Appendices

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Send all correspondence and completed forms to:

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ICDD Grant-in-Aid Proposal for Reference Pattern Production

Please choose one of the following Grant-in-Aid cycles

_____ Cycle I _____ Cycle II

Schedules for the cycles may be found on page 7 of the Guidelines for Grant-in-Aid Proposals.

Title of Proposal:

1. Principal investigator _____

   Position and/or title _____

   Address _____

   _____

   _____

   Telephone _____ FAX number _____

   E-mail address _____

2. Names and work experience of prospective investigators:

   The principal investigator should attach a brief curriculum vitae.

3. Number of patterns to be prepared _____

   Funds requested for grant _____

4. Describe the phases or class of phases for which you propose to prepare XRD reference patterns.

Indicate subfile(s):

❑ Inorganic ❑ Metals & Alloys ❑ Mineral ❑ Organic

Indicate the data type and the number of patterns to be generated of each type:

❑ Experimental patterns No. of patterns_____

❑ Experimental patterns with supporting calculated patterns No. of patterns_____

❑ Calculated patterns No. of patterns_____

5. List the phases proposed for the preparation of XRD reference patterns. This information is requested to avoid duplication of effort among grant recipients. Should you wish to deviate from this list, please inform ICDD. Attach a separate page for additional phases.

	Name	**Formula**	**Comments**
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			

6. Describe the current contents of the **PDF** related to the class of phases you propose to study. List the set numbers of the **PDF** you reviewed and search methods used. What other literature search has been conducted?

7. Indicate why these phases are important technically or scientifically and other criteria which entered into their recommendation. In particular, why are these phases important to users of the **PDF**?
   - ◆ Examples of the latter would be: missing minerals, alloy phases, common medications, frequently encountered phases, or common organic reagents, since these categories fit into current ICDD subfiles.

8. Describe any additional support and research activities in which you are involved that support the preparation and characterization of the phases for which you propose to produce reference patterns.
   - ◆ One of the best uses of ICDD funds is to build upon either institutional or external support to the principal investigator or colleagues since it is uncommon that the ICDD can provide sufficient funds to cover all aspects of preparation, characterization, XRD, and reporting.

9. Besides X-ray diffraction, how will you characterize your samples for phase purity and elemental composition?

   ❑ Optical microscopy ❑ Thermal analysis

   ❑ Melting point ❑ Spectroscopy

   ❑ Chemical analysis method ❑ Other _____

   Additional details:

10. Have you received ICDD grant funds previously?

❑ Yes—most recent grant completed:

Project title: _____

Date of final report: _____

Class of compounds: _____

Total number of completed patterns submitted: _____

Years that other grants were completed: _____

❑ No—Please attach two examples of reference pattern data sets you have produced.

◆ Note: New funds will not be approved until the number of promised patterns is delivered and a final report is received and accepted.

11. Appendices attached:

An **Instrumentation Summary** (see page 5) and an **Experimental Techniques Summary** (see page 6) are required for all grant proposals. If you prepared these summaries for a grant last year and no major changes have occurred, indicate that below and attach a copy of last year's summary to the proposal.

❑ Instrumentation Summary

□ Yes—A copy of summary is attached.

□ No major changes since last grant; a copy of last year's summary is attached.

❑ Experimental Techniques Summary

□ Yes—A copy of summary is attached.

□ No major changes since last grant; a copy of last year's summary is attached.

❑ List of relevant publications

❑ Other attachments and names

_____

_____

_____

Instrumentation Summary

Instrument Type

Date: _____

Diffractometer: ❑ Manual ❑ Automated

Camera: ❑ Guinier ❑ Debye Scherrer ❑ Gandolfi

Other _____

Brand and/or Type number _____

If diffractometer: Monochromator: ❑ Primary Beam ❑ Secondary Beam ❑ None

Detector: ❑ Scintillation ❑ Si(Li) ❑ Other

Intensity Measurement

❑ peak height ❑ peak area

❑ counter ❑ dead-time corrected

❑ microdensitometer ❑ visual

Comments

Experimental Techniques Summary

Experimental techniques: Date: _____

Specimen preparation:

Methods to reduce preferred orientation:

Specimen rotation/oscillation:

Describe the technique used for producing reference data:

Internal Standard:

External Standard:

Routine instrument performance checks:

Data analysis methods (List method and/or software name, if applicable):

Peak-finding method:

Background determination method:

Calibration method:

Cell determination:

Cell refinement:

Acknowledgement of Agreement for the Approved Grant

Acknowledgement

Commencing _____, _____accepts from the JCPDS-International Centre for Diffraction Data, a grant-in-aid to be known as _____.

JCPDS-International Centre for Diffraction Data agrees to fund this grant in the amount of _____, payable in four quarterly installments of _____each. It is understood that this funding will not be used as salary compensation for the grant's principal investigator if he/she is an active member of the ICDD Board of Directors or the Grant-in-Aid Committee, nor shall the funding be used by the principal investigator to compensate another active ICDD member unless prior written approval is obtained from the ICDD Board of Directors. The grantee agrees to abide by the report schedule as described in the *Guidelines for Grant-in-Aid Proposals.*

The grant-in-aid will be administered by _____ in accordance with the policies and procedures of _____.

_____	_____
Date	Grant Administrator
_____	_____
Date	Authorized Officer
_____	_____
Date	General Manager International Centre for Diffraction Data

Publishing Agreement

1. It is our custom to request authors to vest the worldwide copyright of their powder diffraction data and any supporting information submitted as part of the grant-in-aid report with the JCPDS—International Centre for Diffraction Data for the full term of copyright and we would be grateful if you would confirm your acceptance of these terms by signing and returning the agreement below. We will not withhold permission for any reasonable request from you to publish, in connection with any other work by you, any part of the powder diffraction data and any supporting information which were submitted as part of the grant-in-aid report, provided the usual acknowledgements are given regarding copyright notice and reference to the original publication.

2. If it is appropriate, the author's employer may sign this agreement, and in any event the employer may reserve the right to use the powder diffraction data and any supporting information submitted as part of the grant-in-aid report internally or for promotional purposes only, by so indicating on this agreement. It is understood that proprietary rights other than copyright (including patent rights) are reserved.

3. If the author is a U.S. Government employee and this work was done in that capacity, the assignment applies only to the extent allowable by U.S. law. If at least one coauthor is not a government employee, said author should sign the agreement.

4. If the author is an employee of the British Government, then HMSO will grant a nonexclusive license to publish this powder diffraction data and any supporting information submitted as part of the grant-in-aid report in the **Powder Diffraction File** in any form or media, provided British crown copyright and user rights (including patent rights) are reserved.

5. The author warrants that the powder diffraction data and any supporting information submitted as part of the grant-in-aid report is the author's original work, and has not been published before. (If excerpts from copyrighted works are included, the author will obtain written permission from the copyright owners and show credit to the sources.) The author also warrants that the information contains no libelous or unlawful statements, and does not infringe on the rights of others.

6. If the work was prepared jointly, the author agrees to inform coauthors of the terms of the agreement, and to sign on their behalf.

_____

With the qualifications listed above, I assign to JCPDS-International Centre for Diffraction Data the copyright of my powder diffraction data and any supporting information submitted as part of the grant-in-aid entitled " _____

_____"

for publication in the **Powder Diffraction File**.

_____Signed

_____Date

Appendix 3

The Standard Data Form for Powder Diffraction Data

ICDD Grant-in-Aid Report Form

Due at ICDD by

_____

Grant # _____

Title of Proposal: _____

_____

1. Principal investigator _____

   Affiliation _____

2. Names of additional investigators contributing to this report and reference data:

3. Number of patterns proposed _____

   Number of patterns prepared_____

   Funds approved for grant_____

4. Indicate why these phases are important technically or scientifically, or any other criteria which entered into their selection. In particular, why are these phases important to users of the **PDF**?
   - Examples of the latter would be: missing minerals, alloy phases, common medications, frequently encountered phases, or common organic reagents, since these categories fit into current ICDD subfiles.

5. If major changes occurred in instrumentation or experimental techniques used in the production of reference patterns that were not indicated in your original proposal, please update the relevant summary and attach the summary to this report.

   Instrumentation Summary

   - ❑ Yes—Changes have occurred and a copy of the updated summary is attached.
   - ❑ No major changes since proposal.

   Experimental Techniques Summary

   - ❑ Yes—Changes have occurred and a copy of the updated summary is attached.
   - ❑ No major changes since proposal.

6. Describe the phases or class of phases for which you prepared XRD reference patterns.

Indicate the number of patterns completed and submitted to ICDD.

Experimental patterns _____

Experimental patterns with supporting calculated patterns_____

Calculated patterns _____

7. Please complete the following information for each phase you have submitted under this grant.

Pattern Number	Chemical Formula	Name: Chemical Common, Mineral, etc.	CAS No.	New or Redone	Experimental or Calculated	PDF-3 data (yes/no)	Date Submitted	Other

Attach separate page for additional patterns.

- Note: Pattern number is in the following form: Grant number/###, where Grant number is an identification number assigned by ICDD and ### is the sequential number of the submission.

8. Additional Comments.

A. List any published reports or journal articles which were supported to any extent by your grant-in-aid.
   - Please note: Acknowledgment of ICDD's financial support in preparation of the original article is appreciated.

B. Although grants-in-aid are not meant to fund other activities, you may wish to describe any other activities carried out on behalf of ICDD (e.g., subcommittee participation, lab visits, short courses that demonstrate the level of your support for, and participation in, the work of ICDD).

C. Other comments:

Guidelines for the Preparation of Digitized X-ray Powder Patterns Using Copper Kα Radiation and Bragg-Brentano Diffractometer Geometry

1. **Scan Range:** Should cover at least 6° to 90°/2θ. For large cells, a starting angle of 2° or 3° is recommended.

2. **Step Interval:** 0.02°/2θ

3. **Dwell Time:** (Count time/step): at least one second. The ideal number will vary with the sensitivity of your own diffractometer, but as a rough guide, we would like to accumulate at least 50,000 counts on any line with [I>50] and at least 5,000 counts on any line with [50>I>5]. This would give a counting statistical error of better than 0.5% (1σ) for the strongest lines, and better than 1.5% for the weaker lines.

4. **Internal Standard:** Your sample should not contain an internal standard when the archival digitized scan is made.

5. **Form of Data:** Supply your data in as raw a form as possible using *.PD3* format. The data should not be smoothed, α_2 stripped, background subtracted, or subjected to any other process that would cause significant changes in the true experimental data. If you cannot suppress any of the above treatments, please indicate this fact on your report.

6. **Archiving of Data:** Where possible, files for archiving should be placed on 3.5 inch, high density (1.44 Mbytes) disks. The disk should be labeled with a description and the person's initials. The individual files on the disk should have the label of the material analyzed with an extension of *.PD3*, for raw data, submitted by the originator. The program PDF3FC can be used to transform ASCII files to *.PD3* files. A copy of PDF3FC is available, on request, from ICDD. Also available are the programs PDF3CHK and REFCHK. These programs are designed to check the completeness and format of *.PD3* and *.REF* files.

7. **Description of Experimental Conditions:** Include with your data a list of conditions under which your scans were made. These conditions are shown in Tables 1 and 2. Table 1 is a header from a *.PD3* file and Table 2 is a header from a *.REF* file.

8. **X-ray Source:** We would <u>prefer</u> that data be collected using a fine focus tube at a 6° take-off angle (generator set at 45kV). This will give a beam width of about 0.2 mm at the receiving slit.

9. **External Standard:** Include a scan of an Al_2O_3 external standard, made within two weeks of the submitted scan. This scan should be given a *.REF* extension. The recommended standard is NIST SRM 1976 Instrument Sensitivity Standard.

Table 1—Example of .PD3 File Format

```
&SAMPLE IDENT=Erbium Silicate Chlo
&ICDD-DX=1.00
&DATE=YY-MM-DD
&FILE IDENT=KLA6   .PD3
&SOURCE IDENT=KLA6    .LIS
&CROSS REFERENCE=E_TREF01.REF
&GRANT NO=90-05E_T
&PDFNO=XX-YYYY
&DIV SLIT= 1.0
&WAVELENGTH=  1.540560
&COUNT TIME= 4.000
&STEP SIZE=  .0100
&XUNITS=Deg (2T)
&YUNITS=COUNTS
&YFACTOR=1.0
&FIRSTX= 4.000
&LASTX=140.000
&MAXY= 129168.
&NPOINTS= 13601
&XYDATA=(X++(Y..Y))
  4.000    900.    912.    936.    961.    961.    918.    961.    980.
  4.080    882.    918.    949.    936.    894.    961.    894.    918.
  4.160    882.    986.    900.    918.    936.    847.    864.    870.
  4.240    930.    912.    900.    894.    835.    870.    847.    864.
  4.320    876.    835.    801.    847.    853.    900.    835.    818.
  4.400    882.    829.    853.    847.    864.    778.    790.    835.
  4.480    847.    835.    812.    790.    790.    835.    790.    864.
  4.560    853.    784.    818.    841.    812.    818.    790.    762.
  4.640    795.    784.    790.    829.    818.    767.    818.    762.
  4.720    751.    795.    801.    795.    784.    795.    778.    807.
  4.800    790.    784.    812.    751.    807.    740.    807.    778.
  4.880    784.    708.    773.    762.    708.    729.    778.    773.
```

Table 2—Example of .REF File Format

```
&SAMPLE IDENT=REF STD # SRM-1967
&ICDD-DX=1.00
&DATE=90/11/06
&FILE IDENT=E_TREF01.REF
&SOURCE IDENT=ko1190  .STD
&GRANT NO=90-05E_T
&GENERATOR TYPE=CP
&ANODE=Cu
&GEN KV=   45.
&GEN MA=   30.
&DIFFRACTOMETER=BBR
&DIV SLIT=1.0
&REC SLIT=0.20
&DETECTOR=Scint
&MONOCHROMATOR=PG(DB)
&WAVELENGTH=1.540600
&COUNT TIME=  4.0
&STEP SIZE=  .0100
&XUNITS=Deg(2T)
&YUNITS=Counts
&YFACTOR= 1.0000
&FIRSTX= 4.000
&LASTX=140.000
&MAXY=  36634.
&NPOINTS= 13601
&XYDATA=(X++(Y..Y))
  4.000    650.    666.    692.    655.    718.    697.    655.    686.
  4.080    635.    692.    650.    666.    676.    645.    671.    630.
  4.160    697.    625.    600.    650.    620.    635.    650.    681.
  4.240    630.    645.    630.    666.    610.    581.    615.    625.
  4.320    605.    645.    595.    586.    605.    590.    610.    595.
  4.400    610.    620.    605.    671.    620.    605.    590.    557.
  4.480    595.    571.    605.    645.    595.    590.    552.    625.
  4.560    586.    562.    600.    562.    620.    576.    562.    515.
  4.640    562.    566.    557.    524.    571.    625.    557.    534.
  4.720    557.    552.    534.    566.    571.    566.    552.    548.
  4.800    552.    605.    538.    562.    557.    511.    515.    515.
  4.880    581.    562.    552.    581.    557.    576.    538.    515.
```