THE STANDARD DATA FORM FOR POWDER DIFFRACTION DATA

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RECOMMENDATIONS

1. We recommend the establishment of publication standards for powder diffraction data through the use of a standard data-form. We have designed such a form to be used at the author-referee level to ensure that published data are of good quality.

An example of a filled-out form is shown in Appendix A. Suggestions for filling out the data-form are given in Appendix B.

The data-form itself is divided into five sections: Sample Characterization, Technique, Unit Cell Data, References, and the Powder Data. Items on the form that are considered essential documentation are indicated by bold-face print. The other items are highly desirable information; however, it is recognized that some experimental procedures will not yield data on some of these items. Hence, omissions of data for these items should not of themselves preclude publication of the powder data.

This list of items is not all-inclusive: an all-inclusive list might be longer than the paper accompanying it. But this list does include the data most important to today's instrumentation. It represents, we believe, what the referee and editor can reasonably expect of experimenters seeking to publish their data. However, we do not intend that the list be used to stifle either new techniques in powder diffraction or the publication of powder data obtained under difficult conditions, e.g., low and high temperatures, high pressure, ultra-small quantities of materials.

We have several specific recommendations pertaining to the reporting, dissemination, and archiving of powder diffraction data:

(a) The published powder pattern should be as complete as possible; for example, it should include weak as well as strong diffraction lines. An element of doubt often arises in phase identification when the observed strong diffraction lines match those of a particular reference pattern, but some weak lines are observed that are not listed in the reference pattern. Complete reference patterns would be a benefit in these cases and generally would make identification easier. Furthermore, the ease of computer-indexing of powder patterns depends, in most cases, on the accuracy and completeness of the data. Ordinarily, 40 lines permit ready characterization of the material.* In the absence of 40 lines, the data should extend to (at least) 100° 2θ (Cu $K\alpha$ radiation).

(b) Researchers should report the experimentally observed 2θ values, in degrees and corrected for systematic instrumental errors. The d values are usually not an adequate substitute for the primary data, the 2θ values. Reporting the 2θ's does not distort information, but reporting the derived quantities, d's and Q's ($Q = 1/d^2$), usually does. For example, even the d values reported by NBS (for the example in Appendix A) do not match exactly their observed 2θ values. We had to add additional significant figures to the reported d values (in Appendix A) to accurately accomplish an otherwise simple transformation: 2θ's to d's. Hence, we believe that the magnetic-tape version of the PDF should contain 2θ values, which can be readily converted to d's, if necessary, by a user program. (Anyone using the magnetic-tape version, would have computer capability.) The PDF file cards, on the other hand, should probably continue to list d values.

(c) Intensities should be reported numerically, and on a scale from 0 to 100. This will avoid the non-quantitative notations $I < 1$ and $I \ll 1$ that sometimes occur with the present scale of 1-100. For example, 0.7 might be used instead of < 1. This new scale will require no additional digits. The recommended scale does not preclude the use of a scale from 1-10 or 1-100, when appropriate, nor should it be understood to imply an increased accuracy for $I < 1$.

(d) The reproducibility of the measured 2θ and I's should be indicated. These data should be obtained by multiple mountings of the sample material.

(e) Indexing of the powder diffraction data should be required for all but the rarest and best-defended cases. Without indexing, there is little proof that the reported pattern is for a single phase. Patterns can often be automatically indexed with appropriate computer codes [4, 5, 16]. Authors should report a figure of merit based on accuracy of the 2θ measurements and completeness of their data. As mentioned above, a figure of merit has been proposed for this purpose.

(f) Information concerning line breadth of the sample should be supplied. This could be full-width at half-maximum (FWHM) of a resolved $K\alpha_1$ line in the region 30-60° 2θ.

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*Although 40 lines are typically reported on a data card in the Powder Diffraction File, the author should not limit the published pattern to this number if more data is available. Patterns should be reported to at least 100°2θ. If no distinct lines can be detected beyond the high angle limit of the reported data, this point should be stated. Patterns with a small number of lines should be reported to the limit of the experimental method used.

(g) Additional information of value to future users should be supplied. Such data could be standard deviations, Chemical Abstracts Service Registry number, Crystal Data index number, etc.

We believe the use of a standard data-form has a number of benefits including:

- This data-form can be used as a checklist. Authors can use the form to ensure the necessary characterization of their data. Also, familiarity with the standard form will encourage authors to record complete information on their measurements.
- The completeness of the experimental data will be easy to evaluate. Because information pertaining to the experimentation will be collected in one place in the manuscript, referees or journal editors will be able to readily judge the future usefulness of the contribution. Moreover, an editor could send the powder data to an expert in powder diffraction for refereeing and send the rest of the manuscript to another reviewer.
- The data form could be recast into a camera-ready format for direct printing in the journals. In this way, data-base managers could receive the powder data exactly as sent by the authors.*

2. This subcommittee has also given some thought as to *what* constitutes publishable powder diffraction data. We believe that to justify being published, powder diffraction data must be potentially useful to someone else and must constitute in some way an original contribution to the literature. To constitute an original contribution, the data must be the first published for a well-characterized phase, must be a significant correction to or an improvement on published data, or must relate to the phase in a previously uncharacterized condition, e.g., at elevated temperatures or pressure. A powder pattern calculated from single-crystal structure data does not in itself meet the criterion of originality. Such data, we hope, will continue to be contributed directly to the data-base managers under guidelines being established by a JCPDS committee.

3. To help gain acceptance for publication standards within the world-wide powder diffraction community, we recommend that the ACA request the International Union of Crystallography (IUCr), through the Commission on Journals, to now consider implementation of these recommendations in all IUCr journals. As a first step in that process, the Commission on Journals may elect to refer these recommendations to the Commission on Crystallographic Data for review and approval. It is envisioned that a mutually acceptable version could be included in *Notes for Authors* and in the *Handbook for Co-Editors.* If the IUCr journals enact publication standards in the near future, other journals will in time follow that lead and the quality of published powder diffraction data will improve.

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*JCPDS has offered to referee powder data and to receive powder data directly if they are not to be published by the journal.

APPENDIX A: EXAMPLE OF COMPLETED DATA FORM

POWDER DIFFRACTION DATA FOR PHASE CHARACTERIZATION

(Data from Swanson, H. E., et al. (1971). NBS Monograph No. 25, Section 9, p. 25)

BOLDFACE ITEMS ARE CONSIDERED ESSENTIAL

SAMPLE CHARACTERIZATION

NAME (CHEMICAL,MINERAL, Trivial) Magnesium Aluminum Oxide (Spinel)

EMPIRICAL FORMULA $MgAl_2O_4$

CHEMICAL ANALYSIS NO x **YES**

SOURCE/PREPARATION Synthetic; Fusion of binary oxides

CHEMICAL ABSTRACT REGISTRY NO. 12068-51-8 PEARSON PHASE DESIGNATION cF56

OTHER **Index of Refraction = 1.718 (Isotropic)**

TECHNIQUE

RADIATION TYPE, SOURCE X-rays, Cu **λ VALUE USED** 1.54056 Å K α_1

λ DISCRIM. (Filters Mono, Etc.) Diffracted beam, curved LiF mono.

λ DETECTOR (Film, Scint, Position Sensitive etc.) Geiger

INSTRUMENT DESCRIPTION (Type, Slits, etc.) 17 cm Vertical Diffractometer **DIV** 1° **REC** 0.003"

SOLLER Yes **No.** 1 **Position** Inc. **Aperture** q = 1.2

INSTRUMENTAL PROFILE BREADTH 0.10 °2θ **TEMP (°C)** 25 ± 1

SPECIMEN FORM/PARTICLE SIZE Edge loaded powder/< 10 μm particle size for I's, packed for **2θ's**

RANGE OF 2θ FROM 5 **°2θ to** 165.0 °2θ **SPECIMEN MOTION** None

INTERNAL/EXTERNAL 2θ STD (if any) Ag (internal) **LATTICE PARAMETER OF 2θ STD** 4.08641 Å

2θ ERROR CORRECTION PROCEDURE Linear interpolation from nearest **2θ**'s of std.

INTENSITY MEAS. TECHNIQUE Strip chart record **(peak heights)** **ERROR (~)** 5% **PEAK** X **INTEGRATED**

MINIMUM INTENSITY THRESHOLD (IN RELATIVE INTENSITY UNITS) **0.3**

INTENSITY STD USED α - Al_2O_3 hkℓ's OF INTENSITY STD 113

INTENSITY RATIO I/I_c 1.70 (5) CONVERSION FACTOR IF CORUNDUM NOT USED

RESOLUTION (FWHM) FOR THIS MATERIAL: 0.10 °2θ AT 59.37 °2θ

2θ REPRODUCIBILITY FOR THIS MATERIAL: ± 0.02 °2θ AT All °2θ

UNIT CELL DATA

METHOD OF CELL DETN. **Cell and structure known from Ref. 1**

CELL REFINEMENT METHOD **Least-squares. See Ref. 2**

a = 8.0831 (1) Å ; b = ( ) Å ; c = ( ) Å

α = °(°) ; β = °(°) ; γ = °(°)

Z = 8 ; D_m = ( ) g cm^{-3} ; D_x = 3.578 g cm^{-3} ; V = 528.1 $Å^3$; Formula Wt = 142.25

CRYSTAL SYS. Cubic SPACE GROUP Fd3m [227] CRYSTAL DATA INDEX NO. 8.0831

FIGURE OF MERIT TYPE F_N. See Ref. 3 VALUE F_{29} = 58(0.015, 33)

REFERENCES

1. Bragg, W. H. (1915). Nature, 95, 561.
2. Appleman, D. E., Evans, H. T. (1973). NTIS Document No. PB-216188
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( ) INDICATES STANDARD DEVIATION IN LEAST SIGNIFICANT DIGIT(S)

POWDER DATA

ESSENTIAL		DESIRED		
2θ EXP (DEGREES)	I/I_0	d_{EXP} (Å)	hkℓ	$\Delta 2\theta$* (DEGREES)
19.02	35	4.66	111	+.019
31.27	40	2.858	220	-.003
36.84	100	2.437	311	-.009
38.53	3	2.335	222	-.021
44.83	65	2.020	400	+.016
55.64	9	1.650	422	-.020
59.37	45	1.5554	511	+.008
65.24	55	1.4289	440	-.001
68.64	3	1.3662	531	+.006
74.13	3	1.2780	620	+.003
77.32	8	1.2330	533	-.029
78.40	1	1.2187	622	-.013
82.64	5	1.1666	444	+.006
85.76	2	1.1320	711	-.012
90.97	5	1.0802	642	-.009
94.10	12	1.0524	731	-.005
99.34	7	1.0104	800	-.006
107.90	2	0.9527	822	-.020
111.22	8	0.93343	751	-.014
112.32	1	0.92738	662	-.035
116.91	6	0.90384	840	-.025
120.50	1	0.88722	911	+.004
121.69	0.9	0.88203	842	-.021
126.76	0.8	0.86161	664	+.013
130.74	8	0.84737	931	-.011
138.07	17	0.82488	844	+.033
142.97	0.4	0.81232	933	+.024
152.70	2	0.79266	10.2.0	-.033
160.65	11	0.78139	951	+.025

* $2\theta_{EXP} - 2\theta_{CALC}$

Appendix B. Information to Aid in the Completion of the Powder Diffraction Data Form

The underlined items below correspond to each of the items on the Powder Diffraction Data-Form. Authors may not be able to complete every item, but are urged to be as complete as possible.

SAMPLE CHARACTERIZATION

Chemical Name. Names should be consistent with the conventions of the journal in which the pattern is to be published. Such nomenclature is often in accord with IUPAC conventions [1-3]. This name should include polymorphic phase identification (e.g., β-manganese dioxide).

Mineral Name. The mineral name should be included for all minerals or synthetic compounds known to have mineral counterparts (e.g., lead sulfide, galena).

Trivial. Common names like Rochelle salt or methylene blue, which may be of value, should be included.

Empirical Formula. The empirical or stoichiometric formula should, if possible, connote structural information such as functional groups (e.g., $(PO_4)_2$). Such stoichiometric formulas can be readily converted to strictly empirical formulas by computers. The stoichiometric formula should, however, be consistent with the conventions of the journal in which the pattern is to be published.

Chemical Analysis. The results of a partial or full chemical analysis should be communicated either on this form or in the journal article.

Source. List the source of the material. If it is a natural mineral, the location must be given. If the material is commercially obtained, state the supplier.

Preparation. State method and pertinent conditions of preparation.

Other Data. Give any other data that will help to assure that the specimen is well characterized apart from the diffraction pattern. (A high-quality diffraction pattern of an improperly identified material is of less than no use.) Such data might include: melting point or transformation temperatures, the fact that single-crystal studies were done; structure type (e.g., NaCl type), color, indices of refraction, etc.

Chemical Abstracts Service Registry Number. This number is uniquely assigned to each compound by Chemical Abstracts. It is very useful in cross correlating between various computer data bases. This number should be included it if is known to the author. For further information see reference [4].

Pearson Phase Designation. A method for classifying structures of metals and alloys is described by W. B. Pearson [5]. The "Pearson Symbol" is quite useful and, if known, should be included (e.g., α-Se has the symbol mP32, Al_3Zn has the symbol tI16). The three parts of the Pearson symbol are: the first, lower-case letter (a, m, o, t, h, c) designating crystal system, the second, capital letter (P, C, F, I, R) designating Bravais lattice type, and a number indicating the number of atoms in the conventional unit cell: NOTE: This is not the number of formula units.

TECHNIQUE

Radiation Type. X-rays, neutrons, electrons, etc.

Radiation Source. X-ray target material, neutron source, electron accelerating voltage, etc.

λ Value Used. The numerical value of the wavelength used in calculating d values. List $\lambda(K\alpha_1)$ if the α_1 component was fully resolved throughout the pattern or if analysis leads to 2θ's for the α_1 component. List λ(Kα) if the Kα doublet was not resolved. If both λ(Kα) and $\lambda(K\alpha_1)$ were used, indicate the dividing line and list both wavelengths.

λ Discriminator (Filters, Mono, etc.). Give the method used to monochromatize the beam. State whether the monochromator or filter was used in the incident or the diffracted beam.

λ Detector (Film, Scint., Position Sensitive, etc.). The type of radiation detector used and any unusual electronic processing should be given. (e.g., pulse-height discrimination for a scintillation or proportional detector is conventional and need not be stated.)

Instrument Description (Type, Slits, etc.). State the type and size of instrument used (e.g., 114.6-mm Debye-Scherrer camera, 17-cm diffractometer, 200-mm Guinier camera, etc.) and the conditions for the experiment. For diffractometers, state the divergence angle (Div) of the incident beam, receiving-slit width (Rec), and whether or not different or continuously varying divergence slits were used for various 2θ ranges.

Soller (Number, Position, Apertures). For diffractometers, give the number of sets of Soller slits, their positions (e.g., in incident or diffracted beam or in both) and aperture(s), because axial divergence can be a source of large profile displacement or broadening. Aperture is best characterized by $q = R\Delta/h$, where R is the radius of the diffractometer; h is 1/2 the axial extension of the sample, and

$$\Delta = \frac{\text{spacing between Soller foils}}{\text{length of Soller foils}}$$

Instrumental Profile Breadth. This parameter is the full width at half maximum (FWHM) of a reference sample that has minimal intrinsic broadening. It allows the instrument used to be compared with others and indicates the resolving ability. A recommended procedure is to measure the 311 reflection ($d_{311} = 1.638$ Å) of a well-annealed Si specimen whose particle size is between 1 and 20 μm. The FWHM of only the $K\alpha_1$ peak should be reported (for Cu $K\alpha_1$ radiation, this occurs at 56.12° 2θ). If another material or peak is chosen, it should be stated. If the α_1 and α_2 peaks are not resolved, report that fact along with the FWHM for the peak observed.

Temperature. State the temperature of the sample when the pattern was obtained.

Specimen Form. Indicate if the sample is a loose powder, sintered compact, metallographic mount, etc. State how the specimen was prepared for diffraction analysis (e.g., side loaded or vertically packed into a diffractometer holder, dusted onto a substrate, packed into a capillary). If different preparations were used in the measurement of d values and intensities, indicate both methods.

Particle Size. Give the average or maximum particle size in the specimen (e.g., 10 μm, <325 mesh, <20 μm).

Range of 2θ. Indicate the range of 2θ that was examined. All peaks above the stated intensity threshold (given below) in the reported 2θ range should be included under Powder Data. Include a minimum of 40 peaks or all data up to $2\theta = 100°$.

Specimen Motion. State type of sample motion during the diffraction experiment (e.g., sample spinner, rotated cylinder, Gandolfi motion, none).

Internal/External 2θ Standard (if any). If the instrument was calibrated with a known standard before or after the pattern was obtained, state "external--(material of standard)." If an internal standard was mixed with the sample to check for a specimen displacement and/or 2θ zero error, state "internal--(material of standard)." A standard material recommended for both procedures is National Bureau of Standards SRM silicon [6].

Lattice Parameter of 2θ Standard. State the numerical value of the lattice parameter used in calculating the expected 2θ positions of the lines of the standard.

2θ Error Correction Procedure. State the method used to correct the observed 2θ values for systematic instrumental error. A recommended procedure is to use one or more external standards such as Si (ref. [6]) and determine the $\Delta 2\theta$ ($2\theta_{exp} - 2\theta_{calc}$) vs 2θ curve. These data may be fitted using a least squares polynominal regression (e.g., a third-degree equation: $\Delta 2\theta = a_0 + a_1 2\theta + a_2 2\theta^2 + a_3 2\theta^3$). If this procedure is used, state the coefficient (a_i's) of the polynominal. A cubic standard having a large cell dimension (>10 Å) is recommended for patterns having many lines at low angles.

Intensity Measuring Technique. Indicate the method used to determine diffracted intensities: peak heights or integrated areas from a strip chart, or densitometer or step-scan data. Indicate if monochromator polarization effects have been removed from the I/I_0 values reported under Powder Data.

Minimum Intensity Threshold (in Relative Intensity Units). Indicate the relative intensity (see I/I_0 below) not considered to be a real peak. Typically this threshold is taken as the background reading plus 2.5 times $\sqrt{\text{background}}$. NOTE: Because this value will differ in regions of high and low background, limiting values should be reported.

Intensity Standard Used. Indicate the material used in determining the reference intensity ratio. Intensity standards allow for the direct comparison of intensities in different materials. Methods involving the use of corundum as an intensity standard for direct quantitative phase analysis have been described [7-9].

hkl's of Intensity Standard. Give the hkl(s) of the line(s) used for determining the I/I_c standard value.

Intensity Ratio I/I_c. Give the value of the ratio of the strongest line in the pattern to the strongest line of corundum (d_{113} = 2.085 Å) in a 50-50 wt percent mixture of the two [7-9], and give the reproducibility, $\alpha(I/I_c)$.

Conversion Factor if Corundum not used. If a-Al_2O_3 is not used as the intensity standard, then the conversion factor between the standard used and corundum should be listed. This conversion factor can be used to calculate the I/I_c for the specimen.

Resolution (FWHM) for this Material. The full width at half maximum, in degrees 2θ, for a well-resolved α_1 line (if possible) should be given along with the 2θ of this line. This peak should be chosen between 30° and 60° 2θ to avoid wavelength broadening effects. This value, of course, includes the instrumental profile breadth given above. If α_1 and α_2 are not resolved, this fact should be stated.

2θ Reproducibility for this Material. State any measured reproducibility of the positions of the diffraction maxima; if measured for one line, state the 2θ.

UNIT CELL DATA

Method of Cell Determination. If the unit cell was obtained from the literature, give the reference. If the unit cell was derived from the powder data, state the method or program used.

Cell Refinement Method. If the cell was refined, state the method and computer program used (if any).

Cell Parameters a, b, c, α, β, and γ. These values should be reported to the proper number of significant digits; put the standard deviations, in terms of the least significant digit, in parentheses (parameters in angstroms, angles in degrees).

D_m: experimentally determined density in g/cm^3

D_x: calculated density; from $D_x = Z\ FW/N_oV$, where Z is the number of formula units in each unit cell, FW is formula weight, N_o = Avogadro's number (0.60225×10^{24}), and V is the volume of the unit cell in cm^3

Space Group. If the space group has been determined by single-crystal studies, give the Hermann-Mauguin symbol followed in brackets by the space-group number as listed in reference [10]. For space group determinations that are not your own, a reference should be given below. For unit cells determined solely from powder diffraction data, give the diffraction aspect as defined in reference [11] (pp. S-8 to S-18).

Crystal Data Index Number(s). List the axial ratios, derived from the cell parameters, that would be used to locate this material in Crystal Data Determinative Tables [11]. Warning: these axial ratios are based on a unit cell defined by Crystal Data, which may not be the cell for which the parameters are given above. Reference [10] describes how the Crystal Data cell is obtained. These rules have been incorporated in the powder pattern evaluation program AIDS [12].

Figure of Merit Type and Value. List the type of figure of merit used (e.g., M_{20} or F_N--see references [13] and [14]) and its value. The figure M_{20} is defined as:

$$M_{20} = \left(\frac{Q_{20}}{2\,|\overline{\Delta Q}|}\right)\left(\frac{1}{N_{poss}}\right),$$

where Q_{20} is the Q value (= $1/d^2$) of the 20th observed line, $|\overline{\Delta Q}|$ is the average absolute discrepancy between Q_{obs} and Q_{calc}, and N_{poss} is the number of independent diffraction lines possible up to the 20th observed line. The figure F_N is defined as:

$$F_N = \left(\frac{1}{|\overline{\Delta 2\theta}|}\right)\left(\frac{N}{N_{poss}}\right) ,$$

where $|\overline{\Delta 2\theta}|$ is the average absolute discrepancy between observed and calculated 2θ values; and N_{poss} is the number of independent diffraction lines possible up to the Nth observed line.

With regard to the figures of merit, some guidelines for the counting of possible independent diffraction lines are in order:

Systematic absences caused by symmetry elements and lattice type are excluded in the tallying of N_{poss}.

Only one plane from the complete set of planes related by crystal symmetry is counted in N_{poss}. For example, in the cubic system, the 100 line is counted as one independent line although it is composed of diffracted intensities from all six planes of that crystallographic form.

Some forms, though not related by symmetry, have exactly the same spacing and would give rise to the same line in the powder pattern (e.g., 333 and 511 in the cubic system). Forms of this kind are also counted as one independent line. Note that this rule means that the higher-symmetry Laue group is always assumed. When a lower-symmetry Laue group is definitely known from single-crystal studies, this rule is not strictly correct for some of the diffraction lines. However, for most of these cases, the effect on the value of the figure of merit for the overall pattern should be insignificant.

For the case of accidental degeneracy (i.e., nonequivalent forms which have spacings so nearly identical that the individual lines would not be experimentally resolved), all lines in such a cluster are counted as possible independent lines; the line having the smallest Δ2θ is used in the calculation of $|\overline{\Delta 2\theta}|$ for the pattern, and the other line or lines are listed as not observed, thereby increasing N_{poss}.

The function is reported in the form

$$F_N = \text{Value}\ (|\overline{\Delta 2\theta}|,\ N_{poss}).$$

For example,

$$F_{30} = 73.5(0.012, 34).$$

We recommend that N in F_N be the 30th observed line or the last line if there are fewer than 30 lines in the pattern.

Note that if the space group or the diffraction aspect is not determined, N_{poss} can only be reported as a maximum value; i.e., no allowance would have been made for systematic absence, a fact which could appreciably reduce M_{20} or F_N.

REFERENCES

Cite pertinent literature references for previous x-ray or preparative studies.

POWDER DATA LISTING

$2\theta_{exp}$. Report the experimentally observed 2θ values, in degrees and corrected for systematic instrumental errors. If multiple determinations of each peak were made, then the standard deviation of each 2θ value should follow in parentheses.

I/I_0. List the relative intensities I/I_0 of the diffraction lines on a numeric scale; $I/I_0 = I/I_{max} \times$ scale, where I_{max} = the numeric value chosen for the most intense refection. The scale value should be chosen such that the maximum intensity value is not greater than 100. Intensities less than 1 are reported as decimal fractions. If the standard deviation of each intensity was computed, place the value in parenthesis after each I/I_0 value.

d_{exp}. List the d values in angstroms, derived from the observed 2θ values using Bragg's law: $d = \lambda/[2 \sin (2\theta/2)]$, where λ is the value of the wavelength stated under Technique. In reporting d's, the number of significant figures given should be sufficient to allow recomputation of the experimental 2θ's to their measured accuracy, i.e., the number of significant figures should be in accordance with $\sigma(d) = (d/2) \cot \theta\sigma(2\theta)$.

hkl. List the Miller indices of the diffraction lines determined from the known unit cell. If several peaks are overlapped such that separate peak position measurements cannot be made, the hkl's should be grouped together and given for the single intensity value. The hkl's of all peaks can be listed, or a "+" can be post-scripted to the last one given to indicate others not listed.

Δ2θ. List the difference, with sign, between the experimental and calculated 2θ values: $\Delta 2\theta = 2\theta_{exp} - 2\theta_{calc}$. A useful rule of thumb is that, if systematic errors have been removed completely, the absolute value of each Δ2θ should be less than $3\sigma(2\theta)$. Alternatively, a small bar-graph can be presented showing Δ2θ (with sign) versus line number. The reader, if interested, could reconstruct more accurate values of Δ2θ from the experimental 2θ's and the reported lattice parameters.

References, Appendix B

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[3] Fletcher, J. H., Dermer, O. C., and Fox, R. B., *Nomenclature of Organic Compounds, Principles, and Practice*, Advances in Chemistry Series No. 126 (American Chemical Society, Washington, DC, 1974).

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[6] Hubbard, C. R., Swanson, H. E., and Mauer, F. A., A silicon powder diffraction standard reference material, J. Appl. Cryst. *8*, 45 (1975).

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[9] Hubbard, C. R., Evans, E. H., and Smith, D. K., The reference intensity ratio, I/I_c, for computer simulated powder patterns, J. Appl. Cryst. *9*, 169-174 (1976).

[10] *International Tables for X-ray Crystallography*, N. F. M. Henry and K. Lonsdale, eds., Vol. 1, 2nd edition (Kynoch Press, Birmingham, England, 1965).

[11] Donnay, J. D. H. and Ondik, H. M. (eds.), *Crystal Data Determinative Tables*, Third Ed., Vols. 1-4 (National Bureau of Standards and Joint Committee on Powder Diffraction Standards, Publishers, 1972, 1973, 1978).

[12] Mighell, A., et al., Single crystal and powder data evaluation and standardization program, U.S. National Bureau of Standards, to be published.

[13] de Wolff, P. M., A simplified criterion for the reliability of a powder pattern indexing, J. Appl. Cryst. *1*, 108 (1968).

[14] Smith, G. S. and Snyder, R. L., F_N: a criterion for rating powder diffraction patterns and evaluating the reliability of powder pattern indexing, J. Appl. Cryst. *12*, 60 (1979).

Discussion

Question (Zwell): Many analysts have asked for publication of all diffraction patterns. Wouldn't your proposal decrease the number of patterns being published and thereby reduce the information available? (Would the improvement in quality of pattern compensate for the loss of data?)

Comments (Calvert, et al.): It would be hard to predict the effect with any degree of confidence. A similar set of publication recommendations made earlier by the single-crystal community resulted in a marked improvement in the average quality of papers published in Acta and many other journals. Some journals continue to accept lower quality data but the prestige of publication in the lead crystallographic journals is such that authors generally strive to meet these standards. That result can't be bad.

Question (Zwell): It appears that it is implied that 2θ would be for Cu radiation. Wouldn't the use of such angles reduce the value of data for those persons using other radiations chromium, cobalt, molybdenum? (d's are constant and independent of radiation.)

Comments (Calvert, et al.): Two theta would be recorded as measured. These data would not be transformed to a different wavelength since that process could potentially introduce noise.

POWDER DIFFRACTION DATA FOR PHASE CHARACTERIZATION

BOLD FACE ITEMS ARE CONSIDERED ESSENTIAL

SAMPLE CHARACTERIZATION

NAME (CHEMICAL MINERAL, Trivial) ________________

EMPIRICAL FORMULA ________________

CHEMICAL ANALYSIS. NO____ **YES** ________________

SOURCE/PREPARATION ________________

____

CHEMICAL ABSTRACT REGISTRY NUMBER ________ PEARSON ALLOY DESIGNATION ________

TECHNIQUE

RADIATION TYPE, SOURCE ________________ **λ VALUE USED** ________

λ DISCRIM. (Filters Mono, Etc.) ________________

λ DETECTOR (Film, Scint, Position Sensitive, etc.) ________________

INSTRUMENT DESCRIPTION (Type, Slits, etc.) ________________ **DIV =** ________ **REC =** ________

INSTRUMENTAL PROFILE BREADTH ________________ **°2θ** ________ **TEMPERATURE (°C)** ________

SPECIMEN FORM/PARTICLE SIZE ________________

RANGE OF 2θ FROM ________ **°2θ TO** ________ **°2θ, SPECIMEN MOTION** ________________

INTERNAL/EXTERNAL 2θ STD (if any) ________ **LATTICE PARAMETER OF 2θ STD** ________

2θ ERROR CORRECTION PROCEDURE ________________

INTENSITY MEAS. TECHNIQUE ________________ **PEAK** ____ **INTEGRATED** ____

MINIMUM INTENSITY THRESHOLD (IN RELATIVE INTENSITY UNITS) ________________

INTENSITY STD USED ________________ 2θ's OF INTENSITY STD ________________

INTENSITY RATIO I/I_c ________ CONVERSION FACTOR IF CORUNDUM NOT USED ________________

RESOLUTION (FWHM) FOR THIS MATERIAL. ________________ °2θ AT ________________ °2θ

2θ REPRODUCIBILITY FOR THIS MATERIAL: ________________ °2θ AT ________________ °2θ

CELL DATA

METHOD OF CELL DETN. ________________

CELL REFINEMENT METHOD ________________

a = ________________ (   ) Å; b = ________________ (   ) Å; c = ________________ (   ) Å

α = ________________ ° (°); β = ________________ ° (°); γ = ________________ ° (°)

Z = ________; D_m = ________ (   ) g cm^{-3}, D_x = ________ g cm^{-3}, V = ________ Å^3; Formula Wt = ________

CRYSTAL SYS. ________________; SPACE GROUP ________________; CRYSTAL DATA INDEX No. ________________

FIGURE OF MERIT TYPE ________________ VALUE ________________

REFERENCES

(   ) INDICATES STANDARD DEVIATION IN LEAST SIGNIFICANT DIGIT/S

POWDER DATA

ESSENTIAL		DESIRED		
2θ EXP (DEGREES)	I/I_o (.1-100)	d_{EXP} (Å)	$hk\ell$	$\Delta 2\theta$* (DEGREES)

*$2\theta_{EXP}-2\theta_{CALC}$