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INDEXING REFLECTIONS OBTAINED WITH A FOUR-CIRCLE DIFFRACTOMETER

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ABSTRACT

A procedure for indexing reflections obtained when setting up a crystal on a four-circle diffractometer is described, together with a computer program which has been written for implementing part of the procedure.

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The following descriptors have been selected from the INIS Thesaurus to describe the subject content of this report for information retrieval purposes. For further details please refer to IAEA-INIS-12 (INIS: Manual for Indexing) and IAEA-INIS-13 (INIS: Thesaurus) published in Vienna by the International Atomic Energy Agency.

CRYSTALLOGRAPHY; CRYSTAL LATTICES; COMPUTER CODES; X-RAY DIFFRACTOMETERS; NEUTRON DIFFRACTOMETERS; CRYSTALS; NEUTRON DIFFRACTION; X-RAY DIFFRACTION;

CONTENTS

1. INTRODUCTION

After obtaining a suitable crystal, the first step in the determination of its crystal structure with a computer-controlled four-circle diffractometer is to find and identify a number of reflections. Once this has been done, existing computer programs can be used to determine the UB matrix, which is needed by the control computer for calculating the axial angular settings that will enable the intensities of other reflections to be measured. Here 'find' means determine the angular settings of the four axes of the diffractometer that will result in the observation of a reflection. This is done by trial and error, guided perhaps by the morphology of the crystal.

This report is concerned with identification of the reflections once they have been found; primarily it is addressed to students and research workers using the computer-controlled neutron and X-ray diffraction facilities at Lucas Heights. Some familiarity with the reciprocal lattice and the construction and principle of operation of a four-circle diffractometer is assumed; suitable introductions to these subjects can be found in Cullity [1965] and Ellis and Pryor [1968].

Note that the axial definitions of Ellis and Pryor used here are not the same as those described in the International Tables for X-ray Crystallography [Hamilton 1974].

2. USE OF STEREOGRAPHIC PROJECTION

In many cases, there will be prior knowledge of the unit cell of the crystal being investigated. When this so, it should be possible to assign a consistent set of indices to the observed reflections by inspecting a stereographic projection of the normals to the reflecting planes, utilising the relations observed between plane normals. The procedure for drawing a stereographic projection is illustrated in Figures 1(a) and 1(b).

In Figure 1(a), V represents the vertical direction in laboratory space and X and D the directions of the incident and diffracted beams, respectively, for a right-handed diffractometer. N is the normal to the reflecting planes. The angle $(X \wedge D) = 2\,\theta_B$, where θ_B is the Bragg angle for the reflection concerned, and $(D \wedge N) = 90 - \theta_B$. This serves to locate N in laboratory space. Figure 1(b) shows how N can be related to arbitrary axes fixed in the crystal. The arbitrary axes chosen are the directions parallel to V, X and the mutually perpendicular direction, T, when the crystal is oriented such that $\omega = \chi = \phi = 0$; ω, χ and ϕ are, respectively, the angular settings of axes 2, 3 and 4 of the four-circle diffractometer.

The crystal can be brought into this orientation by performing the following sequence of operations:

(a) Rotate through ω about the vertical axis. N is brought to N'; the axis of rotation of the χ-circle is then parallel to the incident beam direction, X.

(b) Rotate through χ about X. N' moves on a small circle to N". χ is the angle between great circles passing through X and N' or N". If χ is negative, as is commonly the case, the sense of rotation will be as shown in Figure 1(b). After this rotation, the ϕ axis will be parallel to V.

(c) Rotate through $-\phi$ about the vertical axis. N" moves on a small circle to N"', which is the orientation of the reflecting plane normal when the crystal is in standard orientation, i.e. with $\omega = \chi = \phi = 0$.

For a left-handed diffractometer, the positive senses of 2θ, ω and ϕ are reversed, but the sense of χ is unaltered. Of the four diffractometers in use at the AAEC Research Establishment, the X-ray and 2TanB diffractometers are right-handed instruments and 6HA and 2TanA are left-handed.

If the parameters of the unit cell are known, it will generally be possible to determine the form of each reflection {h k l}, by comparing the observed interplanar spacing, d, with the values calculated from the unit cell parameters. It then only remains to assign a consistent set of specific indices, (h k l), to the reflections; this can be done by noting zone relations between the reflecting plane normals and using the zone law.

Even if the unit cell is not known, it is still possible to assign a consistent set of indices to the reflections by use of the zone law, but this involves an arbitrary choice of indices for some of the reflections, which is equivalent to an arbitrary choice of unit cell. There can be no guarantee that a cell chosen in this way possesses the full symmetry of the lattice or, if it does, that it is the smallest cell with that symmetry; in such cases, it is safer to work with the reciprocal lattice. An example of two sets of indices that could be assigned to the same set of reflections is shown in Figures 2(a) and 2(b). It is not immediately apparent that the unit cell of Figure 2(b) is monoclinic whereas that of Figure 2(a) is only triclinic.

3. USE OF RECIPROCAL LATTICE

A stereographic projection does not make full use of the information available since it records only the directions of the reflecting plane normals and not the d spacings of the planes. All the available information is, however, recorded in the reciprocal lattice. The coordinates of a point in reciprocal space can be obtained by determining the direction cosines, a_1, a_2 and a_3, of the reflecting plane normal relative to an arbitrary set of orthogonal axes, e.g. V, X and T, and multiplying them by the length of the reciprocal lattice vector. The direction cosines can be either measured on a stereographic projection or calculated from the observed axial angles.

However, it is not always clear how to interpret the coordinates. The basic problem is that the reciprocal lattice is a three-dimensional structure and spatial relations between the reciprocal lattice points may not be apparent in a projection onto an arbitrary plane. The problem can be simplified by choosing as the plane of projection a plane containing a significant number of reciprocal lattice points, i.e. a significant number of reflecting plane normals. Hopefully, the remaining points will then be seen to lie on a small number of planes parallel to the first plane and a suitable choice of unit cell may become obvious. This is illustrated in Figures 3(a) and 3(b), which show the reciprocal lattice of the data points of Figures 2(a) and 2(b) projected onto (a) the plane containing X and T and (b) the plane containing the reciprocal lattice points indexed as 1 0 0 and 0 0 1 in Figure 2(b). The coordinates normal to the plane of projection are marked beside each point. Note that if two or more orders of reflection have been observed from the same set of lattice planes, there will be two or more reciprocal lattice points corresponding to a single plane normal in the stereographic projection.

It is possible to discern a pattern in Figure 3(a) but it is very much clearer in Figure 3(b). Furthermore, it is apparent that in Figure 3(b) there are prominent reciprocal lattice rows normal to the plane of projection, which itself contains a number of prominent lattice rows, and therefore it is possible to choose a unit cell in which two of the interaxial angles are 90°. One way in which this can be done is indicated in Figure 3(b). This unit cell corresponds to the indices of Figure 2(b).

When Figures 2(a) and 2(b) were plotted, the plane normals for reflections with $X > 0$ were reversed so that they lay in the half-space above the plane of projection, to enable zone relations to be seen more easily. This reversal must be borne in mind when assigning indices to the reflections actually observed, but it is of no other significance since observation of a reflection implies the existence of its inverse; in other words, the reciprocal lattice possesses a centre of inversion symmetry at the origin. The corresponding reciprocal lattice points in Figures 3(a) and 3(b) have not been reversed as can be seen from the negative heights above the plane of projection in Figure 3(a). This explains an apparent lack of consistency between Figures 2(a,b) and 3(a,b). By taking advantage of the symmetry of the reciprocal lattice, each point could be plotted at its inversion position as well; in some cases this might make the pattern of the reciprocal lattice more apparent.

It should be noted that the symmetry of the lattice is seen more easily in Figure 3(b) than it would be in any other projection because, in this example, there is only a single axis of rotational symmetry and, furthermore, a reflection from the planes normal to this axis was not observed. On the other hand, the pattern of the lattice is more readily seen in Figure 3(a) than it would generally be in a projection onto an arbitrary plane because, in this example, the plane X-T is very close to a plane containing a significant number of reflecting plane normals.

On the basis of the information contained in Figure 3(b), it is possible to guess at the space group of the structure, but it must be remembered that the reflections observed have been found in a random search and that what appear to be systematic absences may only be the result of chance. To identify the space group, it is necessary to make a systematic search of reciprocal space, e.g. by using photographic film techniques.

4. COMPUTER PROGRAM INDX

The sequence of operations described in Section 3 is straightforward but time-consuming. In particular, plotting of projections of the reciprocal lattice is a tedious task and, as we have seen, more than one projection will probably be required. The situation is made worse by the fact that the diffractometer will be standing idle while indexing is being done, therefore it seemed worthwhile to devise a computer program to plot a stereographic projection of the reflecting plane normals and as many projections of the reciprocal lattice as might be useful.

It is necessary first to establish whether a right- or left-handed diffractometer is being used. A data card in I2 format must be supplied, with the number 1 allocated for a right-handed, or -1 for a left-handed diffractometer. The program then reads the axial angles for each reflection observed. A data card, in 4F10.3 format, must be provided for each reflection, giving the angles 2θ, ω, χ and ϕ, in that order. The program will handle up to twenty reflections; any data cards beyond the twentieth will be ignored. The input data, serially numbered, are first printed on the line printer and each reflecting plane normal is plotted on a stereographic projection in standard orientation. The direction cosines and length of the reciprocal lattice vector for each reflection are then printed to enable the plotted normals to be identified.

It is probable that there will be cases where two or more orders of reflection have been observed from the same set of lattice planes, and also that the separate observations of each normal do not exactly coincide. The program assumes that normals within about 2.5° of one another are, in fact, the same normal and averages them to give a mean orientation. It then looks to see which of these normals lie in the same zone. The procedure is to calculate a zone normal for each pair of reflecting plane normals and then assume that zone normals that lie within about 8° of each other are in fact the same normal; the most likely orientation is then obtained by averaging. The number of plane normals in a zone can be deduced from the number of zone normals that are judged to be the same. Axes of prominent zones, i.e. those containing three or more plane normals, are plotted on the stereographic projection, using the symbol + to distinguish them from plane normals (plotted as o), numbered serially and the zone drawn. This completes the stereographic plot.

The program then draws a projection of the reciprocal lattice parallel to each prominent zone axis. For each projection, the coordinates of each reciprocal lattice point are calculated by applying a matrix rotation to its direction cosines in standard orientation and then multiplying the results by the length of the reciprocal lattice vector. The Z axis is, of course, parallel to the zone axis, but the X and Y axes can be chosen arbitrarily in the plane normal to this axis; the rotation applied is such that the direction in the zone initially in the plane of the stereographic projection becomes the Y axis.

As we have seen (Figure 3(b)), many of the reciprocal lattice points will lie in the plane of projection and the remainder will lie in a small number of planes parallel to this plane. For each projection, the program looks at the z-coordinates of all points, groups them according to the level of the plane in which they lie and determines a value for the distance between neighbouring planes. The symbol -, o or + is plotted at the projected position of each point, according to whether it is below, in or above the plane containing the origin of the reciprocal lattice; the z-coordinate of the point, expressed as a multiple of the spacing between reciprocal lattice planes, is drawn beside the symbol if it is + or -. A large O is drawn at the origin of the reciprocal lattice. The coordinates of each point in each projection are also printed on the line printer.

The JCL statements for the program are given, together with a sample set of data, in Appendix A, the program is set out in Appendix B, and the line printer output and stereographic plots are given in Appendix C.

It should be emphasised that this program does not assign indices to any of the reflections. Rather it presents the available information in a form that makes a rational choice of indices easier, but the choice must be made by the operator.

5. ACKNOWLEDGEMENT

I am grateful to Dr F.H. Moore for reading the manuscript and making some useful comments.

6. REFERENCES

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Ellis, P.J. and Pryor, A.W. [1968] - AAEC/E191.

Hamilton, W.C. [1974] - International Tables for X-ray Crystallography, 4 : 273. The Kynoch Press, Birmingham, England.

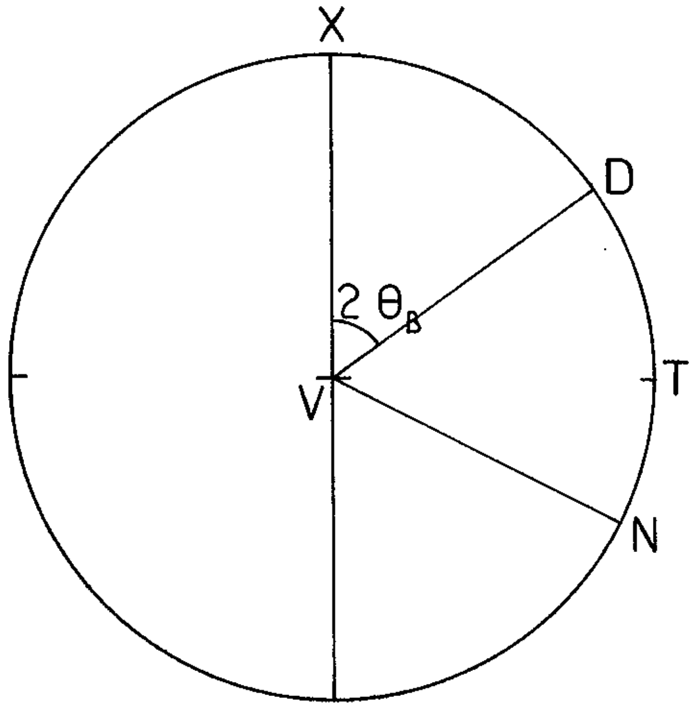


FIGURE 1(a) Stereographic projection showing: X, direction of incident beam; D, direction of diffracted beam; N normal to reflecting planes; V, vertical direction, parallel to axes 1 and 2 of diffractometer; T, direction perpendicular to V and X.

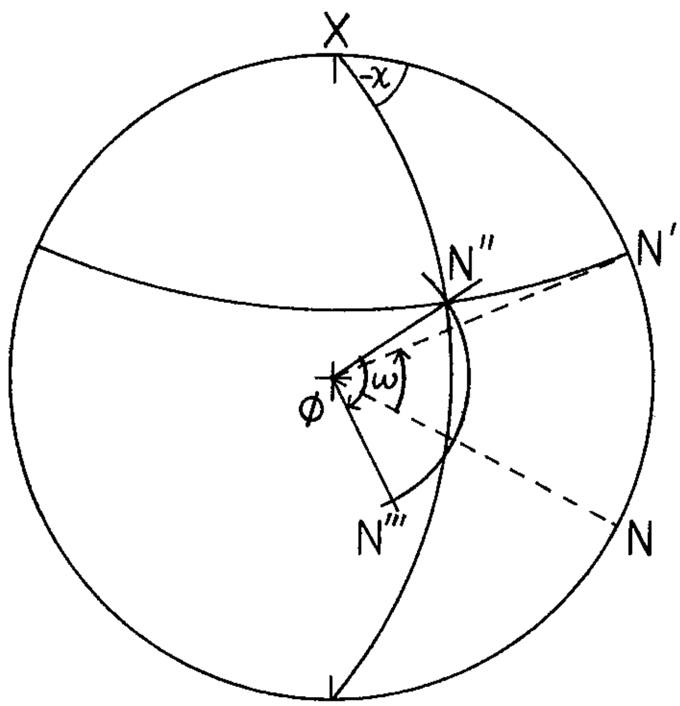
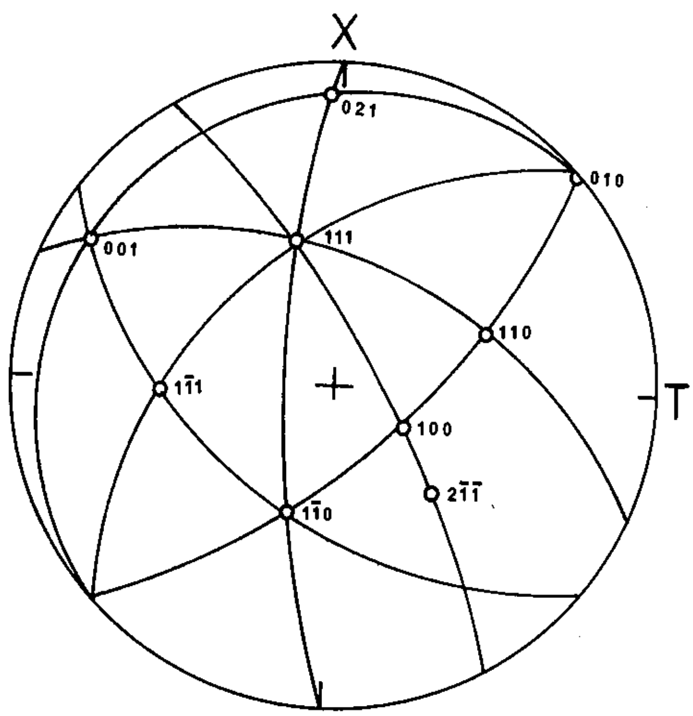
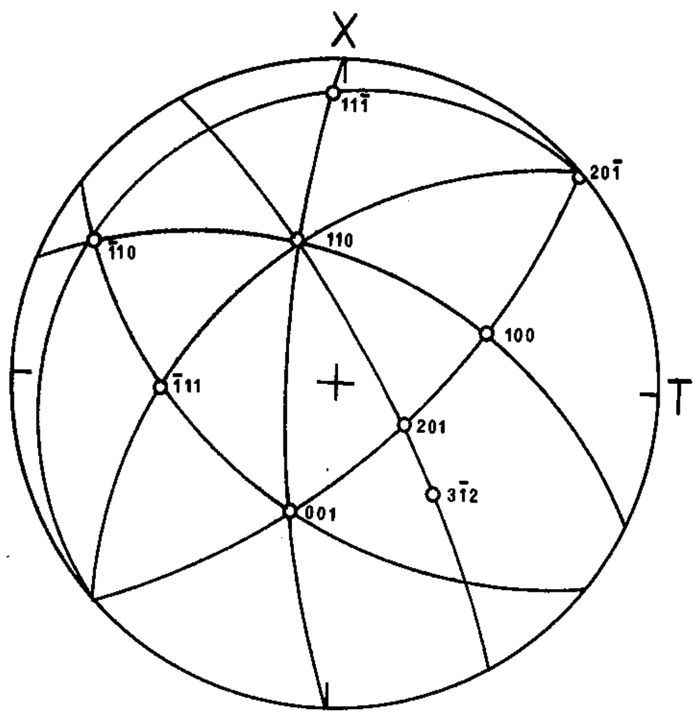


FIGURE 1(b) Stereographic projection showing movement of N as crystal is rotated into standard orientation.



(a)



(b)

FIGURE 2(a,b) Alternative ways of indexing the same set of reflections, corresponding to different choices of unit cell.

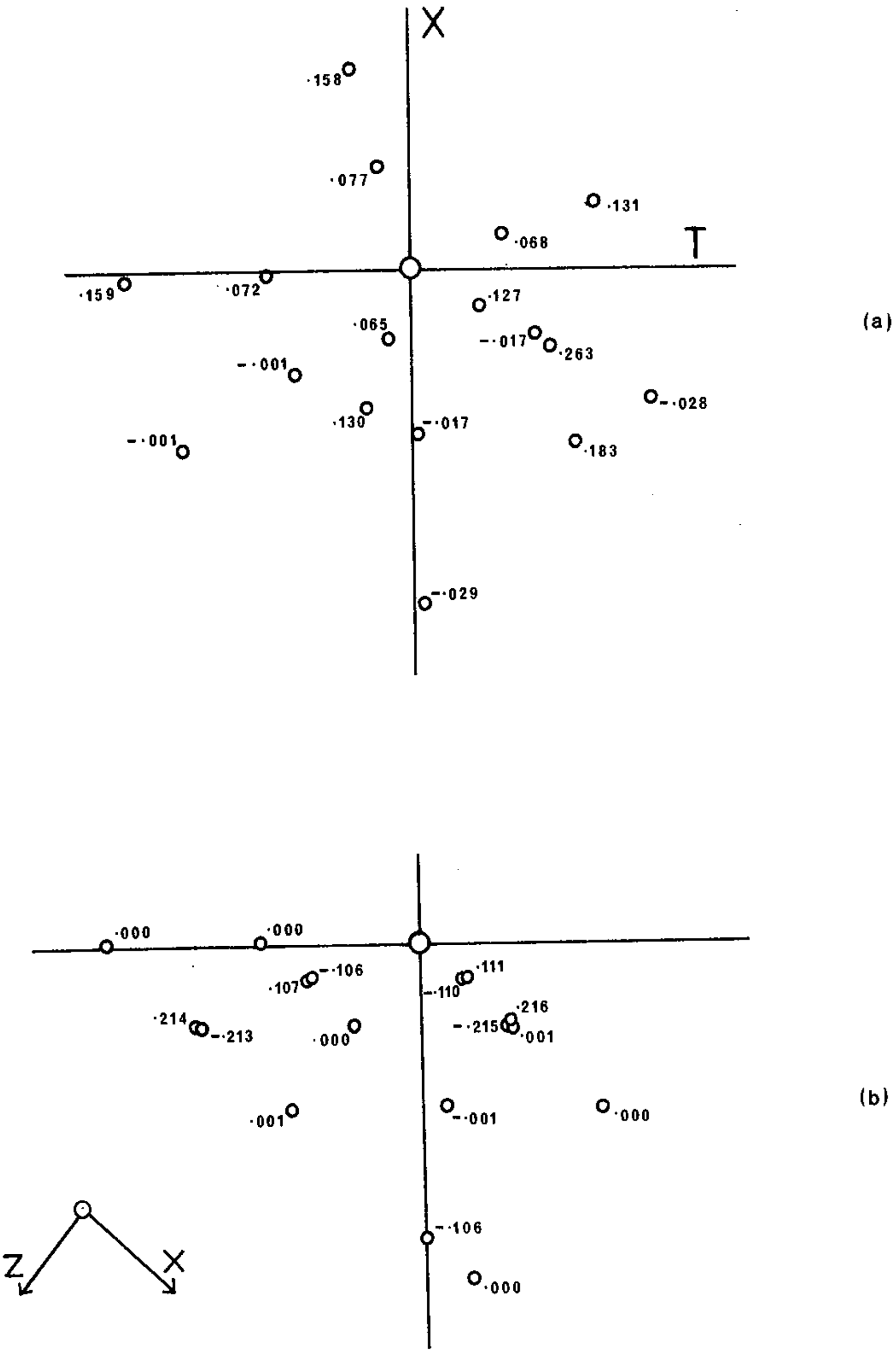


FIGURE 3 Reciprocal lattice points of reflections shown in Figure 2(a,b) projected onto (a) plane normal to V, and (b) the plane containing plane normals indexed as 001 and 100 in Figure 2(b). The reciprocal lattice axes X and Z corresponding to the indices of Figure 2(b) are as shown; the Y axis is normal to the plane of projection.

APPENDIX A

JCL AND SAMPLE DATA FOR PROGRAM INDX

```
//GJBINDX   JOB ('********/N31MA001',E3),G.J.BALL,
//                   CLASS=F,TIME=1
/*JOBPARM L=1
//      EXEC BUFFPROG,OUT=F
//      EXEC PGM=INDX
//STEPLIB   DD DSN=MAT.CJB.PLIB,DISP=SHR
//FT01F001  DD DDNAME=SYSIN
//FT03F001  DD SYSOUT=A
//AEPLOT    DD SYSOUT=F
//SYSIN     DD *
  1
         10.519       5.009     -45.219     107.664
         12.299       6.309     -37.599     -19.779
          5.314       2.424     -44.469     107.664
         13.809       6.659       6.709      28.274
          7.019       3.239     -39.209    -106.604
          7.019       3.249       8.079      28.274
         13.799       6.629     -41.040    -106.604
          6.319       3.289     -38.329     -19.779
          8.389       4.039       6.719      87.989
         16.989       8.279       5.589      87.989
          7.949       3.999       0.369     137.744
         15.559       7.814       0.189     137.744
          8.479       3.959     -31.479     177.149
         17.030       8.219     -32.389     177.149
         15.969       7.799     -41.259      46.379
          8.269       3.999     -61.960      28.559
         17.079       8.204     -62.489      28.559
/*
//
```

APPENDIX B

PROGRAM INDX

```
      DIMENSION A(4,20),B(4,20),Z(4,400),W(4,400)
      DIMENSION G(10),NG(10)
      DATA MINUS/'-'/,ZERO/'0'/,PLUS/'+'/
  100 FORMAT(I2)
  101 FORMAT(4F10.3)
  102 FORMAT(I4,1X,4F14.4)
  103 FORMAT(3X,'J',12X,'X',13X,'Y',13X,'Z')
  104 FORMAT(////3X,'J',11X,'A1',12X,'A2',12X,'A3',12X,'K')
  105 FORMAT(1H1)
  106 FORMAT(//20X,'ZONE',I3/)
  107 FORMAT(3X,'J',9X,'2THETA',9X,'OMEGA',9X,'CHI',11X,'PHI')
  108 FORMAT(' DATA CARD ERROR')
      READ(1,100) ND
      IF(ND*ND.EQ.1) GO TO 110
      WRITE(3,108)
      STOP
  110 CONTINUE
C
C  SET UP STEREOGRAPHIC PLOT
      CALL GPSEND(1)
      CALL GPLCT(0.,0.,1)
      CALL GPLCT(1.,1.,2)
      CALL GPLCT(7.,2.45,3)
      CALL GPLCT(7.,8.45,4)
      CALL GPLCT(4.,5.45,3)
      CALL GPLCT(10.,5.45,4)
      DO 120 J=5,360,5
      XST=3.*COS(J/57.29578)+7.
      YST=3.*SIN(J/57.29578)+5.45
      CALL GPLCT(XST,YST,4)
  120 CONTINUE
C
C  READ DATA AND PLOT ON STEREOGRAPHIC PROJECTION
      WRITE(3,107)
      J=1
    1 READ(1,101,END=3) TH,OM,CHI,PHI
      WRITE(3,102) J,TH,OM,CHI,PHI
      SINA=SIN((0.5*TH-OM)/57.29578)
      COSA=COS((0.5*TH-OM)/57.29578)
      SINF=SIN(PHI/57.29578)
      COSF=COS(PHI/57.29578)
      SINX=SIN(CHI/57.29578)
      COSX=COS(CHI/57.29578)
      A(1,J)=COSF*SINA+SINF*COSX*COSA
      A(2,J)=ND*(COSF*COSX*COSA-SINF*SINA)
      A(3,J)=-ND*SINX*COSA
      A(4,J)=2.*SIN(TH/114.592)
      IF(A(4,J).LT.0.) A(4,J)=-A(4,J)
      IF(A(3,J).GE.0.) GO TO 2
      A(4,J)=-A(4,J)
      A(3,J)=-A(3,J)
      A(2,J)=-A(2,J)
      A(1,J)=-A(1,J)
```

Continued

```
    2 R=3.*TAN(0.5*ARCOS(A(3,J)))
      Q=ARCCS(A(1,J)/SQRT(A(1,J)*A(1,J)+A(2,J)*A(2,J)))
      IF(A(2,J).LT.0.) Q=-Q
      XST=7.+R*SIN(Q)
      YST=5.45-R*COS(Q)
      CALL GPLOT(XST,YST,3)
      CALL GPTEXT(ZERO,0,1,0.)
      J=J+1
      IF(J.LE.20) GO TO 1
    3 CONTINUE
C
C   PRINT DIRECTICN COSINES OF PLANE NORMALS
      WRITE(3,104)
      NA=J-1
      DO 11 J=1,NA
      WRITE(3,102) J,(A(K,J),K=1,4)
   11 CONTINUE
      NP=60/(NA+4)
      NL=0
C
C   IDENTIFY ANC AVERAGE IDENTICAL PLANE NORMALS
      B(1,1)=A(1,1)
      B(2,1)=A(2,1)
      B(3,1)=A(3,1)
      B(4,1)=1.
      NB=1
      DO 6 J=2,NA
      DO 4 K=1,NB
      IF(A(1,J)*B(1,K)+A(2,J)*B(2,K)+A(3,J)*B(3,K).GE.0.999*B(4,K))
     C GO TO 5
    4 CONTINUE
      NB=NB+1
      B(1,NB)=A(1,J)
      B(2,NB)=A(2,J)
      B(3,NB)=A(3,J)
      B(4,NB)=1.
      GO TO 6
    5 B(1,K)=B(1,K)+A(1,J)
      B(2,K)=B(2,K)+A(2,J)
      B(3,K)=B(3,K)+A(3,J)
      B(4,K)=B(4,K)+1.
    6 CONTINUE
      DO 7 J=1,NB
      B(4,J)=SQRT(B(1,J)*B(1,J)+B(2,J)*B(2,J)+B(3,J)*B(3,J))
      B(1,J)=B(1,J)/B(4,J)
      B(2,J)=B(2,J)/B(4,J)
      B(3,J)=B(3,J)/B(4,J)
    7 CONTINUE
C
C   TAKE AVERAGED NORMALS IN PAIRS AND DETERMINE DIRECTION
C   COSINES OF CORRESPCNDING ZCNE AXIS
      J=1
      DO 10 K=2,NB
```

Continued

```
      KL=K-1
      DO 10 L=1,KL
      Z(1,J)=B(2,K)*B(3,L)-B(3,K)*B(2,L)
      Z(2,J)=B(3,K)*B(1,L)-B(1,K)*B(3,L)
      Z(3,J)=B(1,K)*B(2,L)-B(2,K)*B(1,L)
      Z(4,J)=SQRT(Z(1,J)*Z(1,J)+Z(2,J)*Z(2,J)+Z(3,J)*Z(3,J))
      IF(Z(3,J).LT.0.) GO TO 8
      Z(1,J)=Z(1,J)/Z(4,J)
      Z(2,J)=Z(2,J)/Z(4,J)
      Z(3,J)=Z(3,J)/Z(4,J)
      GO TO 9
    8 Z(1,J)=-Z(1,J)/Z(4,J)
      Z(2,J)=-Z(2,J)/Z(4,J)
      Z(3,J)=-Z(3,J)/Z(4,J)
    9 J=J+1
   10 CONTINUE
C
C     IDENTIFY AND AVERAGE IDENTICAL ZONE AXES
      NZ=J-1
      W(1,1)=Z(1,1)
      W(2,1)=Z(2,1)
      W(3,1)=Z(3,1)
      W(4,1)=1.
      NW=1
      DO 16 J=2,NZ
      DO 14 K=1,NW
      IF(Z(1,J)*W(1,K)+Z(2,J)*W(2,K)+Z(3,J)*W(3,K).GE.0.99*W(4,K))
     C GO TO 15
   14 CONTINUE
      NW=NW+1
      W(1,NW)=Z(1,J)
      W(2,NW)=Z(2,J)
      W(3,NW)=Z(3,J)
      W(4,NW)=1.
      GO TO 16
   15 W(1,K)=W(1,K)+Z(1,J)
      W(2,K)=W(2,K)+Z(2,J)
      W(3,K)=W(3,K)+Z(3,J)
      W(4,K)=W(4,K)+1.
   16 CONTINUE
C
C     PLOT AXES OF ZONES CONTAINING THREE OR MORE PLANE NORMALS
C     AND DRAW GREAT CIRCLE
      L=0
      DO 19 K=1,NW
      IF(W(4,K).LT.2.) GO TO 19
      L=L+1
      Z4=SQRT(W(1,K)*W(1,K)+W(2,K)*W(2,K)+W(3,K)*W(3,K))
      Z1=W(1,K)/Z4
      Z2=W(2,K)/Z4
      Z3=W(3,K)/Z4
      IF(Z3.GE.0.) GO TO 17
      Z3=-Z3
```

Continued

```
      Z2=-Z2
      Z1=-Z1
   17 R=3.*TAN(0.5*ARCOS(Z3))
      Q=ARCCS(Z1/SQRT(Z1*Z1+Z2*Z2))
      IF(Z2.LT.0.) Q=-Q
      XST=7.+R*SIN(Q)
      YST=5.45-R*COS(Q)
      CALL GPLCT(XST,YST,3)
      CALL GPTEXT(PLUS,0,1,0.)
      CALL GPNUMB(L,'(1H ,I2)',2,0.)
      XST=7.+3.*COS(Q)
      YST=5.45+3.*SIN(Q)
      CALL GPLCT(XST,YST,3)
      R1=TAN(0.5*ARSIN(Z3))
      R2=0.5*(1.-R1*R1)/R1
      R=R1+R2
      F1=ATAN(1/R2)
      DO 18 J=1,40
      F=F1*(1.-0.05*J)
      X=R2-R*CCS(F)
      Y=R*SIN(F)
      XST=3.*(X*SIN(Q)+Y*COS(Q))+7.
      YST=3.*(Y*SIN(Q)-X*COS(Q))+5.45
      CALL GPLCT(XST,YST,4)
   18 CONTINUE
   19 CONTINUE
      CALL GPSEND(2)
C
C  DETERMINE COORDINATES OF RECIPROCAL LATTICE POINTS W.R.T.
C  AXES RELATEC TC ZCNE AXES
      DO 30 K=1,NW
      IF(W(4,K).LT.2.) GO TO 30
      Z4=SQRT(W(1,K)*W(1,K)+W(2,K)*W(2,K)+W(3,K)*W(3,K))
      Z1=W(1,K)/Z4
      Z2=W(2,K)/Z4
      Z3=W(3,K)/Z4
      Z4=SQRT(1.-Z3*Z3)
      Z5=Z1/Z4
      Z6=Z2/Z4
C  PAGE FORMAT CONTROL
      IF(MOD(NL,NP).EQ.0) WRITE(3,105)
      NL=NL+1
      WRITE(3,106) NL
      WRITE(3,103)
C
C  SET UP PLCT OF RECIPROCAL LATTICE
      XL=0.
      XH=0.
      YL=0.
      YH=0.
      DO 20 J=1,NA
      B(1,J)=(Z3*Z5*A(1,J)+Z3*Z6*A(2,J)-Z4*A(3,J))*A(4,J)
      B(2,J)=(Z5*A(2,J)-Z6*A(1,J))*A(4,J)
```

Continued

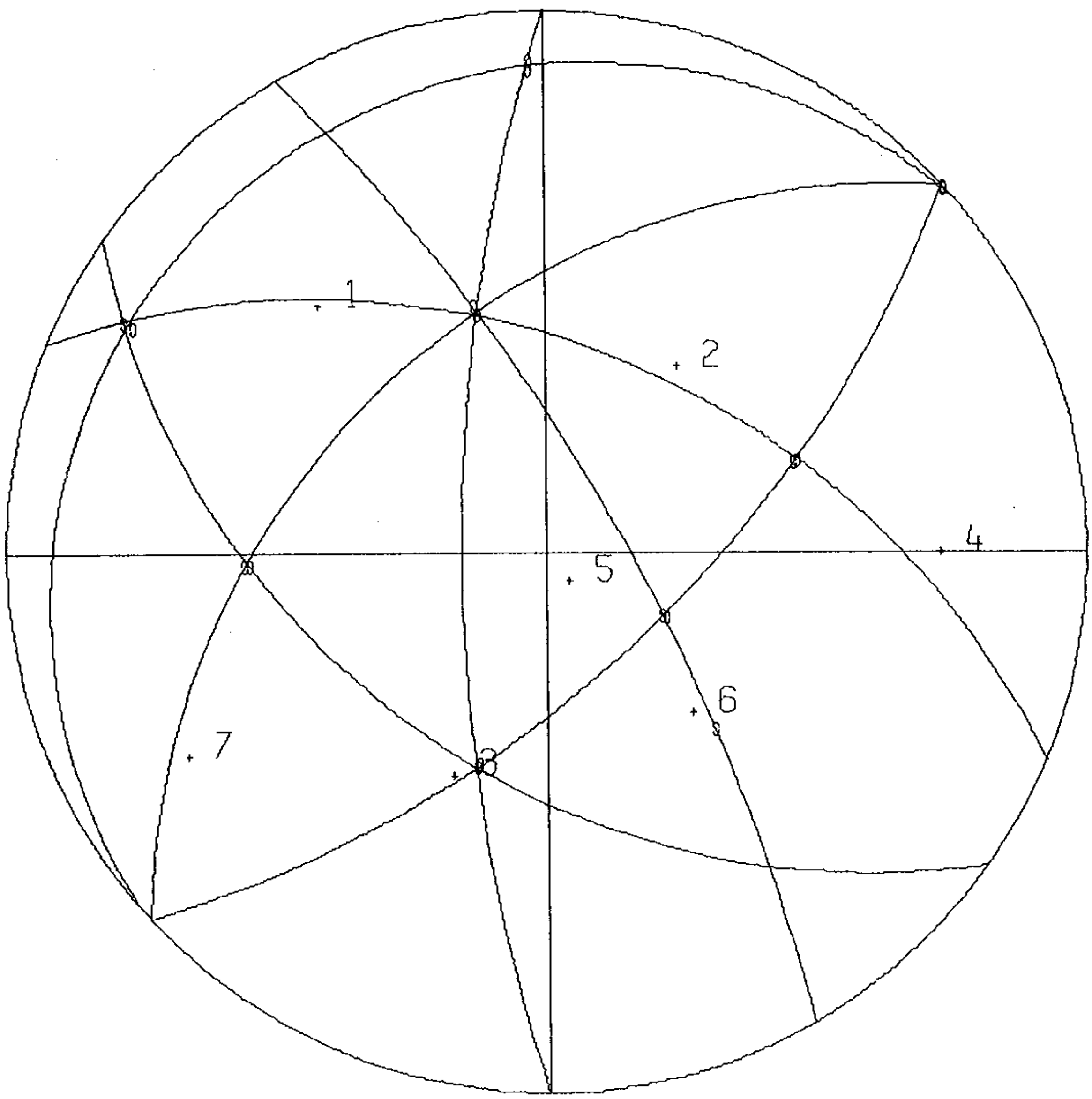
```
      B(3,J)=(Z4*Z5*A(1,J)+Z4*Z6*A(2,J)+Z3*A(3,J))*A(4,J)
      B(4,J)=B(3,J)
      IF(B(4,J).LT.0.) B(4,J)=-B(4,J)
      IF(B(1,J).LT.XL) XL=B(1,J)
      IF(B(1,J).GT.XH) XH=B(1,J)
      IF(B(2,J).LT.YL) YL=B(2,J)
      IF(B(2,J).GT.YH) YH=B(2,J)
   20 CONTINUE
      R=XH-XL
      IF(YH-YL.GT.R) R=YH-YL
      R=0.125*R
      CALL GPSEND(1)
      CALL GPLOT(2.,0.3,3)
      CALL GPNUMB(NL,'(5HZONE ,I2)',3,0.)
      CALL GPLOT(R,R,2)
      XPLT=R-XL
      YPLT=R-YL
      CALL GPLOT(XPLT,YPLT,3)
      CALL GPLOT(0.,0.,1)
      CALL GPTEXT(ZERO,0,2,0.)
C
C  ORDER Z COORDINATES OF RECIPROCAL LATTICE POINTS
      J=1
   21 IF(B(4,J).GT.B(4,J+1)) GO TO 22
      J=J+1
      IF(J.EQ.NA) GO TO 23
      GO TO 21
   22 TEMP=B(4,J)
      B(4,J)=B(4,J+1)
      B(4,J+1)=TEMP
      IF(J.EQ.1) GO TO 21
      J=J-1
      GO TO 21
   23 CONTINUE
C
C  GROUP Z COORDINATES IN PLANES AND DETERMINE SPACING BETWEEN PLANES
      DO 230 L=1,10
      G(L)=0.
      NG(L)=0
  230 CONTINUE
      L=1
      G(L)=B(4,1)
      NG(L)=1
      DO 25 J=2,NA
      IF(B(4,J)-G(L)/NG(L).LT.0.01) GO TO 24
      G(L)=G(L)/NG(L)
      L=L+1
   24 G(L)=G(L)+B(4,J)
      NG(L)=NG(L)+1
   25 CONTINUE
      G(L)=G(L)/NG(L)
C
C  PLOT RECIPROCAL LATTICE POINTS IN PROJECTION PARALLEL TO ZONE AXIS
```

Continued

```
      DO 29 J=1,NA
      WRITE(3,102) J,(B(M,J),M=1,3)
      CALL GPLOT(B(1,J),B(2,J),3)
      ZPLT=B(3,J)
      IF(ZPLT*ZPLT.LT.1.E-5) GO TO 28
      IF(ZPLT.LT.0.) GO TO 27
      I=ZPLT/G(2)+0.1
      CALL GPTEXT(PLUS,0,1,0.)
C
C  WRITE Z COORDINATE OF POINT AS A MULTIPLE OF INTERPLANAR SPACING
   26 CALL GPNUMB(I,'(1H ,I2)',2,0.)
      GO TO 29
   27 I=-ZPLT/G(2)+0.1
      CALL GPTEXT(MINUS,0,1,0.)
      GO TO 26
   28 CALL GPTEXT(ZERO,0,1,0.)
   29 CONTINUE
      CALL GPSEND(2)
   30 CONTINUE
      STOP
      END
```

APPENDIX C

COMPUTER OUTPUT FOR EXAMPLE GIVEN IN APPENDIX A

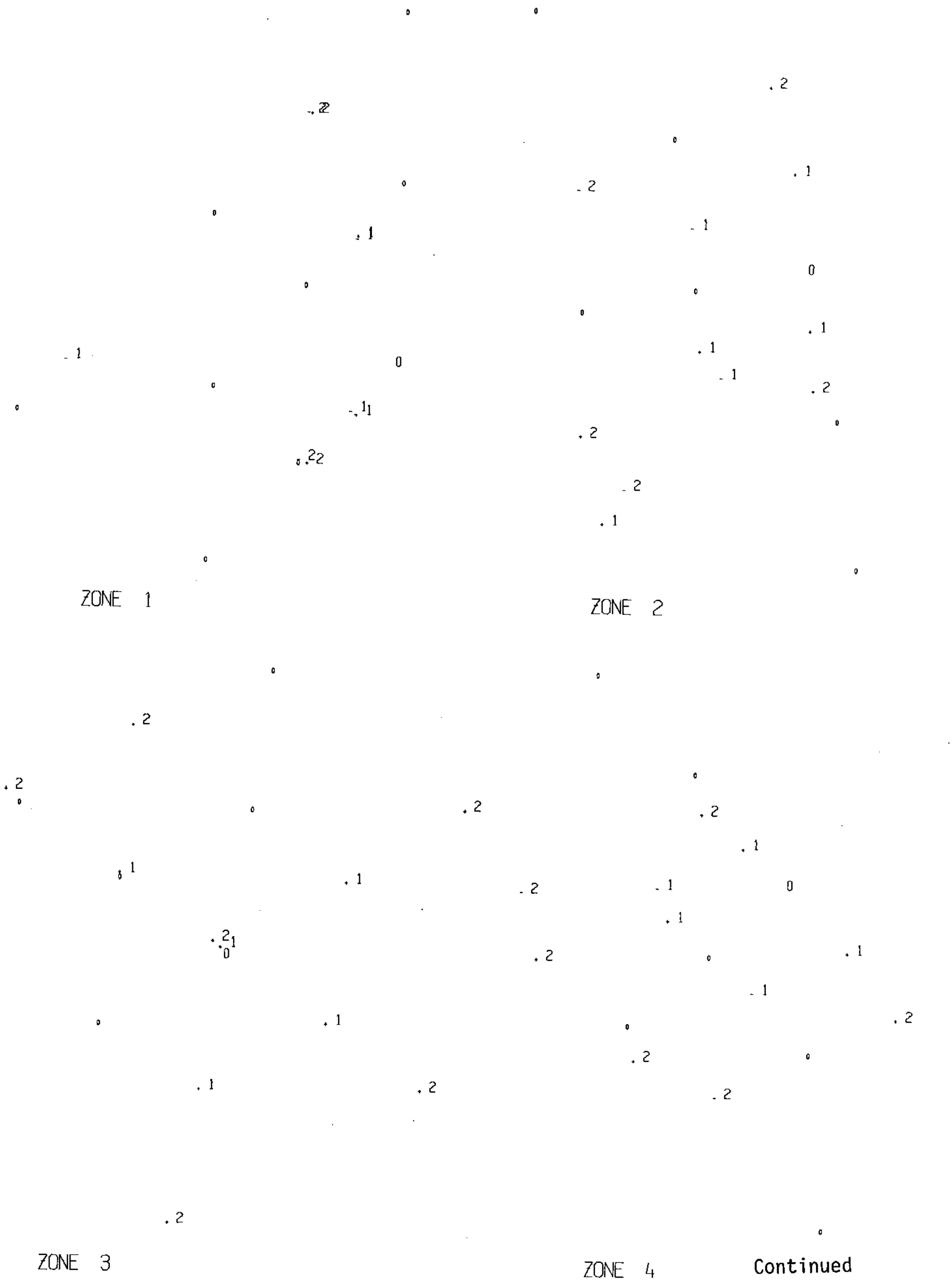


Continued

J	2THETA	CMEGA	CHI	PHI
1	10.5190	5.0090	-45.2190	107.6640
2	12.2990	6.3090	-37.5990	-19.7790
3	5.3140	2.4240	-44.4690	107.6640
4	13.8090	6.6590	6.7090	28.2740
5	7.0190	3.2390	-39.2090	-106.6040
6	7.0190	3.2490	8.0790	28.2740
7	13.7990	6.6290	-41.0400	-106.6040
8	6.3190	3.2890	-38.3290	-19.7790
9	8.3890	4.0390	6.7190	87.9890
10	16.9890	8.2790	5.5890	87.9890
11	7.9490	3.9990	0.3690	137.7440
12	15.5590	7.8140	0.1890	137.7440
13	8.4790	3.9590	-31.4790	177.1490
14	17.0300	8.2190	-32.3890	177.1490
15	15.9690	7.7990	-41.2590	46.3790
16	8.2690	3.9990	-61.9600	28.5590
17	17.0790	8.2040	-62.4890	28.5590

J	A1	A2	A3	K
1	0.6699	-0.2179	0.7098	0.1833
2	-0.2707	0.7446	0.6101	0.2142
3	0.6787	-0.2204	0.7005	0.0927
4	-0.4742	-0.8726	0.1168	-0.2404
5	-0.7439	-0.2169	0.6321	0.1224
6	-0.4730	-0.8698	0.1405	-0.1224
7	-0.7241	-0.2110	0.6566	0.2403
8	-0.2676	0.7374	0.6202	0.1102
9	-0.9926	-0.0321	0.1170	-0.1463
10	-0.9948	-0.0312	0.0974	-0.2954
11	-0.6727	0.7398	0.0064	-0.1386
12	-0.6729	0.7397	0.0033	-0.2707
13	0.0375	-0.8520	0.5222	0.1479
14	0.0368	-0.8436	0.5357	0.2961
15	0.5464	0.5163	0.6595	0.2778
16	0.2268	0.4118	0.8826	0.1442
17	0.2260	0.4029	0.8869	0.2970

Continued



Continued

.2

.2 .1 o

.1 .4 o

.2

0 .3

.1

.1 o

.2

o

.2 o

o

ZONE 5

.2

o .1 .2

o .4 .1

.2

0 .4

.1

o .1

.2

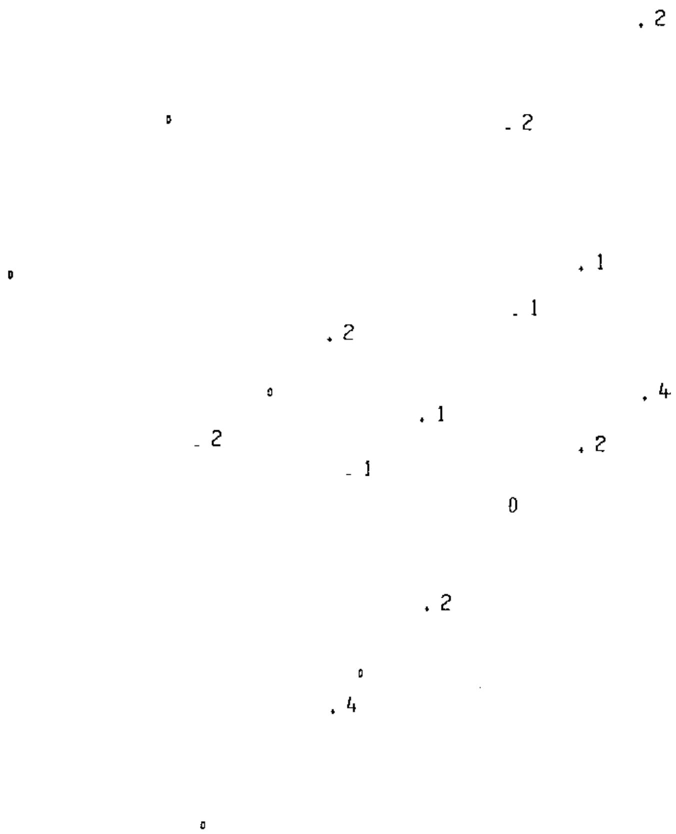
o

.0 .2

o

ZONE 6

Continued



ZONE 7

Continued

ZONE 1

J	X	Y	Z
1	-0.1448	0.1124	0.0010
2	-0.1460	-0.1568	-0.0001
3	-0.0727	0.0576	-0.0003
4	-0.0753	-0.0776	-0.2148
5	-0.0315	-0.0420	0.1106
6	-0.0356	-0.0394	-0.1103
7	-0.0690	-0.0802	0.2157
8	-0.0760	-0.0798	0.0007
9	-0.0337	0.0947	-0.1063
10	-0.0733	0.1918	-0.2124
11	0.0011	0.1386	0.0001
12	0.0013	0.2707	0.0006
13	-0.0331	0.0966	0.1069
14	-0.0706	0.1915	0.2146
15	-0.2569	-0.0031	-0.1056
16	-0.1426	-0.0217	-0.0009
17	-0.2939	-0.0428	0.0004

ZONE 2

J	X	Y	Z
1	-0.1792	-0.0385	0.0024
2	0.0037	-0.0965	0.1912
3	-0.0906	-0.0197	0.0001
4	0.0400	-0.2371	0.0008
5	-0.0144	0.0744	0.0962
6	0.0224	-0.1204	-0.0017
7	-0.0345	0.1420	0.1907
8	0.0006	-0.0492	0.0986
9	-0.0684	-0.0879	-0.0948
10	-0.1429	-0.1776	-0.1880
11	-0.0938	0.0297	-0.0976
12	-0.1838	0.0579	-0.1901
13	-0.1093	0.0995	-0.0016
14	-0.2207	0.1974	0.0007
15	-0.1597	-0.2048	0.0986
16	-0.0858	-0.0673	0.0943
17	-0.1786	-0.1364	0.1941

Continued

ZONE 3

J	X	Y	Z
1	-0.0108	0.0106	0.1827
2	-0.1742	0.1247	0.0017
3	-0.0043	0.0055	0.0925
4	0.0370	0.2376	-0.0009
5	-0.1068	-0.0597	-0.0031
6	0.0210	0.1206	-0.0024
7	-0.2115	-0.1140	0.0006
8	-0.0900	0.0636	0.0021
9	0.1010	0.0605	0.0868
10	0.2001	0.1221	0.1798
11	0.0846	-0.0585	0.0929
12	0.1646	-0.1143	0.1820
13	-0.0215	-0.1140	0.0916
14	-0.0468	-0.2262	0.1853
15	-0.0798	0.1910	0.1853
16	-0.0899	0.0674	0.0904
17	-0.1855	0.1363	0.1876

ZONE 4

J	X	Y	Z
1	-0.1362	-0.1228	0.0017
2	-0.0755	0.0578	0.1920
3	-0.0681	-0.0629	0.0004
4	0.0910	-0.1143	0.1909
5	-0.0818	0.0911	-0.0014
6	0.0490	-0.0581	0.0960
7	-0.1656	0.1740	0.0004
8	-0.0401	0.0294	0.0984
9	0.0177	-0.1452	-0.0010
10	0.0301	-0.2939	-0.0004
11	-0.0307	-0.0931	-0.0980
12	-0.0607	-0.1819	-0.1911
13	-0.1121	-0.0054	-0.0962
14	-0.2276	-0.0106	-0.1891
15	-0.1304	-0.1520	0.1925
16	-0.1029	-0.0328	0.0955
17	-0.2140	-0.0673	0.1946

Continued

ZONE 5

J	X	Y	Z
1	0.0490	-0.1101	0.1382
2	0.0409	0.1590	0.1376
3	0.0254	-0.0564	0.0691
4	0.2246	0.0857	0.0028
5	-0.0963	0.0389	0.0648
6	0.1144	0.0435	-0.0015
7	-0.1854	0.0743	0.1336
8	0.0206	0.0810	0.0719
9	0.1148	-0.0906	-0.0014
10	0.2314	-0.1836	0.0030
11	0.0045	-0.1386	-0.0003
12	0.0087	-0.2706	0.0003
13	-0.0874	-0.0995	0.0658
14	-0.1741	-0.1972	0.1360
15	0.1815	0.0107	0.2101
16	0.0453	0.0240	0.1348
17	0.0913	0.0475	0.2786

ZONE 6

J	X	Y	Z
1	-0.0439	-0.1124	0.1380
2	-0.0432	0.1566	0.1395
3	-0.0213	-0.0576	0.0695
4	0.1828	0.0776	0.1356
5	-0.1150	0.0420	-0.0028
6	0.0948	0.0393	0.0667
7	-0.2265	0.0802	0.0019
8	-0.0232	0.0798	0.0724
9	0.0915	-0.0947	0.0637
10	0.1810	-0.1918	0.1330
11	0.0002	-0.1386	-0.0010
12	-0.0002	-0.2707	-0.0014
13	-0.1119	-0.0966	-0.0001
14	-0.2259	-0.1915	0.0037
15	0.0246	0.0031	0.2767
16	-0.0414	0.0217	0.1364
17	-0.0876	0.0428	0.2805

ZONE 7

J	X	Y	Z
1	-0.1003	0.0872	0.1263
2	-0.1703	0.0281	-0.1269
3	-0.0497	0.0447	0.0642
4	-0.0063	0.2026	-0.1294
5	-0.0804	-0.0923	-0.0005
6	-0.0004	0.1028	-0.0664
7	-0.1631	-0.1764	0.0016
8	-0.0885	0.0144	-0.0642
9	0.0343	0.1287	0.0605
10	0.0639	0.2603	0.1243
11	0.0366	0.0307	0.1301
12	0.0706	0.0599	0.2544
13	-0.0448	-0.0572	0.1288
14	-0.0941	-0.1135	0.2568
15	-0.1899	0.2028	0.0001
16	-0.1321	0.0577	-0.0007
17	-0.2728	0.1173	0.0011