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SXD Diffuse Scattering Data Analysis Manual

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Appendix to the User Guide for the SXD Instrument

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1. Introduction

The SXD diffractometer has been descibed in detail in the SXD Instrument Manual [1]. One of the key roles of the instrument is the rapid measurement of diffuse scattering over large portions of reciprocal space. The interpretation of diffuse scattering is dependent on the ability to perform rigourous corrections on the raw data. Data from SXD are collected in terms of number of neutron counts for each given (x,z,t) where x and z are the coordinates referring to a given pixel on a given detector and t is the neutron time-of-flight. The data analysis task is therefore two-fold: first to correct the raw data for instumental effects and secondly to convert the (x,z,t) corrected data into a form which is suitable for further interpretation, usually into (h,k,l) reciprocal lattice planes.

This manual describes the various routines currently available for the correction of diffuse scattering type data measured on SXD. The first set of routines incorporate a quick correction of data which adds spectra in time-of-flight, subtracts background and divides by a vanadium run. The second set of routines are more rigorous and are intended for those who require accurately normalised structure factor data or data which are not in the equatorial plane of the instrument. These routines are based around the NORM routines used on LAD and SANDALS and the CORRECT package which was developed at Oxford and are designed to compute a normalised structure factor in any reciprocal lattice plane impinging on the detector. These routines create very large files, are very cpu intensive and are currently only installed on the SXD alpha computer as a developmental package.

Throughout this manual we will use demonstration data from C_{60} measured by W. I. F. David *et al* in December 1995. C_{60} in its high temperature, orientationally disordered phase (above T=260K) has a face-centred cubic structure with $a\approx$14.1Å. It is known to show significant diffuse scattering with a prominent broad ring of scattering at $|Q|\approx 6Å^{-1}$. The data used here are from two crystal orientations (ω=22° and 67°) at T=266K with the detectors set at 2θ=53.7°, $L2$=180mm and 2θ=124.5°, $L2$=159mm respectively. Figure 1 shows four 'Laue' plots from these data for the two orientations and two detectors. These are produced by integrating over neutron time-of-flight (or λ) for each pixel on the detector and dividing by an integration over an equivalent vanadium run to remove the effect of the relative pixel efficiency. These plots show the scale of the data analysis problem. Even with only two 'frames' there is an enormous volume of data to process.

2. Quick Correction Routines

These routines will only correct the data from an approximation to the equatorial plane of the detectors. It is implicitly assumed that the data of interest are equatorial or nearly equatorial and that data out of this plane are less important. This assumption makes the data correction much more straightforward since the equatorial plane is perpendicular to the axis of rotation and the reciprocal lattice plane contained in the equatorial plane of the

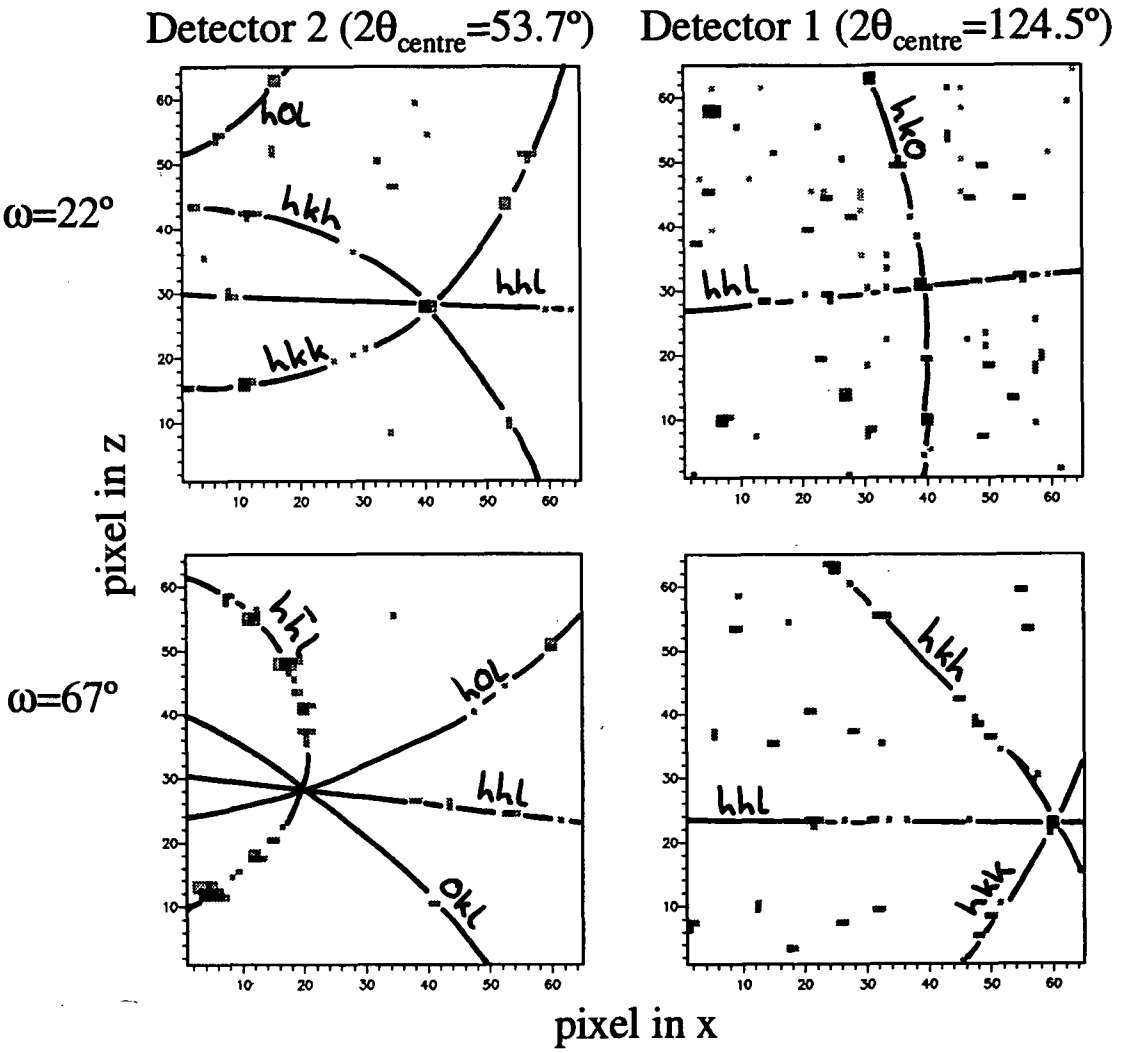

Figure 1. Laue plots from two 'frames' of data from C_{60} (ω=22° and 67°), showing the positions of Bragg reflections on the face of each detector. Various principle reciprocal lattice planes are shown.

instrument will be a so-called zeroth layer line. A sample mounted with cubic symmetry and a [00l] vertical has the *hk*0 reciprocal lattice plane equatorial, a [1 $\bar{1}$ 0] vertical has the *hhl* plane equatorial etc. This has two additional advantages in so far as the t-o-f direction for a given pixel in the equatorial plane of the instrument is equivalent to the |*Q*| direction in the reciprocal lattice plane and hence the relationship between, for example, (*x*,*z*=0,*t*) and (*hkl*) is straightforward. Also, any slight misalignment of the sample still results in an approximately linear path of the reciprocal lattice plane across the detector (the C_{60} sample was not aligned with [1 $\bar{1}$ 0] perfectly vertical, but the *hhl* plane, although not in the centre of the detectors, describes a straight line - see Figure 1).

Hence these routines correct a linear strip of detectors which may not be horizontal across the detector. They are run inside a command file, an example of which is listed below:

```
$! *** An example command file for SXD data analysis ***
$! *** producing a reciprocal lattice grid file for ***
$! *** plotting using dp:plot3d_100100 ***
$! *** last altered (DAK) 08/10/96
$!
$ ASSIGN/NOLOG SXD$DUA0:[SXDMGR.DAVE.PROGS] DP
$!
$! Define the required working subdirectory
$ SET DEF SXD$DISK1:[SXD.sample]
$!
$! Initialise GENIE and run the correction routines
$! parameter#1 - first run number
$! parameter#2 - last run number
$! parameter#3 - vanadium run number
$! parameter#4 - background run number (0 for none)
$! parameter#5 - 1:subtract background or 2:don't
$! parameter#6 - centre pixel at low 2theta detector edge
$! parameter#7 - centre pixel at high 2theta detector edge
$! parameter#8 - which detector - (1=s1-s4096, 2=s4101-s8196)
$! parameter#9 - vertical width of strip
$!
$ GENIE
> @DP:SUMV_1_5 8426 8426 8430 8419 1 -12 -14 1 7
> @DP:SUMV_1_5 8427 8427 8430 8419 1 -13 -15 1 7
> EXIT
$!
$! Run the routine to convert tof-angle to qx,qy,qz
$! row#1 - L1, L2 (m) and 2theta (middle of detector):'
$! row#2 - pixel size (mm) and no of pixels (16 or 64):'
$! row#3 - min and max column usable:'
$! row#4 - 'lattice' parameters a,b,c
$! row#5 - reciprocal lattice plane measured, i.e.
$! (1) x:[100] v y:[010]
$! (2) x:[010] v y:[100]
$! (3) x:[100] v y:[001]
$! (4) x:[001] v y:[100]
$! (5) x:[110] v y:[001]
$! (6) x:[001] v y:[110]
$! row#6 - grid spacings along axes low w, high w (in r.l.u)
$! row#7 - grid to start from qx,qy (in r.l.u.)
$! row#8 - run number
$! row#9 - measured omega angle
$! row#10 - zero omega angle (defines lowest w angle axis)
$! row#11 - y: cont fr. row#8
$!          n: then y: cont. from row#1 with diff. detector angles
$!          n: then n: give output file name on next line
$ RUN DP:QCALC_1_4
8.0 .1973 125.89
3 64
1 64
6.1 6.1 6.1
5
0.1 0.1
-3.0 -3.0
8426
-20.0
-104.0
y
8427
1.0
-104.0
n
n
TEMP.DAT
```

This example command file (DP:SV_1_5.COM) may be copied into the working area and used as a template. SUMV_1_5 adds together a vertical strip of detector elements of width governed by parameter #9 centred on a straight line defined to run across the detector from parameter #6 at the low 2θ edge to parameter #7 at the high 2θ edge. Parameter #6 and #7 are defined with respect to the centre (equatorial) row of the detector (pixel 32 in Figure 1). It then does the same for a background run (if required) and a vanadium run. It then subtracts the background from each strip and divides by a smoothed vanadium. Each run is scaled by dividing by the integrated monitor count. The resulting file (SXD0nnnn.COR) contains corrected time-of-flight data for each scattering angle (2θ) of the detector in the equatorial scattering plane.

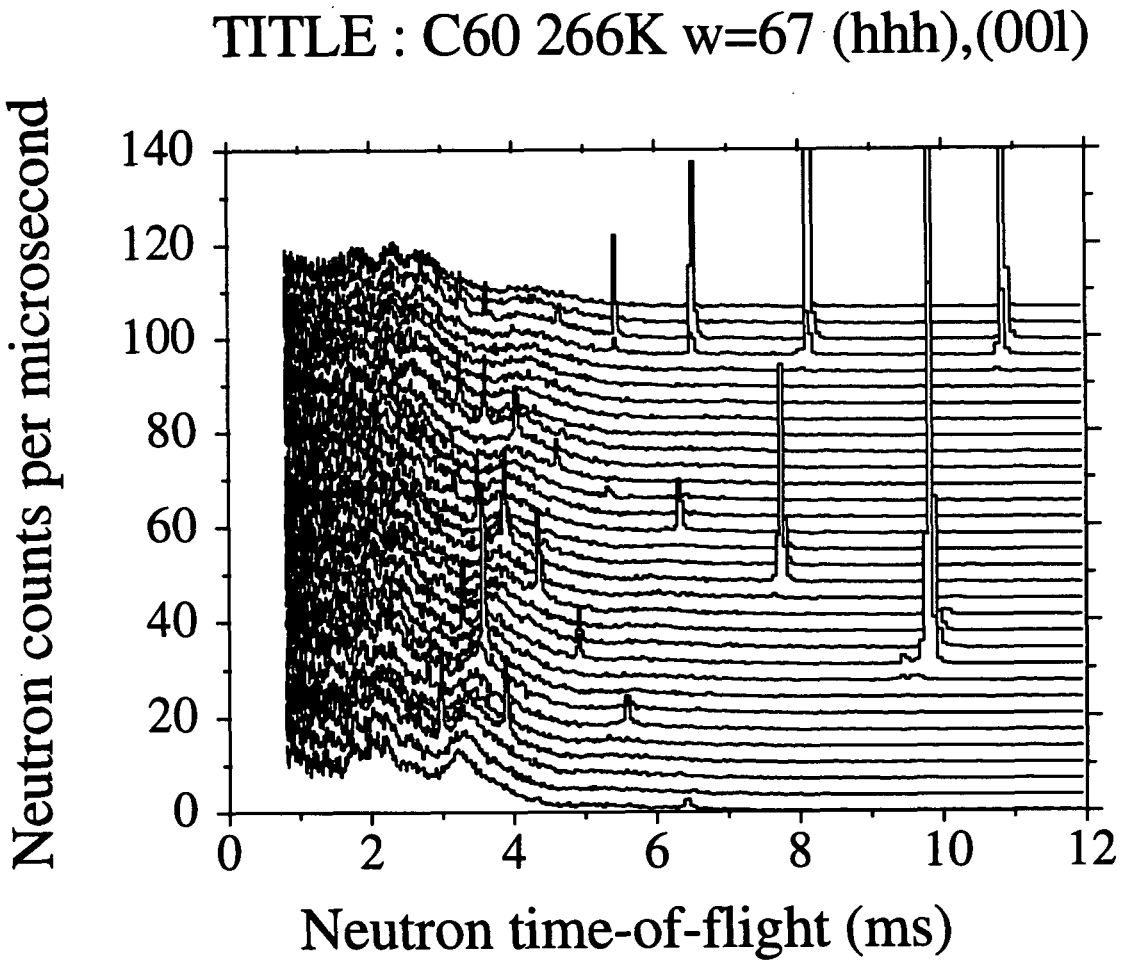

Figure 2. Raw summed data from C_{60}, ω=67°, high-angle detector 1 showing spectra from the equatorial *hhl* plane.

Figure 2 shows the raw data from a summed strip of pixels between z=-10 and -3 from detector 1 with ω=67°. The spectra at the bottom of the plot correspond to the low-2θ side of the detector and 2θ increases with successive spectra. As can be seen from Figure

1, this includes the *hhl* reciprocal lattice plane and the *hhh* reflections which appear as a line of reflections within two or three spectra at the top of Figure 2. No correction has been made for the different pixel efficiencies or the incident neutron flux or instrumental background. Figure 3 shows the same data after correction for these effects using the routines described above. The data are more even and the characterstic flux shape has been removed. The data are more noisy at the high t-o-f since this is where the incident neutron flux is low.

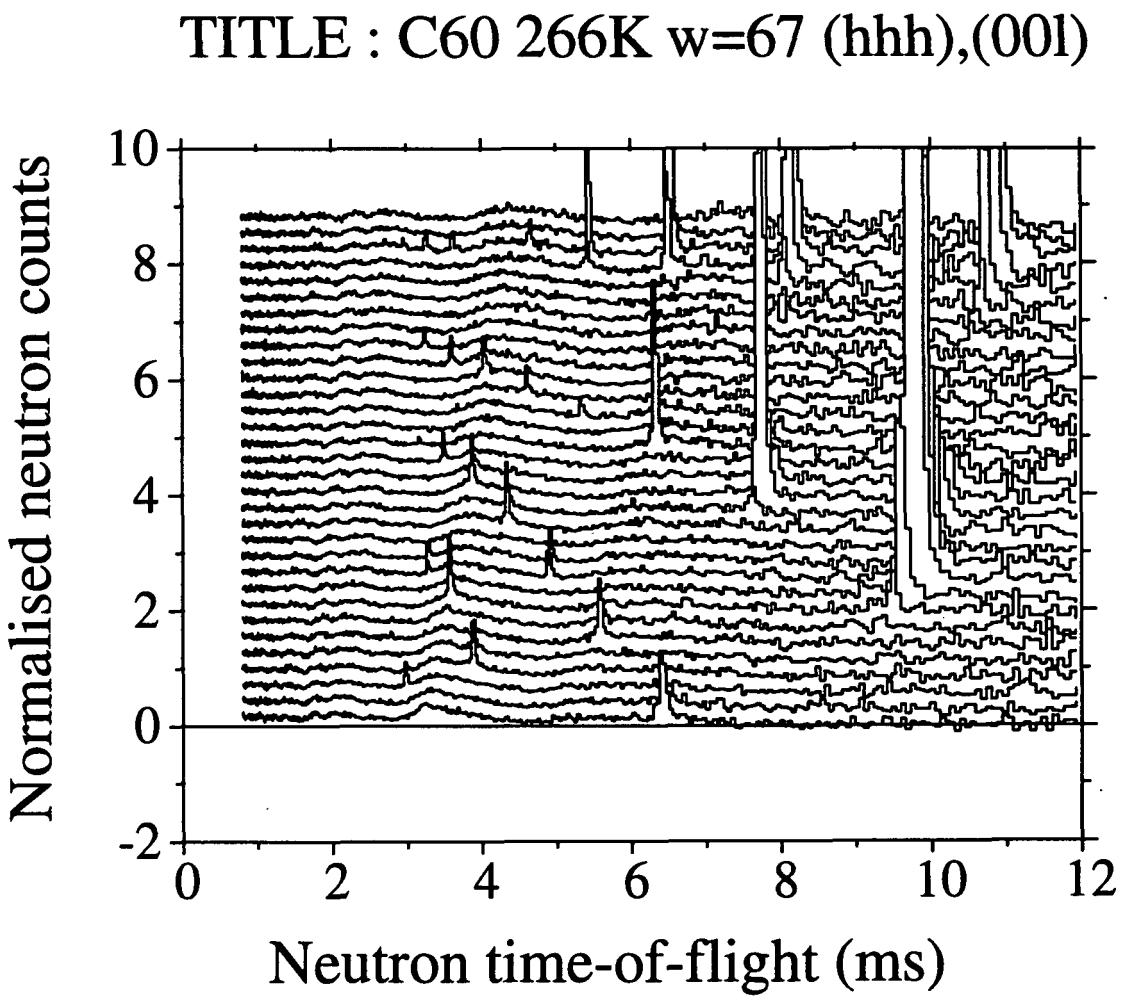

Figure 3. Normalised summed data from C_{60}, ω=67°, high-angle detector 1 using SUMV_1_5 and the same spectra in the *hhl* equatorial plane as Figure 2.

QCALC_1_4 takes the output from SUMV_1_5 and using extra information converts time-of-flight - 2θ data to binned Q_x - Q_y data in the measured reciprocal lattice plane. The binning works by placing each t-o-f - 2θ point in a Q_x - Q_y bin of a certain size, adding up the total intensity that occurs in that bin and dividing by the total number of times a t-o-f - 2θ point has been placed in that bin. It calculates the reciprocal lattice plane between 0 and 100x(grid spacing - specified in row#6) in reciprocal lattice units. It is possible to specify

an offset for this calculation by giving the Q_x - Q_y lower left-hand corner position other than (0,0) - in row#7.

For example, for a cubic sample with [1 $\bar{1}$ 0] vertical and 0.1 grid spacing a plane between 10 10 0 and 0 0 10 will be calculated, provided the zero is specified as (0,0). If instead (5,0) had been given, the plane would have been calculated between 5 5 0 and 15 15 0 along x and between 0 0 0 and 10 0 0 along y. The 'lattice parameters' (row#4) assume a 90° angle between the two principle rows measured in the equatorial plane of the detector. For a cubic system this would be for example (100) and (010) for [001] vertical or (110) and (001) for [1 $\bar{1}$ 0] vertical. For a hexagonal system, the lengths a,b,c must be chosen to give the correct scaling. They essentially define the reciprocal lattice spacing along each of the plot axes. To decide between the options given in row#5, the 'x-axis' corresponds to the principle row measured at the smallest ω-angle. The 'zero-angle' (row#9) defines the ω-angle that the sample must be rotated to such that the x-axis defined by row#5 would scatter into the centre of the detector. If a number of runs are being added together, this parameter may be adjusted from run to run to take into account any backlash in the ω-drive and may simply be calculated from the positions of the Bragg peaks measured in each run.

It is advisable to run this correction command file in batch mode e.g.

```
$ SUBMIT/QUE=SXD$BATCH/NOPRINT/LOG=SXD$DISK1:[SXD.sample] SV_1_5.COM
```

The output from the QCALC_1_4 routine is shown in Figure 4 using data from the two frames and from both detectors. The whole reciprocal lattice plane is not completely covered using these two frames such that there are two narrow gaps in the plot in Figure 4. These result both from the small gap between the two detectors and from using a large change in ω between frames. The data, however, are well corrected with a smooth variation of diffuse scattering across the reciprocal lattice plane. These routines therefore give a good approximation to the diffuse scattering in the reciprocal lattice plane corresponding to the equatorial plane of the SXD instrument. It should be noted however that taking a fixed number of pixels vertically (specified by parameter#9 in SUMV_1_5) will include a variable amount of reciprocal space in the direction perpendicular to Q_x and Q_y. Reciprocal space becomes more compressed at high-|Q|. In our C_{60} example with a [1 $\bar{1}$ 0] direction vertical, the angular separation between the 222 and the 312 reflection (222+1 $\bar{1}$ 0) from the next layer above it is 22.2° whereas there is only 4.7° between the 10 10 10 and the 11 9 10 reflections (10 10 10 +1 $\bar{1}$ 0). This means that there is a tendency to include insufficient pixels in a Q_x-Q_y bin at low-|Q| and too many at high-|Q|. The consequence of this is to see Bragg reflections from out-of-plane peaks at high-|Q| and poor statistics at low-|Q|.

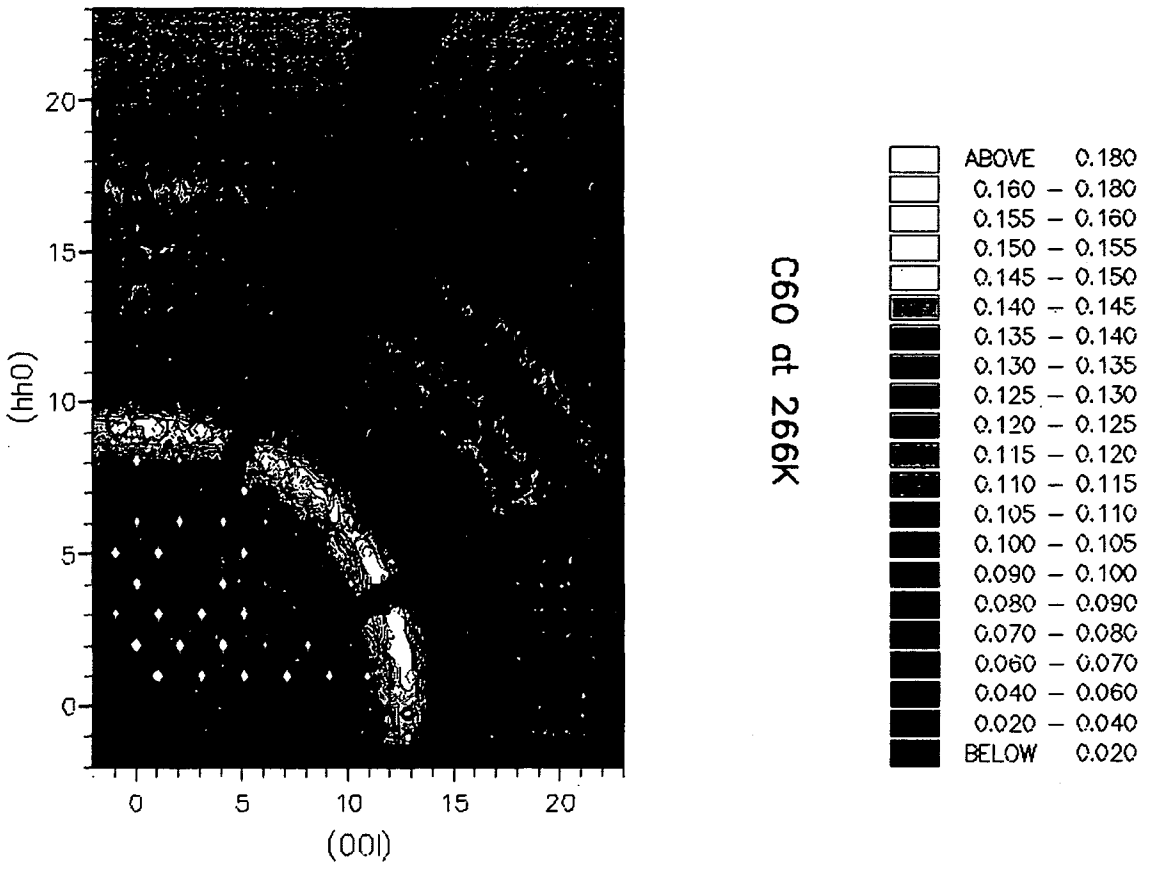

Figure 4. The *hhl* reciprocal lattice plane from C_{60} using equatorial data from both frames and both detectors and the routines SUMV_1_5 and QCALC_1_4.

3. Correction Routines for Producing Normalised Structure Factor Data

These routines are more time consuming and are intended for those who are going to use the data in a more quantitative manner such as model fitting or those who require data which are not only in the equatorial plane of the detectors. There are three stages in this procedure: first the individual time-of-flight spectra are corrected for electronic dead-time, time-offsets, divided by the monitor spectrum and converted to $|Q|$. This is carried out using the NORM routines (described in the ATLAS manual by Soper *et al* 1989). The second stage normalises the data which have been added together by NORM, taking into account absorption in the sample, multiple scattering and subtraction of sample container scattering, backgrounds etc. A vanadium run and appropriate background runs in the same experimental configuration are required for this stage. These corrections are carried out using the CORRECT programme developed by M. A. Howe, based on programmes largely developed at Oxford [3]. These routines are designed for cylindrical sample geometries and will not work for some more complex sample environments. There is a brief description of the CORRECT routines in Appendix A. The final stage performs a similar task to QCALC_1_4 described above, only it converts the $(x,z,|Q|)$ data which are output from CORRECT into binned data for the required reciprocal lattice plane. All the

programs and command files described in this section are found in SXD$DISK1:[SXD.C60.DEC95.CORRECT] which, for convenience, can be assigned to diff_cor using the following command:

```
$ ASS/NOLOG SXD$DISK1:[SXD.C60.DEC95.CORRECT] diff_cor
```

3.1 Stage One - NORM

SXD routinely runs with two position sensitive detectors, and it is most convenient to correct the data from each detector separately. It is suggested that two subdirectories are created e.g. SXD$DISK1:[SXD.sample] and SXD$DISK1:[SXD.sample.DET2]

NORM requires three input files as well as the data files, and these must usually be set up for each experimental configuration and placed in the working directory SXD$DISK1:[SXD.sample]. ENCODERS.DAT contains information about which detector elements go into which encoder. Only two encoders are used on SXD (one for each detector) and an example ENCODERS.DAT file would be:

```
2
1 4096
4101 8196
```

DETECTOR.CALIB files contain information about the detector positions and performance. This file may be created by running DETECTOR.COM, suitably edited for the experimental setup being used, and will have information about both detectors and monitor(s) (all 8196 spectra). It is recommended that this file be called DETECTOR_DATA.CALIB. A similar file, but with only the information about the second detector (spectra 4101-4196) should also be created in SXD$DISK1:[SXD.sample.DET2] for use in stage three (DETECTOR_DATA2.CALIB). GROUPS.DAT files describe which detector elements are added together by NORM, although, for these routines there is no grouping and a one-to-one correspondence is established by running GROUPS. File names GROUPS_1.DAT and GROUPS_2.DAT are recommended for detectors one and two respectively. Using these parameters, NORM may be run on each of the data files and the background file. This must be done in batch and an example command file is shown below (NORM.COM):

```
$ ASS/NOLOG SXD$DISK1:[SXD.C60.DEC95.CORRECT] diff_cor
$ on error then continue
$ @diff_cor:SETUPNORM
$ ASS/NOLOG SXD$DISK1:[SXDMGR.DIFFUSE.PROGS] norm_par
$ NORM:==RUN G_F:NORM_5_2_1_AXP.EXE
$ SET DEF SXD$DISK1:[SXD.sample]
$ COPY DETECTOR_DATA.CALIB DETECTOR.CALIB
$! running NORM for the first detector
$ NORM
> SET INST SXD
> SET DISK SXD$DISK1:
> SET DIR [SXDMGR.DATA]
> SET EXT RAW
> SET WORK 70 2000
> SHOW DEF
> CAL
```

```
> GR GROUPS_1.DAT

> RUN 8108
> BEGIN

0.025
25
> EXIT
$! running NORM for the second detector
$ NORM
> SET INST SXD
> SET DISK SXD$DISK1:
> SET DIR [SXDMGR.DATA]
> SET EXT RAW
> SET WORK 70 2000
> SHOW DEF
> CAL
> GR GROUPS_2.DAT

> RUN 8108
> BEGIN

0.025
25
> EXIT
$ RENAME SXD08108.NRM [.DET2]SXD08108.NRM
```

The two parameters after > BEGIN define the $|Q|$ bin size and the maximum $|Q|$ (in $Å^{-1}$) respectively. A full description of the commands can be found in the ATLAS manual (Soper *et al* 1989). The output files are named SXD0nnnn.NRM and contain data for each group defined by GROUPS.DAT as a function of $|Q|$ in GENIE format with the .NRM file for the second detector redirected to [.DET2]. Another file, SXD0nnnn.MON contains the corrected monitor spectrum. These files can be inspected in GENIE using the READ command i.e.

```
>> READ Wn SXD0nnnn.NRM n
```

will read the n^{th} block into workspace n. This corresponds to the detector elements summed in the n^{th} group as defined by GROUPS.DAT.

The data from the vanadium run must be treated slightly differently in order to improve the counting statistics. This is because it is not appropriate to collect data from a vanadium sample for the length of time required to obtain suitable statistics for an individual pixel. Hence neighbouring pixels are added together and then scaled to produce the correct overall time-integrated intensities. To do this NORM uses an extra parameter which scales the data and with different GROUPS.DAT files to sum neighbouring spectra on the detector. This aspect is very time-consuming, but does provide much smoother data. First a V_'run_no'.CALIB file is created in GENIE by running INT_VAN.COM. Two GROUPS.DAT files are then created by running GROUPS_V.EXE (Suggested filenames are GROUPS_V1.DAT and GROUPS_V2.DAT for detector one and two respectively). This information is then incorporated into DETECTOR_V.CALIB by running DETECTOR_V.EXE. NORM is then run for the vanadium file in the usual way with appropriate changes of input file names, e.g.

```
$ ASS/NOLOG SXD$DISK1:[SXD.C60.DEC95.CORRECT] diff_cor
$ on error then continue
$ @diff_cor:SETUPNORM
$ ASS/NOLOG SXD$DISK1:[SXDMGR.DIFFUSE.PROGS] norm_par
$ NORM:==RUN G_F:NORM_5_2_1_AXP.EXE
$ SET DEF SXD$DISK1:[SXD.sample]
$! appropriate detector.calib for vanadium files only
$ COPY DETECTOR_V.CALIB DETECTOR.CALIB
$! running NORM for the first detector
$ NORM
> SET INST SXD
> SET DISK SXD$DISK1:
> SET DIR [SXDMGR.DATA]
> SET EXT RAW
> SET WORK 70 2000
> SHOW DEF
> CAL
> GR GROUPS_V1.DAT

> RUN 8112
> BEGIN

0.025
25
> EXIT
$! running NORM for the second detector
$ NORM
> SET INST SXD
> SET DISK SXD$DISK1:
> SET DIR [SXDMGR.DATA]
> SET EXT RAW
> SET WORK 70 2000
> SHOW DEF
> CAL
> GR GROUPS_V2.DAT

> RUN 8112
> BEGIN

0.025
25
> EXIT
$ RENAME SXD08112.NRM [.DET2]SXD08112.NRM
```

3.2 Stage Two - CORRECT

The SXD0nnnn.NRM files from NORM for each sample run together with background and vanadium runs can now be used by the CORRECT programme. To run this programme type (or set this up in a command file to run as a batch job):

```
$ RUN SXD$DISK1:[SXDMGR.DIFFUSE.PROGS]CORRECT_MANYSPECTRA
CORRECT> @file.COM
```

More details on this programme are found in Appendix A, including some of the adaptations that were necessary for SXD data. However, an example file.COM is shown below:

```
INST SXD
X Q
VANADIUM SXD01868.NRM 0.3
BACKGROUND SXD01831.NRM/SAMPLE
SAMPLE SXD01816.NRM 0.44/TEMP=293/DEN=0.01648
OUTPUT SXD01816.COR
COMPONENT 1.00    207.19 11.11 0.17
TITLE "Lead at T=293K"
BEAM 0.9 0.9
RANGE 0.5 7.0
PATHS 8.0 0.23
SPECTRUM 1    80.74   0.233037/DET=SCINT
SPECTRUM 2    81.96   0.232285/DET=SCINT
SPECTRUM 3    83.18   0.231638/DET=SCINT
SPECTRUM 4    84.41   0.231098/DET=SCINT
SPECTRUM 5    85.65   0.230665/DET=SCINT
SPECTRUM 6    86.89   0.230339/DET=SCINT
SPECTRUM 7    88.13   0.230122/DET=SCINT
SPECTRUM 8    89.38   0.230014/DET=SCINT
SPECTRUM 9    90.62   0.230014/DET=SCINT
SPECTRUM 10   91.87   0.230122/DET=SCINT
SPECTRUM 11   93.11   0.230339/DET=SCINT
SPECTRUM 12   94.35   0.230665/DET=SCINT
SPECTRUM 13   95.59   0.231098/DET=SCINT
SPECTRUM 14   96.82   0.231638/DET=SCINT
SPECTRUM 15   98.04   0.232285/DET=SCINT
SPECTRUM 16   99.26   0.233037/DET=SCINT
.
.
.
.
. etc.
EXECUTE
```

It should be noted that, unlike the usual CORRECT routines, each spectrum has a scattering angle *and* secondary flight path associated with it. These values are taken from the entry in DETECTOR_DATA.CALIB for each pixel by running CREATE_CORFILE.EXE and remembering to create a different file.COM for each detector (with corresponding different L2's and 2θ's). The output from CREATE_CORFILE must be edited onto an appropriate file.COM header portion. The output file from this programme is again in GENIE format (and is usually called SXD0nnnn.COR) and can be inspected in GENIE in the same way as the SXD0nnnn.NRM files (see above). The data which are equivalent to those shown in Figures 2 and 3 are shown in Figure 5. These data are plotted against $|Q|$ and have been absolutely normalised.

3.3 Stage Three - conversion to reciprocal space

This is the part where the single crystal nature of the scattering is taken into account. In order to convert the $(x,z,|Q|)$ points to relevant (h,k,l) points with appropriate binning, an accurate knowledge of the crystal orientation is required. The crystal orientation with respect to the SXD diffractometer is described using the so-called UB matrix and a knowledge of the SXD geometry. This matrix and instrument geometry may be determined and refined using the location in (x,z,t) of the Bragg peaks and is described in detail in the SXD manual for Bragg peak intensity extraction [4]. Having obtained this information (in a file called SXD0nnnn.USE), any given (h,k,l) may be directly mapped on

to an $(x,z,|Q|)$ point in detector space. However, within a given $(h\pm\delta h,k\pm\delta k,l\pm\delta l)$ bin there may be many $(x,z,|Q|)$ points and the number is strongly $|Q|$ dependent. In the PLANESETUP program, the 8 extremes of x, z and λ (or t) are determined for each

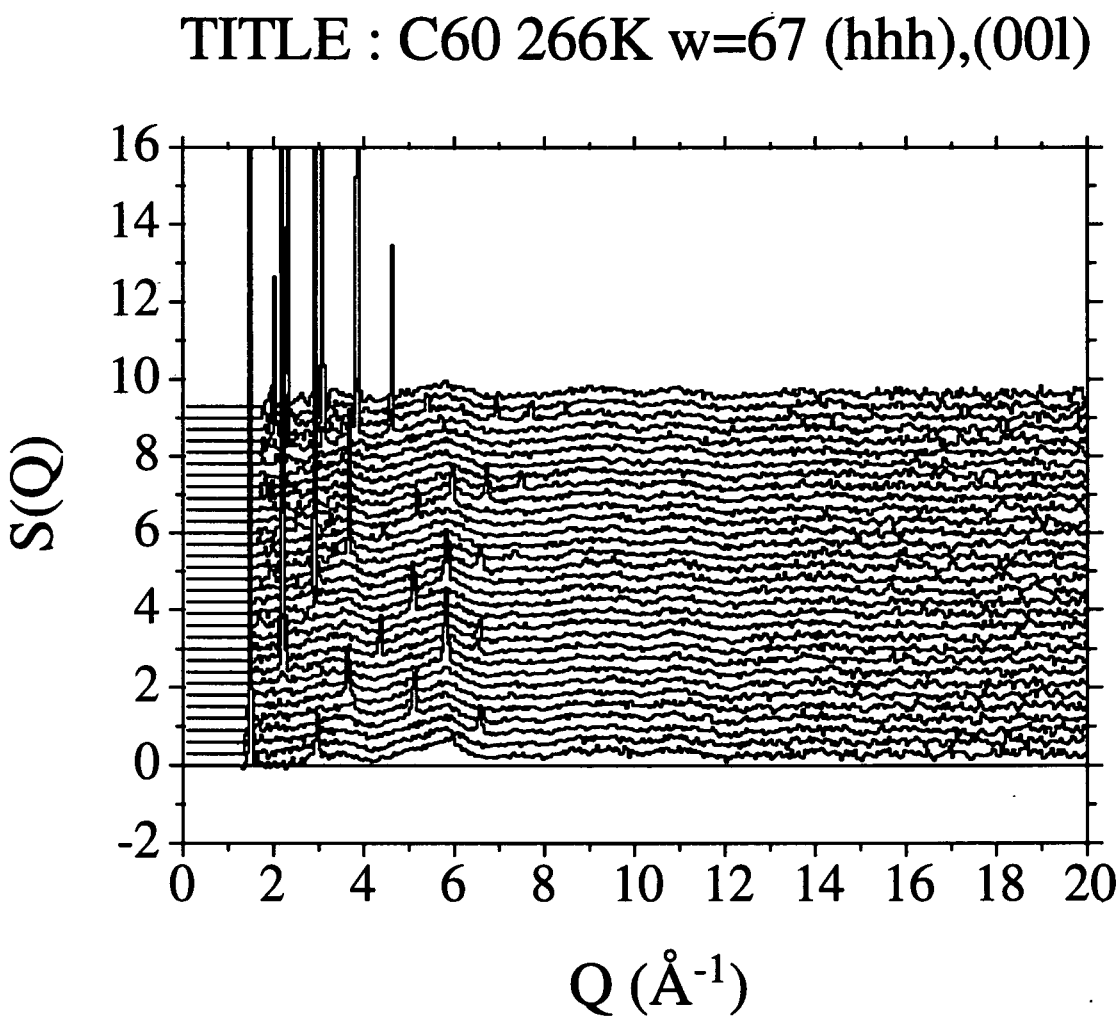

Figure 5. Normalised data from C_{60}, ω=67°, high-angle detector 1 using NORM and CORRECT and summing the same spectra in the *hhl* equatorial plane as in Figure 2.

$(h\pm\delta h,k\pm\delta k,l\pm\delta l)$ bin in the reciprocal lattice plane to be calculated. A circle of radius r and centred on the (x,z) corresponding to (h,k,l) is constructed which includes all the pixels which contain a contribution to the $(h\pm\delta h,k\pm\delta k,l\pm\delta l)$ bin. All data from the pixels within this circle and between q_{min} and q_{max} are included in the $(h\pm\delta h,k\pm\delta k,l\pm\delta l)$ bin where q_{min} and q_{max} have been determined from the the extremes of λ. Clearly this is only a close approximation to the true shape of the $(h\pm\delta h,k\pm\delta k,l\pm\delta l)$ bin in $(x,z,|Q|)$ space, but it is computationally straightforward, reasonably fast and is only unsuitable for crystal systems with very dissimilar axes.

```
$! an example of running PLANESETUP
$ ASS/NOLOG SXD$DISK1:[SXD.C60.DEC95.CORRECT] diff_cor
$ RUN diff_cor:PLANESETUP_001
detector_data.calib                    ! DETECTOR.CALIB file:
sxd08080.use                           ! Input .USE file:
0 15                                   ! hmin, hmax
0 15                                   ! kmin, kmax
0 0                                    ! lmin, lmax
0                                      ! Other conditions 0: no,1: h=k, 2: h=-k
.1 .1                                  ! Spacing in r.l.u.
-25 25                                 ! Use pixels between zmin and zmax:
peaks.prep                             ! Output file
```

The output from PLANESETUP (e.g. a file called PEAKS.PREP) is a list of *h,k,l* with the spectrum numbers and q_{min} and q_{max} which contribute to each given *h,k,l*. PEAKS.PREP is then used by GENERALQCALC to extract the relevant ($x,z,|Q|$) from the SXD0nnnn.COR file output from CORRECT. GENERALQCALC will also allow you to combine data from different crystal orientations. The output from GENERALQCALC contains the data in final analysed

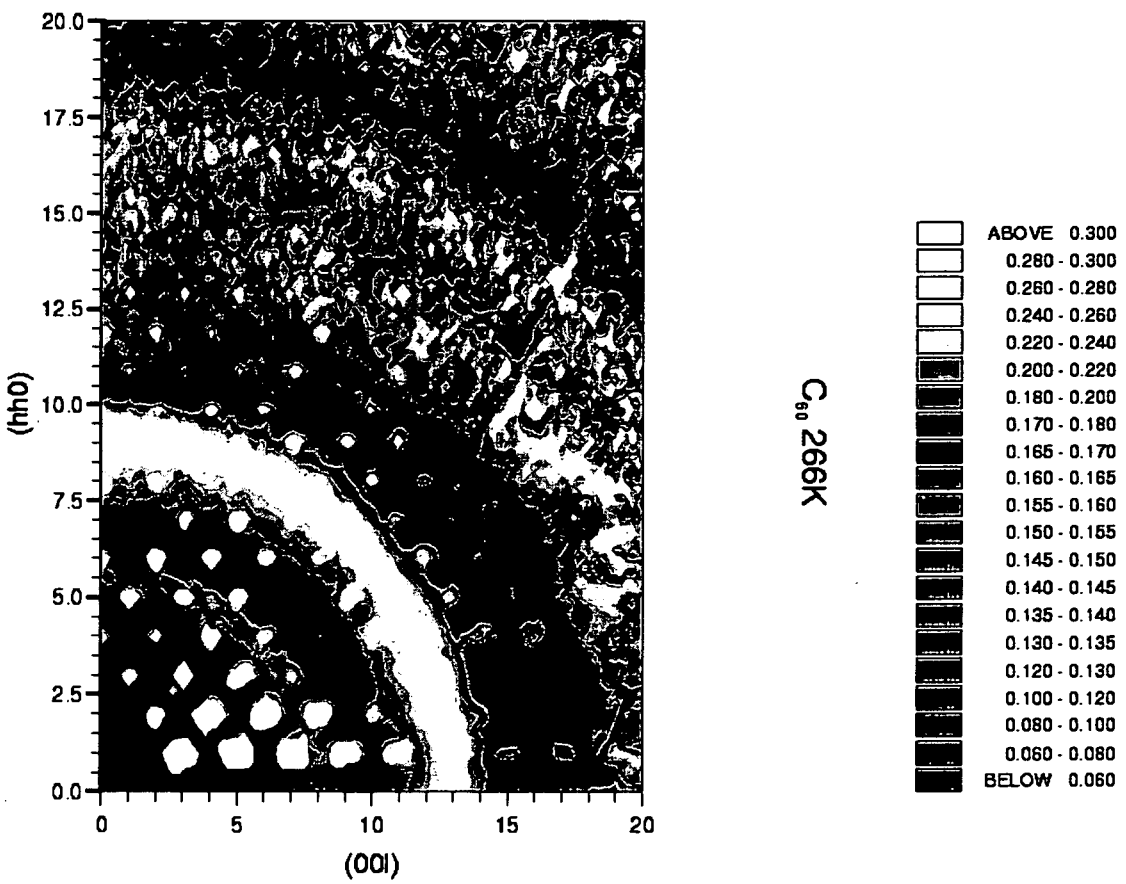

Figure 6. The *hhl* reciprocal lattice plane from C_{60} using all equivalent *hhl* data from both frames and both detectors

form ready for plotting and interpretation. PLANESETUP and GENERALQCALC (both written in Fortran) are not yet completely general and it is possible that small modifications to the routines will be necessary for less common planes.

```
$! example file for the running of GENERALQCALC
$ ASS/NOLOG SXD$DISK1:[SXD.C60.DEC95.CORRECT] diff_cor
$ SET DEF SXD$DISK1:[sxd.sample]
$ RUN diff_cor:GENERALQCALC
1                                   ! 1:l, 2:k, 3:h constant, 4:h=k, 5: all h,k,l varying
.333333 .333333 .333333             ! grid spacing r.l.u.
0 0 0                               ! zero offset
sxd08108.cor                        ! corrected data filename
peaks.prep_8108_001                 ! file output from PLANESETUP
groups_1.dat                        ! GROUPS.DAT file
y                                   ! another data file?
[.det2]sxd08109.cor                 ! corrected data filename
[.det2]peaks.prep_8109_001          ! file output from PLANESETUP
groups_1.dat                        ! GROUPS.DAT file
n                                   ! another data file
c60_20K_hk0.dat                     ! output file
```

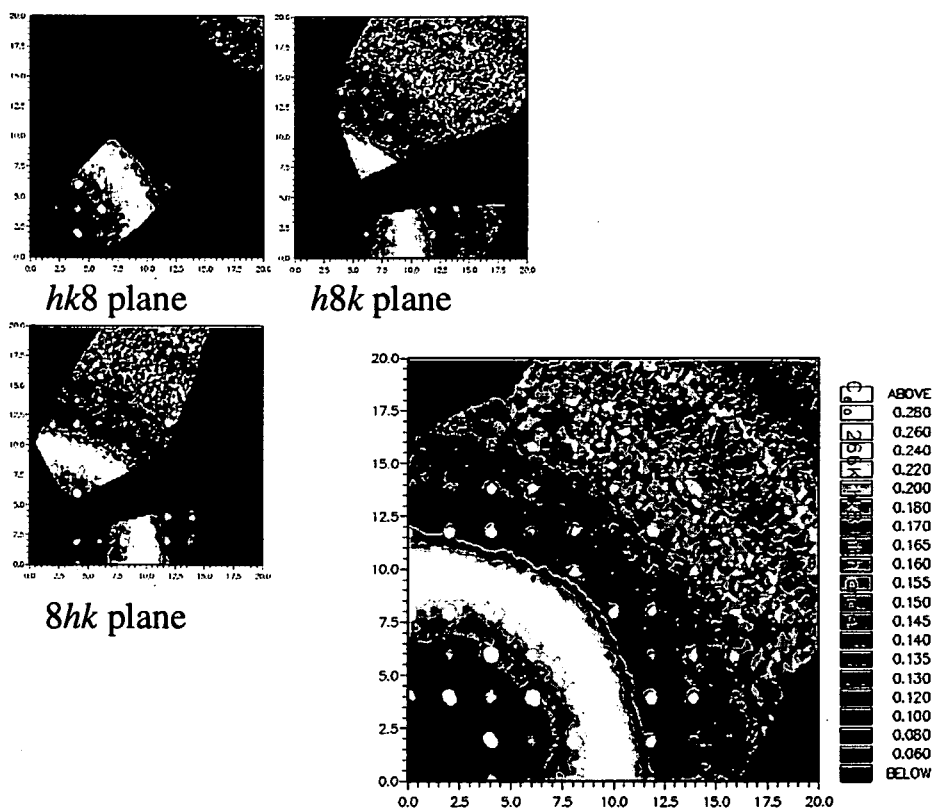

Figure 7. The *hk*8 reciprocal lattice plane from C_{60} at 266K summed from three symmetry equivalent planes, which are also shown. High angle detector, 2 crystal settings only.

The equivalent data to those shown for the quick corrections routines in Figure 4 are shown in Figure 6. There is very little difference between the two plots although three things should be noted. First, the gaps which are in Figure 4 have been filled in using data from parts of the equivalent *hkh* and *hkk* reciprocal lattice planes which occur out of the equatorial plane. Secondly, the Bragg peaks around the [00*l*] direction are broader in Figure 6 due to the inclusion of the low-angle scattering of the $h\bar{h}\,l$ plane which have more significantly resolution broadened reflections than elsewhere (see Figure 1). Thirdly the low-Q scattering in Figure 6 is more reliable due to the inclusion of significantly more data than was used in Figure 4. Moreover it is possible to combine more data and to calculate data from reciprocal lattice planes which are not in the equatorial plane. Figure 7 and Figure 8 shows the *hk*8 reciprocal lattice plane and how it has been obtained from a combination of small sections of symmetry equivalent reciprocal lattice planes which impinge on the two detectors. Complete flexibility may be obtained using this diffuse scattering data correction approach.

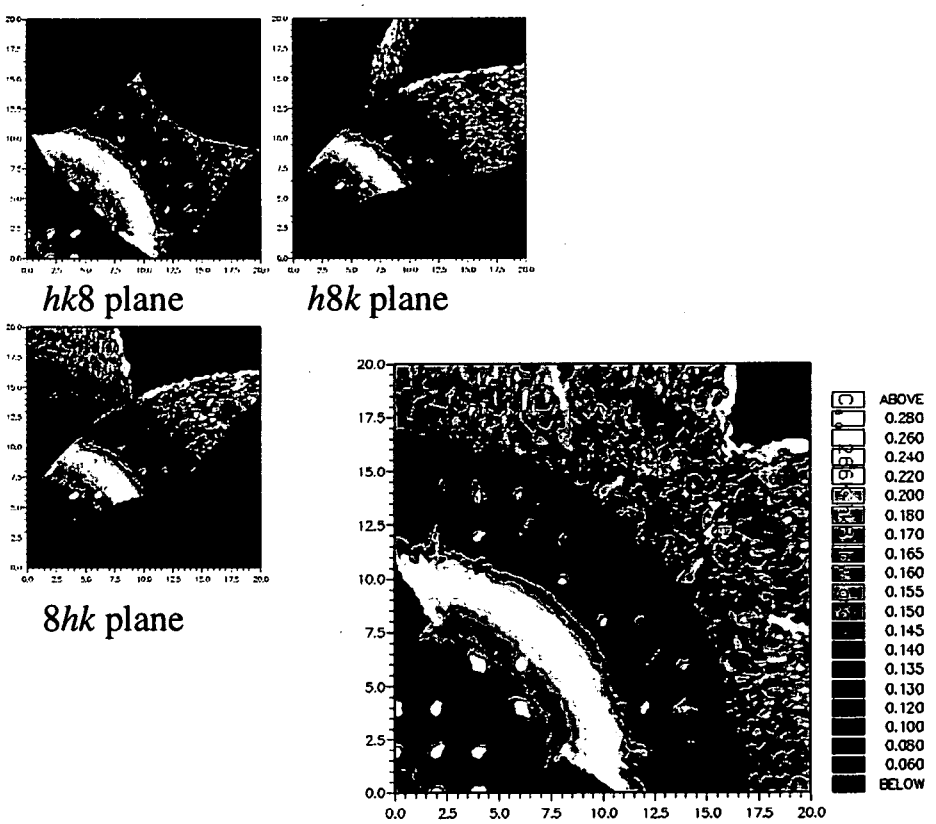

Figure 8. The *hk*8 reciprocal lattice plane from C_{60} at 266K summed from three symmetry equivalent planes, which are also shown. Low angle detector, 2 crystal settings only.

4. Plotting Output Data

This is facilitated by running `DP:PLOT3D_100100` and the input to this routine is largely self-explanatory. To obtain a black and white hard-copy of a plot which you are happy with `GROUTE> SE MPOST;EX` will create a file `POST.DAT`. This may be sent to a POSTSCRIPT printer by using the command e.g.

```
$ PRINT/QUE=POST$LSR0/DELETE POST.DAT
```

Note that colour option 3 selects a grey scale for shading. For a colour hard-copy `GROUTE> SE HNECPSA4;EX` also produces a file called `POST.DAT`, which may be sent to the colour postscript printer using the command

```
$ PRINT/QUE=COLOUR$PHASER0/DELETE POST.DAT
```

5. References

[1] D. A. Keen and C. C. Wilson, *Rutherford Appleton Laboratory Report* RAL-TR-96-083, (1996)

[2] A. K. Soper, W. S. Howells and A. C. Hannon, *Rutherford Appleton Laboratory Report* RAL-89-046, (1989)

[3] M. A. Howe, R. L. McGreevy and W. S. Howells, *J. Phys.:Condens. Matter*, **1** (1989) 3433

[4] C. C. Wilson, *to be published as a Rutherford Appleton Laboratory Report* (1996)

A. Brief notes on CORRECT

These notes are based on a manual written by M. A. Howe, a copy of which can be obtained from the instrument scientists. It is the intention of this appendix to describe the mechanics of running the routines, rather than the underlying physics which govern the correction processes. As such, only a description of the relevant commands and associated parameters will be given here and the readers are referred to reference [3] for a description of *why* such corrections are necessary. Details of how to run the CORRECT program and an example command file are found in Section 3.2.

A1 Details of commands

The default values assumed by CORRECT are given in square brackets.

BACKGROUND - Defines the file containing the background

parameter	The file [background for everything].
/FURNACE	Defines the background for furnace only.
/CONTAINER	Defines the background for container only.
/SAMPLE	Defines the background for sample only.
/VANADIUM	Defines the background for vanadium only.

BEAM - Defines the beam dimensions (in cm)

parameter1	Beam height [3cm]
parameter2	Beam width [1.5cm]

COMPONENT - Defines the properties of one of the components of the sample. This command must be used once for each component.

parameter1	The *relative* proportion of the component
parameter2	The atomic weight
parameter3	The atomic scattering cross-section (barn)
parameter4	The absorption cross-section (barn) at 2200ms^{-1} (1.798Å)

CONTAINER - Defines the file containing the container data and some of its properties

parameter1	The file
parameter2	The container external radius (cm)
/ABSORPTION=value	The absorption cross-section of the container at 2200ms^{-1}. [Value for vanadium.]
/DENSITY=value	The number density of the container (in Å^{-3}). [Value for vanadium.]
/MULTIPLIER=value	A 'correction' factor by which the container scattering (after subtraction of any background) is multiplied. [1]
/SCATTERING=value	The scattering cross-section of the container. [Value for vanadium.]

EXECUTE - Performs the correction

/NOLIST	Supresses listing of program parameters
/OUTPUT=file	Sends output to file

FURNACE - Defines the file for the furnace and some of its properties

parameter1	The file
parameter2	The furnace heater internal radius (cm).
parameter3	The furnace heater external radius (cm).
/ABSORPTION=value	The absorption cross-section of the furnace at 2200ms^{-1}. [Value for vanadium.]
/DENSITY=value	The number density of the furnace (in Å^{-3}). [Value for vanadium.]
/SCATTERING=value	The scattering cross-section of the furnace. [Value for vanadium.]

INSTRUMENT - Defines the instrument used - should be set to SXD

OUTPUT - Defines the output file

parameter	The file (usually SXDnnnnn.COR)

PATHS - Defines the incident and scattered path lengths (m).

parameter1	The incident flight path (moderator to sample) [10m] - the SXD incident flight path is 8m.
parameter2	The scattered flight path (sample to detector) [1.043m]. parameter2 is superseded by the values in SPECTRUM.

RANGE - Defines the range of wavelengths to be used (Å).

parameter1	Minimum wavelength [0.1Å].
parameter2	Maximum wavelength [3Å].

SAMPLE - Defines the sample and some of its properties

parameter1	The file
parameter2	The sample radius (cm)
/DENSITY=value	The number density of the sample (in Å^{-3}).
/FULLNESS=value	The fullness of the container [1].
/TEMPERATURE=value	The temperature of the sample (in K) [300K].
/CS_FILE	A file containing the total cross-section of the sample as a function of wavelength, typically in 0.5Å intervals up to 10Å. [Calculate this from COMPONENT information.]

SPECTRUM - Defines the spectrum to be corrected

parameter1	The spectrum number
parameter2	The scattering angle (2θ)
parameter3	The secondary flight path (m).
/DETECTOR	Either =HE_GAS or =SCINTILLATION [HE_GAS]

TITLE - Defines a title for the file

parameter	The title

VANADIUM - Defines the vanadium data

`parameter1`	The vanadium file
`parameter2`	The vanadium radius (cm)
`/ABSORPTION=value`	The absorption cross-section of vanadium at 2200ms^{-1}. [5.08barn]
`/DENSITY=value`	The number density of vanadium (in Å^{-3}). [0.0722Å^{-3}]
`/SCATTERING=value`	The scattering cross-section of vanadium. [4.953barn]

WRITE - Defines what should be written to the output file

`parameters`	`ABSORPTION`, `CALIBRATION`, `PLACZEK` [corrected data only].

X - Defines what the input data are recorded as a function of [Q]