DO NOT ATTEMPT TO COLLECT SINGLE CRYSTAL DATA WITHOUT

HAVING READ THIS DOCUMENT THROUGH PAGE 25

Draft. Any comments by users would be welcome for final writeup.

AXDA - ANDA

USERS INFORMATION MANUAL

A. L. Bednowitz, Brookhaven National Laboratory

Writeup modified by W. C. Hamilton, 2/69

Automatic X-Ray Data Acquisition and Automatic Neutron Data Acquisition

AXDA and ANDA are programs written for X-ray and Neutron data acquisition with the BNL Multiple Spectrometer Control System. The basic programs provide the necessary computations for the startup and continuation of a data acquisition run. These programs provide for obtaining data in one of several modes of operation as desired by the experimenter. They have been designed to operate four-circle single crystal diffractometers. But, with small modifications they have also been applied to three-circle and even two-circle diffractometers.

Version 2/69

Contents

Warnings 1

Brief operational procedures 2-4

Data output 4-10

Control Integer Table 5

Details of control integers . . . . . 11 ff

IC and IC+2 (Sequencing) 11-13

Table of IC and IC+2 14

IC+1 (Parity) 15-16

IC+3 (Extinctions) 17

IC+4 (Scan range) 18

IC+5 (Optimization) 19

IC+6 (Scan mode) 20

IC+7 (Reference reflections: display or true values) . . 20

IC+8 (Output option) 21

IC+9 (Settings check or collect). 22

FORMAT of data list 23-24

DISPLAY OUTPUT 25

Appendices . . . . . 26 ff

AXDA-ANDA (SATT, MAIN, XRAY)

****

It is assumed that the user is familiar with the MSCS operating system, especially all mnemonics available on the console typewriter. For convenience however, these are listed in Appendix 4. Be sure that you understand an instruction before using it.

****

Before starting an experiment, be sure that all limit switches are set to prevent damage to either the equipment or the experiment for all possible motion of the various circles.

****

Before starting the experiment, it is well to zero all motors and then check and reset display to zero. This is accomplished by addressing each motor in turn with the two commands (for motor 3, for example)

ZRA3

CHA3

If the check axis command is given while the motor is still moving to zero, the message

ERROR ZERO ENABLED

will be typed out.

****

The method for calculation of the orientation matrix is described in Appendix 1.

****

THIS IS A COMPLEX SET OF PROGRAMS. DO NOT ATTEMPT ANY CHANGES WITHOUT THOROUGH UNDERSTANDING OF INTERRELATIONSHIPS OF PROGRAMS WITH ONE ANOTHER AND WITH THE SYSTEM, WHICH IS NOT PROTECTED AGAINST USER ERRORS. UNDER NO CIRCUMSTANCES SHOULD A SUBROUTINE BE CHANGED WITHOUT CONSULTATION WITH WCH.

Procedure

(1) Obtain the appropriate paper tape (MAIN, SATT, XRAY) in one of the two following ways.

(1) Use the master tape available at the MSCS. This will usually be found in the drawer labelled W. Hamilton. The programs are always the current operational versions but will include cell parameter for KBr and instrument constants and wave lengths which are probably incorrect. (As soon as possible, prepare your own tape.)

(2) Assemble a tape on the CDC 924 computer for your problem. Always use the current source deck obtained from the crystallographic program source tape. Replace the standard parameters with the parameters appropriate for your problem.

(2) Have the listing of the assembled program in front of you before you begin. The listing of the master tape is available in the control room. If you have assembled your own program, you will have the listing.

(3) Insert the tape in the paper tape reader (after you know how to use it) Load the program as follows:

LDP0000MAIN (or SATT or XRAY)

Now check the listing for the location called LAST. It is probably around 1224. The next program on the tape is loaded into this location, for example,

LDPI224VECT.

(4) Reset the program flag

RSP

(5) Change parameters as necessary by use of ALW,INT,DEC,LDW,LDI,LDD commands. All alterable parameters are fully described below.

(6) STP0000

(7) Honor all requests received on typewrite . If data is requested and that already stored in the computer is correct, simply restart program at the location indicated on the typewriter.

(8) At one point in the procedure, the metric and the orientation matrix will be typed out. If these appear to be incorrect, corrections to reference reflections may be made and the program restarted at the location CARE. This can be done as often as one likes at this point in procedure DO NOT ATTEMPT TO RESTART HERE AT ANY LATER STAGE WITHOUT RELØADING PRØGRAM.

(9) The program next zeroes all the motors and then requests the indices of the first reflection, from which data collection will be initiated. A restart location (XXXX) will also be typed out. Enter the indices as follows, for the (65,-58,1) reflection for example:

INT0001 65,-58,1

STP in location directed. (Probably about 371)

(10) From this point on the only acceptable restart procedure is exactly that given in item (9). DO NOT START THE PRØGRAM IN ANY OTHER LOCATION. The program may be stopped at any time with the

HLP

instruction. Never restart before giving an

RSP

instruction.

(11) At some point, the message will be typed out:

DATA COLLECTIØN TERMINATED etc.

This will happen after the first reflection if the single reflection mode has been specified by the appropriate control integer. If no data has appeared on typewriter at this point, it means either that breakpoint D at the station is down, or that the expected reflection lies outside the range of the internal "limit switches". (See discussion of parameters below.) and that IC+9 = 0 or 1.

(12) In the normal multiple reflection operating mode, data acquisition will be terminated after a complete independent set of data has been collected. Instructions for continuation will be given on the typewriter. However, if breakpoint switch C has been left in the down position, data collection will be restarted (usually in the negative hemisphere) at a point determined by one of the input parameters (see below).

(13) Please observe that data output modes are selected by one of the control integers (IC+8).

DATA INPUT

The following discussion describes all items of data which must be set according to the desires of the experimenter. The data in each block must be in exactly the order indicated.

DATA BLOCK 1 (integer format)

DAY, MONTH, XTAL — These three integers are used solely to identify output on magnetic tape. For easy identification of data, it is wise to reload program and change these when important program parameters are changed. They cannot be changed without reloading program. Alternatively the location DATE in BCD format may be later changed with an ALW instruction.

DATA BLOCK 2 (integer format)

IC → IC-9 CONTROL INTEGERS which control practically all options in program [illegible] the following Table 1.

Table 1. Control Switches

Switch #	Symbolic Address	Description of Use
1	IC	Defines the lattice type (See Table 2, pg. 14) Controls segment of reciprocal space to be examined. 0 for triclinic.
2	IC + 1	Axis parity definition (See Table 3, pg. 16)
3	IC + 2	.LE.0 Continuous (Table 2, pg 14) .GT.0 Call Finish after each data output for one reflection
4	IC + 3	Systematic lattice extinctions (Table 4, pg. 17) 0 for primitive cell
5	IC + 4	.LE.0 Normal fixed scan interval .GT.0 Variable scan interval 1 = $\alpha/\cos\theta$ variation; 2 = $\alpha+\alpha(\tan\theta)K$
6	IC + 5	.EQ.4 For no optimization .LT.4 Optimize 1 or 2 axes before scan (see pg. 19)
7	IC + 6	SCAN MODE (see pg. 20) 0 = Phi Scan Mode 4 = Peak Height Mode 2 = θ - 2θ Scan Mode 1 = Omega Scan Mode
8	IC + 7	.EQ.0 Reference reflection settings angles are input as read from display counter* .GT.0 Reference reflection settings angles are true (or absolute) values based on the x-ray beam and diffractometer axes.
9	IC + 8	.EQ.0 For only tape output .GT.0 For alternate outputs (see pg. 21)
10	IC + 9	.EQ.0 For normal operation** .GT.0 For calculation of angle settings. 1 Settings after zero and parity correction. 2 Settings before zero and parity correction.

* Except that on neutron machines, subtract true θ from ($\omega+\theta$) reading before entry.

** If startup in this mode, most input requests will be skipped.

DATA BLOCK 3 (floating point format)

WAVE	wavelength of α_1 or highest point of incident spectrum. Used in calculating metric from reference reflections.
DSTAR	maximum d^* to be calculated.
DSTMIN	minimum d^* to be calculated.
SETWAV	wavelength for center of θ-2θ scan. May set to 0. If so, WAVE is used for center of scan.

DATA BLOCK 4 (integer format)

STEP	step scan interval (degrees x 100). MUST BE EVEN INTEGER for the neutron machines if θ-2θ scan is used.
STPTIM	monitor setting for each step (prescaler number is 1 for stepscan, 2 for peak heights). But prescaler number is 0 for H6S3.
BKGTIM	number of steps for each background (n on each side of the step scan) pertinent only for integrated on-line output. Complete scan is available on tape.
SCNDEL	step-scan range (degrees x 100). May be left at any value if IC+4 .GT.0
ATSCL	scale factor x 100 for no attenuator
ATSCL1, ATSCL2, ATSCL3	attenuator scale factors (XRAY only) These may be modified in data processing programs. Useful to have for on-line output.
RTCØMP	Monitor scale factor for linearity check, one prescaler is used (XRAY only).
RATE	Maximum count for linearity (XRAY only).
FILTNM	filter number (XRAY only) 0, 1, or 2 (Zr = 1)

[The following 6 software limit switch settings should be somewhat inside the true (hardware) limit switches. If a hardware limit switch is hit, the program will stop and be rendered inactive. The software limit switches prevent attempted data collection for any reflection which would lie outside the limits. These software limits are always entered as display values and in degrees x 100.]

LIMS	positive 2θ limit
LIMS+1	Negative 2θ limit
TUBELM=ØHMLIM	positive (ω+θ) limit
TUBELM+1	negative (ω+θ) limit
CNTERO=CHILIM	X-ray: omega vs counter limits
CNTER+1	neutron: chi limits
DELPHI	added to calculated Ø to get display value
DELØHM	added to calculated ω to get display value
DELTWØ	added to calculated 2θ to get display value
DELCHI	added to calculated χ to get display value
TIME	delay time for filter settings ≈ 8 (XRAY only)
STDSCN	minimum scan range in variable scan range mode (degrees x 100). This is α. See Table 1.

DATA BLOCK 5 (Floating point format)

FACTØR (double precision) constant used in the tangent formula for scan range. The variable scan range is

STDSCN/100 + FACTØR*tanθ*STDSCN/100 for IC+4=2.

DATA BLOCK 6 (integer format)

Three reference reflections.

21 pieces of data in the following order.

H, K, L, PHI, OMEGA, TWOTHETA, CHI	for first reflection.
ditto	for second reflection.
ditto	for third reflection.

These three reflections are used to determine the metric and orientation matrix. If H, K, L, and DSTAR for the third reflection are entered as zero, the first two reflections and the input cell constants are used to calculate the orientation matrix and a new metric. If the true TWOTHETA is entered as less than or equal to zero for any one of the three reflections, the TWOTHETA calculated from the input cell constants is used. Otherwise the calculated value is ignored and the observed value is used.

These reflections may be entered as either "true" or "display" values, depending on the value of IC + 7.

If entered as DISPLAY values

Enter the encoder readings as displayed on the diffractometer SCS-1 console, with the following exception. On the neutron machines, the value displayed is $\theta + \omega$, not ω alone. The user must insert the displayed value of $(\theta + \omega)$ minus the true value of θ. For each angle, the zero of that angle will be subtracted from the displayed value, and any requested changes in axis parity will be made to provide true angles for a positive machine in the sense defined in Appendix 1.

If entered as TRUE values

The true values of the angles for a positive Hamilton machine should be entered. If three reference reflections are used, the parity does not really matter, and an internally consistent orientation matrix will be put out. If only two reference reflections are used, the routine will calculate the third axis in a right-handed system, and parity conventions must be strictly adhered to.

DATA BLOCK 7 (floating point format)

A^*, B^*, C^* in reciprocal angstroms, $\cos\alpha^*$, $\cos\beta^*$, $\cos\gamma^*$ in absolute units. This should correspond to a right-handed system. These cell constants are ignored if three reference reflections with positive 2θ's are supplied. The program will recalculate the cell constants to be consistent with the reference reflections, and the new values may be typed out by the user. (See the listing for locations).

DATA BLOCK 8 (integer format)

STDHA STDKA STDLA	indices of first standard reflection for intensity monitoring
STDHB STDKB STDLB	indices of second standard reflection for intensity monitoring
STDNUM	The number of reflections to be observed between runs on the standard reflections.
ØPTSCN	Scan range in degrees x 100 for an optimization run.
STDFLG	Current count for number of reflections observed between standards. Initial value should be less than STDNUM.

ØPTSTP	Step-scan size for optimization run (see page 19)
BADCHI	if true χ for (hkℓ) is less than this number, program will attempt to collect the ($\bar{h}\ \bar{k}\ \bar{\ell}$) reflection at 180 + χ but the same Ø.
PHIMØT	= 0 for AXDA, = 3 for ANDA
ØMEGA	= 1 for AXDA, = 2 for ANDA
TWØM T	= 2 for AXDA, = 1 for ANDA
CHIMØT	= 3 for AXDA, = 0 for ANDA
LABEL	Blank
SIZENM	Character size and number of word in display, usually 00000307
CØØRD	Vertical coordinate of display, usually 00000400

<u>Other data which should be checked</u>

CODEI7		BCI 1,I3	last integer <u>must</u> be the experiment number
	or	I2	
	or	I7	
CØDEN		BCI 1,SATT	or any other word which will appear on magnetic tape data
	or	,MAIN	
	or	,XRAY	
DATA		must be in this location. Date will be merged in here by program.	
CØDE		BCI 1,I2I3	last integer must again be experiment number
		,I2I4	
		,I2I7	
LIST		in the program VECT should contain coded restart indices for continuation when switch is down. See page 13.	

Control Integers

The various options in this program are controlled for the most part by a set of synthetic switches. The assembled set of control integers is typed out before the second input request at startup time. At that time the integers may be altered freely. In addition they may be altered at any time after halting the program (HLP). The various options currently available are given in the following notes.

Control Integer 1 IC

Lattice type. This integer limits data collection to certain segments of reciprocal space depending on the symmetry of reciprocal space. (see Table 2, page 14)

Control Integer 2 IC + 1 (See page 15)

Control Integer 3 IC + 2

Sequencing

= 1 CALL FINISH after each reflection.

= 0, -1, -2 Sequence through reciprocal space as follows.

	Layers	Rows	Spots
= 0	H	K	L
=-1	L	H	K
=-2	K	L	H

≤-4 Special list of reflections is provided. (see page 13)

The automatic choice of hkℓ values is controlled by the integer (IC+2). There are four modes of operation. When the integer is greater than zero the program will terminate after observing one reflection and outputting the data for that reflection. Another reflection can be inserted with ALW instructions and the program restarted. (The location for restarting is typed out by the RESTAR routine.)

In the three automatic sequencing modes (see Table 2 the hkℓ values are chosen in a zig-zag fashion through one level at a time.

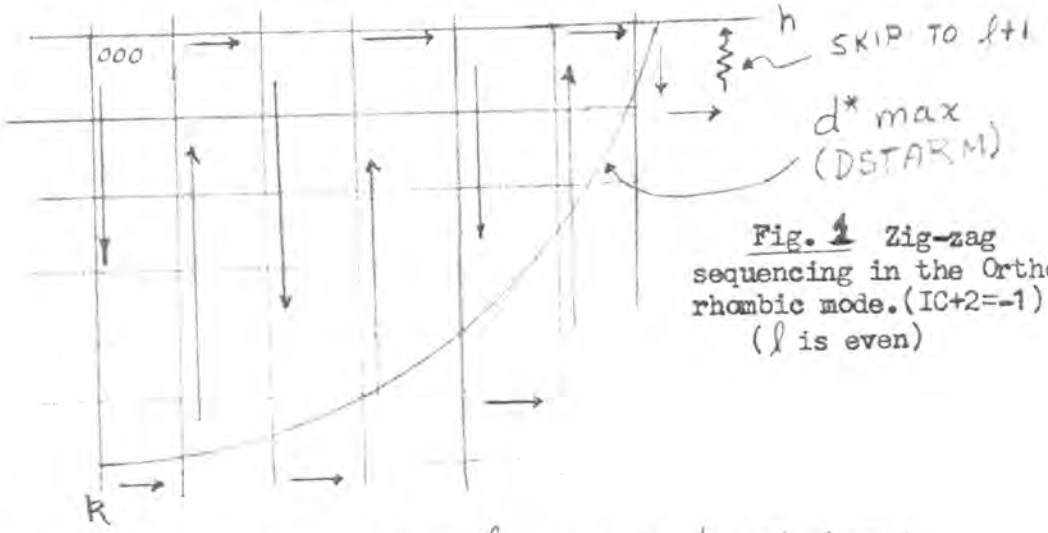


Fig. 1 Zig-zag sequencing in the Orthorhombic mode. (IC+2=-1) (ℓ is even)

For IC+2 =0 the sequencing is most rapid on ℓ, next on k and slowest on h. In Fig. 2 the effect of setting the sequence control integer to (-1) is shown. For the conditions in Fig. 2. The operation is as follows

1) increment ℓ if h+ℓ is even
2) decrement ℓ if h+ℓ = 2n+1
3) if DSTARM is exceeded
   a) Increment h if ℓ = 2n
   b) Decrement h if ℓ = 2n+1
4) if DSTARM is still exceeded

   Increment ℓ and repeat 1)

5) if in repeated trials DSTARM is still exceeded, reverse the sign of ℓ and test breakpoint 'C'.

6) if ℓ has been negated and breakpoint 'C' is up the data aquisition run terminates.

7) if 'C' is down the data aquisition run continues in sequence from the first reflection in the special list(**must be inserted by ALW at run time). If this reflection has negative indices, reflections will be collected in the negative hemisphere.

Special List

If IC + 2 $\leq$ -4 the program requires a special list of reflections whose number is limited by the available user area at the end of ANDA (in the BUFFER array). This option is not available in AXDA.

The special list of reflections is to be inserted only when requested by the program. It cannot be precompiled into the program.

The reflection list consists of packed octal words (right adjusted octal integers). Each index is represented by a two digit octal integer, i.e.: $10_8 = 8_{10}$, $71_8 = -6_{10}$. Several examples follow:

00177705 $\equiv$ h=15, k=$\bar{1}$, ℓ=5

00772019 $\equiv$ h=$\bar{1}$, k=16, ℓ=15

00770167 $\equiv$ h=$\bar{1}$, k=1, ℓ=$\bar{9}$

The last reflection in the list should be 00000000, this is a terminator flag. When reaching the terminator the program will type a "programmed stop" message and go inactive.

This list can be inserted either by using the ALW or LDW commands or by assembling a program on the CDC 924 to obtain an input tape

eg.

```
LIST    IDEN
HKL1    ØCT     777701
HKL2    ØCT     777703
KKL3    ØCT     770177
HKLAST  PZE
        ABSO
        END
```

The tape will be loaded by an LDP (LIST) LIST, command.
address

If IC + 4 $\geq$ -2, the first word in LIST should contain the indices of the reflection from which data collection in the negative hemisphere is to be collected. This one word may be precompiled.

Table 2. Data collection sequencing and limitations as determined by IC and IC + 2.

Lattice IC	Laue Groups	Fraction of space examined	Sequence IC+2	Condition on h, k, ℓ assuming start with zero or positive slow varying index: Layers	Rows	Spots		Conditions on h, k, ℓ assuming start with negative slow varying index: Layers	Rows	Spots
0	$\bar{1}$	1/2	0	+h	±k	±ℓ		-h	±k	±ℓ
			-1	+ℓ	±h	±k		-ℓ	±h	±k
			-2	+k	±ℓ	±h		-k	±ℓ	±h
1	2/m	1/4	0	h	k	±ℓ		-h	k	±ℓ
			-1	ℓ	±h	k		-ℓ	±h	k
			-2	k	±ℓ	h		-k	±ℓ	h
2	mmm	1/8	0	h	k	ℓ		-h	k	ℓ
	4/m	1/8	-1	ℓ	h	k		-ℓ	h	k
	6/m	1/2 or 1/6	-2	k	ℓ	h		-k	ℓ	h
3	$\bar{3}$	1/6	0	h	±k	±ℓ	$\lvert h\rvert = \min(\lvert h\rvert, \lvert k\rvert, \lvert \ell\rvert)$	-h	±k	±ℓ
	$\bar{3}$m {Twice unique set}	1/6	-1	ℓ	±h	±k	$\lvert \ell\rvert = \min(\lvert h\rvert, \lvert k\rvert, \lvert \ell\rvert)$	-ℓ	±h	±k
			-2	k	±ℓ	±h	$\lvert k\rvert = \min(\lvert h\rvert, \lvert k\rvert, \lvert \ell\rvert)$	-k	±ℓ	±h
4	4/mmm	1/16	0	h	k	ℓ		-h	k	ℓ
			-1	ℓ	h	k	$\lvert h\rvert \leq \lvert k\rvert$	-ℓ	h	k
			-2	k	ℓ	h		-k	ℓ	h
5	m3	1/24	0	h	k	ℓ	$\lvert h\rvert = \min(\lvert h\rvert, \lvert k\rvert, \lvert \ell\rvert)$	-h	k	ℓ
	m3m {Twice unique set}	1/24	-1	ℓ	h	k	$\lvert \ell\rvert = \min(\lvert h\rvert, \lvert k\rvert, \lvert \ell\rvert)$	-ℓ	h	k
			-2	k	ℓ	h	$\lvert k\rvert = \min(\lvert h\rvert, \lvert k\rvert, \lvert \ell\rvert)$	-k	ℓ	h
6	6/mmm	1/24	0	h	k	ℓ	$\lvert h\rvert \leq \lvert k\rvert$	-h	k	ℓ
			-1	ℓ	h	k	$\lvert h\rvert \leq \lvert h+k\rvert$	-ℓ	h	k
			-2	k	ℓ	h		-k	ℓ	h

Control Integer 2 IC + 1

Parity. Parity bit for each axis is 0 conventions in Appendix 1 for right-handed machine are followed. 1 if reversed. Parity is applied to display values of reference reflections and to calculated angles. If three reference reflections are used, an internally consistent system will be obtained for any parity setting but parity bits appropriate to the particular diffractometer are recommended.

The parity bits may be used with care to collect different data sets for special orientations of the crystal, but never reverse 2θ on the neutron machines.

Recommended procedure:

(1) Insert appropriate parity bits in program.

(2) For display reference reflections, insert display values.

(3) For true reference reflections inset values corresponding to conventional Hamilton +++ convention. These are obtained from display value by subtracting zero correction and changing signs as demanded by parity.

For 2 reference reflections, it is very important to follow these instructions, for a right-handed coordinate system is assumed.

For 3 reference reflections, a self-consistent set is always obtained, but it is nevertheless best to follow the standard procedure above.

If the orientation matrix is used in the absorption programs, it will then always have the same meaning.

The following parity bits are recommended:

H6S3	0011	χ positive, ω and ϕ negative
H6M4	1011	pure negative machine
XRY7	1011	pure negative machine

Table 3

Sense Indicators for Axis Parity

BIN	OCT	DEC	AXIS
0000	0	0	NONE
0001	1	1	Φ
0010	2	2	ω
0100	4	4	2θ
1000	10	8	χ
0011	3	3	Φ, ω
0101	5	5	$\Phi, 2\theta$
0110	6	6	$\omega, 2\theta$
0111	7	7	$\Phi, \omega, 2\theta$
1001	11	9	Φ, χ
1010	12	10	ω, χ
1011	13	11	Φ, ω, χ
1100	14	12	$2\theta, \chi$
1101	15	13	$\Phi, 2\theta, \chi$
1110	16	14	$\omega, 2\theta, \chi$
1111	17	15	$\Phi, \omega, 2\theta, \chi$

Control Integer 4 IC+3

Systematic Extinctions

Provision has been made for systematic extinctions due to lattice centering. No provision has been made for extinctions due to screw axes or glide planes as these are much less numerous and their specification would be rather more complicated.

Table 4

Systematic Extinctions for Centered Cells

BIN	OCTAL	DEC	Type of Centering
0000	0	0	none
0001	1	1	A
0010	2	2	B
0100	4	4	C
0111	7	7	F
1000	10	8	I
1111	17	15	ggg Only*

* Only those reflections for which h, k, ℓ are all even will be collected.

Control Integer 5 IC+4

Variable Scan Interval

It is often necessary to vary the scan range as a function of the scattering angle. Three modes of operation are available (see Table 1).

1. Fixed scan mode.

In this mode the scan interval is set by placing the desired total range in the location SCNDEL. The range is in degrees x 100.

2. Cosine variation

The minimum scan range is inserted into STDSCN. The requisite scan as computed from formula (1) will be inserted by the program into SCNDEL.

$$\Delta 2\theta = \alpha/\cos\theta \tag{1}$$

where $\Delta 2\theta$ is the scan range and α is the minimum scan range (STDSCN).

3. In the tangent variation mode the minimum range is again inserted into STDSCN. In addition the factor $\mathcal{K}$ in eq. 2. must be inserted in double precision decimal into FACTØR.

$$\Delta 2\theta = \alpha + \alpha\,\mathcal{K}\,\mathrm{Tan}\theta \tag{2}$$

The third mode (tangent variation) is quite flexible and has been found to give satisfactory results. These constants are usually determined empirically with the aid of the display.

As an example, in one problem on the H6S3, we found that $\alpha = 300$, $\mathcal{K} = 2.0$ gave satisfactory results.

Control Integer 6 IC+5

Axis Setting Optimization

If desired the scanning axis plus one other can be optimized before proceeding with the step scan. For example, it might be desired to optimize phi before performing a θ-2θ scan. This can be accomplished by setting the control integer (IC+5) to zero (see Table 6).

Often it is important to set the scan axis to an optimum setting before optimizing any of the other axes. Eg. if the program is in the θ-2θ step scan mode and if optimization on phi is desired, it may be necessary to first set 2θ to an optimum position. This is accomplished by "resetting" the sign bit in the control integer. For the control integer IC+5 = 40000000 (octal) the optimization sequence is:

1) Optimize Scan axis (IC+6)
2) Optimize Phi
3) Scan axis given by (IC+6)

The following parameters are pertinent:

OPTSCN	optimization scan length
OPTSTP	optimization step size

Optimization Options

For a Single Optimization

0	Optimize Phi before data scan
1	" Omega " " "
2	" Two-Theta " " "
3	" Chi " " "
4	No optimization

Optimization is particularly useful in emergency situations when crystal orientation is sloppy or encoders are giving trouble.

Control integer 7 IC+6

Scan Mode

There are four scan modes available, one for each axis. The most useful of course will be the omega and theta/two-theta scans. The phi scan will usually be used only for optimization. The scan mode is set by control integer (IC+6).

A peak height mode for IC+6 = 4 is now available. Counting will be at a single point.

0 ϕ scan

4 peak height only

2 θ, 2θ scan

1 ω scan

In the peak height mode, count is taken at $(\theta, 2\theta)_0$ and $(\theta, 2\theta)_0 \pm \Delta/2$, i.e. it is a three-point $(\theta, 2\theta)$ scan. For a single point at calculated position only use θ - 2θ scan mode with a SCNDEL = 0 and STEP > 0.

Control Integer 8 IC+7

0 Reference reflections as input from display

1 True values of reference reflection.

See pages 8, 9 and 15.

Control Integer 9 IC+8

Output Options

The standard output device for the MSCS system is magnetic tape. There are, however, two other forms of output which may be useful for monitoring the data. Limited output on the CRT and/or typewriter can be obtained by specifying IC + 8 as indicated below.

To operate in the 'tape only' mode the control integer (IC+8) is set to zero. If while operating in this mode a typeout is desired the breakpoint 'd' can be thrown up . With breakpoint 'd' up the (IC+8) = 0 mode is equivalent to the (IC+8) = 1 mode, i.e. type plus tape output.

Integer	Result
0	Tape output with an option for typing if breakpoint (D) is in * UP * position.
1	Tape + Type output
2	Type only
3	Display + Tape + Type output
4	Display + Type output
5	Display + Tape with an option for typing if breakpoint (D) is in the * UP * position.

We normally use #5 when collecting data.

Control Integer 10 IC+9

Angle Settings Check

The angle settings check consists of two options used in conjunction with the sequence option. With the sequence integer (IC+2) set at a positive value the program will terminate after printing out the setting angles. With the angle setting check integer (IC+9) set at (2) the absolute or "true" settings will be output. With the integer set at (1) the corrected (or display) values of the settings will be output. A zero value for this integer will result in the setting and scan of the current reflection with an output of the results after completion of the run.

If the angle settings check integer is zero at the time the program is loaded the majority of the input requests and intermediate output will be bypassed.

Magnetic Tape Output Data List

When the program is started from scratch in location 0001 or 0000 a request is made for the data and crystal number. When the program is restarted in the requested location (DATER) the tape label will be set up.

tape label

ANDA0806 05I2

PROGRAM DAY MONTH

These are set up in the locations CØDEN, DATA, CØDE.

The magnetic tape output consists of one record. The output list is given below:

Physical	Logical			
11	1	PROGRAM NAME	BCI	LABEL
12	2	MONTH/DAY	BCI	LABEL
13	3	XTAL/I NUMBER	BCI	LABEL
	4	H	h	
	5	K	k	
	6	L	l	
	7	STEP	Step interval	
	8	STPTIM	Monitor setting for step	
	9	BKGTIM	Number of steps for background	
	10	SCNDEL	Scan range	
	11	ATSCL(ATTN)	Attenuator scale factor	
	12	NWØRDS	Points in the step scan	

13	SIGNAL	Integrated Intensity
14	BACKRN	Scaled background
15	PHI	Ø
16	ØHM	ω
17	TWØTHE	2θ
18	CHI	χ
19	DSTAR(A)	Double precision
20	DSTAR(B)	D^* in binary form
21	INDEX	Reflection data count
22	ARRAY	First word in the array
•		
•		
•		
21+NWORDS	ARRAY+NWORDS-1	Last scan step
•		
•		
•		
21+NMAX		Maximum array size
+1	TOTAL(a)	Double precision
+2	TOTAL(b)	scan total.
•		
•		
•		

*****NOTE**** This is an output data LIST only, the actual physical record on tape will contain control words and a padded-out 400 word record. Please check the MSCS manual and newsletters for details on the physical tape format.

These tapes are processed using the sequence: ØRACLE, PRØCH, DATAPH, SØRTH

Display Output

The display output enables the operator to monitor at once the entire step scan. The data array is scaled so that the maximum point is on the top of the screen and so that the first and last points fill the screen horizontally. Therefore, the intensity of the peak must be judged from the statistics or the labeled intensity.

The label on the display contains, H,K,L, SIGNAL and BACKRN, where (SIGNAL) = Integrated signal

The display is observed by placing the CRT in storage mode with the control integer (IC+8) set at 3, 4 or 5.

Appendix 1

Hamilton's notes on diffractometer settings. pp 27-38

Appendix 2

Bednowitz' old notes on setting angles pp 39-44

Appendix 3

Library routines pp 45-60

Appendix 4

MSCS mnemonics.

Appendix 5

Sample of assembled data input.

Appendix 6

Some sample output

Appendix 7

Some photographs

Appendix 8

Assembly of programs

Appendix 9

Some miscellaneous comments.

Appendix 10

Some auxiliary programs.

APPENDIX 1

Angle Settings for Four-circle Diffractometers

Walter C. Hamilton

I. The Diffractometer

The conventional four-circle diffractometer (Figure 1) may be defined as follows: The three points--source, crystal, and detector--define a plane which we may call the diffraction plane. The axis passing through the crystal and perpendicular to this ~~axis~~ plane is the main axis of the instrument. The detector rotates around this axis on a circle of constant radius. The angle subtended at the crystal by the source and detector is $180 - 2\theta^{\circ}$, the complement of twice the Bragg angle 2θ; we may thus call the main axis the 2θ axis. The bisector of this angle is the direction of the diffraction vector.

We now define an axis passing through the crystal and parallel to the plane of diffraction; this is the χ-rotation axis. A third axis passing through the crystal corresponds to the spindle axis of the goniometer head and is the ϕ axis. The ϕ axis makes an angle χ with the main axis of the instrument; the goniometer head thus may travel in a circle--the χ circle perpendicular to the χ axis. A fourth angle ω defines the angle between the plane of the χ circle and the diffraction vector; the ω axis is coincident with the 2θ axis. In the typical three circle diffractometer, ω is always zero so that the χ circle always lies in the perpendicular plane which includes the diffraction vector.

It is useful to adopt conventions concerning the sense of rotation of the several angles. These conventions are unimportant in the development that follows as long as internal consistency is maintained. The engraved scales or odometers on existing instruments correspond to all possible choices

of axis parities. One common convention is as follows

ω is positive if its rotation direction is opposite to that of 2θ.

χ is positive if it rotates counter-clockwise when viewed from the direction of the incident beam when $\omega = 0$.

ϕ is positive if its rotation is opposite to that of 2θ when $\chi = 0$.

We may define a fifth angle ψ --the azimuthal angle around the diffraction vector. Few diffractometers have been built where this corresponds to a physical axis. It is often convenient to rotate around this axis by a combination of motions of the other angles.

II. Coordinate Systems

Let us define the following coordinate systems:

A. An orthonormal system $\underset{\sim}{A}_D$ fixed to the diffractometer is the following way:

$\underset{\sim}{b}_D$ is parallel to the diffraction vector and hence bisects the angle $(180 - 2\theta)$ between the incident and diffracted beams.

$\underset{\sim}{a}_D$ is directed toward the source from the crystal when $2\theta = 0$. (1)

$\underset{\sim}{c}_D$ is coincident with the main axis of the instrument and has a direction such that

$$\underset{\sim}{A}_D \equiv (\underset{\sim}{a}_D, \underset{\sim}{b}_D, \underset{\sim}{c}_D)$$

is a right-handed system.

B. An orthonormal system $\underset{\sim}{A}_G$ fixed in the goniometer head and hence with respect to the crystal such that $\underset{\sim}{c}_G$ is coincident with the phi axis. (2)

The system $\underset{\sim}{A}_G$ is coincident with $\underset{\sim}{A}_D$ when all the setting angles are zero. (2)

C. The reciprocal axis system of the crystal $\underset{\sim}{A}^*$ (3)

In matrix notation, we may write these as

$$\underset{\sim}{A}_D = \begin{pmatrix} \underset{\sim}{a}_D \\ \underset{\sim}{b}_D \\ \underset{\sim}{c}_D \end{pmatrix} \quad \text{Diffractometer fixed} \quad \text{with metric} \begin{pmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix} \tag{4}$$

$$\underset{\sim}{A}_G = \begin{pmatrix} \underset{\sim}{a}_G \\ \underset{\sim}{b}_G \\ \underset{\sim}{c}_G \end{pmatrix} \quad \text{Goniometer fixed} \quad \text{with metric} \begin{pmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix} \tag{5}$$

$$\underset{\sim}{A}^* = \begin{pmatrix} \underset{\sim}{a}^* \\ \underset{\sim}{b}^* \\ \underset{\sim}{c}^* \end{pmatrix} \quad \text{Crystal reciprocal axes} \quad \text{with metric } M^{-1} = \begin{pmatrix} a^{*2} & a^*b^*\cos\gamma^* & a^*c^*\cos\beta^* \\ a^*b^*\cos\gamma^* & b^{*2} & b^*c^*\cos\alpha^* \\ a^*c^*\cos\beta^* & b^*c^*\cos\alpha^* & c^{*2} \end{pmatrix} \tag{6}$$

The coordinates of a reciprocal vector in these systems are

$$\begin{aligned} \underset{\sim}{X}_G &= (x_g \; y_g \; z_g) \\ \underset{\sim}{X}_D &= (x_d \; y_d \; z_d) \\ \underset{\sim}{h} &= (h \; k \; \ell) \end{aligned} \tag{7}$$

such that a reciprocal vector $\vec{h}$ may be written

$$\vec{h} = \underset{\sim}{h}\underset{\sim}{A}^* = \underset{\sim}{X}_D\underset{\sim}{A}_D = \underset{\sim}{X}_G\underset{\sim}{A}_G \tag{8}$$

The systems $\underset{\sim}{A}_G$ and $\underset{\sim}{A}_D$ are identical when $\omega = \chi = \phi = 0$. When these angles are not zero

$$\underset{\sim}{A}_G = \underset{\sim}{F}\underset{\sim}{A}_D \tag{9}$$

where

$$\underset{\sim}{F} \equiv \underset{\sim}{R}_\phi \underset{\sim}{R}_\chi \underset{\sim}{R}_\omega \tag{10}$$

$$= \begin{pmatrix} \cos\phi & \sin\phi & 0 \\ -\sin\phi & \cos\phi & 0 \\ 0 & 0 & 1 \end{pmatrix} \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos\chi & \sin\chi \\ 0 & -\sin\chi & \cos\chi \end{pmatrix} \begin{pmatrix} \cos\omega & \sin\omega & 0 \\ -\sin\omega & \cos\omega & 0 \\ 0 & 0 & 1 \end{pmatrix} \tag{11}$$

It follows also that

$$\underset{\sim}{X}_G = \underset{\sim}{F}\underset{\sim}{X}_D \; . \tag{12}$$

Orientation

It is convenient to introduce an orientation matrix U such that

$$\underset{\sim}{A}^* = \underset{\sim}{U}\underset{\sim}{A}_G \tag{13}$$

[Note that $UU' = M^{-1}$ (14)

from which all parameters may be drawn]

The matrix U (which is not necessarily symmetric) has nine independent elements which are related to the six cell constants and three angles describing the crystal orientation.

Conditions for a reciprocal vector to be in diffracting position.

If ω, χ, ϕ are such that the diffraction conditions are met, then

$$\underset{\sim}{X}_D = \begin{pmatrix} 0 \\ d^* \\ 0 \end{pmatrix} \tag{15}$$

and

$$\underset{\sim}{X}_G = d^* \begin{pmatrix} \cos\phi \ \sin\omega + \sin\phi \cos\chi \cos\omega \\ -\sin\phi \ \sin\omega + \cos\phi \cos\chi \cos\omega \\ -\sin\chi \cos\omega \end{pmatrix} \tag{16}$$

Since

$$\underset{\sim}{H}\underset{\sim}{A}^* = \underset{\sim}{H}\underset{\sim}{U}\underset{\sim}{A}_G \tag{17}$$

and

$$\underset{\sim}{H}\underset{\sim}{A}^* = \underset{\sim}{X}_G\underset{\sim}{A}_G \tag{18}$$

we have

$$\underset{\sim}{H}\underset{\sim}{U} \equiv \underset{\sim}{X}_G \tag{19}$$

Knowing $\underset{\sim}{H}$ and $\underset{\sim}{U}$, we can calculate the components of $\underset{\sim}{X}_G$ and hence the setting angles. One of the angles is redundant and may be varied to provide rotation around the scattering vector. Table 1 gives eight alternate settings for observing a given reflection in a given aspect.

Table 1. Various setting for observing a reflection on a four-circle diffractometer.

I	χ	ϕ	2θ	ω
II	$-\chi$	$180 + \phi$	2θ	$180 + \omega$
III	$180 + \chi$	ϕ	-2θ	$-\omega$
IV	$180 - \chi$	$180 + \phi$	-2θ	$180 - \omega$
V	$180 - \chi$	$180 + \phi$	$+2\theta$	$-\omega$
VI	$180 + \chi$	ϕ	2θ	$180 - \omega$
VII	$-\chi$	$180 + \phi$	-2θ	ω
VIII	χ	ϕ	-2θ	$180 + \omega$

Each of the settings above is for the (h,k,ℓ) reflection. The $(-h, -k, -\ell)$ reflection is obtained in each case by either reversing the sign of 2θ or by adding 180° to ω.

Methods of Solution

In the calculation of setting angles, particularly when programming algorithms for a digital computer, care must be taken with regard to special values of all of the angles and to principal values defined in the trigonometric function routines used. The effect of inaccuracy in the function $\sin^{-1}\chi$ and $\cos^{-1}\chi$ for values of χ near 1 must also be guarded against. It is usually most convenient to calculate χ and ϕ as a function of ω. It is convenient to define a unit vector

$$\underset{\sim}{T} = \frac{1}{d^*} \underset{\sim}{X}_G \qquad (20)$$

with components $(t_1\ t_2\ t_3)$.

For $\omega \neq 90^\circ$, equation (16) may be conveniently solved for the setting angles as follows:

$$\sin\chi = -t_3/\cos\omega \qquad (21)$$

(If $t_3 > \cos\omega$, the reflection cannot be observed for the chosen value of ω.) With no loss of generality, we choose as the standard solution that which places χ in the range

$$-\pi/2 \leq \chi \leq \pi/2$$

ϕ may now be obtained as follows:

Define $q = \sin\omega$ and $r = \cos\omega\cos\chi$ (22)

Then

$$\cos\phi = (qt_1 + rt_2)/(q^2 + r^2) \qquad (23)$$

$$\sin\phi = (t_1 - q\cos\phi)/r$$

ϕ may cover a complete circle.

(Since $q^2 + r^2 = t_1^2 + t_2^2$, a solution for ϕ is always possible.)

If $\omega = 90^{\circ}$, a different solution is required since $\cos\omega = 0$.

$$\begin{aligned} \cos\phi &= t_1 \\ \sin\phi &= -t_2 \\ (t_3 &= 0) \end{aligned} \tag{24}$$

Only one plane of reciprocal space may be examined. For values of ω near 90°, a more convenient solution is obtained by setting $\chi = 90^{\circ}$ and calculating ω and ϕ from

$$\begin{aligned} \cos\omega &= -t_3 \\ \sin\phi \sin\omega &= t_2 \\ \cos\phi \sin\omega &= t_1 \end{aligned} \tag{25}$$

Rotation around the diffraction vector ψ

Rotation of the crystal around the diffraction vector is sometimes of interest.

Define an angle of rotation ψ about the diffraction vector. This may lie between 0 and 2π. Define χ_0 and ϕ_0 to be the angles at which the crystal diffracts for $\omega = 0$ and for $\psi = 0$. The following equations may be used in a variety of ways to determine the settings for other values of ψ.

Let

$$\begin{aligned} k_1 &= \cos\chi_0 \sin\phi \\ k_2 &= \cos\chi_0 \cos\phi_0 \\ k_3 &= -\sin\chi_0 \\ k_4 &= \sin\chi_0 \sin\phi_0 \\ k_5 &= \sin\chi_0 \cos\phi_0 \\ k_6 &= \cos\chi_0 \end{aligned} \tag{26}$$

Then

$$\sin\omega\cos\phi + \cos\omega\cos\chi\sin\phi = k_1 \tag{27}$$

$$-\sin\omega\sin\phi + \cos\omega\cos\chi\cos\phi = k_2 \tag{28}$$

$$-\cos\omega\sin\chi = k_3 \tag{29}$$

$$-\sin\psi(\cos\omega\cos\phi - \sin\omega\cos\chi\sin\phi) + \cos\psi\sin\chi\sin\phi = k_4 \tag{30}$$

$$\sin\psi(\cos\omega\sin\phi + \sin\omega\cos\chi\cos\phi) + \cos\psi\sin\chi\cos\phi = k_5 \tag{31}$$

$$-\sin\psi\sin\omega\sin\chi - \cos\psi\cos\chi = k_6 \tag{32}$$

One way of using these equations is as follows: Combination of equations (27), (28), (30), (31) results in

$$\tan\chi_0 \tan\omega = -\sin\psi. \tag{33}$$

This may be used to solve for ω. Then ϕ may be obtained from one of the following:

$$\sin\omega = \cos\chi_0 \sin(\phi_0 - \phi) \tag{34}$$

$$-\sin\psi\cos\omega = \sin\chi_0 \sin(\phi_0 - \phi) \tag{35}$$

Finally χ may be obtained from

$$\cos\omega\sin\chi = \sin\chi_0 \tag{36}$$

An alternative way of solving the equations is to solve equation (33) for and then return to the formalism beginning on page (5) for calculating ϕ and χ, given ω.

Determination of U, the orientation matrix

At any value of ω, measurement of χ, ϕ, and d^* for a reflection $\underset{\sim}{h}$ is a measurement of three independent quantities which may be used in the determination of U. The measurement of three non-coplanar reflections

thus serves to completely determine U, even when the cell constants are completely unknown.

For three reflections $\underset{\sim}{h}_1$, $\underset{\sim}{h}_2$, $\underset{\sim}{h}_3$ we have

$$\begin{aligned} \underset{\sim}{h}_1\underset{\sim}{A}^* &= \underset{\sim}{X}_{G1}\underset{\sim}{A}_G \\ \underset{\sim}{h}_2\underset{\sim}{A}^* &= \underset{\sim}{X}_{G2}\underset{\sim}{A}_G \\ \underset{\sim}{h}_3\underset{\sim}{A}^* &= \underset{\sim}{X}_{G3}\underset{\sim}{A}_G \end{aligned} \tag{37}$$

Let us define the following matrices

$$\underset{\sim}{H} = \begin{pmatrix} \underset{\sim}{h}_1 \\ \underset{\sim}{h}_2 \\ \underset{\sim}{h}_3 \end{pmatrix} \equiv \begin{pmatrix} h_1 & k_1 & \ell_1 \\ h_2 & k_2 & \ell_2 \\ h_3 & k_3 & \ell_3 \end{pmatrix} \tag{38}$$

$$\underset{\sim}{X} = \begin{pmatrix} X_{G1} \\ X_{G2} \\ X_{G3} \end{pmatrix} \equiv \begin{pmatrix} x_1 & y_1 & z_1 \\ x_2 & y_2 & z_1 \\ x_3 & y_3 & z_3 \end{pmatrix} \tag{39}$$

Then

$$\begin{aligned} \underset{\sim}{H}\underset{\sim}{A}^* &= \underset{\sim}{X}\underset{\sim}{A}_G \\ \underset{\sim}{A}^* &= \underset{\sim}{H}^{-1}\underset{\sim}{X}\underset{\sim}{A}_G \end{aligned} \tag{40}$$

But since

$$\underset{\sim}{A}^* = \underset{\sim}{U}\underset{\sim}{A}_G \tag{41}$$

we have

$$\underset{\sim}{U} = \underset{\sim}{H}^{-1}\underset{\sim}{X} \tag{42}$$

The matrix $\underset{\sim}{X}$ depends only on ϕ, χ, ω and d^* for the three reflections. The matrix $\underset{\sim}{H}$ depends only on the indices. Hence we may solve for $\underset{\sim}{U}$. If some account of known cell constants is to be taken, calculated rather than observed values of d^* may be used in the elements of $\underset{\sim}{X}$.

Determination from two reflections.

It is sometimes convenient when the cell constants are known to specify the crystal orientation by data from two reflections. (Two reflections actually give us 6 pieces of data--three cell constants and three orientation angles. The three cell constants are the lengths of the two reciprocal vectors and the angle between them.) One way of using these two reflections is to supplement the two by taking the cross products

$$\begin{aligned} \underset{\sim}{h}_3 &= \underset{\sim}{h}_1 \times \underset{\sim}{h}_2 \\ \underset{\sim}{X}_{G3} &= \underset{\sim}{X}_{G1} \times \underset{\sim}{X}_{G2} \end{aligned} \tag{43}$$

and using the vectors $\underset{\sim}{h}_1$, $\underset{\sim}{h}_2$, $\underset{\sim}{h}_3$, $\underset{\sim}{X}_{G1}$, $\underset{\sim}{X}_{G2}$, $\underset{\sim}{X}_{G3}$ as in the formalism when three vectors are actually observed. The known cell constants serve to determine the lengths of the three vectors, but this approach will fit the direction of the two vectors exactly, so that if the angle between the vectors is not in agreement with the cell constants, the observations will prevail.

An alternative approach (suggested by Busing and Levy) which maintains the cell constants as given is to take the following vector set as the three reflection input to the general procedure

$$\begin{aligned} \underset{\sim}{h}_1 &= \underset{\sim}{h}_1(\text{obs}) \\ \underset{\sim}{h}_3 &= \underset{\sim}{h}_1(\text{obs}) \times \underset{\sim}{h}_2(\text{obs}) \\ \underset{\sim}{h}_2 &= \underset{\sim}{h}_3 \times \underset{\sim}{h}_1 \end{aligned} \tag{44}$$

Least Squares Refinement

It is generally useful to refine the components of $\underset{\sim}{U}$ by a least squares procedure when the quantities χ, ϕ, d^* have been observed for several reflections. Rather than directly refine the elements of $\underset{\sim}{U}$, it

seems most interesting to refine the six cell constants and three orientation parameters. The three orientation parameters are conveniently taken as three angles ω_s, χ_s, ϕ_s through which the crystal must be rotated from the orientation when $\omega = \chi = \phi = 0$ to obtain some standard orientation. A convenient standard orientation and corresponding set of orientation angles may be defined as follows:

Consider an orthogonal axis system $\underset{\sim}{A}_o$ fixed in the crystal such that

$\underset{\sim}{a}_o$ is in the direction of $\underset{\sim}{b}_o \times \underset{\sim}{c}_o$

$\underset{\sim}{b}_o$ is in the direction of $\underset{\sim}{b}^*$

$\underset{\sim}{c}_o$ is in the direction of $\underset{\sim}{a}^* \times \underset{\sim}{b}^* = \underset{\sim}{c}$

We may write

$$\underset{\sim}{A}^* = \underset{\sim}{V}\underset{\sim}{A}_o \tag{45}$$

where

$$\underset{\sim}{V} = \begin{bmatrix} a^* \sin\gamma^* & a^* \cos\gamma^* & 0 \\ 0 & b^* & 0 \\ (a^* c^* \cos\beta^* - v_{12}v_{32})/v_{11} & c^* \cos\alpha^* & (c^{*2} - v_{32}^2 - v_{31}^2)^{1/2} \end{bmatrix} \tag{46}$$

Starting with the crystal mounted on the goniometer head and $\phi = \chi = \omega = 0$ consider three rotations which bring the orthogonal system $\underset{\sim}{A}_o$ into coincidence with the diffractometer system A_D. First a rotation ϕ_s about ϕ which brings $\underset{\sim}{c}_o$ into the plane of the χ circle, secondly a rotation χ_s about the χ axis to bring $\underset{\sim}{c}_o$ vertical, then a rotation ω_s about the ω axis to bring $\underset{\sim}{b}_o$ coincident with $\underset{\sim}{b}_D$. With the orthogonal coordinate system defined as we have done here, this places the crystal such that the (0k0) reflections are in the diffraction position, and with the (hk0) plane parallel to the plane of the diffractometer. We may then write

$$\underset{\sim}{A}_G = \underset{\sim}{F}_s \underset{\sim}{A}_D$$

where

$$\underset{\sim}{F}_s = \underset{\sim}{R}_{\phi_s} \underset{\sim}{R}_{\chi_s} \underset{\sim}{R}_{\omega_s} \tag{47}$$

as defined in equations (10) and (11).

After the rotation

$$\underset{\sim}{A}_G = \underset{\sim}{F}_s \underset{\sim}{A}_D \tag{48}$$

and

$$\underset{\sim}{A}^* = \underset{\sim}{U}\underset{\sim}{F}_s \underset{\sim}{A}_D \tag{49}$$

Now

$$\underset{\sim}{A}^* = \underset{\sim}{V}\underset{\sim}{A}_o \tag{50}$$

But under these circumstances,

$$\underset{\sim}{A}_o = \underset{\sim}{A}_D \tag{51}$$

Hence

$$\underset{\sim}{V} = \underset{\sim}{U}\underset{\sim}{F}_s \tag{52}$$

and

$$\underset{\sim}{U} = \underset{\sim}{V}\underset{\sim}{F}_s^{-1} \tag{53}$$

Knowing $\underset{\sim}{V}$ and $\underset{\sim}{F}_s$ we can determine $\underset{\sim}{U}$.

But also knowing $\underset{\sim}{V}$ and $\underset{\sim}{U}$, we may determine $\underset{\sim}{F}_s$ by

$$\underset{\sim}{F}_s = \underset{\sim}{U}^{-1}\underset{\sim}{V} \tag{54}$$

and hence the orientation angles may be obtained when $\underset{\sim}{U}$ is known.

Appendix 2. Bednowitz original notes on U matrix determination. Note that he normalizes and transposes U with respect to Hamilton definition.

Definition #1

Crystal Lattice

Oblique system defined by a, b, c, cosa, cosb, cosc

Definition #2

Reciprocal Lattice: $a^*, b^*, c^*, \cos a^*, \cos b^*, \cos c^*$

Right handed oblique system inverse to the crystal lattice.

Definition #3

Real Metric: $\underline{\underline{G}}$

$$= \begin{pmatrix} a^2 & a\,b\,\cos c & a\,c\,\cos b \\ ab\cos c & b^2 & bc\cos a \\ ac\cos b & cb\cos a & c^2 \end{pmatrix}$$

Definition #4

Reciprocal Lattice Metric: $\underline{\underline{G}}^{-1}$

$$= \begin{pmatrix} a^{*2} & a^*b^*\cos c^* & a^*c^*\cos b^* \\ a^*b^*\cos c^* & b^{*2} & b^*c^*\cos a^* \\ a^*c^*\cos b^* & b^*c^*\cos a^* & c^{*2} \end{pmatrix}$$

Definition #5

Length of Reciprocal vector: $d^{*2} = \underline{H}\,\underline{\underline{G}}^{-1}\underline{H}^{\tau}$

Where $\underline{H} = (h,k,l)$

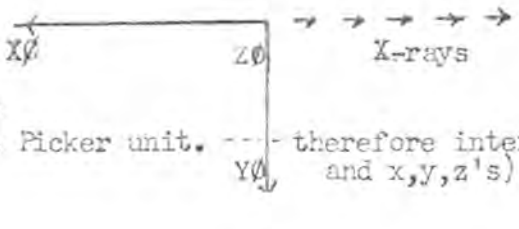
Definition #6

Spectrometer-Fixed Coordinate Axes: XØ, YØ, ZØ

XØ is along the X-ray beam when $\theta = 0$, with the rays traveling (-).

ZØ is coincident with the diffractometer main axis.

YØ = ZØ × XØ



ϕ, ω, χ are positive clockwise on Picker unit. --- therefore internal results (U-matrix, and x,y,z's) dont agree with definiti

Definition #7 x, y, z, are the

coordinates of a reciprocal lattice point in the XØ, YØ, ZØ system

When Chi = 0, Phi = 0, omega = 0, and theta = 0.

Definition #8

Rotation matrix relating (GONI) and (DIFF) coordinates:

$$\begin{pmatrix} x \\ y \\ z \end{pmatrix} = \underline{\underline{F}} \begin{pmatrix} X\emptyset \\ Y\emptyset \\ Z\emptyset \end{pmatrix} = R_{\emptyset} R_{\chi} R_{\omega} \begin{pmatrix} 0. \\ d^{*} \\ 0. \end{pmatrix}$$

Definition #9

The three Eulerian rotation matrices:

$$R_{\omega} = \begin{pmatrix} \cos\omega & \sin\omega & 0. \\ -\sin\omega & \cos\omega & 0. \\ 0. & 0. & 1 \end{pmatrix}, \quad \begin{pmatrix} x \\ y \\ z \end{pmatrix} = R_{\omega} \begin{pmatrix} X\emptyset \\ Y\emptyset \\ Z\emptyset \end{pmatrix}$$

$$R_{\chi} = \begin{pmatrix} 1. & 0. & 0. \\ 0. & \cos\chi & \sin\chi \\ 0. & -\sin\chi & \cos\chi \end{pmatrix}, \quad \begin{pmatrix} x \\ y \\ z \end{pmatrix} = R_{\chi} \begin{pmatrix} X\emptyset \\ Y\emptyset \\ Z\emptyset \end{pmatrix}$$

$$R_{\emptyset} = \begin{pmatrix} \cos\emptyset & \sin\emptyset & 0. \\ -\sin\emptyset & \cos\emptyset & 0. \\ 0. & 0. & 1. \end{pmatrix}, \quad \begin{pmatrix} x \\ y \\ z \end{pmatrix} = R_{\emptyset} \begin{pmatrix} X\emptyset \\ Y\emptyset \\ Z\emptyset \end{pmatrix}$$

$$R_{\chi} R_{\omega} = \begin{pmatrix} C_{\omega} & S_{\omega} & 0. \\ -C_{\chi} S_{\omega} & C_{\chi} C_{\omega} & S_{\chi} \\ S_{\chi} S_{\omega} & -S_{\chi} C_{\omega} & C_{\chi} \end{pmatrix}$$

$$R_\phi R_\chi R_\omega = \begin{pmatrix} C_\phi C_\omega - S_\phi C_\chi S_\omega & C_\phi S_\omega + S_\phi C_\chi C_\omega & S_\phi S_\chi \\ -S_\phi C_\omega - C_\phi C_\chi S_\omega & -S_\phi S_\omega + C_\phi C_\chi C_\omega & C_\phi S_\chi \\ S_\chi S_\omega & -S_\chi C_\omega & C_\chi \end{pmatrix}$$

$$\frac{x}{d^*} = \cos\phi \sin\omega + \sin\phi \cos\chi \cos\omega = t_1$$

$$\frac{y}{d^*} = -\sin\phi \sin\omega + \cos\phi \cos\chi \cos\omega = t_2$$

$$\frac{z}{d^*} = -\sin\chi \cos\omega = t_3$$

$$\sin\chi = -t_3 / \cos\omega \qquad \cos\omega \geqq t_3$$

Let $q = \sin\omega$ $\qquad r = \cos\omega \cos\chi$

then $\cos\phi = (qt_1 + rt_2)/(q^2 + r^2)$

$\sin\phi = (t_1 - q\cos\phi) / r$

Computation of the Orientation Matrix

The U matrix relates the reciprocal lattice coordinates (equation 1) with a set of Cartesian coordinates fixed in the goniometer head (see definitions in Appendix I)

The definition of the U matrix is given by,

$$\begin{pmatrix} x \\ y \\ z \end{pmatrix} = \underset{\sim}{U} \begin{pmatrix} h|a^*| \\ k|b^*| \\ \ell|c^*| \end{pmatrix} \tag{2}$$

The relationship between the coordinate axes is thus,

$$\overbrace{(\underline{X},\underline{Y},\underline{Z})}^{\text{unit axes fixed in head}} = \underbrace{\left(\frac{\underline{a}^*}{|a^*|}, \frac{\underline{b}^*}{|b^*|}, \frac{\underline{c}^*}{|c^*|} \right)}_{\text{normalized reciprocal axes.}} \underset{\sim}{U}^{-1} \quad . \tag{3}$$

Upon post multiplying by U we obtain,

$$(\underline{X},\underline{Y},\underline{Z})\, \underset{\sim}{U} = \left(\frac{\underline{a}^*}{|a^*|}, \frac{\underline{b}^*}{|b^*|}, \frac{\underline{c}^*}{|c^*|} \right) \tag{4}$$

where X, Y, Z are the unit vectors.

Taking the transpose of each side results in the relation (5)

$$\underset{\sim}{U}^{\tau} \begin{pmatrix} \underline{X} \\ \underline{Y} \\ \underline{Z} \end{pmatrix} = \begin{pmatrix} \dfrac{\underline{a}^*}{|a^*|} \\ \dfrac{\underline{b}^*}{|b^*|} \\ \dfrac{\underline{c}^*}{|c^*|} \end{pmatrix} \tag{5}$$

For an arbitrary reciprocal lattice vector h we know by definition,

$$\underline{h} = (h,k,\ell)\begin{pmatrix}\underline{a}^* \\ \underline{b}^* \\ \underline{c}^*\end{pmatrix} \equiv (x,y,z)\begin{pmatrix}\underline{X} \\ \underline{Y} \\ \underline{Z}\end{pmatrix} \tag{6}$$

where x,y and z are the coordinates of the reciprocal vector in the X,Y,Z system.

For our three independent reference reflections we can set up the matrix relation (7).

$$\underset{\sim}{H}\begin{pmatrix}\underline{a}^* \\ b^* \\ c^*\end{pmatrix} = \begin{pmatrix}h_1k_1\ell_1 \\ h_2k_2\ell_2 \\ h_3k_3\ell_3\end{pmatrix}\begin{pmatrix}\underline{a}^* \\ \underline{b}^* \\ \underline{c}^*\end{pmatrix} = \begin{pmatrix}x_1y_1z_1 \\ x_2y_2z_2 \\ x_3y_3z_3\end{pmatrix}\begin{pmatrix}\underline{X} \\ \underline{Y} \\ \underline{Z}\end{pmatrix} \tag{7}$$

Multiplying both sides of (7) by $\underset{\sim}{H}^{-1}$

$$\begin{pmatrix}\underline{a}^* \\ \underline{b}^* \\ \underline{c}^*\end{pmatrix} = \underset{\sim}{H}^{-1}\begin{pmatrix}x_1y_1z_1 \\ x_2y_2z_2 \\ x_3y_3z_3\end{pmatrix}\begin{pmatrix}\underline{X} \\ \underline{Y} \\ \underline{Z}\end{pmatrix} \tag{8}$$

$$= \underset{\sim}{W}\begin{pmatrix}X \\ Y \\ Z\end{pmatrix}$$

Normalizing the reciprocal axes we obtain

$$\begin{pmatrix}\frac{\omega_{11}}{|a^*|} & \frac{\omega_{12}}{|a^*|} & \frac{\omega_{13}}{|a^*|} \\ \frac{\omega_{21}}{|b^*|} & \frac{\omega_{22}}{|b^*|} & \frac{\omega_{33}}{|b^*|} \\ \frac{\omega_{31}}{|c^*|} & \frac{\omega_{32}}{|c^*|} & \frac{\omega_{33}}{|c^*|}\end{pmatrix}\begin{pmatrix}\underline{X} \\ \underline{Y} \\ \underline{Z}\end{pmatrix} = \begin{pmatrix}\frac{\underline{a}^*}{|a^*|} \\ \frac{\underline{b}^*}{|b^*|} \\ \frac{\underline{c}^*}{|c^*|}\end{pmatrix} \tag{9}$$

Appendix 3. Brief description of some library routines.

Utility Subroutines

Several utility subroutines (stored on the drum) are described briefly below.

a) DSCM - computes the 1/d value and the integer 2θ x 100 when given an integer set of hk ℓ values.

b) CSET - computes the chi and phi settings for a given omega value. The usual case is when omega is zero.

c) ØTØC - converts an octal integer to BCD in the (B), (A) registers. The least significant half of the integer will be in the (A) register. This subroutine differs from ØCTØ in that negative numbers are presented as decimal signed integers rather than in octal or complement forms.

d) UTIL - converts the H, K, L, Signal, and Background and settings data for typewriter and/or display output.

e) ARR Y - sets up an output array for double precision data. It is used to check the computations at the program startup. This routine is suppressed when (IC + 9) = 0.

f) INTN - computes and corrects the integrated count for scale factors and background. When OPTFLG is positive then this subroutine will find the peak maximum and adjust the value of each angle accordingly. If the calling sequence is headed by NOP DEGREE the peak maximum angle will be obtained from a smoothed step scan (subroutine SLUR is called).

g) CØMS - computes the scan parameters for the SCAN subroutine. It computes the scan length when the variable scan length option is used and the number of steps in the scan. Also computed are the beginning and end of the scan, and parameters for optimization runs. The order of the data words is important in several arrays. Included among these is the array begun by NWØRDS. (See Appendix III).

h) SCHK or CHKS - computes and applys the zero corrections to the 'true' setting angles. This subroutine also checks to see if any limits are in danger of being hit, then a flag is set to zero to so indicate that fact. Parity is applied here.

MESS and MES2 - To provide storage for an unlimited number of BCI messages. Two subroutines have been placed on the drum.

Calling Sequence: EAX NUMBER

CALL MESS or MES2

or

LDX NUMBER

CALL MESS or MES2

or

EAX 7

CALL MESS or MES2

NUMBER DEC Message Number, Eg. 7 There are two sequences one for MESS and another for MES2

Results: The location of the message address will be found in the (A) register. The location of the number of BCI words in the message will be in the (B) register. The actual address of the message will be in the (X) register.

(A) NØP Message Address Location

(B) NØP Location of (Decimal) Number of Words in Message

(X) NØP Address of the Message

B. IMPT - To set up the request messages for output on the typewriter.

Calling Sequence: NØP IM RETN

NØP MESSAG

NØP DATA - FIRST

NØP NUMBCI

```
NØP      RENTRY-FIRST
CALL     IMPT
```

Return from the IMPT routine is accomplished by a BRM* to the location containing the address of the typing routine (IMRETN).

```
MESSAG   PZE      MESSAGE
MESSAGE  BCI      INPUT REQUIREMENTS (14 words)
DATA     BSS
FIRST    BRU      RESTAR    LOCATION (0000) IN USER AREA
NUMDAT   BCI      1,NUM.    EG. -10-
RENTRY   NOP                RENTRY LOCATION
IMRETN   NOP      **        FIRST WORD OF TYPING ROUTINE
```

(See Appendix IV)

C. GCØM

Given three (or 2) reference reflections, GCØM will compute the orientation matrix $\underset{\sim}{U}$, the metric $\underset{\sim}{G}$, and new cell constants.

Calling Sequence:

```
NØP      IC
NØP      ZRCORR
NØP      HREF

NØP      AS
NØP      METRIC
NØP      UMTX
CALL     UCØM
SKG      ZERO
BRU      LØC. XXXX
CALL     MESS      ERRØR RETURN
BRM      IM RETN
```

ZRCORR	BSS	4	ZERØ CORRECTIONS
HREF	BSS	21	REFERENCE REFLECTION DATA
AS	BSS	12	RECIPRØCAL LATTICE CØNSTANTS
METRIC	BSS	18	RECIPRØCAL METRIC
UMTX	BSS	18	U MATRIX
IC	BSS	10	CØNTRØL SWITCH ARRAY
LØC. XXXX			NØRMAL RETURN FRØM THE SUBRØUTINE

An error indication is typed out if the orientation matrix cannot be computed.

D. DSCM

Computes 1/d and 2θ for integer hkℓ indices

Calling Sequence:

	NØP	TWØT
	NØP	WAVE
	NØP	METRIC
	NØP	H
	NØP	DSTAR
	CALL	DSCM
TWØT	DEC	XXXX	TWØ-THETA (INTEGER FØRM)
WAVE	DED	XXXX	D.P. WAVELENGTH
METRIC	BSS	18	RECIPRØCAL METRIC
H	BSS	9	INTEGER HKL values
HF	EQU	H+3	D.P. HKL values
DSTAR	DED	XXXX	1/d for HKL

E. ZZAG

Purpose: Routine for automatically choosing hkℓ indices.

Calling Sequence:

```
NØP     IC
NØP     TWØT
NØP     WAVE
NØP     METRIC
NØP     H
NØP     DSTAR
CALL    ZZAG
SKE     ZERO
BRU     * + 2
BRU     **** CØNTINUE PRØGRAM
CALL    MESS
BRM     IMPRETN   ERROR MESSAGE
```

Details: Reflections are chosen in a zig-zag fashion through the reciprocal lattice. When the DSTAR maximum is exceeded the k index is altered. If DSTAR is still too large the h index is altered and the next layer is studied.

```
IC      BSS     10          CØNTROL INTEGERS
TWØT    DEC     XXXX        TWØ-THETA (INTEGER FØRM)
WAVE    BSS     4               (DØUBLE PRECISIØN)
DSTARM  EQU     WAVE + 2    MAXIMUM ØBSERVABLE 1/d
METRIC  BSS     18          RECIPROCAL METRIC
H       BSS     9           INTEGER HKL values
DSTAR   BSS     2           1/d
```

F. CSET

To compute the Phi and Chi axis settings (given omega) for one set of HKL indices.

Calling Sequence:

```
        NØP     PHI
        NØP     AS
        NØP     DSTAR
        NØP     H
        CALL    CSET
PHI     BSS     4       Setting Angles
UMTX    BSS     18              U- Matrix
AS      BSS     12      RECIPRØCAL LATTICE CØNSTS.
DSTAR   BSS     2       1/d HKL
H       BSS     9       Integer HKL
HF      EQU     H + 3   D.P. HKL
```

G. SCHK

Checks the settings and scan limits for possible contact with the omega and 2θ limit switches. If all limit switches are safe, then all four axes can be set. Also makes zero corrections.
CHKS is the version for the neutron spectrometer (ANDA)

Calling Sequence:

```
        NØP     STDSCN
        NØP     IC
        NØP     DELZER
        NØP     PHI
        NØP     LIMS
        NØP     SCNDEL
        CALL    SCHK    or CHKS for ANDA
```

```
            SKG       M1
            BRU       ZAG        ERRØR BRANCH NO. 1
            SKE       ZERØ
            BRU       SET        ERRØR BRANCH NO. 2
            CONTINUE
IC          BSS       10         CØNTRØL INTEGERS
PHI         BSS       4          SETTINGS ANGLE
LIMS        BSS       4          LIMITS ØN 2θ, -2θ, ω, -ω
DELZER      EQU       LIMS + 4   INSTRUMENT ZERØ CØRRECTIØNS
SCNDEL      DEC       XXXX       SCAN RANGE
M1          DEC       -1
ZAG         XXX       XXXXXX     ERRØR RETURN FØR TWØ THETA LIMIT SWITCH
SET         XXX       XXXXXX     ERRØR RETURN AFTER MØDIFYING ØMEGA
                                 BECAUSE ØF A PØSSIBLE LIMIT SWITCH ENCØUNTER
STDSCN      DEC       XXXX       Minimum Scan Range
```

H. ØTØC

To convert an octal word to two BCI words containing one signed decimal integer.

Calling Sequence:

```
            LDA       M
            CALL      ØTØC
```

M is INTEGER TØ BE CØNVERTED TØ BCI IN (B,A) registers

Results: The first half of the decimal integer is in (B), the second half is in (A).

I. UTIL

Converts the H,K,L, Signal, Background and settings angles to BCI typewriter format.

Calling Sequence:

```
NØP     H
NØP     PHI
CALL    UTIL
```

Follow with

```
RESET     FLGTYP
BRM       IMPRETN
SET       FLGTYP
CONTINUE
```

or with

```
ADD       =26
NØP       CØØRD
NØP       SIZENM
NØP       LABEL
CALL      FØNT
CALL      DISPLY
YIELD
```

This calling sequence will result in a type output and a return of control to the location specified by (CONTINUE).

```
H         BSS     11        H,K,L in integer
HF        EQU     H + 3
SIGNAL    EQU     H + 9
BACKRN    EQU     H + 10
PHI       BSS     4         PHI, OMEGA, TWØ-THETA and CHI
FLGTYP    PZE     **        FLAG FØR RETURN FROM TYPING RØUTINE
```

Subroutines Used:

```
MESS      Message number 7
ØTØC
```

J. ARRY

Converts double precision floating point data into BCI and stores in an output array which can be typed out with the typing routine. The location at which the program is to be restarted is also typed out.

Calling Sequence:

```
NØP     RENTRY-FIRST
NØP     ARRAY
NØP     LINES
NØP     CØLUMNS
CALL    ARRY
BRM     IMRETN
```

* * * * * * * * * * * * * *

FIRST	BRU	RESTAR	LOCATION (0000)
ARRAY	BSS	XXXX	DATA ARRAY
LINES	DEC	N	NUMBER OF TYPEWRITTEN ØUTPUT LINES (25 MAXIMUM LINES)
CØLUMNS	DEC	M	NUMBER ØF CØLUMNS ØF ØUTPUT (M = 1,2, 3, 4 <u>only</u>)

For a 3x3 matrix output format use

```
LINES     DEC     3
CØLUMNS   DEC     3
```

K. ICNM

Sets up the tape code containing the day, month and crystal number. In addition it sets up the type output of control integers.

Calling Sequence:

```
NØP     DATE
NØP     DAY
NØP     IC
CALL    ICNM
RESET   FLGTYP
BRM     IMRETN
```

```
* * * * * * * * * * * * * * * * * * * *
DATE    BCI     1,      LØC. OF DATE IN TAPE CODE WORD
DAY     DEC     XX      Day
MØNTH   DEC     XX      Month
XTAL    DEC     XX      Crystal number
IC      BSS     10      Control numbers
```

Example of Output:

```
CØNTRØL INTEGERS:   1  2  3  4  5  6  7  8  9  10
                    0  0  1  7  0  0  2  1  4  0
```

Return of control is to the next instruction

L. CØMS

Will check for optimization run, set up the correct motor number, compute the upper and lower limits of the scan. It will use SETWAV to find the midpoint of the θ-2θ scan. If SETWAV = 0., the current 2θ will be used as midpoint of the scan.

Calling Sequence:

```
        NØP     IC
        NØP     DELTWØ
        NOP     ERROR
        NØP     ØPTSCN
        NØP     MØTNUM
        NØP     NWØRDS
        NØP     SETWAV
        NØP     STEP
        NØP     METRIC
        NØP     H
        NØP     PHI
        NØP     ØPTFLG
        CALL    CØMS
      * * * * * * * *
IC      BSS     10          Control Integers
DELTWØ  DEC     XXX         Zero correction for two-theta
ERRØR   PZE     **          SET WHEN THE COMPUTED SCAN
                            RANGE REQUIRES MORE THAN
                            NMAX STEPS
ØPTSCN  DEC     **          CONSTANT ANGULAR SCAN
                            RANGE FOR AN OPTIMIZA-
                            TION RUN
MØTNUM  DEC     XXX         LOCATION OF MOTOR NUMBER
```

M. INTI

This routine computes the signal and background. It also finds the optimum angle setting for the current axis scanned. If it is an optimization scan, then all it does is the latter.

Calling Sequence:

```
        NØP     DEGREE
        NØP     IC
        NØP     ØPTFLG
        NØP     PHI
        NØP     MØTNUM
        NØP     ARRAY
        NØP     NWØRDS
        NØP     ATTN
        NØP     SIGNAL
        NØP     STEP
        NØP     TOTAL
        CALL    INTN
```

* * * * * * * * *

IC	BSS	10	Control Integers
ØPTFLG	PZE	**	Optimization flag
PHI	BSS	4	Settings angles
MØTNUM	DEC	XX	Motor number
ARRAY	BSS	100	Step scan data array
NWØRDS	BSS	7	Number of Steps + other data
ATTN	DEC	XX	Attenuation number
SIGNAL	EQU	H + 9	Signal=Integrated-Background
BACKRN	EQU	SIGNAL+1	Background
STEP	BSS	23	Step size + Other data
TOTAL	DED	XX	Integrated Scan Count

Order Important	NWØRDS	DEC	XXX	COMPUTED number of steps
	NMAX	DEC	100	Maximum number of steps
	PRE	DEC	1	Number of Prescalers
	ØPTFLG	PZE	**	Optimization Flag
	DELTA	PZE	**	Step size when optimizing
	UPLIM	PZE	**	Beginning of Scan
	ENDSCN	PZE	**	End of Scan
	SETWAV	DED	***	Wavelength to be in center of Scan.
	STEP	DEC	XX	Step in 1/100 deg Step size for regular scan
	STPTIM	DEC	XX	---
	BKGTIM	DEC	XX	---
	SCNDEL	DEC	XX	Scan Range for data taking run.
	METRIC	BSS	18	Reciprocal Metric
	H	BSS	9	H,K,L + 6 locations for D.P. H,K,L
	PHI	BSS	4	Setting angles
	DEGREE	DEC	3	Degree of smoothing

Additional System Subroutines Called by AXDA-ANDA

GETG - Computes the reciprocal Metric from the cell constants.

DELA - Real time delay routine 1 sec. per call.

FILT - Sets the attenuator and filter wheel on the X-ray unit.

TAPE - Tape output routine.

MINA - Finds the minimum or maximum in an array

PLTF - Displays an array of data.

FONT - Displays a BCD message.

SPLY - Used in setting up displays.

SETX - Sets up the breakpoint tests

BKPT - Breakpoint program

Suggested Consol Typewriter Output
Routine for Use with MESS, MES2 and IMPT

```
IMRETN    NØP     **
          STA     AREG
          STB     BREG
          LDX*    AREG
          SXA     MADDR
          LDA*    BREG
          STA     BREG
          STA     AREG
LØØP      LDX     AREG
MADDR     LDA     **,2
          XMA     BUFFER,2
          SKR     AREG
          BRU     LØØP
          NOP     TYPNUM
          NØP     BUFFAD
          NØP     BREG
          CALL    TYPE
          YIELD
          SKN     FLGTYP
          CALL    FINISH
          SET     FLGTYP
          BRR     IMRETN
BUFFAD    PZE     BUFFER
TYPNUM    DEC     2        Typewriter number
BREG      PZE     **
AREG      PZE     **
BUFFER    BSS     100
FLGTYP    PZE     **       ~RETURN FLAG
```

Appendix 4. MSCS mnemonics.

A single line may contain up to 72 characters.

LLLL denotes a location--four octal digits--relative to the beginning of the current users area.

N always denotes a decimal digit.

{ } enclose different options.

[ ] encloses argument which may be repeated separated by commas.

I. Input-Output Instructions

ALW LLLL [{XXXXXXXXA / XXXXXXXXR}]

words beginning in location LLLL are replaced by the octal digits indicated for A format. For R format, the beginning of the current users area is added to the address portion of the word before entry. Multiple entries may be made, separated by commas. Mixed A and R format is possible.

LDW LLLL

same as ALW but words are on paper tape, separated by commas and terminated by Carriage return.

TYW LLLL {NR / NA}

N words ($N \leq 6$) starting in location LLLL are typed out in either absolute (A) or relative to first word of users area (R). Type-out is eight-octal digits.

INT LLLL [M]

loads signed integers M starting in location LLLL. $|M| \leq 2^{23}$ (82, 116, 608). Multiple entry permitted.

LDI LLLL

Same as INT, but integers are on paper tape, separated by commas and terminated by CR.

TYI LLLL N

N integers ($N \leq 6$) starting at location L are typed out.

DEC LLLL [{±} .ME {±} NN]

stores a double precision floating point number in the two words starting at LLLL.
M is a decimal mantissa of up to 13 digits
NN is a two-digit exponent. The number is $\pm 0.M \times 10^{\pm NN}$
Multiple entry permitted.

LDD LLLL

Same as DEC but numbers on tape, separated by commas and terminated by CR.

TYD LLLL N

N floating point decimal numbers ($N \leq 3$) beginning in location LLLL are typed out.

LDP LLLL NAME

The assembled program NAME is located into core at the location LLLL.

II. Status enquiries

TYS a status report is typed out.

TYM motor positions and activity are typed out.

III. Program Control

STP LLLL
the string of command starting at location LLLL is executed.

HLP the program is stopped on next entry.

RSP all program flags are lowered. Always give this command before STP.

IV. Motor and Counter Control

STA [N {±} M]
motor number N is set to location ±M (mod36000) where M is a decimal integer in degrees x 100. Multiple entries are permitted.

LDA same as STA, but data is on paper tape.

STE [N {±} M]
motor position readin is changed but motor is not driven.

LDE same as STE, but data is on paper tape.

SDP same as STE

ZRA N motor N is driven to mechanical zero.

CHA N encoder is set to 0 and old reading is printed out as ERRØR ØF XXX.XXX DEGREES

STC N M

Counters are started with N (≤ 2) prescalers and will halt when monitor reaches M counts.
($M = (1,2,4,8) \times (10^3, 10^4, 10^5)$).

HLC the counters will be stopped.

RSC the counters are reset.

Appendix 5. Sample of Assembled Data Input for AXDA

DATE	IDEN		For Tape Output Code Word
DAY	DEC	25	
MONTH	DEC	3	
XTAL	DEC	1	
	ABSO		
	END		

DAT1	IDEN		Control Integers
IC	DEC	0	
	DEC	0	
	DEC	0	SEE
	DEC	0	
	DEC	1	
	DEC	1	
	DEC	3	TABLE I
	DEC	0	
	DEC	0	
	DEC	0	Page 33.
	DEC	1	
	ABSØ		
	END		

DAT2	IDEN		Wavelength and DSTAR Maximum
WAVE	DED	.7094	X-Ray Wavelength
DSTARM	DED	1.20	Maximum
DSTMIN	DED	0.02	Minimum 1/d desired
SETWAV	DED	0.7104	Wavelength for Scan setting
	ABSO		
	END		
DAT3	IDEN		Instrument Constants
STEP	DEC	10	Scan Step in Degrees x 100
STPTIM	DEC	1000	Monitor Time Per Step in Pulses
BKGTIM	DEC	3	Number of steps for Background
SCNDEL	DEC	100	Step Scan Range in Degrees x 100
ATSCL	DEC	100	Attenuation Scale X 100
ATSCL1	DEC	400	First Attenuator Scale Factor x 100
ATSCL2	DEC	1600	Second Attenuator Scale Factor x 100
ATSCL3	DEC	6400	Third Attenuator Scale Factor x 100
RTCOMP	DEC	10000	Monitor Time for Linearity Check
RATE	DEC	1000	Maximum Count for Linearity
FILTNM	DEC	1	The Filter Number
LIMS	DEC	11400	2θ Limit Position in Degrees x 100
	DEC	-6600	Negative 2θ Limit
TUBELM	DEC	5700	Positive omega limit
	DEC	-6000	Negative omega limit
CNTER	DEC	3300	Positive omega-counter limit
	DEC	-5600	Negative omega-counter limit
DELPHI	DEC	0	Zero Correction on the Phi Circle
DELOHM	DEC	0	Zero Correction on the Omega Circle
DELTWO	DEC	0	Zero Correction on the Two Theta Axis

DELCHI	DEC	0	Zero Correction on the Chi Circle
TIME	DEC	4	Delay Time for Filter Setting
STDSCN	DEC	100	Minimum 2θ Scan
FACTOR	DED	1.0	Constant in variable scan formula
	ABSO		
	END		
DAT4	IDEN		Reference Reflection Settings
H1	DEC	1	
K1	DEC	0	
L1	DEC	0	
PHI1	DEC	0	
ØHM1	DEC	0	
TWØT1	DEC	2049	
CHI1	DEC	0	
H2	DEC	0	
K2	DEC	1	
L2	DEC	0	
PHI2	DEC	6000	
ØHM2	DEC	0	
TWØT2	DEC	2049	
CHI2	DEC	0	
H3	DEC	0	
K3	DEC	0	
L3	DEC	1	
PHI3	DEC	0	

```
ØHM3        DEC        0
TWØT3       DEC        883
CHI3        DEC        9000
            ABSØ
            END
```

```
DAT5        IDEN                  Reciprocal Lattice Constants
AS          DED        0.2        1/d(100)
BS          DED        0.2        1/d(010)
CS          DED        0.1        1/d(001)
CØSAS       DED        0.0        Cosine of ALPHA*
COSBS       DED        0.5        Cosine of BETA*
COSCS       DED        0.0        Cosine of GAMMA*
            ABSØ
            END
```

DAT6	IDEN		Standard Reflections
STDHA	DEC	1	H_1
STDKA	DEC	1	K_1
STDLA	DEC	1	L_1
STDHB	DEC	1	H_2
STDKB	DEC	3	K_2
STDLB	DEC	1	L_2
STDNUM	DEC	30	Number of reflections to be measured between standard scans.
ØPTSCN	DEC	20	Scan range for an optimization scan in deg x 100
STDFLG	DEC	20	Counter location for number of reflections measured.
OPTSTP	DEC	2	Step size for the optimization mode.
BADCHI	DEC	-4500	Limit of allowable negative CHI settings.
	ABSO		
	END		

APPENDIX 6

Sample of Console Typewriter On-Line Output

```
ALB7        NØT UNDER CØMPUTER CØNTRØL


ALB7
            TIME:  06408  SEC
LDP 0000 AXDA
            DØNE

LDP 1217 VECT
            DØNE

STP 0001
            DØNE


  INPUT DATA FØR EXP.    7

            ****DAY, MØNTH, AND CRYSTAL NUMBER**** INTEGER FØRMAT

  BEGINS IN LØCATIØN  0012

  INSERT    3  ITEM(S)

  STP IN  0016

  ALW 0012 00000003A
            DØNE

ALW 0013 00000012A
            DØNE

ALW 0014 00000006A
            DØNE
```

```
STP 0016
          DØNE

CØNTRØL INTEGERS   1   2   3   4   5   6   7   8   9  10
                   3   0   0   0   2   4   2   1   3   0
$

  INPUT DATA FØR EXP.    7

  INSERT ØR ALTER THE ***CØNTRØL INTEGERS*** AS REQUIRED

  BEGINS IN LØCATIØN  1035

  INSERT   TEN ITEM(S)

  STP IN  0037

ALW 1046 00000002A
          DØNE

STP 0037
          DØNE

  INPUT DATA FØR EXP.    7

  **WAVELENGTH, DSTAR MAX., DSTAR MIN., AND SETWAV** D.P.

  BEGINS IN LØCATIØN  1063

  INSERT  FØUR ITEM(S)

  STP IN  0051
```

```
STP 0134
          DØNE

          DATA ØUTPUT FØR EXPERIMENT NØ.   7
RESTART PRØGRAM AT LØCATIØN, 0175

 .33185908900-01      .11698032887-01      .11698032887-01

 .11698032887-01      .33185908900-01      .11698032887-01

 .11698032887-01      .11698032887-01      .33185908900-01

STP 0175
          DØNE

          DATA ØUTPUT FØR EXPERIMENT NØ.   7
RESTART PRØGRAM AT LØCATIØN, 0271

 .75387885851 00      .75387885852 00      .75387885852 00

 .65701344480 00     -.32850672241 00     -.32850672241 00

 .0                   .56899033384 00     -.56899033383 00
```

```
STP0271
          DØNE

TYM
          ST 7
          MØTØR PØSITIØNS          0 ØFF      1 ØFF      2 ØFF      3 ZRØ
                                  +001.09    +001.00    -008.55    -001.90

          DØNE

TYM
          ST 7
          MØTØR PØSITIØNS          0 ZRØ      1 ØFF      2 ØFF      3 ØFF
                                  +027.11    +000.00    +000.00    +000.00

          DØNE

**        KEYBRD ØVTM
          TIME:  07930  SEC

  INPUT DATA FØR EXP.    7

  WE'RE READY TØ GØ. WHAT H,K,L IS YØUR DESIRE? (INTEGER)

  BEGINS IN LØCATIØN  0001

  INSERT    3  ITEM(S)

  STP IN  0376


ALB7
          TIME:  08450  SEC
TYM
          ST 7
          MØTØR PØSITIØNS          0 ØFF      1 ØFF      2 ØFF      3 ØFF
                                  +000.00    +000.00    +000.00    +000.00
```

**FØR EXPERIMENT NØ. 7

H	K	L	SIGNAL	BACKRN	PHI	ØMEGA	2THET	CHI
2	2	-7	276	1642	199	1383	3969	12626

**FØR EXPERIMENT NØ. 7

H	K	L	SIGNAL	BACKRN	PHI	ØMEGA	2THET	CHI
2	2	-6	8115	3247	956	1383	3278	12389

**FØR EXPERIMENT NØ. 7

H	K	L	SIGNAL	BACKRN	PHI	ØMEGA	2THET	CHI
2	2	2	8395	4261	12939	1383	2523	24

EXP NØ. 7 AXIS NØ 0 ERRØR = 2

**FØR EXPERIMENT NØ. 7

H	K	L	SIGNAL	BACKRN	PHI	ØMEGA	2THET	CHI
0	-2	2	241902	27974	3940	1383	823	9023

**FØR EXPERIMENT NØ. 7

H	K	L	SIGNAL	BACKRN	PHI	ØMEGA	2THET	CHI
2	2	-5	161	2532	2125	1383	2606	12153

**FØR EXPERIMENT NØ. 7

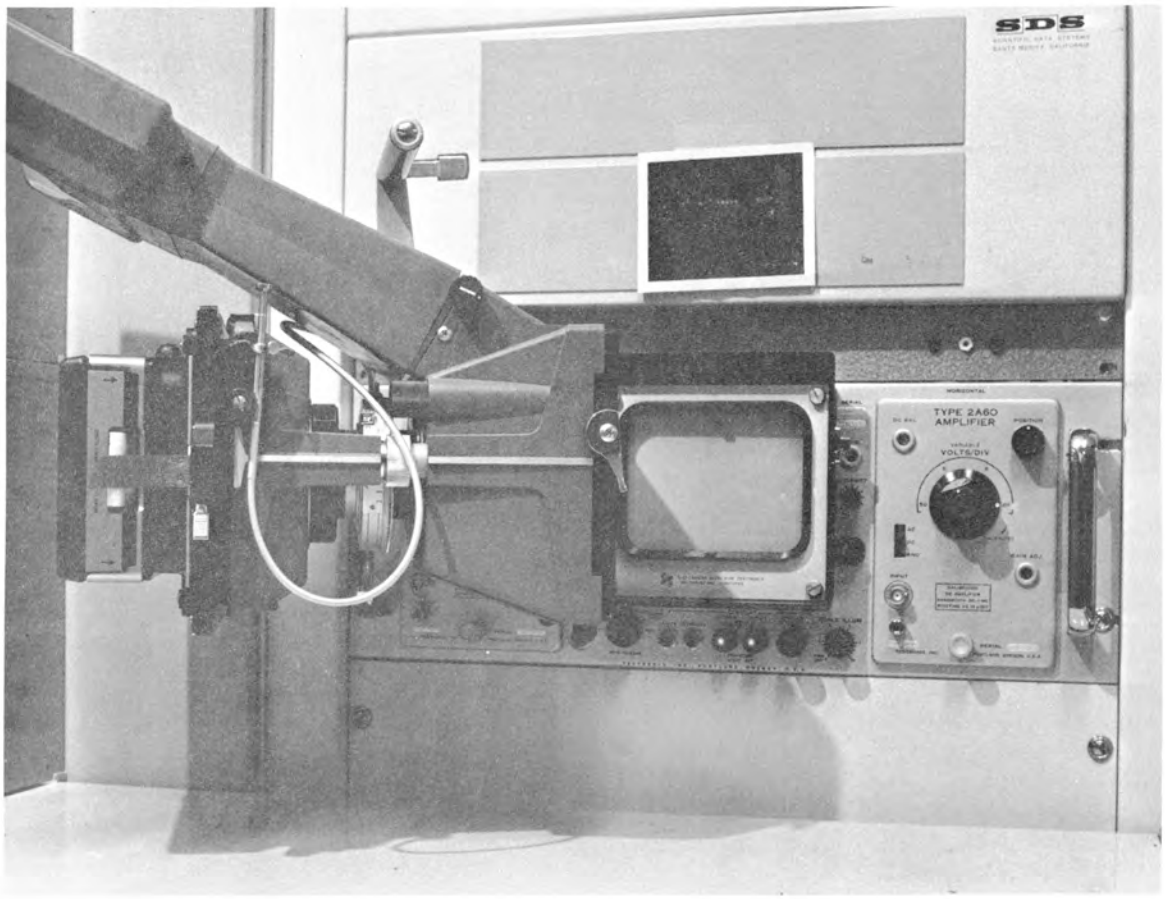
H	K	L	SIGNAL	BACKRN	PHI	ØMEGA	2THET	CHI
2	2	-4	32314	5980	3940	1383	2087	12024

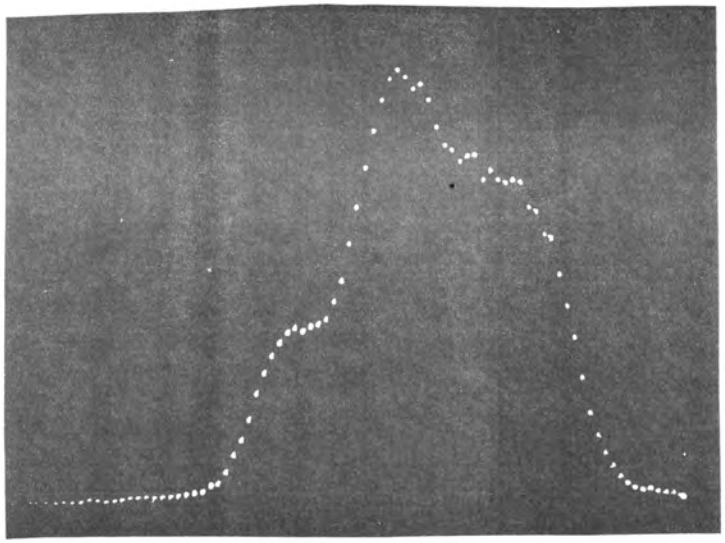
**FØR EXPERIMENT NØ. 7

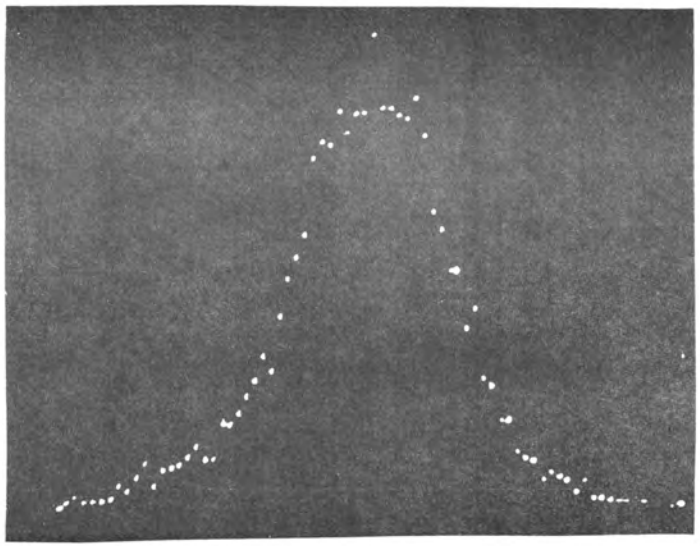
H	K	L	SIGNAL	BACKRN	PHI	ØMEGA	2THET	CHI
2	2	-3	1	3059	6405	1383	1620	12267

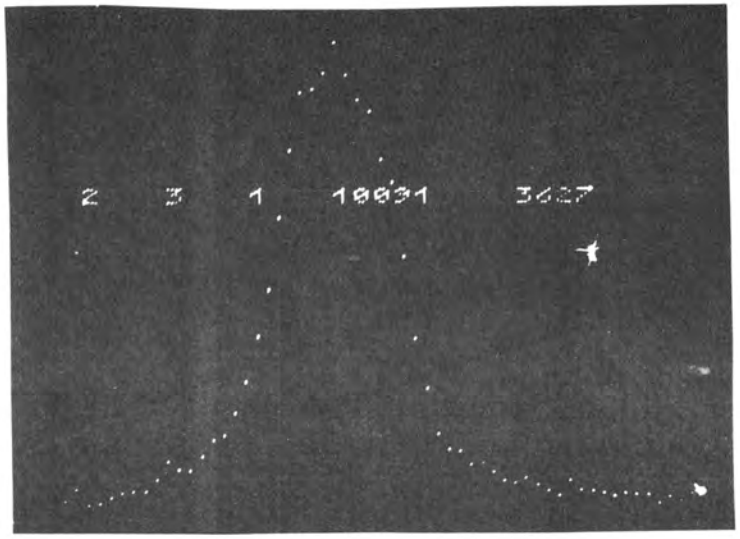
APPENDIX 7

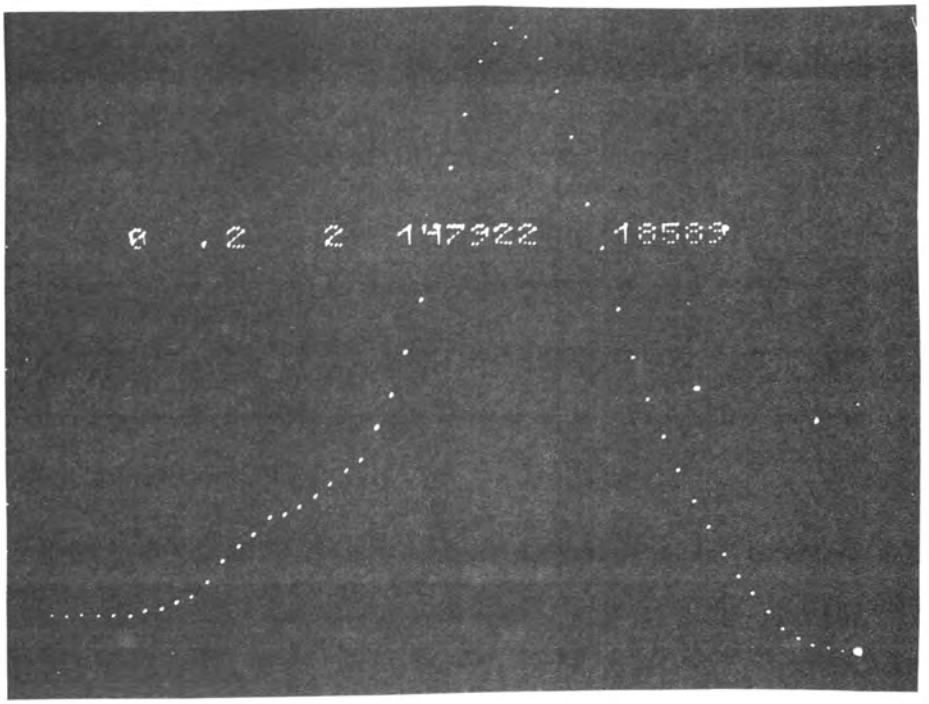
Photographs of the CRT Installation and Examples of the CRT Display Output

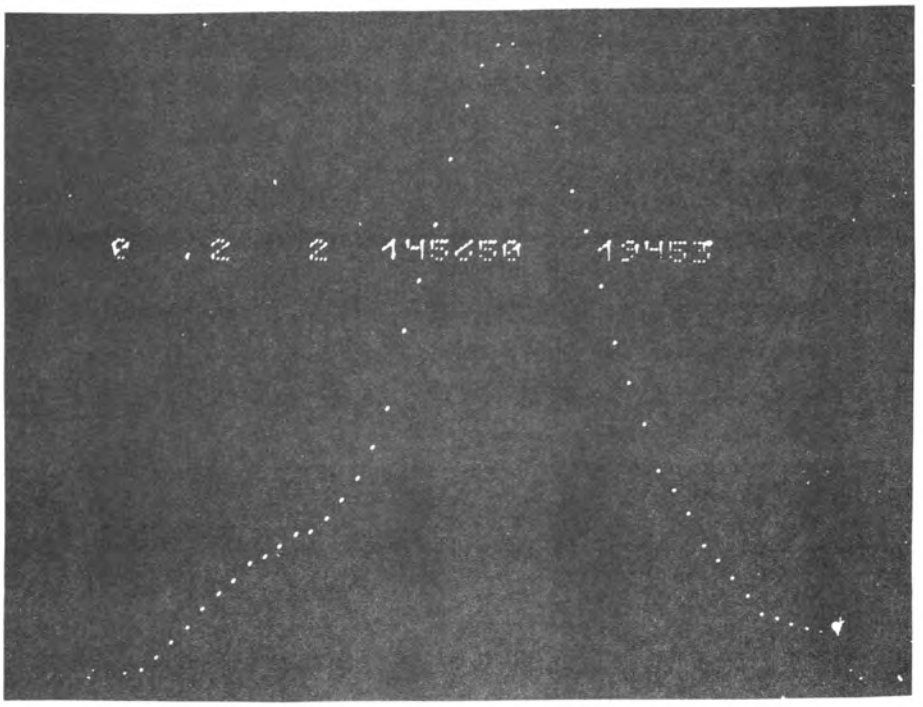

SDS
TYPE 2A60
AMPLIFIER
VOLTS/DIV

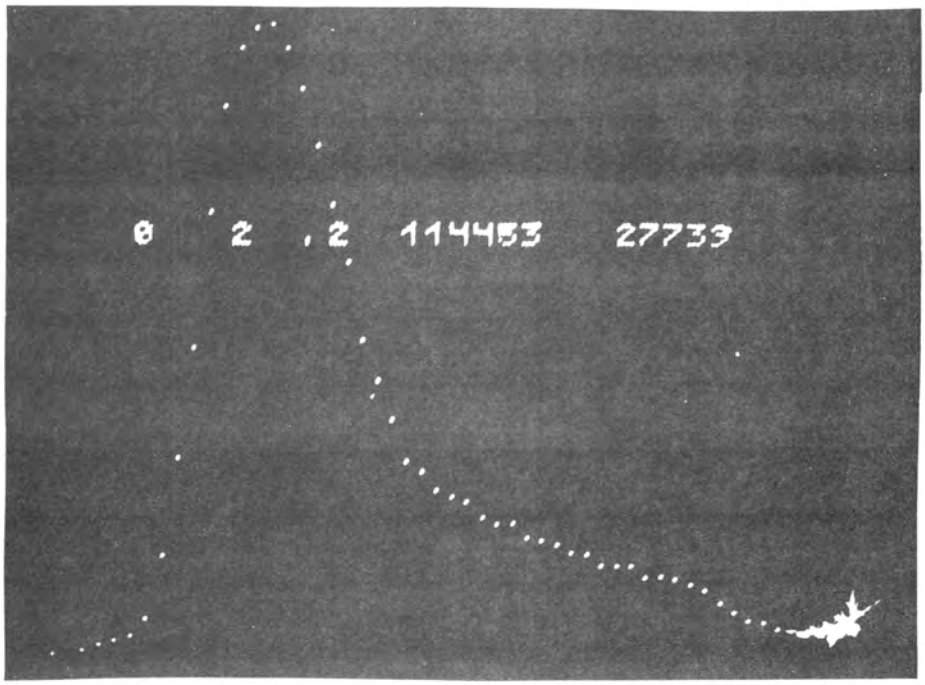


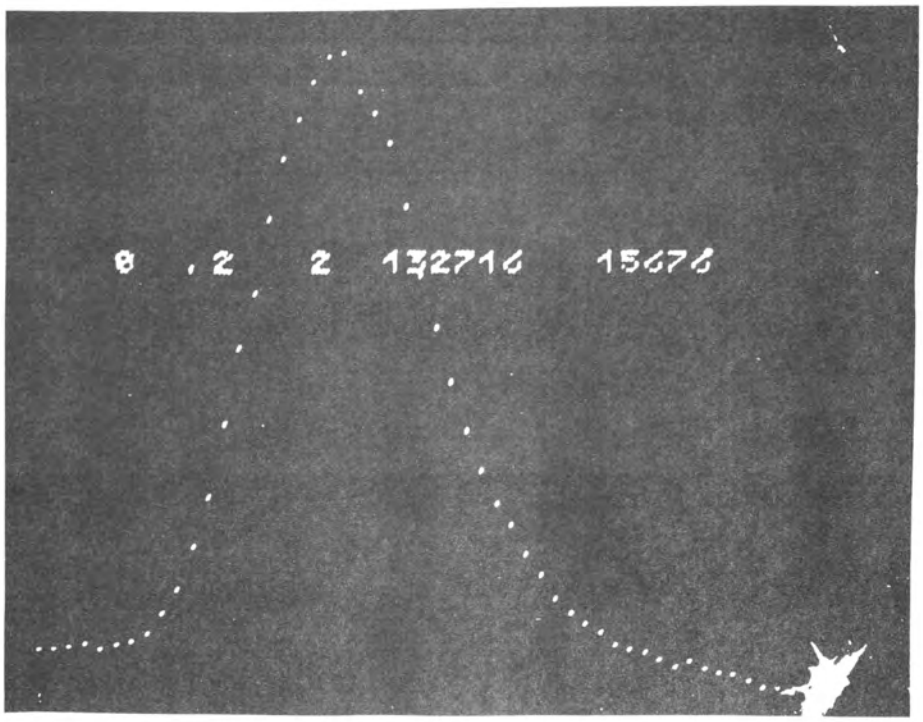



2 3 1 10091 3627


0 .2 2 147922 .18583


0 .2 2 145450 .19453


0 2 .2 114453 27733


0 .2 2 132716 15676

Appendix 8. Assembly on the CDC 924

Card Order

1. CDC 924 Request card "Request Assembly Mode Prolibe #1"

2. Col 1 Col 8
   XXXX IDEN

   Program identifier

3. ⋮ Body of Program

4. ABSØ - prepares an end of load mark on the paper tape.

5. END - signals assembler for end of symbolic deck.

6. Normal End of File Card

Appendix 9. Some miscellaneous controls.

Restart Routine (RESTAR)

In order to reduce the accumulation of axis setting errors the axes are zeroed (driven to encoder zero) after STDNUM reflections. After zeroing each axis in turn the CHECK AXIS subroutine is used to reset the display angle to zero. In addition (for the X-Ray instrument) AXDA will close the shutter and place a lead stop in front of the counter before beginning to zero the axes.

After the above operations are complete the program will type out a request for h,k, ℓ and indicate a new reentry location. If the program has been started from scratch the h,k, ℓ locations will all be zero at this point. The RESTAR routine is also used between standard reflections. If the latter is the case no message will be typed out and the program will continue to take data.

Note that for the X-Ray machine the lead stop is on the attenuator wheel and it is set (as well as the shutter being closed) by using attenuator number four (4) in the FILT subroutine. There is a delay after signaling the attenuator relay which is controlled by the DELA subroutine. The constant TIME can be varied to control the delay required. One full turn of the attenuator wheel can be accomplished in eight seconds.

Restarting the Program after Data Insertion

Each input request specifies the location at which the program is to be restarted after insertion of the data. If no data are to be inserted or altered the program may be restarted in the indicated location. As the operator becomes familiar with the current version of the program (and if his data are assembled with it, he may skip the input requests (3) $\longrightarrow$ (6) and the intermediate matrix data output by using a zero for IC+9 in the assembly.

Warning Do not skip the first two input requests. This part of the program must be executed in order to set up the magnetic tape output label.

Housekeeping Routines

The housekeeping routines begin at location ENT3A. When skipping the input requests 3 ⟶ 6 start the program in ENT3A.

WARNING Do not repeat any previous step unless the program tape is completely reloaded.

The first housekeeping routine zeros locations 0 ⟶ 20. The erasure and reuse of the locations allocated to the request routines allow more data to be stored in the running program. Note that the housekeeping routines are overlayed (by the step scan array). This procedure is necessary because of the limited user area.

The Reciprocal Metric is computed by subroutine GETG and also stored over the input requests.

The subroutines MESS and MES2 contain all the typewriter requests and error messages. The subroutine IMPT picks up these messages and sets up the form of the input request for output on the consol typewriter. Using the three reference reflections inserted as data, computes the matrix relating the reciprocal lattice coordinates to those fixed with respect to the goniometer head. The U matrix is also overlayed upon the PRIMER routines. To repeat a warning; if the program is to be restarted from scratch it must be reloaded.

ALFA ALFA ALFA

THE PROGRAM ALFA CAN BE USED TO OPTIMIZE THE SETTING ANGLES ON A DIFFRACTOMETER. THE PARTICULAR ANGLES OPTIMIZED ARE CONTROLLED BY THE BREAKPOINT SWITCHES.

SWITCH A DOWN	OPTIMIZE MOTOR Ø (ZERO)
SWUTCH B DOWN	OPTIMIZE MOTOR 1 (ONE)
SWITCH C DOWM	OPTIMIZE MOTOR 2 (TWO)
SWITCH D DOWN	OPTIMIZE MOTOR 3 (THREE)

WHEN MORE THAN ONE SWITCH IS DOWN, THE OPTIMIZATION IS ALWAYS IN THE ORDER OMEGA, PHI, TWO-THETA,CHI.

THE PROGRAM FOR THE INDIVIDUAL DIFFRACTOMETER MUST BE USED, SINCE MOTOR IDENTICFICATION VARIES AND SINCE ONLY THE H6S3 MACHINE IS EQUIPPED WITH AN AUTOMATIC LEFT-RIGHT, TOP-BOTTOM DEVICE.

FOR OPTIMIZATION OF PHI AND OMEGA, THE PROGRAM FINDS THE HALF-MAXIMA ON EITHER SIDE OF THE PEAK(OR THE END OF THE SCAN IF HALF-MAXIMUM IS NOT REACHED) AND TAKES THE AVERAGE AS BEING THE PEAK POSITION,.

FOR OPTIMIZATION OF TWOTHETA, SCANS ARE MADE WITH THE LEFT-HALF CLOSED, WITH THE RIGHT HALF CLOSED, AND THE POINT OF INTERSECTION (AVERAGE OF THE TWO CLOSEST HALF-MAXIMA POSITIONS) IS TAKEN AS HE PEAK POSITION.

FOR OPTIMIAZATION OF CHI, THE SAME PROCEDURE IS FOLLOWED AS FOR TWOTHETA, BUT USING THE TOP-BOTTOM APERATURES RATHER THAN THE LEFT-RIGHT.

THE PROGRAM REITERATES CONTINUOUSLY, AND SWITCHES FOR THE NEXT RUN MAY BE SET DURING THE CURRENT RUN.

THE RESULTS ARE DISPLAYED ON THE CRT.

IMPORTANT CONSTANTS IN THE PROGRAM ARE

NAME	LOCATION	PRIMED AT	MEANING
DELØ	1Ø43	5	~~DELTA~~ Ø
MONØ	1Ø44	1ØØØ	MONITOR FOR MO Ø
CHIRA	1Ø71	175	HALFRANGE MO Ø
DEL1	1Ø51	5	STEP MOTOR 1
MON1	1Ø52	1ØØØ	MONITOR MOTOR 1
THERA	1Ø72	5Ø	HALFRANGE MOTOR1
DEL2	1Ø57	2	~~DELTA~~ MOTOR 2
MON2	1Ø6Ø	1ØØØ	MONITOR MOTOR 2
OMERA	1Ø73	25	HALFRANGE MOTOR 2
DEL3	1Ø65	2	STEP MOTOR 3
MON3	1Ø66	1ØØØ	MONITOR MOTOR 3
PHIRA	1Ø7Ø	25	HALFRANGE MOTOR 3

THE NUMBER OF PRESACLERS IS PRIMED TO Ø AND FOR EACH MOTOR IS LOCATED IMMEDIATELY FOLLOWING THE MONITOR SCALE FACTOR

Δ;) OPERATING INSTRUCTIONS-FOR-ALFA

L.

1. LDPØØØØALFA

2. MANUALLY SET REFLECTION SOMEWHERE NEAR PEAK.

3. SET STATION BREAKPOINT SWITCHES AS DESIRED (SEE ABOVE)

4. STPØØØØ

5. ;PROGRAM NOW WILL OPTIMIZE DESIRED MOTORS AND REITERATE UNTIL AN HLP COMMAND IS ENTERED.

PROGRAM IS PRESENTL COMPILED ONLY FOR THE H6MS.....

FØR THE H6M4 machine, use the program BETA

FØR THE XRY7 machine, use the program GAMS

Program BPEX

Calls the subroutine BKPT which can be used for certain trivial operations useful in alignment.

```
LDP 0000 BPEX
STP 0000
```

Before starting, be sure all station breakpoint switches are up.

Arguments in routine start are as follows

```
DELTA3   DEC 500 An increment
MØNUM1   DEC 3   A motor number
PRE      DEC 0   A prescaler number
MØN      DEC 1000  A monitor scale factor
DELTA1   DEC 10  Step-scan step size
DELTA2   DEC 500 Scan-length
MØNUM2   DEC 1   A motor number
NUMBER   DEC 4   Number of motors at station
```

Breakpoint switches B, C, D control program options taken as in the following table.

Select appropriate breakpoint settings. Put switch A down to initiate program, back up to terminate. Select new program only when 999.99 is displayed in position 7. You may also go to manual operation while 999.99 is displayed. Program will be reentered simply on going to computer control.

For the counting options, cumulative count will be displayed in position 4.

Breakpoint Settings

(B)	(C)	(D)	CODE	FUNCTION
up	up	up	000	zero all motors and check zero for each
up	up	dn	001	Step Scan forward (Range=DELTA2) Step=DELTA1, MØNUM2
up	dn	up	010	Step Scan reverse Range = DELTA2, Step=DELTA1, ØNUM2. Do not store results.
up	dn	dn	011	Increment MØNUM1 by DELTA3
dn	up	up	100	Decrease MØNUM1 by DELTA3.
dn	up	dn	101	Repetitive count at a single point (accumulating) using MON and PRE in Position 4.
dn	dn	up	110	Single Count using MØN and PRE
dn	dn	dn	111	Do a complete step scan, recording results in an ARRAY (location 62 → 62 + (49)10 and cumulative, count in position 4. Plot.

Program SLIT

Sets filter wheel on H6S3 by use of breakpoint switches.

Operation.

```
LDP 0000 SLIT
STP 0000
```

(1) Key in an octal integer on switches B, C, and D.
(2) Put switch A down to position wheel.
(3) To select another position put switch A up and repeat 1 and 2.

Breakpoint Program

To enter the breakpoint program the main program should be started with a command STP0000. If the breakpoint (A) is set in the down position the breakpoint program will be entered, otherwise entry occurs at the RESTAR routine.

Once in the breakpoint program, exit from it and reentry to the main program can be accomplished by setting breakpoints (B), (C) and (D) down, then flipping (A) (up first, and down) to activate the branch.

The functions available with the breakpoint program are listed in Table 8. These functions allow the experimenter to align a crystal and check out the reference and standard reflections from the spectrometer control station. The required constants, such as, axis number and monitor setting are in the same locations as the corresponding constants in the main program.

STEP	step size
SCNDEL	scan length (x100)
STPTIM	monitor setting
PRE	number of prescalers
MOTOR	actual motor number