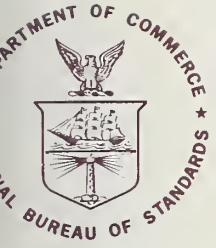

NBS TECHNICAL NOTE 1112

U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

User Evaluation of Crystal Data Products and Services: Questionnaire Analysis and Impact

NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards[1] was established by an act of Congress on March 3, 1901. The Bureau's overall goal is to strengthen and advance the Nation's science and technology and facilitate their effective application for public benefit. To this end, the Bureau conducts research and provides: (1) a basis for the Nation's physical measurement system, (2) scientific and technological services for industry and government, (3) a technical basis for equity in trade, and (4) technical services to promote public safety. The Bureau's technical work is performed by the National Measurement Laboratory, the National Engineering Laboratory, and the Institute for Computer Sciences and Technology.

THE NATIONAL MEASUREMENT LABORATORY provides the national system of physical and chemical and materials measurement; coordinates the system with measurement systems of other nations and furnishes essential services leading to accurate and uniform physical and chemical measurement throughout the Nation's scientific community, industry, and commerce; conducts materials research leading to improved methods of measurement, standards, and data on the properties of materials needed by industry, commerce, educational institutions, and Government; provides advisory and research services to other Government agencies; develops, produces, and distributes Standard Reference Materials; and provides calibration services. The Laboratory consists of the following centers:

Absolute Physical Quantities[2] — Radiation Research — Thermodynamics and Molecular Science — Analytical Chemistry — Materials Science.

THE NATIONAL ENGINEERING LABORATORY provides technology and technical services to the public and private sectors to address national needs and to solve national problems; conducts research in engineering and applied science in support of these efforts; builds and maintains competence in the necessary disciplines required to carry out this research and technical service; develops engineering data and measurement capabilities; provides engineering measurement traceability services; develops test methods and proposes engineering standards and code changes; develops and proposes new engineering practices; and develops and improves mechanisms to transfer results of its research to the ultimate user. The Laboratory consists of the following centers:

Applied Mathematics — Electronics and Electrical Engineering[2] — Mechanical Engineering and Process Technology[2] — Building Technology — Fire Research — Consumer Product Technology — Field Methods.

THE INSTITUTE FOR COMPUTER SCIENCES AND TECHNOLOGY conducts research and provides scientific and technical services to aid Federal agencies in the selection, acquisition, application, and use of computer technology to improve effectiveness and economy in Government operations in accordance with Public Law 89-306 (40 U.S.C. 759), relevant Executive Orders, and other directives; carries out this mission by managing the Federal Information Processing Standards Program, developing Federal ADP standards guidelines, and managing Federal participation in ADP voluntary standardization activities; provides scientific and technological advisory services and assistance to Federal agencies; and provides the technical foundation for computer-related policies of the Federal Government. The Institute consists of the following centers:

Programming Science and Technology — Computer Systems Engineering.

[1]Headquarters and Laboratories at Gaithersburg, MD, unless otherwise noted; mailing address Washington, DC 20234.
[2]Some divisions within the center are located at Boulder, CO 80303.

User Evaluation of Crystal Data Products and Services: Questionnaire Analysis and Impact

J. K. Stalick
A. D. Mighell
R. J. Boreni

Center for Materials Science
National Measurement Laboratory
National Bureau of Standards
Washington, DC 20234

* Technical Note

U.S. DEPARTMENT OF COMMERCE, Philip M. Klutznick, Secretary

Luther H. Hodges, Jr., Deputy Secretary

Jordan J. Baruch, Assistant Secretary for Productivity, Technology and Innovation

NATIONAL BUREAU OF STANDARDS, Ernest Ambler, Director

Issued June 1980

National Bureau of Standards Technical Note 1112
Nat. Bur. Stand. (U.S.), Tech. Note 1112, 42 pages (June 1980)
CODEN: NBTNAE

U.S. GOVERNMENT PRINTING OFFICE
WASHINGTON: 1980

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402
Price $2.25
(Add 25 percent additional for other than U.S. mailing)

TABLE OF CONTENTS

USER EVALUATION OF CRYSTAL DATA PRODUCTS AND SERVICES: QUESTIONNAIRE ANALYSIS AND IMPACT

J. K. Stalick, A. D. Mighell, and R. J. Boreni

Center for Materials Science
National Measurement Laboratory
National Bureau of Standards
Washington, D. C. 20234

A survey was made of the needs of the users of Crystal Data Determinative Tables and related products and services of the NBS Crystal Data Center. The results indicate a high frequency of use of Crystal Data, with particular application to materials analysis and design. The survey suggests a need for the development of a rapid and inexpensive method of unit-cell determination as well as education of the scientific community in the use of the single-crystal method for identification of unknown materials. More complete coverage and consolidation of entries are also suggested. The necessity for computer-based methods of data base dissemination is indicated, with particular importance for research applications.

Key Words: Computer dissemination; Crystal Data; identification; materials analysis; materials design; NBS Crystal Data Center; user evaluation.

1.0 Introduction

The NBS Crystal Data Center is responsible for the evaluation and compilation of crystallographic data on inorganic substances for which unit-cell data have been reported, and for the dissemination of data on both organic and inorganic substances to the scientific community. This activity is in accordance with the Standard Reference Data Act of 1968, which states in Section 1 that "The Congress hereby finds and declares that reliable standardized scientific and technical reference data are of vital importance to the progress of the Nation's science and technology. It is therefore the policy of the Congress to make critically evaluated reference data readily available to scientists, engineers, and the general public." The Crystal Data Center is sponsored jointly by the Office of Standard Reference Data and by the Ceramics, Glass, and Solid State Science Division of the Center for Materials Science.

The principal output of the Crystal Data Center has been the publication of Crystal Data Determinative Tables. In 1978 Volumes 3 and 4 of the 3rd Edition series were published jointly by the U. S. Department of Commerce, National Bureau of Standards, and the JCPDS--International Centre for Diffraction Data. In the Determinative Tables the compounds are ordered by crystal system and by Crystal Data cell ratios. The Crystal Data Space-Group Tables, published in 1977 in the Journal of Physical and Chemical Reference Data, contains a tabulation of compounds appearing in Volumes 1 and 2 of the Determinative Tables arranged by space group, and the Crystallochemical Identification Tables (in preparation) will contain all compounds in the Crystal Data File ordered by reduced cell parameters.

In the eleven years since the Standard Reference Data Act was passed, the demand and need for standard reference data as compiled and evaluated by experts at various data centers has increased substantially. There are three main reasons for this:

(1) Data explosion. Standardized and critically evaluated data subsets are essential because the entire set of physical and chemical data is so large that it is not subject to facile evaluation and dissemination. Chemical Abstracts has now registered 5 million compounds, with a yearly increase of 360,000 substances. Only the data centers can deal in a meaningful way with specified subsets of this data.

(2) Quality improvement. Data centers have developed sophisticated computer algorithms to evaluate published data, in addition to evaluation by experts in the field. Errors are detected and corrected so that the compiled data are significantly better than the data in the primary literature. Such evaluated data are invaluable for scientific research and computer modeling.

(3) Data dissemination. Recent developments in computer hardware and software have totally changed the way in which data can be disseminated. Computer-based data files make possible a much closer interaction between the data center and the user (e.g. it is possible to supply any desired subset of the data base) and greater interaction among various data centers to permit the merging of selected data parameters. Computer methods also make possible the rapid on-line dissemination of data throughout the United States, as well as the incorporation of the data base directly into the instrument that collects the data. In addition, research using the evaluated data sets is possible through the use of data base management systems.

The NBS Crystal Data Center is now in a period of transition from a hand-based to a computer-based operation. This will permit a second transition from emphasis on a single product to a variety of products and modes of data dissemination. The questionnaire on Crystal Data was designed to gain the information required to assist the Crystal Data Center in this transition stage, particularly with respect to the design and content of the new computer file structure. In view of the limited resources available for the data compilation effort, it was deemed necessary to obtain information on the cost-effectiveness of the inclusion of various data items and products and to set priorities for the allocation of these resources while still meeting the prime needs of the users. An additional benefit would be the location of users willing to help with the Crystal Data project.

Consequently a survey was conducted to find out who the users are, what data they want, the products and services they want, and how they use crystallographic data in their work. This information will be of great value in determining the directions in which the NBS Crystal Data Center should concentrate its efforts.

2.0 Analysis of Response

The questionnaire was distributed in December of 1978 and January of 1979 by the NBS Associateship of the JCPDS-International Centre for Diffraction Data. Since no mailing list of users or purchasers of Crystal Data existed, the questionnaire mailing list was selected from that of the JCPDS and, in a few cases, from the World Directory of Crystallographers (Fifth Edition, 1977). The total number of questionnaires distributed was 1058. The number returned was 235, or 22%. Taking into account the difficulty in compiling the mailing list, the out-of-date addresses of the recipients (many addresses on the list were more than 10 years old), and the extreme length of the questionnaire, this represents a remarkable return rate and indicates a high degree of interest in the Crystal Data publications.

The questionnaire was divided into six sections. The first section requested information describing the respondent. The next three sections dealt with the use and content of Crystal Data publications, and the final two sections concerned published products and computer-based distribution of the NBS Crystal Data File. A copy of the questionnaire is given in Appendix A, with a summary of the answers shown for each question.

2.1 Description of Respondent

The first seven questions requested information on nature of employment and personal data. In all cases the totals add up to greater than 100% of those responding, as one or more categories could be checked.

2.1.1 Type of Work and Employer

Most respondents classified their type of work as basic research (71%) and/or applied research (44%), with significant numbers also involved in education (33%) and analysis (26%) (Table 1). The types of employers are given in Table 2, with university or college (43%) and industrial (37%) predominating.

Table 1. Present Type of Work (I-1)*

Category	Number of Respondents (%)
Basic research	168 (71%)
Applied research	104 (44%)
Education	78 (33%)
Analysis	62 (26%)
Product development	29 (12%)
Quality control	19 (8%)
Production	13 (6%)
Other[a]	8 (3%)

[a]Catalyst development, consulting, customer service, data base production, legal, production support, student, trouble shooting.

Table 2. Type of Employer (I-2)

Category	Number of Respondents (%)
University or college	102 (43%)
Industrial	86 (37%)
Government	37 (16%)
Research institute	11 (5%)
Other[a]	5 (2%)

[a]Clinical laboratory or hospital (2), emeritus, non-profit technical services to government, personal consultant.

The respondents were asked to characterize their employer further by checking one or more of a group of listed Standard Industrial Categories (SIC), with the results given in Table 3. The results indicate a broad range of industrial classifications, with educational services (97 responses) and chemicals and allied products (51 responses) predominating. Forty-three respondents checked the box "Other [please describe]" and their descriptions are also given in Table 3.

Table 3. Standard Industrial Categories of Employer (I-3)

SIC No.	Category	Number of Times Checked
82	Educational services	97
28	Chemicals and allied products	51
36	Electrical and electronic equipment	18
32	Stone, clay, glass, & concrete products	16
34	Fabricated metal products	13
80	Health services	12
97	National security and international affairs	12
14	Mining of nonmetallic minerals	8
33	Primary metal industries	8
12	Bituminous coal and lignite mining	7
29	Petroleum refining	6
95	Environmental quality and housing	6
10	Metal mining	5
13	Oil and gas extraction	5

*Numbers in parentheses indicate question number.

Table 3 (continued)

38	Instruments and related products	5
35	Machinery, except electrical	4
84	Museums, art galleries, botanical and zoological gardens	3
91	Executive, legislative, and general government	3
11	Anthracite mining	2
81	Legal services	1
92	Justice, public order, and safety	1
	Other[a]	43

[a]The following descriptions of industrial categories were given:

Energy research (6 responses)
Government basic research (4 responses)
Communications (3 responses)
Research in earth sciences (USGS) (3 responses)
Pharmaceuticals (2 responses)
Photographic materials (2 responses)
Polymer products (2 responses)
Refractory materials (2 responses)
Textiles (2 responses)
Aerospace products
Automotive
Biochemistry - pharmacology
Consulting services
Contract research
Crystal growth for optical uses
Explosives research
Extractive metallurgy - corrosion - resource recovery
Manufacturing
Materials research
Metallurgy research and secondary materials research
Military
Nuclear energy
Ore processing
Plutonium metallurgy
Soap and detergent industry
Stimulation and support of basic research (granting agency)

2.1.2 Type of Diffraction and Materials

The respondents were also asked to characterize their work by the type of diffraction and type of materials involved in their work, with the results given in Tables 4 and 5. Both x-ray powder diffraction and single-crystal diffraction were checked as being used by 80% and 71%, respectively, of the respondents, while the types of materials involved were primarily inorganics and minerals (Table 5).

Table 4. Type of Diffraction Used (I-4)

Type	Number of Respondents (%)
X-ray powder diffraction	189 (80%)
X-ray single-crystal diffraction	167 (71%)
Electron diffraction	44 (19%)
Neutron diffraction	21 (9%)

Table 5. Type of Materials Used (I-5)

Type	Number of Respondents (%)
Inorganics	177 (75%)
Minerals	118 (50%)
Organics	81 (34%)
Ceramics	73 (31%)
Metals and intermetallics	67 (29%)
Small molecules	64 (27%)
Organometallics	59 (25%)
Catalysts	51 (22%)
Drugs	29 (12%)
Large molecules or proteins	21 (9%)
Pesticides and herbicides	13 (6%)
Other[a]	14 (6%)

[a]Polymers (3 responses), corrosion products (2 responses), explosives (2 responses), air pollutants, biological molecules, environmental materials, gaseous samples, metal salts of organics, natural products especially toxins, high explosive radioactive materials.

2.1.3 Name and Address of Employer and Respondent

The name and address of the employer were given by 177 (75%) of the respondents, and these are given in Table 6. In addition, 169 (72%) listed their own name and address, indicating a willingness to be contacted for further information (question I-7).

Table 6. Personal Employment Data (I-6)

Industrial

AMP Inc.
Abbott Laboratories
Allied Chemical Corp. (2)
Basic Inc.
Bell Telephone Laboratories, Inc. (2)
Borg-Warner
Cleveland Crystals, Inc.
Corning Glass Works
Dow Chemical Co. (3)
Dow Corning Corp.
Dresser Industries, Canadian Refractories Division
E. I. du Pont de Nemours & Co. (5)
Eastman Kodak Co. (2)
Engelhard Industries
Exxon Research and Engineering Co.
Ford Motor Co.
GTE Laboratories (2)
GTE Sylvania
General Electric
B. F. Goodrich Co.
Gould Inc.
W. R. Grace and Co.
A. P. Green Refractories Co.
The Harshaw Chemical Co.
Louis C. Herring & Co.
Hoffmann-LaRoche
Lockheed Research
The Lubrizol Corp.
3M Co.

Table 6 (continued)

Medtronic, Inc.
Olin Corp.
PPG Industries, Inc.
Phelps Dodge Corp.
Phillips Petroleum Co.
Polaroid Corp.
The Procter and Gamble Co. (2)
RCA Corp.
Raychem Corp.
Reynolds Metals
Rockwell International (2)
Shell Development Co.
Sherwin-Williams Co.
Sperry Research Center
J. P. Stevens & Co., Inc.
Sun Production Co.
Thiele Kaolin Co.
U. S. Steel
Union Carbide Corp. (2)
Union Oil Co. of California
Upjohn Co.
Warner Lambert/Parke Davis

University or College

Baylor University
Bethune-Cookman College
Brigham Young University
Brown University
California Institute of Technology
Carnegie-Mellon University
Case Western Reserve University
Colorado School of Mines (2)
Cornell College
Georgia Institute of Technology
Harvard University
Huston-Tillotson College
Indiana University
Iowa State University (2)
Johns Hopkins University
Loyola University
McGill University (2)
McMaster University
Menlo College
Michigan State University (2)
Montana State University
The Ohio State University
Pennsylvania State University
Portland State University
Purdue University
Queen's University
Roanoke College
Rutgers University
Simon Fraser University
State University of New York at Buffalo (3)
Texas A&M University (3)
University of Alaska
University of Arizona
University of British Columbia
University of California at Berkeley
University of California at Los Angeles

Table 6 (continued)

University of Cambridge
University of Chicago (4)
University of Colorado (2)
University of Connecticut
University of Heidelberg
University of Illinois at Chicago
University of Iowa
University of Louisville
University of Michigan
University of Minnesota
University of Nebraska
University of New Brunswick, Fredericton
University of New Hampshire
University of North Carolina
University of Oregon
University of Pittsburgh
University of Rhode Island
University of Southern California
University of Vermont
University of Virginia
University of Waterloo
University of Wisconsin at Milwaukee
University of Wyoming
Vanderbilt University
Virginia Polytechnic Institute & State University
Wake Forest University

Government and National Laboratories

Air Force Office of Scientific Research
Argonne National Laboratory
Armament Research and Development Command (ARRADCOM)
Brookhaven National Laboratory
Geological Survey of Canada
Institute of Forensic Sciences
Lawrence Berkeley Laboratory
Lawrence Livermore Laboratory (2)
Los Alamos Scientific Laboratory
National Aeronautics and Space Administration
National Research Council of Canada (2)
Naval Research Laboratory (4)
Naval Surface Weapons Center, White Oak Laboratory
Oak Ridge National Laboratory (2)
Sandia Laboratories (4)
U. S. Army Engineer Waterways Experiment Station
U. S. Bureau of Mines (2)
U. S. Geological Survey (4)

Research Institutes

Battelle Northwest Laboratories
Colorado School of Mines Research Institute
Denver Research Institute
Midland Macromolecular Institute
Royal Ontario Museum

Other

The Aerospace Corp.
Mt. Sinai Hospital

2.2 Use of Crystal Data

In this section eleven questions were asked concerning the use of Crystal Data Determinative Tables and Crystal Data Space-Group Tables.

2.2.1 Use of Crystal Data Determinative Tables

Question II-1 asked, "How often do you use Crystal Data Determinative Tables in the following way [use 3 = frequently, 2 = occasionally, 1 = rarely, 0 = not at all]". Of the 212 respondents answering all or part of this question, 103 (49%) use Crystal Data frequently in one or more ways, and another 92 (43%) use it occasionally. Only 17 respondents (8%) reported that they use Crystal Data rarely. Also, 172 respondents (81%) reported occasional or frequent use in more than one category, so that the combined usage is high. The responses indicating relative frequency of use are summarized in Table 7.

It can be seen that Crystal Data is most commonly used as a reference book, primarily to obtain structural information; that is, to find cell dimensions, to find out if a particular structure has been determined, or to obtain information about structure types. It is used to identify unknown substances using the determinative ratio less often. Seventeen respondents checked the box marked "other" and their responses are also given in Table 7.

Table 7. Use of Crystal Data Determinative Tables (II-1)

Number of Respondents	Average Rating[a]	Type of Use
212	1.8	To find cell dimensions or space group of a particular substance
211	1.7	To find out if the structure of a particular substance has been determined.
205	1.5	To find information about structure types or to locate isostructural materials.
209	1.4	To locate literature references containing crystal data on a particular substance or structural group.
204	1.1	To identify an unknown substance using the determinative ratio.
202	0.9	To obtain information about physical properties of a substance
17		Other[b]

---

[a] In this and succeeding questions the average rating was computed according to the following equation, where N(i) represents the number of times a given response [3,2,1,0] was checked:

$$\text{average rating} = \frac{3[N(3)] + 2[N(2)] + N(1)}{N(3) + N(2) + N(1) + N(0)}$$

[b] The following responses were given:

1. Educational purposes for non-specialists
2. To find the formula of a mineral
3. As a literature stockroom
4. To find similar mineral classifications (e.g. asbestos types)
5. To check out cell dimensions of compounds

6. To assist in indexing and generating powder diffraction data when only single crystal reference data is available
7. For qualitative identification, not using the determinative ratio
8. For phase determinations
9. I use it when I am baffled
10. To determine degree of orientation
11. Basic reference in teaching/research crystallography lab
12. For research [this response is given in full in the discussion of question II-5]. Much more work of this sort could be based on Crystal Data.
13. In the study of polytypism, e.g. SiC
14. Routine use in characterization of solids
15. To check conventions regarding axial lengths etc.
16. When powder data fails for identification
17. To check powder data and other crystal data quoted in the literature

The next question (II-2) requested information on the areas in which the respondents use Crystal Data, with the results summarized in Table 8. The type of study checked was predominantly crystal structure analysis (143), with significant use in phase equilibria (53), materials processing (42), high or low temperature studies (40), corrosion (33), and catalysis (29). The types of materials most frequently checked were ceramics (51) and semiconductors (29), although magnetic materials, optical materials, and energy conversion materials were also important.

Table 8. Areas of Use of Crystal Data (II-2)

No. of Times Checked	Type of Study
143	Crystal structure analysis
53	Phase equilibria
42	Materials processing
40	High or low temperature studies
33	Corrosion
29	Catalysis
17	Fracture or wear studies
17	High pressure studies
16	Diffusion in metals
11	Failure prevention
10	Materials durability

No. of Times Checked	Type of Material
51	Ceramic materials
29	Semiconductor materials
23	Magnetic materials
23	Optical materials
20	Energy conversion materials

2.2.2 Duplication of Crystal Structures and Unit Cell Determinations

The next two questions concerned duplication of effort that in many cases could have been prevented if the appropriate reference data had been consulted or had been available. Question II-3 asked, "Have you ever solved a structure or collected data and then found that your work was unnecessary because the same or similar substance had already been solved? If yes, how often has this happened? What is your dollar estimate of the cost involved (computer time, lost hours, etc.) for each occurrence?" "No" was checked by 159 respondents; of the 64 respondents who checked "yes", 59 explained further. Table 9 summarizes the number of structure duplications reported. Of the more than 67 structures redetermined, 10 were commented on as still being of value, and another 10 redeterminations could not have been prevented as the data had not yet been published. The cost in computer time (excluding equipment costs and personnel costs) was estimated by 29 respondents, and ranged from $300 to $15,000 per structure with a median of $2000. Only 12 respondents attempted to estimate the time lost per structure, ranging from "trivial" to "4 years of graduate school", with a median of two weeks.

Table 9. Frequency of Crystal Structure Redetermination (II-3)

Number of Respondents	Number of Structures Redetermined
159	0
31	1
12	2
2	3
1	6
5	Not given (but checked "yes")
4	Don't know (but checked "yes")
6	Not often or occasionally
3	Other[a]

[a]Once a year; 5% of the time; 1-2 per year.

The respondents were next asked to give the same type of information with respect to the redetermination of unit cell parameters (rather than complete structure determinations). The response indicates that the redetermination of cell parameters is not as serious as the redetermination of a crystal structure. Although the number of unit cell redeterminations reported was comparable to that reported for crystal structures, several respondents indicated that this in fact represents a saving of time and money as the compound involved was not what was originally suspected and thus the crystal structure was not duplicated. Of the 63 respondents who checked "yes", 55 commented on the number of occurrences or cost. Table 10 summarizes the number of unit cell redeterminations reported. The cost was estimated by 28 respondents and was an order of magnitude less than that for a crystal structure determination, ranging from $5 to $5000 with a median cost of $200. The time involved was not indicated.

Table 10. Frequency of Unit Cell Redetermination (II-4)

Number of Respondents	Number of Cells Redetermined
158	0
12	1
6	2
5	3
2	6
19	Not given (but checked "yes")
7	Not often or occasionally
6	Done for qualitative identification
2	Don't know (but checked "yes")
4	Other[a]

[a]Several times; 5% of the time; one per year; 3-4 per year

2.2.3 Specific Applications of Crystal Data Determinative Tables

Question II-5 asked the respondents to describe one or more specific applications of Crystal Data Determinative Tables. The 106 responses were more general than specific, but provided some very interesting statistics and uses not indicated in the preceding questions. The responses are given in full in Appendix B, grouped by user categories. An attempt to group the responses into various areas of application is summarized in Table 11.

Table 11. Areas of Application of Crystal Data Determinative Tables (II-5)

Number of Respondents	Area of Application
43	Materials analysis, qualitative identification
20	Aid in research applications
19	Assign structure type, search for isostructural materials, or find trial structure
8	For literature search or location of cell constants
7	To find out if a structure has been determined
5	As an aid in teaching and design of experiments
4	Other[a]

[a]See question II-1 (3); need for a computer readable data base.

The response indicates that Crystal Data is used in a complex variety of ways, with some users seeking only bibliographic information, some only cell data, some only structure information, etc. However, the value of Crystal Data as a general purpose reference book was stressed by several respondents. One professor at a major university gave the following uses: "Educational purpose: to find a compound suitable for basic training of students in course work. To find possible isostructural or related compounds pertaining to current research. Many of my colleagues use the Tables, which I keep accessible to them in my office. There is such a large variety of occasions where the Tables come in handy, I cannot list them all."

2.2.3.1 Materials Analysis and Qualitative Identification

The most frequent use of Crystal Data is seen to be in the area of materials analysis and qualitative identification (Table 11). Although the response to question II-1 indicated that Crystal Data is not primarily used to identify an unknown by using the determinative ratio, many users have some idea of the chemical composition and use the indexes to locate possible materials. A consultant for a private firm said in part, "I often have an unknown crystal for which I have chemical information . . . and limited optical crystallographic data . . . Using Crystal Data as a source of information I may be able to pick out likely possibilities and be thus aided in choosing the next step towards identifying the unknown."

Two further examples help to illustrate the use of Crystal Data for materials analysis. A university professor stated, "Local phosphoric acid plant was having trouble with an unknown compound precipitating in pumps and pipes. Was able to identify compound and advise on how to manage settling ponds to avoid problem." Also, an industrial user commented, "We use our x-ray diffraction mainly to identify reaction compounds which form in refractories while they are in use. The data is used to suggest refractories which might provide better service in such applications, or it may aid in the development of improvements in the refractory products."

In other cases, the investigator does not know what type of material to expect. For example, one university respondent told the following: "We made a high pressure run in an attempt to prepare a specific new compound. An x-ray powder photograph of the result showed that a reaction had occurred and that none of the original material was present. The starting elements were Nb and Te. The powder photograph showed a cubic material with a = 4.38 to 4.40Å. A quick look through Crystal Data revealed that the 'new' material was almost certainly NbN (with the N from BN in reaction chamber)." In another case, a user at a large manufacturing company said, "A small (approximately 1000Å) crystallite was isolated in a piece of human tissue. The crystal provided an electron diffraction pattern. Only elemental information and two-dimensional diffraction data [were available]. The crystallite was identified with the aid of Crystal Data."

2.2.3.2 Research Applications

Another major area of use of Crystal Data is in research applications (see Table 11).

A primary crystallographic research area involves structure types and relationships. One respondent indicated the importance of Crystal Data in this field with the following comments: "Enclosed are reprints of two papers in which references to Crystal Data play an important part. The paper on formates shows some of the shortcomings of Crystal Data [inconsistent data on rare-earth formates in Crystal Data was the impetus for further structural work], and the paper on the F51 type shows how much information can be gleaned from Crystal Data [various structure types reported were explained on the basis of information given in Crystal Data]. Much more work of this sort could be based on Crystal Data."

Several other examples were given in which the structural relationships found in Crystal Data can be used in materials research. These include the following three responses, each from a representative of a major industrial corporation: (1) "A search for a number of isotropic substances was desired. Searching in the cubic section was most useful, yielding many compounds which fit our particular needs." (2) "[Crystal Data was used to] look for oxides having spinel structure and having lattice parameter within a certain range. These spinels would be possible substrates for epitaxial growth of other compounds." (3) "Searched for all compounds crystallizing with a given a and b dimension (indeterminate) and a given angle between a and b for use in layer stacking studies."

The responses also indicate other areas of research in which the information contained in Crystal Data is useful. These include theoretical calculations, bonding electron distribution studies, interpretation of radial distribution functions and extended x-ray absorption fine structure data, correlation of polytypes with luminescence of SiC, and calculation of theoretical powder patterns.

2.2.3.3 Isostructural Materials and Assignment of Structure Type

The third major area of use of Crystal Data given in Table 11 is to assign a structure type, to search for isostructural materials, or to find a trial structure for a crystal structure determination. A typical response was, "Having a unit cell, I would use the Tables to see if the structure could be easily guessed by comparing it to known compounds." The following two comments were from university professors: (1) "A new mineral, maricite, $NaFePO_4$, had its structure determined by careful checking against axial ratios and cell dimensions of similar compounds." (2) "[Crystal Data was used] to determine if a cubic organomercury compound had been reported. Found that cell edge and space group were identical to that of $Ba(NO_3)_2$. Powder patterns proved to be practically identical. These data were used to determine the most probable arrangement of the ligands in the unknown."

The remaining areas of application of Crystal Data given in Table 11 are primarily bibliographic. These include using Crystal Data to find literature references or cell dimensions, to find out if a structure has been determined, and to aid in teaching and the design of experiments.

2.2.4 Use of the Single-Crystal Method and the Powder Method of Identification

The next two questions dealt with the use of the single-crystal method and the powder method of identification. Question II-6 asked, "Do you need to identify unknown crystalline substances in your work? If yes, do you rely mainly on the powder method (ASTM-JCPDS file) or the single-crystal method?" Thirty respondents checked "no". Of the 200 respondents who checked "yes", most relied primarily on the powder method of identification. The results are given in Table 12.

Table 12. Use of the Single-Crystal Method and the Powder Method (II-6)

Number of Respondents	Method of Identification Used
105	Use mostly powder method but also use Crystal Data for confirmation or for literature references
60	Use powder method exclusively
46	Use primarily single-crystal method

In an attempt to find out why the powder method is preferred for identification of unknown materials, the following question was asked: "Do you grind up single crystals in order to use the powder method of identification? If yes, why do you not use the single-crystal method [check as many as apply]?" Most respondents (132) answered "no", but a significant number (87, or 40%) checked "yes". The reasons given for using the powder method when single crystals are available are summarized in Table 13.

Table 13. Reasons for Not Using the Single-Crystal Method (II-7)

Number of Respondents	Reason
30[a]	Powder method is easier, faster, routine
28	Too difficult to obtain a good single crystal
25	Do not have the necessary equipment
19	Tradition
14	A trained technician is required
9	Crystal Data is not up to date
6	Too difficult to calculate a unit cell
6	The equipment is too expensive
5[a]	Use a Gandolfi camera instead
4	Too complicated to transform the unit cell to the Crystal Data cell to get the determinative ratio
2[a]	The PDF file is available for computer search
20	Other[b]

[a]The responses occurring more than once under "other" have been included in the table; the number of times these responses might have been checked if they had been included in the questionnaire is unknown, but probably much greater than indicated.

[b]The following reasons were given:

1. Used as standard for powder diffraction.
2. Usually have mixtures, rather than pure substances, for analysis.
3. We involve all supporting techniques.
4. Different type of data in powder file. Somehow, one has the feeling that if you fit 20-40 powder lines (both d and I) it is surer than fitting 6 unit cell dimensions. But we have been successful using cell constants too.
5. We are usually given powders.
6. If there are several crystals, why not?
7. Educational training.
8. Use single crystal when we can't find diagram in powder file.
9. Single crystal [equipment] is usually in use by another individual.
10. To grind up single crystals to powder is useful for "fingerprinting purposes" of complex compounds to compare batches etc. during syntheses.
11. Generally powder patterns for comparison are more readily available.
12. Small sample.
13. To reduce size of sample crystallites.
14. We may have a group of crystals expendable.

Table 13 (continued)

15. The powder data gives 20 or more pieces of information and therefore gives normally a completely unique identification. Lattice constants and their ratios typically do not give a unique identification. Also, data are not as complete. Many substances have published powder patterns but no lattice constants.
16. To confirm solid solution etc.
17. Macromolecules rarely yield large single crystals.
18. For comparison of samples not crystallized [microcrystalline], need powder patterns.
19. Too many students and not enough time for equipment available.
20. The excuses listed above are those of people who are either ill-informed, lazy, or prejudiced.

It can be seen from Table 13 that the powder method is used, rather than the single-crystal method, primarily because it is easier, faster, and more routine, or because equipment necessary for the single-crystal method is not available. Since the single-crystal method of identification is a powerful technique, it is hoped that these objections will be overcome. Responses such as numbers 4 and 15 listed under "other" also indicate a need for education, even among crystallographers; the fact that the Crystal Data file is roughly twice the size of the powder data file and the fact that the unit cell parameters give fewer possibilities for an unknown than given by comparison of powder data need to be publicized more.

Since many substances may appear in either the Powder Diffraction File or the Crystal Data File, but not both, the powder method and the single-crystal method for identification should be used in a complementary fashion. The following example of this type of use was given in response to question II-5: "Using the JCPDS file, a material was identified as a compound it could not possibly be. The cell constants for that material were used [to calculate] the determinative ratios and Crystal Data consulted for compounds around that ratio. The correct material was found easily. The pattern for this material did not exist in the JCPDS file."

2.2.5 Use of Crystal Data Space-Group Tables

The next question concerned the use of Crystal Data Space-Group Tables and Nowacki's Systematic Tables, in which compounds are grouped according to crystal system and space group. Question II-8 asked, "Do you use the Crystal Data Space-Group Tables or Nowacki's Systematic Tables? If yes, briefly describe one or more applications to your work." Most respondents (150) answered "no". However, 66 (roughly one-third the number of users of Crystal Data Determinative Tables) checked "yes", and 38 commented on their use. Again, the responses were more general than specific; the responses are given in full in Appendix C, grouped by user categories. An attempt was made to group the responses by area of application, with the results summarized in Table 14.

Table 14. Areas of Application of Crystal Data Space-Group Tables and Nowacki's Systematic Tables (II-8)

Number of Respondents	Area of Application
10	To locate isostructural compounds, compounds with specific space groups, or related materials
8	As a research tool; relation of symmetry to structure
7	To determine or verify space groups

Table 14 (continued)

4	As an aid to identification
2	As an aid to teaching
7	Other (used occasionally, or only for interest)

Although the number of respondents was not large, the areas of application indicated can be quite useful in materials science. For example, one industrial user responded: "Compounds with certain space groups were predicted to have properties which we wanted to study. The Space-Group Tables provided a starting point: determining what compounds had those space groups so that substituted compounds might also be studied." Another respondent at a government laboratory indicated that the Space-Group Tables were used to "search for isomorphous compounds of explosive or oxidizer nature so as to make mixed crystals of same." Areas of research application indicated include group-subgroup relationships, correlation between chemical class and space group, theoretical relations of symmetry to structure, and use in single-crystal ESR and NMR work.

2.2.6 Suggestions for Improvement of Crystal Data Determinative Tables

The respondents were next asked to comment on ways in which Crystal Data Determinative Tables could be improved (question II-9). There were 81 responses, and those given two or more times are summarized in Table 15. It is obvious that many users are concerned about the out-of-date coverage of the inorganic materials, with 30 respondents requesting more complete coverage, keeping Crystal Data current, or annual updates between editions. Sixteen respondents commented that they were satisfied with Crystal Data as it is. The existence of multiple entries for several compounds was commented on by eight respondents, with the suggestion that these entries be consolidated, the out-of-date data eliminated, and the data evaluated so that the best data are indicated.

Table 15. Ways To Improve Crystal Data Determinative Tables (II-9)

Number of Respondents	Comment
30	Keep up to date; more complete coverage; annual updates between editions.
16	Crystal Data is a good book as it is.
8	Consolidate entries; eliminate out-of-date data; evaluate the entries and indicate the best data.
4	Add cross-references to other data bases (Cambridge, JCPDS, Structure Reports, etc.).
3	Include more information on physical properties.
3	Include more optical data.
2	Make the data base available for computer search.
2	Reduce the cost of the books, so that they may be purchased for personal use.
2	Include data on solid solutions.
2	Add powder data, or references to powder data.

2.2.7 Evaluation of Data Prior to Publication

The next question (II-10) asked, "If a paper submitted for publication contains crystal data, do you think the data should be evaluated (e.g. checked for inconsistencies) prior to publication as part of the journal review process?" Of the 223 responses to this question, 184 (83%) said "yes", and 39 (17%) answered "no". Several of those who felt this evaluation should not be done added comments that they were concerned about the time it would take and the cost.

2.2.8 Assistance to the Crystal Data Project

Finally, the respondents were asked if they would be willing to assist the Crystal Data project (question II-11). Most respondents (137) said they would be willing to send in crystal data directly prior to publication, but a significant number (65) said they would not. When the respondents were asked if they would be willing to help by abstracting a class of compounds from the published literature or by abstracting crystal data for all compounds from a given journal, 141 replied "no" and 51 replied "yes". The respondents who are willing to help with the project (nearly 22% of those returning the questionnaire) represent a valuable resource for future editions of Crystal Data, as well as indicating the interest in and value of this compendium.

2.3 Supplementary Content of Crystal Data Entries

In addition to the basic Crystal Data information (unit cell, space group, number of molecules per unit cell, information on whether or not the structure has been determined, measured and calculated densities, chemical name and formula, and literature reference), the respondents were asked to indicate the desirability of including additional data in a typical Crystal Data entry. Question III-1 asked for the importance to the user of seven types of information, with the results summarized in Table 16. The rating scale was 3 = high value, 2 = moderate, 1 = marginal, and 0 = no value. As indicated in the table, most information was checked as being moderately useful, with the source of cell data considered most valuable. Of particular interest to this study was the lack of confidence in the Chemical Registry Number as assigned by Chemical Abstracts, which was rated as only marginally important. Two respondents added comments expressing dissatisfaction with the Chemical Registry Number.

Table 16. Importance of Supplementary Data (III-1)

Number of Respondents	Average Rating[a]	Item
208	2.5	Source of cell data (single-crystal or powder work)
204	2.2	An indication that indexed powder data may be found in the literature reference
207	2.1	An indication that recorded powder data may be found in the literature reference
204	2.1	Cross-reference to the JCPDS powder data file
203	1.8	Specific method of obtaining cell data
202	1.6	Refined R-value
191	0.9	Chemical Registry Number

[a]See definition in Table 7.

The next question concerned the importance of supplementary information that has traditionally been included in Crystal Data Determinative Tables. The respondents were asked to indicate if it is useful to have information in the commentary on several data items, with the results given in Table 17. The data items were rated as being moderately useful to marginally useful, with the same rating scheme as used in question III-1. The two data items that concern the primary crystallographic parameters, the existence of sub-, super-, or pseudocells and the temperature of data taking, were rated as most useful.

Table 17. Rating of Data Items (III-2)

Number of Respondents	Average Rating[a]	Item
197	2.0	Existence of sub-, super-, or pseudocells
198	2.0	Temperature of data taking
196	1.9	Melting point
200	1.8	Color
192	1.8	Indication of neutron or electron diffraction data
194	1.8	Twinning
199	1.7	Habit
199	1.7	Refractive indices
195	1.6	Solid solution studies
202	1.5	Source of polymorphic forms
195	1.5	Cleavage planes
196	1.3	Goniometric axial ratios
193	1.3	Thermal expansion studies
191	1.2	Locality for minerals
192	1.1	Pleochroism
191	1.0	Compressibility studies

[a]See definition in Table 7.

2.4 Literature Coverage

Two questions were asked concerning the handling of references when more than one paper has appeared containing cell constants for a given material. Question IV-1 stated, "Many publications containing unit-cell dimensions do not give new cell or structural data, but may provide additional crystallographic information such as an indexed powder pattern, solid solution data, thermal expansion data, or cell data for a mineral of slightly variant composition. References of this type should be treated as follows [check one]". The responses checked are given in Table 18. The majority of respondents answering this question (145, or 69%) checked that these papers should be given only as a reference with an indication of the type of information to be found, while 43 (20%) felt that they should be reported in full, including unit-cell dimensions.

Table 18. Treatment of Multiple References to a Given Material (IV-1)

Number of Respondents	Response Checked
145	Given only as a reference with an indication of the type of information to be found in the paper
43	Reported in full, including unit-cell data
15	Given only as a reference
6	Omitted from Crystal Data
2	Other[a]

[a]Significant new data may be a useful criterion; the underlined data [powder pattern] I would expect to find in the JCPDS Powder Diffraction File.

The next question (IV-2) asked if, to facilitate data handling, it would be acceptable to publish annotated references of the above type as a supplement to Crystal Data, with appropriate indexes. This format was checked as being acceptable by 145 respondents, while 41 checked that this would not be desirable.

2.5 NBS Crystal Data Center Published Products

The respondents were asked to rate potential Crystal Data products based on importance to their work, using the scale 3 = high value, 2 = moderate, 1 = marginal, 0 = no value. The results are summarized in Table 19.

Question V-1 concerned Crystal Data Determinative Tables. The publication of a supplement to the inorganic section for 1970-1978 was rated at high to moderate value, with 114 respondents (59%) indicating this would be of high value to their work. This result is in agreement with the response to question II-9, and underlines the importance of bringing Crystal Data up to date. The publication of a cumulative name and formula index for the inorganic volumes was rated as being of moderate value, with one respondent commenting that "a cumulative index begins to be of value if more than two volumes are covered".

The respondents were asked to give the value of bringing Crystal Data up to date more rapidly for certain categories (question V-2). Only the category of refined inorganic quantitative structures was rated as of moderate value, with the special publication of Crystal Data for minerals, metals and intermetallics, or oxides only rated as of marginal to moderate value. Since these publications would be of interest to smaller groups of users, this reply is to be expected. The following numbers of respondents felt that these separate publications would be of high value: refined inorganic quantitative structures, 76 (41%); minerals, 68 (36%); oxides, 40 (22%); metals and intermetallics, 35 (20%). Two respondents expressed concern about these special publications. One commented, "I disapprove in principle of this kind of approach. If you give a person a book with data on minerals only, he or she will implicitly assume that everything to be sought can be found in it and that isn't true".

The publication of the Identification Tables (question V-3) was rated as being of slightly greater than moderate value, with 79 respondents (41%) rating it as of high value to their work and 58 (30%) as of moderate value. The response indicates that this could be a valuable new publication.

Finally, a second issue of Crystal Data Space-Group Tables (question V-4) was rated as being only of marginal to moderate value, with 22 respondents (12%) rating it as being of high value. This result is in agreement with the lack of general use indicated in question II-8.

Table 19. NBS Crystal Data Center Published Products (V-1, V-2, V-3, V-4)

Number of Respondents	Average Rating[a]	Product
		V-1. Crystal Data Determinative Tables
192	2.4	A supplement to the inorganic section of Crystal Data covering the literature for 1970-1978
188	2.0	A cumulative name and formula index for the 3rd Edition of Crystal Data for Volume 2 (inorganic literature through 1966) and Volume 4 (inorganic literature 1967-1969)
		V-2. Crystal Data for special categories
187	2.0	Crystal Data for refined inorganic quantitative structures only (1970-1978)
188	1.7	Crystal Data for minerals only (1970-1978)
178	1.5	Crystal Data for oxides only (1970-1978)
178	1.3	Crystal Data for metals and intermetallics only (1970-1978)
40		Other[b]
		V-3. Crystallochemical Identification Tables
192	2.1	This is a new product designed for the rapid identification of unknown materials. For a given compound, an entry would consist of one line of key crystallographic and chemical data (reduced cell, space group, formula, density, and literature reference). Data from all volumes of Crystal Data would be included.
		V-4. Crystal Data Space-Group Tables
178	1.4	A second issue of Crystal Data Space-Group Tables including data coincident with Volume 3 (organic) and Volume 4 (inorganic) of Crystal Data Determinative Tables. The first issue (J. Phys. Chem. Reference Data, 6, 675, 1977) is coincident with Volumes 1 and 2 of the 3rd Edition of Crystal Data.

[a] See definition in Table 7.

[b] Organics and organometallics (17), sulfides (5), proteins and biological macromolecules (4), fluorides (2), zeolites (2), phosphates (2), silicates, pharmaceutical biochemicals, sulfates, semiconductors, hydroxides, halides, polymers, explosives

2.6 Crystal Data File Searches and Analyses

This section concerned computer-based methods of dissemination and use of the Crystal Data file. The increased use of computer search techniques can be seen in the responses given. As one user commented, "the size of Crystal Data makes computer methods imperative."

2.6.1 Computer-Based Methods of Dissemination

Questions VI-1 and VI-2 asked the respondents to compare three potential methods for computer search of the data base:

Method A. On-line Interactive Search System

> The Crystal Data file will soon be available to the public as a component of CIS (Chemical Information System of NIH/EPA). This system is an on-line interactive search system; the user queries the file via a terminal and receives an answer in a very short time. For example, one could find all compounds with a given cell, space group, or formula within a few minutes.

Method B. Batch Search System at Central Location

> Such a system would be operated at a central location with a resident crystallographic expert to interface between the user's query and the batch-operated computer system. Questions would be received and answered via mail or telephone. Several days might be required for the process.

Method C. User's Organization to Buy Computer Data Base

> In this system the organization would pay for the data base, appropriate software, and yearly updates. The organization and the users would then decide on the most appropriate way to use the file.

The large size of the Crystal Data file makes computer searches a valuable asset. In question VI-1, the respondents were asked to indicate how important such a search ability would be in their work for methods A, B, and C [3 = high value, 2 = moderate, 1 = marginal, 0 = no value], and to indicate the maximum acceptable cost if possible. The results are summarized in Table 20. It can be seen that method A was preferred by most users, with 64 respondents (33%) rating the on-line interactive search method as being of high value, and 60 (31%) as of moderate value in their work. For method B (batch search system at a central location), 21 respondents (11%) indicated high value and 44 (24%) indicated moderate value; for method C (user's organization to buy computer data base) 25 respondents (15%) indicated high value and 26 (15%) indicated moderate value.

Table 20. Methods of Computer File Search (VI-1)

Method	Number of Respondents	Average Rating[a]	Number of Respondents	Median Cost
A	194	1.8	82	$10 per query
B	184	1.2	47	$10 per query
C	168	1.0	29	$500 for file

[a] See definition in Table 7.

Many fewer respondents attempted to estimate the maximum acceptable cost for the file search or purchase. For method A, the cost per query ranged from $1 to $500, with a median of $10, and for method B the cost per query ranged from $1 to $100 with a median of $10. Only 29 respondents attempted to estimate the acceptable cost for purchase of the file (method C); the values given ranged from 'free' (since funded through government sources) to

$3000, with a median of $500.

In question VI-2 the respondents were asked to comment on the relative merits of the above approaches (A, B, C) with respect to their needs. Again, the on-line interactive search system (method A) was preferred by 52 respondents (48% of those commenting); method B was preferred by 20 (19%), and method C was preferred by 18 respondents (17%). A manual search through the book was felt to be adequate by 11 respondents, 4 respondents commented that computer searches would be too costly, and 3 respondents remarked that the preferred method would depend on the frequency of use and local computer facilities. The advantages and disadvantages of the three methods given in the comments are summarized in Table 21.

Table 21. Relative Merits of Computer Search Methods (VI-2)

Method	Advantages	Disadvantages
A	Best and cheapest for intermittent use; easiest to use; many users are already using CIS	Needs support to continue; problems with proprietary information
B	Best when individual use is low; allows guidance for new users; do not need computer access	May be too expensive; no immediate answer; problems with proprietary information
C	Best for frequent use or large group of users; necessary for proprietary information	Too expensive to implement and maintain; need staff member to oversee project

It can be seen from Table 21 that the advantages of the three methods depend largely on the frequency of use. The batch search (method B) would be preferred for low use, the on-line search (method A) would be preferred for occasional or intermittent use, and the in-house data base (method C) would be best for frequent use or for a large group of users. The public nature of the file access procedures for methods A and B might be a problem for queries concerning proprietary information.

2.6.2 Research Using a Computer Data File

Finally, the last question (VI-3) asked, "Assume that the Crystal Data file, software, and computer time are all available at a centrally supported site. If funding were available to support you for one month to one year, would you consider doing research taking advantage of the data in the file?" Nearly one-half of the 176 respondents to this question (85, 48%) answered "yes". This represents a potentially large and unexplored research resource. As one respondent commented, "this is an essential ingredient in promoting use of the file."

3.0 Summary of Results and Conclusions

The questionnaire on Crystal Data was distributed in December of 1978 and January of 1979 by the NBS Associateship of the JCPDS. A total of 235 responses were received from representatives of industry (37%), universities and colleges (43%), and government (16%). Most respondents characterized their work as basic and/or applied research, and used both powder diffraction and x-ray single-crystal diffraction in their work.

The overall frequency of use indicated for Crystal Data Determinative Tables was high, with 81% of the respondents reporting occasional or frequent use in more than one category. The major area of application was in crystal structure analysis (143 responses), with significant use in phase equilibria (53), materials processing (42), high or low temperature studies (40), corrosion (33), and catalysis (29). The types of materials most frequently checked were ceramics (51) and semiconductors (29), although magnetic materials, optical

materials, and energy conversion materials were also important.

Specific examples of the use of Crystal Data could be grouped into the following areas of application: materials analysis and qualitative identification; aid in research applications; to assign structure type or search for isostructural materials; for literature search or location of cell parameters; to find out if a structure has been determined; as an aid in teaching and design of experiments. Examples of use given in the area of materials analysis included identification of unknown compounds in a phosphoric acid plant, refractories, and human tissue. Research applications mainly involved use of the structural relationships found in Crystal Data for the design of materials with given properties, and extended to such diverse areas as theoretical calculations, bonding electron distribution studies, interpretation of radial distribution functions and extended x-ray absorption fine structure, correlation of polytypes with luminescence, and calculation of theoretical powder patterns. Several examples were also given in which a crystal structure could be assigned through the location of isostructural materials.

The usage of Crystal Data directly for the identification of unknowns is lower than the use of the powder method; the powder method was preferred primarily because it is easier, faster, and more routine, or because equipment necessary for the single-crystal method is not available in the respondent's laboratory. This indicates (1) that the development of a rapid and inexpensive method for unit-cell determination would be desirable, and (2) that there is a need for education of the scientific community, as the single-crystal method of identification is in many cases more powerful than the powder method owing to the uniqueness of the unit cell and the larger size of the Crystal Data file (~60,000 entries vs. ~32,000 in the Powder Diffraction File).

Crystal Data Space-Group Tables or Nowacki's Systematic Tables were used by roughly one-third the number of users of Crystal Data Determinative Tables. Examples of use in the area of materials design included location of materials in a given space group to study properties related to that space group, and location of isomorphous substances so as to make mixed crystals with desired properties. Research applications included group-subgroup relationships, correlation between chemical class and space group, theoretical relations of symmetry to structure, and use in single-crystal ESR and NMR work.

The value of the inclusion of various supplementary data items was rated by the respondents, and these ratings will be considered for future data input. Also, several data items previously abstracted, such as goniometric axial ratios and compressibility information, were rated as relatively unimportant, and these may be deleted.

The most frequent suggestion for improvement was that the coverage should be more complete, or annual updates be published between editions; the necessity for complete coverage was further indicated by the fact that over one-fourth of the respondents reported that they had needlessly duplicated crystal structure determinations at a median cost of $2000. It was also suggested that entries be consolidated; most respondents felt that duplicate references to the same material should be given only as a reference with an indication of the type of information to be found, and indicated that it would be acceptable to publish annotated references of this type as a supplement to Crystal Data. The development of the computer-based data file currently being implemented should make it possible to overcome these deficiencies, particularly if a suitable data base management system is included. Adequate computer typesetting capability is also required.

The respondents also indicated a willingness to help with the Crystal Data project. Most said that they would be willing to send in data directly prior to publication, and 51 respondents indicated that they would be willing to help with abstracting; this represents a valuable resource for future Crystal Data publications and indicates the interest in and value of this compilation. The expansion of funding for abstracters is thus desirable; this would help to alleviate the problem of out-of-date coverage.

Potential published products rated as of moderate to high value by the respondents were the following:

(1) A supplement to the inorganic section of Crystal Data covering the literature

for 1970-1978. The publication of this supplement was given as most important, again indicating the need for complete coverage.

(2) The Crystallochemical Identification Tables with key crystallographic and chemical information for all compounds in the Determinative Tables. This new product is ready for typesetting, and should be made available soon for identification of unknowns both as a printed version and a computer file for automated search.

(3) A cumulative name and formula index. The feasibility of such a publication is currently being assessed.

(4) Crystal Data for refined quantitative inorganic structures (1970-1978). This product would help eliminate duplication of crystal structure determinations. Other subfiles (minerals, metals and intermetallics, oxides) were rated as of high value to smaller groups of users, as would be expected.

The preferred method of computer-based dissemination of the Crystal Data file was an on-line interactive search technique, particularly for moderate or intermittent use. A subfile of Crystal Data is currently being tested for distribution on the Chemical Information System (CIS) network. A batch search at a central location was felt to be desirable for low usage or those without computer access, and an in-house data file and search capability was preferred for frequent use or for a large group of users. All three methods of dissemination appear to be valuable to various groups of users; also, great interest was indicated in the possibility of doing research taking advantage of a computer-based Crystal Data file. It is proposed that the file be made available at NBS for on-line search, for batch search with NBS acting as a regional data center, and for research using the file.

APPENDIX A

QUESTIONNAIRE ON CRYSTAL DATA

[The number of responses has been added for each item.]

I. Description of Respondent

1. Your present type of work [check as many as appropriate]:

168	Basic research	19	Quality control
104	Applied research	62	Analysis
29	Product development	78	Education
13	Production	1	Student
7	Other (please specify) _____		

2. Employer:

86	Industrial	102	University or college
37	Government	11	Research institute
5	Other (please specify) _____		

3. Please check one or more of the following Standard Industrial Classifications which best describe your employer:

5	10 Metal mining
2	11 Anthracite mining
7	12 Bituminous coal and lignite mining
5	13 Oil and gas extraction
8	14 Mining of nonmetallic minerals, except fuels
51	28 Chemicals and allied products
6	29 Petroleum refining
16	32 Stone, clay, glass, and concrete products
8	33 Primary metal industries
13	34 Fabricated metal products
4	35 Machinery, except electrical
18	36 Electrical and electronic equipment
5	38 Instruments and related products
12	80 Health services
1	81 Legal services
97	82 Educational services

3 □ 84 Museums, art galleries, botanical and zoological gardens

3 □ 91 Executive, legislative, and general government

1 □ 92 Justice, public order, and safety

6 □ 95 Environmental quality and housing

12 □ 97 National security and international affairs

43 □ Other [please describe] _____

4. In your work do you use [check as many as appropriate]:

189 □ X-ray powder diffraction 167 □ X-ray single-crystal diffraction

21 □ Neutron diffraction 44 □ Electron diffraction

5. Please check one or more of the types of materials routinely involved in your work:

177 □ Inorganics 73 □ Ceramics

67 □ Metals and intermetallics 64 □ Small molecules

118 □ Minerals 21 □ Large molecules or proteins

51 □ Catalysts 13 □ Pesticides and herbicides

81 □ Organics 29 □ Drugs

59 □ Organometallics 14 □ Other (please specify) _____

6. Name and address of present employer [optional]:

177 _____

_____

_____

7. Your name, address, and telephone number [optional]:

169 _____

_____

_____

II. Use of Crystal Data

1. How often do you use Crystal Data Determinative Tables in the following ways [use 3 = frequently, 2 = occasionally, 1 = rarely, 0 = not at all]:

3 / 2
23/54 To identify an unknown substance using the determinative ratio

58/77 To find out if the structure of a particular substance has been determined

35/81 To find information about structure types or to locate isostructural materials

62/81 To find cell dimensions or space group of a particular substance

9/48 To obtain information about physical properties (e.g. color, habit, mp) of a substance

30/74 To locate literature references containing crystal data on a particular substance or structural group (e.g., phosphates, cyanates, etc.)

17 Other (please specify) _____

2. Do you use Crystal Data in one or more of the following areas [check as many as apply]:

143 ☐ crystal structure analysis	29 ☐ catalysis
42 ☐ materials processing	23 ☐ magnetic materials
33 ☐ corrosion	20 ☐ energy conversion materials
16 ☐ diffusion in metals	29 ☐ semiconductor materials
53 ☐ phase equilibria	51 ☐ ceramic materials
11 ☐ failure prevention	23 ☐ optical materials
17 ☐ fracture or wear studies	17 ☐ high pressure studies
10 ☐ materials durability	40 ☐ high or low temperature studies

3. Have you ever solved a structure or collected data and then found that your work was unnecessary because the same or similar substance had already been solved?

159 ☐ No

64 ☐ Yes. If yes, how often has this happened? What is your dollar estimate of the cost involved (computer time, lost hours, etc.) for each occurrence?

59 _____

_____

4. Have you ever determined cell parameters for a compound or group of compounds and then discovered that your work was a duplication of previously published data?

158 ☐ No

63 ☐ Yes. If yes, how often has this happened? What is your dollar estimate of the cost involved for each occurrence?

55 _____

_____

5. Please describe one or more specific applications of Crystal Data Determinative Tables, showing the type of information needed and how it was used.

106 _____

_____

_____

_____

_____

6. Do you need to identify unknown crystalline substances in your work?

30 □ No

200 □ Yes. If yes, do you rely mainly on the powder method (ASTM-JCPDS file) or the single-crystal method [check one]:

60 □ Use powder method exclusively

105 □ Use mostly powder method but also use Crystal Data for confirmation or for literature references

46 □ Use primarily single-crystal method

7. Do you grind up single crystals in order to use the powder method of identification?

132 □ No

87 □ Yes. If yes, why do you <u>not</u> use the single-crystal method [check as many as apply]:

6 □ Too difficult to calculate a unit cell

25 □ Do not have the necessary equipment

6 □ The equipment is too expensive

14 □ A trained technician is required

28 □ Too difficult to obtain a good single crystal

4 □ Too complicated to transform the unit cell to the Crystal Data cell to get the determinative ratio

9 □ Crystal Data is not up-to-date

19 □ Tradition

57 □ Other (specify) _____

8. Do you use the Crystal Data Space-Group Tables or Nowacki's Systematic Tables?

150 □ No

66 □ Yes. If yes, briefly describe one or more applications to your work

38 _____

_____

_____

9. In what ways could Crystal Data Determinative Tables be improved to serve you? Please give us your comments.

81 _____

_____

_____

10. If a paper submitted for publication contains crystal data, do you think the data should be evaluated (e.g. checked for inconsistencies) prior to publication as part of the journal editorial review process?

184 □ Yes 39 □ No

11. Would you be willing to assist the Crystal Data project:

By sending us your crystal data directly prior to publication?

137 □ Yes 65 □ No

By abstracting a class of compounds from the published literature or by abstracting crystal data for all compounds from a given journal?

51 □ Yes 141 □ No

III. Supplementary Content of Crystal Data Entries

1. Indicate the importance to you of the following types of information that might be found in a given Crystal Data entry [use 3 = high value, 2 = moderate, 1 = marginal, 0 = no value]:

3 / 2	
135/50	Source of cell data (single-crystal or powder work)
53/74	Specific method of obtaining cell data (e.g., "refined on 30 reflections from single-crystal diffractometer data").
86/75	An indication that recorded powder data may be found in the literature reference
85/81	An indication that indexed powder data may be found in the literature reference
85/61	Cross-reference to the JCPDS powder data file
13/36	Chemical registry number
43/63	Refined R-value (for quantitative structures)

2. In order to speed up processing and obtain an up-to-date file as soon as possible, we are considering the simplification or elimination of certain data items. Please indicate if it is useful to have information in the commentary on the following [use 3 = high value, 2 = moderate, 1 = marginal, 0 = no value]:

3 / 2		3 / 2	
32/47	Goniometric axial ratios	58/72	Color
50/67	Indication of neutron or electron diffraction data	61/49	Refractive indices
66/80	Existence of sub-, super-, or pseudo-cells	21/40	Pleochroism
31/91	Habit	59/67	Melting point
29/69	Cleavage planes	43/62	Solid solution studies
44/77	Twinning	24/51	Thermal expansion studies
69/68	Temperature of data taking	12/36	Compressibility studies
36/29	Locality for minerals	46/59	Source of polymorphic forms

IV. Literature Coverage

1. Many publications containing unit-cell dimensions do not give new cell or structural data, but may provide additional crystallographic information such as an indexed powder pattern, solid solution data, thermal expansion data, or cell data for a mineral of slightly variant composition.

References of this type should be treated as follows [check one]:

43 □ Reported in full, including unit-cell data

145 □ Given only as a reference with an indication of the type of information to be found in the paper

15 □ Given only as a reference

6 □ Omitted from Crystal Data

2 □ Other (please specify) _____

2. To facilitate data handling, annotated references of the above type could be published as a supplement to Crystal Data, with appropriate indexes.

This format would be 145 acceptable 41 not desirable

V. NBS Crystal Data Center Published Products

For each of the following published products, please indicate how important the given product would be in your work [use 3 = high value, 2 = moderate, 1 = marginal, 0 = no value]:

1. Crystal Data Determinative Tables [Indicate importance 3, 2, 1, 0]:

3 / 2
114/48 A supplement to the inorganic section of Crystal Data covering the literature for 1970-1978 (∿20,000 entries)

65/67 A cumulative name and formula index for the 3rd Edition of Crystal Data for Volumes 2 (inorganic literature through 1966) and 4 (inorganic literature 1967-1969).

2. Crystal Data for special categories (these may be brought up to date more rapidly) [Indicate importance 3, 2, 1, 0]:

3 / 2
68/33 Crystal Data for minerals only (1970-1978)

35/27 Crystal Data for metals and intermetallics only (1970-1978)

76/47 Crystal Data for refined inorganic quantitative structure only (1970-1978)

40/44 Crystal Data for oxides only (1970-1978)

40 Crystal Data for other classes (please specify) _____

_____

3. Crystallochemical Identification Tables [Indicate importance 3, 2, 1, 0]:

3 / 2
79/58 This is a new product designed for the rapid identification of unknown materials. For a given compound, an entry would consist of one line of key

crystallographic and chemical data (reduced cell, space group, formula, density, and literature reference). Data from all volumes of Crystal Data would be included.

4. Crystal Data Space-Group Tables [Indicate importance 3, 2, 1, 0]:

3 / 2
22/56 A second issue of Crystal Data Space-Group Tables including data coincident with Volume 3 (organic) and Volume 4 (inorganic) of the Crystal Data Determinative Tables. The first issue (J. Phys. Chem. Reference Data, 6, 675, 1977) is coincident with Volumes 1 and 2 of the 3rd Edition of Crystal Data.

VI. Crystal Data File Searches and Analyses

1. The large size of the Crystal Data file makes computer searches a valuable asset. For the following methods, please indicate how important such a search ability would be in your work [use 3 = high value, 2 = moderate, 1 = marginal, 0 = no value]. If possible, also indicate the maxmium acceptable cost.

A. On-line Interactive Search System

3 / 2
64/60 The Crystal Data file will soon be available to the public as a component of CIS (Chemical Information System of NIH/EPA). This system is an on-line interactive search system; the user queries the file via a terminal and receives an answer in a very short time. For example, one could find all compounds with a given cell, space group, or formula within a few minutes.

$ 10 Maximum cost per query (median of 82 responses)

B. Batch Search System at Central Location

3 / 2
21/44 Such a system would be operated at a central location with a resident crystallographic expert to interface between the user's query and the batch-operated computer system. Questions would be received and answered via mail or telephone. Several days might be required for the process.

$ 10 Maximum cost per query (median of 47 responses)

C. User's Organization to Buy Computer Data Base

3 / 2
25/26 In this system the organization would pay for the data base, appropriate software, and yearly updates. The organization and the users would then decide the most appropriate way to use the file.

$500 Maximum cost for file (median of 29 responses)

2. Please comment on the relative merits of the above approaches (A, B, C) with respect to your needs 52A 20B 18C.

3. Assume that the Crystal Data file, software, and computer time are all available at a centrally supported site. If funding were available to support you for one month to one year, would you consider doing research taking advantage of the data in the file?

85 ☐ Yes 91 ☐ No

APPENDIX B

SPECIFIC AREAS OF APPLICATION OF CRYSTAL DATA DETERMINATIVE TABLES (II-5)

INDUSTRIAL

1. A search for a number of isotropic substances was desired. Searching in the cubic section was most useful, yielding many compounds which fit our particular needs.

2. Can often index a powder pattern as cubic quickly with a slide rule (B/D scale method with inverted slide) then check in Crystal Data for possible or isomorphous compounds with the same or similar unit cell.

3. A small (approximately 1000Å) crystallite was isolated in a piece of human tissue. The crystal provided an electron diffraction pattern. Only elemental information and two-dimensional diffraction data [were available]. The crystallite was identified with the aid of Crystal Data.

4. We use our x-ray diffraction mainly to identify reaction compounds which form in refractories while they are in use. The data is used to suggest refractories which might provide better service in such applications, or it may aid in the development of improvements in the refractory products.

5. In attempting to prepare analog structures use Crystal Data as a source as to what has been reported. Example: eulytite, $Bi_4Si_3O_{12}$ - check how many A and B site substitutions are known. Same use for other mineral analogs.

6. Searched for all compounds crystallizing with a given a and b dimension (indeterminate) and a given angle between a and b for use in layer stacking studies.

7. We have been interested in certain cubic, hexagonal, and orthorhombic space groups and have used the Crystal Data Determinative Tables as a source of materials crystallizing in the desired space groups.

8. Searching for isostructural materials to aid in identification. The structure type is recognized, lattice parameter(s) determined, and lattice constant or ratio used to search for compound possibility - usually elemental data is also available to aid in the search.

9. Crystal Data is used exclusively for identification of multiphase samples. The Tables are used to determine semi-quantitative mineralogical analyses by process of elimination of diffraction peaks published by JCPDS.

10. If the electron diffraction data and the card file match disagree, the Determinative Tables usually give us the isomorph.

11. 1. Identification of fillers in polymer products.
    2. Determination of crystallinity and orientation in polymers.
    3. Identification of corrosion products on plated copper conductor.
    4. Determination of d-spacing in carbon blacks.

12. Look for oxides having spinel structure and having lattice parameter within a certain range. These spinels would be possible substrates for epitaxial growth of other compounds.

13. I have had several problems involving an unknown substance mixed with kaolin. I have solved these problems by the Determinative Tables.

14. Identifying inorganic components in a polymer composite.

15. [Used for identification] without the aid of chemical data when d-spacings are close together.

16. We use both Crystal Data and Pearson's handbook of metals and alloys as a backup reference source to our PDF file. These books cut down the volume of literature searching which might otherwise have been done for research projects. We usually do not use them for identifying unknowns.

17. Identification of corrosion products.

18. To identify structurally isomorphous materials of a particular structure type.

19. In addition to the uses in II-1, have indexed an unknown powder pattern with known chemistry and looked up the axial ratio to see if the structure was known or discovered.

20. 1. Axial ratio was used to find alloy analog of $(Tl_{0.75}Pb_{0.75})_4Cl_5$ viz Zr_5Si_4 prior to determining structure.
    2. Everyday use is for getting references and unit cell data.

21. a. Identification of elements by x-ray diffraction identification of ashed residues.
    b. Analysis of commercial products.
    c. Fingerprinting of compounds.

22. Calculation of a theoretical [powder] pattern for a simple compound (cubic, orthorhombic, tetragonal, or hexagonal) not found in the JCPDS-ASTM file. Information needed: unit cell parameters, angles, and space group.

23. Practically all my work involves comparisons of lattice constants among isostructural compounds, such as the spinel structure, the KH_2PO_4 structure, etc. Therefore, the important thing is the completeness of coverage of different compounds within the structure type, as is evaluation of the data (i.e. inclusion of poor data where better data are available is not useful).

24. 1. Once obtaining lattice and space group of an unknown, (a) search to make sure structure not already solved; (b) see if there are analogous structures.
    2. To generate powder diagrams via computer using crystallographic data.

25. To find any similar compounds or related structures during new mineral description.

26. In some cases cell constants and space groups are given for a material cited in the PDF which are not indexed on the PDF card. This saves a lot of trouble trying to index that material. Often, cell data from a number of investigations are cited for a single substance. One doesn't always get these cross references from the PDF file.

27. My most frequent use of the Tables is to find whether the structure of particular substances has been done. In this context, the formula index is the first most useful part of the book.

28. Identification of different minerals in ore or separation of caustive [sic] agents in unit operations.

29. When a sample is identified as having elements that could not be present, Crystal Data is used to find isostructural compound that could be possible.

30. Identification of crystals based on ratios of a to b to c (unit cell dimensions).

31. Identification of phases.

32. Identify unknown powder patterns.

33. Generally used to check out identification of unknown single crystals.

34. Mainly used for identification of compounds not in the JCPDS file.

35. Data on unit cells of $KMnO_4$ and $KClO_4$ was consulted to develop a teaching experiment on solid solutions of these two salts.

36. Adduct - cell dimensions. Other proprietary [compounds] also [identified by] unit cell dimensions.

37. Identifying simple crystals present in a mixture.

38. Having a unit cell, I would use the Tables to see if the structure could be easily guessed by comparing it to known compounds. Used many times for a quick reference check.

39. Checking for unknown. Checking for oxygen states.

40. By use of unit cell ratio or cell size, have located a material for identification of an unknown.

41. To determine that a crystalline precipitate was CrO_3 from the axial ratios.

42. Mostly powder work. Clarification of some powder data or if powder data is not available. For references. To help in solid solution evaluation.

UNIVERSITY or COLLEGE

1. Local phosphoric acid plant was having trouble with an unknown compound precipitating in pumps and pipes. Was able to identify compound and advise on how to manage settling ponds to avoid problem.

2. We made a high pressure run in an attempt to prepare a specific new compound. An x-ray powder photograph of the result showed that a reaction had occurred and that none of the original material was present. The starting elements were Nb and Te. The powder photograph showed a cubic material with a = 4.38 to 4.40Å. A quick look through Crystal Data revealed that the "new" material was almost certainly NbN (with N from BN in reaction chamber).

3. We use crystallographic data to help interpret radial distribution functions and extended x-ray absorption fine structure data from amorphous materials. Our work is aimed at determining atomic arrangements in amorphous materials.

4. Enclosed are reprints of two papers in which references to Crystal Data play an important part. The paper on formates shows some of the shortcomings of Crystal Data [inconsistent data on rare-earth formates in Crystal Data was the impetus for further structural work], and the paper on the F51 type shows how much information can be gleaned from Crystal Data [various structure types reported were explained on the basis of information given in Crystal Data]. Much more work of this sort could be based on Crystal Data.

5. 1. Educational purpose: to find a compound suitable for basic training of students in course work.
   2. To find possible isostructural or related compounds pertaining to current research.
   3. Many of my colleagues use the Tables, which I keep accessible to them in my office.
   4. There is such a large variety of occasions where the Tables come in handy, I cannot list them all.

6. Usually used as a screen before starting a structure determination on a given material.

7. A new mineral, maricite, $NaFePO_4$, had its structure determined by careful checking against axial ratios and cell dimensions of similar compounds.

8. To determine if a cubic organomercury compound had been reported. Found that cell edge and space group were identical to that of $Ba(NO_3)_2$. Powder patterns proved to be practically identical. These data were used to determine the most probable arrangement of the ligands in the unknown.

9. 1. Use of ratio to identify unknown in standard way.
   2. Use of ratio and chemical formulas in Tables to look for potentially isomorphous materials as a guide to structure of an unknown.

10. [For work on] bonding electron distribution, need fairly simple structures having no peculiarities (disorder etc.) and only moderate symmetry (monoclinic, orthorhombic). Search Crystal Data for likely candidates.

11. My books are frequently borrowed by the theoretical chemists who use the distance and angle determinations.

12. A "new" mineral was very rare and only the axial ratios were obtained (besides color and habit). No composition was known. The one crystal measured was tiny! Crystal Data identified it for me.

13. We routinely examine the Tables every time we analyze a crystal to see if isostructural (or identical) compounds are known. The Tables serve as an analytical tool.

14. Comparisons of forms of Se. Searched for information on chalcogenide halides, metal complexes of organic acids (and the organic acids).

15. We have often determined cell constants and space groups of complexes which we have synthesized and used Crystal Data as an analytical device, i.e., if we suspect that we know what the complex might be the cell constants are very useful. The information needed is simply space group, cell constants, and density.

16. 1. A terpene, trigonal, $P3_121$ - impossible space group for the compound. Found a compound with roughly the same axial ratio in Crystal Data, which was $P3_1$ twinned-solved the problem (but not the structure!).
    2. Mineral names [used to get] formulas (must be geological tables which do this more efficiently, but Crystal Data is handy on my office bookshelf).

17. Description of unit cell transformation for the reduced cell in relation to the original literature work is very often helpful, particularly in the triclinic system in relation to handling of single crystal diffraction films.

18. Mainly used to look up space group and cell dimensions to see if a crystal structure has been done.

19. Checking variation of cell dimensions and symmetry with composition. Checking unknowns.

20. Checking new structure of unknown substance to see if ratios were similar to known listed one.

21. Used to find cell constants for all nitrates studied.

22. 1. Easiest access to structure literature.
    2. Used in instruction for determinative mineralogy (mostly for cubic).
    3. Use Nowacki tables for lectures on relation of symmetry to structure.

23. Currently attempting to prepare unusual metal complexes containing I, TCNQ, or TCNE. A check of unit cell dimensions on crystals obtained provides information on whether we have indeed prepared something new.

24. A reference book for problem solving in x-ray crystallography class.

25. Used in surveying references of compounds similar to one whose structure determination we are carrying out.

26. Mostly to find unit cell and space group of compounds similar to those being studied by single crystal methods in our own lab. Also for identification of unknowns (powder data) in teaching instrumental analysis.

27. See II-1, items 2, 3, and 4.

28. To find out if a structure was known - most recently that of alanine. Crystal Data, although not up to date of course, is a quick and easy reference work to use for this purpose.

29. I have only used the Tables to see if our new crystals of human hemoglobin might be isomorphous with hemoglobin crystals from other species.

30. In experimental mineralogy, to determine what compounds have been described previously.

31. Axial ratios of knowns to suggest structure type of unknown.

32. Comparison of Crystal Data values with own experimental values (material analysis).

33. In teaching crystallography.

34. How many crystal forms of L-tartaric acid are known? From formula index to Tables for unit cell data.

35. Identification. Determination of known structures. Literature search.

36. Setting up a powder diffraction experiment for undergraduate lab, I searched for cubic F cells with similar cell dimensions.

37. Occasionally we identify very small mineral samples by single crystal x-ray oscillation photos. Also a source book.

38. Needed cell parameters to check against my determined values.

39. Used primarily as a way to identify unknowns (minerals). Quick reference for properties.

40. There must be a computer readable data base of cell parameters, volume, etc. for instant access. This has been needed for years.

41. See II-1 on page 2.

42. As already indicated in II-1.

43. Only seek molecular structure information.

44. To identify simple crystals present in a mixture.

GOVERNMENT and NATIONAL LABORATORIES

1. 1. It was suspected that hexagonal $(NH_4)_2SiF_6$ (bararite) was present in a sample, but no pattern was listed in the JCPDS Powder Diffraction File. Unit cell data from Crystal Data were used to calculate a pattern.
   2. Unit cell data for minerals are sometimes calculated from powder diffraction data, then compared with values for synthetic materials from Crystal Data to estimate substitutions. Useful for work with solid solution series.

2. Identification of new superconducting materials - binary and ternary intermetallics.

3. X-ray data for polycrystalline SiC doped with boron were correlated with the luminescence (different polytypes have different emission spectra).

4. We have used Crystal Data to search the literature for information on cell constants for KBr, which we have adopted as a wavelength calibration standard for our neutron beams of the High Flux Beam Reactor. Also to locate data on metals, for comparison with M-M bond lengths obtained in work on metal cluster compounds. Generally use inorganic index most frequently.

5. Search of isomorphous compounds of explosive or oxidizer nature so as to make mixed crystals of same.

6. $Eu_{11}As_{10}$ was found to be orthorhombic, $a \approx b$; traced to related tetragonal $Sm_{11}Ge_{10}$. Eu_5As_4, orthorhombic, was traced to related Sm_5Ge_4 which is not isostructural.

7. 1. Quick location of cell constants and space group.
   2. Quick check of potential structure types for a specific compound.
   3. Check if there are several crystalline forms (types) of a particular compound.
   4. In one case, identification of unknown compound made in small quantities when preparing another compound.

8. Salt crystals [were obtained] in a protein crystallization; cell constants determined only for identification.

9. Crystal Data tables are used to search for a structural and/or chemical analog of a particular new mineral which is too fine grained for single crystal analysis.

10. Lattice constants to provide clues for identification. Chemical formulas to identify if material has been worked on.

11. Using the JCPDS file, a material was identified as a compound it could not possibly be. The cell constants for that material were used [to calculate] the determinative ratios and Crystal Data consulted for compounds around that ratio. The correct material was found easily. The pattern for this material did not exist in the JCPDS file.

12. See enclosed reprint (copy is enclosed, with the paragraph of interest circled). [The crystal structure of a mineral was solved by finding a known structure with similar axial ratios, and using this structure as a trial structure for the unknown structure.]

13. Data on zeolites and related alkali and alkaline earth alumino-silicate hydrates - also SiO_2 and silica hydrates - sundry mineral group relations. Synthetic inorganic mineral analogs.

14. Identify powder patterns not in the JCPDS file or find other compounds that might yield a similar pattern. Cell dimensions, space group, name, chemical formula.

15. By using Crystal Data as described under II-1 (above) I have avoided problems described under II-3 and II-4 (above).

16. Whether the material on hand has been characterized crystallographically and in particular to look for the original paper through references given.

17. Tend to use the Tables mainly to find out previously determined information.

18. 1. To identify certain materials with measured cell parameters.
    2. To check similar type structures.

OTHER

1. Can't recall a specific example, but I often have an unknown crystal for which I have chemical information (elemental analysis by electron microprobe) and limited optical crystallographic data, e.g., whether substance is anisotropic and if so, whether uniaxial or biaxial, plus some idea of refractive index. Using Crystal Data as a source of information I may be able to pick out likely possibilities and be thus aided in choosing the next step towards identifying the unknown.

2. 1. Find possibly similar structures to one being determined.
   2. Find if structural or symmetry information on a particular compound is available.
   3. Search for information on modes of twinning, either listed in Crystal Data or in literature references cited.

APPENDIX C

APPLICATIONS OF CRYSTAL DATA SPACE-GROUP TABLES AND NOWACKI'S SYSTEMATIC TABLES (II-8)

INDUSTRIAL

1. Compounds with certain space groups were predicted to have properties which we wanted to study. The Space-Group Tables provided a starting point: determining what compounds had those space groups so that substituted compounds might also be studied.

2. To establish that phases $Sr_7Sn(PO_4)_6$, $Sr_7Zn(PO_4)_6$, etc. were isostructural with $Bi_4(SiO_4)_3$.

3. Searching for compounds with similar structures.

4. a) When unit cell parameters and space group are necessary for new compounds.
   b) Identification of possible isomorphs.

5. To simplify identification of space group for technician.

6. For developed alloys, to determine if alloy is what was expected.

7. Identifying crystal variants of VO_2.

8. For locating isostructural compounds.

9. To assist in space group determinations.

10. Occasionally.

UNIVERSITY or COLLEGE

1. Interested in possible correlation between chemical class and space group.

2. Group-subgroup relationships. Also for quick check of our early results.

3. Theoretical relations of symmetry to structure.

4. To determine space group of an organometallic molecule, prior to complete single-crystal structural analysis.

5. Use in single-crystal ESR, NMR work.

6. Frequency analysis.

7. Crystal structure determination.

8. Whenever I determine the space group of an unknown crystal. I use Donnay tables in Crystal Data.

9. Occasionally to check or verify or establish a space group.

10. Mainly to provide illustration examples and data for lectures.

11. See my reviews of the 1st and 2nd editions for comments on uses, and examples in reprints. [See response 4 (University or College) in Appendix B.]

12. To find similar material for comparison.

13. Very occasionally I have needed names of other compounds in the same space group to help out a colleague in physics.

14. Mineral identification.

15. To find illustrations of different crystal structures for teaching.

16. Checking unknowns.

17. Statistical use.

18. Seldom.

19. Both are on the shelf adjacent to the Determinative Tables but rarely get used. The Space Group Tables get a little use. Nowacki's table just doesn't seem conducive to use.

20. Rarely and only for look-up purposes.

21. Only for interest.

GOVERNMENT and NATIONAL LABORATORIES

1. A very useful means of quickly finding related minerals (and inorganic compounds).

2. Search for isomorphous compounds of explosive or oxidizer nature so as to make mixed crystals of same.

3. To look for likely isostructural materials and/or trial structures.

4. Space group determination of new minerals.

5. Nowacki is interesting but has not proven very useful.

OTHER

1. Theoretical study of crystal systematics.

2. Sometimes.

NBS-114A (REV. 9-78)

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET	1. PUBLICATION OR REPORT NO. NBS TN 1112	2. Gov't. Accession No.	3. Recipient's Accession No.

4. TITLE AND SUBTITLE

USER EVALUATION OF CRYSTAL DATA PRODUCTS AND SERVICES: QUESTIONNAIRE ANALYSIS AND IMPACT

5. Publication Date June 1980

6. Performing Organization Code

7. AUTHOR(S)

J. K. Stalick, A. D. Mighell, and R. J. Boreni

8. Performing Organ. Report No.

9. PERFORMING ORGANIZATION NAME AND ADDRESS

NATIONAL BUREAU OF STANDARDS
DEPARTMENT OF COMMERCE
WASHINGTON, DC 20234

10. Project/Task/Work Unit No.

11. Contract/Grant No.

12. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS *(Street, City, State, ZIP)*

Same as item 9.

13. Type of Report & Period Covered Final

14. Sponsoring Agency Code

15. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

16. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)*

A survey was made of the needs of the users of Crystal Data Determinative Tables and related products and services of the NBS Crystal Data Center. The results indicate a high frequency of use of Crystal Data, with particular application to materials analysis and design. The survey suggests a need for the development of a rapid and inexpensive method of unit-cell determination as well as education of the scientific community in the use of the single-crystal method for identification of unknown materials. More complete coverage and consolidation of entries are also suggested. The necessity for computer-based methods of data base dissemination is indicated, with particular importance for research applications.

17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)*

Computer dissemination; crystal data; identification; materials analysis; materials design; NBS crystal data center; user evaluation;

18. AVAILABILITY ☒ Unlimited

☐ For Official Distribution. Do Not Release to NTIS

☒ Order From Sup. of Doc., U.S. Government Printing Office, Washington, DC 20402

☐ Order From National Technical Information Service (NTIS), Springfield, VA. 22161

19. SECURITY CLASS (THIS REPORT)	21. NO. OF PRINTED PAGES
UNCLASSIFIED	42
20. SECURITY CLASS (THIS PAGE)	**22. Price**
UNCLASSIFIED	$2.25

USCOMM-DC

☆ U.S. GOVERNMENT PRINTING OFICE :1980 O-311-046 (122)

NBS TECHNICAL PUBLICATIONS

PERIODICALS

JOURNAL OF RESEARCH—The Journal of Research of the National Bureau of Standards reports NBS research and development in those disciplines of the physical and engineering sciences in which the Bureau is active. These include physics, chemistry, engineering, mathematics, and computer sciences. Papers cover a broad range of subjects, with major emphasis on measurement methodology and the basic technology underlying standardization. Also included from time to time are survey articles on topics closely related to the Bureau's technical and scientific programs. As a special service to subscribers each issue contains complete citations to all recent Bureau publications in both NBS and non-NBS media. Issued six times a year. Annual subscription: domestic $17; foreign $21.25. Single copy, $3 domestic; $3.75 foreign.

NOTE: The Journal was formerly published in two sections: Section A "Physics and Chemistry" and Section B "Mathematical Sciences."

DIMENSIONS/NBS—This monthly magazine is published to inform scientists, engineers, business and industry leaders, teachers, students, and consumers of the latest advances in science and technology, with primary emphasis on work at NBS. The magazine highlights and reviews such issues as energy research, fire protection, building technology, metric conversion, pollution abatement, health and safety, and consumer product performance. In addition, it reports the results of Bureau programs in measurement standards and techniques, properties of matter and materials, engineering standards and services, instrumentation, and automatic data processing. Annual subscription: domestic $11; foreign $13.75.

NONPERIODICALS

Monographs—Major contributions to the technical literature on various subjects related to the Bureau's scientific and technical activities.

Handbooks—Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

Special Publications—Include proceedings of conferences sponsored by NBS, NBS annual reports, and other special publications appropriate to this grouping such as wall charts, pocket cards, and bibliographies.

Applied Mathematics Series—Mathematical tables, manuals, and studies of special interest to physicists, engineers, chemists, biologists, mathematicians, computer programmers, and others engaged in scientific and technical work.

National Standard Reference Data Series—Provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated. Developed under a worldwide program coordinated by NBS under the authority of the National Standard Data Act (Public Law 90-396).

NOTE: The principal publication outlet for the foregoing data is the Journal of Physical and Chemical Reference Data (JPCRD) published quarterly for NBS by the American Chemical Society (ACS) and the American Institute of Physics (AIP). Subscriptions, reprints, and supplements available from ACS, 1155 Sixteenth St., NW, Washington, DC 20056.

Building Science Series—Disseminates technical information developed at the Bureau on building materials, components, systems, and whole structures. The series presents research results, test methods, and performance criteria related to the structural and environmental functions and the durability and safety characteristics of building elements and systems.

Technical Notes—Studies or reports which are complete in themselves but restrictive in their treatment of a subject. Analogous to monographs but not so comprehensive in scope or definitive in treatment of the subject area. Often serve as a vehicle for final reports of work performed at NBS under the sponsorship of other government agencies.

Voluntary Product Standards—Developed under procedures published by the Department of Commerce in Part 10, Title 15, of the Code of Federal Regulations. The standards establish nationally recognized requirements for products, and provide all concerned interests with a basis for common understanding of the characteristics of the products. NBS administers this program as a supplement to the activities of the private sector standardizing organizations.

Consumer Information Series—Practical information, based on NBS research and experience, covering areas of interest to the consumer. Easily understandable language and illustrations provide useful background knowledge for shopping in today's technological marketplace.

Order the **above** *NBS publications from: Superintendent of Documents, Government Printing Office, Washington, DC 20402.*

Order the **following** *NBS publications—FIPS and NBSIR's—from the National Technical Information Services, Springfield, VA 22161.*

Federal Information Processing Standards Publications (FIPS PUB)—Publications in this series collectively constitute the Federal Information Processing Standards Register. The Register serves as the official source of information in the Federal Government regarding standards issued by NBS pursuant to the Federal Property and Administrative Services Act of 1949 as amended, Public Law 89-306 (79 Stat. 1127), and as implemented by Executive Order 11717 (38 FR 12315, dated May 11, 1973) and Part 6 of Title 15 CFR (Code of Federal Regulations).

NBS Interagency Reports (NBSIR)—A special series of interim or final reports on work performed by NBS for outside sponsors (both government and non-government). In general, initial distribution is handled by the sponsor; public distribution is by the National Technical Information Services, Springfield, VA 22161, in paper copy or microfiche form.

BIBLIOGRAPHIC SUBSCRIPTION SERVICES

The following current-awareness and literature-survey bibliographies are issued periodically by the Bureau:

Cryogenic Data Center Current Awareness Service. A literature survey issued biweekly. Annual subscription: domestic $25; foreign $30.

Liquefied Natural Gas. A literature survey issued quarterly. Annual subscription: $20.

Superconducting Devices and Materials. A literature survey issued quarterly. Annual subscription: $30. Please send subscription orders and remittances for the preceding bibliographic services to the National Bureau of Standards, Cryogenic Data Center (736) Boulder, CO 80303.

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
Washington, D.C. 20234

OFFICIAL BUSINESS

Penalty for Private Use, $300

POSTAGE AND FEES PAID
U.S. DEPARTMENT OF COMMERCE
COM-215

SPECIAL FOURTH-CLASS RATE
BOOK