Publishing Crystal Structures

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Outline

Part 1

- Crystals
  - Quality of crystals
  - Growing crystals

Part 2

- Submitting a sample for analysis
- Determining quality of data
- Preparing data for publication
- Submitting data for publication
- Crystallography programs

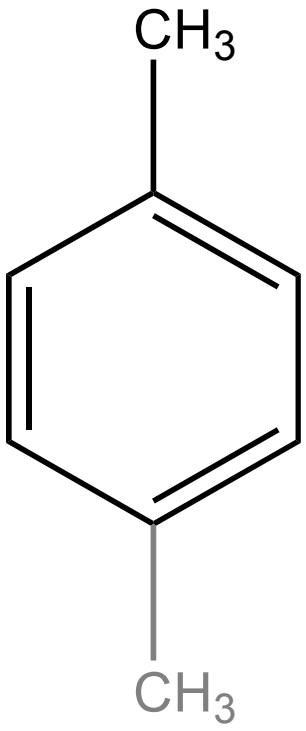
Submitting a Sample for Analysis

- Vessel has to be clearly labeled
- Form needs to be filled out
  - Label on sample must match sample code on form
  - Location of sample
  - Stability and/or Toxicity
  - Synthetic route
    - Starting materials
    - Byproducts
  - Proposed molecular formula and structure
  - Solvents used in crystallization

NO FORM = NO DATA

Submitting a Sample for Analysis

- Sample submitted does not equal a crystal structure
  - Crystal quality
    - Poor crystallinity
    - Too small
  - Data collection
    - Poor diffraction
    - Long collection times
  - Solving and refining data
    - Incompleteness
    - Twinning
    - Disorder
    - Correct structure, but not publishable
    - Inability to determine correct structure



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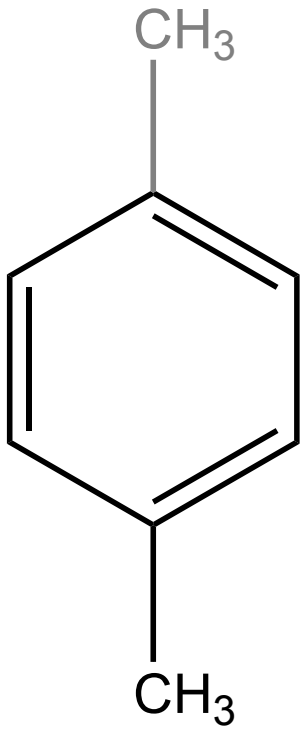

Submitting a Sample for Analysis

- Inability to collect data or poor data quality
  - Data collection
    - Remount sample
      - Sample will be consumed entirely unless noted in form
    - Alternate data collection strategy
  - Recrystallize
    - Purify sample
    - Alternate solvent
    - Alternate method of crystal growing

Determining Quality of Data

- Data collection
  - Close to 100 % Completeness
    - Collected most of the reflections in the asymmetric unit
- Refinement
  - Low R1 values (below 10 %)
    - Measure of how well the refined structure predicts the observed data.
  - Low wR_2 values (below 20 %)
    - Least-squares residual; measure of how intensities calculated for the X-ray reflections match those measured experimentally.
    - High values could be attributed to:
      - Incorrectly assigned atoms
      - Unaccounted electron density
      - Disorder

Determining Quality of Data

- Low R(int) values (below 10 %)
  - Measure of how well the data merges.
    - Incorrect Laue group
    - Twinning
    - Crystal decomposition
    - Bad or missing absorption correction
- Residual electron density is close to 0 %
- Final structure makes chemical sense

Successful Data Analysis

- Obtain crystal structure which refines well
- Resolve twinning or disorder
- Resolve any other issues present

- Once data is satisfactory you will receive:
  - Crystallographic information file or CIF
  - Copy of the CIF check results
  - Crystallographic table
    - Information regarding structure refinement
    - Bond lengths and angles
  - Labeled and numbered crystal structure
  - Sample

Preparing Data for Publication

- Crystallographic information file or CIF
  - Standard file for the archiving and distribution of crystallographic information.
    - Used to validate the quality of data
      - IUCr CHECKCIF
      - Alerts must be addressed
        - Wrong space group
        - Incomplete data
        - Disorder

Preparing Data for Publication

- Prepare crystallography tables
  - Use a CIF file once all of the alerts have been addressed
    - Table of crystallographic parameters
      - Formula
      - Formula weight
      - Space group
      - Unit cell
      - Volume
      - Temperature
      - Wavelength
      - Refinement values
    - Table of bond lengths and angles
      - Must include Estimated Standard Deviation values or esd's
        - 1.555(6) Å = 1.555 ± 0.006 Å

Preparing Data for Publication

- Deposit CIF to Cambridge Crystallographic Data Centre (CCDC)
  - Acquiring deposition numbers
    - Email CCDC the following information:
      - Author list
      - Title of article
      - Journal
      - CIF
  - Deposition numbers need to be included in your experimental details

Preparing Data for Publication

- Include in your experimental section:
  - Details on the X-ray Diffractometer
    - Radiation source (Mo or Cu)
    - Wavelength (Mo = 0.71073 Å; Cu = 0.15443 Å)
    - Type of detector (CCD)
    - Sample mounting (Copper pin; crystal covered in silicone oil)
    - Low temperature device (Oxford Cryosystems)
  - Details on structure refinement
    - Programs used for solving, scaling, refining, handling disorder
    - Special information on structure refinement
      - Twinning
      - Disorder

Crystallography Programs

- Apex II
  - Mounting
  - Solving and Refining
    - CIF file and Table
- Mercury
  - Reads CIF files for visualization of crystal structures
    - Measure bond lengths and angles
    - Measure inter- and intramolecular interactions
    - Make pictures
- Encifer
  - Edit CIF files for publication

www.ccdc.cam.ac.uk/free_services

Conclusions

- The quality and meaningfulness of your results is directly dependent on the quality of your sample crystal.

  Garbage in = Garbage out

- You can get information from a bad crystal structure, but it will be difficult to publish.
- Spend the time and effort to determine the best method of crystallization before submitting a sample for analysis.
- Consult your crystallographer on preparing and submitting data for publication.
- Don’t let your crystals dry out!