NASA TECHNICAL MEMORANDUM

NASA TM X-73,092

NASA TM X-73,092

IDENTIFYING UNKNOWN MINERALS AND COMPOUNDS FROM X-RAY DIFFRACTION PATTERNS USING THE JOHNSON AND VAND FORTRAN IV COMPUTER PROGRAM

Frank T. Kyte and Maxwell B. Blanchard

San Jose State University
San Jose, California 95192

and

Ames Research Center
Moffett Field, California 94035

APR 1976
RECEIVED
NASA STI FACILITY
INPUT BRANCH

January 1976

(NASA-TM-X-73092) IDENTIFYING UNKNOWN MINERALS AND COMPOUNDS FROM X-RAY DIFFRACTION PATTERNS USING THE JOHNSON AND VAND FORTRAN 4 COMPUTER PROGRAM (NASA) 44 p HC $4.00 CSCL 08G
N76-20575
Unclas
G3/42 20732

1. Report No. NASA TM X-73,092	2. Government Accession No.	3. Recipient's Catalog No.

4. Title and Subtitle
IDENTIFYING UNKNOWN MINERALS AND COMPOUNDS FROM X-RAY DIFFRACTION PATTERNS USING THE JOHNSON AND VAND FORTRAN IV COMPUTER PROGRAM

5. Report Date

6. Performing Organization Code

7. Author(s)
Frank T. Kyte and Maxwell B. Blanchard

8. Performing Organization Report No.
A-6401

9. Performing Organization Name and Address
San Jose State University, San Jose, Calif. 95192
Ames Research Center, Moffett Field, Calif. 94035

10. Work Unit No.
195-21-04-02

11. Contract or Grant No.
NCA 2-OR 675-514

12. Sponsoring Agency Name and Address
National Aeronautics and Space Administration
Washington, D. C., 20546

13. Type of Report and Period Covered
Technical Memorandum

14. Sponsoring Agency Code

15. Supplementary Notes

16. Abstract

The contents of this document are designed to serve two purposes. First, to provide automated computer identification of minerals and compounds from unknown samples for those staff members (mineralogists and geochemists) in the Planetary Science and Applications Branch, Ames Research Center, who have been using the more traditional manual identification technique employing the Fink index. Second, to provide detailed instructions and worked examples for use in graduate level courses in mineralogy (Geol 211) and X-ray analysis applications (Geol 255) at San Jose State University. This document presumes the reader has either had prior course work or direct experience in mineral and rock type identification employing X-ray diffraction and emission techniques.

Appendix I, a compendium of computer printouts describing worked examples discussed in Section IV of this report, is available from either Dr. Marshall Maddock (San Jose State University, Department of Geology) or Maxwell Blanchard (Ames Research Center, Planetary Science and Applications Branch).

17. Key Words (Suggested by Author(s))
Mineral identification
X-ray diffraction
Johnson-Vand program

18. Distribution Statement
Unclassified - Unlimited

STAR Category - 42

19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 44	22. Price* $3.25

*For sale by the National Technical Information Service, Springfield, Virginia 22161

PREFACE

The contents of this document are designed to serve two purposes. First, to provide automated computer identification of minerals and compounds from unknown samples for those staff members (mineralogists and geochemists) in the Planetary Science and Applications Branch, Ames Research Center, who have been using the more traditional manual identification technique employing the Fink index. Second, to provide detailed instructions and worked examples for use in graduate level courses in mineralogy (Geol 211) and X-ray analysis applications (Geol 255) at San Jose State University. This document presumes the reader has either had prior course work or direct experience in mineral and rock type identification employing X-ray diffraction and emission techniques.

Appendix I, a compendium of computer printouts describing worked examples discussed in Section IV of this report, is available from either Dr. Marshall Maddock (San Jose State University, Department of Geology) or Maxwell Blanchard (Ames Research Center, Planetary Science and Applications Branch).

ACKNOWLEDGEMENTS

The authors wish to express their gratitude for assistance rendered by Dr. David W. Andersen, Geology Department, San Jose State University, and to Harry D. Shade, LFE - Environmental Analysis Laboratories, Richmond, Calif., for support services under contract NAS 2-7764. Sincerest appreciation is expressed to Gary Cunningham, San Jose State University (now at University of Oregon) for the early interpretation efforts, and to Jessie C. Gaspar and Ramon Cañas, Scientific Applications and Analysis Branch (Computer Division), Ames Research Center, for their excellent and helpful computer programming assistance. A special thanks to Audrey Summers, Theoretical Studies Branch, ARC, for her helpful discussions when we seemed destined to encounter immovable obstacles, and to Dr. Marshall E. Maddock, San Jose State University, who was first to apply the program's output in his mineralogy class.

This effort was supported by a NASA - Ames University Consortium Agreement (#NCA 2-OR 675-514).

TABLE OF CONTENTS

IDENTIFYING UNKNOWN MINERALS AND COMPOUNDS FROM X-RAY DIFFRACTION PATTERNS USING THE JOHNSON AND VAND FORTRAN IV COMPUTER PROGRAM

Frank T. Kyte* and Maxwell B. Blanchard

Ames Research Center

I. INTRODUCTION

The Johnson and Vand FORTRAN IV Program for Identification of Multiphase Powder Diffraction Data can be a useful tool to interpret complex diffraction patterns from mixtures of minerals. The program compares d-spacings and intensities of unknown diffraction patterns with every compound in the ASTM X-ray diffraction file. It then prints out every compound that satisfies the input parameters and data. These parameters include: chemical information, intensity match requirements, allowable error in pattern measurement, number and 2θ range of d-spacings observed, and background intensity. The 10 standards having the best match are then subtracted from the original unknown pattern, leaving a residual pattern.

This report describes the program operation and how to interpret the output. Input parameters are discussed in enough detail that a new user can understand why patterns are accepted or rejected by the computer. A principal reference for this report is "FORTRAN IV Program (Version 10) for the Identification of Multiphase Unknown Powder Diffraction Patterns" by G. G. Johnson, Jr. and V. Vand, published and distributed by ASTM, 1969.

II. JOHNSON - VAND PROGRAM DESCRIPTION

A. Input Parameters

Successful use of this program depends on the choice of input parameters. The most important of these are the error window and the log intensity match, which define how closely a standard pattern must match the unknown in its d-spacings and intensities. The parameters log intensity match, number, and percent of line matches apply only to the range of d-spacings within the input high and low range of 2θ. These five parameters interact in a very complicated way. When the d-spacing range is unusually narrow, number and percent should probably be decreased and log intensity match increased.

(1) Subfile option- The subfiles are alloys, organics, inorganics, minerals, and dual (inorganic compounds). The subfile option allows the operator to limit the search, and thus the output, to subfiles in which he is interested.

---

*Geology Department, San Jose State University, San Jose, Calif. 95192.

(2) Resolution- This parameter instructs the program that a high-resolution Gunier camera has been used and the intensities are "fuzzed" to give a "normal" resolution. This parameter is not used for Debeye-Scherrer diffraction patterns or for diffractometer scans.

(3) Error window- The error window is defined by a variable d^* which, unlike $d(\text{Å})$, has a linear relationship with θ and best approximates a constant error window for the observed range of θ values. The error window is a very important parameter because it defines how closely a standard line must match a d-spacing in the unknown pattern.

Errors in the diffracted line measurement (in Angstroms) are roughly proportional to d^2. A constant error window is best approximated by transformation to reciprocal spacings that are 1/d. When used by the program, the reciprocal spacings are multiplied by 1000, rounded down to the nearest integer, and added to 0.5. Thus, input d-spacings from 200 Å to 0.6 Å are represented by a set of numbers from 5.5 to 1666.5 (e.g., 5.5, 6.5, 7.5, . . ., 1666.5). This new number, d^*, allows the program to perform integer arithmetic for much faster calculations and defines a constant error window for any value of θ.

$$d^* \cong 1/d(\text{Å}) \times 1000$$

Figure 1 shows a plot of $d(\text{Å})$ and d^* versus °θ for $\text{CuK}\alpha = 1.5418$.

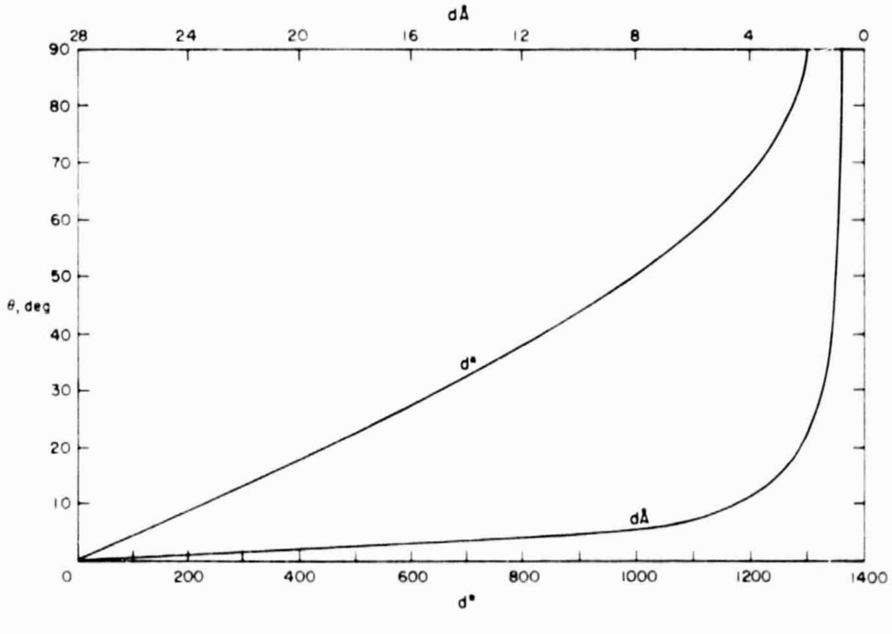


Figure 1.- Plot of d^* and dÅ versus °θ for $\text{CuK}\alpha = 1.5418$. (To convert from dÅ to d^* read d in Å from upper scale using line labeled dÅ which corresponds to a °θ value on left hand scale. Using this °θ value read the corresponding d^* using the line labeled d^* whose value is given on the lower scale. For example, a d-value of 2.98Å corresponds to an angle of 15° θ and is equivalent to 335 d^*.)

To determine the optimum error window for the instruments at Ames Research Center, a mixture of approximately equal weights of flourite, galena, rutile, chromite, and olivine was X-rayed using the diffractometer and the Debeye-Scherrer camera.

To see how much error could be expected, d* was calculated for multiple measurements on the Debeye-Scherrer powder films and for each of the diffractometer scans shown in Tables 1 and 2, respectively. The diffractometer scans were corrected because the goniometer was consistently about 0.05° θ too high. For 99 percent (508 measurements) of the line measurements, the maximum variation was 4d*, or an error of ±2d*. Of the remaining 1 percent (5 measurements) of line measurements, the maximum variation was 6d*.

A nominal error window of ±2 would seem to be appropriate for most identifications. However, inaccuracies in the ASTM file itself, or variations in d-spacings caused by solid solution, may necessitate the use of a larger window (±3 or greater). One should be cautious of using too large an error window because it can produce a large number of possibilities, and, in some cases, the windows could even overlap, creating a continuous coverage of several degrees 2θ. Usually, a broad error window of ±4 is suitable only for data in conjunction with chemical information.

TABLE 1.- DEBEYE-SCHERRER CAMERA FILMS

Sample pattern	Camera radius	Sample grain size	Quality of lines	Times measured	Total lines	Date
ASTM 1	114.6 mm	coarse	spotty	4	120	7/22/75
ASTM 2	114.6 mm	fine	continuous	4	112	7/24/75
ASTM 2A	57.3 mm	fine	continuous	4	66	7/28/75
ASTM 3	57.3 mm	coarse	spotty	4	95	7/29/75
ASTM 5	57.3 mm	fine	continuous	1	---	8/19/75

TABLE 2.- DIFFRACTOMETER SCANS

Sample pattern	°2θ/min	cps	Slits	Peaks	Date
ASTM 4A	1	250	1/2	14	7/30/75
ASTM B	1	100	1/2	18	8/4/75
ASTM C	1	250	1	22	8/4/75
ASTM D	1/2	250	1	22	8/4/75
ASTM E	1/2	250	1/2	13	8/5/75
ASTM F	1/4	100	1/2	31	8/5/75
BSTM 1	1/4	250	1	---	10/3/75
BSTM 2	1	250	1	---	10/9/75

(4) Log intensity match- This very important parameter is a requirement for intensity agreement between the unknown and an ASTM standard. All intensities are input on a 3-digit scale of 1 to 100, but all program calculations are on a 1-digit log scale of 0 to 9. The 3-digit (I_3) intensities are converted to the 1-digit (I_1) log scale by the formula

$$I_1 = 5 \log I_3$$

The conversion scale thus becomes

I_3	0-1	2	3	4-6	7-10	11-15	16-25	26-39	40-63	64-100
I_1	0	1	2	3	4	5	6	7	8	9

Thus, an input intensity of 40 is equivalent to an input of 63 and very different from an input of 39. Program users should keep this in mind when inputting intensities.

Log intensity match is defined as

$$\text{log intensity match } (I_1) = 9 - (I_{1STD} - I_{2UNK})_{MAX}$$

where $(I_{1STD} - I_{2UNK})_{MAX}$ is the maximum intensity disagreement between any line in the standard and its counterpart in the unknown pattern. When selecting an input value for log intensity match, the user must remember that any standard pattern with a match of this value or less will be rejected as a possibility.

For example, when log intensity match is input with a value of $2(I_3)$ it is converted to a log scale value of $1(I_1)$, and then intensities of every line in the standard are compared with corresponding intensities of lines in the unknown (which may be zero if no line is present in the unknown to compare with one in the standard pattern), and log intensity match is calculated for each line in the standard. If the log intensity match of any single line in the pattern has a value of $1(I_1)$ or lower, then the standard will be rejected as a possibility. Therefore, in this example, it is necessary that

$$9 - (I_{1STD} - I_{1UNK})_{MAX} > 1$$

or that

$$(I_{1STD} - I_{1UNK})_{MAX} \neq 8 \text{ or } 9$$

Essentially what this means, in returning to the I_3 scale, is that lines with intensities of 40 to 100 in the standard must match with corresponding lines in the unknown. Failure to do so results in rejection, even if every other line in the standard pattern except one, say with an intensity of 40, matches perfectly.

(5) Number- This is the minimum number of line matches that the program requires for an identification. If this minimum is set too high, a pattern may not be identified (e.g., diamond has only 5 lines).

(6) Percent of line matches- This parameter allows the operator to select the minimum percent of line matches between an unknown and a standard. In most cases, 33 percent should be sufficient, requiring that at least 1/3 of the lines in the standard must match the unknown pattern.

(7) Icycle- This parameter instructs the program to go through a second cycle, during which time, all previous parameters are increased. The amount of increase is specified by another set of parameters described in Section III. In general, it is suggested that this parameter *not* be used because *all* parameters must be increased. If Icycle were used to increase the error window, a larger number of standard lines could be compared, but rejection of previous matches could occur from stricter requirements for log intensity match or number.

(8) Observed range of d-spacings- This is defined by two parameters, DHI and DLO, which are the range of d-spacings capable of being observed in the diffraction experiment. DHI must be higher than the highest input d-spacing. For example, a 3Å line might be the largest observed spacing when the experimental range was up to 20Å. If DHI were input as 3Å rather than 20Å, then standards with strong lines between 3 and 20Å could be matched when they should have been rejected. DLO should equal the lowest d-spacing in the unknown pattern. Note that back reflections (from Debeye-Scherrer camera patterns) are often omitted in the ASTM file. It is desirable to include all of the unknown patterns down to about 1.1Å.

(9) Background intensity- This parameter instructs the program to disregard all lines in the standard pattern of this value or smaller. This is

difficult to quantify on powder films and should be given as about half the lowest intensity observed. On diffractometer scans, it is a good idea to set the counter scale so that the most intense peak(s) is at about 100 on the chart. Then the background (as well as other peaks) can be read directly off the chart.

(10) Option- This parameter uses chemistry. If the input is MAJOR, all the compounds (regardless of chemistry) will be printed. If the input is CHEM or SOLID, then only compounds which are chemically correct will be printed in the output as potential identifications. If the input is MINOR: (a) all compounds fitting parameters for a match will be printed, and (b) if a compound is chemically correct, the intensity agreement will not be applied.

(11) Chemical information- Chemical information can be used in this program. It is possible to list all elements and functional groups which are known to be present and those which are known to be absent. Chemical information is highly desirable because it may narrow down a list of over 100 possibilities to less than 10 or 20. It is strongly suggested that chemistry be used whenever possible.

(12) Preconceived possibilities- This option is to be used for phases which are *definitely* known to be present. It can be very useful in that part of the output (described below) where standard phases are subtracted from the unknown pattern. Preconceived possibilities are always subtracted first. Thus, if a match was selected on the basis of certain lines in the unknown pattern generated by the preconceived possibility, when it is subtracted from the residual unknown pattern, it will have fewer lines to compare with and it will be scaled down to have a minimal effect on the residual pattern. This will be discussed in more detail in the next section.

B. Program Output

The program output comprises of five sections: INPUT, OUTPUT, REPORT, MATCHD, and SUBSTI. Figures 2 through 8 show a portion of the output obtained by Johnson and Vand as their own example. It was generated by a known mixture of equal weights of CaF_2, SiO_2, ZnS, $CaCO_3$, and PbS. The pattern was obtained from a Norelco diffractometer at 1°/min, using Cu radiation and peak intensities.

(1) INPUT- This is a summary and description of the input parameters and data (fig. 2), and should always be checked carefully for correctness. If this section is not checked, the user might miss an error comment such as, "YOU MAY HAVE KEYPUNCHED INPUT DATA WRONG - A SPACING IS OUT OF ORDER - PROGRAM ORDERS AND WILL CONTINUE."

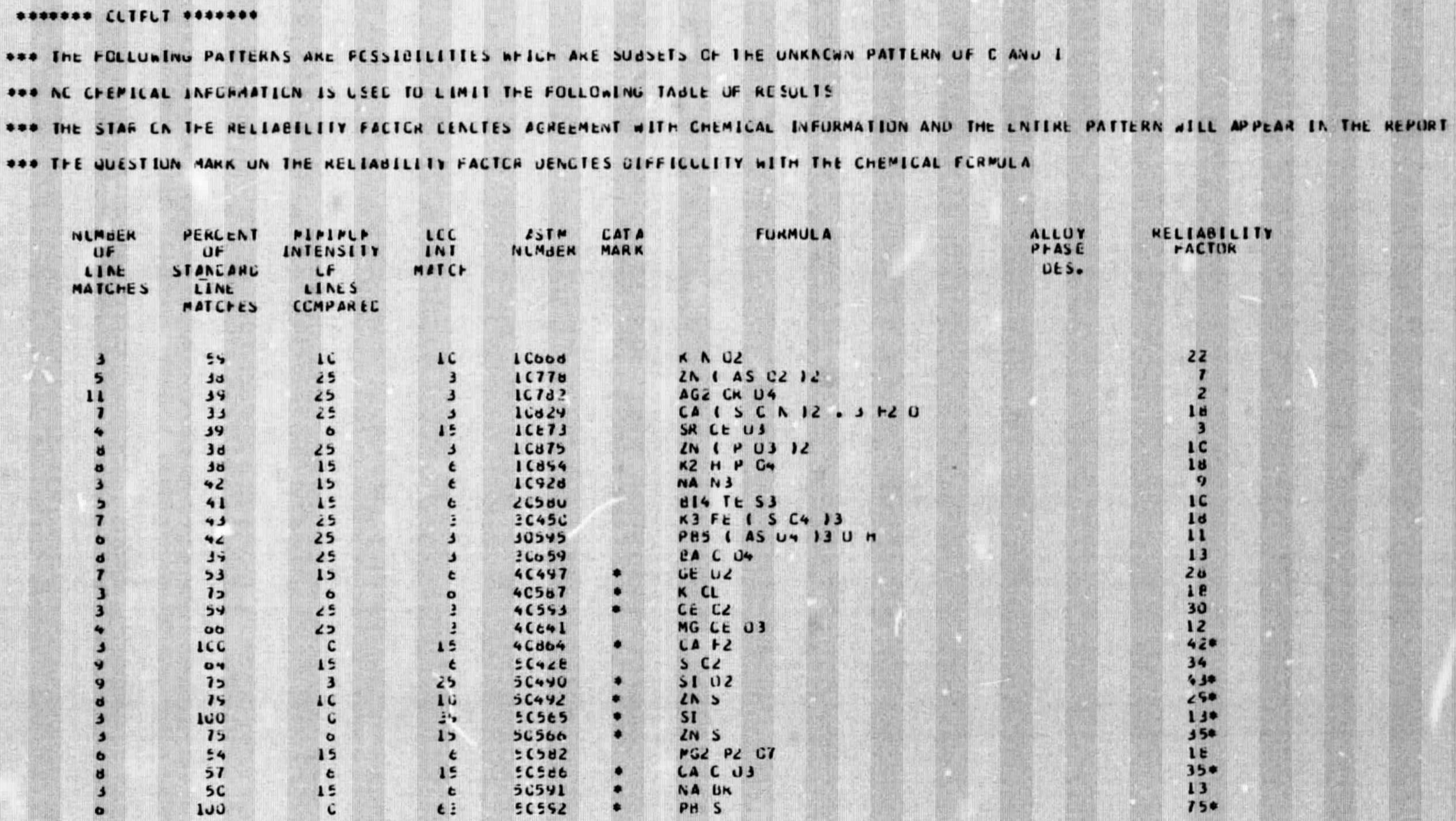
(2) OUTPUT- The OUTPUT section (fig. 3) lists every compound, regardless of chemical information (unless CHEM or SOLID options are used), that fits the input data and parameters. It also lists the various numerical measures of degree of fit of the unknown to the standard patterns, as well as a reliability factor which is a composite index calculated from the percentage of line

```
******* INPUT *******

**COMMENT** THE FOLLOWING OUTPUT WAS OBTAINED USING THE JOHNSON-VAND X-RAY POWDER DIFFRACTION SEARCH AND MATCH SYSTEM VERSION 10

**COMMENT** PATTERNS IDENTIFIED WITH A LOG INT MATCH OF  2 OR LESS OF STANDARD IN RANGE CONSIDERED WILL NOT BE PRINTED

**COMMENT** PATTERNS WITH LESS THAN   3 LINE MATCHES OF STANDARD ABOVE BACKGROUND IN RANGE CONSIDERED ARE NOT PRINTED

**COMMENT** PATTERNS WITH LESS THAN 33 PERCENT MATCH OF STANDARD ABOVE BACKGROUND IN RANGE CONSIDERED ARE NOT PRINTED

**COMMENT** BACKGROUND RADIATION IS  2 (THRESHOLD MEASUREMENT VALUE)

**COMMENT** ONLY D-SPACINGS LESS THAN  22.000 ARE USED IN THIS PHASE

**COMMENT** ONLY D-SPACINGS GREATER THAN   1.450 ARE USED IN THIS PHASE

*********** VALUES ABOVE ARE EXPERIMENTAL RANGE

*********** EXPERIMENTAL RANGE IS ALWAYS GREATER THAN MEASURED D RANGE

**COMMENT** THE FOLLOWING ELEMENTS ARE KNOWN TO BE PRESENT CA SI  F  O ZN  S  C PB

**COMMENT** THE FOLLOWING ELEMENTS ARE KNOWN TO BE ABSENT  H HE LI BE  B  N NE NA MG AL  P CL AR  K SC TI  V CR MN FE CO NI CU GA GE
                                                           AS SE BR KR RB SR  Y ZR NB MO TC RU RH PD AG CD IN SN SB TE  I XE CS BA LA
                                                           CE PR ND PM SM EU GD TB DY HO ER TM YB LU HF TA  W RE OS IR PT AU HG TL BI
                                                           PO AT RN FR RA AC TH PA  U NP PU AM CM BK CF ES FM

**COMMENT** THE FOLLOWING FUNCTIONAL GROUPS ARE KNOWN TO BE ABSENT S O2       P O

**COMMENT** MAJOR COMPONENT OPTION BEING USED

**COMMENT** WINDOW FOR THIS RUN IS + OR -   2

SAMPLE     SAMPLE NUMBER 5 CAF2 SIO2 ZNS CACO3 PBS                                              UNIQUE ID.=        37  01/20/72

UNKNOWN SPACINGS AND INTENSITIES WITH ERROR WINDOW
         4.2700           14           4.3011    4.2283
         3.8600            6           3.8835    3.8241
         3.4300           61           3.4542    3.4072
         3.3500           77           3.3727    3.3276
         3.3200           26           3.3385    3.2949
         3.1500           28           3.1658    3.1299
         3.1340           34           3.1456    3.1104
```

Figure 2.- INPUT. (This section should be checked carefully for correctness and for any possible error messages.)

```
3.0390        100       3.0534   3.0160
2.9730         77       2.9855   2.9542
2.9310         22       2.9455   2.9112
2.4990          5       2.5094   2.4845
2.4600          5       2.4722   2.4480
2.2870         12       2.2962   2.2753
2.2340          3       2.2447   2.2247
2.1360          6       2.1350   2.1209
2.1010         49       2.1119   2.0942
1.9416          3       1.9512   1.9361
1.9325         21       1.9355   1.9247
1.9141         32       1.9212   1.9066
1.8777         11       1.8850   1.8709
1.8203          5       1.8265   1.8132
1.7915         25       1.7969   1.7841
1.7674          5       1.7746   1.7621
1.7151         13       1.7197   1.7079
1.6734          4       1.6793   1.6681
1.6524          6       1.6570   1.6461
1.6328         23       1.6360   1.6273
1.5415          6       1.5468   1.5373
1.4860          5       1.4914   1.4826
```

Figure 2.- Concluded.



```
******* OUTPUT *******

*** THE FOLLOWING PATTERNS ARE POSSIBILITIES WHICH ARE SUBSETS OF THE UNKNOWN PATTERN OF C AND I

*** NO CHEMICAL INFORMATION IS USED TO LIMIT THE FOLLOWING TABLE OF RESULTS

*** THE STAR ON THE RELIABILITY FACTOR DENOTES AGREEMENT WITH CHEMICAL INFORMATION AND THE ENTIRE PATTERN WILL APPEAR IN THE REPORT

*** THE QUESTION MARK ON THE RELIABILITY FACTOR DENOTES DIFFICULTY WITH THE CHEMICAL FORMULA


   NUMBER    PERCENT    MINIMUM      LOC     ASTM   DATA            FORMULA                    ALLOY      RELIABILITY
     OF        OF      INTENSITY     INT    NUMBER  MARK                                       PHASE        FACTOR
    LINE     STANDARD     OF        MATCH                                                      DES.
   MATCHES     LINE      LINES
             MATCHES    COMPARED


      3        59         10         10     10668           K N O2                                             22
      5        38         25          3     10778           ZN ( AS O2 )2                                       7
     11        39         25          3     10782           AG2 CR O4                                           2
      7        33         25          3     10829           CA ( S C N )2 . 3 H2 O                             18
      4        39          6         15     10873           SR CE O3                                            3
      8        38         25          3     10875           ZN ( P O3 )2                                       10
      8        38         15          6     10854           K2 H P O4                                          18
      3        42         15          6     10928           NA N3                                               9
      5        41         15          6     20580           BI4 TE S3                                          10
      7        43         25          3     30450           K3 FE ( S O4 )3                                    18
      6        42         25          3     30595           PB5 ( AS O4 )3 O H                                 11
      8        39         25          3     30659           BA C O4                                            13
      7        53         15          6     40497    *      GE O2                                              28
      3        75          6          6     40587    *      K CL                                               18
      3        59         25          3     40593    *      CE O2                                              30
      4        66         25          3     40641           MG CE O3                                           12
      3       100          0         15     40864    *      CA F2                                              42*
      9        64         15          6     50428           S O2                                               34
      9        75          3         25     50490    *      SI O2                                              43*
      8        79         10         10     50492    *      ZN S                                               29*
      3       100          0         35     50565    *      SI                                                 13*
      3        75          6         15     50566    *      ZN S                                               35*
      6        54         15          6     50582           MG2 P2 O7                                          18
      8        57          6         15     50586    *      CA C O3                                            35*
      3        50         15          6     50591    *      NA BR                                              13
      6       100          0         63     50592    *      PB S                                               75*
```

Figure 3.- OUTPUT. (This section lists all the possibilities which fit the input parameters. All intensities are I_3 values.)

```
 3     75     25      3     50667   *   CU2 O                                      11
 3     75     25      3     50702   *   RE                                         16
 3     59      6     15     50724       GA2 SE3                                    12
 3     59     25      3     60329       ( PR CL .83 ) 11 .                  32F    24
 3     75      6     15     60344   *   CU CL                                      44
 5     50     15      6     60347   *   Y O F                                      14
15     46     25      3     60366       BA2 SI O4                                  12
 6     33     25      3     60381       PB8 O7 BR2 . 5 H2 O                        11
 3     33     25      3     60395   *   ( SN O )                             4T    16
 4     66      6     15     60399   *   BA ZR O3                                   17
 5     55     15      6     60400   *   BA2 CA W O6                                33
 3     50     25      3     60640       ( MG B2 )                            3H     6
 5     62     15      6     60644   *   ( MO B )                             8O    17
 5     55     25      3     70205   *   TL2 SI F6                                  12
 3     55     15      6     70261       NA CA CD Y F8                               5
 7     58     15      6     70271   *   NA N O3                                    17
 3     37      3     25     70279   *   MG2 ( O H ) CL O3 . 3 H2 O                 13
 5     55     15      6     80200       ( MO5 CR6 FE18 )                    58B    11
 5     62     25      3     80376       PU O2                                      21
 4     57     25      3     80383       ( LI MG ZN )                        24C    13
 6     50     15      6     80455   *   BA MO O4                                   27
 4     39     25      3     80457   *   BA W O4                                    18
 5     45     25      3     80468   *   HG2 BR2                                     7
 9     40     25      3     80474   *   NA I O3                                    11
 3     75      6      6     80487   *   ( SN TE )                            8F    24
 6     33     15      6     80497   *   GA P O4                                     5
 3     59     15      6     90012       ( B N )                              4H    13
 6    100      0     15     90096       ( BE5 PD )                          24F    44
 6     46     15      6     90104       ( CS2 O )                            9R    15
 6    100      0     10     90313       ( BE5 PT )                          24F    37
 8     42     25      3     90447       BI2 TE2 S                                   3
10     41     25      3     90477   *   AU AG TE4                                  11
 5     55     25      3    100111       K3 CR F6                                   23
 5     35     25      3    100225       ( TI SI2 )                          12Q     4
 5     35     15      6    100423       AL P O4                                     5
 8     68     15      6    100434       ZN S                                      32*
 5     71     10     10    110213   *   BA B6                                      18
 8     44     15      6    110345       CA ( MN, MG ) ( C O3 )2                     5
 8     75     15      6    110513       ( ZNO .43 MNO .31 FEO .26 ) S              25
 3     50     25      3    110608       GD2 O3                                     15
14     33     25      3    110641       ( AG P O3 )4                               18
 4     39     25      3    110690   *   SB2 O5                                     21
 6    100      0     10    120042       K2 U F6                                    18
11     36     25      3    120300       K2 CR2 O7                                  15
 5     41     25      3    120338       BA MG GE2 O6                               22
```

Figure 3.- Concluded.

```
******* REPORT *******

THE FOLLOWING REPORT IS BASED ON THE ABOVE TABLE AND CHEMICAL INFORMATION (BOTH POSITIVE AND NEGATIVE).

IF NO CHEMICAL INFORMATION WAS GIVEN AS INPUT, THE 10 PATTERNS THAT FOLLOW HAVE THE HIGHEST RELIABILITY FACTOR

THE ASTM NUMBER AND THE RELIABILITY PRECEED THE CHEMICAL FORMULA

THE TRUE RESULTS COULD BE A MIXTURE OF THE FOLLOWING POSSIBILITIES

THE 10 POSSIBILITIES WITH THE HIGHEST RELIABILITY FACTORS WILL BE LISTED IN MATCHD IN ORDER OF DECREASING RELIABILITY FACTOR

THE PRECONCEIVED POSSIBILITIES HAVE A RELIABILITY FACTOR OF 999


   40864                50490                50492                50565                50566

      42                   43                   29                   13                   35


   CA F2                SI O2                ZN S                 SI                   ZN S
             *4                                        *4                   *

  1.932  100- 63       3.335  100- 63       3.306  100- 63       3.140  100- 63       3.120  100- 63
  3.150  100- 63       4.264   39- 25       3.130  100- 63       1.921   63- 39       1.910   63- 39
  1.646   39- 25       1.817   25- 15       2.928  100- 63       1.638   39- 25       1.633   39- 25
  1.115   25- 15       1.542   15- 10       1.910  100- 63       1.108   25- 15       2.706   10-  6
  1.365   15- 10       2.281   15- 10       1.765   63- 39       1.246   15- 10       1.103   10-  6
  1.252   10-  6       2.460   15- 10       1.630   63- 39       0.918   15- 10       1.240   10-  6
  0.864   10-  6       1.375   15- 10       2.275   39- 25       0.859   10-  6       1.350    6-  3
  0.923   10-  6       1.373   10-  6       1.296   15- 10       1.045   10-  6       0.914    6-  3
  1.051   10-  6       2.130   10-  6       1.103   15- 10       1.358   10-  6       1.040    6-  3
  0.966    6-  3       1.382   10-  6       1.599   15- 10       0.828    6-  3       0.855    3-  2
  0.833    3-  2       1.671   10-  6       1.210   10-  6       0.960    6-  3       0.956    3-  2
  0.910    1-  0       1.978    6-  3       1.654   10-  6                            0.824    2-  1
                       2.235    6-  3       0.840   10-  6                            1.208    2-  1
                       1.200    6-  3       0.885   10-  6                            1.561    2-  1
                       1.082    6-  3       1.161   10-  6
                       1.180    6-  3       0.915   10-  6
                       1.184    6-  3       0.955    6-  3
                       1.255    6-  3       0.998    6-  3
                       1.288    3-  2       1.072    6-  3
                       1.452    3-  2       1.251    6-  3
```

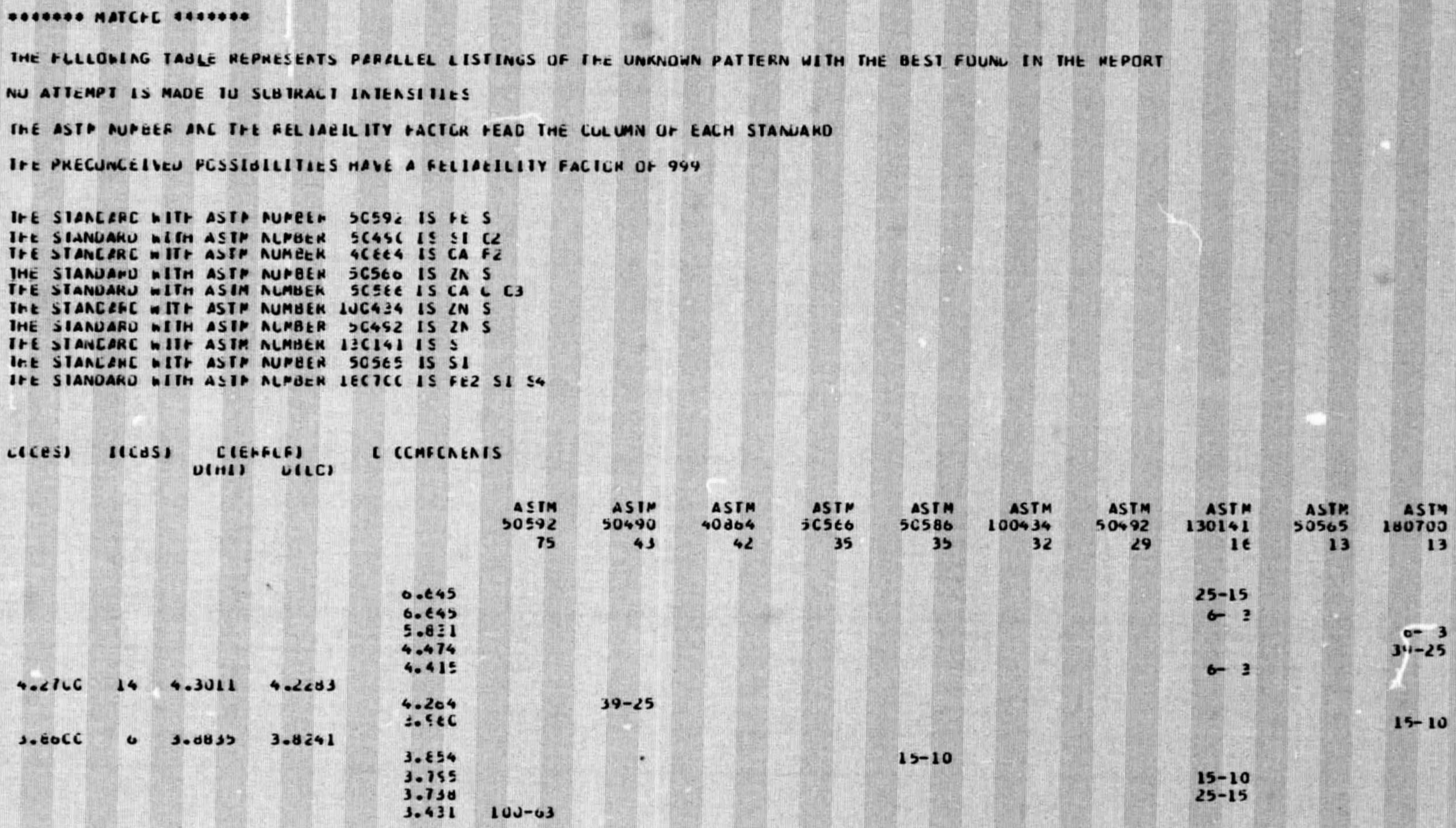
Figure 4.- REPORT. (This section lists d-spacings and intensities (I_3 based on the I_1 scale) for all the possibilities which match the chemical information. If no chemical information is used, then the 10 with the highest reliability factors are printed. The asterisk and/or number to the right of the formula denotes data quality and subfile.)

```
                    1.660      3-  2          0.918      6-  3
                    0.961      2-  1          1.040      6-  3
                    0.988      2-  1          1.463      6-  3
                    0.990      2-  1          1.170      6-  3
                    1.015      2-  1          1.226      3-  2
                    1.035      2-  1          0.908      2-  1
                    1.043      2-  1          1.564      2-  1
                    1.048      2-  1          0.977      1-  0
                    1.153      2-  1          1.137      1-  0
                    1.197      2-  1          1.413      1-  0
                    1.228      2-  1
                    0.928      1-  0
                    0.976      1-  0
                    0.978      1-  0
                    1.063      1-  0
                    1.114      1-  0
                    1.141      1-  0
                    1.417      1-  0
                    1.609      1-  0
                    1.800      1-  0


  50586                 50592                 100434                130141                180700

     35                    75                     32                    16                    13

  CA C O3               PB S                  ZN S                  S                     PB2 SI O4
                 *4                    *4                    4                     4

3.035    100- 63      2.972    100- 63      2.928    100- 63      3.295    100- 63      2.911    100- 63
2.094     25- 15      3.431    100- 63      3.306    100- 63      6.645     25- 15      4.474     39- 25
2.266     25- 15      2.099     63- 39      1.910    100- 63      3.738     25- 15      2.677     39- 25
1.874     25- 15      1.791     35- 25      1.765     63- 39      1.452     25- 15      2.099     25- 15
1.914     25- 15      1.327     25- 15      3.130     63- 39      3.795     15- 10      3.396     25- 15
2.497     15- 10      1.714     25- 15      1.630     63- 39      3.044     15- 10      3.960     15- 10
3.854     15- 10      1.211     10-  6      2.275     63- 39      3.101     10-  6      1.732     15- 10
1.604     10-  6      1.361     10-  6      1.599     25- 15      2.491     10-  6      2.014     10-  6
1.440      6-  3      1.485     10-  6      1.654     15- 10      1.635      6-  3      2.541     10-  6
1.526      6-  3      0.990      6-  3                            1.857      6-  3      3.306     10-  6
1.525      6-  3      1.142      6-  3                            2.460      6-  3      3.252     10-  6
```

Figure 4.- Continued.

```
1.044   6-  3      1.004   6-  3                 4.415   6-  3      1.775   6-  3

1.515   6-  3      0.895   6-  3                 1.432   6-  3      1.903   6-  3
1.625   6-  3      0.535   6-  3                 1.566   6-  3      2.245   6-  3
0.577   3-  2      0.823   3-  2                 1.787   6-  3      3.063   6-  3
1.048   3-  2      0.631   3-  2                 1.871   6-  3      1.951   6-  3
1.154   3-  2      1.045   3-  2                 2.181   6-  3      5.831   6-  3
1.180   3-  2      0.905   2-  1                 2.442   6-  3      2.195   2-  1
1.421   3-  2      0.857   1-  0                 2.998   6-  3
1.509   3-  2                                    6.645   6-  3
2.645   3-  2                                    1.599   2-  1
0.566   2-  1                                    1.679   2-  1
1.012   2-  1                                    1.708   2-  1
1.036   2-  1                                    1.896   2-  1
1.235   2-  1                                    1.932   2-  1
1.256   2-  1                                    2.095   2-  1
1.340   2-  1                                    2.601   2-  1
1.474   2-  1
1.566   2-  1
0.576   1-  0
0.565   1-  0
0.590   1-  0
1.023   1-  0
1.061   1-  0
1.124   1-  0
1.142   1-  0
1.248   1-  0
1.265   1-  0
1.356   1-  0
```

Figure 4.- Concluded.

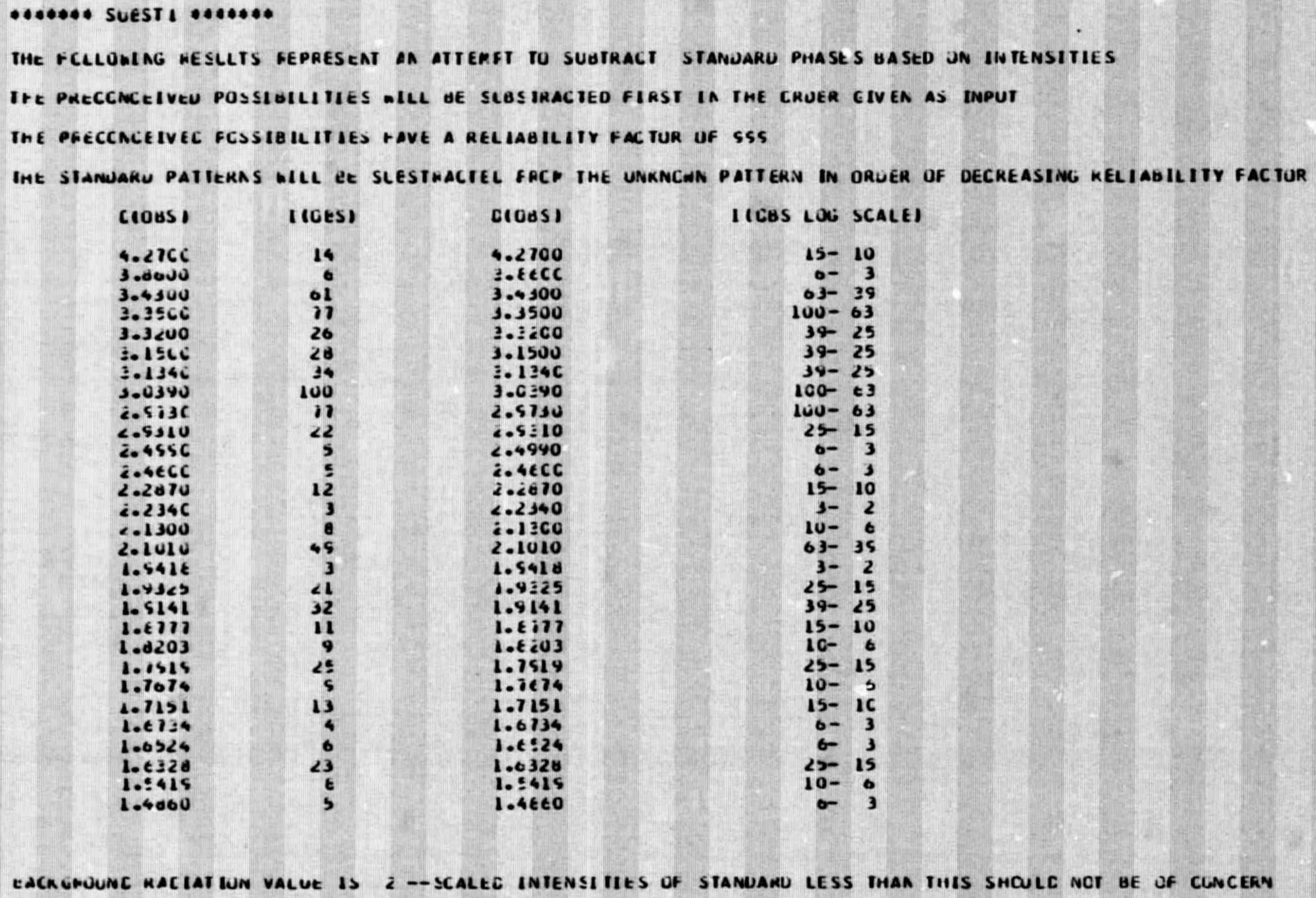


```
******* MATCHD *******

THE FOLLOWING TABLE REPRESENTS PARALLEL LISTINGS OF THE UNKNOWN PATTERN WITH THE BEST FOUND IN THE REPORT

NO ATTEMPT IS MADE TO SUBTRACT INTENSITIES

THE ASTM NUMBER AND THE RELIABILITY FACTOR HEAD THE COLUMN OF EACH STANDARD

THE PRECONCEIVED POSSIBILITIES HAVE A RELIABILITY FACTOR OF 999


THE STANDARD WITH ASTM NUMBER  50592 IS FE S
THE STANDARD WITH ASTM NUMBER  50490 IS SI O2
THE STANDARD WITH ASTM NUMBER  40864 IS CA F2
THE STANDARD WITH ASTM NUMBER  50566 IS ZN S
THE STANDARD WITH ASTM NUMBER  50586 IS CA C O3
THE STANDARD WITH ASTM NUMBER 100434 IS ZN S
THE STANDARD WITH ASTM NUMBER  50492 IS ZN S
THE STANDARD WITH ASTM NUMBER 130141 IS S
THE STANDARD WITH ASTM NUMBER  50565 IS SI
THE STANDARD WITH ASTM NUMBER 180700 IS FE2 SI S4


D(OBS)   I(OBS)    D(ERROR)        D COMPONENTS
                  D(HI)   D(LO)

                                                 ASTM     ASTM     ASTM     ASTM     ASTM     ASTM     ASTM     ASTM     ASTM     ASTM
                                                 50592    50490    40864    50566    50586   100434    50492   130141    50565   180700
                                                    75       43       42       35       35       32       29       16       13       13


                                        6.645                                                                   25-15
                                        6.645                                                                    6- 3
                                        5.831                                                                                       6- 3
                                        4.474                                                                                      39-25
                                        4.415                                                                    6- 3
 4.2700    14   4.3011   4.2283
                                        4.264             39-25
                                        3.980                                                                                      15-10
 3.8600     6   3.8835   3.8241
                                        3.854                                       15-10
                                        3.755                                                                   15-10
                                        3.738                                                                   25-15
                                        3.431   100-63
```

Figure 5.- MATCHD. (The 10 best matches are listed in order of decreasing reliability factor. The intensities below the ASTM number correspond to d-spacings to the left.)

3.4300	61	3.4542	3.4072											
				3.356										25-15
3.3500	77	3.3727	3.3278											
				3.339		100-63								
3.3200	26	3.3384	3.2945											
				3.308							100-63			
				3.306										10-6
				3.308						100-63				
				3.299								100-63		
				3.252										10-6
3.1500	28	3.1696	3.1299											
				3.150			100-63							
				3.140									100-63	
3.1340	34	3.1496	3.1104											
				3.130							100-63			
				3.130						63-39				
				3.120				100-63						
				3.101								10-6		
				3.063										6-3
				3.044								15-10		
3.0350	100	3.0534	3.0166											
				3.035					100-63					
				2.998								6-3		
2.9730	17	2.9855	2.9542											
				2.972	100-63									
2.9310	22	2.9455	2.9112											
				2.928							100-63			
				2.928						100-63				
				2.911										100-63
				2.845					3-2					
				2.706				10-6						
				2.677										39-25
				2.601								2-1		
				2.541										10-6
2.4950	5	2.5094	2.4845											
				2.497					15-10					
				2.491								10-6		
				2.460								6-3		
				2.460		15-10								
2.4600	5	2.4722	2.4480											
				2.442								6-3		
2.2870	12	2.2962	2.2753											
				2.286					25-15					
				2.281		15-10								
				2.275						63-39				

Figure 5.- Concluded.



```
******* SUBSTI *******

THE FOLLOWING RESULTS REPRESENT AN ATTEMPT TO SUBTRACT  STANDARD PHASES BASED ON INTENSITIES

THE PRECONCEIVED POSSIBILITIES WILL BE SUBSTRACTED FIRST IN THE ORDER GIVEN AS  INPUT

THE PRECONCEIVED POSSIBILITIES HAVE A RELIABILITY FACTOR OF 999

THE STANDARD PATTERNS WILL BE SUBSTRACTED FROM THE UNKNOWN PATTERN IN ORDER OF DECREASING RELIABILITY FACTOR

       D(OBS)         I(OBS)          D(OBS)              I(OBS LOG SCALE)

       4.2700           14            4.2700                   15- 10
       3.8600            6            3.8600                    6-  3
       3.4300           61            3.4300                   63- 39
       3.3500           77            3.3500                  100- 63
       3.3200           26            3.3200                   39- 25
       3.1500           28            3.1500                   39- 25
       3.1340           34            3.1340                   39- 25
       3.0390          100            3.0390                  100- 63
       2.9730           77            2.9730                  100- 63
       2.9310           22            2.9310                   25- 15
       2.4990            5            2.4990                    6-  3
       2.4600            5            2.4600                    6-  3
       2.2870           12            2.2870                   15- 10
       2.2340            3            2.2340                    3-  2
       2.1300            8            2.1300                   10-  6
       2.1010           49            2.1010                   63- 39
       1.9418            3            1.9418                    3-  2
       1.9325           21            1.9325                   25- 15
       1.9141           32            1.9141                   39- 25
       1.8777           11            1.8777                   15- 10
       1.8203            9            1.8203                   10-  6
       1.7519           25            1.7519                   25- 15
       1.7674            9            1.7674                   10-  6
       1.7151           13            1.7151                   15- 10
       1.6734            4            1.6734                    6-  3
       1.6524            6            1.6524                    6-  3
       1.6328           23            1.6328                   25- 15
       1.5419            8            1.5419                   10-  6
       1.4860            5            1.4860                    6-  3


BACKGROUND RADIATION VALUE IS  2 --SCALED INTENSITIES OF STANDARD LESS THAN THIS SHOULD NOT BE OF CONCERN
```

Figure 6.- SUBSTI. (I_3 values for the unknown pattern are converted to the log scale.)

```
                                                                          RESULTING RESIDUE PATTERN
D(OBS)    D(HI)     D(LO)   I(OBS)       D(STANDARD)       I(STANDARD)       D(OBS)        I(OBS)

4.2700    4.3011    4.2283    15                                             4.2700          15
3.8600    3.8835    3.8241     6                                             3.8600           6
                                         3.431             80
3.4300    3.4542    3.4072    63                                             3.4300         -17
3.3500    3.3727    3.3278   100                                             3.3500         100
3.3200    3.3389    3.2949    39                                             3.3200          39
3.1500    3.1656    3.1259.   39                                             3.1500          39
3.1340    3.1496    3.1104    39                                             3.1340          39
3.0390    3.0534    3.0166   100                                             3.0390         100
2.9730    2.9855    2.9542   100                                             2.9730          19
                                         2.972             80
2.9310    2.9455    2.9112    25                                             2.9310          25
2.4990    2.5094    2.4845     6                                             2.4990           6
2.4600    2.4722    2.4480     6                                             2.4600           6
2.2870    2.2962    2.2753    15                                             2.2870          15
2.2340    2.2447    2.2247     3                                             2.2340           3
2.1300    2.1390    2.1209    10                                             2.1300          10
2.1010    2.1119    2.0942    63                                             2.1010          12
                                         2.099             50
1.9418    1.9512    1.9361     3                                             1.9418           3
1.9325    1.9399    1.9249    25                                             1.9325          25
1.9141    1.9212    1.9066    39                                             1.9141          39
1.8777    1.8850    1.8709    15                                             1.8777          15
1.8203    1.8265    1.8132    10                                             1.8203          10
1.7919    1.7969    1.7841    25                                             1.7919          -5
                                         1.791             31
1.7674    1.7746    1.7621    10                                             1.7674          10
1.7151    1.7157    1.7079    15                                             1.7151          -4
                                         1.714             20
1.6734    1.6793    1.6661     6                                             1.6734           6
1.6524    1.6570    1.6461     6                                             1.6524           6
1.6328    1.6380    1.6273    25                                             1.6328          25
1.5419    1.5468    1.5373    10                                             1.5419          10
1.4860    1.4914    1.4826     6                                             1.4860          -1
                                         1.485              8

THE  STANDARD WITH ASTM NUMBER  50592 HAS A SCALING FACTOR OF  0.807 AND A FORMULAE OF PB S
```

Figure 7.- SUBSTI. (Intensities for the standard with the highest reliability are scaled down and subtracted from the unknown pattern. The next standard will be scaled down to the residue pattern.)

```
                                                                              RESULTING RESIDUE PATTERN
D(OBS)   D(HI)    D(LO)   I(OBS)     D(STANDARD)      I(STANDARD)               D(OBS)       I(OBS)

4.2700   4.3011   4.2283   19                                                   4.2700        -11
                                      4.264               27
3.8600   3.8835   3.8241    6                                                   3.8600          6
3.4300   3.4542   3.4072  -17                                                   3.4300        -17
3.3500   3.3727   3.3278  100                                                   3.3500         30
                                      3.339               70
3.3200   3.3389   3.2949   39                                                   3.3200         39
3.1500   3.1656   3.1299   39                                                   3.1500         39
3.1340   3.1496   3.1104   39                                                   3.1340         39
3.0390   3.0534   3.0166  100                                                   3.0390        100
2.9730   2.9855   2.9542   19                                                   2.9730         19
2.9310   2.9455   2.9112   25                                                   2.9310         25
2.4950   2.5054   2.4845    6                                                   2.4950          6
                                      2.460               10
2.4600   2.4722   2.4480    6                                                   2.4600         -4
2.2870   2.2982   2.2753   15                                                   2.2870          4
                                      2.281               10
                                      2.235                4
2.2340   2.2447   2.2247    3                                                   2.2340          0
2.1300   2.1390   2.1209   10                                                   2.1300          3
                                      2.130                7
2.1010   2.1115   2.0942   12                                                   2.1010         12
                                      1.978                4
1.9418   1.9512   1.9361    3                                                   1.9418          3
1.9325   1.9399   1.9249   25                                                   1.9325         25
1.9141   1.9212   1.9066   39                                                   1.9141         39
1.8777   1.8850   1.8709   15                                                   1.8777         15
1.8203   1.8265   1.8132   10                                                   1.8203         -7
                                      1.817               17
                                      1.800                0
1.7919   1.7969   1.7841   -5                                                   1.7919         -5
1.7674   1.7740   1.7621   10                                                   1.7674         10
1.7151   1.7197   1.7079   -4                                                   1.7151         -4
1.6734   1.6793   1.6681    6                                                   1.6734          0
                                      1.671                7
                                      1.660                2
1.6524   1.6570   1.6461    6                                                   1.6524          6
1.6328   1.6380   1.6273   25                                                   1.6328         25
                                      1.609                0
                                      1.542               10
1.5419   1.5468   1.5373   10                                                   1.5419          0

1.4860   1.4914   1.4826   -1                                                   1.4860         -1
                                      1.452                2

THE  STANDARD WITH ASTM NUMBER  50490 HAS A SCALING FACTOR OF  0.703 AND A FORMULAE OF SI O2
```

Figure 7.- Concluded.

```
******* SUBSTI FINAL REPORT *******

THE FOLLOWING TABLE REPRESENTS PARALLEL LISTINGS OF THE UNKNOWN PATTERN WITH THE BEST FOUND IN THE REPORT

THE FOLLOWING RESULTS REPRESENT AN ATTEMPT TO SUBTRACT  STANDARD PHASES BASED ON INTENSITIES

THE PRECONCEIVED POSSIBILITIES WILL BE SUBSTRACTED FIRST IN THE ORDER GIVEN AS INPUT

THE STANDARD PATTERNS WILL BE SUBSTRACTED FROM THE UNKNOWN PATTERN IN ORDER OF DECREASING RELIABILITY FACTOR


THE  STANDARD WITH ASTM NUMBER   50592 HAS A SCALING FACTOR OF  0.807 AND A FORMULAE OF PB S                 **
THE  STANDARD WITH ASTM NUMBER   50490 HAS A SCALING FACTOR OF  0.703 AND A FORMULAE OF SI O2                **
THE  STANDARD WITH ASTM NUMBER   40864 HAS A SCALING FACTOR OF  0.293 AND A FORMULAE OF CA F2                **
THE  STANDARD WITH ASTM NUMBER   50566 HAS A SCALING FACTOR OF  0.481 AND A FORMULAE OF ZN S                 **
THE  STANDARD WITH ASTM NUMBER   50586 HAS A SCALING FACTOR OF  0.544 AND A FORMULAE OF CA C O3              **
THE  STANDARD WITH ASTM NUMBER  100434 HAS A SCALING FACTOR OF  0.093 AND A FORMULAE OF ZN S                  *
THE  STANDARD WITH ASTM NUMBER   50492 HAS A SCALING FACTOR OF  0.012 AND A FORMULAE OF ZN S                 **
THE  STANDARD WITH ASTM NUMBER  130141 HAS A SCALING FACTOR OF  0.184 AND A FORMULAE OF S                     *
THE  STANDARD WITH ASTM NUMBER   50565 HAS A SCALING FACTOR OF  0.0   AND A FORMULAE OF SI                    *
THE  STANDARD WITH ASTM NUMBER  180700 HAS A SCALING FACTOR OF  0.095 AND A FORMULAE OF PB2 SI S4              *


D(OBS)   I(OBS)    D(WINDOW)       D COMPONENTS
                 D(HI)   D(LO)

                                            ASTM   ASTM   ASTM   ASTM   ASTM   ASTM   ASTM   ASTM   ASTM   ASTM  RESIDUE INT.
                                           50592  50490  40864  50566  50586 100434  50492 130141  50565 180700


                                   6.845                                                      4
                                   6.645                                                      1
                                   5.631                                                                    0
                                   4.474                                                                    3
                                   4.415                                                      1
 4.2700   15   4.3011   4.2263                                                                                  -11
                                   4.264          27
                                   3.960                                                                    1
 3.8800    8   3.8835   3.8241                                                                                   -1
                                   3.854                               8
                                   3.795                                                      2
                                   3.738                                                      4
                                   3.431   80
 3.4300   63   3.4542   3.4072                                                                                  -17
```

Figure 8.- SUBSTI FINAL REPORT. (The 10 best possibilities are listed with their scaling factors. All the subtractions of SUBSTI are listed in parallel.)

				3.358										2	
3.3500	100	3.3727	3.3278												30
				3.339		70									
3.3200	29	3.3389	3.2949												10
				3.308							1				
				3.306										0	
				3.308						9					
				3.295								16			
				3.252										0	
3.1500	29	3.1658	3.1299												10
				3.150			29								
				3.140									0		
3.1340	29	3.1456	3.1104												-12
				3.130							1				
				3.130						5					
				3.120				48							
				3.101								1			
				3.063										0	
				3.044								2			
3.0390	100	3.0534	3.0166												43
				3.035					54						
				2.998								1			
2.9730	100	2.9895	2.9542												19
				2.972	80										
2.9310	25	2.9455	2.9112												5
				2.928							1				
				2.928						9					
				2.911										9	
				2.845					1						
				2.706				4							
				2.677										3	
				2.601								0			
				2.541										0	
2.4990	6	2.5044	2.4845												-2
				2.457					8						
				2.451								1			
				2.480								1			
				2.460		10									
2.4600	8	2.4722	2.4480												-4
				2.442								1			
2.2870	15	2.2962	2.2753												-13
				2.286					13						
				2.281		10									
				2.275						5					
				2.275							0				

Figure 8.- Continued.

```
                                  2.245                                                           0
                                  2.235           4
2.2340    3   2.2447    2.2247                                                                          0
                                  2.195                                                           0
                                  2.181                                         1
2.1300   10   2.1390    2.1205                                                                          3
                                  2.130           7
2.1010   63   2.1115    2.0942                                                                         -1
                                  2.099                                         0
                                  2.099     56
                                  2.095                                                           2
                                  2.094                       13
                                  2.014                                                           0
                                  1.978           4
                                  1.951                                                           0
1.9418    3   1.9512    1.9361                                                                          2
1.9325   25   1.9399    1.9249                                                                         -4
                                  1.932                 29
                                  1.932                                         0
                                  1.929                        3
                                  1.921                                               0
1.9141   34   1.9212    1.9066                                                                        -11
                                  1.914                       13
                                  1.910                    30
                                  1.910                                   1
                                  1.910                             9
                                  1.903                                                           0
                                  1.896                                         0
1.8777   15   1.8850    1.8709                                                                          0
                                  1.874                       13
                                  1.871                                         1
                                  1.857                                         1
1.8203   16   1.8265    1.8132                                                                         -7
                                  1.817          17
                                  1.800           0
1.7919   25   1.7969    1.7841                                                                         -5
                                  1.791     31
                                  1.787                                         1
                                  1.779                                                           0
1.7674   16   1.7740    1.7621                                                                          2
                                  1.765                             5
                                  1.765                                   0
                                  1.732                                                           1
1.7151   15   1.7197    1.7079                                                                         -3
                                  1.714     20
```

Figure 8.- Continued.

```
                                    1.708                                                        0
                                    1.675                                                        0
1.6734    6   1.6793   1.6681                                                                                           0
                                    1.671         7
                                    1.660         2
                                    1.654                                           1
                                    1.654                                                  0
1.6524    6   1.6570   1.6481                                                                                          -3
                                    1.646                    11
                                    1.636                                                                0
                                    1.635                                                        1
1.6326   25   1.6380   1.6273                                                                                           0
                                    1.633                           18
                                    1.630                                           5
                                    1.630                                                  0
                                    1.625                                    3
                                    1.605         0
                                    1.604                                    5
                                    1.599                                           2
                                    1.599                                                  0
                                    1.599                                                        0
                                    1.566                                    1
                                    1.566                                                        1
                                    1.564                                                  0
                                    1.561                            0
                                    1.542        10
1.5410   10   1.5468   1.5373                                                                                           0
                                    1.520                                    3
                                    1.519                                    3
                                    1.509                                    1
1.4860    6   1.4914   1.4826                                                                                          -1
                                    1.485  6
                                    1.474                                    1
                                    1.463                                                  0
                                    1.452                                                        4
                                    1.452         2

IF THE ABOVE RESULTS DO NOT RESULT IN 'AN IDENTIFICATION', IT IS SUGGESTED THAT THE PARAMETERS BE CHANGED AND THIS PHASE REPEATED

THE EXECUTE TIME FOR THIS RUN WAS      2632HUNDREDTHS OF SECOND(CPU TIME)

THE EXECUTE TIME FOR THIS RUN INCLUDING ALL I/O WAS      3058 HUNDREDTHS OF SECOND
```

Figure 8.- Concluded.

matches, intensity agreement, and spacing agreement. Experience has shown that a reliability factor greater than 33 and a log intensity match greater than 25 indicate a strong probability of the presence of that phase. If the input parameters are too restrictive (e.g., error window of 2 and log intensity match of 4 when the pattern has only 6 to 8 lines) and *no* possibilities match the input data, then an error message will be printed in this section. It can be identified by the statement "ABEND CODE IS: SYSTEM 0OC5."

(3) REPORT- The entire pattern for those compounds which are chemically correct is given (fig. 4). If no chemical information is provided, then the 10 compounds with the highest reliability factors will be printed. Each compound listed is headed by its ASTM number, reliability factor, formula, and to the right is a number denoting its subfile, as follows:

blank = An inorganic pattern
2 = Organic
3 = Dual
4 = Inorganic, mineral
5 = Greater than 10Å (organic)
6 = Organic, mineral
7 = Greater than 10Å (inorganic)
8 = Cross-indexed minerals
9 = Greater than 10Å (dual)

If chemical information is used as input but no chemical formula occurs on the ASTM card, then the compound will not be listed in REPORT, MATCHD, or SUBSTI. Thus, the OUTPUT section should be visually scanned for possibilities with high reliability factors and intensity matches, but without chemical formulae.

(4) MATCHD- The 10 best matches are listed in order of decreasing reliability (fig. 5). The numbers and asterisk to the right denote the subfile and data quality. Below this to the left, the unknown pattern, with intensities and error windows, is listed. The next column lists the d-spacings of the 10 best matches which are then listed to the right. Each possibility is headed by its ASTM number and reliability factor. Under the heading are the I_3 intensities (grouped according to the I_1 scale) which correspond to the standard d-spacings to their left.

(5) SUBSTI- This section attempts to subtract standard phases from the unknown pattern, one at a time, based on intensities. This results in a final residual pattern. Intensities of the unknown are converted to the same log scale as the standards (figs. 6 and 7) before execution. SUBSTI FINAL REPORT (fig. 8) lists d-spacings and scaled intensities of all the standard together.

A scaling factor is used to scale down the intensities of the standards to those of the unknown. This is calculated as

$$\text{Scaling factor} = \frac{\sum I_{\text{unknown}}}{\sum I_{\text{standard}}}$$

where $I_{unknown}$ are the intensities of the unknown pattern whose d-spacings match those of the standard to which it is compared. These values may be negative if already subtracted by a previous standard with a higher degree of confidence. It is here that a preconceived possibility is useful. A known phase will be subtracted first, scaling down any later possible phases to insignificance if their identification relied on lines belonging to the known phase. SUBSTI FINAL REPORT has the same format as MATCHD, except that now scaling factors have been calculated, standard intensities have been scaled down, and each standard has been subtracted from the unknown pattern.

III. PROGRAM INPUTS AND OPERATION

The Johnson-Vand program is simple to operate. A minimum of 8 data cards (described below) are placed inside a deck of 15 job cards. The data follow the first 14 job cards and the final job card is placed at the back of the deck. The deck is then given a job header card (see fig. 9). Table 3 describes the data card formats.

TABLE 3.- DATA CARD FORMATS

DATA CARD 1	This card may contain any information (not beyond column 76) to *identify* the run.
DATA CARD 2	The parameter card. The program will supply a reasonable set of parameters, default parameters if a value of zero or blank is given. The parameters are:
Col. 1(A)	*IFL*, the subfile of the ASTM powder diffraction file to be searched. Set IFL as follows: blank = all subfiles A = alloy O = organic I = inorganic D = dual (e.g., both inorganic and organic) default = blank (entire file) M = mineral
Col. 3(I)	*RESOL*, resolution condition 0 = normal 1 = Guinier default = 0
Col. 5-8(I)	*MM*, the number of lines in unknown pattern default = computer will count number of peaks

TABLE 3.- Continued.

Col. 9-12(I)	*IW*, the error window default = 2
Col. 13-16(I)	*KUTØFF*, the minimum acceptable value of log intensity match default = 2
Col. 17-20(I)	*NUMBER*, the minimum acceptable number of line matches default = 3
Col. 25-28(I)	*IPERCT*, the minimum acceptable number of line matches default = 33
Col. 29-32(I)	*ICYCLE*, the number of cycles default = 0
Col. 33-36(I)	*IWCY*, the increase of IW per cycle default = +1
Col. 37-40(I)	*KUTOCY*, the increase of KUTØFF per cycle default = +5
Col. 41-44(I)	*NUMCY*, the increase of NUMBER per cycle default = +1
Col. 45-48(I)	*IPERCY*, the increase of IPERCT per cycle default = +10
Col. 49-52(I)	*JBKGR*, the background intensity (0 to 100 scale) default = +1 or half lowest given intensity
Col. 53-60(F)	*DHI*, the upper limit of the d-spacing range observed in the diffraction pattern default = error window above largest measured d
Col. 61-68(F)	*DLO*, the lower limit of the d-spacing *range* observed default = error window below smallest measured d

TABLE 3.- Continued.

Col. 69-73(A)	OPTION SOLID = Solid solution MINOR = Minor phase MAJOR = Major component default = Major
DATA CARD 3	The positive element and preconceived possibilities card. *Chemical elements* on this card and on Card 4 must be right justified, two columns per element; that is, single letter elements must be punched with a blank, then the letter: for example, a list of sodium, hydrogen, lithium, iron, nickel, oxygen, and platinum would be punched NA HLIFENI OPT With the first N in column 1 and none beyond column 50. RE (Rare Earth) punched in columns 49 and 50 will cause all the rare earth elements from La to Lu, inclusive, to be used as positive elements, in addition to any others punched in columns 1 through 48. The letters TU (transuranium) punched in columns 49 and 50 of the position element card will cause all the elements from Ac to Fm to be included automatically in the positive element list. A Q in column 1 calls for the periodic table. *Preconceived possibilities*- The ASTM card numbers patterns which are to be printed out even though not found by the search program. Each must consist of six digits with no spaces. (A maximum of five patterns can be listed as preconceived possibilities. As an example, if patterns 1-24, 6-432, 11-4, 12-100, and 16-432, were known to be present, then the card would contain 010024060432110004120100160432 (starting in column 51). In other words, the six numbers are divided into a two-digit field for the card set and a four-digit field for the card in that set. The minimum contents for this card is 000000.)
DATA CARD 4	The negative element card. These must be punched right justified, two columns per element as in Card 3 with none beyond column 80. A Q punched in column 1 will cause all elements (through Fermium) not specifically designated as positive elements to be used as negative elements.
DATA CARD 5	Positive Functional Groups. Those functional groups which are known to be *present* in the unknown (from spot or other chemical tests).

TABLE 3.- Concluded.

DATA CARD 6	Negative Functional Groups. Those functional groups which are known to be *absent* in the unknown (from spot or other chemical tests). NOTE: The input for both these cards are as follows: Punch chemical group exactly as the group appears in figure 3 (***OUTPUT***) under FORMULA. Notice the spaces - this is extremely important. Eight groups can be external, starting in columns 1, 11, 21, etc.
DATA CARD 7 thru N	These cards contain the pairs of d-spacings and intensities measured on the unknown pattern. These need not be in order. Punch the d-spacing, then its intensity (scale 1-100). The d-spacing *must* contain a decimal point and the intensity *must not* contain a decimal point. These cards are of the format (8(F7.3,I3)). *Then one blank card.* Cards 1-N may be repeated as many times as unknown diffraction patterns are available.

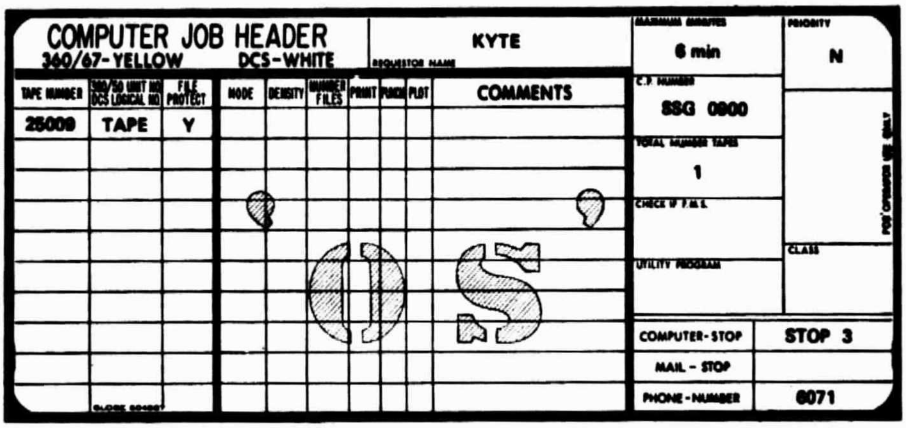
A printout can be obtained with only a data set of spacings and intensities (Card 7-N) along with 7 blank cards in the proper places. The program provides a reasonable set of parameters (by default) if none are input.

On Card 2, all numerical parameters should be right justified in their fields. For example, an error window (IW) of 3 which has a field of columns 9-12 should be punched into column 12. Remember that a zero on this card is read as a blank and a default parameter will be used. It is impossible to give a parameter a value of zero.

On Cards 7-N, the diffraction data cards, the format is (8(F7.3,I3)). That is, for each card which has 80 columns, there are 8 fields of 10 columns each for paired spacings and intensities. Each of these 10 column fields is divided into a 7- and a 3-column field for the spacing and intensity, respectively. In the 7-column field, if no decimal is used, all blanks are counted as zeros and a decimal is placed automatically before the three right-hand digits. If a decimal is used, the spacing value may be placed anywhere within this field. The 3-column intensity field *must* be occupied by an integer which *must* be right justified. All blanks in this field are read as zeros. At least one of the intensities *must* equal 100.

All data cards 1-N must be followed by a blank card to end the search.

A typical run would probably use the mineral subfile. The error window would be defaulted to 2, or set at 3. All other parameters would probably be defaulted except perhaps the log intensity match, which might be set at 4 (or

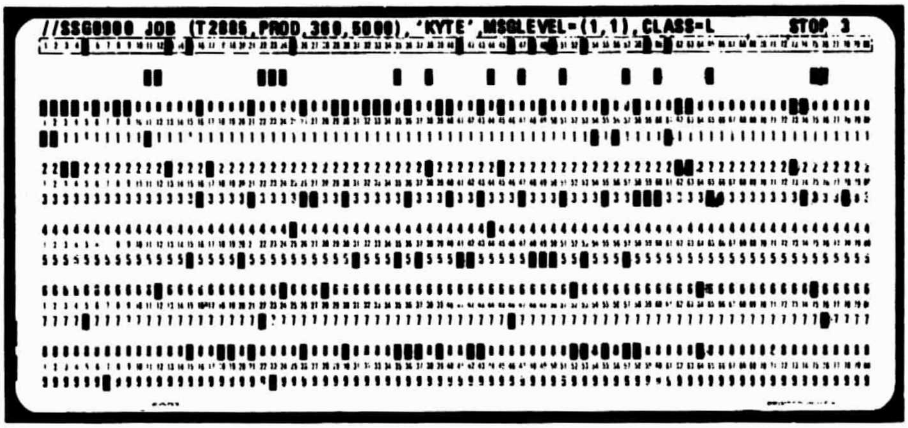


No. 1
JOB HEADER CARD

COMPUTER JOB HEADER
360/67-YELLOW DCS-WHITE

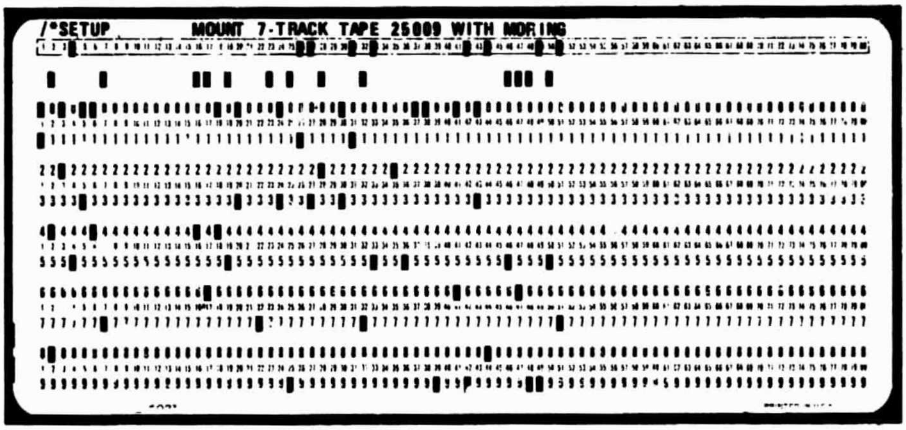
REQUESTOR NAME: KYTE
MAXIMUM MINUTES: 6 min
PRIORITY: N
C.P. NUMBER: SSG 0900
TOTAL NUMBER TAPES: 1
CHECK IF F.M.S.:
UTILITY PROGRAM:
CLASS:
FOR OPERATOR USE ONLY
COMPUTER-STOP: STOP 3
MAIL - STOP:
PHONE-NUMBER: 6071

TAPE NUMBER	360/50 UNIT NO DCS LOGICAL NO	FILE PROTECT	MODE	DENSITY	NUMBER FILES	PRINT	PUNCH	PLOT	COMMENTS
25009	TAPE	Y							



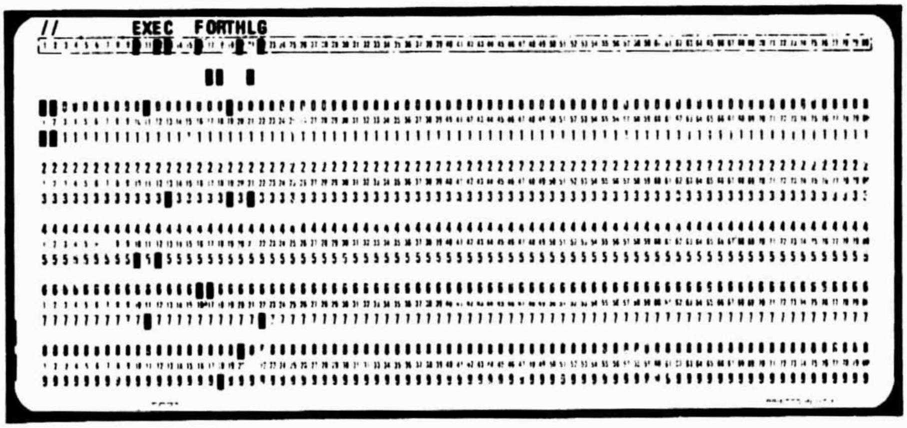
No. 2
JOB CARD-1

```
//SSG0900 JOB (T2885,PROD,360,5000),'KYTE',MSGLEVEL=(1,1),CLASS=L       STOP 3
```



No. 3
JOB CARD-2

```
/*SETUP          MOUNT 7-TRACK TAPE 25009 WITH NORING
```

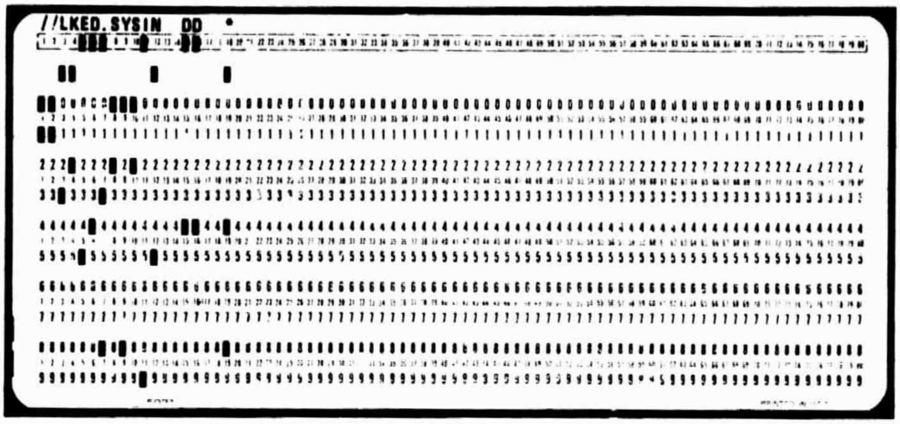


No. 4
JOB CARD-3

```
//        EXEC  FORTHLG
```

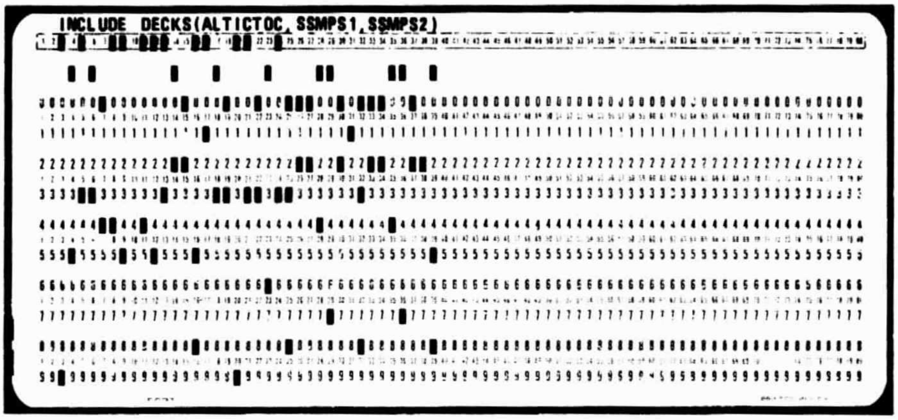
Figure 9.- Illustration of input cards.

No. 5
JOB CARD-4



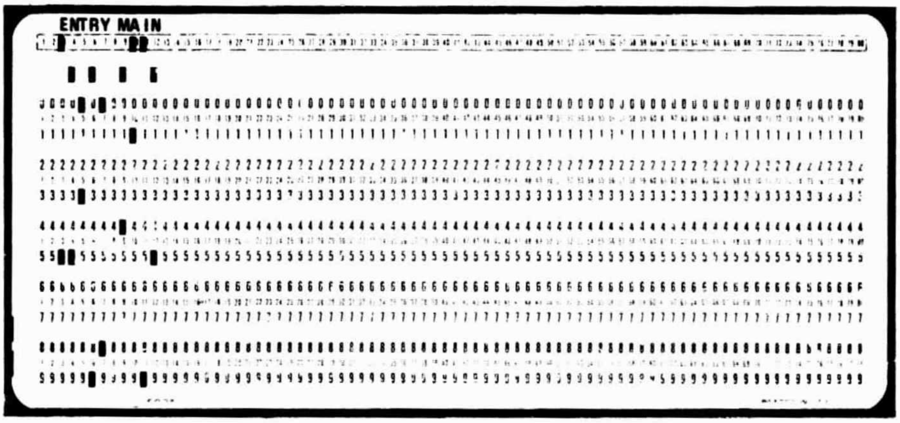
```
//LKED.SYSIN DD *
```

No. 6
JOB CARD-5



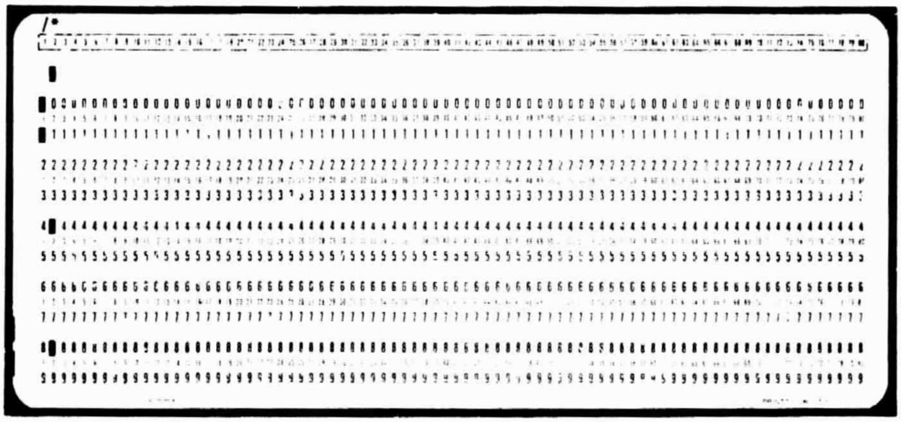
```
 INCLUDE DECKS(ALTICTOC,SSMPS1,SSMPS2)
```

No. 7
JOB CARD-6



```
 ENTRY MAIN
```

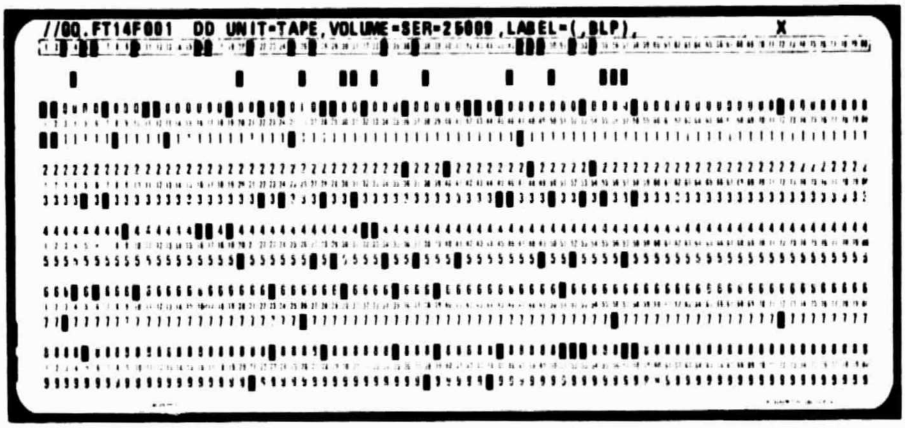
No. 8
JOB CARD-7



```
/*
```

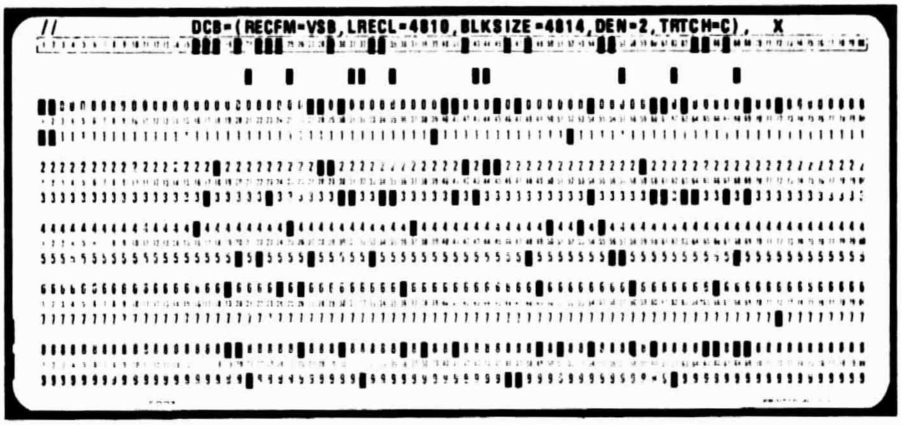
Figure 9.- Continued.

No. 9
JOB CARD-8



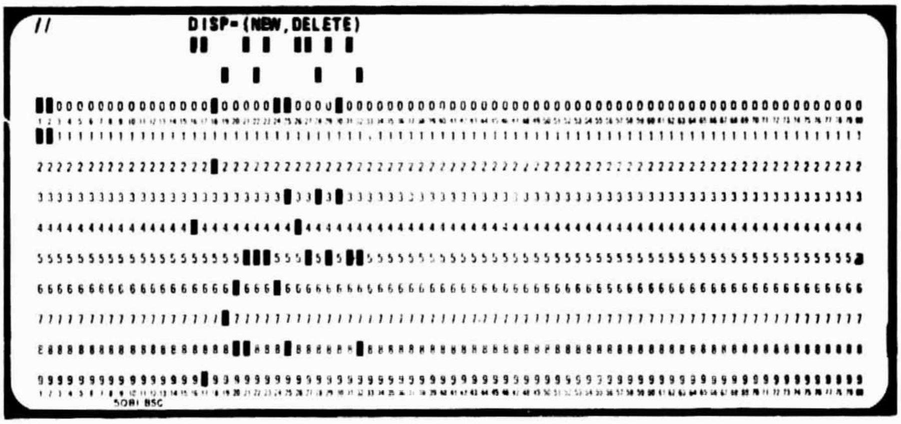
```
//GO.FT14F001  DD UNIT=TAPE,VOLUME=SER=25009,LABEL=(,BLP),               X
```

No. 10
JOB CARD-9



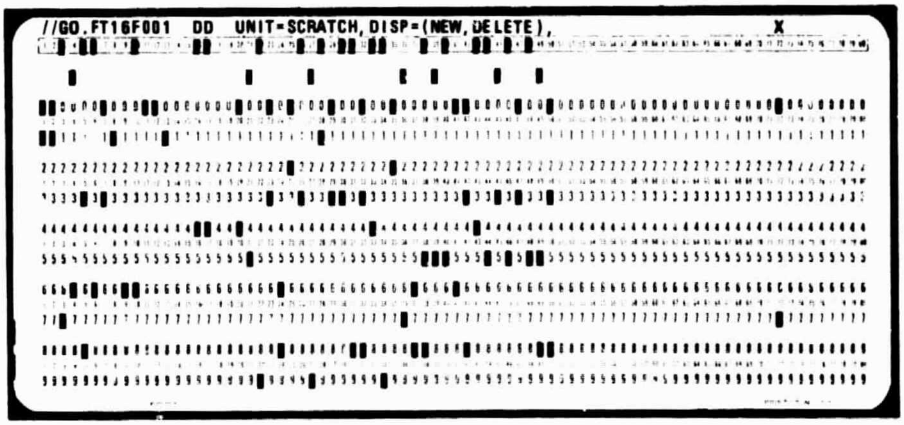
```
//             DCB=(RECFM=VSB,LRECL=4810,BLKSIZE=4814,DEN=2,TRTCH=C),   X
```

No. 11
JOB CARD-10



```
//             DISP=(NEW,DELETE)
```

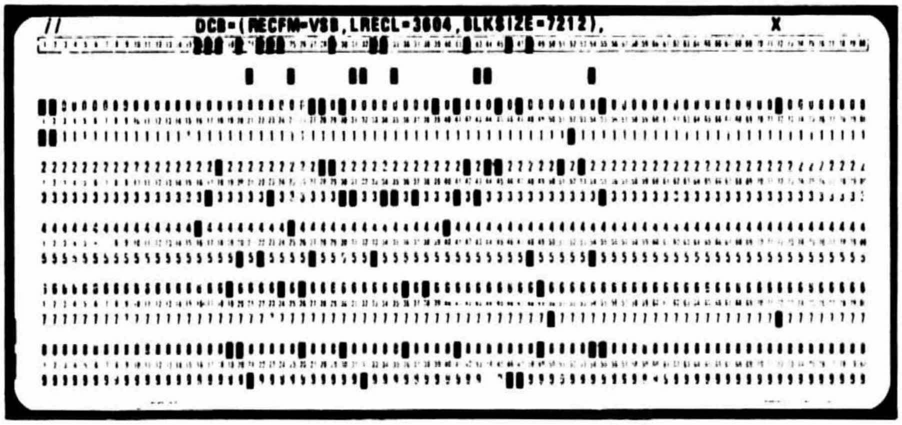
No. 12
JOB CARD-11



```
//GO.FT16F001  DD   UNIT=SCRATCH,DISP=(NEW,DELETE),                     X
```

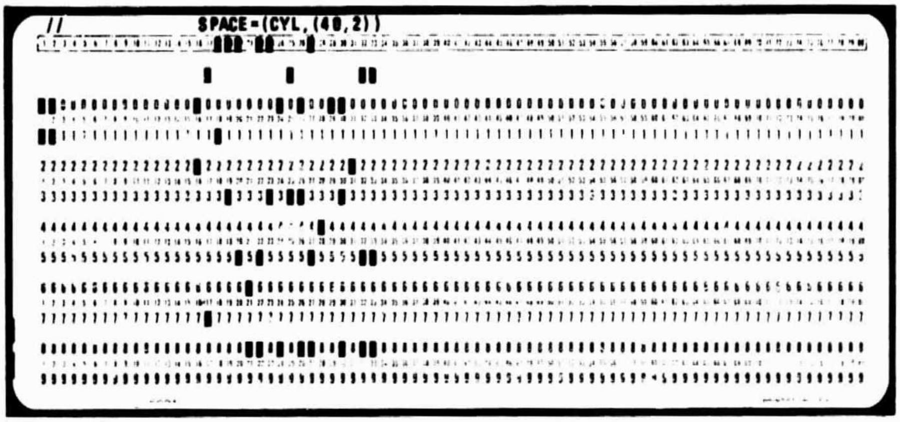
Figure 9.- Continued.

No. 13
JOB CARD-12



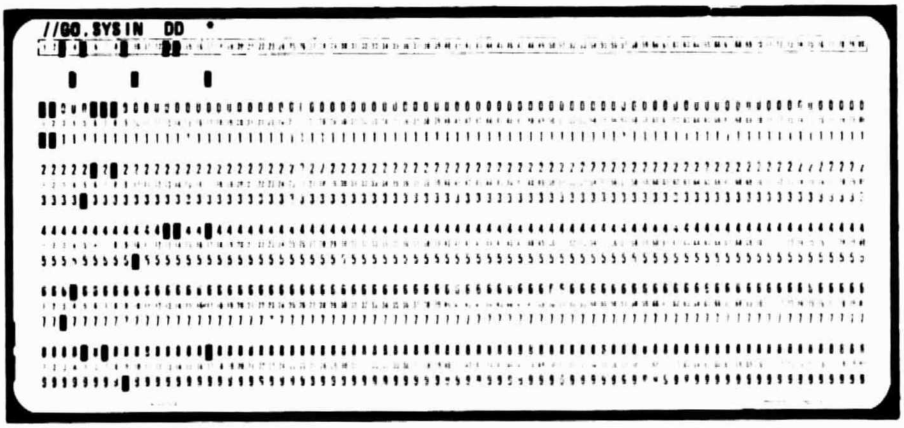
```
//            DCB=(RECFM=VSB,LRECL=3604,BLKSIZE=7212),                    X
```

No. 14
JOB CARD-13



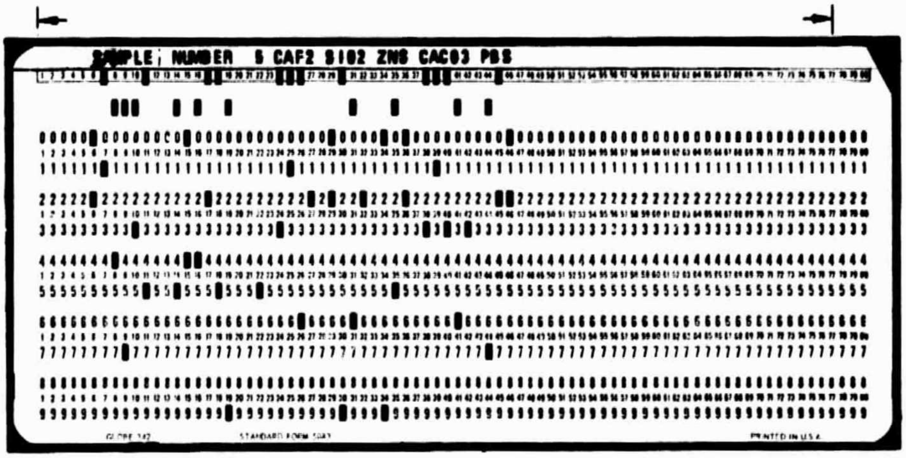
```
//            SPACE=(CYL,(40,2))
```

No. 15
JOB CARD-14

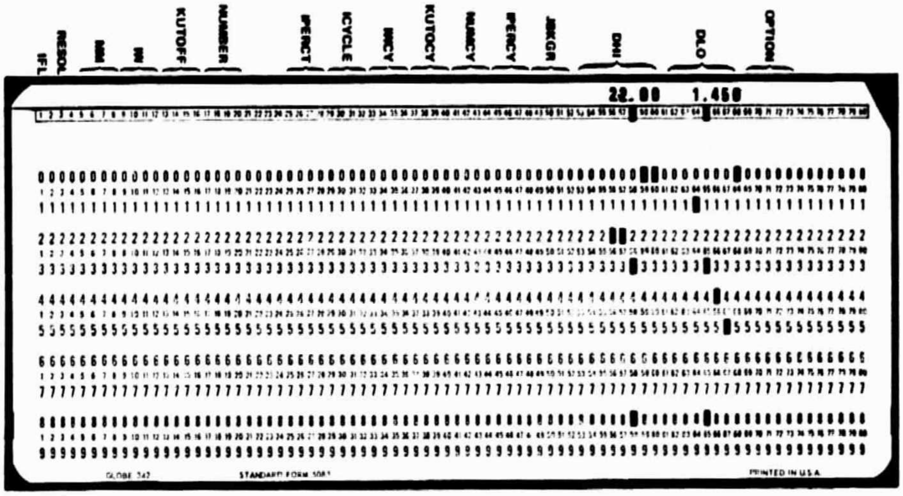


```
//GO.SYSIN  DD  *
```

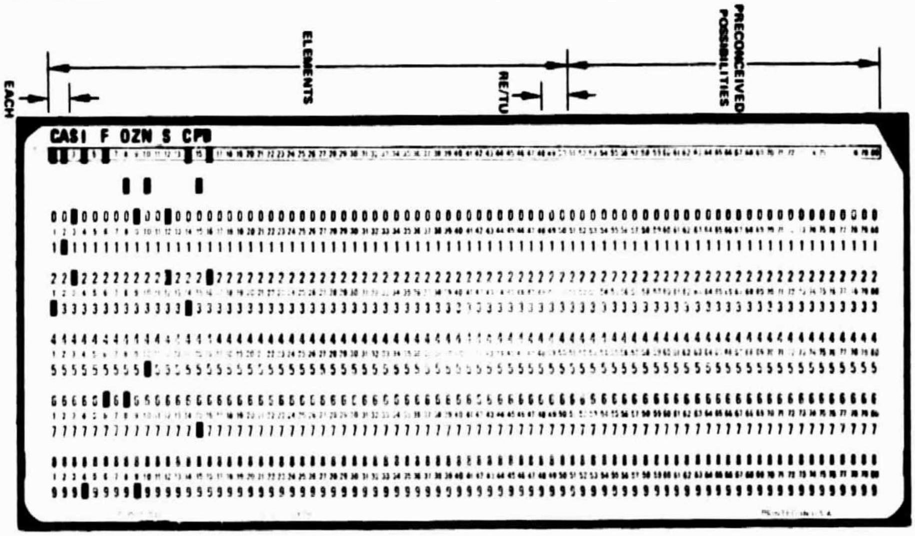
Figure 9.- Continued.



No. 16
DATA CARD (TITLE)-1

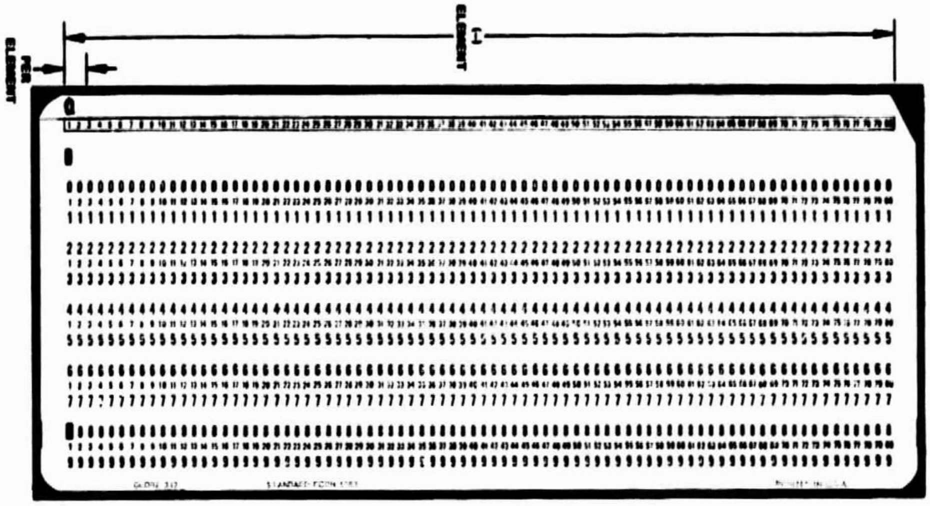


No. 17
DATA CARD (PARAMETERS)-2

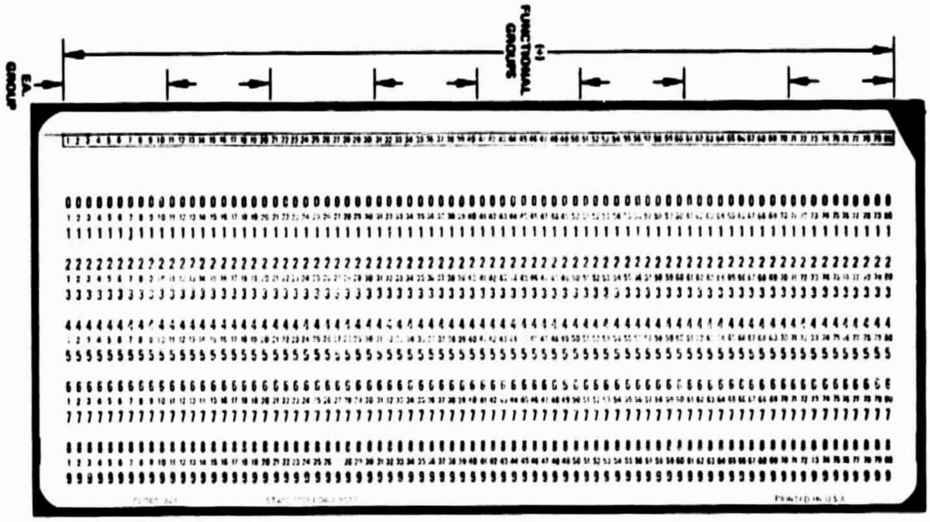


No. 18
DATA CARD (POSITIVE ELEMENTS)-3

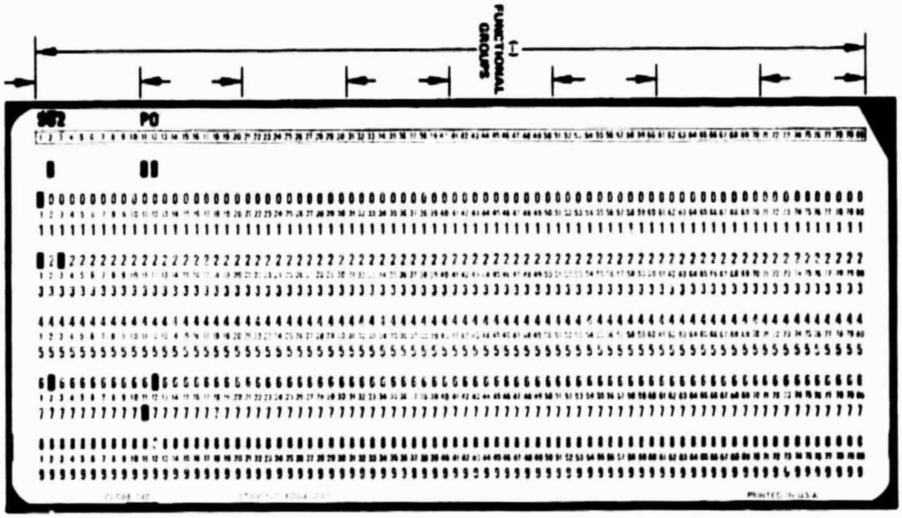
Figure 9.- Continued.



No. 19
DATA CARD (NEGATIVE ELEMENTS)-4

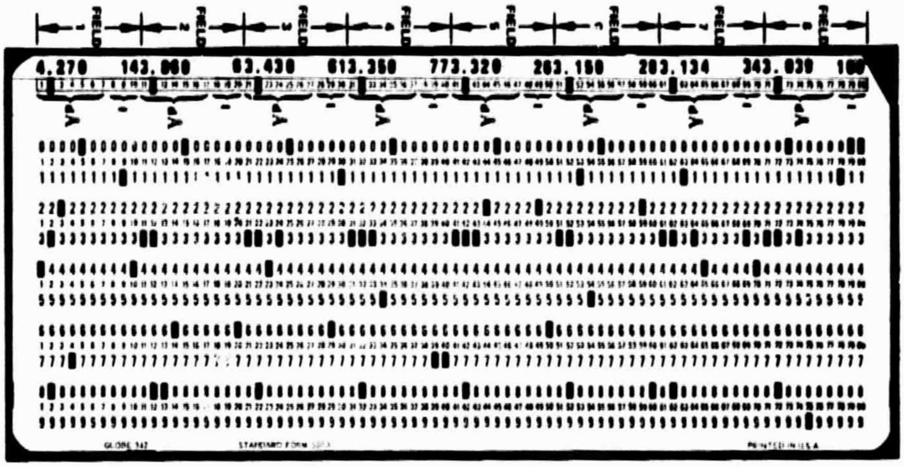


No. 20
DATA CARD (POSITIVE FUNCTIONAL GROUPS)-5

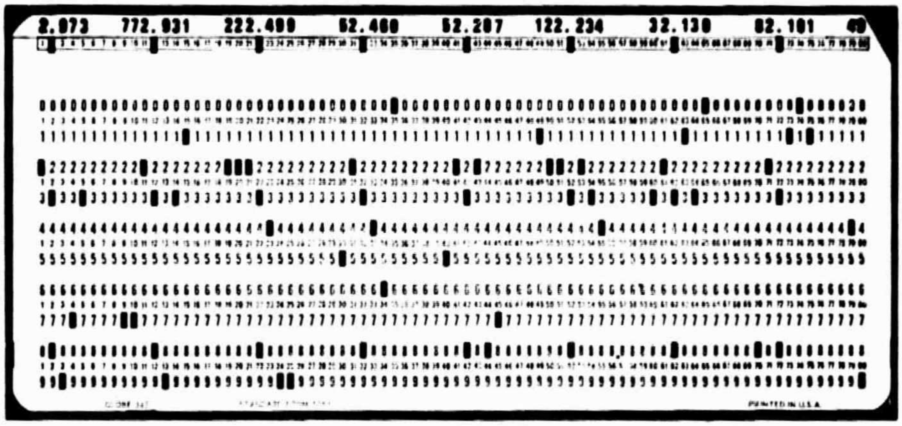


No. 21
DATA CARD (NEGATIVE FUNCTIONAL GROUPS)-6

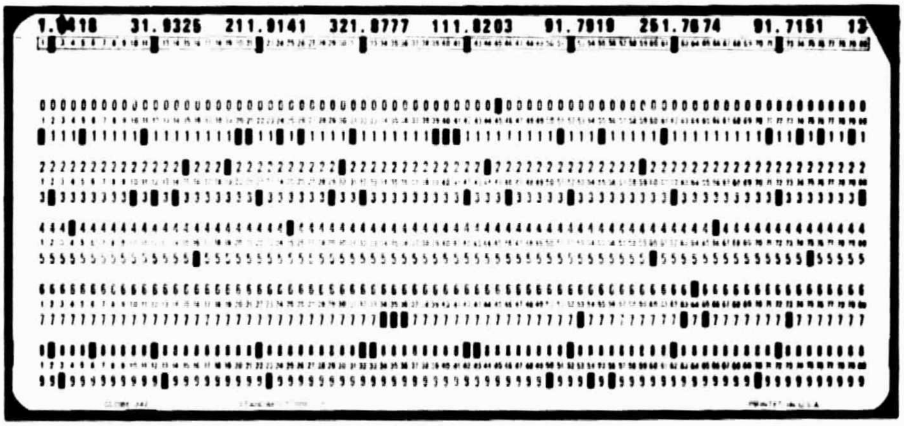
Figure 9.- Continued.



No. 22
DATA CARD (d SPACINGS & INTENSITIES)-7



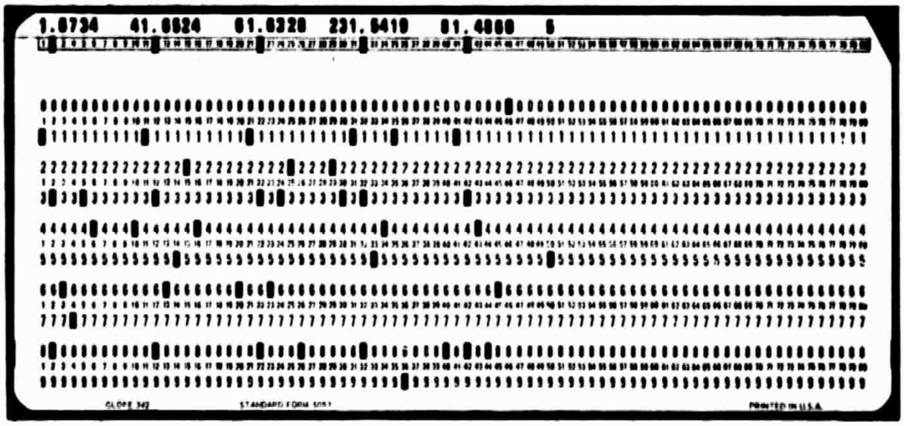
No. 23
DATA CARD (d SPACINGS & INTNESITIES)-8



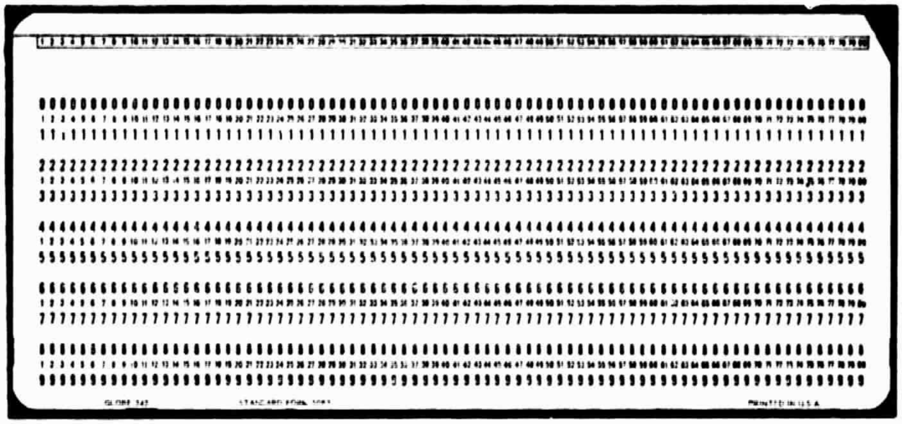
No. 24
DATA CARD (d SPACINGS & INTENSITIES)-9

Figure 9.- Continued.

No. 25
DATA CARD (d SPACINGS & INTENSITIES)-10



No. 26
DATA CARD (BLANK)-11



No. 27
JOB CARD (FINAL)-14

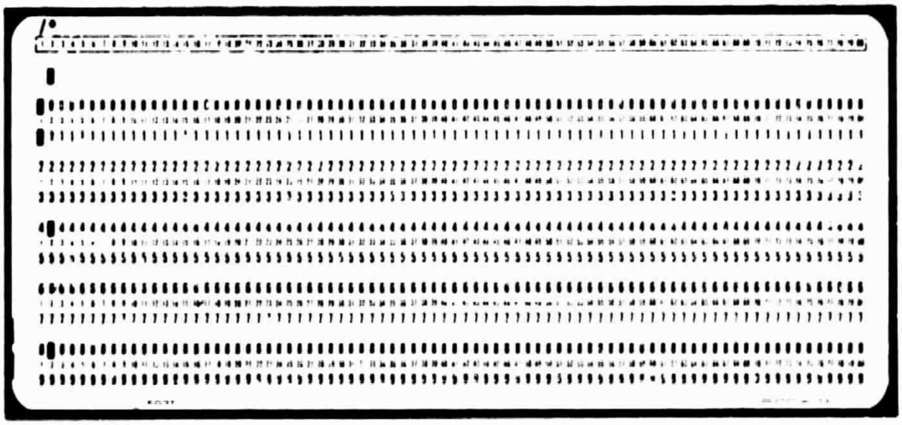


Figure 9.- Concluded.

7 for a good diffractometer scan). If an identification did not result, data should be rechecked and the error window should be expanded and other parameters increased by small amounts to limit the amount of output. With experience, an operator should get a feel for what the capabilities of the system are.

IV. A WORKED EXAMPLE

A set of worked examples has been prepared. The reader is referred to Appendix I which is a package of computer printouts. This package is available on loan from Max Blanchard at Ames Research Center or Dr. Marshall E. Maddock at San Jose State University. Appendix I comprises nine printouts obtained from Johnson and Vand's example (figs. 2-8) and from two mixtures of minerals (Tables 1 and 2) which were X-rayed with the Debeye-Scherrer powder cameras (57.3- and 114.7-mm diameters) and the X-ray diffractometer (at different scan speeds).

A. Sample Number 5 (CaF_2, SiO_2, ZnS, $CaCO_3$, PbS

This is the entire output of the Johnson and Vand example. The entire inorganic file was searched. Chemical information was used. The only input parameters were the range of observed d-spacings. The effect of using chemical information can easily be appreciated here as it reduced a total of 170 possibilities to only 11. From there, it easily identified the five phases present.

B. ASTM 2, 2A, 5, and 4F

These four printouts were obtained from a mixture of approximately equal amounts of PbS, CaF_2, TiO_2, $(Mg,Fe)_2SiO_4$, and $Fe(Cr,Al)_2O_4$. Unlike the Johnson and Vand example, two minerals in this mixture are subject to considerable solid solution and standard patterns in the Powder Diffraction File, for these minerals represent only points along a solid-solution sequence. Thus, standard intensities and d-spacings could vary significantly from those observed in an unknown sample.

Data for ASTM 2 were obtained from a large Debeye-Scherrer powder camera. All of the parameters were defaulted and the mineral subfile was searched. CaF_2 and PbS were easily identified in OUTPUT with high reliabilities and intensity matches. TiO_2 had the next highest reliability but a low-intensity match. $(Mg,Fe)_2SiO_4$ and $Fe(Cr,Al)_2O_4$ were not identified because their strong lines at 2.27Å and 2.068Å, respectively, were not measured. Inspection of the X-ray film confirmed their existence but their intensities were very weak and spotty. The data were not good enough to identify these phases. In SUBSTI FINAL REPORT, cordierite and montmorillonite were listed as good possibilities, as well as the other three phases. Here, chemical information would have been helpful in determining the actual phases present. The program was, however, successful, using default parameters, in searching the approximately 2000 mineral subfile and listed five good possibilities without the use of chemical information. Three of these five were correct.

Data for ASTM 2A were obtained from a short exposure on a small Debeye-Scherrer camera. All the parameters were defaulted and the mineral subfile was searched. None of the possibilities listed had confidence levels high enough to be considered as an identification. It is assumed that the exposure time was too short to darken the film and that too few diffraction lines were measured for an identification. Thus another, longer, exposure was taken and named ASTM 5.

Output for ASTM 5 was from data for 28 lines, whereas ASTM 2A had only 17 lines for the same range of d-spacings. The data were run at an error window of ±3, and only the mineral subfile was searched. Results were comparable to those on the large camera. TiO_2, PbS, and CaF_2 were easily identified, but the two components with solid solution were missed again. Attempts at broadening the error window were futile because too many possibilities were selected. In general, a broad window of ±4 is suitable only for data with chemical information.

Data for printout ASTM 4F was obtained from a slow diffractometer scan at 1/4° 2θ/min. This was much more successful than with the powder cameras. This is attributed to the diffractometer's ability to resolve weak lines and the greater accuracy of line location and peak height which are inherently better on diffraction charts. These data were processed with default parameters and only the mineral subfile was searched. Without chemical information only 20 possibilities were listed in OUTPUT. Six of the 20 represented the 5 known mineral phases and 2 others (spinels) were isostructural with the chromite. SUBSTI FINAL REPORT lists all except olivine as having a high probability, and the only incorrect phase listed there is BeO, an unlikely component.

Observation of the log-intensity matches listed led to the conclusion that the output could be improved if this parameter were increased. Another computer run was made with a log-intensity match of 4. This is ASTM 4FA. A brief glance at OUTPUT and SUBSTI FINAL REPORT indicates an excellent match of all phases except olivine, with the only other phases listed being BeO and $NiFe_2O_4$, again, a spinel isostructural with chromite.

C. BSTM 1 and 2

Another mixture, this time composed of equal weights of rutile (TiO_2), galena (PbS), zircon ($ZrSiO_4$), Flourite (CaF_2), and calcite ($CaCO_3$) was prepared. Of these, only the calcite exhibits solid solution with the replacement of Ca by Mg, and it was a relatively Ca-pure sample. Two diffractometer scans were run at 1/4° 2θ/min and at 1° 2θ/min and labeled BSTM 1 and BSTM 2, respectively.

Both data sets were recorded in two ways. One was to read directly off the diffraction charts (BSTM 1 and 2) and the other (BSTM 1A and 2A) was to subtract 0.05° θ to compensate for misalignment of the goniometer. (Note: 0.05° θ is equivalent to about 1d*.) These four data sets were then input with default parameters except for log-intensity match at 4 and DHI at 8.845Å corresponding to an angle of 10° 2θ. Background radiation was read directly off the charts and only the mineral subfile was searched.

BSTM 1 at 1/4° 2θ/min had an output of only 10 possibilities uncorrected and 13 when corrected by 0.05° θ. In both cases, all five known phases were identified. The corrected output had higher reliability factors in every case except zircon, which matched with 11 lines instead of 12 after being corrected.

In both cases, SUBSTI FINAL REPORT listed the five known phases as the only ones with scaling factors above 0.1, the lowest of these being calcite at 0.206. The remaining possibilities had been scaled down so low because their identifications had been dependent on lines generated by those phases with higher reliabilities. When it came time to calculate a scaling factor for these incorrect phases, there was little or no pattern left for them to compare to.

BSTM 2 at 1° 2θ/min uncorrected had an output of eight possibilities, correctly identifying every phase except calcite. The corrected data set listed only 10 possibilities, including calcite, and reliability factors for the known phases were greatly enhanced. Once again, in both cases, the known phases were the only ones listed with scaling factors greater than 0.1 and a low factor for calcite at 0.189. Some very successful runs indeed!

IV. SUMMARY AND SUGGESTIONS

It has been demonstrated that this program can easily identify mixtures of minerals which can be quite difficult to resolve manually. There is still some difficulty in identifying phases which are subject to solid solution, it is hoped that when the master file is updated from Set 19 to Set 24 this will be less of a problem. The update will nearly double the data file and this should be accomplished in the near future. When a number of spacings have not been identified, solid solution should be suspected. The best way to resolve this would be to use chemical information, a wider error window (perhaps ±4), and the OPTION parameter. Remember, though, that it is possible that a phase is not identified because its pattern is not in the master file.

When inputting data, default parameters should be sufficient in most cases. An error window of ±3 might be needed when there is a poor resolution of lines. With confidence in intensity measurements a log-intensity match of 4 will greatly reduce the number of possibilities, requiring that standard intensities down to 26 must match with lines of the unknown pattern. A second cycle is usually not desirable!

Remember to input the entire range of d-spacings, especially DHI. When inputting intensities, keep in mind the log scale the program uses. Note that, in the worked examples, all intensities are input on this scale. Remember, at least one intensity *must* equal 100.

As an operator uses the program more often, he will become more familiar with its capabilities. He should always keep in mind, however, that the program merely lists possibilities with different degrees of confidence. The worked examples presented were quite successful; however, at other times incorrect identifications with high reliabilities have been observed. In the end, it is the responsibility of the operator, *not* the computer, to decide which phases are actually present.