NOTICE TO AUTHORS

Publication of *X*-Ray Crystallographic Work in the Journal of the Chemical Society

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In September 1982 a working party was set up by the Society's Journals Committee to consider updating the Society's approach to the publication of crystallographic structure determinations. This working party has received and evaluated comments from over thirty chemists and crystallographers including members of the Society's Chemical Crystallography Group, and has produced the following set of guidelines, which are framed as a direct replacement for section 5 of Instructions for Authors (see *Perkin Transactions*, 1983, Issue 1; *Dalton Transactions*, 1983, Issue 3). These guidelines were approved by Journals Committee in June 1983, and should be followed as from 1st January 1984 by all authors submitting crystallographic results for publication.

5.0 Publication of *X*-Ray Crystallographic Work

Crystallographic work will be assessed mainly for its chemical interest. Thus crystallographic work carried out as part of a wider chemical study should not normally be submitted for publication separately from the results of that study. However, papers reporting only the results of crystal structure determinations may be accepted for publication provided that these results are considered to possess specifically chemical significance.

The description of a crystallographic structure determination should be as brief as possible, consistent with the following guidelines, and should be included at the end of a paper (or at the end of the Experimental section, if this precedes the Discussion). If the procedures for data collection and structure analysis were routine, their description should be particularly concise.

5.1 Title and Summary

The title of a paper reporting a crystallographic structure determination should normally contain the words ' Crystal Structure of . . .'

The summary need not contain cell dimensions and other crystal data, but should make clear that a crystal structure analysis has been performed.

5.2 Presentation of Crystal Data

The following sequence should be employed (although not all items will be required in all cases): (1) chemical formula and formula weight (M); (2) crystal system; (3) unit cell dimensions (Å or pm, degrees) and volume, with estimated standard deviations, method of determination, and temperature (if not ambient); (4) type and wavelength of radiation used; (5) space group symbol (if non-standard give related standard setting); (6) measured density (D_m), no. of molecules in unit cell (Z), and calculated density (D_x) (with any implications for ' molecular symmetry '); (7) colour, size, and shape of crystal used for data collection; (8) linear absorption coefficient (μ).

5.3 Data Collection and Processing

The description of data collection should contain sufficient information for the reliability of the determination to be assessed. The following should normally be included:

(1) Type of diffractometer, measuring mode, and temperature if not ambient (with reference if possible to an earlier full description).

(2) Theta range and reciprocal lattice segment.

(3) Number of data measured, number observed, and $I/\sigma(I)$ criterion used (if more than one asymmetric unit is recorded, the merging R value should be given).

(4) Absorption correction and method used (with reference).

(5) Stability of intensity controls and any action taken.

5.4 Structure Analysis and Refinement

The information provided should fulfil the same criteria as for data collection (section 5.3). This would normally include:

(1) Method of solution (direct, heavy atom, or combination) and development.

(2) Mode of refinement (full matrix, blocked, block diagonal, anisotropic, isotropic, or mixed), treatment of hydrogen atoms, and weighting scheme.

(3) Final values of R and R_w (and their definitions), with a statement of whether unobserved reflections were included (and if so, how), and if possible some estimate of the accuracy of the F_o data.

(4) Programs or packages and computers used (with references) and source of f data (also f', f'').

(5) Range of standard deviations for bond lengths and angles.

(6) Any procedures used for definition of chirality (quotation of a single Hamilton ratio without specifying the R values from which the ratio was derived, and the conditions under which these were obtained, will not normally be acceptable).

When the analysis has not been of a routine nature, authors should explain concisely all procedures used.

5.5 Example of Presentation

The following example demonstrates the application of the recommendations in the preceding sections (5.2 to 5.4):

Experimental

Crystals were prepared as described in ref. 11 and sealed under nitrogen in Lindemann capillaries.

Crystal Data.—$C_{24}H_{26}Cl_3Re_3Si_6$, M = 1 028.0. Monoclinic, a = 12.021(3), b = 20.489(5), c = 18.194(5) Å, β = 91.03(3)°, V = 4 480 Å^3 (by least-squares refinement on diffractometer angles for 15 automatically centred reflections, λ = 0.710 69 Å), space group $P2_1/n$ (alt. $P2_1/c$, No. 14), $Z = 4$, D_x = 1.76 g cm^{-3}. Dark blue, air-sensitive tablets. Crystal dimensions (distance to faces from centre): 0.313(211,$\bar{2}\bar{1}\bar{1}$) × 0.163(010,$\bar{0}\bar{1}\bar{0}$) × 0.063(101,$\bar{1}\bar{0}\bar{1}$) mm, μ(Mo-K_α) = 85.12 cm^{-1}.

Data Collection and Processing.[12]—CAD4 diffractometer, $\omega/2\theta$ mode with ω scan width = 0.85 + 0.35 tan θ, ω scan speed 1.3—6.8 deg min^{-1}, graphite-monochromated Mo-K_α radiation; 6 383 reflections measured ($1.5 \leqslant \theta \leqslant 25°$, $+h,k,l$), 5 797 unique [merging R = 0.024 after absorption correction (max., min. transmission factors = 0.37, 0.10)], giving 4 170 with $I > 2\sigma(I)$. Linear and approx. isotropic crystal decay, *ca.* 37%, corrected during processing.

Structure Analysis and Refinement.—Direct methods (Re and Cl atoms) followed by normal heavy atom procedures. Full-matrix least squares refinement (in two blocks for final, anisotropic cycles) with all non-hydrogen atoms anisotropic and hydrogens in calculated positions with one, overall, refined μ_{iso} [= 0.08(2) Å^2]. The weighting scheme $w = 1/[\sigma^2(F_o) + 0.0078F_o^2$, with $\sigma(F_o)$ from counting statistics [12] gave satisfactory agreement analyses. Final R and R_w values are 0.062, 0.064. Programs and computers used and sources of scattering factor data are given in ref. 12.

References

11 Ref. to prep of compound.

12 For full details of experimental techniques used see ref. to previous paper giving full details of data collection procedures *etc.* used in author's laboratory.

5.6 Presentation of Tables and Diagrams

A clear distinction should be made between material to appear in print and material for deposition as a Supplementary Publication (see section 4.0).

5.6.1 *For Publication in the Journal.*—(1) A table of final *fractional atomic co-ordinates* (labelled x, y, z) must be included. If the origin chosen differs from that adopted in International Tables this should be mentioned and justified. Where the asymmetric unit consists of a discrete molecule (or molecules) co-ordinates should refer to atoms which are all in the same molecule. Hydrogen atom co-ordinates should be included only when experimentally determined or refined; when they have been determined only by calculation, deposition is appropriate (but not mandatory).

(2) A table of selected *bond lengths and angles*, with estimated standard deviations. This should be restricted to significant dimensions only (for example it is rarely necessary to include data for phenyl rings). Average values may be given (with a range of e.s.d.s) for chemically equivalent groups or for similar bonds. As an alternative to tabular presentation it is often clearer to give important dimensions in a structural diagram. Differences from expected norms should be noted.

(3) A conventional *line drawing* of the structure must be included except in the simplest cases and one *perspective diagram* (or *stereo pair*) if appropriate. Packing diagrams should not be included unless required to illustrate a specific chemical point. The *atom numbering system* should be clearly shown in one of the diagrams. Any differences from that required by standard rules of chemical nomenclature should be pointed out. Each atom of the asymmetric unit should be assigned an arabic numeral in parentheses following the chemical symbol: C(2), O(1″), *etc.*; it is often convenient to associate a particular number of primes with a particular asymmetric unit. Alternatively, roman numeral superscripts can be employed: $C(2^{I}) \cdots C(2^{IV})$.

5.6.2 *For the Referees and/or for Deposition.*—(1) Any *calculated co-ordinates* (*e.g.* hydrogen).

(2) A full list of *bond lengths and angles* with estimated standard deviations.

(3) A full list of *thermal parameters* in the form B_{ij} or U_{ij} (in Å^2 or pm^2).

(4) Details of any *least squares planes* used to provide parameters for the paper.

(5) A legible table of *structure factors* (F_o, F_c) (for the referees only; not for deposition).*

5.6.3 *Deposition of Material at the Cambridge Crystallographic Data Centre.*—The table of final *fractional atomic co-ordinates* and the full list of *bond lengths and angles* will also be deposited at the Cambridge Data Centre (or at the University of Bonn for molecules not containing 'organic' carbon). These tables may be submitted as computer printout; the data need not be retyped if clearly legible. However, a print program should be used which yields concise tables of atomic and derived parameters. The content of the tables should be non-redundant and their interpretation immediately obvious.

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* Authors should preserve copies of structure factor tables in their own records. During 1984—85 the Society will also keep copies in the editorial office (photocopies available on request), in order to assess the demand for this material.