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ABSTRACT

This report describes the programs developed over about five years at Lucas Heights for the processing of crystallographic data from manual and computer-controlled diffractometers. The description, theory, and input and output formats are given, but not the program listings.

Sections 2 and 3 describe the programs for handling, editing and reducing the 5-hole tape produced by manually-operated machines. Section 4 describes the revised versions of the programs associated with the computer-controlled diffractometers. It replaces the previous report AAEC/E191 as an operating manual for these instruments. Section 5 describes the programs for sorting, absorption-correcting and evaluating standard deviations. Section 6 describes the Job-Control procedures.

* Australian Institute of Nuclear Science and Engineering

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CONTENTS (continued)

1. INTRODUCTION

This report describes the programs used for data-handling and processing in the neutron-diffraction field at Lucas Heights (the AAEC's Solid State Physics Section, and the Neutron Diffraction Group of the Australian Institute of Nuclear Science and Engineering (AINSE)). Experimental procedure is omitted as well as angle-calculating procedures such as GASP (AAEC/TM500) or TASCAN, the computer-control programs MUD (AAEC/E191), and data-interpretation programs such as the refinement programs ORFLS and LINUS and the Fourier program. Descriptions are given of all programs and procedures for handling data between the experiment stage and the interpretation stage.

A variety of instruments, which we describe as 'manual', though they are really semi-automatic, produce data on 5-hole paper tape via the Series 150 (see AAEC/M73) automatic recording system. This data is usually very lengthy, because it contains the monitor and detector count for each individual step by the instrument. It often contains mis-punchings due to operational errors of the instruments. It must be transferred to magnetic tape, inspected, reduced and edited. For some instruments that completes the data-handling. For others the data passes on to processing programs. These arrangements for 5-hole tape are illustrated diagrammatically in Figure 1 and described in Sections 2 and 3. Programs handling the data are identified by 'FAF'.

The Computer-Controlled Diffractometers (CCD) produce 8-hole tape output in a much smaller volume because a record is produced for each Bragg reflection rather than for each step by the instrument. This record is transferred to magnetic tape, edited and interpreted. The program CODRUB which produces and refines orientation data for the CCD is also described. The flow is illustrated in Figure 2 and described in Section 4.

Bragg-intensity data requires further processing for absorption, Lorentz-polarization factor, and averaging of equivalents. These arrangements are illustrated in Figure 3 and described in Section 5.

Section 6 describes the Job Control Language procedures for running the programs.

2. THE ACQUISITION, HANDLING AND EDITING OF FIVE-HOLE TAPE FROM MACHINES CONTROLLED BY THE SERIES 150 EQUIPMENT

2.1 Data Acquisition

The 5-hole paper tape outputs are produced by several instruments controlled by the Series 150 equipment for data acquisition:

(a) 4-circle Manual Diffractometers – used for measuring Bragg intensities.

(b) The Small Angle Scattering Equipment – used for measuring spin interactions.

(c) The Long Wavelength Mechanical Chopper, X78 – used for measuring total cross sections.

(d) The Triple Axis Spectrometer 6H1 – used for measuring phonons.

(e) The Powder Spectrometers 4H1, 6H1 – measuring Bragg intensities from powders.

The programs process data from all of these to the point where the data is available to the user as a line-printer listing. Processing of the data from the first three machines is completed by running the appropriate programs: FAFPAC and FAFMIP for (a), FAFSAS for (b) and FAFMEL with the X78 option for (c). Detailed descriptions of these programs are given in Section 3. Final processing programs for (d) are not essential, though the FAFPAC program may be useful in locating the peak in the phonon scan. For (e) no processing program is available beyond the FAFMEL listings.

For all of these machines an 8-digit 'heading' is set manually on tumbler switches and punched at the beginning of the scan. The starting positions, step size and monitor counts are also manually set. For each step the monitor count M, the detector count C, and a third channel, 'field', are punched. ('Field' is used only by instruments (b) and (c)).

In this report the word 'record' means a set of three numbers: monitor, detector and field. A 'scan' is a heading, followed by a number of 'records'.

(a) Four-Circle Manual Diffractometers

The initial angle settings for a particular Bragg reflection (hkℓ) are calculated on the site computer using GASP. The details for running this program are given in AAEC/TM500. GASP also codes hkℓ into a 6-digit number which, with the user's experiment number (supplied by AAEC*) gives an 8-digit heading, which is used in the processing programs to identify each particular scan. The 'field' is always zero and is not used. FAFPAC and FAFMIP programs decode the heading into indices and experiment number.

(b) Small Angle Scattering Equipment

The last 2-digits of the heading must be the experiment number supplied by AAEC, but the user is free to allocate the first 6-digits. The 'field' indicates whether the magnetic field at the specimen is vertical or horizontal. It is used in the final processing program FAFSAS which determines the difference in counts between the two settings. 'Field' is reset whenever the scattering angle is changed.

(c) The Long Wavelength Mechanical Chopper, X78

'Field' may contain a cycle number from 1 to 6, or a time value, depending on the experimental conditions. When the cyclic mode is used the experiment number must be '78'. (For further detail see AAEC).

(d) The Triple Axis Spectrometer, 6H1

The heading, starting angles, step sizes and monitor count are calculated using program TASCAN. The 'field' is always zero. The scans are shorter than for most instruments, being usually 20 records. This may require special provision in FAFEDT.

(e) Powder Spectrometers 4H1 and 6H1

Apart from the last 2-digits, which must be '99', the heading is the user's choice or in the case of 4H1 the appropriate sequential job number obtained from the 4H1 log book. No program is needed to determine the starting position etc.

2.2 The Data-handling Arrangements

All 5-hole output tapes are processed by AAEC through the following programs:

PTPART – transfers the paper tape onto magnetic tape.

PTPGM – translates the punch code and performs a data format check; finds and labels the headings; and converts any illegal punch codes to X.

FAFEDT – performs straightforward editing, checks the monitor value for constancy and notes any inconsistent statistical variations between consecutive or cyclically related detector counts. A data set containing the reconstructed data is written.

FAFMEL – (without editing) – it lists the count and the cumulative count for each scan.

FAFHEL – gives a short list of the headings with the number of records in each.

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* In this context 'supplied by AAEC' will normally mean supplied by the Solid State Physics Section, Materials Division.

The programs PTPGM, FAFEDT, FAFMEL and FAFHEL are normally combined in a single procedure called NDFAFRUT.

The output listings of FAFEDT and FAFMEL are given to the user. From the FAFEDT listing he decides if he wishes to edit any records before further processing by FAFMEL, FAFPAC or FAFSAS (whichever is appropriate). If editing is required, the FAFEDT listing, with the necessary corrections clearly indicated and the next processing program (of the three above) stated, is returned to AAEC who will edit the data using FAFMEL and run the next program using the edited data, temporarily written on a disk by FAFMEL, as input. If no editing is necessary, the FAFEDT listing should be returned to AAEC who will run the next program using the FAFEDT output tape as input. The FAFEDT listing is not usually returned to the user.

Although it is not recommended, the user may edit his data himself as explained in Section 2.4 on FAFMEL. However the user must consult AAEC to find out which magnetic tape his data is on and to obtain extra editing cards to bypass other users' data which may be on the same tape.

At the date of this report the FAFEDT output is available on magnetic tape for editing and final processing for approximately two weeks after the paper tape is given to AAEC. After this time the magnetic tapes are required for more recent data. By prior arrangement with AAEC a particular magnetic tape may be kept for longer than two weeks. However a user should remember that this speeds up the turn-round on the remaining magnetic tapes, and other users may be inconvenienced. Data will always be available for at least one week.

2.3 Programs PTPGM and FAFEDT

PTPGM This is a program not normally run by the user so the details of the program description, input and output are relegated to Appendix 1.

FAFEDT (EDits Tape) is an updated version of ND5B1, originally written by Mrs. S. Hogg. It reads the PTPGM output, a scan at a time, looks for errors and corrects as many as it can (see below). It adds a serial number, checks the monitor value for constancy and the count for statistical errors, and stores the record for subsequent output on DSR 12* . Whenever a heading is found, the previous data is output and the serial number is reset to zero. If less than 'NMIN' records occur for a particular heading, no output of that scan on DSR 12 is made and the message 'this heading is too short, all . . . . . of its records deleted' is included in the listing. NMIN is normally set to 21, but a smaller value may be read in from the data card. As the program can only store 21 records before a batch output is made, NMIN must not exceed 21.

Provision exists in the program for excluding up to three specific headings from processing. The default option is 12345678. Users are requested to set this number on the Series 150 whenever they are adjusting their equipment, so that if any output onto the tape occurs by accident it will automatically be thrown away by FAFEDT.

It is also possible to process part of a tape by specifying the first and last headings required on an input card. This is done only in special cases, possibly when a second run of FAFEDT is required to recover short records.

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* DSR is used throughout this manual for Data Set Reference numbers. It is fully defined in Section 6.

2.3.1 X78 Data

The long wavelength mechanical monochromator X78 can take data for several specimens on a cyclic basis, in which case the third channel contains the specimen number. For this data the cycle of specimen numbers has to be determined for each heading and subsequently checked. The count has to be compared with the equivalent count in the previous cycle. These sections of the program are only entered when the experiment number is 78, for which reason users will never be allocated 78 as an experiment number. (In the program listing these sections are identified by 'X78ONLY' in cols 73–79).

2.3.2 Monitor Count Check

If the monitor count differs from that of the previous record by two or more the 'monitor count error' message is printed. If during the first 19 records after a heading, 10 consecutive monitor errors involving the same new monitor count are found, the monitor value is assumed to be the new value for subsequent tests and the message 'monitor reference changed to . . . . . from . . . . . .' is written. If a new monitor is not found in the first 19 records the program scans the count field to see if the monitor and count fields have been interchanged. If this is so the message 'first and second fields changed for this heading' is written, the 19 records already read have these fields interchanged and in all subsequent records for this heading the fields are interchanged before any checks on the data are made. If a satisfactory monitor value cannot be found the message 'monitor count too varied for heading . . . . . which is therefore skipped' is written, no output to DSR 12 is made and the input tape is read without processing until another heading is found. If the number of records in a scan is less than 19, the check for field interchange cannot be applied and it is possible (if NMIN<19) to output such scans on DSR 12. However, since all subsequent programs assume a constant monitor in the first field, short scans are not recommended.

2.3.3 Detector Count Check

If C is the present count, PC is the previous count (or equivalent count in the previous cycle for X78) and M is the monitor, the message 'count appears spurious (Comparison PC)' is written if

$$|C - PC| > 3(C + C^2/M)^{1/2} \quad .$$

2.3.4 Error Records

Error records are always read in 'A' format by using the second identifying digit of the previous record. If the first digit of the record indicates that the record is a heading containing errors it is converted to a heading if there is only one error (the error being converted to zero) or a dummy heading is inserted in its place.

For all other error records a heading is first looked for by scanning the record for:

(i) 'W' followed by 8 digits, then a blank.

(ii) 'W' followed by 8 digits including 1 blank, then a blank, with the correct experiment no.($\neq 0$).

(iii) 'W' followed by 8 digits and correct experiment no. ($\neq 0$).

(iv) 'W' followed by 6, 7 digits and a blank with correct experiment no. ($\neq 0$).

(v) 'X' followed by 7 or 8 characters, and a blank with correct experiment no. ($\neq 0$).

(vi) 8 characters and a blank with correct experiment no. ($\neq 0$).

(vii) 8 characters (no blank) with correct experiment no. ($\neq 0$) starting from the first two positions on the record only

If any of these tests are true the record is assumed to be a heading, and it is either reconstructed or a dummy heading is inserted.

If a heading is not assumed, the record is assumed to be a data record. The number and positions of all blanks and illegal characters (including 'W') are found and if more than half the record is incorrect it is rejected. The beginnings and ends of all non-blank areas of the record are then found. If there are three non-blank areas they are reconstructed as the three fields. If there are more than three, the three longest are used; if there are two, the longer is halved, and if only one, the area is divided into three approximately equal portions. If any non-blank area selected for reconstruction is longer than 6 characters the most significant digits are removed.

Whenever a record is reconstructed, either as a heading or as data, the input record is listed as well as the record in its reconstructed form. For a data record, if a monitor/count interchange has already been indicated for the heading, the interchange occurs before the message showing the reconstructed form.

FAFEDT is not usually run by the user so the Input details are given in Appendix 2.

2.4 Program FAFMEL

FAFMEL (Manual Editing and Listing) reads records from the magnetic tape produced by FAFEDT, edits them as required by the user, and lists individual counts and cumulative counts. The data may have come from the manual diffractometers, or from any other machines using the Series 150 data-recording system, such as the powder diffractometers, the triple-axis spectrometer, the small-angle-scattering instrument or the long-wavelength cross section instrument. It is routinely run through FAFMEL on the first running of NDFAFRUT. The FAFEDT listings are inspected and the normal procedure would be for the user to make editorial comment on the listings. These editorial adjustments would be translated into input instructions (normally by AAEC staff) as explained below, and the data re-run through FAFMEL to incorporate the changes on the output tape.

In the case of the manual diffractometers or triple-axis (6H1) spectrometer the output goes forward to FAFPAC, and in the case of the small-angle scattering instrument to FAFSAS. For the powder diffractometer and the long wavelength cross section instrument (X78) the FAFMEL output ends the computation.

For each scan processed FAFMEL outputs the heading and record tally onto a temporary disk file (DSR 21). FAFHEL (HEading Listing) reads and lists this file.

FAFMEL was written in FORTRAN IV for an IBM 360/50H in 1968 by the late J.R. Thompson to replace ND5B2 which was written by Mrs. S. Hogg. It has been corrected and updated in May 1970 by M. M. Elcombe.

2.4.1 Input for FAFMEL

Two input data sets are used, DSR 11, the FAFEDT output tape, is essential; DSR 1, cards, are necessary only when editing or special options are required. The details of the cards for editing runs are:

Card 1, Title card. (format 20A4), any alphanumeric characters up to column 64. The last 16 columns are used to set specific options as follows:

Cols.	Contents	Meaning
65–68	X78	X78 runs are included in the run. The special input deck for X78SUB must follow the title card and precede any editing cards.
69–72	TIME	A listing of the cumulative sum of the 'field' is given.
73–76	COPY	An edited version of the input tape is written on DSR 12, which would normally be a disk but could be a tape. This would normally be used for an immediate run of FAFPAC or FAFSAS.
77–80	CARD	A card deck of the counts only is produced, format 10I7,I6,I4 where 10I7 = counts, I6 = first 6 characters of heading and I4 is a serial number increasing by 10's. This option is not usual.

(For details of the X78SUB special input deck, reference can be made to the unpublished report on program ND5B2).

Editing cards (format I8,1X,I4,1X,A1,6X,3(1X,I6))

columns	1–8	10–13	15–21	23–28	30–35	37–42
	EDHEAD	EDISER	EDT	IE1	IE2	IE3

EDHEAD = heading of record on the input tape.

EDISER = serial number of record on the input tape (that is, ISER as written by FAFEDT).

EDT = edit code – see below.

IE1,IE2,IE3 = record information for editing.

The possible Edit codes are left justified with only the first character significant. The options are:

(i) END RUN. On reaching EDHEAD/EDISER the run is terminated. EDHEAD/EDISER is not processed. For complete output EDISER=0 or a PROCESS card must be inserted before the END RUN card, for example:

11111111 0000 END RUN – all data before this heading on the tape is processed and output.

11111111 100 PROCESS
11111111 100 END RUN } – all data before record 11111111/100 is processed and output.

(ii) INSERT. The record IE1,IE2,IE3 is inserted before record EDHEAD/EDISER. The following edit card may refer to the same record. To insert records at the end of a run the last record and the new ones must be inserted before the last record, and the last record omitted, for example, to insert 2 records after the last record(100)

11111111 100 INSERT IE1,IE2,IE3 = last record
11111111 100 INSERT IE1,IE2,IE3 = new record
11111111 100 INSERT IE1,IE2,IE3 = new record
11111111 100 OMIT 1 = omit last record

Headings may be inserted only by using the PROCESS option.

(iii) PROCESS. All records before EDHEAD/EDISER are processed and output, and the output serial number NISER is reset, as though a tape heading had been found. The next record processed after PROCESS must be a heading which can be obtained by an INSERT of a heading, or a SKIP TO a heading, or a SKIP TO a record and INSERT a heading, for example,

11111111 100 PROCESS
11111111 100 INSERT IE1,IE2,IE3 = heading record

or 11111111 100 PROCESS
22222222 0 SKIP TO

or 11111111 100 PROCESS
22222222 200 SKIP TO
22222222 200 INSERT IE1,IE2,IE3 = heading record.

(iv) OMIT. IE1 records starting with EDHEAD/EDISER are omitted. If IE1 is blank, 1 is assumed. To omit headings (ISER=0) from the input tape it is necessary to insert the last record before the last record, and then OMIT 2 records, for example,

11111111 100 INSERT IE1,IE2,IE3 = last record
11111111 100 OMIT 2 omit last record and following heading record.

(v) SKIP TO. All records up to but not including EDHEAD/EDISER are skipped. The point from which the skip starts is EDHEAD/EDISER of the preceding edit card, or the beginning of the tape if SKIP TO is the first edit card. If the start point of the SKIP is not being edited a LEAVE edit card must be inserted, for example,

11111111 50 LEAVE
22222222 100 SKIP TO } will omit records 11111111/51 to 22222222/99 inclusive.

or 22222222 100 SKIP TO will omit all records on the tape before 22222222/100.

(vi) REPLACE. The record EDHEAD/EDISER is replaced by IE1,IE2,IE3. Headings may be replaced except for X78 runs (see below).

(vii) LEAVE. The next edit card is read. Mainly used in conjunction with SKIP TO.

When a new heading, which is different from the previous heading, is found on the tape, any edit cards of the previous heading which have not been used are listed under that heading.

If an invalid edit code is found, LEAVE is assumed.

If no special options or editing are required the title card may also be omitted, in which case it is replaced by the date and time of the run.

The maximum experiment length provided for is 2048 data records. If this number is exceeded a PROCESS option is assumed and the 2049th record is treated as a heading.

As FAFMEL is designed to follow FAFEDT it expects the last record on a tape to be a heading. If not, the current heading only is listed and output to tape (if COPY is specified) but not to FAFHEL. All data for that heading is ignored.

(a) To Replace and Insert X78 Headings

The experiment number, 78 must be correct on the input tape for FAFMEL to recognise it. If this is not so, two runs are required, the first one correcting the experiment number and writing a new tape (using the COPY option) and the second one processing the data as X78 data. If the second (code) heading has been omitted but the experiment number (=78) is correct a PROCESS and INSERT before the first record is allowed, and the PROCESS option in this case produces no output, for example,

11111178 1 PROCESS
11111178 1 INSERT IE1,IE2,IE3 – code heading.

X78SUB was originally written as part of a complete IBM7040 Fortran IV program by Mrs. S. Hogg. It has been converted to an IBM 360 Fortran IV subroutine.

2.4.2 Output from FAFMEL

For runs other than from X78, FAFMEL edits them and places the count in COUNTS(1,) and the current cumulative count in COUNTS(2,) and the cumulative field in COUNTS(3,). The whole of the record is stored in OUTPUT. On completion of a scan, COUNTS is listed and (if required) OUTPUT written on DSR 12. (COUNTS(3,) is only listed if TITLE(18) = TIME).

3. FURTHER PROCESSING OF THE FAFEDT TAPES

3.1 Program FAFPAC

FAFPAC (Peak Automatically Counted) integrates the Bragg peaks from the manual diffractometers. It accepts the tapes with the individual counts and does a 4-parameter least-mean-squares fit to them (background, peak amplitude, peak width and peak position) and so obtains a net intensity. It is entirely a computer procedure. Occasionally, on very weak peaks, the fitting procedure diverges. The user may then use FAFMIP (see Section 3.2) to try to recover something from his data.

The main use of the program is in evaluating Bragg intensities. It may also be used to evaluate phonon data originating in the 6H1 or 10H instruments. (Indeed the first draft was written by Mr. A. W. Hewat of Melbourne University for just this purpose).

Cards containing net intensities and their estimated standard deviations (e.s.d.'s)* are produced normally for input into DRACULA or VARISORT.

The program was written by M. M. Elcombe.

3.1.1 General Description of FAFPAC

The input data comes from magnetic tape. Each scan has its heading followed by a maximum of 2048 records each containing a serial number ISER (inserted by FAFEDT), M (the monitor count), C (the detector count) and the 3rd channel (which is used only in FAFSAS). When the serial number ISER returns to zero the scan is complete and the fitting procedure to that peak is initiated. For Bragg peaks the program decodes the heading into Miller indices.

The fitted peak may be Lorentzian or Gaussian.

The monitor count for different peaks may differ. (The editing in FAFMEL should ensure that it is unchanged for any one peak). If so, the final intensities may be set on a common scale of monitor count specified by reading in OALLSC.

If only some of the many scans on one magnetic tape are to be processed, the first and last headings may be specified.

A group-identifying number (1 to 9) may be punched on the output cards if required.

3.1.2 Mathematical Theory

Let n = the step number (ISER)

c_{no} = the observed count at step n.

Then the Gaussian expression for the calculated count c_{nc} at step n is

$$c_{nc} = b + p \exp\left[-4\ell n2 \left(\frac{n - n_p}{n_w}\right)^2\right],$$

* e.s.d. will be used throughout this manual for estimated standard deviation. For any value A it will be represented by $\sigma(A)$.

where b = background (parameter 1)

p = peak height (parameter 2)

n_w = full width at half maximum height (parameter 3)

n_p = position of peak (parameter 4).

The Lorentzian expression is

$$c_{nc} = b + p\left[1 + 4\left(\frac{n - n_p}{n_w}\right)^2\right]^{-1} .$$

The weight W_n is assumed to be that given by the statistics of c_{no}, and the monitor count M,

$$W_n = \left(c_{no} + c_{no}^2/M\right)^{-1} .$$

The function minimized in the analysis is

$$\chi^2 = \frac{1}{N-4} \sum_n W_n (c_{no} - c_{nc})^2 ,$$

where N is the total number of counts.

After fitting, the counts are re-checked to reject spurious, noise-affected, counts. The quantity $W_n^{1/2}\ (c_{no} - c_{nc})$ is assumed to be normally distributed with χ^2 variance. For each point the quantity k_n, given by

$$(n - 4 - 1)\, k_n^2 = (n - 4)\, \chi^2 - W_n (c_{no} - c_{nc})^2 ,$$

is calculated and $S_n = k_n c_{nc}^{1/2}$ is taken as the best estimate of the standard deviation of c_{no}. If $[|c_{no} - c_{nc}|/S_n] > t$ the point is rejected, t being the appropriate value for 0.2 percent probability in a 't'-distribution with $(n - 4 - 1)$ degrees of freedom, and is obtained by interpolation from a statistical table incorporated in the program (see Table 1). If any points are rejected the curve is refitted without them and the significance test is repeated.

Finally the intensity I is calculated from

$$I = A\, p\, n_w ,$$

where $A = (\frac{\pi}{4}\ \ell n 2)^{1/2}$ for the Gaussian curve and $\pi/2$ for the Lorentzian curve. The standard deviation in I is given by

$$\sigma^2(I) = A^2 [n_w^2\, \sigma^2(p) + p^2\, \sigma^2(n_w) + p n_w \sigma(p)\, \sigma(n_w)\, C_{23}] ,$$

where $\sigma(p)$, $\sigma(n_w)$, and C_{23} the correlation between p and n_w, are all obtained in the least-mean-squares fitting of the four parameters.

I and $\sigma(I)$ are also obtained by straightforward numerical summation. In Figure 4a suppose steps n_1 and n_4 are the beginning and end of the scan and the peak occupies the range from step n_2 to step n_3. The program determines n_2 and n_3 as follows:

$$n_2 = n_p - 1.5\, n_w$$

$$n_3 = n_p + 1.5\, n_w .$$

TABLE 1

STATISTICAL TABLE FOR 0.2 PERCENT PROBABILITY AND N DEGREES OF FREEDOM

(a) $N < 5$ SIGLEV = 10 and message written

(b) No interpolation

N =	5	6	7	8	9
SIGLEV =	5.89	5.21	4.79	4.50	4.30

(c) $N \geqslant 10$ Linear interpolation in 120/N

N =	10	12	15	20	24	30	40	60	120	∞
120/N =	12	10	8	6	5	4	3	2	1	0
SIGLEV =	4.14	3.93	3.73	3.55	3.47	3.39	3.31	3.23	3.16	3.09

For example:

if $N = 35$

$$\text{SIGLEV} = 3.31 + \frac{(3.39 - 3.31)}{(4 - 3)} * \left(\frac{120}{35} - 3\right) .$$

(Values are taken from 'Percentage Points of t-Distribution' Cambridge Elementary Statistical Tables by D.V. Lindley and J.C.P. Miller, C.U.P., 1958).

Then if

$N_B = (n_2 - n_1) + (n_4 - n_3)$ = number of background counts

$N_P = n_3 - n_2$ = number of peak counts

$S = N_P/N_B$

$P = \sum_{n_2 \text{ to } n_3} C_{no}$ = total intensity

$B = \sum_{n_2 \text{ to } n_1} C_{no} + \sum_{n_4 \text{ to } n_3} C_{no}$,

the net intensity

$I = P - SB$.

The standard $\sigma(I)$ is given by

$$\sigma^2(I) = \left(\frac{\partial I}{\partial P}\right)^2 \sigma^2(P) + \left(\frac{\partial I}{\partial B}\right)^2 \sigma^2(B) ,$$

where

$$\sigma^2(P) = P + P^2/(M.N_P)$$
$$\sigma^2(B) = B + B^2/(M.N_B) ,$$

so finally

$$\sigma^2(I) = P + S^2B + (P^2 + S^3 B^2)/(M.N_P) .$$

It is this I and $\sigma(I)$ (scaled to the required monitor count) that is the normal card output of FAFPAC. When the fitted limits of the peak lie outside the scan, that is, $n_2 < n_1$ or $n_3 > n_4$ or both, the procedure is not possible and the deck is supplemented by cards, flagged 'TTTT' in cols. 77–80, containing I and $\sigma(I)$ (also scaled) calculated from the fitted parameters. If the user prefers card output calculated entirely from fitted parameters the remainder of the 'TTTT' deck may be obtained by defining DSR 10 as the card punch (see Section 6 on JOB CONTROL).

3.1.3 Discussion of Use

The Lorentzian fit should not normally be used as it sets too low a background.

Regarding the two possible ways of obtaining I and $\sigma(I)$, called briefly 'summed' and 'fitted', it should be noted first that in most cases the two methods agree very well, when the Gaussian option is used, as may be seen by inspecting the FAFPAC listings. When the peak is skew, or double-humped, or when the background is not level on either side of the peak, the 'summed' method is to be preferred. Then the only role of the fitting procedure is to provide the summing range.

If the peaks are so poorly centred in the scans that summing is not possible, then only the fitted intensity is available. But even when the summing range falls inside the scan, unless there is plenty of background on either side it is probably better to use the fitted rather than the summed intensity. Roughly, unless the whole scan contains at least 5 n_w steps, the fitted intensity is preferable to the summed, always excepting the case when the peaks are very skew or double-humped, phenomena which may first be detected by notable discrepancies between the summed and fitted intensities, plus numbers of consecutive counts being rejected as 'noise'.

Sometimes, for low intensities, the fitting procedure diverges because no peak centre n_p can be detected. In such cases the user trying to reclaim such data can only go to FAFMIP, specifying the summing range the best way he can.

3.1.4 Input for FAFPAC

(a) Cards

Card 1 – Title (format 20A4), (default option – date only)

Card 2 – ITYPE, OALLSC, JHEAD, KHEAD, IGP (format I4, 3I8, A1),

where ITYPE = 1 for Gaussian curves, = 2 for Lorentzian curves (default option = 1).

OALLSC = monitor value to which all counts are scaled (default option = 10000).

JHEAD, KHEAD = first and last headings on the magnetic tape, if only part of the tape is to be processed (default option – first and last actually on the tape).

IGP = group number to be punched on cards (default option – blank, which is acceptable to DRACULA if there is only one group).

(The second card, or both cards, may be omitted, in which case all default options are assumed).

(b) Tape (DSR 11) (format I4, 3I6).

This is the magnetic tape from FAFEDT, or, if extra editing has been necessary, from FAFMEL.

3.1.5 Output from FAFPAC

The Listing contains a complete summary of the fitting procedure, which should be self-explanatory as a glossary of terms is given on the first page.

The Cards (one for each reflection) have the format:

'Z', I4, 2I5, 2I8, 24X, A1 if intensities were 'summed',

or 'Z', I4, 2I5, 2I8, 24X, A1, T77, 'TTTT' if 'fitted' and contain hkℓ, $K_M I$, $K_M \sigma(I)$ and IGP.

The 'Z' on every card ensures that DRACULA treats each reflection individually. If groups of reflections are to be averaged in DRACULA the cards must be ordered and these 'Z' flags reset appropriately by a run of VARISORT (see Section 5.1). $K_M I$ and $K_M \sigma(I)$ are the values of I and $\sigma(I)$ scaled to the required monitor, K_M = OALLSC/M.

3.2 Program FAFMIP

FAFMIP (Manual Integrating Program) was a stop-gap program which has now been supplanted by FAFPAC. When FAFPAC doesn't work, which occasionally happens on weak peaks when a peak centre n_p cannot be found, FAFMIP may still be used in order to reclaim the data. Its purpose and output are the same as for FAFPAC.

The program was written by F. H. Moore.

3.2.1 General Description of FAFMIP

A plot of the cumulative listing from FAFMEL may be more or less as shown in Figure 4b. By inspection of the FAFMEL listings the user must decide on the four step numbers n_1, n_2, n_3, n_4, as shown in Figure 4b, where the n_1-n_2 section and the n_3-n_4 section are background, and the n_2-n_3 section contains the whole peak; n_1 and n_4 would commonly be the first and last steps.

From the FAFMEL cumulative listings he then reads S_1, S_2, S_3, S_4, the cumulative counts corresponding to n_1, n_2, n_3, n_4. The set of data hkℓ, M (monitor count), n_1, S_1, n_2, S_2, n_3, S_3, n_4, S_4 is then punched on cards and FAFMIP accepts these cards as input.

The intensities are referred to a common scale of monitor counts read in as OALLSC.

A subroutine PBCORR is available, or may be supplied by the user, to make corrections for 'noisy' counts. However in view of the marginal value of the entire program the details are not described. (They are available from AINSE).

The theory of the calculation of I and $\sigma(I)$ is as given for FAFPAC in Section 3.1.2.

3.2.2 Input and Output for FAFMIP

Card 1 TITLE, OALLSC (format 18A4, F8.0)

Reflection Cards

hkℓ, M, n_1, S_1, n_2, S_2, n_3, S_3, n_4, S_4, IC (format 5X, 3I5, 4X, I6, 4(I3, F7.0), I10),
(where IC = 1 if the noise-correcting subroutine PBCORR is to be called, in which case an additional card is needed).

Terminating Card 'END' in cols. 1–3.

Cards are produced suitable for VARISORT or DRACULA, as described in the output specification of FAFPAC.

3.3 Program FAFSAS

FAFSAS (Small Angle Scattering) is designed to read the output magnetic tape of FAFEDT or FAFMEL. In this case the 'field' channel is used. For each position of the instrument a preset number, L, of count cycles is taken. A cycle consists of two counts, the first taken with the magnetic field horizontal and the second with it vertical. The third channel (field) is ℓ for the first count and $1000 + \ell$ for the second, where $0 < \ell < L$. When $\ell = 0$ the position of the instrument has been changed and the previous data are processed. Isolated errors in ℓ can be dealt with by the program and suitable messages are written. Data processing also occurs when a new heading, which is different from the previous one is found. The processing consists of calculating the difference between the mean counts with the field horizontal and vertical and scaling it to a preset monitor count (OALLSC). At the end of the data on the tape or a new heading or when 66 calculations have been made, two plots are drawn on the line printer, the first of the mean horizontal count H against position (to assist in determining at what point the counter enters the main beam) and the second of the difference (H–V) against position. The shape of the second curve is used in determining the magnetic interaction constants of the specimen.

The position of the instrument is determined from an initial dial gauge reading and its increment. These values can either be read in as data cards, one for each new heading, or derived from the first 6 digits of the heading (the last 2 digits are the user's experiment number).

The program was written by M. M. Elcombe.

3.3.1 Mathematical Theory

N counts C_n are taken under identical conditions. Assuming unit weights, the mean and variance of the counts are calculated as

$$\bar{C} = \frac{1}{N} \sum_{n=1}^{N} C_n$$

$$\sigma^2(\bar{C}) = \frac{1}{(N-1)} \left[\Sigma (C_n)^2 - \frac{1}{N} (\Sigma C_n)^2 \right] .$$

It is then assumed that $(C_n - \bar{C})$ is a NORMAL distribution with $\sigma^2(\bar{C})$ as variance and possible noise points are tested for. For each point, k_n given by $(N-2)k_n^2 = (N-1)\,\sigma^2(\bar{C}) - (C_n - \bar{C})^2$ and $\bar{C}_n$ given by $(N-1)\,\bar{C}_n = N\bar{C} - C_n$ are evaluated and the point is rejected if $\frac{|C_n - \bar{C}_n|}{k_n} > t$,

where t is the appropriate value of the 't'-distribution for 0.2 percent probability with $(N-1)$ degrees of freedom (see FAFPAC, Section 3.1.2).

If any points are rejected, the mean and variance are recalculated without them and the rejection tests repeated, up to a maximum of four times. If points are still rejected at this stage a message is printed to this effect and the user should re-examine his data.

The final mean and variance of the array and the number of points used in the last cycle of calculation are returned to the main program.

This is done for both the horizontal counts, giving $H \pm \sigma(H)$ with NHU points, and the vertical counts, giving $V \pm \sigma(V)$ with NVU points. The values of H–V, $\sigma(H-V) = (\sigma^2(H) + \sigma^2(V))^{\frac{1}{2}}$ and $|100 \times \sigma(H-V)/(H-V)|$ are calculated, scaled by K_M = OALLSC/M, and listed, together with the counts actually used (those rejected are listed as zeros).

3.3.2 Input and Output for FAFSAS

Main input is from DSR 11 which is the FAFEDT magnetic tape output or the FAFMEL DSR 12 output.

The following cards are also required.

Card 1 Format (20A4), TITLE any alphanumeric characters.

Card 2 Format (I4,F8.0, 2I8) contains

ICODE = 0 when position decoded from heading
= 1 when initial position and increment for each heading are to be read in from cards (default option 0)

OALLSC = overall scale factor (default option 10000)

JHEAD = heading with which data processing is to start (default option is beginning of DSR 11)

KHEAD = heading at which data processing is to stop (default option is end of DSR 11).

Card 3 Format (2F10.5) required only if ICODE = 1. One card is required for each new heading.

DIAL = initial dial gauge reading (default value = 0.0)

INC = increment value of dial gauge (default value = 1.0)

If more new headings are found than cards supplied, the default values are assumed.

The Output is a listing only with self-explanatory messages.

4. THE COMPUTER CONTROLLED DIFFRACTOMETERS

4.1 The MUD Instruction Code

The method of operation of the Computer-Controlled Diffractometers is described in AAEC/E191 and the basic approach has not been changed. Two spectrometers have now been interfaced to one PDP8/L computer to which an index register has been added (AAEC/TM525). Both spectrometers run independently of one another, using the same copy of the program, but their own data areas, which are accessed via the index register. In two years of development of the diffractometer program (now retitled MUD – Multiple Use Diffractometer), of the angle refinement program CODRUB, and of the editing program CODOEP, many changes in detail have been introduced which pertain to the problems of collecting and interpreting data. In particular, to understand how CODOEP edits the data it is necessary to understand in detail the peak-measuring routine DCOLL described in Section 4.2.

Table 2 inserted at the end of this report, is a brief summary of the MUD Instruction Code.

Entering an Instruction

Unlisted instructions are not 'echoed' (that is, typed at the console in acknowledgement), nor are invalid characters in data fields.

The items of data are interpreted by the program as 'real' or 'integer' but are not required to be so typed (for example 2.0 will be interpreted as 2 if the program expects an integer, and 90 will be interpreted as 90.0 if it expects a real). Zero data may be omitted.

Each item of data must be terminated by a colon (:). Successful entering of all the data for any instruction is signalled by either a carriage-return-line-feed at the teletype or by the start of motor movements on the spectrometer. All four motors on each spectrometer may move simultaneously, thus before a motor has reached its destination the operator can type commands to move the others.

If a mistake in typing is made, a RUBOUT will delete to the last colon terminator, though it may be better to start the whole instruction again.

All numbers are reduced to an allowed range, so that invalid data can never 'bomb' the program.

Instructions and data may be entered by typing or reading tape. After executing an instruction the computer will always read tape looking for a valid instruction. To enter at the typewriter the operator must first switch off the tape-reader. To interrupt, he must, of course, use the typewriter.

NANG and XANG

The definition of these numbers must always be borne in mind.

The Axis Number NANG = 1 for the 2θ axis

= 2 " " ω "

= 3 " " χ "

= 4 " " ϕ "

= 0 " 2:1 ω:2θ motion.

The Motor-Pulse Number XANG = the number of 200^{ths} of a degree (that is, the number of pulses to the stepping motors).

Instruction to Drive Motors

'Desired Angles'

A call on the motor-driving routines moves all axes to the positions held in the computer as 'desired angles'. These angles are established normally by /CA, but /PA will put the actual current angles in these locations. The 'desired angles' can be changed one at a time by /DM, or incremented by such instructions as /SS, /SC.

/ZA (+4) – Zero Angles

Required when starting from cold, to set the angles in register. Data: the four physical positions of the shafts to an accuracy of ± 10°. (If the angles are fed in ± 0.01° then no ERROR messages occur).

/DM (+2) – Drive Motors

Simply drives the designated axis to the designated position. Further instructions may be typed before the driving is finished. Data: NANG and the Angle. The angle 200° may be specified, say, as 200, 200.0, –160, or even as 560 or –520. If on the 2θ axis it is not accessible to the instrument, a 'LIMIT' message will be typed with no action. NANG = 0 is meaningless for /DM.

/IM (+2) – Increment Motor

Increments the designated motor by the designated number of motor pulses (200^{ths} degree). Data: NANG and XANG, for example, /IM 3:20: moves χ by 0.1°

/DA – Drive to Angles

Drives the motors to the positions held in the 'Desired' locations. The sole use is to follow /CA (see below).

Instructions to Insert Data

/SM (+2) – Set Monitor

Sets the timing for a single count; a pre-requisite to any counting instruction. Data: the first number is 0 if it is desired to count against monitor and 1 if it is desired to count against a real-time clock of 1 kHz. The second number is the number of times 1024 pulses, for example with /SM 1:4: a count is for 4.096 seconds.

/RW (+1) – Read Wavelength and /UB (+9) – UB-matrix

Require as data the wavelength and the 9 elements of the UB matrix by rows. Both instructions are pre-requisite to any call for angle calculations.

/IS (+5) – Insert Standard

In the routines which measure a series of Bragg peaks a standard reflection may be inserted after every N reflections. Data: N, hkℓ, and -m, where the first standard is to occur after m-1 reflections, for example /IS 20:2:::-1: will insert the (200) as a standard, every 20 reflections starting at the first; while /IS 20:2:::1: would insert the (200) every 20 reflections starting at the 4095^{th}! The axis zeros are checked at every standard insertion.

/NS (+1) – Number of Steps

Enters NSTEP the number of 200^{th} degrees in ω in the ω-2θ scan to measure peak intensity. A pre-requisite for any call on DCOLL.

Counting, Scanning, Searching Instructions

/TC – Take Count

Takes a single count and types it. Special programming arrangements reject spurious, noisy counts (Cox 1971).

/SS (+ 2) – Step Scan

Steps and counts on a designated axis. Data: NANG and XANG. Note that /SS:: conveniently takes repeated counts without moving axes.

/SC (+3) – Search for Count

Steps and types the angles and count when the designated count is first exceeded. Data: NANG, XANG, Count.

/CA (+ 3) – Calculate Angles

Calculates and types the angles for a designated reflection. Data: hkℓ. If, having inspected the values, the operator wishes to drive there he will type /DA.

/CR (+1) – Centre Reflection

The steps are: (i) do an ω-scan of from -10 to +10 steps of 0.1° about the current position to find a peak; (ii) centre χ on half-peak positions; (iii) centre ω on half-peak positions; (iv) centre the designated axis on half-peak positions. Data: NANG for the axis of step (iv), normally 1, to centre 2θ. If zero, step (iv) is not executed. Note that near the 'parallel position', where χ-motion will not move the beam vertically in the counter, a reflection cannot be centred. It is advisable to inspect the ω-scan of step (i); if the peak-to-background ratio is not well over 2 the peak cannot be centred. Finally the centred angles are typed and the motors drive to them and a final count is taken. If this final centred count is much greater than the maximum count in the coarse ω-scan it is advisable to repeat the centring.

/MR – Measure Reflection

Calls DCOLL at the 'desired angles': would normally follow instructions like /CR or /CA.

/AS (+1) – Azimuth Scan

Performs an azimuth step-scan starting from the 'desired angles'. Data required is the ψ step in tenths of a degree. As the calculations are incremental ($\delta\omega$, $\delta\chi$, $\delta\phi$ are calculated with $\delta\psi = 0.1°$), this works best when $\chi > 45°$.

Printing Instructions

/TN – Type On and /TF – Type Off

These instructions may be entered at any time without interrupting the operation. In programs such as scanning (/SS, /SC, /AS, /CR) or data-collecting (/MR or data-collecting below) the individual counts are typed in lines of 8 if /TN, and not so if /TF. For example, /SS with /TF types nothing, while the DCOLL routines with /TF would type hkℓ, angles and peak data, but not the individual counts.

/PA – Print Angles

Types the current angle positions in NANG order. It is usually entered with axes stationary.

Interrupting

/OF – Off,, /CP – Cancel Program and /??

/OF interrupts the current program after it finishes what it is doing; for example, /SS would be interrupted after finishing a step, /CD after finishing a reflection. /CP interrupts immediately.

In terms of interruptibility, all instructions may be divided into two classes: Type I(/TN, /TF, /OF), and Type II (all others except /??). When entered from the paper tape reader, the question of interruption does not arise, as the paper tape reader is always disabled until the completion of the current instruction. However, instructions may always be typed in at the keyboard (with the paper tape reader off), and if this is done before completion of the current instruction (that is, before the paper tape reader is willing to read tape), the ensuing action falls into two categories. For Type II instructions, the current instruction is terminated immediately (except for motor movements which continue until the current destination is reached), and the new instruction is immediately acted upon. Type I instructions do not terminate the current instruction, they merely set up a change in the condition of operation.

The instruction /?? is the most drastic terminating instruction; it stops execution of the current instruction, resets all program flags, stops the motors from driving and returns to the waiting loop in the program. It is only used if the program appears to have jammed.

Tape-Managing

/PL – Punch Leader, /DS(+1) Diffractometer Status, and /PH (+ alphameric information) – Punch Heading

Starting a new tape or a new experiment, type /PL, /DS, /PH.

/PL punches three feet of blank leader.

/DS reads the group number, which is transferred eventually to DRACULA; it punches the diffractometer number, the wavelength, the UB-matrix, the standard reflection, the monitor settings and the group number. These are needed by CODOEP.

/PH punches a heading which is terminated by any valid instruction. This heading is preserved through to the CODOEP listings.

Anything punched is also typed. But much that is typed is not punched. The punch is called only by the DCOLL routine, and by the instructions /PL, /DS, and /PH. The punched record is therefore 'clean', containing only the information required by CODOEP. (This applies only when the diffractometer is operating with a separate fast punch. If, as may become common practice, the punched tape is coming from the teletype, the user must keep it as clean as he can).

4.1.1 Data-Collection Instructions

/IR (+3S) – Individual Reflections

Reads sets of $hk\ell$'s, normally from tape, and calls DCOLL.

/IA(+4S) – Individual Azimuths

Reads sets of $hk\ell$, ψ in $(1/10)^{th}$ degree steps from the bisecting position, and calls DCOLL.

/CD (+17) – Collect Data, /CC (+6) – Continue Collection, /RD – Resume Data-collection and /RP (+1) – Read Program

This set of instructions enables the user to select a particular sequence of points in reciprocal space for a program to work on. A number of different programs are available (currently three)

to work on the generated sequence of points, and the program being used may be changed at any time.

The specification of the sequence of points to be used is initiated by using the /CD instruction, together with 17 integers. In the following text, an underlined symbol represents a reciprocal lattice vector which must be entered as three integer fields. The sequence of points is generated by starting at a given point, HKLO, stepping by a given incrementing vector, ΔHKL1, to form the next point, and repeating until the row of points so generated contains a specified number of points, N1. A further row of points is generated by shifting all points of the previous row by a second incrementing vector, ΔHKL2, and repeating until the plane of points so generated contains a specified number of rows, N2. A further plane of points is generated from the previous plane by addition of a third incrementing vector, ΔHKL3, and repeating until the lattice so generated contains a specified number of planes, N3. An over-riding consideration which determines whether or not a new generated point is to be operated on by the current program is that it must lie in a spherical annulus in reciprocal space which is specified by (integral) minimum and maximum counter angles, $2\theta_{min}$ and $2\theta_{max}$.

The /CD instruction of course may be specified to run for a very long time, and it may be required to interrupt it for some purpose, for instance if the output paper tape needs changing, one would want to use a /PL /PL /DS /PH sequence, or one might want to use /SM, /RW, /UB, /RP or even /IR. The current position of the reciprocal lattice point generator is not affected by any instruction other than /CD, /CC, /RD, and if interrupted in any way, data collection may be resumed with the instruction /RD. The first point generated by /RD is the one which was interrupted.

It is possible to specify a volume of reciprocal lattice points and a spherical annulus in reciprocal space with the /CD instruction, and then either immediately, or after some time, start generating a new sequence of points on the same framework, but starting from a point other than the original /CD origin or the current point. To do this, use the /CC instruction (continue collecting data), and specify the new origin HKLO, and the numbers (I1, I2, I3) of points, rows and planes to be generated on the first row, plane and lattice respectively. I1, I2, and I3 must include the restarting point, that is, their minimum values are 1. Second and subsequent rows contain N1 points, the number specified in the original /CD instruction, and similarly, second and subsequent planes contain N2 rows of points.

Two common situations may require the use of this facility; (1) collection of data may have proceeded beyond a point where some equipment malfunction occurred, necessitating restarting at some earlier point, or (2) different subsets of data may need to be collected under different conditions, for example, even and odd reflections of a f.c.c. lattice. After collection of the even reflections, /SM may be used and the data collection restarted with /CC at an odd origin.

Three programs are currently available to operate on the generated points; each is specified by a different value of the single input integer following /RP.

/RP 1: – DCOLL. This uses the DCOLL routine to collect the integrated intensity for each specified point. /IS is operative, and standards are inserted at the specified frequency.

/RP 2: – Diffuse Scan. This uses a routine which takes a single count at each reciprocal lattice point before going to the next point. The counts are printed out under the type option /TN or /TF. A print-out of the indices and setting angles of the first reflection in each row is also obtained. /IS is not operative, and standards are not generated. This routine may be used to do linear scans through diffuse scattering regions of reciprocal space by decreasing the wavelength by some suitable factor and increasing the Miller Indices by the same factor (for example, 100). Beware, however, of unexpected results if any integer greater than 2047 is used or generated.

/RP 3: Print Generated Points. This program does not perform any spectrometer activity, but merely prints the indices of the generated reflections on the teletype so that the user can check that the /CD he proposes to use will generate the points he requires. If the check proves successful, the normal procedure is to change the program with /RP and reload the /CC or /CD instruction to start again. /IS is not operative for this program.

Examples of the Use of the Instructions /CD, /CC, /RD

All of this discussion applies to the collection of integrated intensities using /CD with the /RP 1: program. By a careful choice of incrementing vectors, it is usually possible to arrange to omit from the set of generated points equivalent reflections and systematic absences due to lattice type. The following examples illustrate this.

(1) Collection of data for a monoclinic cell, ranging through values $-9 \leq h \leq 9$, $0 \leq k \leq 5$, $-6 \leq \ell \leq 6$, and $2\theta \leq 70°$. ℓ is to vary fastest and k slowest. This will be achieved by the instruction.

/CD 0:0:1: 1:0:0: 0:1:0: –9:0:–6: –13:–19:–6: 0:70:

(2) If $Ok\ell$ for $k+\ell$ odd data are required to be omitted from the data in example 1, more than one instruction must be used, for example:

/CD 0:0:2: 0:0:0: 0:1:1: 0:0:–6: –7:–1:–6: 0:70:

collects $Ok\ell$ with $k+\ell$ even, and

/CD 0:0:1: 1:0:0: 0:1:0: –9:0:–6: –13:–9:–5: 0:70:

collects all $hk\ell$ for negative h, and

/CC 1:0:–6: –13:–9:–5:

continues this data collection for positive h. Note particularly that I1, I2, I3, N1, N2, and N3 refer to ΔHKL1, ΔHKL2, and ΔHKL3, not to h, k, and ℓ.

(3) Collection of data for a body-centred lattice ($h+k+\ell$ even) with all octants of data ranging up to $h=k=\ell=6$.

/CD 0:0:2: 1:1:0: 2:0:0: –6:–6:–6: –13:–13:–13: 0:120:

Reflections generated are

```
. . .   . . .   . . .   . . .
. . .   002     004     006
. . .   112     114     116
. . .   222     224     226
. . .   . . .   . . .   . . .
. . .   202     204     206
. . .   312     314     316
. . .   . . .   . . .   . . .
```

To generate those with ℓ odd, follow this with

/CC –7:–6:–7: –13:–13:–13:

(4) Collection of data for a face-centred cubic lattice (hkℓ all even or all odd) with $0 \leq h \leq k \leq \ell \leq 10$ specifying that no equivalents should be measured, use

/CD 0:0:2: 0:2:2: 2:2:2: 0:0:0: –6:–6:–6: 0:90:

to measure the even reflections. For the odd reflections use /SM if a different monitor value is required and then

/CC 1:1:1: –5:–5:–5: to collect the odd reflections.

4.2 The Peak-Measuring Procedure, DCOLL

In the MUD program, as in the previous CCD program, the diffractometer measures in an ω-2θ scan about the calculated position, and there is normally no chart display of the peak profile. Therefore, it is essential to check, where possible, that the peak is well covered by the selected scan range. The MUD program determines, for peaks strong enough to make it possible, the half-height positions of the peak profile, and the editing program CODOEP analyses these observations and makes comments on the output listing.

The peak measuring procedure has been changed substantially from that detailed in AAEC/E191. The new procedure gives better optimization of the counting times and produces output which can be more usefully interpreted by a subsequent data processing program. The procedure after calculation and positioning of angles is now as follows.

(1) Print and punch the Miller indices and current angles.

(2) Move ω-2θ by ½ N_P increments of ω in a negative direction to the start of the scan. (One increment = $1/200^{th}$ degree).

(3) Scan the peak in steps of $1/100^{th}$ degree, taking ½ N_P steps altogether. (Round down if N_P is odd). The total cumulative intensity for this first scan is recorded as P1.

(4) Move ω-2θ by a further N_P increments to a position $3/2\ N_P$ increments from the starting position.

(5) Take ½ N_B background counts at the same position to make a total count of B1, where N_B is determined by the criterion

$$N_B^3 \geq \frac{B1 + 2}{P1} : N_P^3$$

with the absolute proviso that N_B must not exceed N_P.

Except for the (B1÷2) term this relation can be derived as the correct way to apportion the background and peak counting times to achieve optimum counting statistics when the total time is constant. This is approximately the present situation where the peak scan counting time is constant. The (B1÷2) term is actually (B1) in the rigorous derivation: the 2 is added here to prevent a low deviate first count (for example, zero) from terminating the background counting prematurely.

The upper limit on N_B is used to ensure that the accidental occurrence of a high count at the first background position does not tie up the spectrometer for a long time on collecting data which will probably turn out to be useless.

(6) Move ω-2θ to the position $(-3/2\ N_P - 1)$ increments from the starting position. (The −1 is so that points on the second peak scan will interleave those on the first).

(7) Count ½ N_B background points again. N_B this time has the value it achieved on the previous background count.

(8) Move N_P increments to a position $(-\frac{1}{2}\ N_P - 1)$ increments from the starting position.

(9) Do a second peak scan, this time searching for values for NH1 and NH2. These are defined to be the number of increments (200^{ths}) from the start of the scan of the count before the first one above 0.16(I1) and the count after the last one below 0.84(I1) respectively, where I1 = P1 − S.B1 is the net peak intensity for the first scan ($S = N_P/N_B$). (This I1 must not be confused with the I1 defined for the /CC instruction in the previous section). The numbers 0.16 and 0.84 are respectively the fractional areas of a Gaussian below −1 and above +1 standard deviations from the mean. Of course to obtain NH1 and NH2 values in this way, the average background count per step is subtracted from each individual peak count before addition to the cumulative peak count.

(10) Evaluate and print the following quantities:

- N_B total number of counts in both backgrounds.
- N_P total number of counts during both peak scans.
- NH1 position of the left peak width indicator.
- NH2 position of the right peak width indicator.
- SB the background count, scaled up to the same count time as I.
- I the net peak count after subtraction of SB from the sum of the two peak scans,

Interpretation of NH1 and NH2

The CODOEP program interprets NH1 and NH2 to determine the width of the peak, and whether any significant part of the peak was outside the nominated scan range. Note that the validity of NH1 and NH2 for this purpose relies on the assumption that both the two background counts and the two peak scans are in agreement. In the case where part of the peak is outside the scan range, CODOEP relies heavily on the assumption of a Gaussian peak shape.

It is often useful to be able to make a quick manual interpretation of NH1 and NH2 at the time the numbers are printed out by the spectrometer; in this regard, the following comments are pertinent. For a peak wholly within the scan range, NH1 and NH2 represent ±1 standard deviations from the peak centre. These are not half-height positions, but for a Gaussian are '0.6 – height' positions, and the full-width at half-maximum height is roughly 20 percent more than (NH2–NH1).

For a peak not wholly within the scan range, the meaning of NH1 and NH2 is more complex, their interpretation is difficult by manual calculation and is best left for CODOEP to cope with properly. To determine if the peak is wholly within the scan range, a rough rule is that both NH1 and NH2 should be at least (NH2–NH1) distant from the ends of the scan. For example, if N_P=300, then NH1=100, NH2=200 is acceptable. NH1=50, NH2=100 is also acceptable, but of course this peak is off centre.

It may be useful to note here the values MUD gives to NH1 and NH2 in the event of unusual values for the counts. If the cumulative intensity of the second peak scan never exceeds 0.16(I1), NH1 has the same value as N_P. If it never exceeds 0.84(I1), NH2 has the value zero. These statements imply that when NH1=0, 0.16(I1) is achieved in the first count of the second peak scan, and when NH2=N_P, 0.84(I1) is exceeded only in the last count of the second peak scan. It might be thought that none of these peculiar circumstances could ever occur, and in fact they are rare for strong peaks; however, for weak peaks, where statistical fluctuations dominate the scene, almost any sequence of counts is possible.

4.3 A Procedure for Orienting a Crystal – Instrument Zeros

There are many ways of establishing the orientation of a crystal on a four-circle diffractometer, but for those not familiar with the geometry or the MUD instruction code, one good method is given here.

First, try to find a strong reflection (which has another normal to it) near $\chi = 90°$. To do this, set up its 2θ and ω values (/CA ..., /DA), set $\chi = 90°$ (/DM 3:90:), and release the ϕ clutch. Spin the ϕ axis manually, watching the ratemeter. If nothing is found, move the χ axis to (say) 88° (/IM 3:–400:) and repeat. Continue this process, decreasing χ by a small amount each time until the peak is found. Engage the ϕ clutch and find the same peak again using /DM 4:... and /SS 4:... or /SC 4:... Centre this peak with /CR 1:

Now try to find the peak normal to this first one by scanning round the zone normal to it. The closer the first peak was to $\chi = 90°$, the closer this scan will be to $\chi = 0$, and hence the fewer the obscurities. Set up the 2θ and ω for the new peak (/CA ..., /DA). Set χ to 90° from its value at the first peak (/DM 3: ...). Scan ω until the peak is found (/SC 2:20: twice background:); this is a scan on the zone normal to the first peak, provided it was found close to the bisecting position; it should be close. Centre the second peak with /CR 1: This second peak will not, in general, be in the bisecting position but CODRUB can cope with this.

Take these two centred reflections, run the CODRUB orientation refinement program and obtain a preliminary UB matrix. Use this to find more reflections for centring, working out to as high 2θ values as accuracy permits. Try to centre some reflections at a range of χ values also. Run CODRUB a second time, including all the orientation data available. This should give a UB matrix which is good enough to start collecting intensity data with. The first few batches of intensity data collected should be run through CODOEP and CODRUB to keep improving on the UB matrix; after a few cycles of this procedure, an orientation matrix should be obtained which enables every peak to be found at its calculated position.

Motor Zero Settings

Associated with each angle drive is a software zero setting which is used to displace all apparent angle readings from their value relative to the hardware zero (which in any case must be

correct within a few degrees). Thus when the spectrometer is aligned it is not necessary to make any mechanical adjustments. No user instruction is provided to change the zero settings as they should rarely need changing and should be contained in an up-to-date tape of the MUD program. The user should consult an AAEC or AINSE staff member before attempting to change them.

The zeros are stored in the computer as octal 200^{ths} of a degree and are usually evaluated from a CODRUB run containing many reflections with a wide range of 2θ and ω values (preferably including negative 2θ values). The values obtained for $2\theta_o$, ω_o, χ_o, should be added to those already in the MUD program at the time of the centring measurements. (ϕ_o has no meaning and would be a redundant parameter in a CODRUB refinement). When the zeros are correct, CODRUB refinement of orientation data should show no zero errors at all. During initial setting up the zero settings may be toggled in to the appropriate locations in the computer memory (first ensuring that the other spectrometer is inactive), and when finally established correctly, should be incorporated in a new copy of the MUD program tape.

4.4 Program CODRUB

The program CODRUB (COmputer Diffractometer Refine UB-Matrix) accepts angle settings from centred reflections and refines them to produce a UB-matrix. It was written by R. J. Dullow and A. W. Pryor in 1967 and the method of calculation is described in some detail in the report AAEC/E191. In 1970 it was extensively modified by A. W. Pryor and G. W. Cox and this version is the one described here.

4.4.1 General Outline

The theory is given in AAEC/E191 and in Busing and Levy (Acta Cryst., 1967, 22: 457). Two reflections known as orienting reflections are chosen to act as a vehicle for the orientation matrix. These are initially the first two reflections provided, and the user should ensure that these are self-consistently indexed, not parallel, and preferably near 90° apart in orientation. If a reasonable orientation matrix cannot be found starting from this pair of orienting reflections, which will be rather rare but can happen in some systems, another pair will be chosen, and the fitting retried. The program will in desperation try all possible pairs of reflections as orienting reflections.

Two reflections are enough to define a UB if the cell parameters are known, and experimenters may find it convenient, having found two reflections, to derive a preliminary UB from them and to use it to find a more extensive set of high angle reflections. With two reflections only, all the K-numbers (Section 4.4.2 below) should be put at zero (that is, one must assume that wavelength, instrument zeros and crystal unit cell are known and refine only for the three orientation parameters).

Additional reflections, which have been centred on one, two or three axes, are used to refine the UB matrix calculated from the orienting reflections. For reflections centred on two or three axes the program calculates the indices from the observed angles and the initial UB matrix, rounds them to the nearest integral values, and uses these integral values in the subsequent refinement. They are listed alongside the ones read from cards, which are listed but otherwise ignored by the program; they serve only to remind the experimenter of his opinions. However for reflections centred on one axis only (always omega), the indices are calculated only if they are not supplied on the card (that is, if they are zero or blank). This is because such data is normally CODOEP output, for which the supplied indices are correct, or at least, consistent.

The observations are weighted in the program as:

2 theta – Assumed standard deviation = 0.2°, that is, wt = 9.10^4

omega – " " " = 0.1°, that is, wt = 36.10^4

chi – " " " = $0.1/\sin\theta$ that is, wt = $36.10^4 \sin^2\theta$,

and $\chi^2 = \Sigma w \Delta^2/(n-p)$ is evaluated after each cycle and listed. w = wt of observation, Δ = discrepancy between an observed and calculated angle, n = number of centred angles and p = number of parameters being refined.

If any observation is more than 3° in error in any setting angle the reflection is omitted from the refinement and labelled 'wrongly indexed'. This avoids the distortion caused by straining to fit mis-punched data, but it should not happen, and must be investigated if it does.

The Busing procedure, using 3 of the 6 angles from the two orienting reflections as parameters to embody the orientation matrix U, means that refinement starts close to the correct value. Cycling proceeds until all parameter shifts are less than half the standard deviation of the parameter, and for not more than three cycles. Usually one cycle is enough.

4.4.2 Input and Output for CODRUB

Input consists of the Data Set and the Refinement Instructions. The Data Set comprises first a title card (format 20A4) and then a number of cards containing:

IG, h, k, ℓ, 2θ, ω, χ, ϕ, in the format (I1, 3I3, 4F10.3) where:

IG = 0 if the reflection has been fully centred (that is, on 3 separate angles, one of which is 2θ).

= 1 if centred as above except for the 2θ centring.

= 2 if only ω is centred.

= 3 if additional data for this run (usually CODOEP output) is to be read from DSR 10 (usually disk). Only one disk data set may be read per run with an IG = 3 card, but it may be read any number of times. This card contains no other data. (The purpose of this option is to allow CODOEP and CODRUB to be run conveniently as one procedure).

= 9 to indicate the end of the reflection data set. This card contains no other data.

Data with IG = 2 are normally supplied by the CODOEP program (as cards or disk records) to improve the UB-matrix as data collection proceeds, but may also be provided directly by the user.

The other quantities on this card have their usual meanings of Miller indices and setting angles in degrees.

The Refinement Instructions comprise three cards containing:

(i) KWAV, WAV in I1, F9.4

(ii) KTTHZ, TTHZ, KOMZ, OMZ, KCIZ, CIZ in 3(I1,F9.4)

(iii) KA, A (1 to 3) and KANG, ANG (1 to 3) in 6(I1,F9.4)

where: WAV = wavelengths in Angstroms

TTHZ = 2-theta zero in degrees

OMZ = omega zero in degrees

CIZ = chi zero in degrees

A(1 to 3) = cell edges in Angstroms

ANG(1 to 3) = cell angles in degrees,

and all the K numbers are 1 if the parameter is to be refined and 0 if not; except that in the case of KA (1 to 3), KA(2) and/or KA(3) are 2 if the corresponding edge is to be refined but constrained to be equal to A(1); similarly for KANG. The three parameters of orientation are always refined; the K numbers define additional parameters. Thus the third card to refine a hexagonal cell might be:

1 5.071 2 5.071 1 7.314 0 90.0 0 90.0 0 120.0

and for a rhombohedral cell:

1 4.83 2 4.83 2 4.83 1 85.2 2 85.2 2 85.2 .

Naturally, one cannot refine both wavelength and cell parameters. Nor can one always obtain satisfactory refinements of the instrument zeros.

The program normally returns to read more Refinement Instructions so that the same data set may be processed in a different way if desired. If KWAV = 2, the program returns to read a new data set, which must be followed by more refinement instructions. If KWAV = 9, the program terminates.

The output consists of a listing of calculated and observed angles for each refinement cycle, and a printout of the final parameter values and the UB-matrix derived from them.

4.5 Program CODOEP

The Output Editing Program, CODOEP processes the paper-tape output of the computer-controlled diffractometers (CCD's) and lists it in an edited form so that unusual or incorrect features of the data are readily noticed by the user.

Apart from this listing, two separate forms of output may be obtained:

(a) Intensity data for all reflections processed; this data is suitable for input to the DRACULA program.

(b) Orientation data for those reflections considered strong enough for the orientation to be well determined. This data is available for feeding back into the CODRUB program to obtain an improved refinement of the UB-matrix. The procedures are described in Section 4.4 on Program CODRUB.

The program was written in 1968 by the late J. R. Thompson and extensively modified in 1970 by G. W. Cox.

4.5.1 General Description of CODOEP

The input to the program is the 8-hole tape from the diffractometer, transferred to magnetic tape by the program PTPART.

Before any reflection input, the program must receive the 'diffractometer status' information. This consists of the following quantities:

NSPEC	the spectrometer number
LAMBDA	the wavelength
UB	the 3 x 3 orientation matrix for the crystal
$(h\,k\,\ell)_{std}$	the Miller indices of the standard reflection
ITIM	1 if counts relative to real time
	0 if counts relative to monitor counter
NMON	number of units of 1024 monitor or clock pulses per CCD count
IGP	group number for punching on DRACULA input cards.

For the first batch of reflections processed by the program, the status may be input from cards, but it is normally provided at the beginning of the paper tape containing the reflections, having been put there by the MUD instruction /DS, which should always be used by the experimenter at the beginning of each run, and each paper tape.

A batch of reflections is terminated by the end of the paper tape or the detection of status information on the paper tape with numeric differences from the current status in the spectrometer number, wavelength or UB-matrix.

The program receives the following quantities as input for each reflection:

h,k,ℓ	Miller indices of the reflection
$2\theta,\omega,\chi,\phi$	the angles used by the CCD as the centre of the scan for the collection of the integrated intensity
N_B	the number of points counted in the background
N_P	the number of steps taken to scan over the peak
NH1	the position of the left 'half height'
NH2	the position of the right 'half height'
SB	the background counts, scaled to the same total count time as I
I	the net peak count after subtraction of SB.

The results of a number of checks and calculations made with this data are printed on one line of output per reflection. Details of these calculations are given below (Section 4.5.2).

The occurrence of a standard reflection is noted on the listing, and all standard reflections are saved and processed together at the end of the batch.

Width and omega errors for strong reflections are saved during the processing and are plotted on the line printer against 2θ at the end of the batch.

4.5.2 Mathematical Details

(a) The Standard Deviation of the Peak Intensity

From the values of I, SB, N_P, N_B, the raw data of actual counts is reconstituted and the standard deviation of the net intensity is calculated.

Total counts in peak = $I + SB$

Total counts in background = $SB/S = B$

and $\sigma^2(I) = I + SB\,(1 + S)$.

If ITIM = 0 (monitor-based counts), correction is made for monitor counting statistics. (See Section 3.1.2).

This is printed under the heading SIGMA PEAK

(b) Classification of Peak as Weak or Strong

This classification is made firstly to establish whether the orientation information derived from NH1 and NH2 is sufficiently well determined for it to be fed back into an orientation refinement, and secondly so that the user can assess the reliability of the orientation information.

A criterion for classification is derived as follows: In the second peak scan by the CCD to determine NH1 and NH2, NH2 is the least well determined, because the intensity collected by the time NH2 is reached is larger than that for NH1. In fact it is well determined only in so far as the standard deviation σ(H2) of the cumulative intensity at NH2 is less than the difference (H1) between that intensity and the total expected cumulative intensity during the second scan. This is because

it is the separation of these latter values which allows NH2 to be different from N_P and hence allows a Gaussian fit of the peak shape to be obtained sensibly.

Two criteria are employed. A peak's orientation is considered worth using for UB refinement if

$$\sigma(H2) < 0.5\ H1.$$

In this case an orientation card is punched; the width and omega-error for that reflection are included in the plots at the end of the batch, and these values on the listing are flagged with a 'P'.

If $\sigma(H2) > H1$, the reflection is flagged as weak ('w') on the listing.

If $\sigma(H2)$ is between these two values, the width and omega-error are not flagged at all.

Note that this classification is completely independent of NH1 and NH2; it depends only on the peak and background. Experience has shown that it is generally possible to fit Gaussian peaks to all of the data not classified as 'w' and to some of the 'w' data, although in the two weak classes, the width and omega-error obtained from the Gaussian fit often appear unreliable.

(c) Calculation of Peak Width and Omega-Error

The values NH1 and NH2 which are output from the CCD are used to evaluate the peak width and to suggest a correction factor which will deviate from 1.0 if the peak has been only partly scanned.

This is done by assuming the peak shape to be Gaussian and fitting it to the data. If the CCD was unable to evaluate one or both of NH1 and NH2, this fact is noted on the listing and none of the calculations of this section are performed.

NH1 and NH2 have the meaning shown on Figure 5 which is a plot of the net cumulative count as the peak is scanned. There are four known points on this curve, namely:

$$(0,0),\ (NH1,\ 0.16\,I),\ (NH2,\ 0.84\,I),\ (N_P,\ I)\ .$$

With a Gaussian peak shape, this curve will have an error function form:

$$y = D\ \mathrm{erf}\left(\frac{x - E}{F}\right) + G\ ,$$

where $$\mathrm{erf}(x) = \frac{2}{\sqrt{\pi}} \int_0^x \exp(-t^2)\ dt$$

and D, E, F, G are free parameters.

The ideal values of D, E, F and G which correspond to a peak which lies wholly but not necessarily symmetrically within the range of the scan are respectively 0.5I, 0.5 (NH1 + NH2), 0.7071 (NH2 – NH1), and 0.5I. After checking for the successful recording of NH1 and NH2 by the CCD, the program starts with the above values for D, E, F and G and attempts to fit them to the four points on the cumulative intensity curve by Newton's approximation. From the values of D, E, F, G so determined, the following quantities are calculated for printout on the listing.

(i) WIDTH of the peak

The full width at half maximum height is calculated from F:

$$\mathrm{WIDTH} = K_A \times \frac{F}{\sqrt{2}} \times 0.005 \text{ degrees.}$$

$F/\sqrt{2}$ steps represents one standard deviation of the Gaussian curve, the constant $K_A = 2.3546$ converts this to the half-height full width, and the 0.005 converts steps to degrees of omega.

(ii) POSITION of the peak

The centre of the scanned peak is given by the value of E. Thus $(E - N_P/2) \times 0.005$ is the difference between the actual centre of the peak and the centre of the scan in degrees, and is listed under the heading 'OMEGA ERROR'.

(d) Correction to Peak Intensity

The full height of the erf curve 2D represents the total intensity of the peak. $2D/I$ expresses this as a correction factor to multiply the observed peak intensity and is listed under the heading 'CORRN'.

It should be noted particularly that the correction factor has not been applied to the intensity. This is because the authors feel that any alteration of the intensity from its measured value should be done manually at the discretion of the user with the correction factor as a guide only. It will be found that the correction factor has little meaning when the peaks are flagged as weak.

The correction factor should be very close to 1. In fact for an accurately centred reflection and N_P equal to the suggested value (see (e) below), its value should be 1.01, as the suggested N_P is calculated on the basis of collecting 99 percent of the intensity in the scan.

(e) Suggested Value of N_P

A value of N_P that would perform a scan to include 99 percent of the peak is calculated assuming that the peak is Gaussian and using the calculated values of the peak width and omega-error, and this is listed under the heading 'NSTEP SUGG'. It is calculated from the formula

$$(K_S \times F/\sqrt{2}) + 2 \mid E - N_P/2 \mid ,$$

where $K_S = 5.15$ converts the standard deviation of a Gaussian into the scan range containing 99 percent of its area. This quantity is useful in enabling the user to determine if his scan range is correct.

Note particularly that a large contribution to 'NSTEP SUGG' comes from the omega-error; thus, if this is large, an improvement in the UB-matrix may obviate an apparent need to increase N_P for subsequent data collection.

It is obviously desirable to operate the spectrometer with N_P as small as possible without excluding too much of the peak, firstly to increase the speed of data collection and secondly to improve the statistics of background subtraction.

(f) General Remarks on Width and Correction Factor

It must be remembered that the shape of the peak is not recorded by the CCD, apart from the position of the four points in Figure 5. The width and correction factor are very sensitive to deviations from the Gaussian shape, and thus the observation of unusual values for them is the best way to detect bad peak shapes. These are commonly caused by high background on one side of the peak (often gives negative intensity) or the existence of two peaks within the scan range (twinned crystal or overlapped peaks).

(g) Checking the Angles Used for Data Collection

The purpose of this check is to compare the angles at which the peak has been scanned with those calculated from the Miller indices and the UB-matrix. If standard procedures have been used, this comparison amounts merely to a check on the CCD calculation.

To allow for the possibility that a non-zero azimuth angle may have been used, and because this situation is not evident in any parameters other than the four angle values, the check is carried out in the following way. (Zero azimuth is defined for this purpose to be at the bisecting position).

A matrix $\underline{R}_1$ which describes the orientation of the crystal relative to the zero position of the spectrometer angles ϕ, χ and ν ($= \omega - \theta$) is calculated from the CCD angles (see AAEC/E191, Equation 5). A similar matrix $\underline{R}_2$ is calculated for the bisecting position from the Miller indices and the UB matrix. The matrix Ψ which rotates $\underline{R}_2$ into $\underline{R}_1$ is then found by solving the matrix equation

$$\underline{R}_1 = \underline{\Psi}\,\underline{R}_2 \quad .$$

If the CCD angles represent a correct setting for the given reflection, $\underline{\Psi}$ should be in the form of an azimuth rotation of ψ about the scattering vector, that is,

$$\underline{\Psi} = \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos\psi & -\sin\psi \\ 0 & \sin\psi & \cos\psi \end{pmatrix} \quad .$$

At this stage it is necessary to allow that the CCD, because of its 'obscure' routine, may have measured $\bar{h}$, $\bar{k}$, $\bar{\ell}$ rather than h, k, ℓ,. In this case Ψ will represent an azimuth rotation combined with a π-rotation about a vertical axis and the matrix element Ψ (1,1) will be -1. If Ψ (1,1) is negative ψ is recalculated for $\bar{h}$, $\bar{k}$, $\bar{\ell}$. The reversal is commented on in the listing and both the listing and the card output contain the correct indices. (Note that the CCD teletype print-out gives the unreversed hkℓ).

The form of Ψ is then checked to see that it is indeed an azimuth rotation about an axis close to the scattering vector by checking that $\Psi(1,1) > 0.99$. If this is so, the angle ψ is extracted from the four elements containing it and the 'correct' setting angles are calculated using this value. If Ψ does not represent an azimuth rotation, setting angles are calculated for the bisecting position. These calculated angles are compared with the angles read in from the CCD tape and if any differ by more than 0.1 degrees they are listed under the heading ERRORS (PDP8--IBM360).

(h) Plots of Peak Widths and Omega Errors

At the end of each batch of reflections processed under the same diffractometer status, plots are presented on the line-printer of the peak widths and omega-errors versus 2θ for all those reflections in the batch where these parameters are considered reliable (flagged P). These plots assist the user to note quickly the existence of bad data and to observe if the correct dependence on 2θ is obeyed. (Each plot contains a program-inserted point at the origin to force the scaling to include the origin).

For example, if the omega errors show no dependence on 2θ, and a small random scatter about zero, it can be assumed that all spectrometer angle zeros and the UB-matrix are correct. The peak widths should show a dependence on 2θ which is a function of the monochromator angle of the spectrometer.

4.5.3 Input for CODOEP

(a) Paper tape input

The CCD produces paper tape which is first read by the program PTPART. This produces a direct copy of the paper tape on magnetic tape, which then forms the CODOEP input. The CCD output tape is of the correct format for CODOEP input. It is detailed in Section 4.6 on PTRESCUE.

(b) Card input

Card input to CODOEP for the diffractometer status information is not required but is allowed. and applies to all reflections before the first diffractometer status record encountered on the magnetic tape input (that is, PTPART output). This facility will be useful in a situation where the diffractometer status information is accidentally omitted or physically damaged on the paper tape. The set of cards need not be complete; the default values indicated apply for any missing cards.

Card 1 Title – Any 80 characters. This appears at the top of each page.
Default value 'NO TITLE PROVIDED'.

Card 2 Format F10.5 LAMBDA, the wavelength.
Default value 1.0.

Cards 3–5 Format:3F10.5 UB-matrix, one row per card.
Default value – identity matrix.

Card 6 Format 3I5 (h, k, ℓ) for standard reflection.
Default values (0,0,0).

Card 7 Format 3I5 ITIM = 1 for counts v. time
= 0 for counts v. monitor
Default value 0.

NMON = number of time or monitor units per count.
Default value 1.

IGP = Group number for punching on output cards.
Default value 1.

4.5.4 Output from CODOEP

Two separate output streams are provided, one for intensity data and one for orientation data. If these are cards, they appear as two separate packs. They may be distinguished by the Z or 2 punching respectively in Column 1. The orientation pack usually contains fewer cards than the intensity pack.

(a) Intensity output – DSR 2

The cards have the format required by DRACULA, that is, ('Z', I4, 2I5, 2I8, 2F10.5, I5, (0X or 5X), I4); and they contain the following information:

$h\,k\,\ell$, $K_M I$, $K_M \sigma(I)$, $\sin 2\theta$, the azimuthal angle in degrees from the bisecting position, the group number and a standard or sequence number. The 'Z' indicates to DRACULA that there is one reflection per set and that the standard deviation is given on the card. $K_M I$ and $K_M \sigma(I)$ are I and $\sigma(I)$ scaled to one unit of time or monitor count. The standard and sequence numbers refer only to the current batch of CODOEP processing and are not used by any subsequent programs. Further details are given in Section 5.2.2.

(b) Orientation data output

For those reflections where the orientation is considered reliable enough to be included in the CODRUB refinement, the position of the peak centre as determined from NH1 and NH2 is output onto DSR's 10 and 11, usually cards and disk. The format of the records is ('2', 3I3, 4F10.3, 20X, I10); and they contain $h\,k\,\ell$, $2\theta, \omega, \chi, \phi$ and the sequence number in the current CODOEP batch – for the user's convenience only. The '2' indicates to CODRUB that this data is centred only on omega. Further details are given in Section 4.4.2.

4.6 Program PTRESCUE

If a malfunction or error causes the paper tape output from the CCD to be lost or destroyed, it is possible to punch the data by hand on cards in fairly free format from the CCD teletype listing and to run PTRESCUE to copy these cards onto a magnetic tape suitable for CODOEP input.

The cards punched must simulate closely the paper tape input. The character set used is the ASCII representation on 8-hole tape of the 26 letters, the 10 numbers and the special characters " # $ % & ' ( ) * + , – . / : ; < = > ? @ · Carriage return and line-feed characters are also used, and are represented by CR and LF in the following text. In addition, three more special characters are defined:

SM	(Octal 201)	Start message character
SMDS	(Octal 202)	Start message character for diffractometer status
EM	(Octal 203)	End of message character.

For numeric input, a number field on the tape consists of a number formed from a string of digits, optionally preceded by a + or – and optionally containing a decimal point in any position. The number field may contain any characters before the number and any number of blanks after the number, and the field must be terminated by a colon.

Two types of numeric records are recognised, each containing a series of number fields:

(i) A diffractometer status record, commencing with the SMDS character, and ending with EM.

(ii) A reflection record, commencing with SM and ending with EM.

The numeric fields in these records are those listed in Sections 4.1 and 4.2.

Information other than that between start and end message characters may appear on the tape and is treated as comments introduced onto the tape by the /PH instruction. Every time a CR is encountered in such information, the preceding comment is printed on one line of the listing. The subsequent character is assumed to be a LF and is ignored. (N.B. If SM or SMDS immediately follow CR it is ignored; the following message is treated as a comment and is not processed, a point to watch when punching paper tape manually, but see below).

A CR with no preceding characters since the last CRLF or EM does not produce a blank line.

The first line of comment after a new diffractometer status message is taken as a title for the batch of reflections and is printed at the top of each page of listing. If a title is not encountered before the first reflection on a page, that page is given the title 'NO TITLE PROVIDED'. To avoid this situation the CCD user should always use the instruction sequence /DS, /PH at the beginning of a new batch of reflections or a new paper tape.

For punching cards, a number of card characters with special meanings are defined:

| (vertical stroke)	SMDS
$	SM
#	EM
:	terminates number field
;	CR
(blank)	LF .

Card boundaries have no meaning for this program; the input is considered to consist of a continuous string of characters, 80 from each card. This input is translated directly to paper tape codes and is written on a magnetic tape for CODOEP input. Blanks have no meaning and are ignored except when embedded in a number (where they are illegal) or in a comment field. It is recommended that reflections be punched one per card for convenience.

Information should be in the order required for CODOEP, that is, a diffractometer status record should appear first, followed by a comment to be used as a title, followed by interspersed comments and reflection data.

The diffractometer status record should start with | , contain 17 number fields and end with #. The reflection data should start with $, contain 13 number fields and end with # :

The comment line (maximum number of characters = 80) should be terminated with ; . The comment extends from the last CRLF or EM to the CR. It is recommended that the # on the reflection cards be punched in Col. 80.

5. SUBSEQUENT PROCESSING OF BRAGG INTENSITIES

5.1 Program VARISORT

VARISORT is a program to re-arrange the order of the reflections. It may be used:

(i) to order the data cards which have been produced by CODOEP (CCD operation) or by either FAFPAC or FAFMIP (manual operation). The data may have been taken in a strange order, or it may be desired to group symmetry-related equivalents together, or to order in terms of sin θ, or whatever,

(ii) to order the data cards produced by DRACULA. It may be desired to set flags on them so that, on the next run, DRACULA will average sets; or to re-order them in some other way for use in other programs,

(iii) to generate symmetry-related equivalents of the reflections that are supplied. This is sometimes needed for Fourier-plotting programs, which may also need the data to be presented in some other order,

(iv) to order data supplied in strange Formats from whatever source.

(v) to order data preparatory to publication.

The program was written by M.M. Elcombe.

5.1.1 General Description of VARISORT

The program accepts cards (usually from DRACULA, CODOEP, FAFPAC or FAFMIP, but the formats are read in, so it is quite general) and produces a new deck ordered in some specified way. Each input card contains hkℓ-indices, on which the ordering is done, plus other data, which is not touched in VARISORT, but simply preserved.

If requested, symmetry-related reflections are generated, which are some specified function of the hkℓ indices of reflection J. These numbers are then sorted, in ascending or descending order, by an optimized routine SORT supplied by Applied Mathematics and Computing Section. The input cards and any generated data are then re-punched in this determined order.

The ordering number JORD may be calculated in a variety of ways in internal subroutines. These are controlled by two supplied numbers HKLORD and ISORT as described in the Input section below. If none of these ways is the desired order the user must supply a routine USORT incorporating his own formulae to calculate JORD from hkℓ.

If the VARISORT output is to be fed into DRACULA (using the DRACULA option INCODE = 2) it may be required in flagged 'sets' (A 'set' of data being several reflections leading to the same value of $|F|$; symmetry-related equivalents for example). With VARISORT option IFLAG = 0, all data with the same value of JORD are put together and the last member of the 'set' has a Z in column 1. With IFLAG = 1 the user may either put blanks in column 1 or keep the original flags, depending on the Format specification for the input and output.

If the VARISORT output is to be used in the AAEC Fourier program ND8A there are several complications. This question will be discussed in a report dealing with the Fourier program. A user-supplied subroutine INDGEN may be needed. This will be described elsewhere.

5.1.2 Input for VARISORT

Card 1 Specification of Input and Output Formats, Format (20A4)

Cols 1–40 give input format. Cols 41–80 give output format; if cols 41–44 are blank the program sets output format = input format. These formats must include the opening and closing bracket, for example, (A1, 3I3, 10A4). Both formats must start with (A1, followed by 3 integer fields, which contain the indices h, k, ℓ on which the sorting is done. A further 10 fields must be supplied, either as 10A4 or in a more specific form if the format is being changed (or if use is to be made of the contents in subroutine INDGEN). If the original flags in col 1 are to be kept they can be read and punched using a T1,A1 format for one of the last 10 fields.

Card 2 Specifying the Sorting Procedure, Format 5I5

Col 5 – HKLORD (default option – 6) in accordance with the table:

HKLORD	I1 (slowest changing)	I2	I3 (fastest changing)
1	ℓ	k	h
2	h	ℓ	k
3	k	h	ℓ
4	k	ℓ	h
5	ℓ	h	k
6	h	k	ℓ

Col 10 – IUP (default value – 0) = 0, for ascending order of JORD
= 1, for descending order of JORD.

Col 15 – IFLAG (default value – 0) = 0, Z flags set for DRACULA
= 1, flags blank or originals kept if so specified on Card 1.

Col 20 – ISORT, specifying the function to be used in calculating JORD from the I1, I2, I3 defined by HKLORD (default value – 1).

ISORT = 0, supply new subroutine USORT (see below).

ISORT = 1, JORD = $10^4 * I1 + 10^2 * I2 + I3$

= 2, JORD = $10^4 * |I1| + 10^2 * |I2| + |I3|$

= 3, JORD = $10^4 * I1 + 10^2 * |I2| + |I3|$

= 4, JORD = $10^4 * |I1| + 10^2 * I2 + I3$

= 5, JORD = 10^4* (max value of $|h|, |k|, |\ell|$) + 10^2* (middle value of $|h|, |k|, |\ell|$) + (min value of $|h|, |k|, |\ell|$)

Col 25 – MULT (default value = 0) = 0, if subroutine INDGEN (for generating equivalents for Fourier program) not needed.

= 1 if INDGEN is needed (in this case an additional card giving the specification for INDGEN is required).

Cards 3 etc. Data Cards (Format specified on Card 1)

The total number (including any that have been generated by INDGEN) must not exceed 3000.

The terminator has an 'L' in col 1 if the output is for DRACULA and a '1' in col 1 if the output is for ND8A, ORFLS, etc.

Complete data sets may be repeated.

The User-supplied Subroutine USORT

This is needed if ISORT = 0 and its purpose is to set up JORD as a function of hkℓ. The form should be:

```
SUBROUTINE USORT (JH, HKLORD, JORD)
DIMENSION JH(3)
INTEGER HKLORD
```

Proceed to set up JORD as the required function of hkℓ (contained in JH) using HKLORD if required

```
RETURN
END
```

How this is incorporated with the existing program is explained in Section 6.

5.1.3 Output from VARISORT

The information from the first three input cards is listed. The input data to be sorted is not listed. The sorted data is listed in the input format, and written on DSR 2 in the output format. This is normally the cardpunch, but can be a disk space for immediate input into another program. The last input card is generated as the last card of the output on DSR 2. The number of reflections sorted is included in the output listing.

Several sets of data may be processed in the one run. They all come out in a single file (FT02F001) separated by the terminators ('L' or '1') which were read in.

5.2 Program DRACULA

The input to DRACULA is the sets of hkℓ, Intensity and its standard deviation which have come from FAFPAC (or FAFMIP) or from CODOEP. (The program will also process data with background rather than standard deviation, supplied in the superseded CDRABS format). The data may have been sorted and flagged in VARISORT.

The output from DRACULA is the data which finally goes into refinement programs like ORFLS or into Fourier programs. It is expected that non-AAEC users will normally be satisfied with the DRACULA output, representing the final word on the I and σ(I) of the Bragg reflections they have measured. However they may care to sort it again in VARISORT.

DRACULA does the following:

(i) Corrects the data for absorption.

(ii) Calculates parameters which may be used in refinements that incorporate extinction.

(iii) Adjusts the standard deviation it is supplied with (which has been calculated for counting statistics alone) for other factors, such as uncertainty in the absorption correction, or personal intuition.

(iv) Averages 'sets' if required, where a 'set' is a number of observations which should give the same $|F|$.

The program incorporates earlier ones by B.M. Craven and G.W. Cox. It was re-written in 1970 by M.M. Elcombe.

5.2.1 General Description of DRACULA

(a) Sets and Groups

The incoming data may be classified into a few large 'groups' taken under the same experimental conditions, such as same crystal, same wavelength, same instrument, though the monitor setting may vary as it would have been adjusted in FAFPAC, or CODOEP. The essence of a group is that it may be related to any other group by a single scaling factor. The separate scaling factors must be supplied with the data; they would normally have been evaluated from the intensities of the standard reflections.

The data is also divided into many 'sets' where the members of a 'set' should all reduce to the same value of $|F|$. They may, for example, be the same reflection, or symmetry-related equivalents of the same reflection, repeated with different crystals or different wavelengths, but members of the same 'set' will frequently turn up in different 'groups'. Usually the 'sets' will have been put together by VARISORT, if they weren't already together, and frequently there are 'sets' containing one reflection only. The program may or may not average the 'sets', depending on how flags are set. Normally a user would want them to be averaged, with appropriate adjustment of the standard deviation of the average, but he may wish to inspect and compare them before averaging, to make sure the discrepancies are not too large. If the data are being fed into programs which refine for extinction, then it is necessary to preserve the symmetry-related equivalents, with their different mean path-lengths, without averaging them. If 'sets' are to be averaged, the necessary flagging would normally be done in VARISORT.

(b) Standard Deviations

Each observed intensity I is presented with its standard deviation $\sigma(I)$, calculated from the counting statistics, in FAFPAC (or FAFMIP) or in CODOEP. The program will also still accept the old CDRABS format where background was supplied and $\sigma(I)$ calculated as explained below.

The absorption factor $A \pm \sigma(A)$, is evaluated (as explained below) and a corrected intensity $Q \pm \sigma(Q)$ is calculated where:

$$Q = AI$$

$$\sigma^2(Q) = \sigma_1^2 + \sigma_2^2 \quad ,$$

where $\sigma_1 = \sigma(I).A$ in the usual case where $\sigma(I)$ was supplied, or

$= (I + 2B)^{\frac{1}{2}} A$ in the obsolete case when background B was supplied for CDRABS. And

$$\sigma_2^2 = (\alpha_i + \beta_i I + \gamma_i I^2)^2 A^2 + I^2 \sigma^2(A) \quad ,$$

where α_i β_i γ_i are constants, defined for each 'group', which are intended to express the user's personal intuition regarding errors in the measurements from non-statistical causes (for example $\beta = 0.05$ is quite a popular value).

An unobservably weak intensity may be flagged by manual punching on the input cards, in which case,

$$Q = I_{mi} A$$

and $$\sigma^2(Q) = S_{mi}^2 A^2 + I_{mi}^2 \sigma^2(A) \ ,$$

where I_{mi} and S_{mi} are constants defined for the i^{th} group, and are related to estimates of the minimum observable intensity. It is usual, in evaluation programs like ORFLS, to treat such observations just like the others, but to retain the flag. The present authors see no merit in this procedure and do not recommend it.

(c) Averaging 'Sets' and Applying Lorentz Polarization Factors

If only one reflection in the set has been measured in group i its mean square structure amplitude $F_i^2 \pm \sigma(F_i^2)$ is given by

$$F_i^2 = K_i L Q$$

and $$\sigma(F_i^2) = L \left[K_i^2 \sigma^2(Q) + Q^2 \sigma^2(K_i)\right]^{\frac{1}{2}} \ ,$$

where $K_i \pm \sigma(K_i)$ is the scale factor for group i

L is (Lorentz polarization factor)$^{-1}$ for the observation

$= \sin 2\theta$ for neutron data

$= 2 \sin 2\theta/(1 + \cos^2 2\theta)$ for X-ray data

and $Q \pm \sigma(Q)$ are defined in the previous paragraph.

If more than one reflection in the set has been measured in group i, $Q \pm \sigma(Q)$ above is replaced by the weighted average $Q_i \pm \sigma(Q_i)$ given by

$$Q_i = \Sigma w Q / \Sigma w \quad \text{with} \quad w = \{\sigma(Q)\}^{-2}$$

and $$\sigma(Q_i) = \{\Sigma w\}^{-\frac{1}{2}} \ ,$$

where the sum is over those reflections in the set, which were measured in group i.

The final mean square structure amplitude for the set $F^2 \pm \sigma(F^2)$ is obtained by averaging the $F_i^2 \pm \sigma(F_i^2)$ over those groups i containing measurements in this set. That is

$$F^2 = \Sigma w_i F_i^2 / \Sigma w_i \quad \text{where} \quad w_i = \{\sigma(F_i^2)\}^{-2}$$

and $$\sigma(F^2) = \{\Sigma w_i\}^{-\frac{1}{2}} \ .$$

If the structure amplitude $F \pm \sigma(F)$ is required the formulae employed are:

$$F = (F^2)^{\frac{1}{2}}$$

$$\sigma(F) = \{F^2 + \sigma(F^2)\}^{\frac{1}{2}} - F \ ,$$

which $\approx \sigma(F^2)/2F$ when $F^2 >> \sigma(F^2)$.

This formula for $\sigma(F)$ has the advantage that it is fully defined down to $F^2 = 0$, where $\sigma(F) = \{\sigma(F^2)\}^{\frac{1}{2}}$. When $F^2 = 3\sigma(F^2)$ the formula is only 6 percent different from the more usual, $\sigma(F) = \sigma(F^2)/2F$.

If $F^2 < 0$ but not less than $-3\sigma(F^2)$ then the output is $F^2 = F = 0$ with $\sigma(F) = \{\sigma(F^2)\}^{\frac{1}{2}}$.

If $F^2 < -3\sigma(F^2)$ then it is assumed that something is seriously wrong in the experiment and no output is produced on DSR 2 or DSR 12. A 'NO OBSERVATION' message is produced on the listing.

It is assumed here that the various scale factors for the groups, $K_i \pm \sigma(K_i)$, can be derived from an initial inspection of the data. After going through DRACULA once, it may be necessary to revise these parameters and repeat the run. To assist in re-evaluating the K_i the following correlation factors are printed out:

$$WS(M,N) = \Sigma\ w_{mn} F_m^2$$

$$SW(M,N) = \Sigma\ w_{mn} \quad ,$$

where

$$w_{mn} = 1/\sigma(F_m^2)\sigma(F_n^2) \quad ,$$

and the sums are only over those reflections within a 'set' which occur in both the m^{th} and n^{th} 'groups'.

If $WS(M,N) \neq WS(N,M)$ the scale factor of the n^{th} group should be multiplied by $\frac{WS(M,N)}{WS(N,M)}$. If more than two groups are involved, similar scaling ratios can be determined.

(d) Absorption Corrections and Extinction Parameters

The absorption correction factor ($A \pm \sigma(A)$) is the reciprocal of the transmission factor ($T \pm \sigma(T)$)

where $$T = \int_{crystal} e^{-\mu r}\, dv \Big/ \int_{crystal} dv \quad ,$$

and $$\sigma(T) = \frac{\partial T}{\partial \mu}\sigma(\mu) = -\left[\int_{crystal} r e^{-\mu r}\, dv \Big/ \int_{crystal} dv\right]\sigma(\mu) \quad .$$

Then $A = 1/T$ and $\sigma(A) = -\sigma(T)/T^2$.

The crystal is divided into 125 volume elements, according to 3-dimensional, 5 point Gaussian integration intervals (for which the values were obtained from standard tables). For each volume element, the sum (r) of the path lengths from its centre to the surface of the crystal in the directions of the incident and scattered radiation is determined. The values of $e^{-\mu r}$ and $re^{-\mu r}$ are calculated and the integrals are evaluated as sums over the volume elements. In order to evaluate the correct path lengths the crystal must have no re-entrant faces or edges. The Zachariasen procedure for extinction correction requires an absorption-corrected mean path length, $\bar{T} = -\frac{1}{T}\partial T/\partial \mu$. These quantities then accompany the reflection and are used in refinement programs incorporating extinction parameters as free variables. $\bar{T}$ is included in the output listing, and, if requested, a card deck containing this and other relevant information for each observation is produced. For anisotropic extinction using the program LINUS, two specific vectors are also required and these can also be obtained from DRACULA.

The faces of the crystal must be specified. This is normally done in terms of the Miller indices of the actual crystal face, plus the distance of that face from some origin within the crystal. Re-entrant angles are not allowed. For the CCD's, where a UB-matrix is, or can be supplied, the user may care to describe his crystal in the PHI-axis system (with all diffractometer axes at zero,

Z is vertical, X points along the incoming beam, Y completes a right-handed set). This would be advantageous for a crystal cut into some simple shape, ignoring crystal axis, and symmetrically mounted on its pin.

For each crystal shape, a data set is produced which is input for the program CRYSPLOT. This program produces a third-angle projection drawing of the crystal on the CALCOMP plotter, so that the user can readily detect any errors in his crystal shape data.

5.2.2 Input for DRACULA

Card 1 Title (Format 20A4).

Card 2 Control card (Format 15I5) containing:

NGP: No of groups of observations $1 \leqslant NGP \leqslant 8$

NOBS: Max. number of observations in a SET ($NOBS \leqslant 200$ for CODOEP format and $NOBS \leqslant 8$ for CDRABS format)

LP:
- = 1 Neutron Data
- = 2 X-Ray Data

INPUT:
- = 1 if the reflection data is on cards in DSR 1
- = 2 if the reflection data is from DSR 11, having been put there by VARISORT for DRACULA or by an earlier DRACULA run with INPUT = 3
- = 3 if the reflection data is on cards in DSR 1 and is to be stored on DSR 11 for later use

NCARD:
- = 1 No 'ORFLS' output
- = 2 'ORFLS' Output of F or $|F|^2$ on cards (DSR 2)
- = 3 'ORFLS' Output of F or $|F|^2$ on DSR 12

NFORM:
- = Blank if NCARD = 1
- = 1 if 'ORFLS' output required in AAEC 'ORFLS' format
- = 2 if 'ORFLS' output required in original 'ORFLS' format

NUB:
- = 1 manual machine
- = 2 if 'UB' matrix supplied; computer controlled machines

NFI:
- = blank if NUB = 1
- = 1 if the indices of the faces are in the crystal system, and
- = 2 if in the PHI system

NTAPE:
- = 1 extinction output on cards, DSR 2
- = 2 extinction output on DSR 10

INCODE:
- = 1 for the CDRABS format (now rarely used)
- = 2 for the CODOEP or FAFPAC format

NFSQ:
- = 1 for output of structure amplitude, F
- = 2 for output of $|F|^2$

NLINUS:
- = 1 for no extinction output
- = 2 for 'LINUS' output
- = 3 partial extinction output ($\overline{T}$ only)

NHAND: is required only if NLINUS = 2, in which case,
= 1 for left-handed spectrometer (positive 2 theta to left of the main beam as seen by the neutron), and
= 2 for right-handed spectrometer.

Cards 3 etc. Group Parameter Cards (Format I5, F10.5, 2I5, F10.2, 2F10.5, 2E10.3).
A card is required for each of the NGP groups containing:

ISC_i: The output label of group i. (DRACULA must have consecutive numbers from 1 to NGP in the input. These may be changed to the ISC_i values. When several groups occur in one averaged set the first group encountered is assumed when setting the output group).

λ_i : Wavelength of radiation

I_{mi}: Minimum observable intensity

S_{mi}: The standard deviation of I_{mi}. Note that I_{mi} and S_{mi} are required only if some of the input has been manually flagged as 'unobservable', a practice we do not recommend.

$\alpha_i, \beta_i, \gamma_i$: The ingredients to adjust the e.s.d.'s in accordance with the intuition of the user (see Section 5.2.1. (b))

K_i: The scale factor of the group, which will normally have been obtained from a comparison of standard reflections.

$\sigma(K_i)$: the e.s.d. of K_i

Card 4 RECIPROCAL CELL CARD (Format 6 F 10.5)
a*, b*, c*, cos α*, cos β*, cos γ*
This card may be blank for CODOEP data with NLINUS = 1 .

Cards 5 ABSORPTION PARAMETER CARDS
There is one set of cards for each of the NGP groups of observations.
Either 5a or 5b.

Cards 5a IF NUB = 1
First card (Format (6 F5.0, I5, 2F10.5, I5)) containing:

HH1: = reciprocal lattice vector which is mounted along the PHI axis (3 components).

HH2: = reciprocal lattice vector which is vertically downwards at AZ = 0 at CHI = 90° (3 components) (that is, it is the $hk\ell$ reference of GASP).

NFACE: = number of plane faces on the crystal. If NFACE is set = 0 an absorption factor of 1 is assumed (maximum value 20).

VMU: = linear absorption factor.

SIGVMU: = estimated standard deviation of VMU.

IGRIDP: = 1 to obtain listing of grid points used in the summation (otherwise 0 or blank).

Subsequent NFACE cards (if NFACE = 0, these cards are not required)
(Format (3F5.0, F10.5) containing:

HKL: = indices of crystal face (not necessarily integers).

DIST: = distance of crystal face from an arbitrary origin inside the crystal.

Cards 5b IF NUB = 2

First card (Format I5, 2F10.5, I5) containing: NFACE, VMU, SIGVMU, IGRIDP as already defined.

Next three cards contain the UB-matrix (by rows) in the format (4X, F8.5, 3X, F8.5, 3X, F8.5).

Subsequent NFACE cards as described above, recalling that if NFI = 1 the HKL indices of the faces are given in the usual crystal system, while if NFI = 2 they are to be given in the PHI–axis system.

Cards 6 REFLECTION DATA (either cards, if INPUT = 1 or 3, or card images on tape or disc, if INPUT = 2. See card 2 description above).

Supply 6a) if INCODE = 1 (CDRABS Format)

or 6b) if INCODE = 2 (CODOEP, FAFPAC and FAFMIP Format).

Cards 6a Format (A1, 3I4, 4(I7, I5, A1, I1)); azimuthal angle format (A1, 8F8.2). Data in this form is now no longer usual, being originally designed for the old CDRABS program of which DRACULA is the new version. CDRABS accepted four reflections per card when the four were members of the same 'set' $hk\ell$ or $\bar{h}\bar{k}\bar{\ell}$. Various azimuthal angles could be accepted only for reflections at CHI = 90°. The cards contained:

FLAG: = 'L' for end of data; blank normally except for NOBS > 4 the flags are 'F' and 'S' on the first and second cards (for the second card data would start in Col. 14); and if azimuthal angles were supplied the flags are 'A' and 'Z' for NOBS < 4 and 'F', 'A', 'Z' for NOBS > 4.

$hk\ell$: indices for the reflections of the 'set'.

Then for each set: I, B, T, G,

where I = intensity; B = background or e.s.d., T is a flag = ' ' (blank), 'S', 'T' if, respectively, B is a background, e.s.d., or the reflection is unobserved; and = '–', 'R', 'U' if ditto, and the reflection is $\bar{h}\bar{k}\bar{\ell}$. G is the group number.

Cards 6b Format (A1, I4, 2I5, 2I8, A2, 8X, F10.5, I5). This is the usual form of data, with one reflection per card, from FAFPAC (or FAFMIP), CODOEP or VARISORT. The cards contain:

FLAG: = 'Z' for the last of a number of consecutive cards to be treated as a 'set', otherwise blank. Thus if any 'sets' present are not to be averaged, every card must have its 'Z'. Programs FAFPAC (or FAFMIP) or CODOEP normally put a 'Z' on every card and to average 'sets' the Z's must either be manually removed or else the data must be put through VARISORT with appropriate options.

$hk\ell$ = indices of the reflection.

I, B = intensity and its standard deviation.

T = data flag as for CDRABS format, if blank, 'S', signifying that B is e.s.d. is assumed. 'T' flags could be manually inserted if the reflection is 'unobserved' but this is not recommended.

AZ = the azimuthal angle measured from the bisecting position. This would normally have been evaluated in CODOEP for the CCD diffractometers. For the manual diffractometers it would be non-zero only for CHI = 90° and would be manually punched.

G = input group number,

5.2.3 Output from DRACULA

Output may be on cards or tape according to the JCL specification. Two decks may be produced on each run, the 'ORFLS' deck and either NZACH or 'LINUS'. The 'LINUS' output includes all the NZACH output, but the NZACH option is faster.

When 'sets' have been averaged, the hkℓ on the card is for the first reflection of the 'set' and the group number on the card is ISC_j, see description of input Card 3, where j is the first group in which data for the particular 'set' is found.

When using the NZACH or NLINUS options it is of course wrong to average 'sets' of symmetry-related equivalents and the output would be useless if it were done.

The output formats are:

(i) Original ORFLS format (NFORM = 2) – (3I9, 1X, F8.3, F9.3, I9) containing hkℓ, F or F^2, $\sigma(F)$ or $\sigma(F^2)$, ISC.

(ii) AAEC ORFLS format (NFORM = 1) – (I5, 2I4, A1, 2F9.3, I3) containing ditto, except that the 'unobserved' flag is in the A1 field.

(iii) Isotropic extinction format (NLINUS = 3) (3I4, 2F9.4, I3, 36X, F6.3) containing hkℓ, F or F^2, $\sigma(F)$, or $\sigma(F^2)$, ISC and $\overline{T}$.

(iv) 'LINUS' format (NLINUS = 2) (3I4, 2F9.4, I3, 7F6.3) containing the above output (iii) plus the two LINUS vectors in the 36X field.

The line printer output contains one page summarising the input data, a minimum of one page per group giving absorption details, and one page containing a glossary of the symbols used. This is followed by the processed reflection data, for which there is one line of output for each input reflection plus one line for each set. A plot of $\sigma(F)$ against F is produced at the end of each run to assist the user in determining the various mathematical constants.

6. JOB CONTROL LANGUAGE (JCL) REQUIREMENTS

6.1 Multiprogramming

JCL cards are the means of directing the computer to associate specific data sets, whether on tape, disk, or cards, with a given program. The JCL cards necessary for running the programs in this manual are given in this section.

At the time of writing, the AAEC's IBM/360 operates under a batch processing system. It is envisaged that in the near future, a multiprogramming system will be implemented and changes in the JCL will be needed which cannot be defined at this stage. For this reason, all of the JCL suggested here for use is in the form of procedures. (A procedure is a set of JCL cards stored on disk which can be called and modified with a user's EXEC card). Any necessary changes to the procedures will be made by the Applied Mathematics and Computing Section at the time of changeover and it is expected that the user's cards will not need to be altered.

6.2 Fortran and JCL

A full resumé of how the JCL works is not given here, but, because most of the programs in this manual are written in Fortran, a brief description of how data sets are accessed from Fortran programs is given. In Fortran, data set reference numbers (referred to as DSR in this manual) are used in Read and Write statements in the program. For example in:

```
      WRITE (10,99) TITLE
   99 FORMAT (20A4)
      READ (11) ARRAY
```

10 and 11 are the data set reference numbers for the WRITE and READ statements; 10 refers to formatted data, and 11 refers to unformatted (binary) data.

For accessing the data referred to, the computer makes two digits of the DSR by adding a zero on the left if necessary and incorporates it in a name of the form FTnnF001 which must appear in the name field of a data definition DD card. This card must appear after the EXEC statement for the job step which executes the program. The text of this DD card must contain the specification of the data set. If the EXEC card is part of a procedure, the DD card is associated with the correct EXEC card by placing the step name plus a fullstop before the DD name. The step name is defined as the name field (first field) on the EXEC card in the procedure. The examples below should clarify this explanation.

6.3 JCL for the Programs in the Manual

In what follows, all text in upper case must be punched exactly as shown; characters in lower case represent variable information which should be replaced by the user's requirements. Take particular care with blanks; in general, text on EXEC and DD cards may contain no blanks. The verbs EXEC and DD must be preceded and followed by one or more blanks and these are the only permissible blanks before the end of the text field. If it is necessary to continue text on more than one card, terminate each card to be continued at any comma in the text and before column 72. Start the next card with // and resume the text, starting anywhere between columns 4 and 16.

The following examples show the use of JCL to run all of the programs in this manual individually or in useful combinations.

For single program runs, use of the FORTHG procedure is recommended; data sets other than card input and output and printer output should be specified on separate DD cards, one for each.

6.3.1 Programs for Handling 'Series 150' Tapes

FAFEDT and PTPGM

FAFEDT will normally be run by the AAEC's computing assistant for Solid State Physics Section using the procedure NDFAFRUT (see Figure 1), which runs the programs PTPGM, FAFEDT, FAFMEL, and FAFHEL in sequence. The single data card for PTPGM is read from disk; data for the other three programs must be supplied. The following deck is used.

```
//A       EXEC  NDFAFRUT,TAPNUM1=xxx ,TAPNUM2=yyy
//FAFEDT.SYSIN DD  *
       data for FAFEDT
/*
//FAFMEL.SYSIN DD  *
       data for FAFMEL
/*
//FAFHEL.SYSIN DD  *
       data for FAFHEL
/*
```

where xxx specifies the 3-digit serial number of the magnetic tape containing the PTPART output (PTPGM input), and yyy is the 3-digit serial number of the tape which is to be written with the FAFEDT output. To obtain these tape numbers, see AAEC.

FAFMEL To run FAFMEL use the deck

```
//A       EXEC  FORTHG,DSN='MATLIB.NDLIB',REF='MATLIB.NDLIB',
//              PRG=FAFMEL
//GO.FT11F001   DD  DCB=CYCFAF,DISP=OLD,UNIT=(280,,DEFER),
//              VOL=SER=TPE06n   n=0 to 4; see AAEC.
//GO.FT12F001   DD  DUMMY
//GO.FT21F001   DD  DUMMY
//GO.SYSIN      DD  *
       editing data if any
/*
```

FAFPAC To run FAFPAC with card output, and a separate deck for the fitted data, use the deck

```
//A       EXEC  FORTHG,DSN='MATLIB.NDLIB',REF='MATLIB.NDLIB',
//              PRG=FAFPAC
//GO.FT02F001   DD  DCB=(RECFM=FA,BLKSIZE=81)
//GO.FT10F001   DD  SYSOUT=B,DCB=*.FT02F001
//GO.FT11F001   DD  DSN=CYCFAF,DISP=OLD,UNIT=(280,,DEFER),
//              VOL=SER=TPE06n   n=0 to 4; see AAEC.
//GO.SYSIN      DD  *
       data cards
/*
```

To delete the fitted data card deck, replace the GO.FT10F001 card with

```
//GO.FT10F001   DD  DUMMY,DCB=*.FT02F001
```

FAFSAS To run FAFSAS, use the deck

```
//A       EXEC  FORTHG,DSN='MATLIB.NDLIB',REF='MATLIB.NDLIB',
//              PRG=FAFSAS
//GO.FT11F001   DD  DSN=CYCFAF,DISP=OLD,UNIT=(280,,DEFER),
//              VOL=SER=TPE06n   n=0 to 4; see AAEC.
//GO.SYSIN      DD  *
       data cards
/*
```

For each of the above three examples, the FT11F001 DD card specifies the FAFEDT or FAFMEL output tape.

FAFMIP To run FAFMIP use a simple FORTHG deck

```
//A       EXEC  FORTHG,DSN='MATLIB.NDLIB',REF='MATLIB.NDLIB',
//              PRG=FAFMIP
//GO.SYSIN      DD  *
       data cards
/*
```

6.3.2 Programs Involving CODOEP (see Figure 2)

CODOEP will normally be run by the AAEC's computing assistant in Solid State Physics Section and users should present him with their MUD paper tapes and/or CODRUB data for all CODOEP runs. CODOEP is optionally preceded by the programs PTPART, PTRESCUE or nothing. It is optionally followed by the CODRUB program or nothing. The procedure NDCODOEP handles all of these possibilities; the various options are specified by the parameters TAPE=, PRG1=, and PRG2= on the EXEC card.

TAPE=nnn specifies the 3-digit serial number of the magnetic tape being used for PTPART output/CODOEP input. PRG1=... specifies the program (if any) which is to precede the CODOEP run. It may be PTPART or PTRESCUE or nothing. If PTRESCUE is coded, the card data for PTRESCUE must follow, enclosed between the JCL cards //PTRESCUE.SYSIN DD * and /* . If PTPART is coded, the paper tape must be provided. If PRG1=... is not coded, CODOEP reads the last data put on the specified tape by PTPART or PTRESCUE.

PRG2=CODRUB specifies that a CODRUB run is to follow the CODOEP run. In this case, CODRUB data should be supplied, enclosed between JCL cards //GO.SYSIN DD * and /* .

The reflection data of this CODRUB run would normally contain a reflection card with IG=3 to request use of the CODOEP orientation data, which is thereby read from the disk. Examples of use of this procedure follow:

1. To run CODOEP with paper tape input, and no CODRUB refinement, and to store the paper tape data on tape 509, that is, to go from A to C and D on Figure 2, use the single card:

```
//A       EXEC  NDCODOEP,PRG1=PTPART,TAPE=509
```

2. To run CODOEP with paper tape input, and follow with CODRUB refinement of the orientation matrix. The CODOEP run is to read some diffractometer status data from cards. This run goes from A to C and E on Figure 2.

```
//A       EXEC  NDCODOEP,PRG1=PTPART,PRG2=CODRUB,TAPE=509
//CODOEP.SYSIN DD  *
       data cards for CODOEP here
/*
//GO.SYSIN     DD  *
       data cards for CODRUB here
/*
```

3. To run CODOEP, starting with MUD output data hand punched on cards, and proceeding through to CODRUB refinement, that is, to go from B to C and E on Figure 2.

```
//A       EXEC  NDCODOEP,PRG1=PTRESCUE,PRG2=CODRUB,TAPE=509
//PTRESCUE.SYSIN  DD  *
       data for PTRESCUE here
/*
//GO.SYSIN     DD  *
       data for CODRUB here
/*
```

CODRUB To run CODRUB on its own, use the FORTHG procedure:

```
//A       EXEC  FORTHG,DSN='MATLIB.NDLIB',REF='MATLIB.NDLIB',
//              PRG=CODRUB
//GO.SYSIN     DD  *
       data for CODRUB here
/*
```

6.3.3 Programs Involving DRACULA (see Figure 3)

Two procedures exist: NDRACULA runs DRACULA on its own, and NDVADRAC runs VARISORT followed by DRACULA.

To run DRACULA on its own, that is, to go from B to C and D on Figure 3, use the deck:

```
//A        EXEC  NDRACULA
//GO.FT09F001  DD       data set for output for CRYSPLOT; needed only if this output
                        is to be other than on disk for an immediately following
                        CRYSPLOT step.
//GO.FT10F001  DD       data set for extinction output; needed only if this output is not
                        on cards (that is, NTAPE=2)
//GO.FT11F001  DD       reflection data set; needed only if INPUT=2 or 3 is specified
//GO.FT12F001  DD       data set (tape or disk) for output for ORFLS; needed only if
                        NCARD=3
//GO.SYSIN     DD  *
        data cards for DRACULA run
/*
```

If any of the FTnnF001 cards are supplied, they should be in the order shown above.

To run VARISORT followed by DRACULA; that is, to go from A to C and D on Figure 3, use the deck:

```
//A        EXEC  NDVADRAC
//VARISORT.SYSIN  DD  *
        data cards for VARISORT run
/*
insert DD cards for DSR's 9, 10 and 12, if required, as defined above
//GO.SYSIN     DD  *
        data cards for DRACULA run
/*
```

To incorporate the user supplied subroutine USORT into VARISORT for the above run, use

```
//S1       EXEC  FORTHL
//LKED.REF      DD  DISP=SHR,DSN=MATLIB.NDLIB
//LKED.SYSLMOD  DD  DSN=&&AAEC(VARISORT)
//LKED.SYSIN    DD  *
            object deck of USORT
            INCLUDE REF(VARISORT)
            ENTRY MAIN
/*
//S2       EXEC  NDVADRAC
//VARISORT.STEPLIB  DD  DISP=OLD,DSN=&&AAEC
//VARISORT.SYSIN    DD  *
          data cards for VARISORT
/*
insert DD cards for DSR's 9, 10 and 12, if required, as defined above
//GO.SYSIN     DD  *
          data cards for DRACULA
/*
```

CRYSPLOT To run the program CRYSPLOT to obtain a plotted picture of the crystal shape specified in the DRACULA run, put the card

```
//B        EXEC  NDCRPLOT
```

after the /* which follows the DRACULA data in either of the above procedures. The program CRYSPLOT will pick up its data from the disk output of DRACULA provided that it is run in the same job. If CRYSPLOT is required without any preceding DRACULA data processing, it is recommended that DRACULA be first run with control data only, in order to set up the disk data for CRYSPLOT.

APPENDIX 1

PROGRAM PTPGM

PTPGM was written by Applied Mathematics and Computing Section and a general description is available from the AAEC. It reads the magnetic tape copy of the paper tape (output of PTPART) from DSR PTIN and translates the 5-hole punch code. Any illegal punch codes are replaced by 'X'. It checks the input record lengths (between 'carriage return' characters) with those specified on the input data card (see below). An 'X' is added to short records and long records are divided into 2 or more records as necessary. An output is written on DSR SYSOUT (usually onto disc for immediate recovery by FAFEDT) which consists of each record preceded by two identifying digits. The first digit (1, 2, 3 or 4) classifies the present record (as correct data, correct heading, incorrect data or incorrect heading respectively) and the second digit (0, 1 or 2) refers to the next record, whether it is alphanumeric, purely numeric or the end of file respectively. These digits are used in FAFEDT.

Input Data Card for PTPGM (Applicable to the 'Series 150' 5-hole tapes)

Starting in column 1 with no embedded blanks the following:

DRL = 21, HRL = 10, SHR = 1, SDR = 1

The order of the four parameters is immaterial but they must be separated either by single blanks or single commas.

Output Listing: A single page containing the JCL and a copy of the input card. If any situation has arisen which causes the job to terminate the reason is given but there is no other indication that the job was terminated.

APPENDIX 2

INPUT AND OUTPUT FOR FAFEDT

Input

The main input of FAFEDT is on DSR 11, which contains the output from PTPGM. Two data cards are required for each input file from DSR 11.

Card 1 – format (20A4) – TITLE any alphanumeric characters, usually contains input and output tape numbers and date.
If TITLE(1) = ENDR the run is terminated, see below.

Card 2 – format (I6, 5I8, I4) INITIAL VALUES – contains

MONCNT = initial monitor count for checking purposes (default value 10000).

LEXC1, LEXC2, LEXC3 = specific headings for which the data is not required (default values 12345678).

LSTART = specific heading with which data processing is to start (default option= beginning of DSR 11).

LEND = specific heading at which data processing is to stop (default option = end of file of DSR 11).

NMIN = minimum number of records between headings which are copied onto DSR 12. NMIN must be less than 21 (default option 21).

On reaching the end of file of DSR 11, remaining data is output on DSR 12 and control transferred to read a new title card. Usually this contains 'ENDR' in the first 4 columns and the run is terminated by writing a dummy heading onto DSR 12 (required by FAFMEL) and stopping.

If the title card does not indicate run termination the program expects another 'INITIAL VALUES' card and further data on file 2 of DSR 11. The output continues on file 1 of DSR 12.

Output

(a) Magnetic Tape: This is all on file 1 of DSR 12, however many input files of DSR 11 are used, and it always ends with the dummy heading 999999xx, where xx is the number of headings that have been reconstructed during the run. Each output record contains ISER, MONITOR, COUNT and FIELD in format I4, 3I6. Whenever ISER = 0, MONITOR and COUNT contain a heading and FIELD = 0.

(b) Listing: The heading, monitor count, number of records in the scan and the maximum count are given. Error messages are printed as they are found.

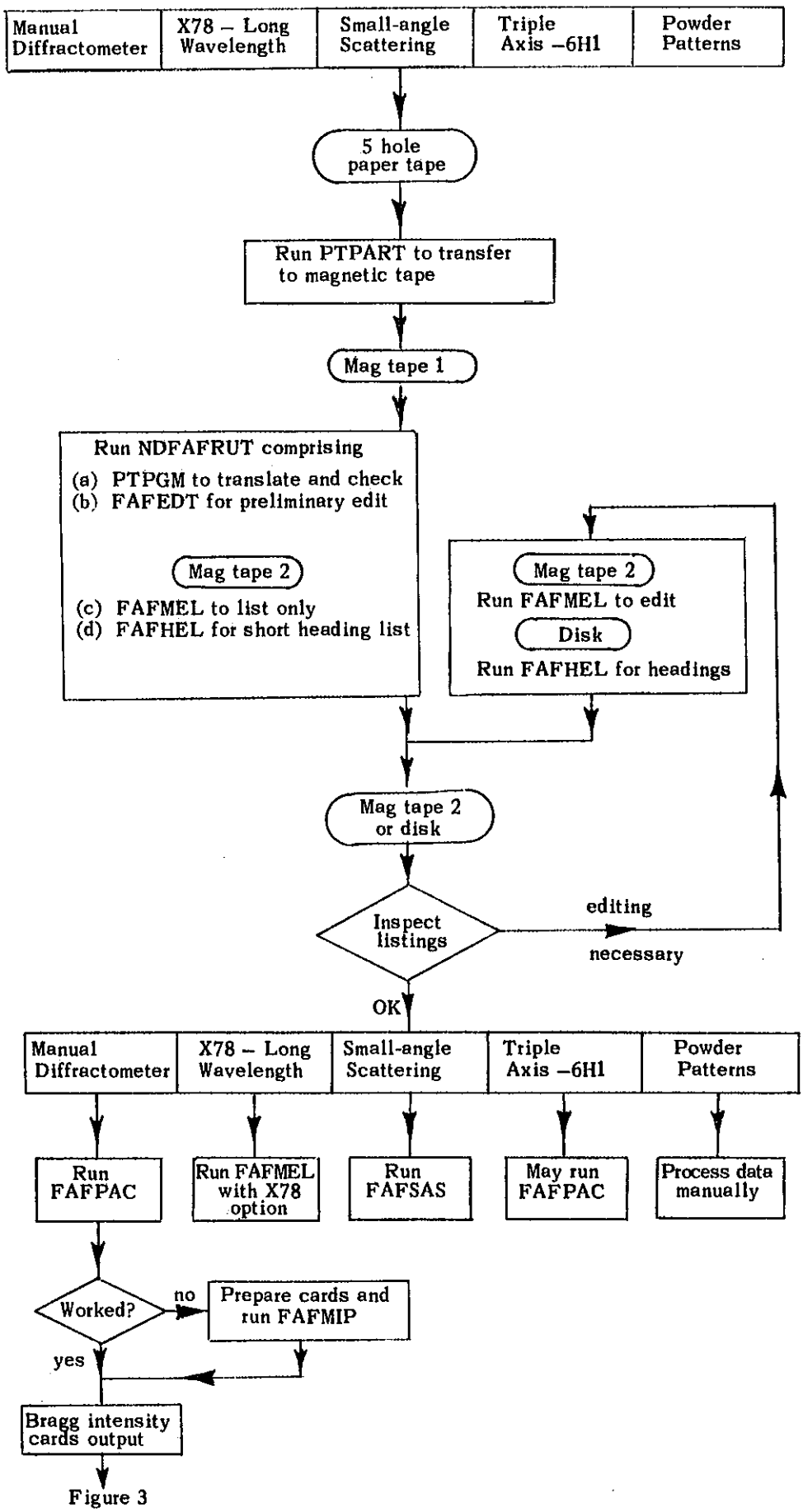

Figure 3

FIGURE 1 'SERIES 150' PROCESSING FLOW CHART

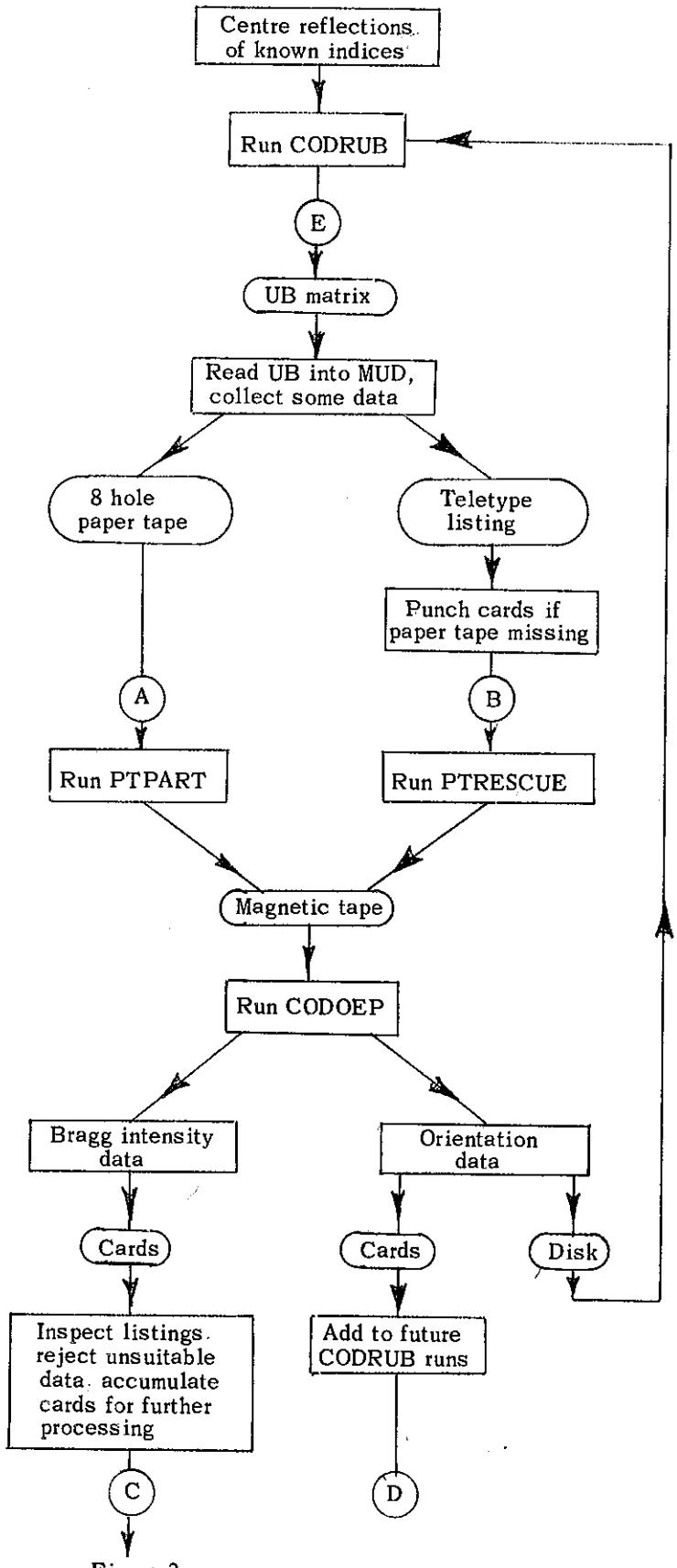

FIGURE 2 CCD's PROCESSING FLOW CHART

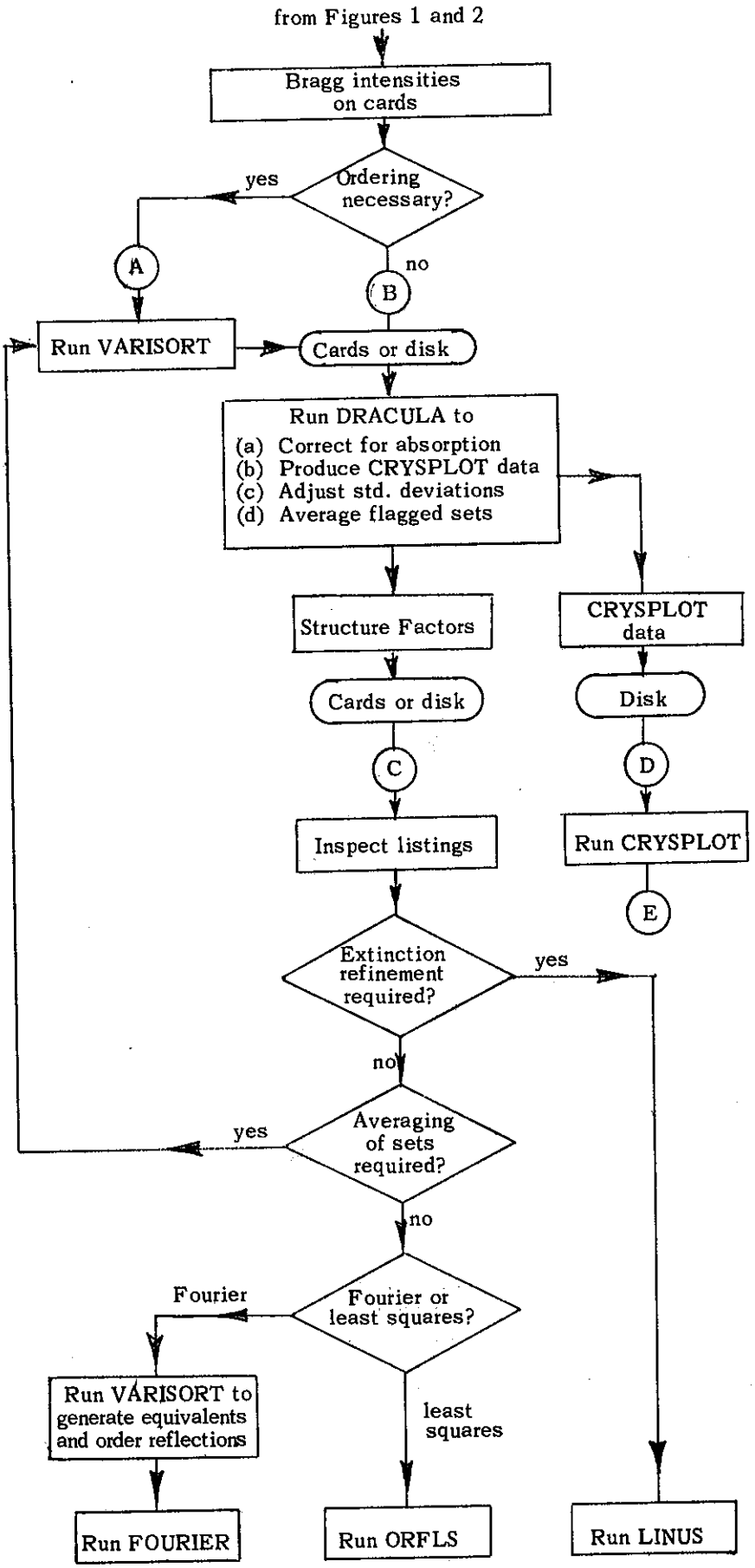

FIGURE 3 BRAGG INTENSITIES PROCESSING FLOW CHART

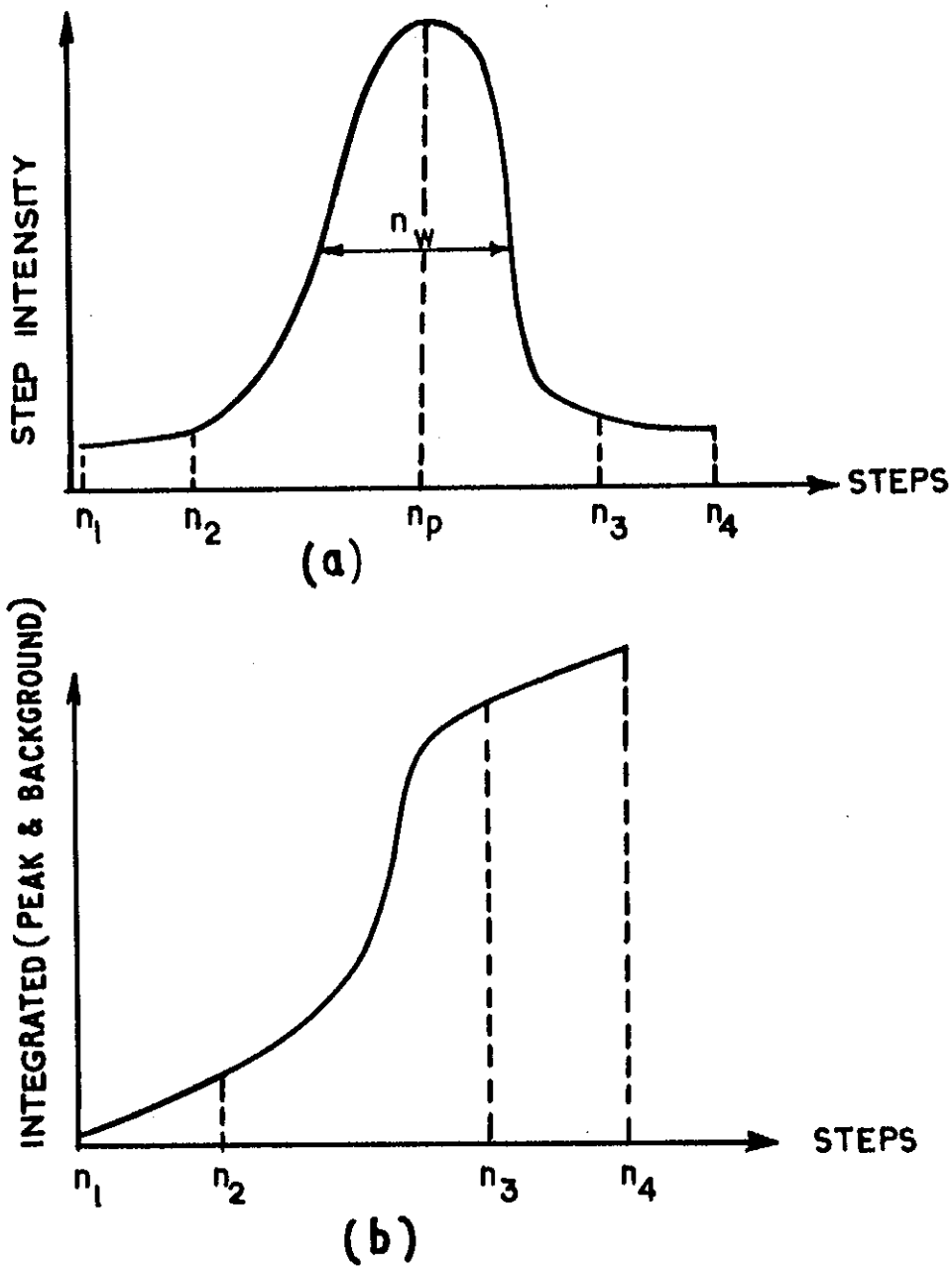

FIGURE 4. TYPICAL APPEARANCE OF THE STEP COUNTS AND THE INTEGRATED COUNTS

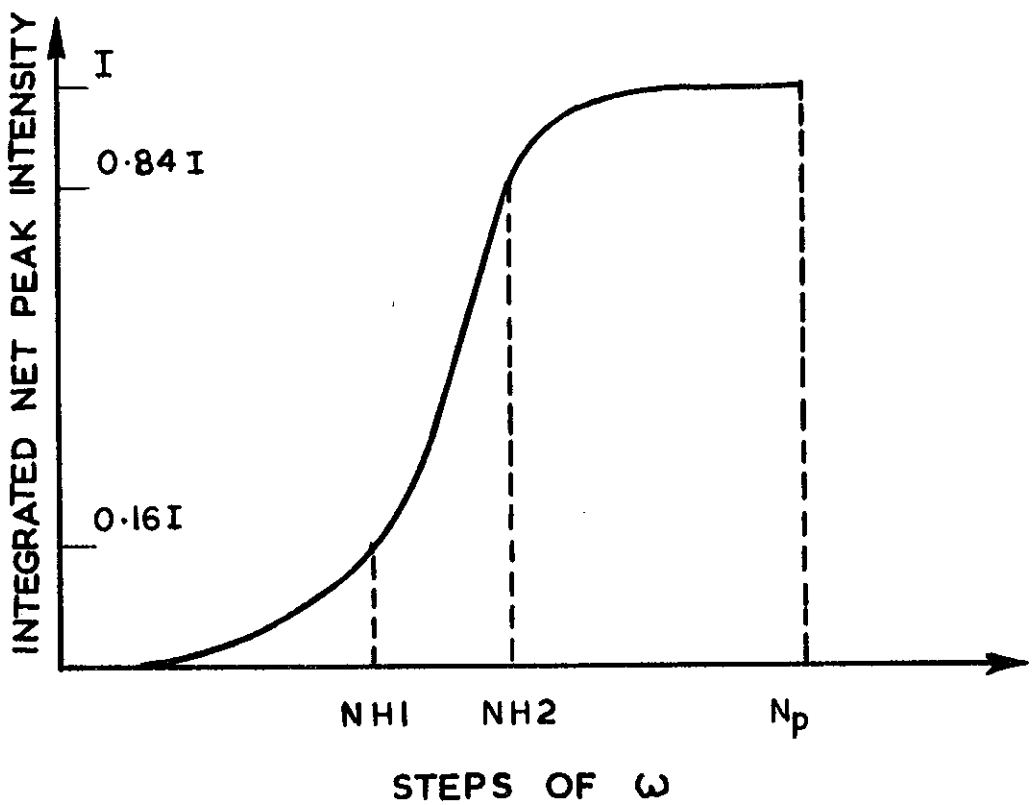

FIGURE 5. TYPICAL SCAN OF INTEGRATED NET PEAK INTENSITY

TABLE 2

SUMMARY OF MUD INSTRUCTION CODE

Class	Name	Code	Data	Data Description
Motor Driving	Zero Angles	/ZA	4	Present axis positions ($\pm 10°$)
	Drive Motor	/DM	2	NANG and angle
	Increment Motor	/IM	2	NANG and XANG
	Drive to Angles	/DA	0	
Data Input	Set Monitor	/SM	2	0 for mon or 1 for real time and number times 1024 pulses
	Read Wavelength	/RW	1	Wavelength
	UB – Matrix	/UB	9	The UB – matrix (by rows)
	Insert Standard	/IS	5	Frequency; hkℓ; –(first insertion)
	Number of Steps	/NS	1	200^{ths} deg. of ω in DCOLL
Counting, Scanning Searching	Take Count	/TC		
	Step Scan	/SS	2	NANG and XANG
	Search for Count	/SC	3	NANG; XANG; count
	Calculate Angles	/CA	3	hkℓ
	Centre Reflection	/CR	1	NANG for axis of final centring (0 if no final centring)
	Measure Reflection	/MR	0	
	Azimuth Scan	/AS	1	Number of $\frac{1}{10}$ th degrees in azimuth step
Printing	Typewriter ON	/TN		
	Typewriter OFF	/TF		
	Print Angles	/PA		
Interrupting	OFF	/OF		
	Cancel Program	/CP		
	Stop it!	/??		
Tape Control	Punch Leader	/PL	0	
	Diffractometer Status	/DS	1	Group number for DRACULA
	Punch Heading	/PH	any	Alphameric heading
Data-Collection	Individual Reflections	/IR	3S	Successive sets of hkℓ
	Individual Azimuths	/IA	4S	Successive sets of hkℓ ψ, with ψ in $\frac{1}{10}$ th degrees
	Collect Data	/CD	17	$\underline{\Delta hk\ell}_1$, $\underline{\Delta hk\ell}_2$, $\underline{\Delta hk\ell}_3$, $\underline{hk\ell}_0$, $-N_1$, $-N_2$, $-N_3$, $2\theta_{min.}$, $2\theta_{max.}$
	Continue Collecting	/CC	6	$\underline{hk\ell}_0$, $-I_1$, $-I_2$, $-I_3$
	Resume Data-Collection	/RD	0	
	Read Program for CD	/RP	1	1 for DCOLL, 2 for TKCNT, 3 for HKLOUT