Information and Suggestions on Presentation of the Results of Crystal Structure Studies

by Professor J. A. Ibers

Abstract

The abstract is a very important part of the paper, both for the busy reader and for eventual data retrieval processes. The abstract should contain the following information:

1. Name and formula of compound(s) studied.

2. Unit cell information on the compound, including space group, *Z*, and measured and calculated densities.

3. A brief statement concerning data collection – for example, 2130 intensities above background were collected by counter methods.

4. A brief statement concerning refinement – for example, the structure was refined by least-squares methods to a conventional R factor of 6.3%.

5. A succinct description of the overall structure. Thus if this is a molecular complex, say so, indicate the molecules are monomeric, that the coordination around the central Fe atom is trigonal bipyramidal, and that the carbonyls are in the basal plane, the triphenylphospines at the apices.

6. A few details on distances or angles of specific interest, including their errors.

7. The relation of the structure to unusual physical or spectroscopic properties.

Experimental Section

1. Give the chemical formula of the compound. If the compound contains unusual ligands (*e.g.*, large organic ligands) then it may be desirable to present a structural representation of the ligand.

2. Give the unit cell dimensions and their errors. State how the errors were obtained. Give the temperature at which the cell was measured. State the radiation used and the wavelength used for the radiation.

3. Give the density calculated for *Z* molecules in the cell. State *Z* explicitly. In making this calculation, be sure that the atomic weight scale agrees with the value used for Avogadro's number. Give the observed density, with estimated error, and indicate how it was measured. If an observed density could not be obtained, state why. On the basis of *Z* and the order of the space group, state specifically any implications of molecular symmetry that are imposed by the crystallographic symmetry.

4. Give the systematic absences and how these were observed (*e.g.*, Weissenberg films of hk0, hk1, hk2 with CuKα radiation). State which space groups are consistent with such absences. If more than one space group is consistent, *e.g.* P2/m and P2, indicate how the choice was made. State whether or not a piezoelectric effect was looked for and what the result was. State whether or not the habit of the crystal is consistent with the choice of space group.

5. If the space group setting is unconventional, *e.g.* I2/a, give the general equivalent positions somewhere in the text. If the origin of the space group is arbitrary (*e.g.* $P2_1$) state explicitly where the origin was chosen. If there are many different types of atoms in special positions in the higher symmetry space groups, then it is desirable to state the Wyckoff notation as well. Example: space group T^3-123; Cu in (2a), Br in (6b), S in (12e), etc.

6. Give specific details on how data were recorded and measured.

(a) If intensities were recorded photographically, give information on type of camera, radiation used, filter used, method of estimation of intensities, method of assigning weights to the reflections, method of scaling the data to a common scale.

(b) If counter data, provide specific information on type of instrument, method of centering reflections, scan technique, if any, extent of scan, method of counting or estimating background, radiation used, filter or filters used, attenuators used (or some statement that coincident losses were negligible), distance of counter from crystal, distance of crystal from source, counter aperture size, take-off angle used, mosaicity of crystal, method of estimating errors in intensities, whether or not equivalent reflections were collected, definition of when a reflection is above background.

(c) In both cases describe the size and habit of the crystal used and how it was mounted with respect to the spindle axis.

7. Give the linear absorption coefficient for the radiation used. Provide information on the absorption correction, if one was applied. Note that the statement "Absorption corrections were neglected because μR was less than 0.5" is uninformative. Give estimates of the maximum effects on the intensities that the neglect of absorption causes.

Refinement and Structure Solving

1. Outline the method of solving the structure, but do not go through each step in agonizing detail, unless there is a specific moral to be stated.

2. Indicate what computer programs were used and be specific about their names, *e.g.* "ORFLS".

3. Indicate the source of atomic scattering factors.

4. Discuss in specific terms the handling of anomalous dispersion. Thus were f′ and f″ included in F_c, what was the source of f′ and f″, etc. This is particularly important for heavy-atom problems in polar space groups.

5. If the data were refined by least-squares techniques, then state specifically the function that was minimized. Define the R and weighted R factors. Be specific about the weighting scheme. What weights were applied to unobserved reflections. Provide information on R and weighted R throughout the course of the refinement. At the end give the standard deviation of an observation of unit weight.

6. Discuss the handling of the extinction problem, or reasons for ignoring it.

7. If an anisotropic refinement was carried out, be sure to give R and weighted R before and after. Be sure to state the form of the thermal ellipsoid. Be sure to state for film data how the scaling of the data was accomplished, keeping in mind that a complete anisotropic refinement will be singular if data are collected around one axis only and if the scale factors are also refined. Justify the anisotropic refinement on the basis of the weighted R before and after, and also on the basis of physical reasonableness. Avoid at all cost meaningless tables of random numbers of anisotropic thermal parameters. Give the root-mean-square amplitudes of vibration, and, if the data seem particularly good, the direction cosines of the thermal ellipsoids. If at all possible, provide a visual representation of these ellipsoides in the figures, for example through the use of Johnson's ORTEP program.

8. Provide an independent assessment of the correctness of the structure. Thus if the structure has been refined by least-squares techniques, compute a final difference Fourier and state the heights of major peaks on it, both in e/Å^3 and in fractions of the heights of various atoms in the structure.

9. If a least-squares plane is put through a series of atoms, be sure to specify the coordinate system to which this plane applies, whether or not the plane calculation takes into account the errors in coordinates (is it a weighted least-squares plane or not) and be sure to give the estimated standard deviations of the distances of atoms from the plane.

Presentation of Results

1. Well-planned figures will obviate the need for long discussions. Figures should have the appropriate atoms labelled. The directions of cell edges and the position of the origin should be clearly shown. This journal will happily publish stereo pairs, and these can be remarkably effective. Most figures should be planned for a single-column width (77 mm wide) and it is essential that the lettering be legible on photoreduction of the drawing to this width. (Height of lettering of 5 and 3 mm allows reduction to $^1/_3$ and $^1/_2$, respectively.

2. Be economical in the ways in which data are presented. Thus extensive tables of meaningful anisotropic thermal parameters should be photoreduced. Moreover, some style in the presentation of parameters and their standard deviations should be adopted that does not occupy too much space. For example, the x of C_1 can be given as 0.1962(12) rather than 0.1962 ± 0.0012, providing that there is a footnote to indicate what the notation means. Here, and elsewhere, be sure to state what the ± 0.0012 means (is it a standard deviation, a probable error, a maximum conceivable deviation, etc.) and how this number was obtained. (Thus for errors of bond distances, has correlation of parameters been taken into account.) The temptation to give a general description of errors is to be avoided. Thus the statement "Errors on carbon atom coordinates vary between 0.0012 and 0.0030" conveys too little information.

3. If a correction is made for thermal motion, then both the uncorrected and corrected bond distances should be given, and the specific formulation of the correction should be stated.

Structure Factor Tables

1. This table should be designed to fit into a space 16 cm wide by 21 cm long. This will occupy a journal page and leave 1.0 cm for title and heading. Such a table should contain about 1600 entries of h, k, *l*, F_o, and if desired, F_c. A table with fewer entries should fit in proportionally less space. Thus a table containing a 1000 entries should fit into a space 16 cm wide by about 13 cm long. Various economies are easily exercised in the production of this table. These include the use of a running index or running indices, elimination of the decimal point by scaling, etc. One should not give more significant figures than are justified. Thus if the R-factor is 10% on F, then entries of F_o and F_c of 300.3 and 301.6 should be replaced by 30 and 30 under a heading giving $10F_o$ and $10F_c$. Unobserved reflections may be entered into this table, with some notation indicating that they were not used in refinement. However, if 5000 data were collected and 4000 were unobserved, then one should indicate that of the 4000 unobserved reflections, only 10 exceeded 2σ above background, or some similar statement. It is important that the scale of the data in this table be indicated.

2. The general experience is that a well-planned table of 1600 entries per journal page will require a photoreduction of about 3:1 from standard listing machine output, and will be readily readable, even for myopic referees. If one is coming from tape or cards to an off-line printer, then it is sometimes convenient to print the table as a one-column table with no space control on the printer. Then this one column can be cut up and pasted on cardboard for eventual photographing and reduction. If one is using a printer that gets heavy usage, then better tables will result if the printing is done in the middle of the platen. It is an easy matter to request that the glossy print be made with high contrast.