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Non-Conventional Scattering Studies using an Image Plate Diffractometer

Lynne Thomas

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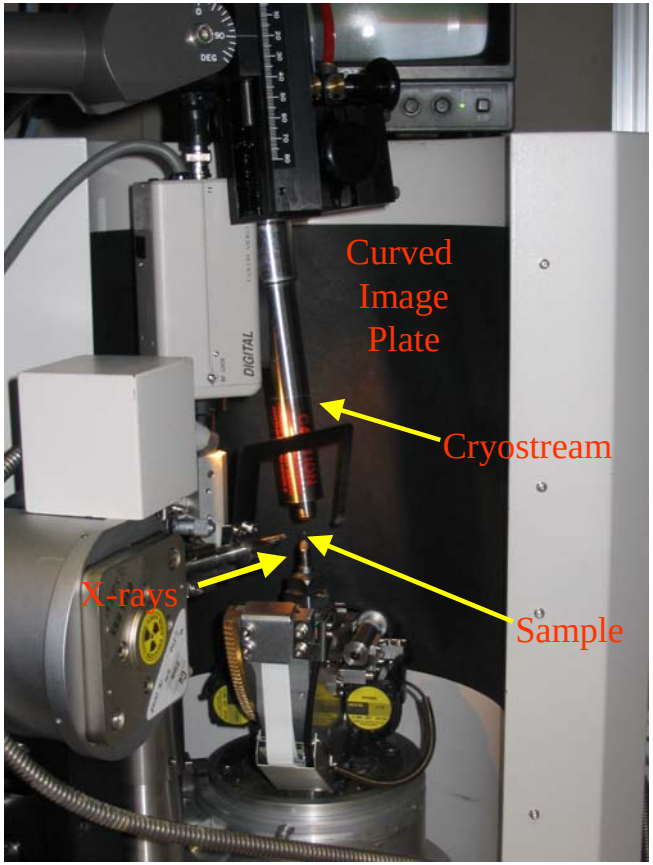
Introduction



- Weak scattering
  - Diffuse Scattering
  - Liquid Scattering
- Image Plate (Rigaku R-axis/Rapid)
  - Large dynamic range
  - No dark current accumulation
  - Long images possible
  - Large regions of reciprocal space accessible in a single image
  - 465 mm x 258 mm active area
  - Mo or Cu radiation

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Diffuse Scattering

- Where local order is present get structured diffuse scattering
- Much weaker than Bragg scattering
  - Often more difficult to see
- Located both around Bragg peaks and between
  - Need to survey large regions of reciprocal space
- This disorder can be important for properties
  - Dielectrics, NLO, etc…

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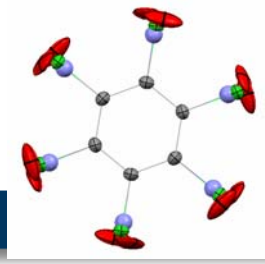
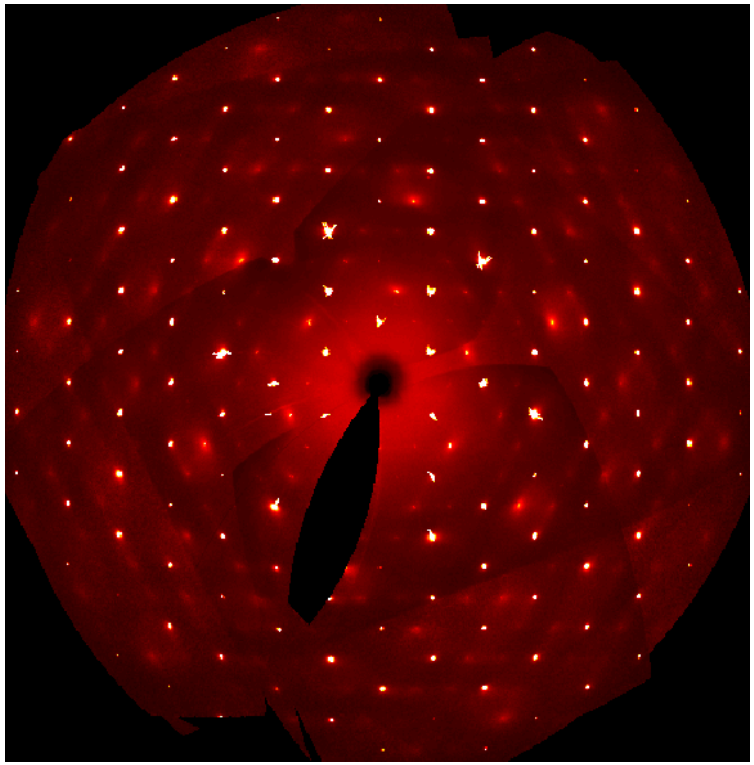
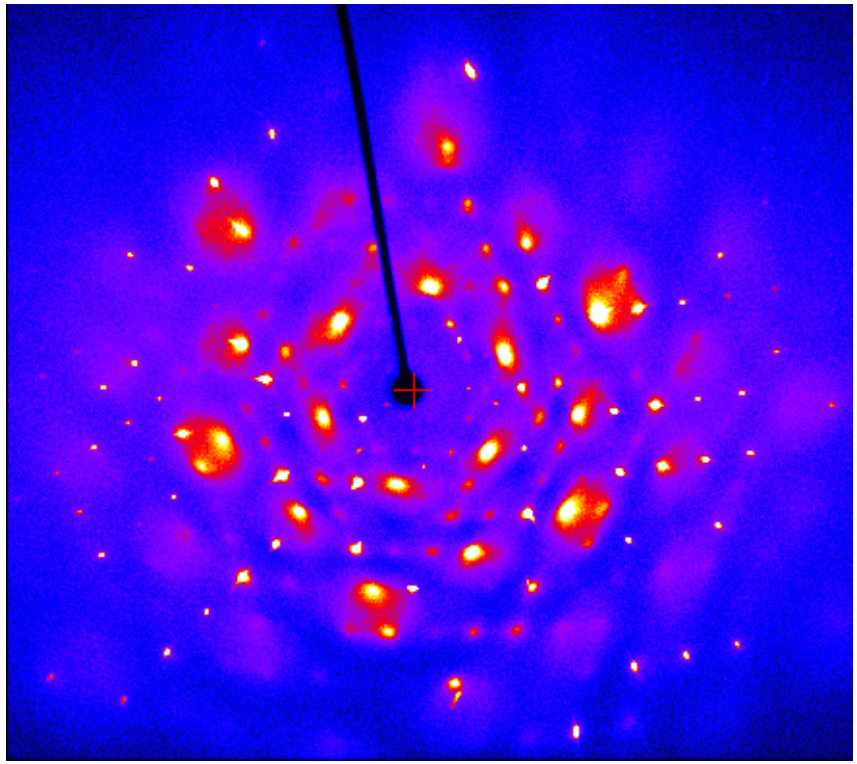


Diffuse Scattering - Pentachloronitrobenzene

Synchrotron



Lab Image plate



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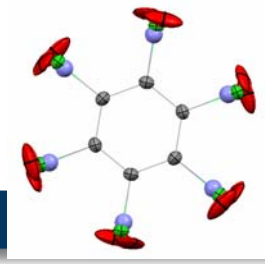

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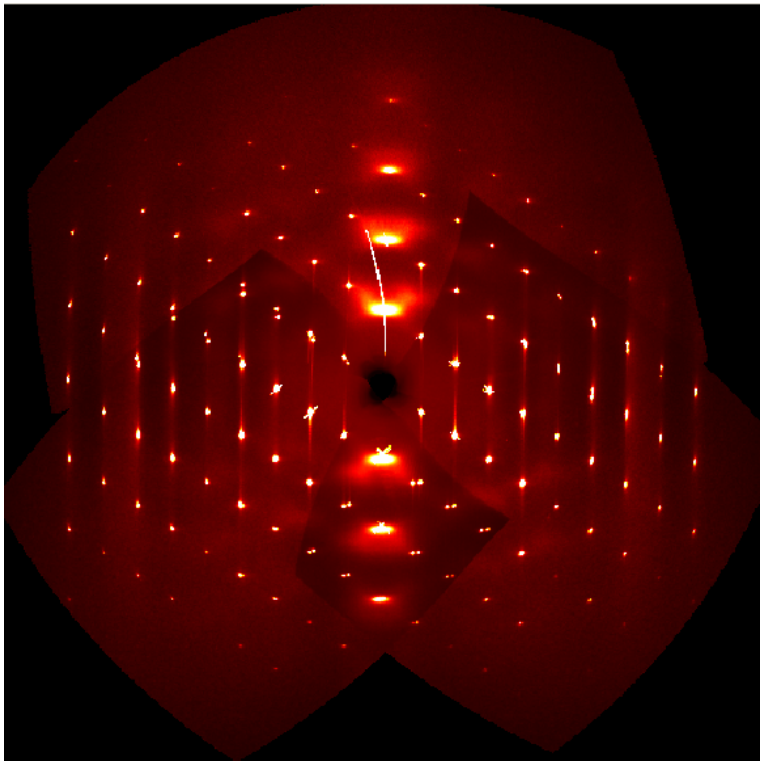

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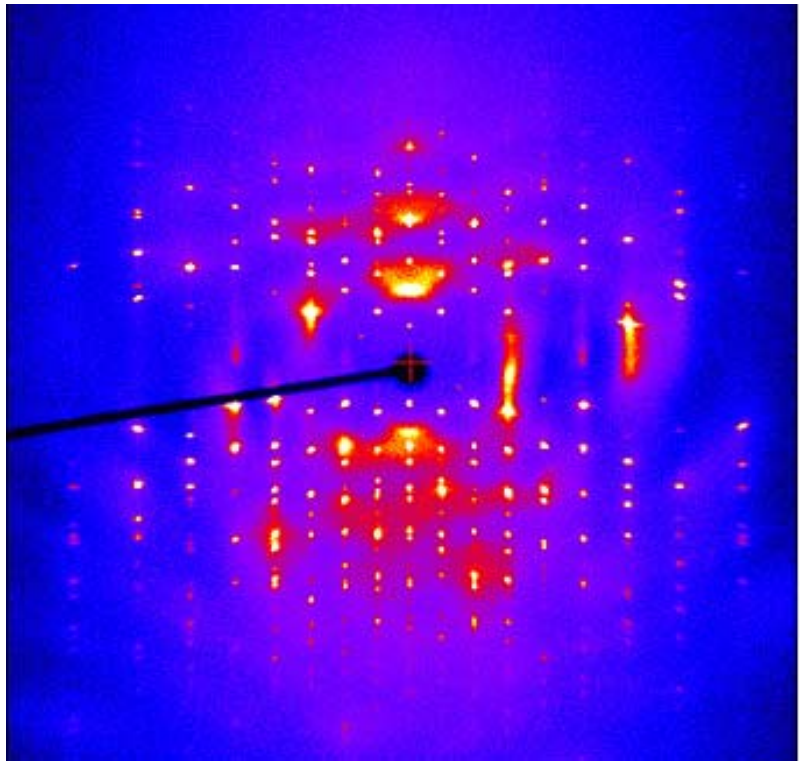

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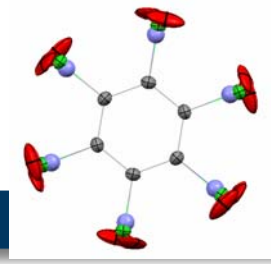
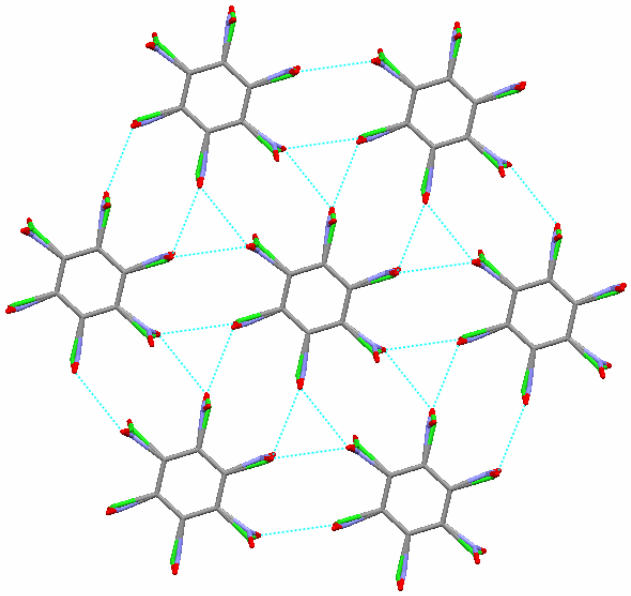
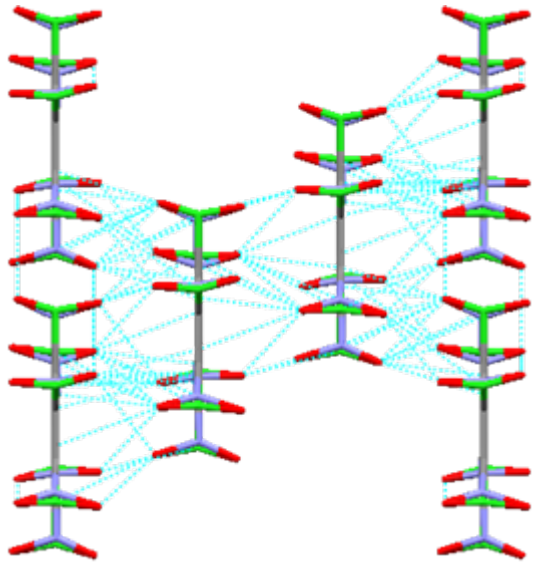
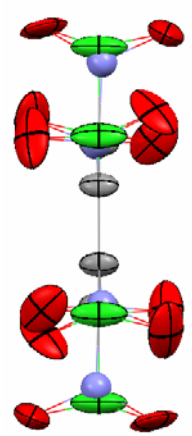


Diffuse Scattering – modelling

- Modelling the disorder
  - Monte Carlo modelling
  - Cluster calculations







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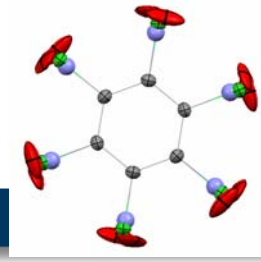

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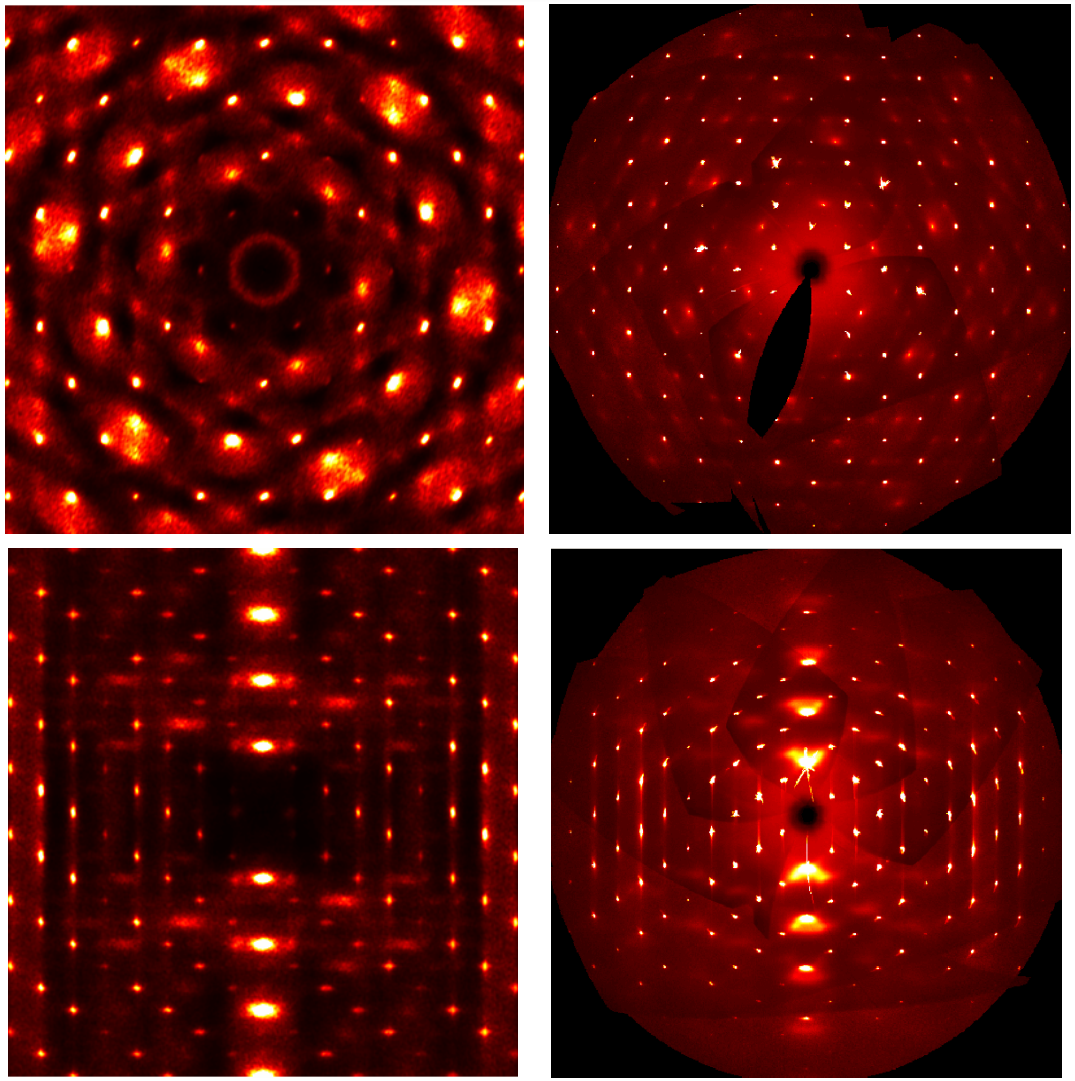

- Successful modelling of diffuse scattering
- Not always the most obvious cause

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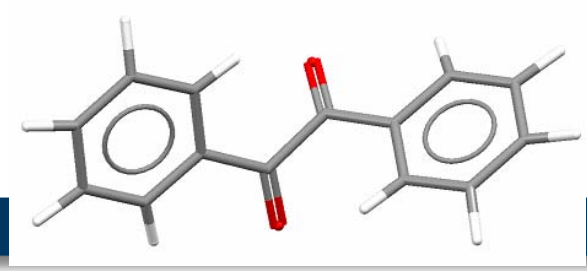
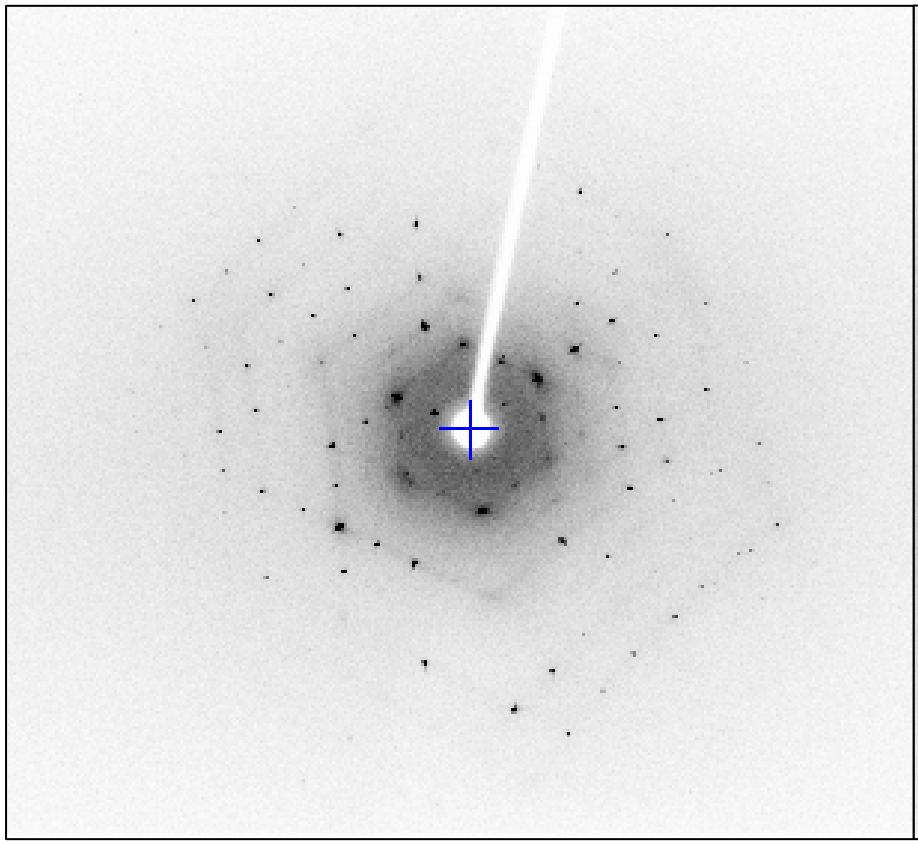
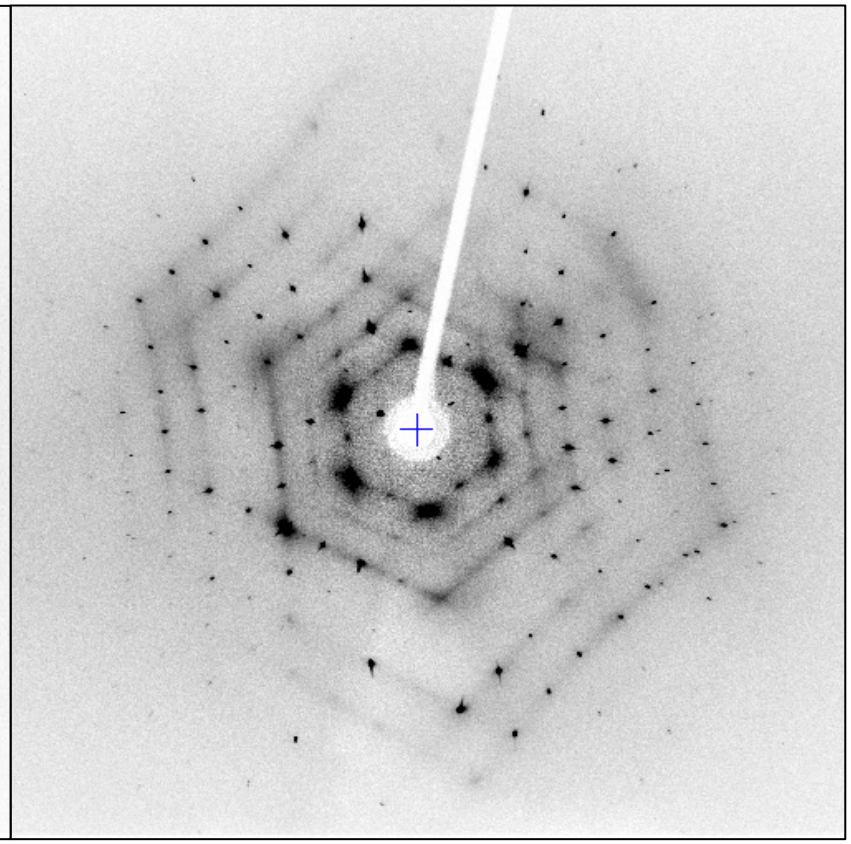
Diffuse Scattering - benzil



Uncorrected

Corrected





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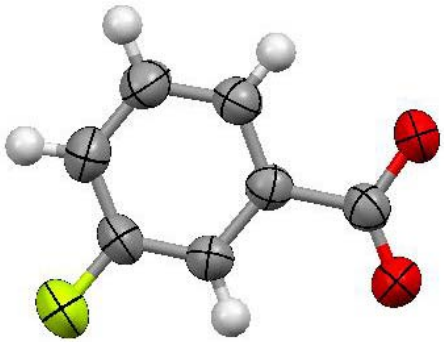
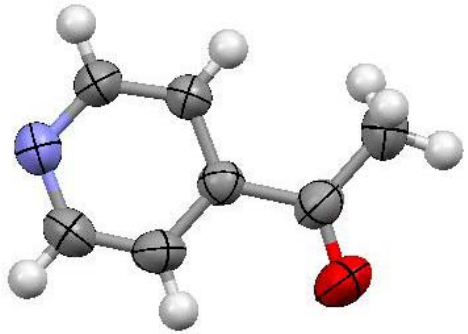
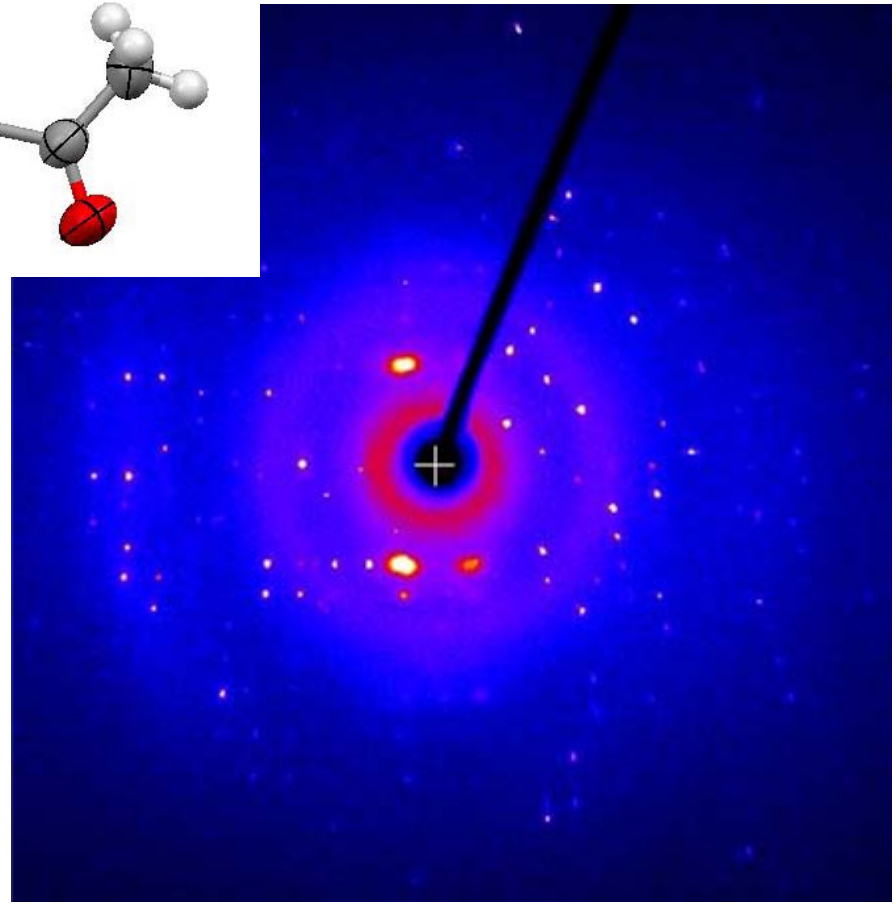
Diffuse Scattering – co-crystals







- 3-fluorobenzoic acid and 4-acetylpyridine
- Similar features to PCNB?
- Another layered material
- Stacking faults?

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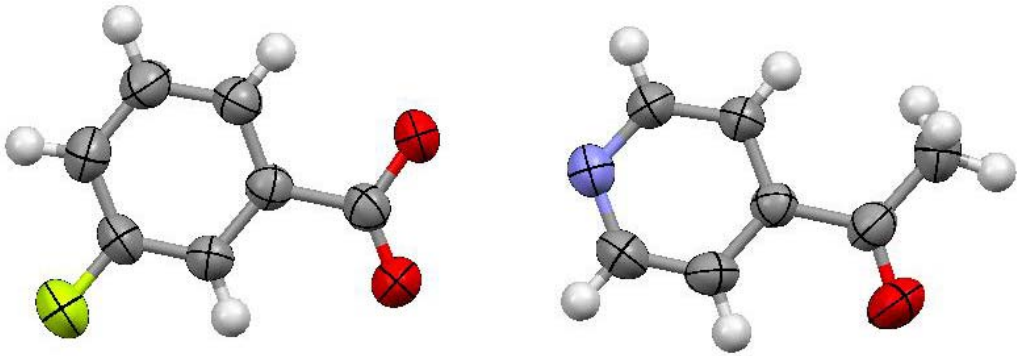
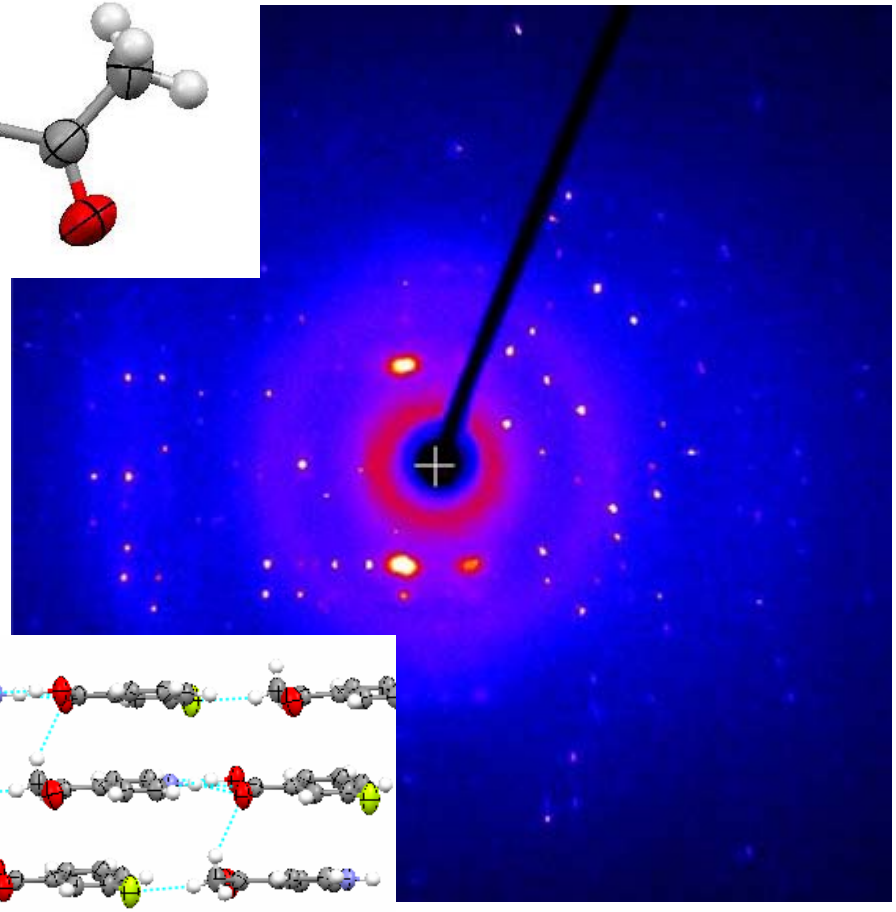
Diffuse Scattering – co-crystals





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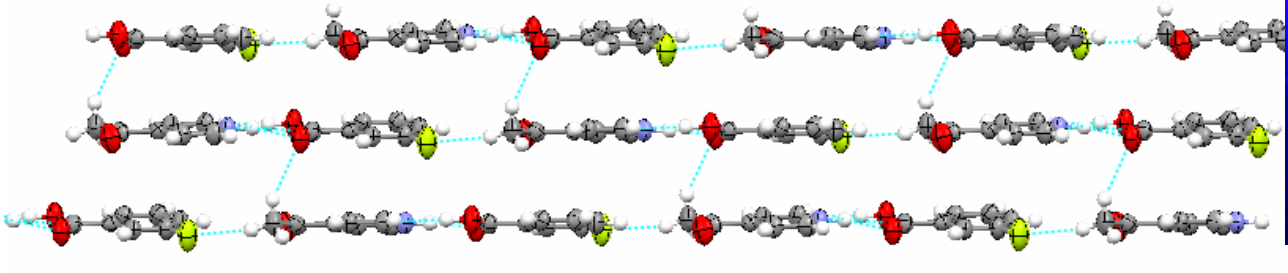

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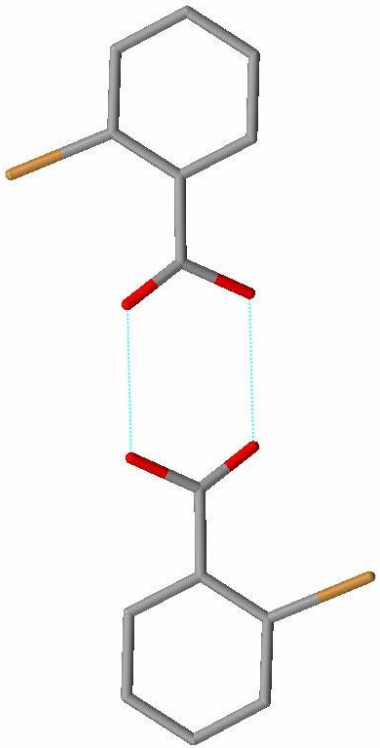
Liquid Scattering



- Nucleation
  - Polymorphism
  - Is there a basic building block that can be consistently detected prior to crystallisation?
- Choose substituted benzoic acids as a standard sample
  - Consistently form dimers in the solid state
  - Do these form in the liquid state? – fingerprint?
  - 1M samples contained in capillary
  - Careful background measurements essential including solvent

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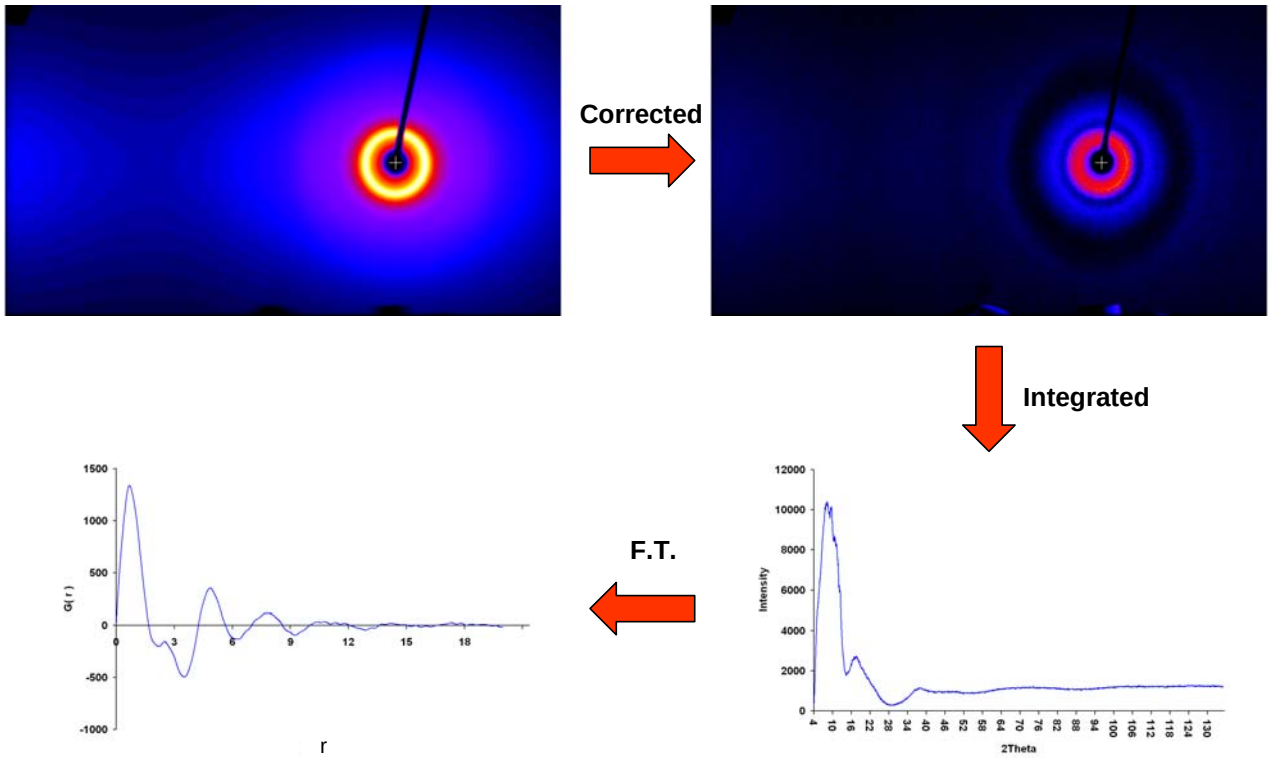
Liquid Scattering – substituted benzoic acids

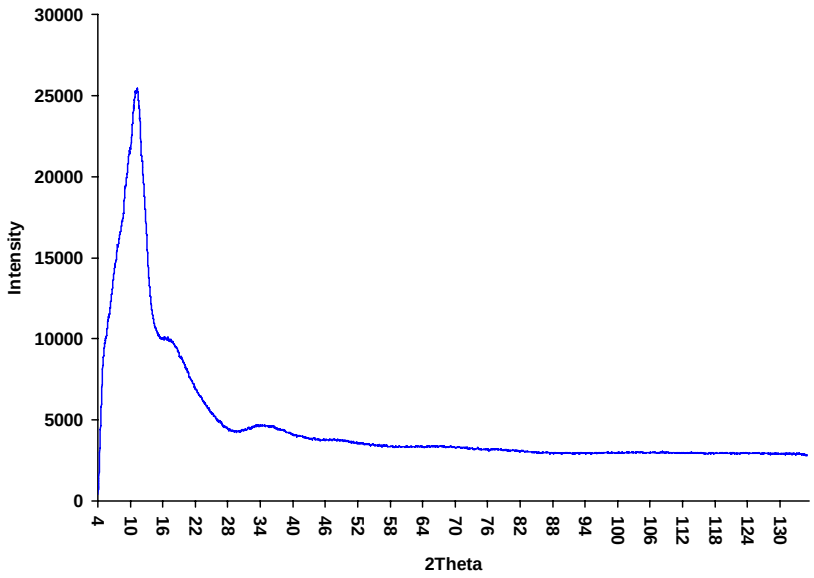
- Benzoic acid methanol solution
- Significant scattering from solution
- Reasonable G(r) - Interpretation on-going



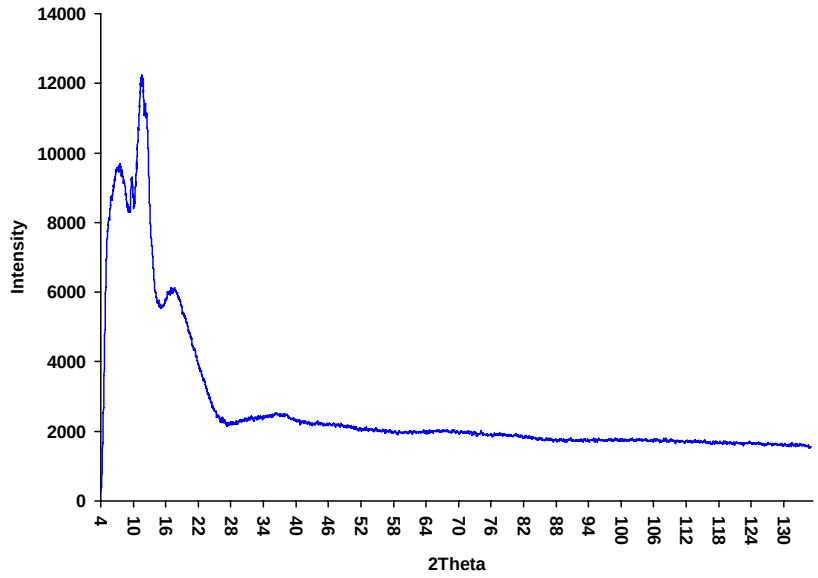
Liquid Scattering – chlorobenzoic acids

- Raw corrected data significantly different
- Need to convert to G(r)
- Careful application of corrections required

3-chlorobenzoic acid



2-chlorobenzoic acid



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Diffuse & Liquid Scattering – what next?

- Diffuse
  - Reciprocal space reconstructions vital
  - Measurement of principal reciprocal space slices in a single image e.g. hk0, h0l, 0kl
  - Important that the Bragg peaks overlay well
- Liquids
  - Reliable way to extract corrected G(r)
  - Long beamstop to gain more small angle data
  - Fingerprint for simple molecular cluster/association
- Modelling
  - Diffuse: increased use of Monte Carlo and RMC
  - Other methods of model generation – supercells, etc
  - Liquid: modelling using EPSR or PDF analysis
  - Neutron data? Liquid-specific PDF program?

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Conclusions

- Can collect good quality diffuse scattering images on the Rigaku R-axis/Rapid
  - Simple corrections enhance the weaker scattering
- To date modelling carried out on synchrotron or specialist instruments
  - Potential to apply to standard laboratory instrument data
- Can measure good quality liquid scattering
  - Potential to identify a fingerprint for interactions
  - Chlorobenzoic acids a promising start

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