

## BRAGG REFLECTION PROGRAM

## User's Guide

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## INFORMAL REPORT

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## BRAGG REFLECTION PROGRAM - User's Guide

The Bragg Reflection Program (BRP) for the PDP-11 system is analogous to the "PAIR" program for the old SDS system. Its general purpose is to automatically scan a series of Bragg reflections to obtain profiles and integrated intensities. The program is used in conjunction with the SUPERVISOR and READ packages and the procedures for using it are similar to those for the Triple-Axis Control program. All the general features of the system, SUPERVISOR and READ packages as described in the Spectrometer Control System User's Guide (SCSUG) have been preserved. To avoid lengthy duplication of material, it will be assumed that the reader is already familiar with these.

### Read Package

The general read variables for the BRP are the same as those listed in Appendix B of the SCSUG except that six additional variables have been defined. These variables are treated just the same as the other general read variables and do not change from scan to scan. They are defined as follows:

ST - Scan type. There are three types of scan that can be performed by the program. With ST = 1, the detector is moved to the scattering angle corresponding to (H,K), and the sample angle is scanned through the peak in steps  $\Delta\psi$ . With ST = 2, a  $\theta:2\theta$  scan is done. For ST = 3, a  $2\theta$  scan is performed, with the sample angle FROZEN. In this mode the sample angle does not change either during a scan or between scans, and the power to the motor may be shut off if desired.

If ST is not set to 1,2 or 3, then the program will be exited and an error message typed.

Note on freezing motors: The sample angle must always be defined ( $0 \leq C2 < 6$ ). ST = 3 provides for freezing the sample angle in the BRP itself. The scattering arm may be frozen via the READ package (i.e. A2 > 6) only when ST = 1. Any other condition will abort the BRP and print an error message. All remaining angles may be either defined or undefined. The elastic condition KI = KF is always assumed in BRP, and the angles which are defined for the monochromator or analyzer (depending on the value of IK) will be moved to the elastic position.

DS - Step in angle  $\Delta\psi$  for sample table when ST = 1 or 2; step in  $2\theta$  when ST = 3. DS can be positive, negative or zero. (Note: the actual step  $\Delta\psi$  used in a scan may be modified by the variable RC below)

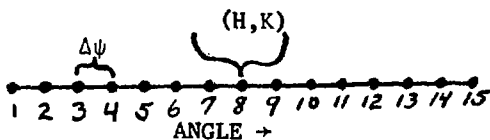
NP - Number of points on either side of (H,K) peak. The total number of points =  $2NP+1$ , and is the same for each scan.

If  $NP < 1$ , the program will issue an error message and return control to the SUPERVISOR.

NB - Number of points (out of total number of points  $2NP+1$ ) on each side of (H,K) which are used for background purposes to calculate the integrated intensity of the peak. Total number of background points =  $2NB$ .

Note: If the user sets  $NB < 1$ , then the program automatically sets  $NB = 1$ . No error message is given.

Example with  $NP = 7$ ,  $NB = 2$ :



Points 1,2,14, and 15 determine background.

Pt #8 corresponds to calculated (H,K) peak position.

MF - Monitor scale factor. This parameter is used only to give an estimate of the total running time. It should be input in units of kilocounts/min. e.g., if the monitor counting rate is 200,000 counts/min., then MF = 200. If MF = 0 no output is printed.

RC - Resolution correction. Since the observed widths of the peaks vary with scattering angle, RC allows the user to adjust the step size  $\Delta\psi$  used in a scan for the resolution of the spectrometer. The algorithm used is

$$\Delta\psi = DS \quad 2\theta \leq 30^\circ$$
$$\Delta\psi = DS \left\{ 1 + RC \left( \frac{2\theta_{H,K} - 30}{70} \right) \right\} \quad 2\theta > 30^\circ$$

Thus for RC = 1, the angle step will double from  $2\theta = 30$  to  $2\theta = 100^\circ$ .

Care is taken to round  $\Delta\psi$  so that it is always an integral multiple of  $0.01^\circ$ . For  $k_1 \sim 2.6 \text{ \AA}^{-1}$ , RC  $\sim 1.3$  would be a good choice; for  $k_1 \sim 4.5 \text{ \AA}^{-1}$ , RC  $\sim 2.5$  (see Fig. 1).

Note: RC has no effect if the scattering arm is frozen.

Preset values for the above variables are:

$$DS = 0.1, NP = 10, ST = 1, NB = 5, MF = 0, RC = 0.$$

The procedure for handling the scan variables in BRP is the same as the triple-axis program. For each scan NS, there are three variables which must be input:

H1 - H

K1 - K

M1 - Monitor

Up to 80 scans can be stored in the computer, and up to 80 scans may be requested at one time for successive execution.

### Execution and Use

The BRP is entered from the SUPERVISOR by typing LG immediately after the \$\$\$. The program responds by typing a heading and requests the scan order desired. The scan order may be entered in one of four ways:

(i), (ii), and (iii) are identical to the Triple-Axis program.

(iv) carriage return - response is determined by the conditions of previous exit from the BRP. If the previous exit occurred after completion of a set of specified scans (normal exit), or if it is the initial entrance into the program, an error message is typed and then the program will again request a new scan order. If the previous exit was caused by a CTRL-C character aborting the BRP, then the program will continue from its point of interruption. BRP is fully reenterable, and parameters such as background counts, total accumulated counts, etc, will be saved.

Example: Scans 1-10 are to be executed. Sometime during scan #5, the user returns to the SUPERVISOR by typing CTRL-C. He may then continue in the BRP where he left off by reentering the BRP and hitting LG when the scan order is requested. The program will finish scan #5 and then continue on to scans 6-10. During the interruption, the user may change the scan variables H1, K1 or M1 for scans 6-10, zero motors, etc, without affecting scan #5. However, if the scan variables for NS = 5 or any of the general read variables are changed, then this could invalidate the statistical results that are tabulated at the end of scan 5 (see Output). If the overlay has to be loaded again (i.e. RS2), then the BRP is automatically initialized.

After the scan order is entered, then the only other enquiry is

#### HEADING?

At this point the user may insert a heading to be typed at the beginning of each scan. If only a carriage return is entered, then the previous heading will be retained and used.

Next the program estimates the total running time needed for the scans requested (unless MF = 0). Then the program types

#### DRY RUN STARTED

The "dry run" checks the scans that have been requested to see if they are executable. A scan will be skipped (and an error message printed) if

- 1)  $H1 = K1 = 0$  or  $M1 = 0$
- 2) The scattering triangle will not close for (H,K) (i.e. the center point of the scan).
- 3) The internal limits of any motor are violated for (H,K).

In addition, individual points in a scan will be omitted if the internal limits are violated for any motor which is defined. (Note: In the dry run, only the first and last points are checked.)

After the scans have been checked, they will be executed. Changes can be made at any time by hitting a CTRL-C and returning to the SUPERVISOR.

#### Error Messages

Upon entering the BRP, a check is made to determine if any of the general variables IM - A3 have nonsensical values. If one or more of these variables have not been properly set, then a self-explanatory error message is typed and control is returned to the SUPERVISOR. The BRP checks its own general variables (DS-RC) and types the error message

#### ILLEGAL DATA

if a problem is found. The following conditions abort the BRP and return control to the SUPERVISOR:

1) The sample motor number (C2) is undefined, or the scattering arm motor (A2) is undefined with  $ST \neq 1$ , or both. A warning message SAMPLE ANGLE FROZEN and/or SCATTERING ANGLE FROZEN is also typed.

2)  $ST$  is not equal to 1,2, or 3.

3)  $NP < 1$

If control is returned to the SUPERVISOR, the user is advised to list the relevant variables to ascertain the problem.

Other error conditions which cause the ILLEGAL DATA error message to be typed but do not abort the programs are:

1)  $H1 = K1 = 0$ , or  $M1 = 0$  for some requested scan. Scan will be skipped.

2) The number of scans requested exceeds 80 or is less than 1. A new scan order is requested.

3) An illegal character is inserted in the scan order list. A new scan order is requested.

In addition, if the scattering triangle cannot be satisfied, or if the calculated angles lie outside the internal motor limits, an error message will be typed and the scan or point will be skipped.

OUTPUT

At the end of each counting interval the observed angles are typed along with the counts  $C_1$  and the accumulated sum (SUM). Upon completion of each scan the following information is given:

H1      K1      M1

$$\text{Total Intensity} = \text{SUM} \left( \sqrt{\text{SUM}} \right) \text{ NPT}$$

$$\text{Background} = \text{BK} \quad (\text{ERRBK}) \quad \text{NBK}$$

$$\text{Net Intensity} = \text{SUM} - \text{BK} * \text{NPT} \quad (\text{NETERR}) \quad .$$

$$\text{Relative Intensity} = \frac{\text{NET INT} * \Delta\psi * \text{SIN} \left( 2\theta_s \right)}{\text{M1}} \left( \frac{\text{NETERR} * \Delta\psi * \text{SIN} \left( 2\theta_s \right)}{\text{M1}} \right)$$

where NBK is the actual number of background points taken, NPT is the total number of points taken, and

$$\text{SUM} = \sum_{i=1}^{\text{NPT}} C_i$$

$$\text{BK} = \left\{ \sum_{i=1}^{\text{NB}} C_i + \sum_{i=2}^{2\text{NP}+1} (\text{NP}+1) - \text{NB} \right\} / \text{NBK}$$

$$\text{ERRBK} = \sqrt{\text{BK} * \text{NBK}} / \text{NBK}$$

$$\text{NETERR} = \sqrt{\text{SUM} + (\text{NPT} * \text{ERRBK})^2} \quad .$$

The statistical errors are enclosed in parentheses.

Note: If one or more points in a scan are skipped due to a motor limit violation, then the above statistical results are still calculated correctly.



Note: If CTRL-C is hit at the end of a counting interval while the line is being typed, the remainder of the line will not be typed, but  $C_i$  has already been added to SUM, and if the scan is continued the statistical results will be correct.

Note: If the scattering arm is frozen, then the Relative Intensity is not corrected for  $\sin(2\theta_s)$ .

Sample outputs for ST=1, ST=2, and ST=3 follow.

SCAN NUMBER 1

SAMPLE OUTPUT WITH IT=1

H= 2.0000 F= 0.0000

MOD= 2.

CALL ANG= 14.508 29.016

STEP= 0.05

CORR ANG FIXED AT 29.02

CAMP ANG	COUNT	CUM
13.76	3.	3.
13.81	2.	5.
13.85	3.	8.
13.89	4.	12.
13.94	3.	15.
14.01	1.	16.
14.06	7.	23.
14.11	0.	23.
14.16	5.	28.
14.21	1.	29.
14.26	7.	36.
14.32	25.	61.
14.37	114.	175.
14.41	450.	625.
14.47	1191.	1816.
14.52	1582.	3398.
14.56	1601.	4999.
14.61	1088.	6087.
14.66	409.	6496.
14.71	94.	6590.
14.76	23.	6613.
14.80	11.	6624.
14.85	10.	6634.
14.91	3.	6637.
14.96	5.	6642.
15.01	2.	6644.
15.07	1.	6645.
15.12	5.	6650.
15.16	1.	6651.
15.21	1.	6652.
15.26	3.	6655.

H= 2.000 F= 0.000 MOD= 2.

TOTAL INT= 6635.0 81.7 31

BK= 2.60 0.50 10

NET INT= 6554.400 82.990

REL INT= 79.4810 1.0060

SCAN NUMBER 1

SAMPLE OUTPUT WITH ST=2

H= 2.0000 K= 0.0000

MON= 2.

CALC ANG: 14.508 29.016

STEP= 0.05

SCAMP ANG	SCATT ANG	COUNTS	TUM
13.75	27.50	2.	3.
13.80	27.52	2.	4.
13.86	27.73	4.	8.
13.92	27.91	3.	11.
13.97	27.93	1.	12.
14.01	28.02	4.	15.
14.07	28.12	0.	16.
14.12	28.23	1.	17.
14.16	28.32	1.	18.
14.21	28.43	4.	22.
14.26	28.52	5.	27.
14.32	28.62	26.	53.
14.36	28.73	123.	176.
14.41	28.81	515.	691.
14.46	28.93	1226.	1917.
14.52	29.03	1572.	3459.
14.56	29.11	1397.	4386.
14.61	29.23	662.	5548.
14.66	29.31	286.	5834.
14.71	29.43	77.	5911.
14.76	29.52	12.	5923.
14.80	29.62	2.	5926.
14.85	29.73	2.	5927.
14.91	29.82	2.	5929.
14.96	29.93	3.	5932.
15.01	30.01	1.	5933.
15.06	30.11	1.	5934.
15.11	30.21	4.	5938.
15.17	30.31	2.	5940.
15.21	30.43	1.	5941.
15.26	30.52	2.	5943.

H= 2.000 K= 0.000 MON= 2.

TOTAL INT= 5943.7 77.1 31

BK= 2.2( 0.5) 10

NET INT= 5874.80( 78.45)

REL INT= 71.240( 0.251)

SCAN NUMBER 1

SAMPLE OUTPUT WITH IT=3

H= 2.0000 F= 0.0000

MON= 2.

CALC ANG: 14.408 29.018

STEP= 0.05

SCAN ANG	CATT ANG	COUNT	TOT
14.40	29.28	1.	1.
14.40	29.32	3.	7.
14.40	29.36	5.	12.
14.40	29.40	3.	14.
14.40	29.44	2.	16.
14.40	29.51	12.	24.
14.40	29.58	39.	49.
14.40	29.61	100.	152.
14.40	29.67	264.	462.
14.40	29.72	490.	822.
14.40	29.77	705.	1227.
14.40	29.82	940.	2067.
14.40	29.86	1216.	3283.
14.40	29.91	1452.	4735.
14.40	29.96	1467.	6202.
14.40	29.01	1338.	8034.
14.40	29.06	1129.	9162.
14.40	29.11	915.	10077.
14.40	29.18	523.	10501.
14.40	29.22	445.	11046.
14.40	29.26	256.	11302.
14.40	29.31	122.	11424.
14.40	29.38	22.	11446.
14.40	29.41	13.	11459.
14.40	29.46	4.	11463.
14.40	29.52	1.	11464.
14.40	29.57	6.	11470.
14.40	29.62	3.	11473.
14.40	29.66	2.	11475.
14.40	29.72	4.	11479.
14.40	29.77	0.	11479.

H= 2.000 F= 0.000 MON= 2.  
TOTAL INT= 11479.0 (107.0) 31  
BK= 3.1 ( 0.6) 10  
NET INT= 11382.90 ( 108.52)  
REL INT= 138.033 ( 1.316)

READ PACKAGE - Bragg reflection variables

DS - Step in angle  $\Delta\psi$  for sample table when

ST = 1 or 2; step in  $2\theta$  when ST = 3. (Note: the actual

$\Delta\psi$  used in the scan may be modified by the variable RC below).

NP - Number of points on either side of (H,K) peak.

Total number of points =  $2NP + 1$  (same for each scan).

ST - Scan type: 1 for sample rotation scan; 2 for  $\theta:2\theta$  scan; 3 for

$2\theta$  scan (with samp angle FROZEN).

NB - Number of points (out of total of  $2NP + 1$ ) on each side of

(H,K) to be used as background to calculate integrated intensity of the peak.

MF - Monitor scale factor (in kilocounts/minute). Used only to give

an estimate of total running time.

RC - resolution correction. RC allows the user to adjust the step

size of each scan to compensate for the change in the resolution

of the spectrometer. The step size  $\Delta\psi$  is determined by

$$\Delta\psi = DS \qquad 2\theta \leq 30^\circ$$

$$\Delta\psi = DS \left\{ 1 + RC \left( \frac{2\theta_{H,K} - 30}{70} \right) \right\} \qquad 2\theta > 0$$

Scan variables: For each scan NS there are three variables which must be input:

H1 - H

K1 - K

M1 - Monitor

Up to 80 scans can be stored in the computer, and up to 80 scans may be requested at one time for successive execution.

