CRYSTALLOGRAPHY in the GLADYSZ GROUP 2012

INTRODUCTION

This document outlines group procedures for preparing a "final report" of a crystal structure. Different journals can have slightly different publishing formats, and frustratingly they change periodically. However, a final report prepared according to the following guidelines will contain enough information to satisfy all journals (and can be directly incorporated into a dissertation).

POINTS TO NOTE IN ADVANCE

1. Solved structures from the Texas A&M Crystallography are code named as follows: "JAG_XYZ_YYMMDD", where the XYZ represents the coworker's initials and YYMMDD the year/month/day in two digit format. The means by which the sample was obtained (crystal growth method) must be clearly indicated in the laboratory notebook.

2. Most crystallography laboratories do not provide a "pure service" in which "ready to submit for publication" output is given to the coworker. Thus, everyone is required to write a report, format the data for publication, print various views of the molecule, analyze the results, etc.

3. Many papers will involve the comparison of two similar crystal structures. As such, the atoms in each compound should be numbered in a similar and sensible fashion. You want to avoid sentences and tables that force a reader to compare the C(24)-O(7)-C(86) angle in one compound with the equivalent C(1)-O(1)-C(2) angle in another. The numbering system is very difficult to change in some crystallography programs, so inform the crystallographer in advance.

4. Coworkers should take appropriate steps to ensure that the crystal submitted does not correspond to starting material, NaCl, etc. This wastes everyone's time as well as money.

THE CIF and checkCIF FILES

A CIF (.cif) file is generated when a structure solution is complete. All journals require that this be submitted as supporting information. Starting in 2012, many journals require that structure factors be included in all CIF files, in order to facilitate the detection of fraudulent crystal data.

Journals also require a checkCIF file, easily generated from a CIF file: http://checkcif.iucr.org/. This raises various alerts to potential problems in the data set. Any "A level" alerts are regarded as serious, and journals require that the crystallographer respond to these (these comments can be included in the cover letter, but some journals may require that they be added to the CIF file. For *Organometallics*, see http://pubs.acs.org/paragonplus/submission/orgnd7/orgnd7_authguide.pdf.

When there is more than one crystal structure in a submitted manuscript, the CIF files must be combined; see http://pubs.acs.org/page/orgnd7/submission/cif.html.

THE NARRATIVE PARAGRAPH

This is page one of the report. It goes into the experimental section of the manuscript and is analogous to what a preparative chemist writes for the synthesis of a new compound. Guidelines are as follows:

1. The first sentence of the narrative paragraph must clearly indicate how the crystals were obtained. The temperature and time scale can be important, and should be specified.

2. Before the days of CCD detectors, microanalyses and density determinations of crystals were required. This information is still useful, particularly if the crystal contains solvent cules that were not present in the material isolated by the "official synthetic procedure".

3. The narrative paragraph should avoid duplicating information given in the crystallographic tables.

4. The next sentences of the narrative paragraph will normally contain the following "routine information":
   - 4.1 how the cell constants were determined (optional: determination of space group);
   - 4.2 corrections applied to the data (footnotes to necessary software);
   - 4.3 methods used to solve the structure (footnotes to necessary software);
   - 4.4 a clear statement about which atoms were refined anisotropically/isotropically;
   - 4.5 how hydrogen atoms were treated (calculated vs. located; refinement style);
   - 4.6 literature sources of scattering factors and similar quantities used.

5. The narrative paragraph must address any of the various non-routine situations that may apply:
   - 5.1 mention any special molecular symmetry, such as a C_2 axis, inversion center, etc.
   - 5.2 mention any significant decay during data collection
   - 5.3 mention the presence of more than one independent molecule in the unit cell.
   - 5.4 mention of any solvent molecules, in particular any disorder or partial occupancy requiring modeling/refinement. The ratio of solvent molecules to the molecule of interest must be clearly stated (e.g. **1**·$(CH_2Cl_2)_2$·$(THF)_{0.5}$), and not put in terms of a nebulous "asymmetric unit".
   - 5.5 information on any disorder, and how it was modeled/refined.
   - 5.6 with non-racemic compounds, how the absolute configuration was determined (include literature reference to method).
   - 5.7 a detailed description of how NO, CO, and CN ligands that are related by a pseudo-symmetry-element are differentiated ("told apart"), and other groups of atoms that have analogous pseudosymmetric relationships.

ORTEP/NUMBERING DIAGRAM

Page two of the report should be an ORTEP diagram ("thermal ellipsoid plot") of the molecule in which all key atoms are numbered. Special points:

1. include a separate diagram for every independent molecule in the unit cell
2. it is not necessary to include both cations and anions, unless there is hydrogen bonding
3. solvent molecules are optional, unless there is hydrogen bonding
4. elements should be colored according to the following table.

element\color	red	green	blue	color
hydrogen	0	0	0	white
boron	255	181	181	pale pink
carbon	145	145	145	grey
nitrogen	177	177	255	blue
oxygen	255	0	0	red
fluorine	194	255	0	green
silicon	209	214	140	pale green
phosphorus	255	170	0	orange
sulfur	255	199	48	yellow

chlorine	0	255	0	green
arsenic				
bromine	190	130	60	pale brown
iodine				
manganese	255	0	255	magenta
copper	204	77	26	copper
rhodium	255	0	255	magenta
tungsten	255	0	255	magenta
rhenium	255	0	255	magenta
platinum	255	0	255	magenta
gold	252	107	0	gold

4. always show the "proper enantiomer" of a racemic rhenium complex (separate handout).
5. some referees are very insistent that the caption contain a statement along the lines of "50% probability level" (e.g., "Themal ellipsoid plot of **23** (50% probability level)".

TABLE OF GENERAL CRYSTALLOGRAPHIC DATA (Table 1)

Examples can be found in recent publications. They should be edited to the ACS (or VCH) journal style, and must conform to the group formatting guidelines.

These are somewhat more work to prepare when several compounds are combined, especially if they come from different crystallography labs. If you are having any difficulty, seek out a trained crystallographer.

OTHER TABLES OF CRYSTALLOGRAPHIC DATA

The traditional series of Tables is given below, but nowadays the uploaded CIF file can take their place.

Table 2. Atomic Coordinates for .....
Table 3. (Bond) Distances and Angles for ...
Table 4. Anisotropic Displacement Parameters for ....
Table 5. Hydrogen Coordinates and isotropic displacement ...
(exact title depends upon how hydrogen atoms were treated)
Table 6. Torsion Angles for .....
(torsion angles should always be determined, but this usually lengthy table is not always required; see JAG)

INCORPORATION OF ADDITIONAL DATA

About half of our structures require additional minor calculations. Typical examples are:

1. Distances between two non-bonded atoms: add these to Table 3.
(see comment on caption below)
2. Closest distances between cations and anions: add anything less than 3 Å to Table 3 (is there a hydrogen bond?)
3. Sometimes we need "angles between two planes" or "deviations of atoms from a least squares plane". This may be given as an extra table, or in an extra paragraph following the narrative paragraph.

FREQUENT TYPES OF MISTAKES

1. If a compound **XX** was solvated, we did not determine "the crystal structure of **XX**". Rather, we determined the crystal structure of "**XX**•$(solvate)_z$"

2. There are semantic issues like "bond lengths" vs. "bond distances" (equivalent and a matter of taste/style). But suppose Table 3 contains a distance between two non-bonded platinum atoms. Then it is not a table of "bond distances" but rather "interatomic distances" or just "distances".

3. Caption errors on ORTEPs (thermal ellipsoid plots) are common. For a neutral molecule, it is hard to go wrong ("Molecular structure of **XX**" or "Crystal structure of **XX**", etc.). Since the PF_6^- anion in a salt is seldom of interest, most authors would only show the cation (unless there is a hydrogen bond). Here, "Structure of the Cation of **XX**" of "Thermal Elipsoid plot of the cation of **XX**" would be the correct captions.

4. When there is an inversion center within a molecule, atoms with unusual labels will appear in the output (e.g., C(6) and C(6)A). Do not change these to "primes" ('), this has a special crystallographic meaning. The "plain English" sense of the footnote that appears on the output is as follows: "Atoms labeled with a "#" suffix are solved via the following symmetry transformation: -x+3, -y-1, -z+1."

5. No table should ever be retyped, **ever**. If there is a problem with an electronic file, a disk crash, or the like, ask the crystallographer for a new electronic file.

CLEAN-UP WORK (after JAG reviews the "final report")

1. We maintain a "data bank of solved structures" on a group disk. A chemdraw structure and minor data entry (trivial name, formula, space group) are required. Make sure that the lab book entry be recorded. JAG periodically updates the data bank to indicate published structures.
   (see the group duty list for the person responsible for the data bank)

2. Deposit the final electronic files
   - 2.1 the narrative paragraph (word file), which may be combined with various usual tables
   - 2.2 any other tables (word files)
   - 2.3 the CIF and checkCIF files and responses to A level alerts
   - 2.4 all of the thermal ellipsoid (ORTEP) diagrams, ideally as editable adobe illustrator files, and any graphics in other formats (e.g., space filling representations)
   - 2.7 the Chemdraw file

3. Make certain that you have carried out a viewing session of both the molecule and lattice with JAG.