cité entre la date de mise en service du montage (1978) et ce jour (1981).

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A technique for loading glass capillaries used in X-ray powder diffraction

The filling of thin-walled glass capillaries is tedious and time consuming especially when the powder tends to agglomerate due to extremely fine grinding (<10 μm) in a micronizing mill. While special devices have been designed for loading capillaries in a dry box (Larsen & Leddy, 1958; Lange & Haendler, 1972), a technique greatly facilitating routine loading is currently in use in our laboratory. It involves placing the capillary in a nearly upright position in a 50 ml Erlenmeyer flask filled with water. The best results were obtained when the water level in the flask was just below the base of the capillary funnel. The flask is then placed in an ultrasonic bath (filled to a depth of one inch with water) where the vibration promotes disaggregation and movement of the particles into the capillary. Occasional clogging of the capillary funnel during loading of highly agglomerating powders can be relieved by insertion of a fine wire probe during agitation. Loading, the rate of which depends in part on capillary diameter and particle size and density, normally requires less than five minutes of ultrasonic agitation. The technique yields a uniform, dense packing of powder with preferred orientation greatly minimized.

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Letter to the Editor

J. Appl. Cryst. (1982). **15**, 357–359

Suggested guidelines for the publication of Rietveld analyses and pattern decomposition studies

Sir,

At the request of the Commission on Journals, we drew up some draft guidelines for the publication of Rietveld analyses and of pattern decomposition studies with powder diffraction patterns. The draft was sent for comment to some 25 persons in Europe, Australia, Japan, and the USA. We are grateful for their responses, which both were generally supportive of the idea that there be guidelines and were most helpful in illuminating oversights and other deficiencies. Not all suggestions were incorporated in the revised draft, of course (in fact, a number were mutually contradictory), but all were carefully considered and many were incorporated in the version which follows.

In presenting these suggested guidelines, we emphasize that we offer them as guidelines, not rigid rules. They are intended primarily to be helpful to the Co-editors; they are not intended to infringe on a Co-editor's judgement of scientific worth of a submitted manuscript, nor should they be allowed to do so. For the most part, these suggested guidelines address matters of format and presentation of details, and not the fundamental question of scientific interest and worth of the submission. It is primarily for the making of such fundamental judgements that the Co-editor system exists; for the health of our science it cannot and should not be replaced with a system of blind rules on a check-off sheet. It is against this background of more overreaching considerations that we offer the following suggestions for guidelines to assist, but not to control or coerce, the Co-editors in their acceptance decisions.

Rietveld method

Definition. Whole-pattern-fitting of calculated to observed powder patterns through least-squares refinement of model(s) for the structure(s), diffraction optics effects, and instrumental factors. A key feature is the feedback, during refinement, between improving knowledge of the structure and improving allocation of observed intensity to individual Bragg reflections.

Suggested guidelines

(1) Deposit the digital data,* starting values of all parameters in the model, the beginning and ending 2θ (or equivalent) values, and the step size.

(2) Specify the instrument type, data type (including step-scan increment and metric), $\lambda(s)$, and monochromator or equivalent data for neutron time-of-flight (TOF) or X-ray energy-dispersive techniques used.

(3) Specify the instrument geometry affecting the instrumental profile. Some type of specification of the actual instrumental profile applicable in this study is desirable. The first paper using data from a particular instrument should contain a discussion of the actual reflection profile shapes observed for well resolved reflections free of diffraction-broadening effects.

(4) Sample
(*a*) Specifications:
(i) source of material, likely impurities, stability;
(ii) preparation, grinding, sieving, *etc.*;
(iii) sample container, dimensions, if sealed or not, precautions against preferred orientation.
(*b*) Environment:
(i) temperature control: relative and absolute precision, calibration method, internal checks, temperature gradient;
(ii) pressure: transmitting medium, measurement and calibration method;
(iii) materials in beam, scattering into counter.

(5) Refinement
(*a*) Computer program used:
(i) cite literature reference;
(ii) note availability if new or newly modified;
(iii) note any unusual features.
(*b*) Reflection profile representation(s):
(i) specify the function or numerical

*Some workers believe this should be optional if full pattern plots and difference plots are provided.

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representation, with reason for selection, and its refinable parameters;

(ii) specify how dependence on 2θ or other scanning variables, asymmetry, and anisotropy (in reciprocal space) were provided for.

(*c*) Background:

(i) if refined simultaneously, what function was used to represent it?;

(ii) if not refined simultaneously, how was it chosen and specified?;

(iii) if not refined simultaneously, what allowance was made for its contribution to the observational weights?

(*d*) Preferred orientation (this item is usually more important for X-ray than neutron studies):

(i) specify function used and its refinable parameters;

(ii) specify actual habits of the particles;

(iii) if no preferred orientation parameters were refined, state what tests were made to detect preferred orientation and their results.

(*e*) Specify how the weights were calculated for each observation ('y_i').

(*f*) Specify the method of calculation of standard deviations, unless that is adequately done in a published and referenced description of the computer program used.

(*g*) Specify lattice parameters, with σ's, and how they were obtained.

(*h*) Specify the structural model(s) being refined:

(i) state what the adjustable parameters in the models were, what constraint relations were used, and which structural parameters were fixed, why, and how their fixed values were chosen;

(ii) specify what precautions were taken to avoid ending in a false minimum, *e.g.* trying different sets of starting parameter values, different weights, *etc.*

(*i*) Also specify:

(i) the atomic scattering factors (lengths) used (including f' and f'');

(ii) the absorption coefficient, how absorption corrections were made, and the maximum correction;

(iii) the λ's and their σ's used in the refinement.

(*j*) Specify:

(i) omitted regions;

(ii) the range over which contributions from a given reflection were considered.

(*k*) How large were the last-cycle shifts relative to the σ's?

(6) Criteria of fit

(*a*) Visual:

(i) give an I (often referred to as y_i) *vs* 2θ plot, or equivalent plot for TOF and energy-dispersive studies, of the entire observed and (final) calculated patterns, superimposed Bragg-reflection markers are welcomed;

(ii) on the same or an enlarged scale, provide a plot of the difference.

(*b*) R's (see definitions at the end of these guidelines):

(i) give final R_{wp} obtained;

(ii) give the statistically expected value for R_{wp};

(iii) R_B is welcomed;

(iv) other R's, *e.g.* R_F and R_p, may be given in addition;

(v) give or cite definitions of all R's reported.

(7) List the final values of all refined parameters with their standard deviations. If the standard cell setting was not used, list the symmetry operations.

(8) Point out, and give the magnitude of the correlation matrix element for, the largest correlations involving any structural parameter.

(9) As for other good papers, assess the physical and chemical reasonableness and significance of the results.

(10) Nomenclature

Preferred: Rietveld analysis/method/refinement

Not acceptable: Profile refinement or profile analysis without the word 'structure'.

$$R_F = \frac{\sum|[I_B(\text{'obs'})]^{1/2} - [I_B(\text{calc})]^{1/2}|}{\sum[I_B(\text{'obs'})]^{1/2}}$$

$$R_p = \frac{\sum|y_i(\text{obs}) - (1/c)y_i(\text{calc})|}{\sum y_i(\text{obs})}$$

$$R_I \text{ or } R_B = \frac{\sum|I_B(\text{'obs'}) - I(\text{calc})|}{\sum I_B(\text{'obs'})}$$

$$R_{wp} = \left\{\frac{\sum w_i[y_i(\text{obs}) - (1/c)y_i(\text{calc})]^2}{\sum w_i[y_i(\text{obs})]^2}\right\}^{1/2}$$

Pattern decomposition method

Definition. Systematic procedure for decomposing a powder pattern into its component Bragg reflections without reference to a structural model [nor, even, need for prior identification of the crystalline phase(s)].

Suggested guidelines

(1) Specify the instrument type, data type (including step-scan increment and metric), $\lambda(s)$, and monochromator or equivalent data for neutron TOF or X-ray energy-dispersive techniques used.

(2) Reflection profile representations

(*a*) What functions or numerical representations were used?

(*b*) How (why) were they chosen or developed (*e.g.* 'learned')?

(*c*) Give the scheme (*e.g.* a formula) for their 2θ dependence and give the values of any parameters in it.

(3) FWHM (full width at half maximum)

(*a*) Give largest and smallest FWHM's.

(*b*) How do α_2 FWHM's compare with α_1 in same 2θ region? (They should be close to the same unless the edge of the monochromator window is at the α_2 wavelength.)

(*c*) Explain any large variation in FWHM from line to line and α_1 to α_2.

(4) Publish or deposit this table and specify the wavelength used:

$d(\sigma)$ or $2\theta(\sigma)$	Integrated intensity (σ)	FWHM (σ)
⋮	⋮	⋮

(5) State how σ's were obtained.

(6) Some sort of test must have been used, and discussed, to see if there are any large correlations between (perhaps incompletely) resolved intensities, 2θ's, FWHM's, and other refined parameters.

(7) The authors must somehow deal with the issue of uniqueness of the decomposition (the use to which the decomposed pattern is put may help in this).

(8) The decomposed pattern must be used for something, *e.g.*

(*a*) Phase identification: indexing is required and other applicable requirements for *Crystal Data* in *J. Appl. Cryst.* (see *Notes for Authors*, and Calvert *et al.*, 1980) must be met.

(*b*) Structure refinement: in this case the weight matrix must use the correlations found among intensities in the decomposition.

(*c*) Studies of crystallite size and inhomogeneous strain.

(9) Nomenclature: Pattern decomposition is acceptable. However, it is recognized that the decomposition may be but one step in the study. Whatever terms are used to identify this general procedure should clearly distinguish it from the Rietveld method.

Finally, let us take note that the pattern decomposition procedure is often used as a step preliminary to the refinement of structure. In this usage, the pattern decomposition followed by structure refinement may be thought of as being related to the Rietveld refinement procedure as is a block-diagonal to a full-matrix refinement (Willis, 1981).

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Crystallographers

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This section is intended to be a series of short paragraphs dealing with the activities of crystallographers, such as their changes of position, promotions, assumption of significant new duties, honours, etc. Items for inclusion, subject to the approval of the Editorial Board, should be sent to the Executive Secretary of the International Union of Crystallography (J. N. King, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England).

Professor **N. V. Belov** died on 7 March 1982. A full obituary will be published in *Acta Crystallographica* Section A in due course.

Professor **Katharina Boll-Dornberger** (née **Schiff**), who died on 27 July 1981, aged 71, was the most prominent X-ray crystallographer in the German Democratic Republic. Drs H. and K. Fichtner write that she started her X-ray work with V. M. Goldschmidt at Göttingen in the early thirties and took her doctor's degree in the city of Vienna after her escape from Germany. After her emigration to England she had the opportunity to work with J. D. Bernal and D. Hodgkin. Returning to Germany after World War II, she established X-ray crystal structure analysis in the GDR. In 1956 she became Professor of Physics at Humboldt University in Berlin. From 1958 to 1969 she was director of the Institute of Crystal Structure Research of the Academy of Sciences of the GDR. She worked in inorganic crystal structure analysis and in protein crystallography. Her OD theory, created in the 1950's, is a geometrical approach to polytypism and stacking disorder. She introduced the concepts of partial coincidence operations and groupoids into crystallography. Crystallographers have lost a colleague of critical intelligence, deep knowledge and decisive influence on a considerable number of scientists in the GDR and in other countries.

Venkatraman Subramanian, a post-doctoral research fellow at the Crystallography Centre of the University of Western Australia, died tragically on 27 December 1981 at the age of 30 as a result of a swimming accident. Dr S. R. Hall, University of Western Australia, and Dr K. Seff, University of Hawaii, write that Subramanian was born in Bombay. He obtained a BSc in Chemistry at Madras University in 1972; an MSc at Birla Institute of Technology in 1974; and a PhD in Chemistry at the University of Hawaii in 1980. In 1974–75 he was a CSIR Research Fellow at the Indian Institute of Science in Bangalore and in 1980 was appointed as an ARGC research fellow at the University of Western Australia for the development of crystallographic computer software for the XTAL System. Subramanian was a diligent research worker with a care for detail. He was highly respected by his colleagues and admired by the many students he went out of his way to assist.

Dr U. W. Arndt, of the Medical Research Council Laboratory of Molecular Biology, Cambridge, Professor **J. D. Birchall**, Senior Research Associate at ICI, Runcorn, and Professor **M. Hart**, Wheatstone Professor of Physics, King's College, London, have been elected Fellows of The Royal Society.

Professor **Gunnar Hägg**, University of Uppsala, Sweden, has been awarded the 1982 Gregori Aminoff Gold Medal by the Royal Swedish Academy of Sciences for his pioneering applications of X-ray crystallography in inorganic chemistry. He will receive the Medal at the June session of the Academy. This is the third time that the Aminoff Prize has been awarded, the first recipient being Professor **P. P. Ewald** in 1979 and the second one Sir **Charles Frank** in 1981.

International Union of Crystallography

J. Appl. Cryst. (1982). **15**, 359

Structure Reports

Volume 46A of *Structure Reports* has recently been published. It covers the literature for metals and inorganic compounds for 1980 (464 pages) and costs 153 Netherlands guilders for subscribers with standing orders. The full price for individual copies is 180 guilders but personal subscribers may buy a copy for their own use at 90 guilders. Orders for these publications may be placed direct with the publisher, D. Reidel Publishing Company, PO Box 17, 3300 AA Dordrecht, The Netherlands, or with any bookseller. Trade orders should be sent to Reidel.

Book Reviews

Works intended for notice in this column should be sent direct to the Book-Review Editor (J. H. Robertson, School of Chemistry, University of Leeds, Leeds LS2 9JT, England). As far as practicable books will be reviewed in a country different from that of publication.

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Advances in X-ray analysis, Vol. 24: Proceedings of the 29th annual conference on the application of X-ray analysis, Denver, Colorado, August 1980. Edited by *D. K. Smith, C. S. Barrett, D. E. Leyden & P. K. Predecki.* Pp. xx + 428. New York: Plenum Press, 1981. Price US $ 49.50.

During the 29th Denver Conference on Applications of X-ray analysis, 74 papers were read, 56 of which are published in this volume. Following the tradition of the conferences there are reports on X-ray diffraction analysis (XRD) and X-ray spectrometry [XRS; mainly X-ray fluorescence analysis (XRF)]. It is difficult to review all the essential results and ideas contained in this volume, but a short summary will be given here.

Nowadays it is possible to obtain refined values of crystal-structure parameters from powder diffraction data by the Rietveld method, including a least-squares refinement procedure for fitting calculated and observed powder diffraction patterns. An example is given for human tooth enamel. Further, advances in the interpretation of diffraction data from amorphous materials are outlined. Qualitative and quantitative phase analysis, collection of crystallographic data, precision and reproducibility of Guinier powder patterns, and the level of XRD in Europe are discussed. The great importance of continuously scanning position-sensitive detectors in modern XRD work is demonstrated by the ten papers on this topic. Application, use and accuracy of such detectors are described; they are capable now of scanning speeds of several hundred degrees per minute, and