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Primary Crystallographic Data*

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A report containing a list of recommendations on the presentation of crystallographic data in primary publications relating particularly to single-crystal work. The more important items of information are discussed in detail with examples. Numerical values of certain constants in common use are recorded.

The increasing number of crystallographic publications, not only in *Acta Crystallographica*, but also in many other journals, has prompted the Commission on Crystallographic Data to draw up the following set of recommendations. The object of these recommendations is to ensure that essential information is given and to suggest a concise arrangement with standardized symbols. Also included are references to the latest numerical values of X-ray wavelengths and of Avogadro's number, for use with the ^{12}C scale of atomic weights. Flexibility in using the standard form is desirable and, in special circumstances, modifications of these recommendations may be necessary.

Previous recommendations were proposed by: Bernal, Ewald & Mauguin (1931); McCrone (1948, 1956); and The Chemical Society of London (1959). Relevant information is also contained in the three volumes of *International Tables for X-ray Crystallography* (1952, 1959, 1962) and in *Crystal Data* (Donnay, Nowacki & Donnay, 1954; Donnay, Donnay, Cox, Kennard & King, 1963). Attention is particularly directed to *Notes for Authors* (1965) and to the *Recommendations of the Commission on Crystallographic Computing* (1962).

For convenience the recommendations will be presented under three headings: (I) *Crystal data*, (II) *Structural data* and (III) *Structure factors*.

I. Crystal data

In this section we wish to suggest a convenient sequence for the essential items of information. Not all entries are applicable in every case; some may be omitted.

Standardized layout

Name(s) of substance. Chemical formula(e). Formula weight F.W. Melting point M.P. Provenance and size of crystals. Crystal system. Goniometric data. Crystal forms. Point group (but only if indicated by morphology). Habit, including twinning if observed. Cell parameters a, b, c, α, β, γ, as necessary (with standard deviations), at stated temperature. Cell volume V (with standard deviation). Measured density D_m in g.cm^{-3} (with method of measurement and limits of error). Number Z of formula units per cell. Calculated density D_x. Type(s) of X-rays used. Numerical value of wavelength used in calculating cell dimensions. Brief note on X-ray methods. Absorption coefficients for X-rays used for intensity measurements. Size of crystal used for such measurements. Total number of observed reflexions, and percentage of radiation sphere explored. Unusual correction factors. Space group. General position coordinates.* Any atoms in special positions. Any implications for molecular symmetry. Anomalous dispersion. Optical data (with wavelength of light used and temperature).

Notes

(1) *Name of substance.* For organic and inorganic compounds, as far as possible the IUPAC rules of chemical nomenclature should be used. Accepted trivial names, which facilitate identification, should also be given, as synonyms. In case of a new compound, the name originally used should be given, together with a reference to the chemical work. For minerals, the recommendations of the International Mineralogical Association are to be followed. Polymorphic forms should be distinguished. (Do not use the word *form* or *modification* to refer to a hydrate or to a twin).

(2) *Chemical formula.* For organic compounds a structural formula should accompany the name, except possibly with simple molecules. In addition, a formula

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* According to current English usage, it is cumbersome to differentiate between a point x,y,z ('position') and the collection of equivalent points ('set of positions'), for which a singular collective noun would be highly desirable. In this report we shall use *site* to designate the point itself and *position* to mean the point set; these terms correspond to the German *Punkt* and *Punktlage* respectively and to the French *point* and *position* respectively.

that can be typed on a single line (such as CH_3CO_2H) is desirable. The alphabetized formula, in the form $C_xH_yA_pB_qD_r\ldots$, should also be given, as a check and to help the indexer. Any abbreviation should be explained (*e.g.*, φ stands for C_6H_5).

For inorganic compounds the formula should be written so as to reflect the conclusions derived from the crystal structure. Use of the dual formula, expressed in terms of oxides, is to be discouraged.

In case of significant incomplete site occupancy, give the non-stoichiometric formula.

For a phase of variable composition, give the formula of the specimen actually studied; for example, $(Fe_{1-x}Ni_x)_2O_3$ as $Fe_{1{\cdot}34}Ni_{0{\cdot}66}O_3$ or $(Fe_{0{\cdot}67}Ni_{0{\cdot}33})_2O_3$. Note that $(Fe_{1{\cdot}34}Ni_{0{\cdot}66})_2O_3$ is not acceptable.

(3) *Formula weight* (*or molecular weight*) F.W. Use the unified scale of atomic weights, based on ^{12}C = 12·0000. Make sure that F.W. refers to the formula actually given. For complicated mineral structures the formula weight of the whole cell is often preferable.

(4) *Point group.* When the point group has been determined from morphology, it should be given, in its oriented Hermann–Mauguin symbol. Usage of the Groth names, based on the general forms, to designate point groups is to be discouraged.

(5) *Habit.* The following adjectives are recommended: acicular, long prismatic, prismatic, short (or stout) prismatic, equant, thick tabular, tabular, thin tabular (or platy), leafy, flaky, pyramidal, dipyramidal and fibrous. The symbol for a face (· · ·) or for an edge [· · ·] follows where appropriate. Thus 'tabular (010)' means that (010) is the largest face, whilst 'prismatic *b*' means that the crystal is elongated in the *b* direction. Note that in this case of an axial direction, the designation *b* is preferred to [010]. 'Lath-shaped (010), elongated *c*' is self-explanatory.

If twinning is observed, it should always be described. For recommended nomenclature consult *International Tables* (1959, Vol. II, section 3). (Note especially that a twin is a heterogeneous edifice composed of two or more crystals – and not the other way round.)

(6) *Cell parameters.* Except for some good reason, the cell to be chosen should be the Bravais-reduced cell, *i.e.* the cell that has its edges along the shortest three lattice translations or lattice symmetry directions whenever such are available. [Donnay, 1943, 1952; see Preface to *Crystal Data* (Donnay, Donnay, Cox, Kennard & King, 1963); *cf.* also *International Tables* (1952, Vol. I, pp. 530–5) for the Delaunay cell]. Note that a triclinic Delaunay-reduced cell may or may not coincide with the Bravais-reduced cell. In every crystal system the set of coordinate axes should be right-handed. The cell can be uniquely oriented according to the following rules: In the triclinic system, choose $c<a<b$ with α and β non-acute.* In the monoclinic system, select the shortest two translations in the net perpendicular to the symmetry direction *b*, take $c<a$, β non-acute, and use appropriate centring. In the orthorhombic system, choose $c<a<b$ and appropriate centring if one-face-centered (*A*, *B*, or *C*). In both monoclinic and orthorhombic systems, this choice may result in a setting different from that used, for illustrating the space group, in *International Tables* (1952), in which case the coordinates of the equivalent sites in the necessary positions will have to be specified. Inasmuch as *International Tables* does not list the coordinates of equivalent points in all possible settings, authors of papers describing crystal structures may find it easier to follow the setting of *International Tables*.

In the high-symmetry systems, the choice of the shortest three lattice translations uniquely defines the cell. A tetragonal cell should be taken as *P* (not *C*) or *I* (not *F*); a hexagonal cell should be primitive; its symbol is now *P* (formerly *C*); the triple cell *H* is not used. In the cubic system, the cell is always a cube (*P*, *I*, or *F*).

If the crystal is better described by means of another cell, the conventional cell should also be given, together with the transformation matrix (from-unconventional-to-conventional). To save space the matrix rows may be given in linear form $uvw/u'v'w'/u''v''w''$. A program, written in FORTRAN, is available to perform this transformation (Takeda & Donnay, 1964).

(7) *Cell dimensions and standard deviations.* Lengths should be given in Å (not kX), angles in degrees and minutes or in degrees and decimal fractions. The cell volume should be given in Å^3.

Pending decision of the International Union of Crystallography, the numerical values of X-ray wavelengths remain those published in *International Tables* (1962).†

All limits of error should be given in the form of a standard deviation σ; *e.g.*, 12·431 ± 11, which means ± 0·011 (*International Tables*, 1959, pp. 85–91). For the given uncertainty to qualify as a standard deviation, it must have been calculated by a least-squares treatment; for example, of the $\sin^2\theta$ values. If the uncertainty given differs from one standard deviation – if, for instance, it has been multiplied by three – this should be made clear: '12·431 ± 33 (three standard deviations)'. Optimistic guesses at the accuracy attained are to be shunned.

(8) *Number Z of formula units per cell and calculated density D_x.* The number *Z* must refer to the formula already specified, with F.W. expressed on the ^{12}C scale. For Avogadro's number, use $N_A = 0{\cdot}602252 \times 10^{24}$; its reciprocal is $1{\cdot}660435 \times 10^{-24}$ (*National Bureau of Standards, Technical News Bulletin*, 1963). Note that

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* In the low-symmetry systems the orientation of the cell is a matter of convention. (Note that, from the viewpoint of systematization, any cyclic permutation, such as $a<b<c$ with β and γ non-acute, is equally acceptable.)

† Note, however, the new values of Bearden (1964). They are expressed in Å*, a unit that is equal to the Å within a few parts per million (probable error), and is defined by the new primary standard for X-ray wavelengths, $\lambda(\mathrm{W}\,K\alpha_1) = 0{\cdot}2090100$ Å*. Their order of accuracy cannot be attained in most crystallographic work.

the new values differ from those in *International Tables*, 1962, pp. 39–45).

(9) *Type*(*s*) *of X-rays*. The nature of the radiation used for intensity measurements should be specified (example: Ni-filtered Cu radiation). The numerical value of the wavelength of the radiation used to determine the cell dimensions should also be reported, to facilitate future corrections should the numerical values change.

(10) *Observed reflexions*. When the observed reflexions conform to the space-group criteria in the orientation of *International Tables*, no statement is needed. If the space group is presented in another orientation, the presence criteria should be stated. They should also be given when they show systematic anomalies.

(11) *Space group*. When the space group is unambiguously indicated by the observed reflexions and the choice of axes is that of the *International Tables*, simply give its oriented Hermann–Mauguin symbol.

When the choice of axes is not that of *International Tables*, the Schoenflies symbol should be added as a means of identifying the space group. Thus *Pnaa*, which appears in *International Tables* as *Pccn* (no. 56), should be followed by D_{2h}^{10}. The matrix of the transformation will also be useful. From *Pnaa* to *Pccn* = 010/001/100.

When the observed absences are compatible with two or more space groups, the use of a condensed diffraction symbol (see Donnay & Kennard, 1964) is recommended. Thus the various possibilities *Pmmm*, *P2mm*, *Pm2m* and *Pmm2* are all covered by the symbol $P***$. When one of the space groups has been chosen, give the reasons for the choice. For example: *Pmmm* was adopted because a statistical study of the intensities indicated centrosymmetry, which was subsequently confirmed by the structure analysis.

(12) *General position*. The coordinates of the equivalent sites should be given only when the labelling of axes differs from that used in *International Tables* (1959). They should be put into the condensed form. For example, for $P2_1/n$: $\pm(x,y,z;\ \tfrac{1}{2}+x,\ \tfrac{1}{2}-y,\ \tfrac{1}{2}+z)$.

(13) *Optical data*. For transparent substances the values of the indices of refraction and the optical orientation should be stated preferably for specified type of light and temperature.

Examples:

Cubic: n (Na, 20°C) = 1·650 ± 2.

Uniaxial: n_E 1·650 ± 2, n_O 1·595 ± 2 (Na, 25°C).

If dichroic: n_E 1·650 ± 2 (colourless), n_O 1·595 ± 2 (blue).

Orthorhombic: Opt. neg. (5893 Å, 25°C): 1·500 (*b*), 1·74 (*c*), 1·77 (*a*), $2V$ 39°; disp. $r > v$, weak.

Monoclinic: Opt. neg. (5893 Å, 25°C): 1·578 ± 2 (*b*), 1·676 ± 2 (+41° to *c*), 1·710 ± 5 (+31° to *a*), $2V$ 58°; disp. $v > r$, strong. An extinction angle with *c* is taken as positive if in the obtuse angle β.

Triclinic: The optical orientation can best be presented by means of a stereographic projection. It may also be of interest to describe the optical properties of the cleavage planes and of the principal faces.

Pleochroism can be indicated after the values of the indices: Opt. neg.: 1·700 (*a*) (pale yellow), 1·750 (*b*) (golden yellow), 1·770 (*c*) (golden yellow).

Note that the given $2V$ (or $2E$) should be the *measured* value.

II. Structural data

(1) *Crystal-chemical unit*. To define the structure of a crystal, coordinates and other parameters must be stated for the atoms in one asymmetric unit (Schoenflies's *fundamental domain*). This asymmetric unit should be chosen so as to contain a chemical entity, even when this means giving, to some of the atoms, coordinates that are negative or improper fractions. Such a domain may be called the *crystal-chemical unit* (CCU). In a molecular compound, it may contain a single molecule, two or more molecules, half a molecule or less. In an ionic compound it may be only part of a cation and part of an anion (as in $PCl_4^+PCl_6^-$).

(2) *Numbering of atoms*. On a structural formula or diagram, the atoms may be numbered irrespective of chemical type [*e.g.*, O(1), O(2), C(3), C(4), C(5)] or according to chemical elements [*e.g.*, O(1), O(2), C(1), C(2), C(3)]. In either case the number should preferably be in parentheses, so as to avoid unwanted chemical implications (O_2, C_2).

(3) *Choice of origin*. The choice of the origin of coordinates should be clearly specified, particularly on the drawings.

(4) *Atomic coordinates*. Use fractional (or trimetric) coordinates x,y,z, defined by $X/a, Y/b, Z/c$, in which a,b,c are the cell edges and X,Y,Z the coordinates in Ångstroms (symbols of *Structure Reports*). Decimal points can be avoided by showing (say) 0·1907 as 1907, with $10^4\ X/a$ as column heading.

Ordinary coordinates (in Å) on orthogonal axes are useful when the symmetry is hexagonal, monoclinic or anorthic. Their symbols X', Y', and Z' must be defined; for example, in the monoclinic case: $X' = X\sin\beta$, $Y' = Y$, $Z' = Z + X\cos\beta$.

The published data should enable a reader to repeat the author's calculations: coordinates should, therefore, be given to one place (or possibly two places) beyond the last significant digit. Since the standard deviation will have been given, there will be no doubt as to which digits are significant.

(5) *Symmetry-related units*. It is often necessary to refer to atoms that lie outside the crystal-chemical unit. *Symmetry-related units*, including those repeated by lattice translations, are best designated by Roman numerals (preferably in lower case). Each of them contains one point equivalent to x,y,z. Example:

CCU	x,y,z
i	$x+\tfrac{1}{2},y-\tfrac{1}{2},z$
ii	$x+1,y,z$
iii	$x+\tfrac{3}{2},y-\tfrac{1}{2},z$

An intermolecular contact can be denoted as C(3) $\cdots$ O(2)(ii). Avoid using a continous line, which may suggest a chemical bond, between two non-bonded atoms.

(6) *Atomic scattering factors.* State the numerical values actually used. When the values given in *International Tables* (1962) are used, a statement to this effect is sufficient.

(7) *Thermal parameters.* If a single isotropic temperature factor e^{-M} has been used for all atoms, its value can be stated in the text. Individual Debye factors (B in Å^2) can be listed by adding a column to the table of coordinates. Individual anisotropic vibrational parameters require six parameters for each atom in a general position; they have been given in various forms. Definitions should be clearly stated. The exponential function in which the b_{ij} values appear, for instance, should be explicitly given. If the author lists the components U_{ij} of the tensor that expresses the ellipsoid representing the mean-square amplitude of vibration of the atom (Cruickshank, 1965), the axes of reference should be specified (a, b', c^*, for example, or some other orthogonal axes). The same remarks apply if librational or translational parameters are given for a molecule or part of a molecule acting as a rigid body.

(8) *Diagrams.* A good diagram is essential. It should show the whole, or a significant part, of the cell, with an indication of the symmetry elements. Such a diagram is a convenient place for giving the numbering of the atoms in the crystal-chemical unit and the numbering of the symmetry-related units.

The origin and the coordinate axes should be clearly marked and their positive senses shown by arrows. As in *International Tables* (1952), the axes should constitute a right-handed set. If feasible, place the origin of an orthogonal projection in the upper left-hand corner.

For example, if a triclinic cell is projected parallel to the c direction, onto a plane perpendicular to the c direction, the axes in the plane of the figure should be labelled b' and a', where $b' = b \sin \alpha$ is directed towards the right-hand side and $a' = a \cos \beta$ (approximately) towards the foot of the page. The positive c direction should be upwards (out of the paper); letting it point downwards (into the paper) has been a source of confusion. The three principal projections are illustrated in Fig. 1.

III. Structure factors

A paper reporting a successful structure analysis always includes a statement of the residual

$$R = \Sigma \, ||F_o| - |F_c|| / \Sigma \, |F_o|$$

(the treatment of non-observed reflexions should be specified). It is also customary to give a table of observed amplitudes $|F_o|$ and calculated structure factors F_c. There are strong arguments in favour of this procedure:

(*a*) The table is the ultimate evidence for the validity of the analysis.

(*b*) With these data, a future worker can always resume the analysis to check it, correct it, or carry out further refinement.

(*c*) The observed amplitudes constitute an important body of data, which it may not always be possible to remeasure. The crystal used may be the only single crystal in existence!

It may be argued that structure-factor tables occupy a good deal of space in a journal and are not of interest to the majority of readers. The problem is sometimes avoided by omitting the table of structure factors altogether, and substituting a statement that it may be obtained from the author or from the Library of Congress or other depository.

It seems desirable that structure factors should continue to be published until some better system of making data available is developed. Space may be saved by economical arrangement of the table. The table is clearer if only the currently changing index is listed in the first column with an indication whenever one of the other two indices is changed.

For a centrosymmetric crystal, the second and third columns will contain $|F_o|$ and F_c.

For a non-centrosymmetric structure, where phases have to be specified, the information can be given as $|F_o|$, followed by:

(1) A_c and B_c, the real and imaginary parts of F_c. (This carries the full information but has the disadvantage that $|F_c|$ is not explicitly given and cannot be compared with $|F_o|$).

(2) $|F_c|$, $\cos \alpha$ and $\sin \alpha$. (This requires one additional column).

(3) $|F_c|$ and α.

Structurally absent reflexions may be omitted provided the calculated values are also small. Any discrepancies should be discussed in the text.

Some bolder suggestions should be included in this Report. It has been pointed out (Lipscomb 1963, 1964) that only $|F_o|$ needs to be recorded, since a reader with access to a computer can recalculate F_c from the structural parameters. Space can be further saved, though with sacrifice of elegance, by placing the $|F_o|$ values

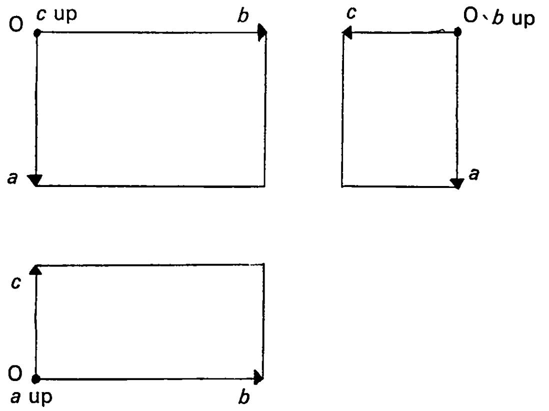

Fig. 1. The three principal projections with the conventional orientation of the axes.

serially along the line, with merely an indication of the changing indices.

In any case a large typescript table should be prepared (*e.g.*, as computer output) and considerably reduced to a photograph no larger than a full page in the journal. Such photographs (one or more) should be suitable for direct photographic reproduction. The editor of *Acta Crystallographica* reports that such a procedure greatly facilitates publication.

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Laue Ellipses and Reciprocal-Lattice Plane Packing Densities

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For each complete ellipse on a Laue photograph one can read off an inclination correction factor (ICF) from a chart and multiply it by the number (N) of reflexions counted on the ellipse. The product $D=N$. ICF is inversely proportional to the unit-cell area of the generating reciprocal-lattice plane and is often sufficient to identify the plane. Use is made of the facts that the reciprocal-lattice lines generating Laue reflexions are uniquely specified by their prime coordinate points in the reciprocal lattice (points with at least two coordinates prime to each other), and that these prime coordinate points are uniformly distributed over any n-dimensional lattice ($n \geq 2$).

Aim

Laue photographs seem to be used mainly to determine angles within the reciprocal lattice, or to find the orientation of a crystal. Another possible use is the identification of the generating reciprocal-lattice plane from its Laue ellipse, by means of counting the number of reflexions on the complete ellipse. It is shown below how this number, N, together with an inclination correction factor, ICF, obtained from a transparency superposed on the ellipse, can give a quick estimate of the relative inverse unit-cell area of the generating reciprocal-lattice plane; this is often sufficient to identify the reciprocal-lattice plane. It is also easy to calculate roughly the angular corrections required to swing that plane into, say, a horizontal orientation.

Terminology

Because complete Laue ellipses are required, a cylindrical camera with its wider angular range is better suited than a planar one; the term 'ellipse' will be retained, however. Reciprocal-lattice point, reciprocal-lattice line and reciprocal-lattice plane will be abbreviated to re point, relline and relplane, respectively.

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