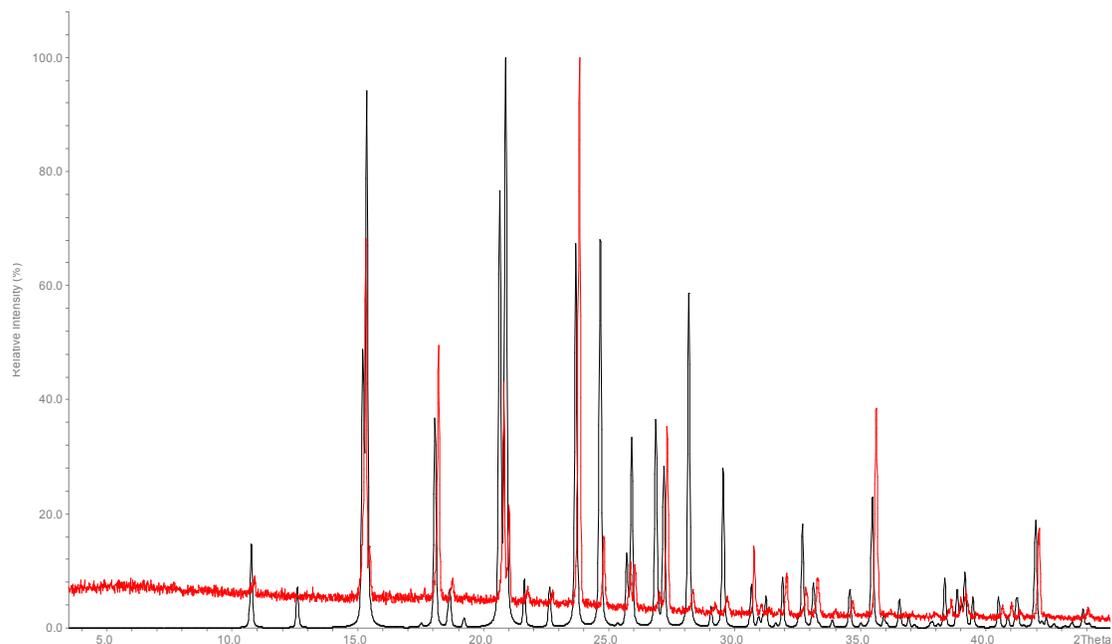
IR spectra for 7.



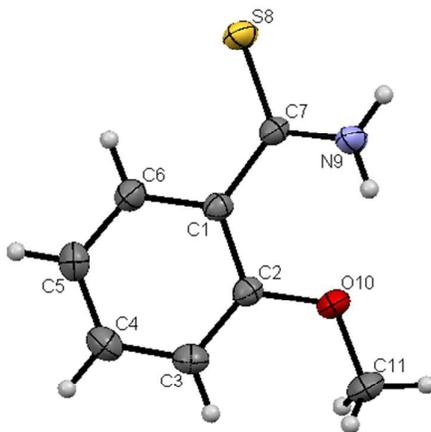
DSC of 7 obtained by solution crystallization (onset temperature 109 °C)



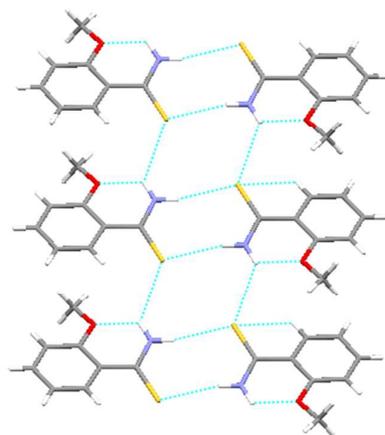
PXRD pattern of **7** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



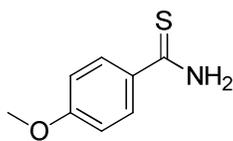
ORTEP plot of **7**(ellipsoids shown at 30% probability).



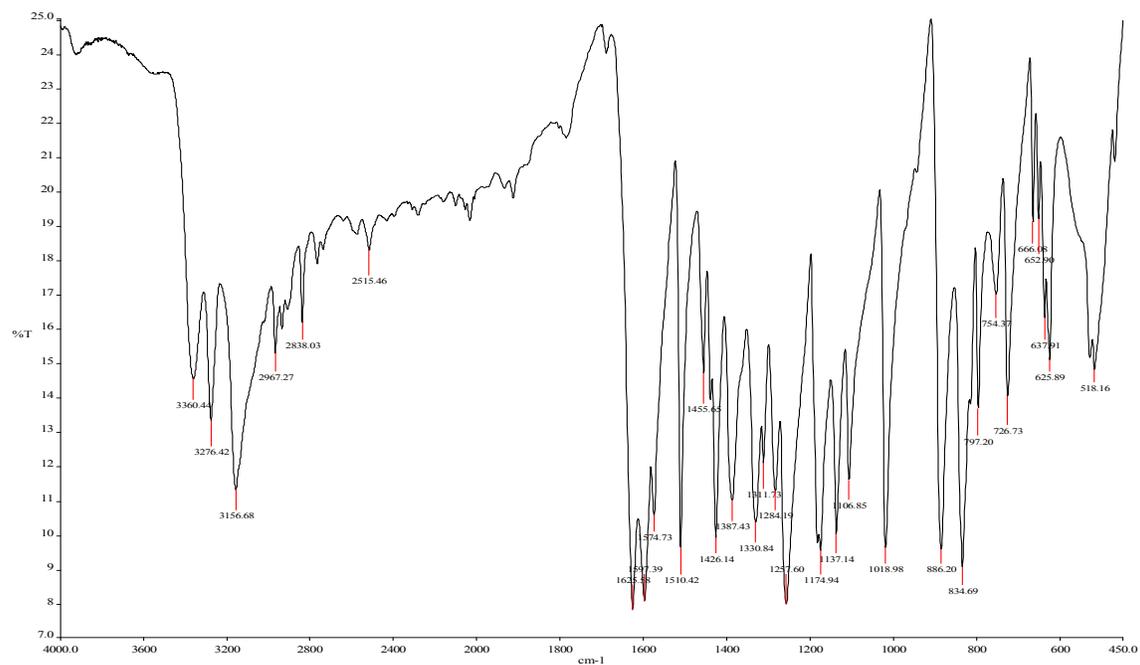
Packing Diagram for 7



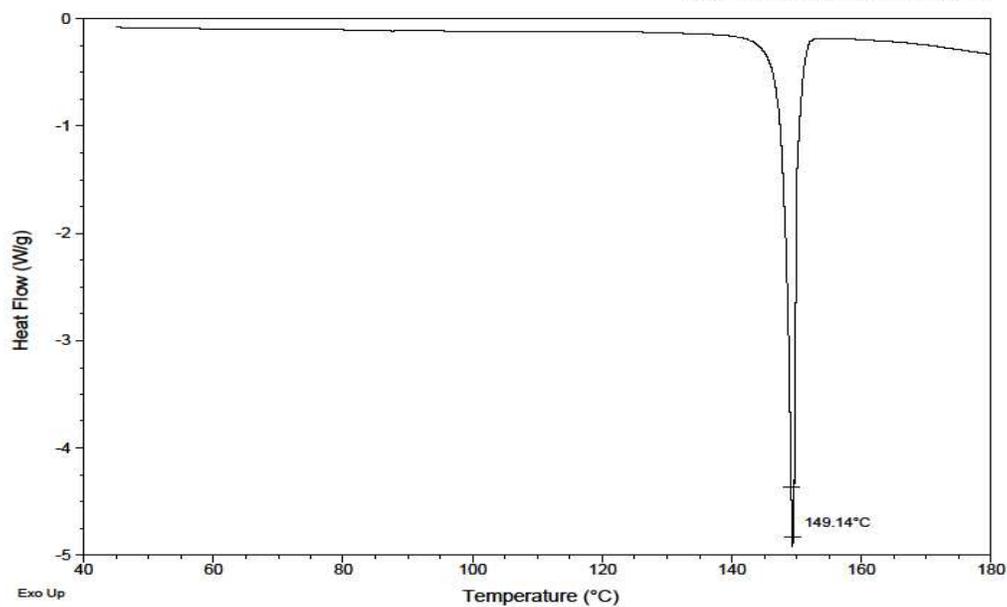
Analytical Data and Figures for 4-Methoxythiobenzamide, **8**



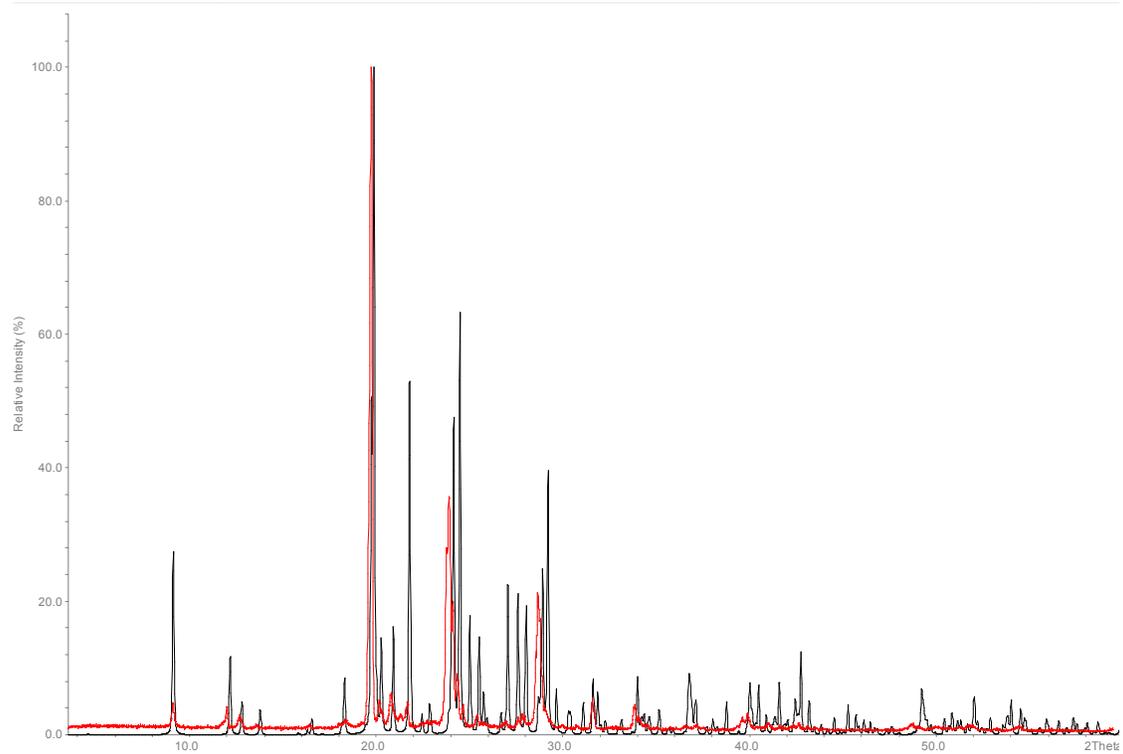
IR spectra for **8**.



DSC of **8** obtained by solution crystallization (onset temperature 147 °C)



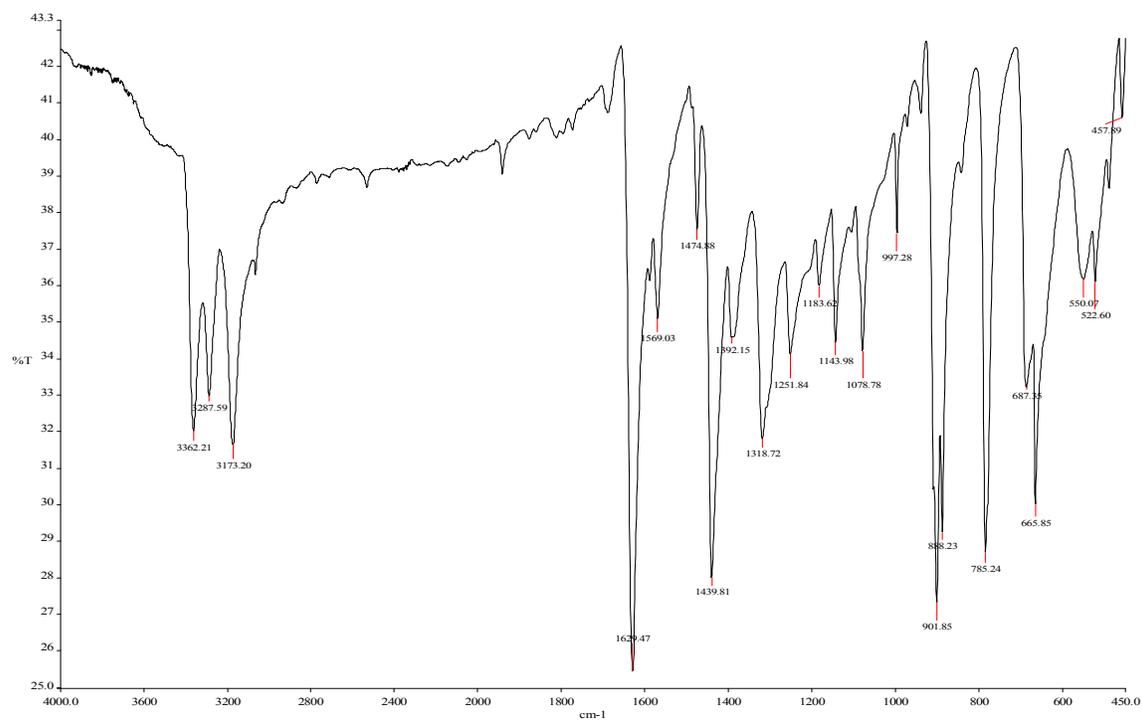
PXRD pattern of **8** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



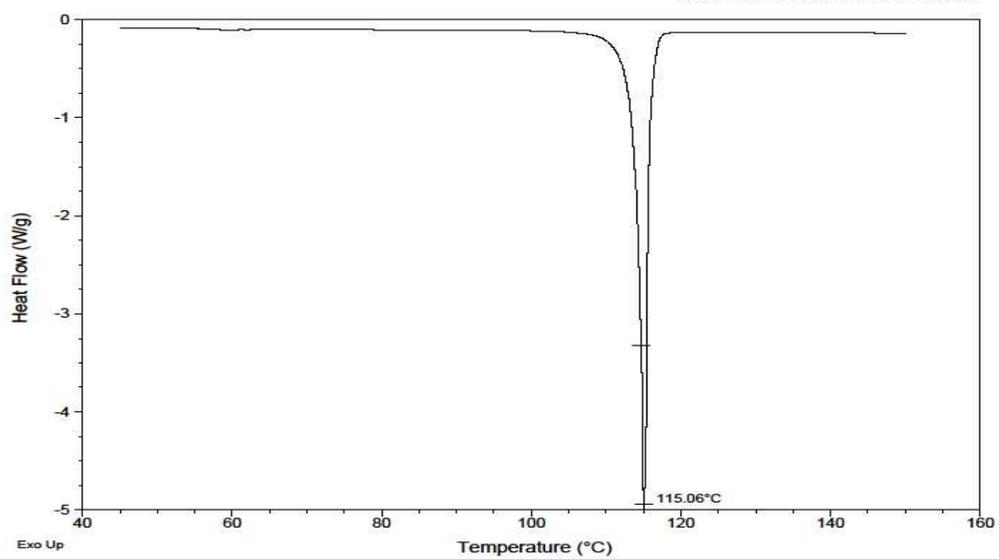
Analytical Data and Figures for 3-Chlorothiobenzamide, 9



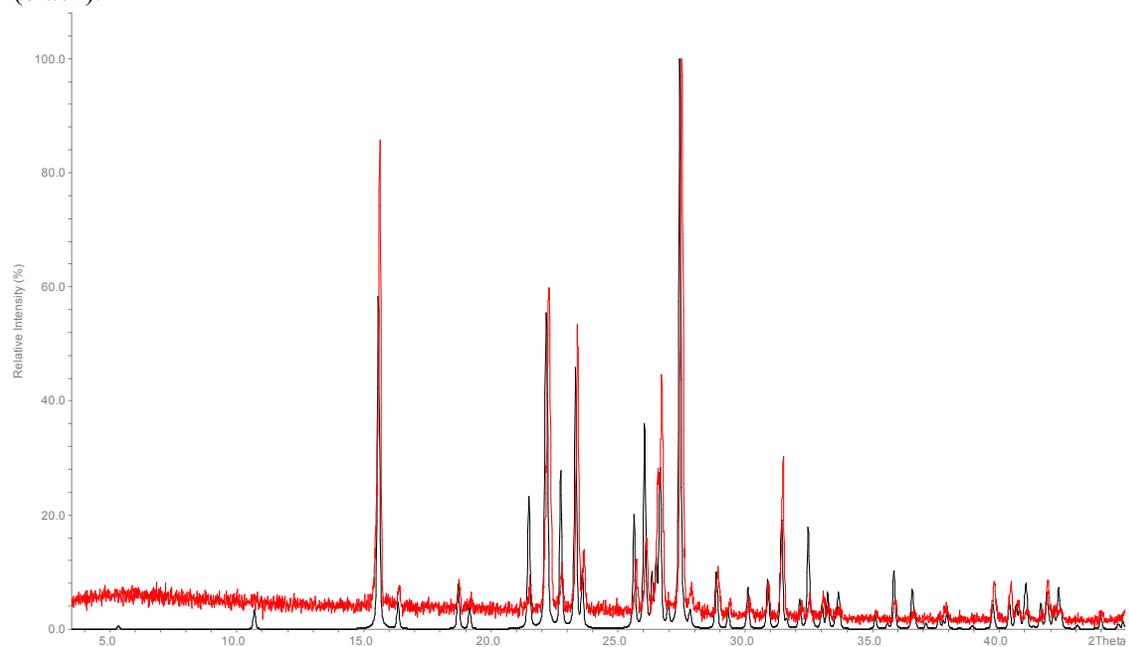
IR spectra for 9.



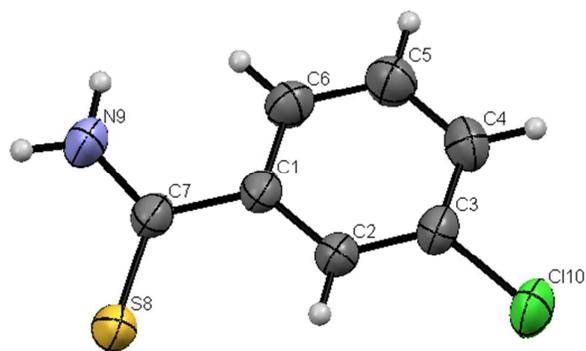
DSC of 9 obtained by solution crystallization (onset temperature 114 °C)



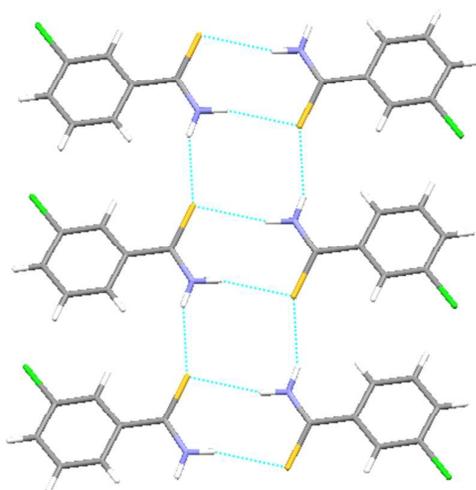
PXRD pattern of **9** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



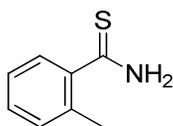
ORTEP plot of **9** (ellipsoids shown at 30% probability).



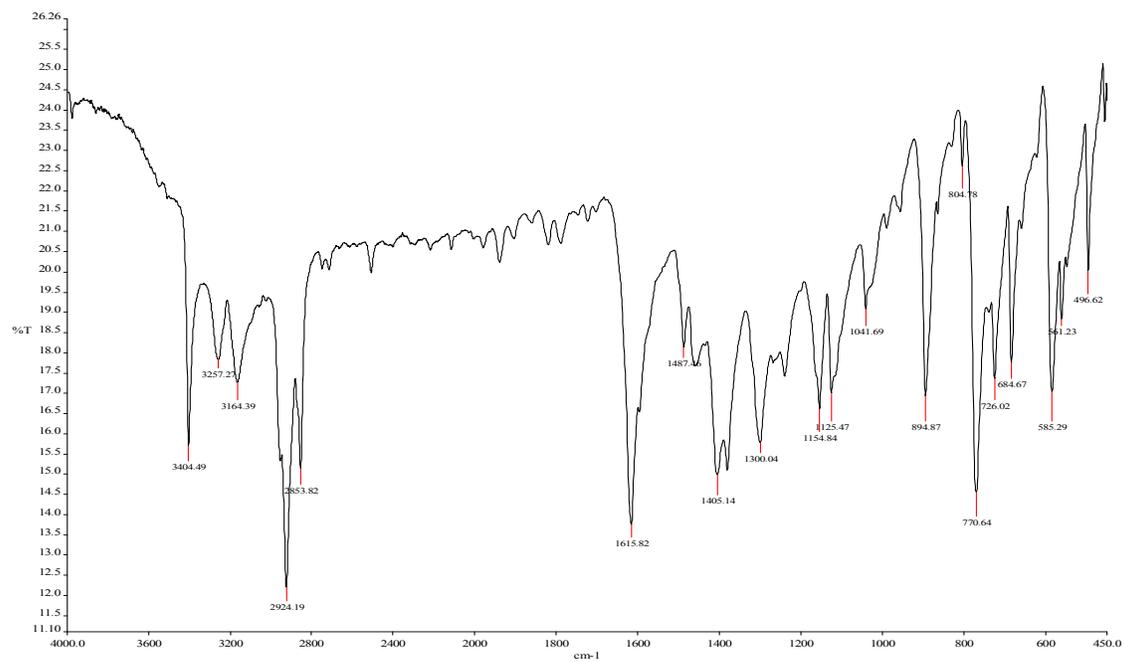
Packing Diagram for **9**



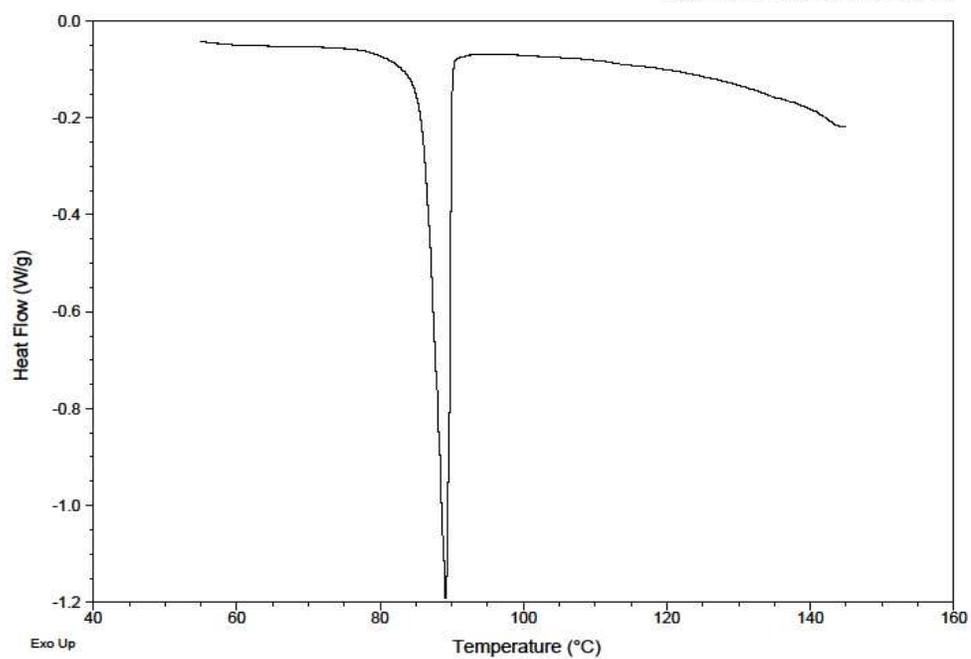
Analytical Data and Figures for 2-Methylthiobenzamide, 10



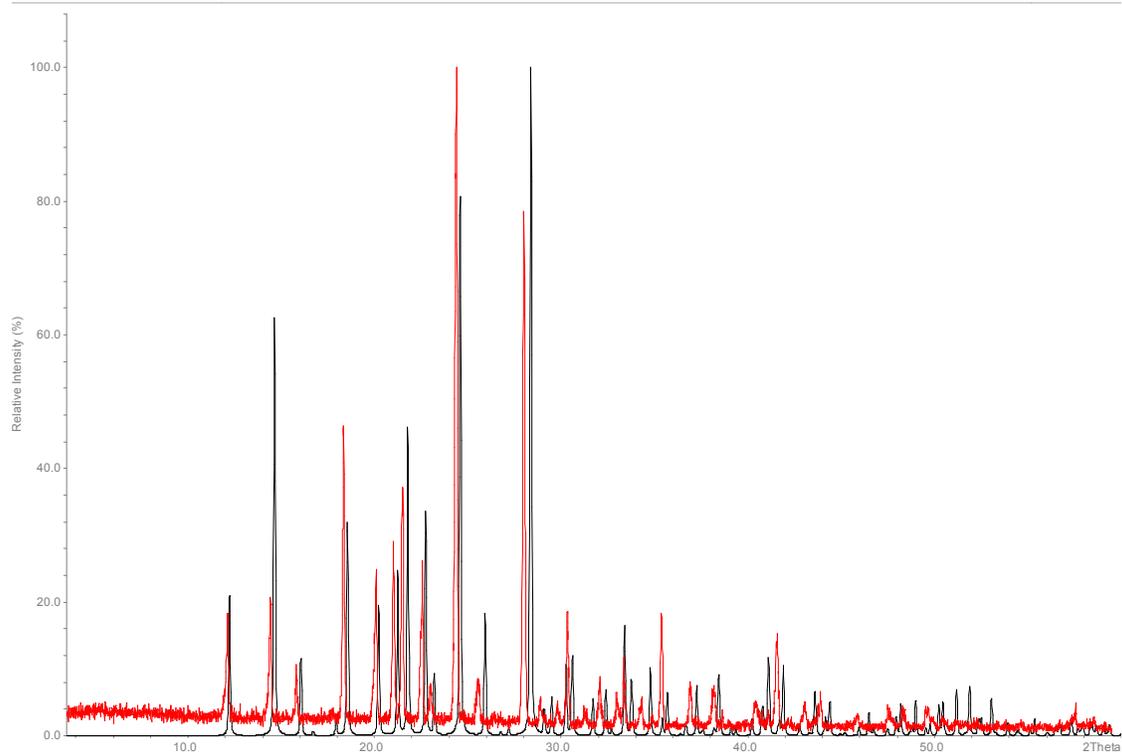
IR spectra for 10.



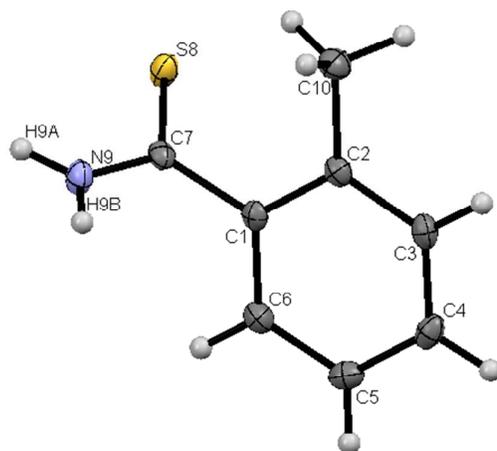
DSC of 10 obtained by solution crystallization (onset temperature 86 °C)



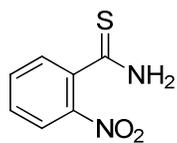
PXRD pattern of **10** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



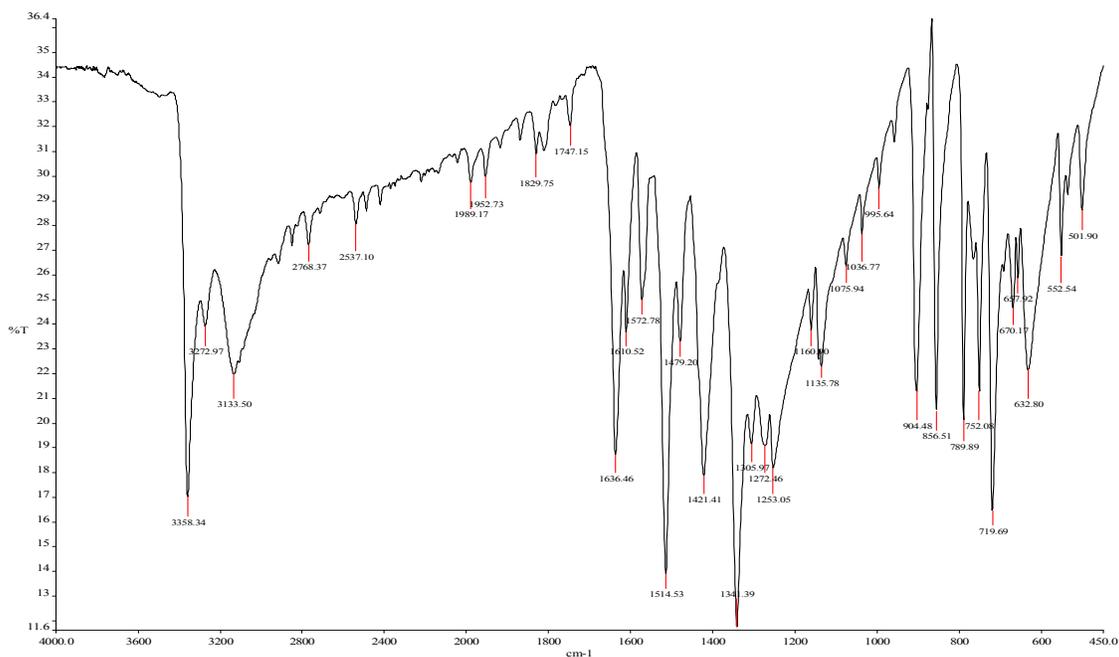
ORTEP plot of **10** (ellipsoids shown at 50% probability).



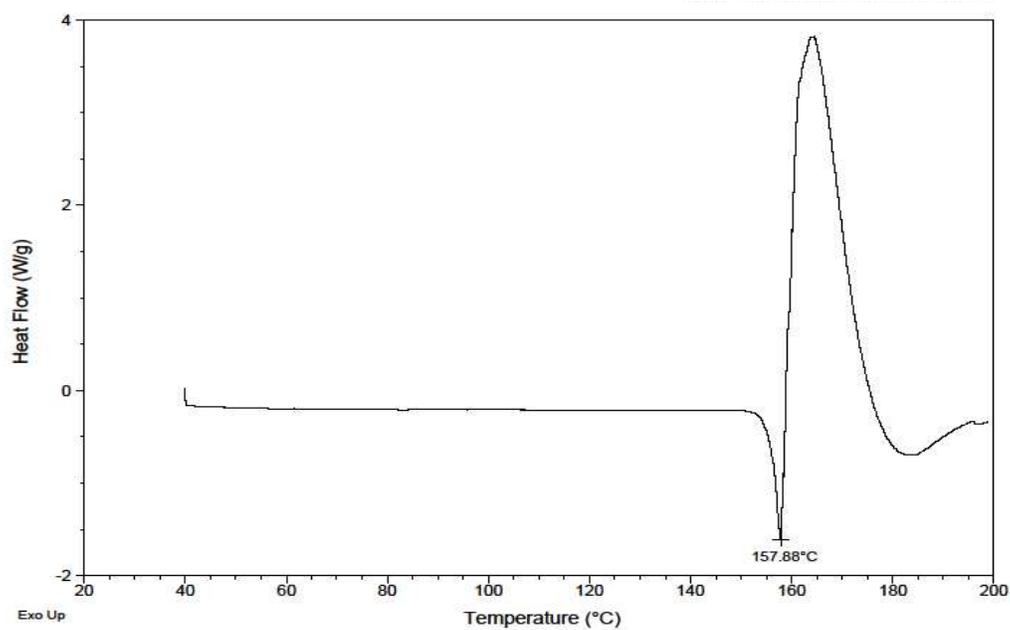
Analytical Data and Figures for 2-Nitrothiobenzamide, 11



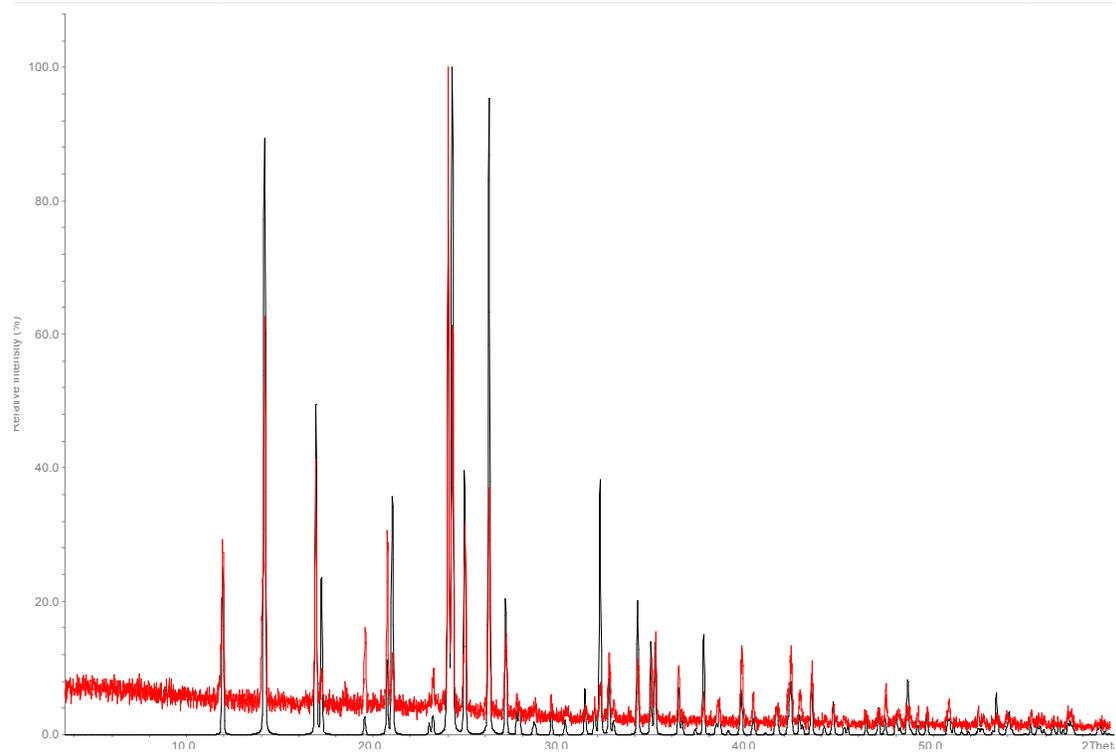
IR spectra for 11.



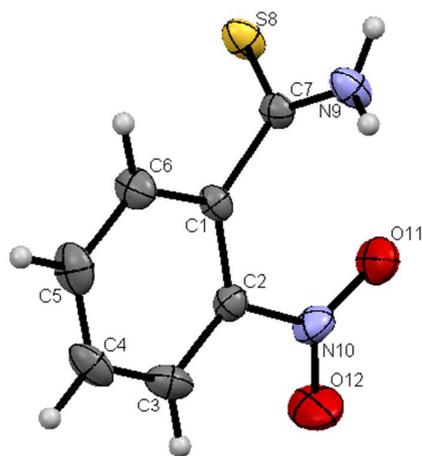
DSC of 11 obtained by solution crystallization (onset temperature 156 °C)



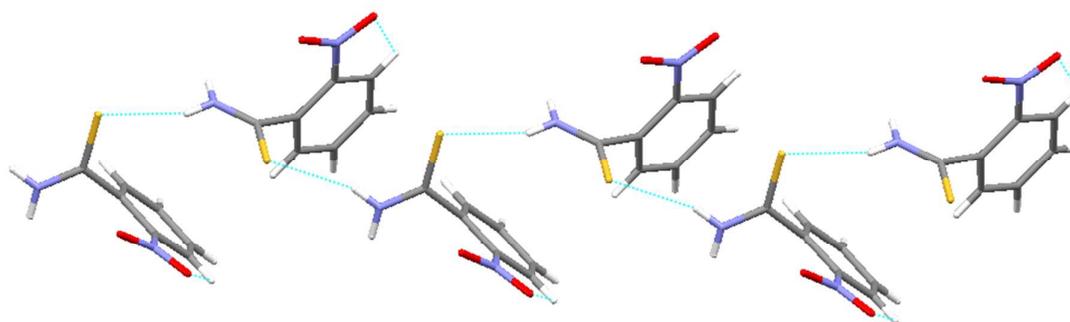
PXRD pattern of **11** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



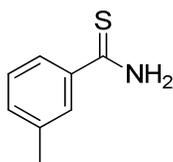
ORTEP plot of **11** (ellipsoids shown at 30% probability).



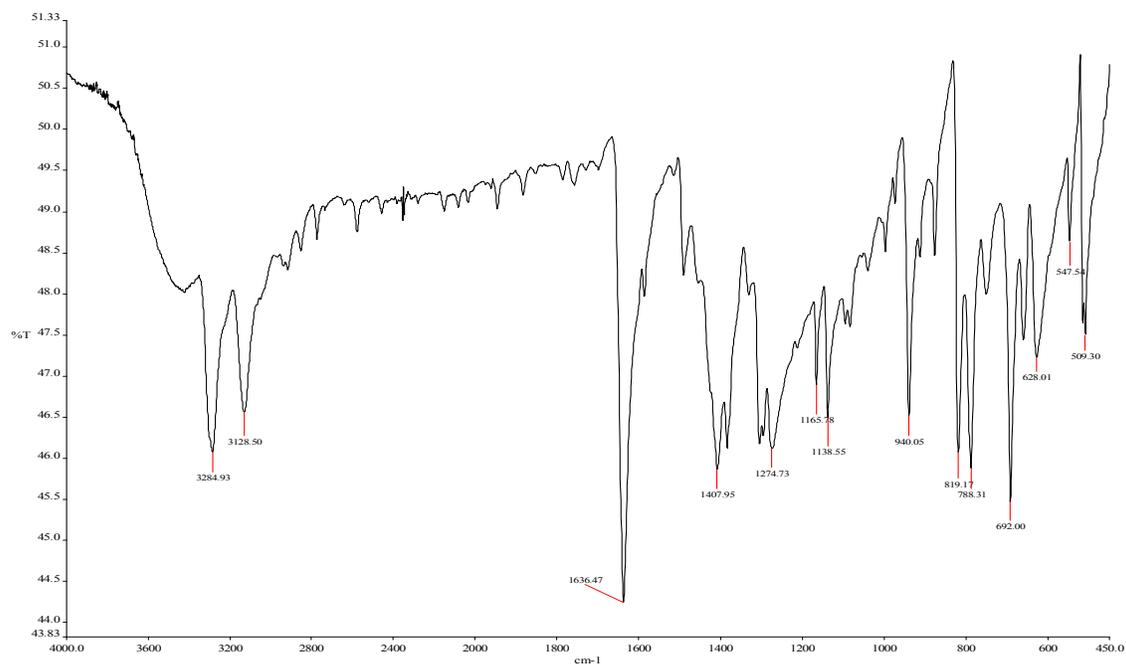
Packing Diagram for **11**



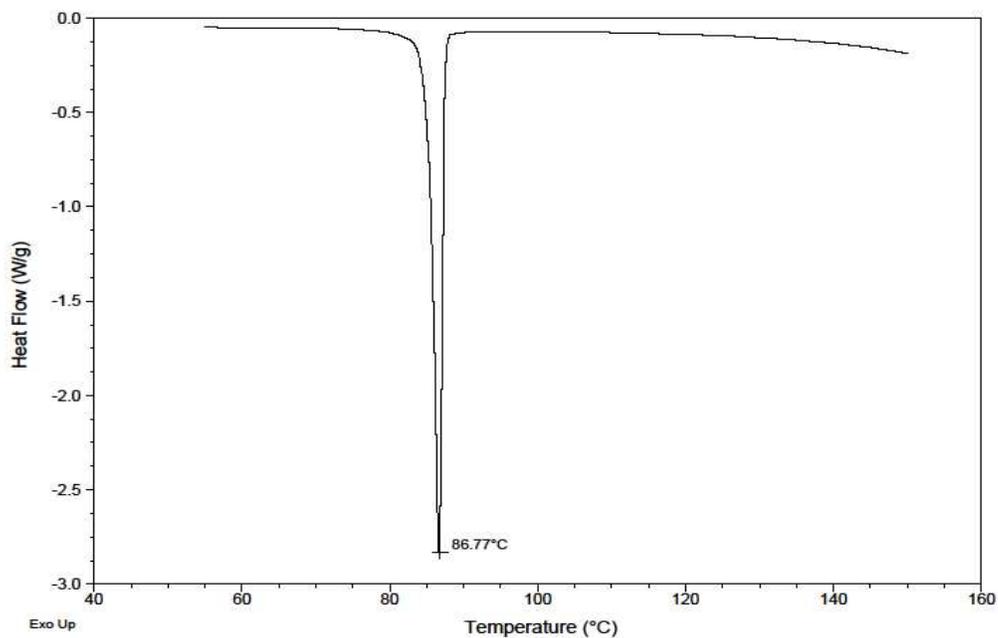
Analytical Data and Figures for 3-Methylthiobenzamide, **12**



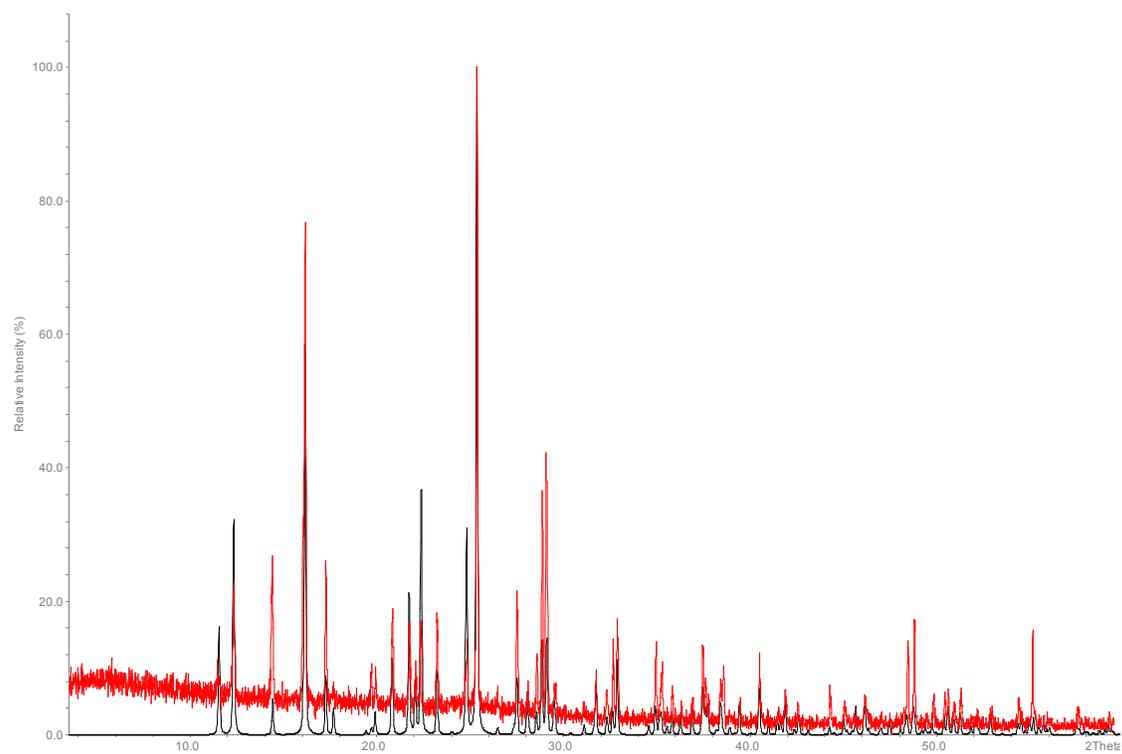
IR spectra for **12**.



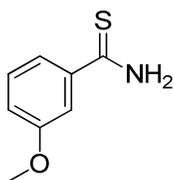
DSC of **12** obtained by solution crystallization (onset temperature 84 °C)



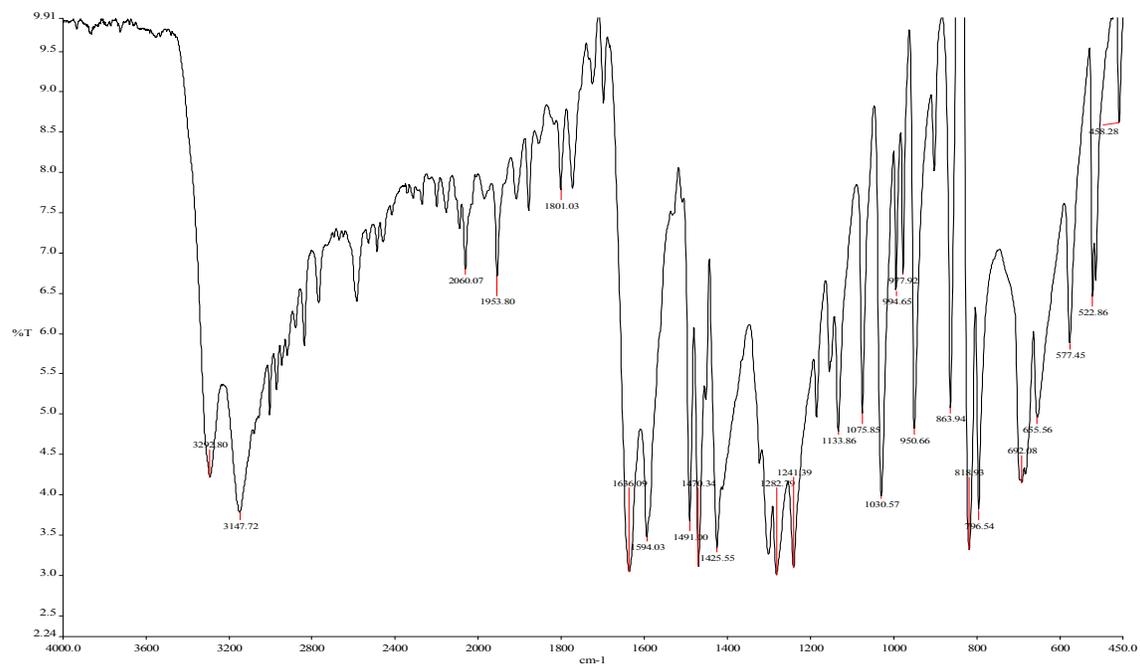
PXRD pattern of **12** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



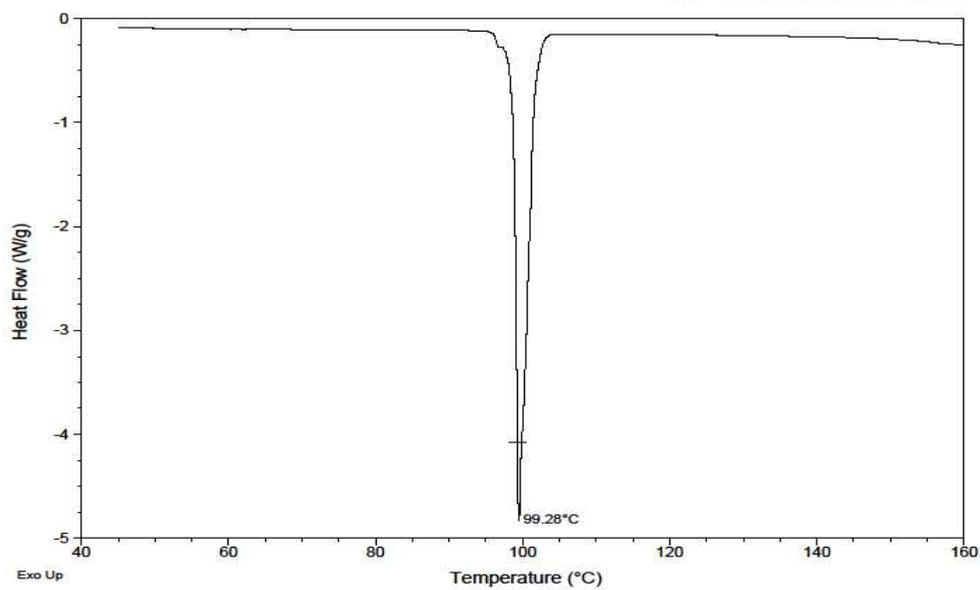
Analytical Data and Figures for 3-Methoxythiobenzamide, 13



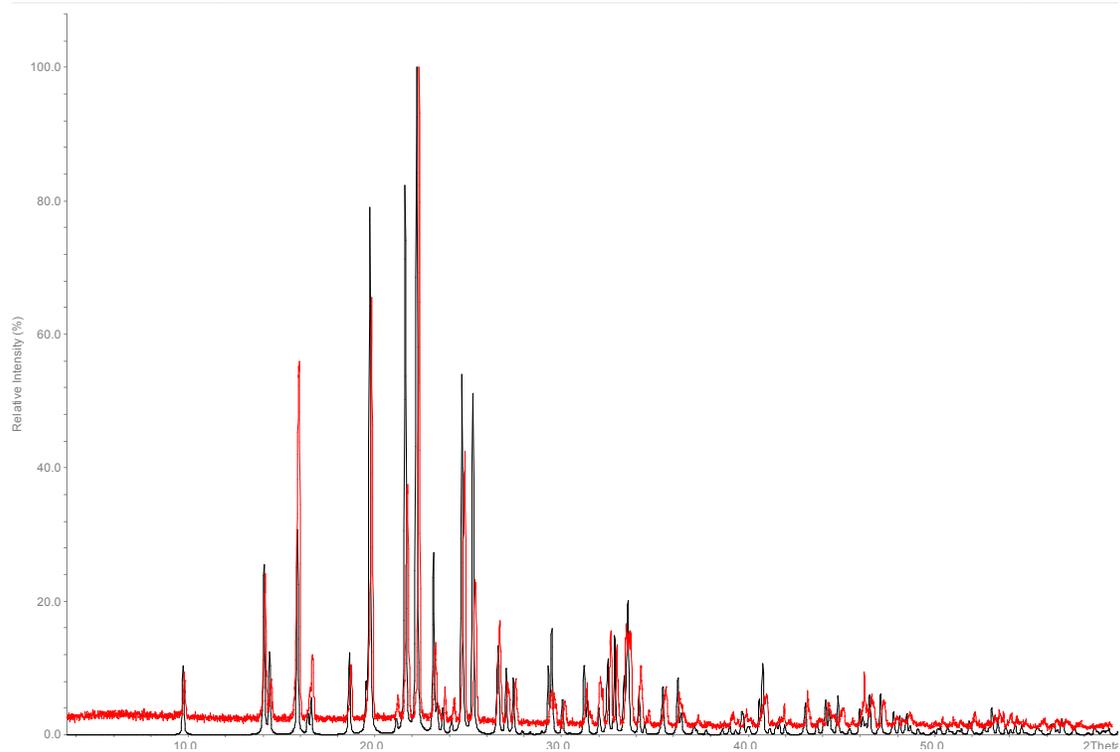
IR spectra for 13.



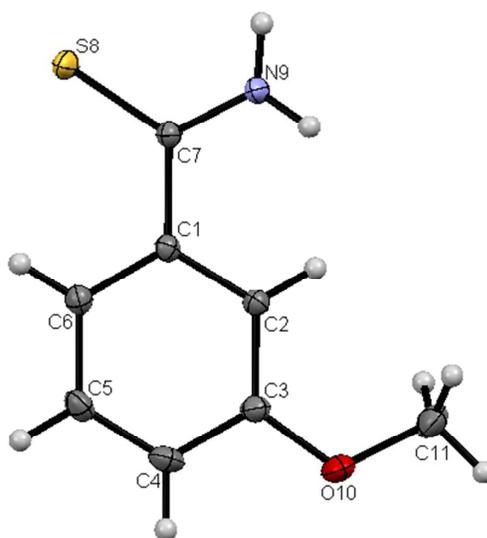
DSC of 13 obtained by solution crystallization (onset temperature 98 °C)



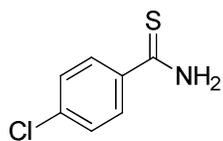
PXRD pattern of **13** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



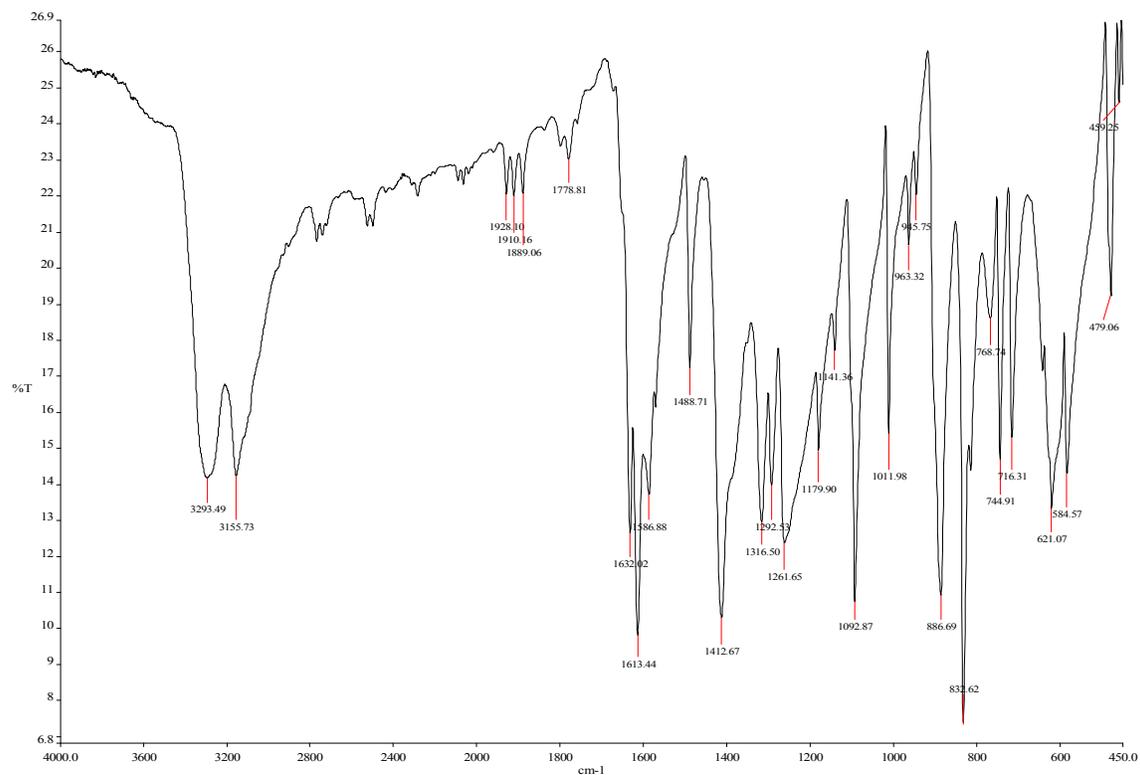
ORTEP plot of **13** (ellipsoids shown at 30% probability).



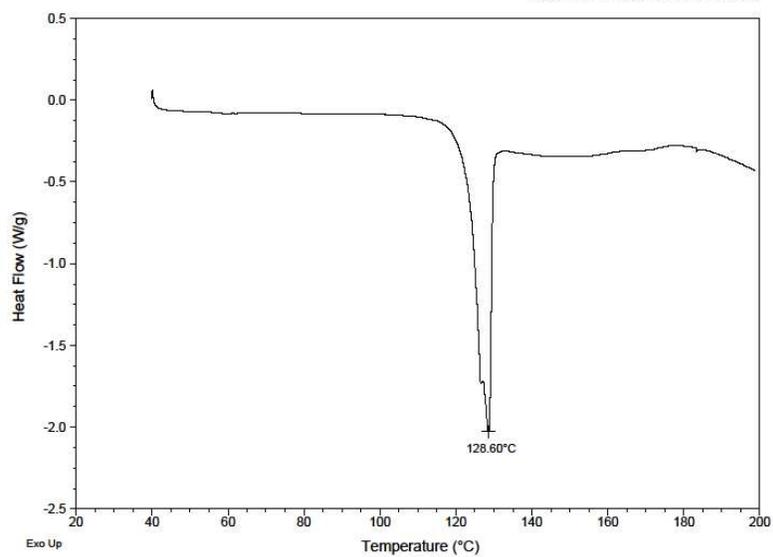
Analytical Data and Figures for the Beta Polymorph of 4-Chlorothiobenzamide, 14 β



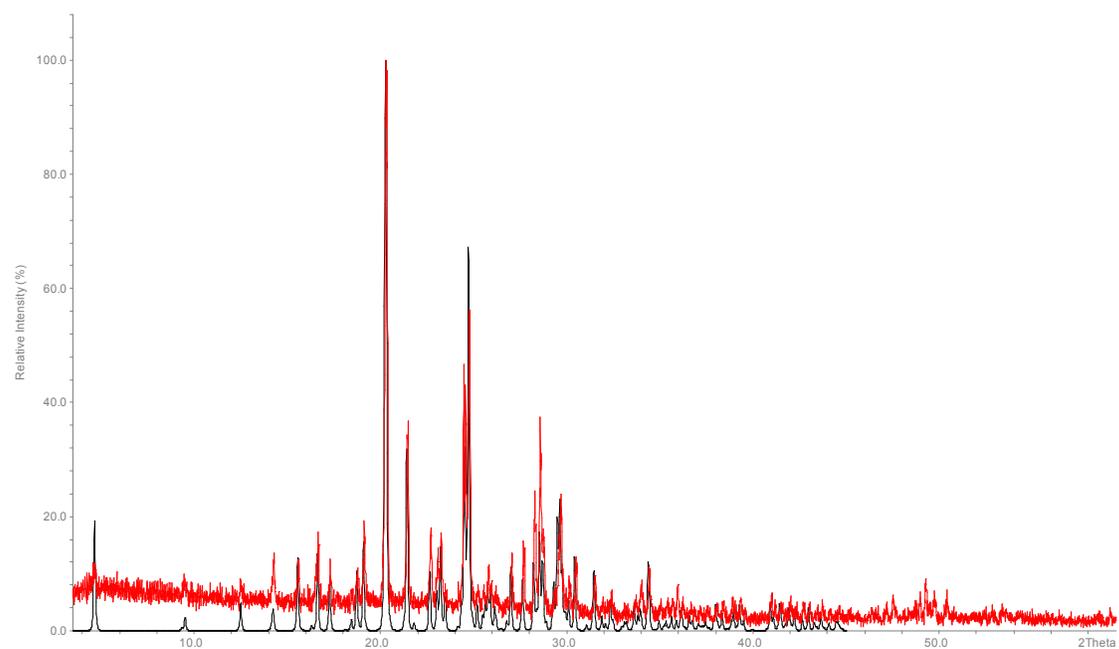
IR spectra for 14 β .



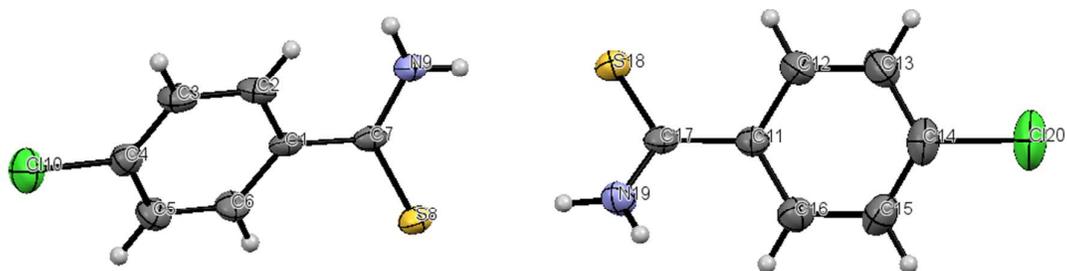
DSC of 14 β obtained by solution crystallization (onset temperature 123 °C)



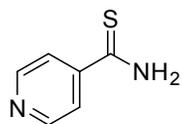
PXRD pattern of **14 β** obtained from solution crystallization (red) overlaid with theoretical pattern (black)



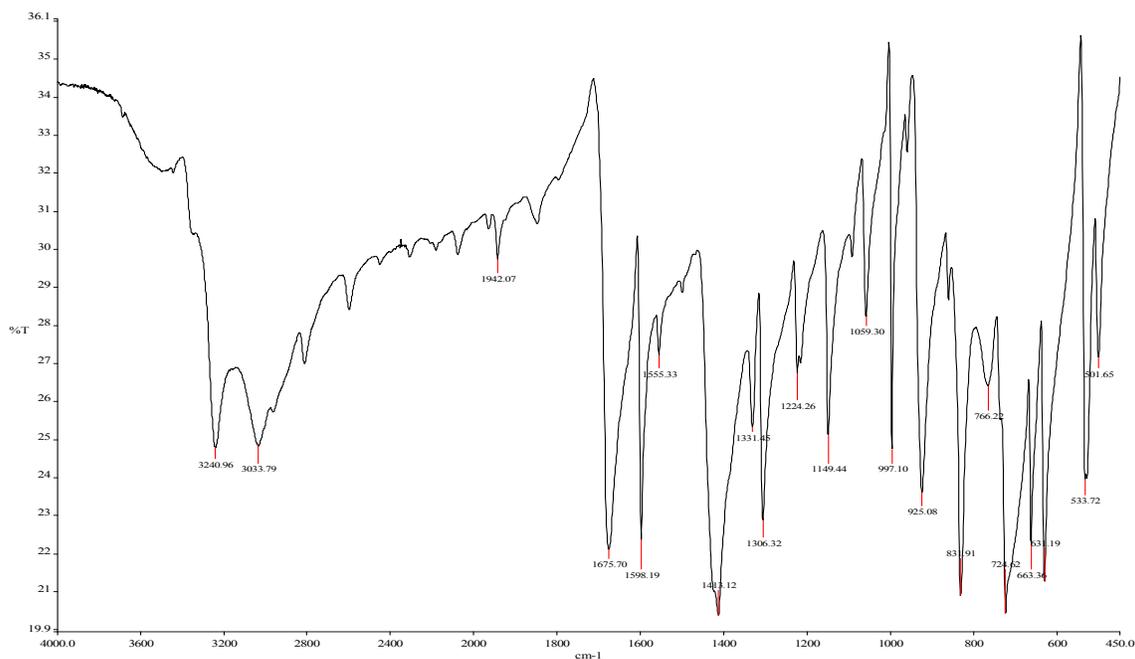
ORTEP plot of **14 β** (ellipsoids shown at 30% probability).



Analytical Data and Figures for Pyridine-4-carbothioamide, 15



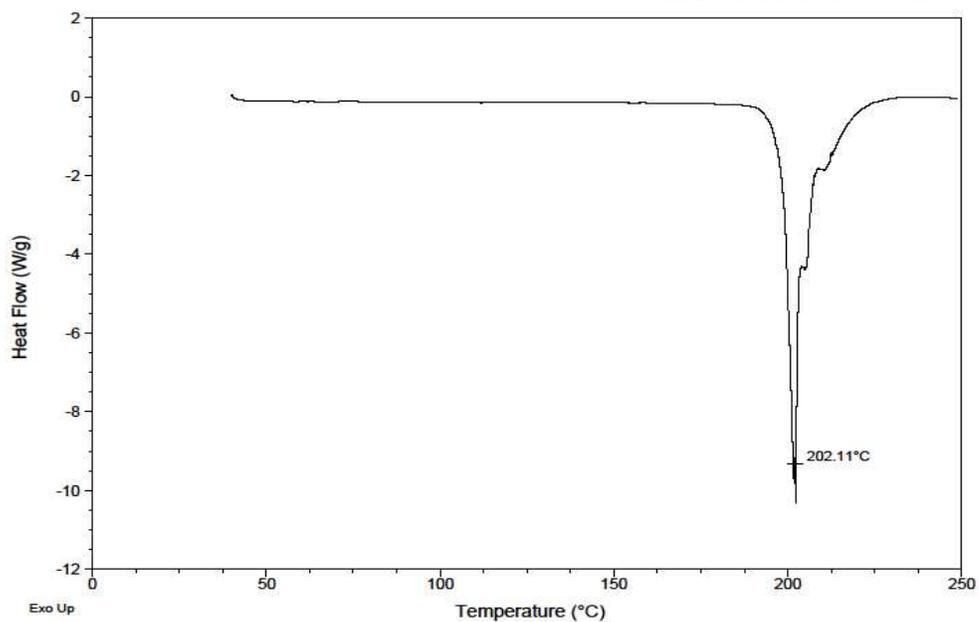
IR spectra for 15.



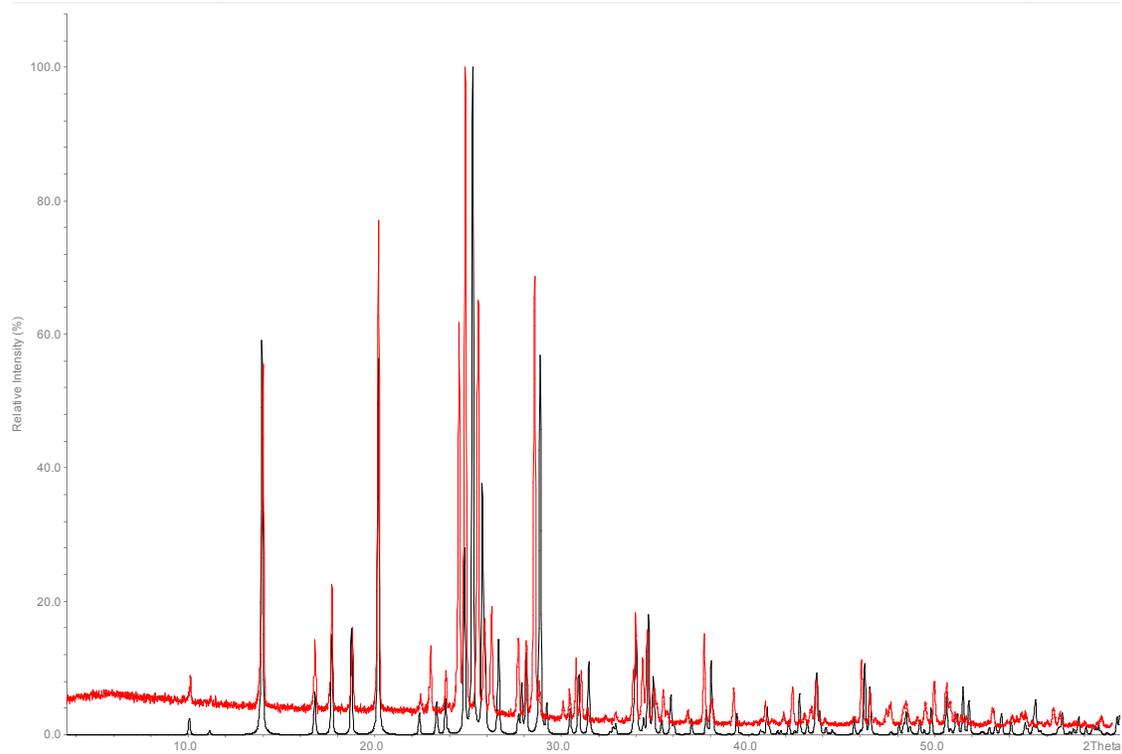
Sample: 4N_TBA
Size: 1.3320 mg
Method: Ramp

DSC

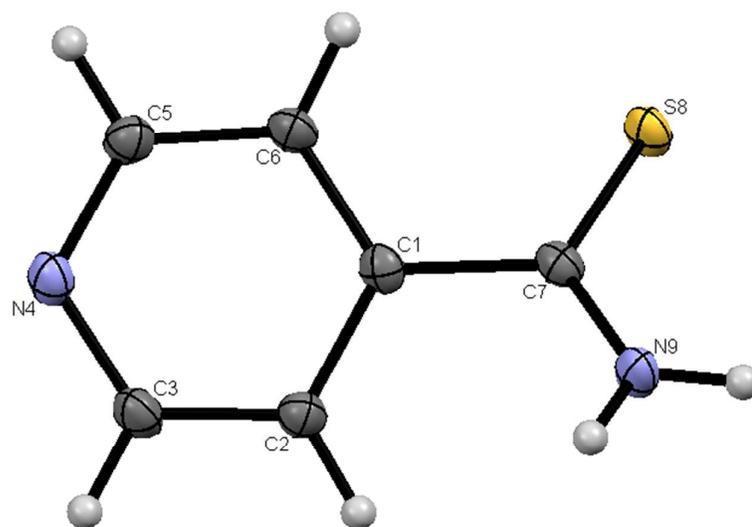
File: C:\...New Folder\4N_TBA.001
Operator: Kevin
Run Date: 07-May-2013 14:12
Instrument: DSC Q1000 V9.9 Build 303



PXRD pattern of **15** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



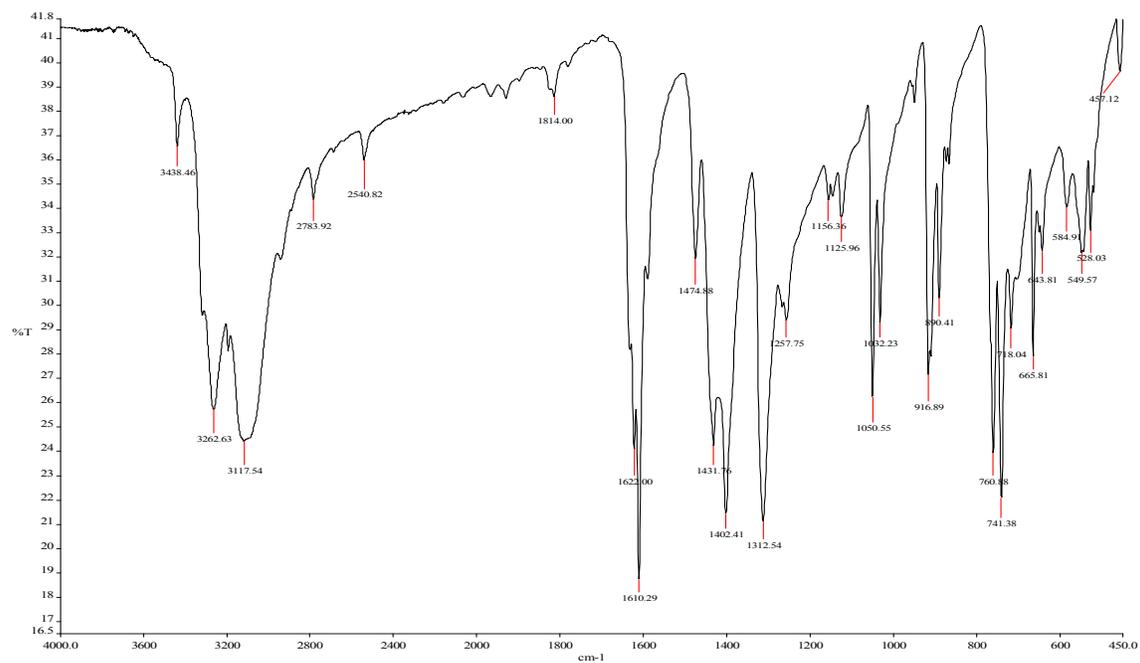
ORTEP plot of **15** (ellipsoids shown at 30% probability).



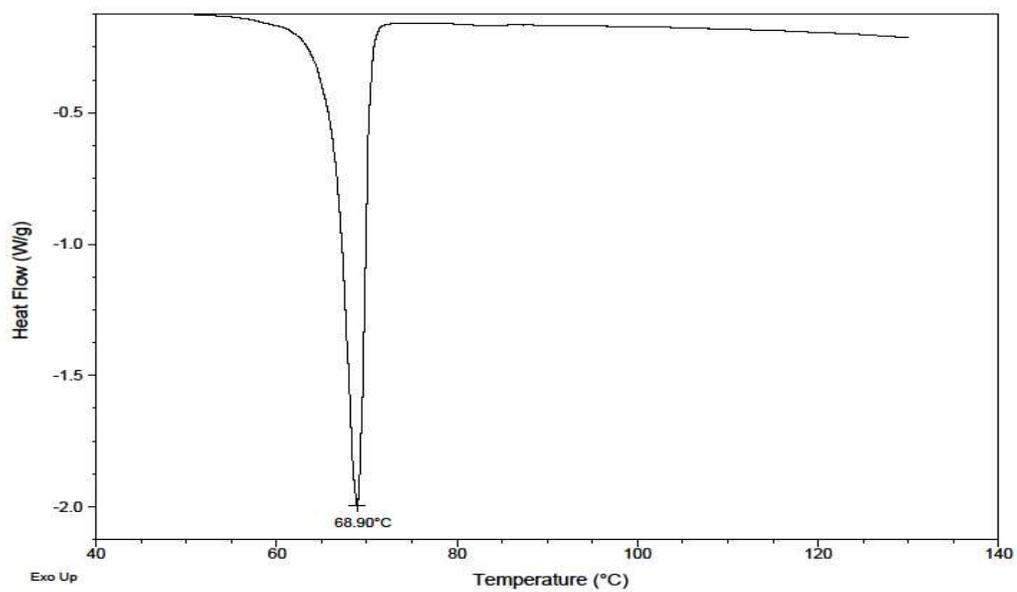
Analytical Data and Figures for 2-Chlorothiobenzamide, 16



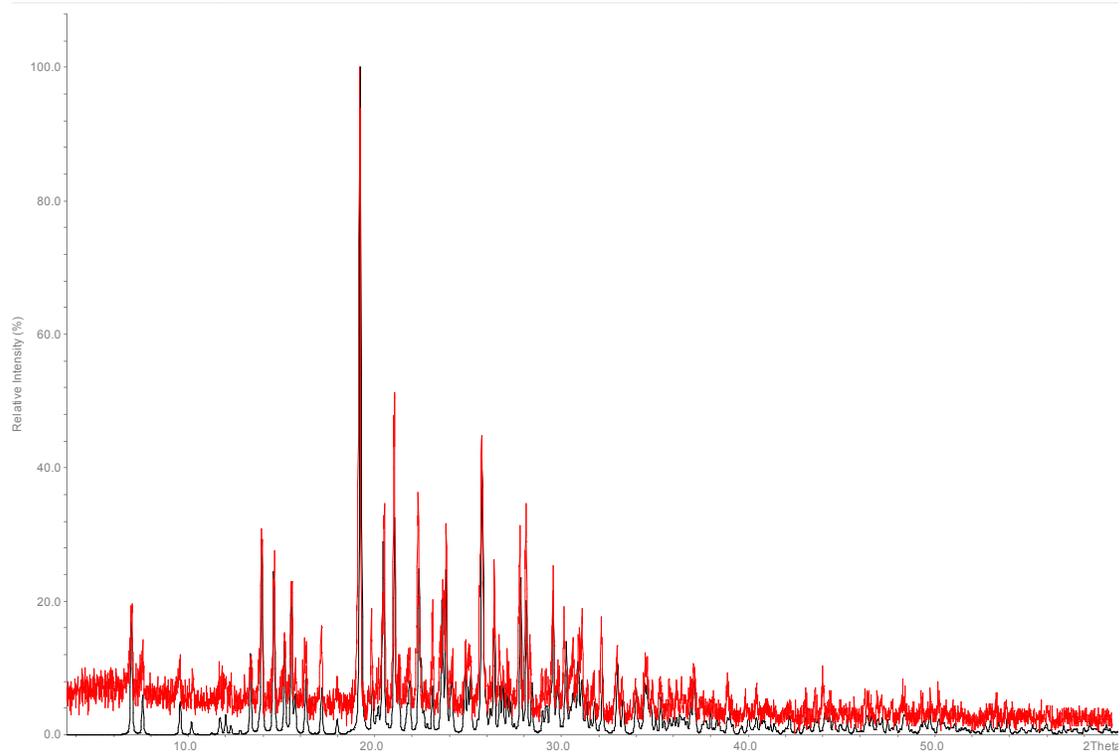
IR spectra for 16.



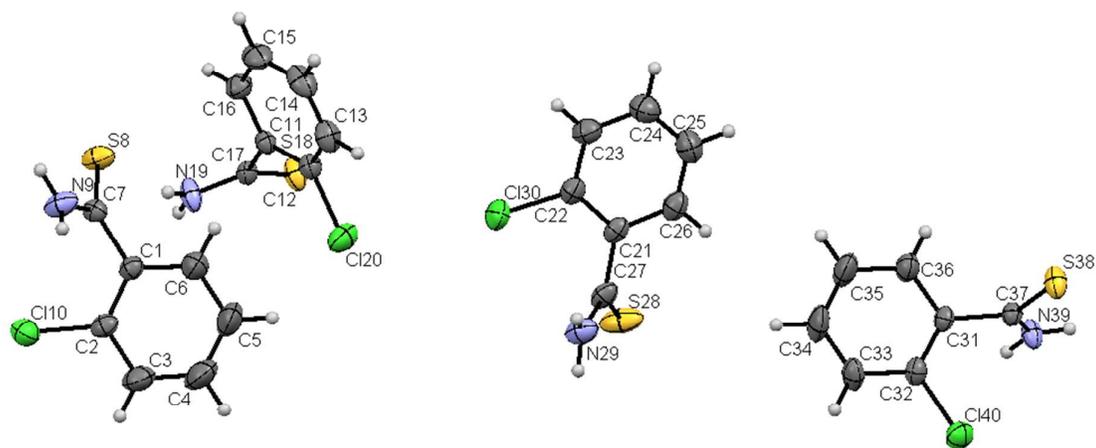
DSC of 16 obtained by solution crystallization (onset temperature 67 °C)



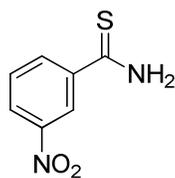
PXRD pattern of **16** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



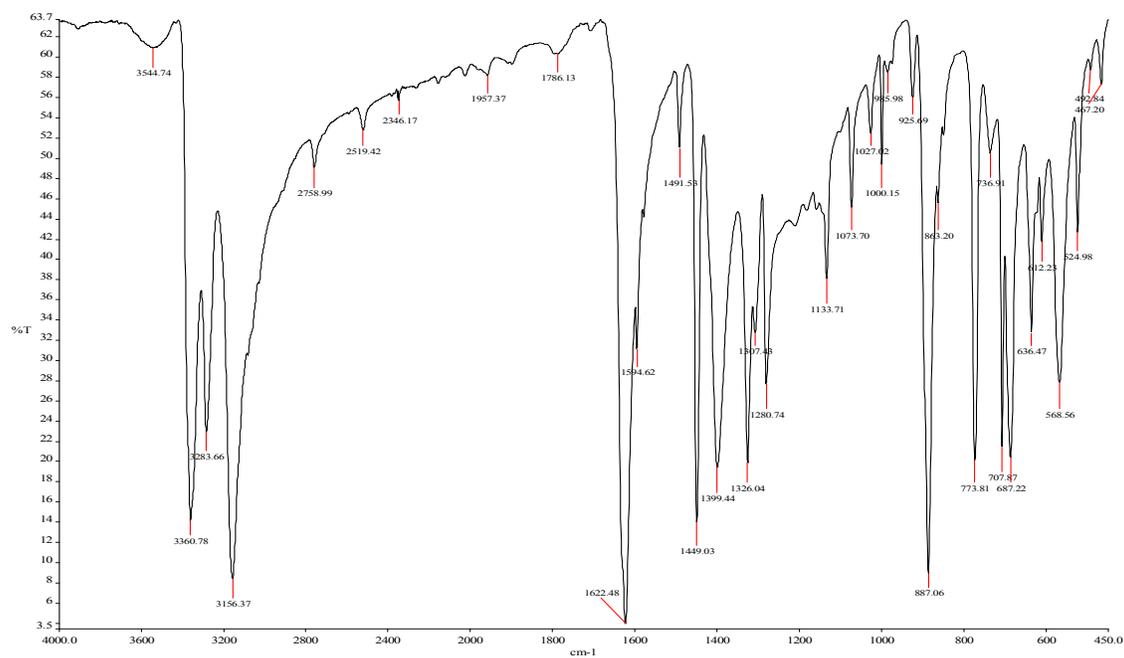
ORTEP plot of **16** (ellipsoids shown at 30% probability).



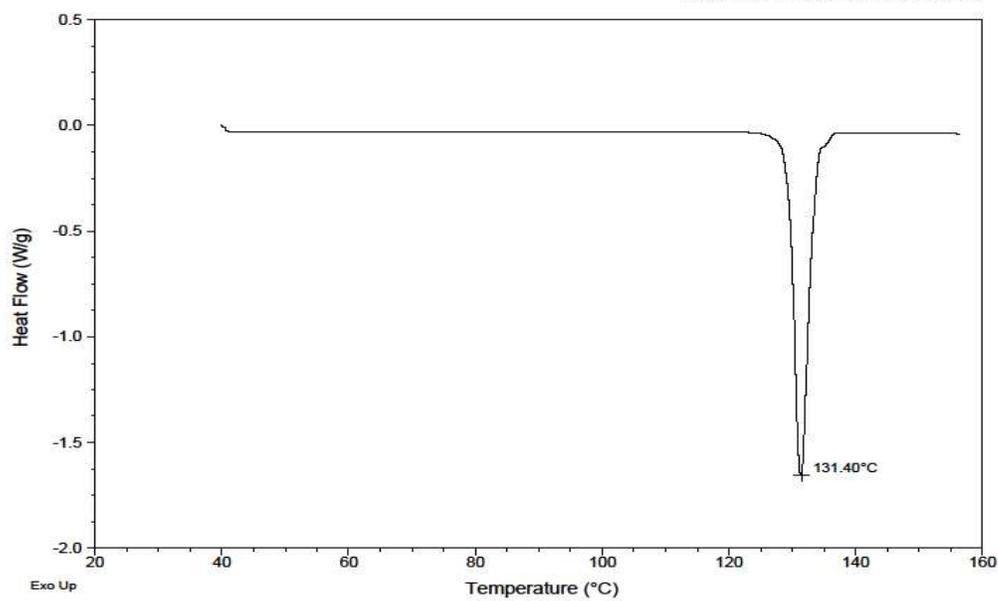
Analytical Data and Figures for 3-Nitrothiobenzamide, 17



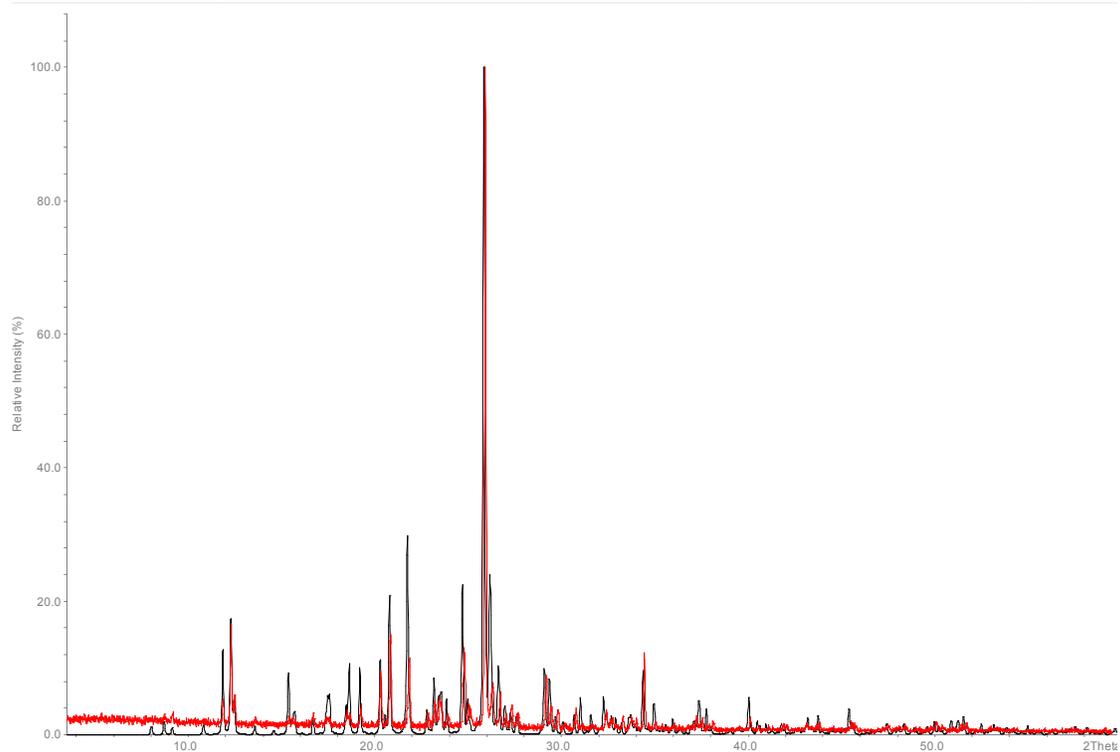
IR spectra for 17.



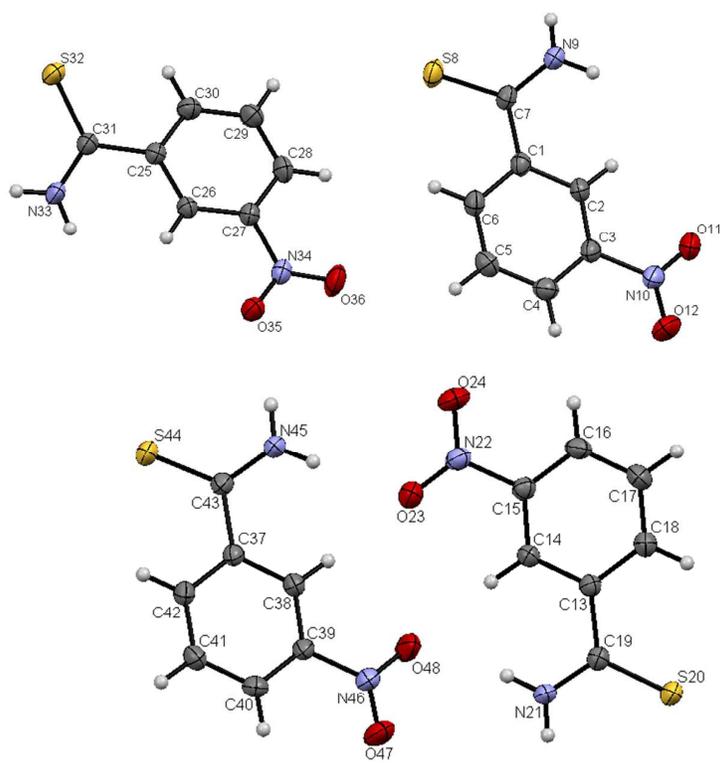
DSC of 17 obtained by solution crystallization (onset temperature 130 °C)



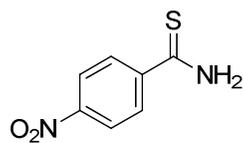
PXRD pattern of **17** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



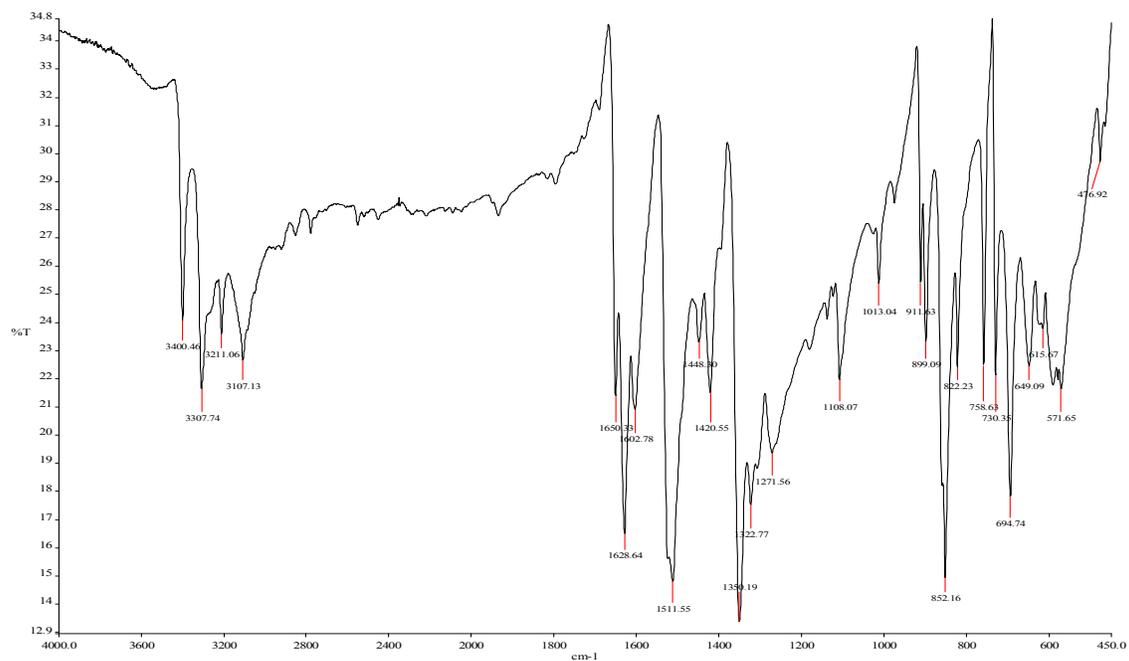
ORTEP plot of **17** (ellipsoids shown at 30% probability).



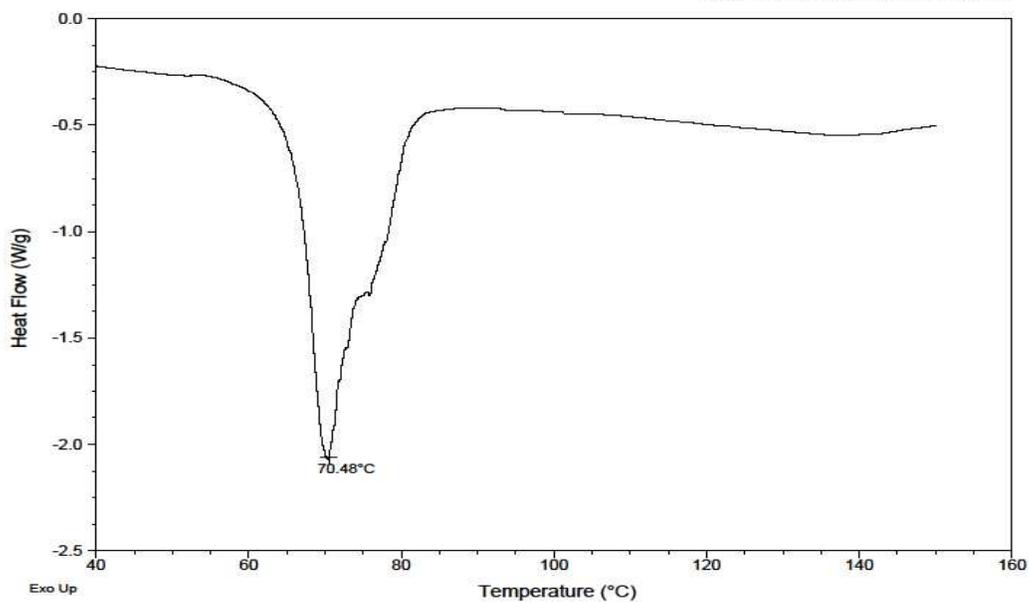
Analytical Data and Figures for 4-Nitrothiobenzamide, **18**



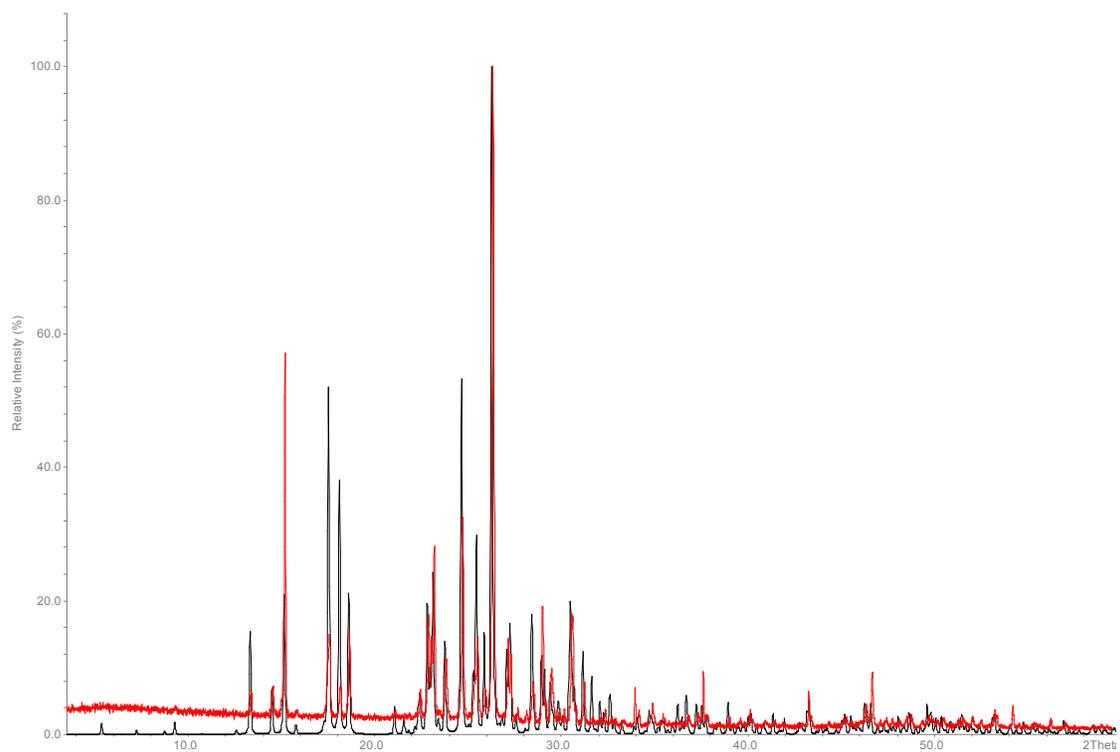
IR spectra for **18**.



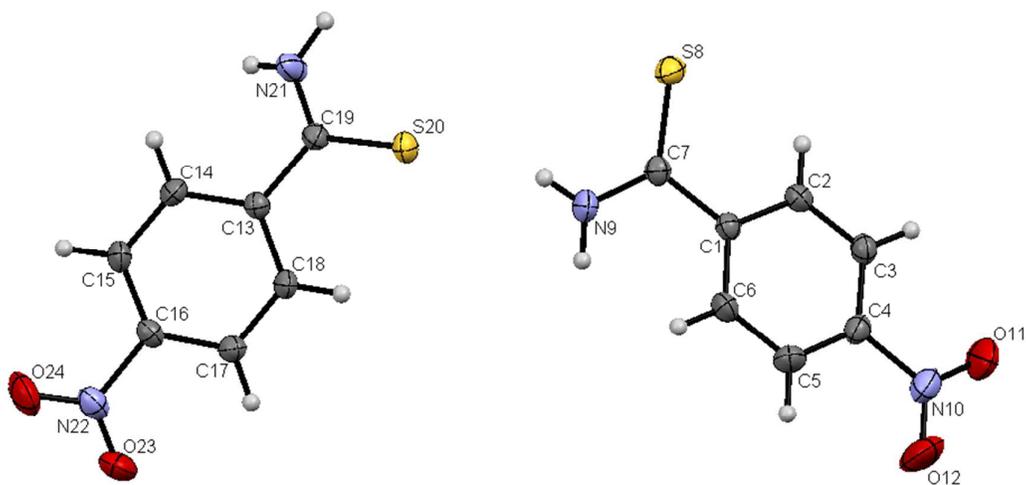
DSC of **18** obtained by solution crystallization (onset temperature 67 °C)



PXRD pattern of **18** obtained from solution crystallization (red) overlaid with theoretical pattern (black).



ORTEP plot of **18** (ellipsoids shown at 30% probability).



Polymorphic Screen

| | Acetone | MeCN | CH ₂ Cl ₂ | EtOH | EtOAc | Toluene |
|-----------|----------------|----------------|---------------------------------|----------------|----------------|----------------|
| 1 | Y ^a | Y ^a | Y ^a | Y ^a | Y ^a | Y ^a |
| 2 | - | Y | - | Y | Y | Y |
| 3 | Y | Y | Y | Y | Y | - |
| 4 | Y | Y | Y | - | - | - |
| 5 | Y | Y | Y | Y | Y | Y |
| 6 | - | Y | Y | Y | - | - |
| 7 | - | - | Y | - | Y | - |
| 8 | - | - | - | - | - | - |
| 9 | Y | Y | Y | Y | Y | Y |
| 10 | Y | Y | Y | Y | Y | Y |
| 11 | - | Y | Y | Y | Y | Y |
| 12 | Y | Y | Y | Y | Y | Y |
| 13 | Y | Y | Y | Y | Y | Y |
| 14 | Y | - | Y | Y | - | - |
| 15 | Y | Y | - | Y | Y | - |
| 16 | - | Y | Y | - | Y | - |
| 17 | - | Y | Y | Y | Y | Y |
| 18 | Y | Y | Y | Y | Y | Y |

Y PXRD data matches the theoretical pattern.

Y^a No theoretical pattern available; the PXRD data match either other.

- The material produces a PXRD pattern insufficient to report on due to either preferred orientation effects, or crystalline material was not obtained.