Sample 3

This sample has a long story. It was question of it at the SDPD mailing list in 1999. The discussion never gave the cell, or space group, but it is clear that reading the emails (N°125 and 196) below provides informations about how to solve the structure :

==========

> In the series of SDPD failures (shame on us, but it is not too late,
> though who knows what will happen after Y2K...):
>
> ---
> Decembre 31, 1999, in case of the structure of $C_{61}Br_2$ is still not
> determined, the powder data will be released to the public,
> including the cell parameters (quite good data recorded at ESRF,
> BM01 with FWHM as low as 0.015°2-theta).
>
> This is the $C_{61}Br_2$ day less 109 !
> N. Dragoe & A. Le Bail

Unfortunately for you, we consider having "solved" that
structure just now. This was done by using ESPOIR on
recent single crystal data, though the powder data give
the "solution" as well. The quotes are meaning that
C_{60} was located as a sphere with a global scattering
factor, and the Br atoms are statistically distributed
over 2 sites. R_F=11% for the 50 first hkl and 7 coordinates
refined (single crystal). R_B=7.5% and R_P=12.8% for the
powder diffraction data by the Rietveld method.

Care to the Y2K BUG,
Merry Christmas and Happy New Year to all of you.

N. Dragoe & A. Le Bail

==========

Anyway, since that result was never published, $C_{60}CBr_2$ was decided to be proposed as the sample 3 of the SDPDRR-2.

N. Dragoe sent me first a very good quality conventional X-ray powder pattern (Rigaku – rotating anode - Cu-Kalpha). That pattern could be indexed by a cubic cell, I-centered. But all efforts in order to solve the structure were unsuccessful by classical approaches. A further synchrotron pattern recorded at the ESRF on BM01 in 1999 did not change a lot the difficulty level, however the extremely good resolution without any peak splitting, confirms the cubic cell. I wasted months on that compound, and the ESPOIR program was built in part in order to try more to solve it (by positioning the whole C_{60} molecule plus the Br atoms). Nothing came when ordered models were built (6 possible space groups : Im3m, I-43m, I432, $I2_13$, Im3, I23, etc). Example of “best” ordered model : putting one $C_{60}CBr_2$ molecule in a general position of the I23 space group results in 24 molecules and we need only 8, but if atoms

overlap exactly 3 by 3, the model could become satisfying. The best model obtained in that way with ESPOIR gives R=27% in the I23 space group on the 50 first reflections – quite deceiving. The light came then with an hypothesis of disordered C_{60} molecule. A global scattering factor taken from FULLPROF, representing a sphere of refinable radius, was used and attempts to locate its position were made, as well as to locate the Br atoms simultaneously, by using an ESPOIR-2 special version. A single crystal was further obtained, But the corresponding data set (m3m Laue class) did not change really the problem since again, no ordered model could be established.

More or less convincing models are proposed below, resulting of many attempts in all possible space groups :

The best C_{60} molecule position is found to be of the x,x,x kind, with x~0.18. The fit is possible only in I-43m or I23 for having 8 molecules per cell. When trying in I23, it is supposed that microtwinning occurs because the Laue group is observed to be m3m with the single crystal data. Two Br atoms, with partial occupancy, are necessary for obtaining small R_F value. In both space groups, one of the positions is near of 0,1/2,1/4.

ESPOIR-2 results

Test for solving in the I23 space group on the single crystal data :

```
    238 moves acc.   90000 tested; Chi**2=0.142    , R=0.142
      5 perm. acc.    9999 tested
     40 events did not improved the fit, dump = 0.000000

 Final coordinates x,y,z and occupation numbers

C60      0.31224    0.31224    0.31224      1.000
Br1      0.56170    0.92827    0.57372      1.000
Br2      0.48838    0.72942    0.99121      1.000
C1       0.44569    0.01086    0.63981      1.000
 14-Dec-1999   Hour: 10 Min: 56 Sec: 29
```

hkl data :

```
  50
   1   1   0    0.98   0.269   1
   2   0   0    8.69   0.039   1
   2   1   1    6.28   0.070   1
   2   2   0    0.66   0.099   1
   3   1   0    1.09   0.124   1
   2   2   2    3.09   0.042   1
   3   2   1    7.36   0.103   1
   4   0   0    7.96   0.030   1
   3   3   0   24.77   0.117   1
   4   1   1   20.21   0.247   1
   4   2   0    0.68   0.184   1
   3   3   2   12.91   0.168   1
   4   2   2   13.18   0.086   1
   4   3   1    4.16   0.112   1
   5   1   0    2.63   0.070   1
   5   2   1    3.91   0.098   1
   4   4   0    7.80   0.086   1
   4   3   3    5.02   0.105   1
   5   3   0    9.64   0.127   1
```

```
   4   4   2   15.05   0.127   1
   6   0   0    4.97   0.090   1
   5   3   2   10.02   0.164   1
   6   1   1    8.60   0.122   1
   6   2   0   11.96   0.148   1
   5   4   1    4.48   0.122   1
   6   2   2    8.15   0.151   1
   6   3   1    5.00   0.140   1
   4   4   4    8.79   0.177   1
   5   4   3    4.25   0.122   1
   5   5   0    5.78   0.061   1
   7   1   0    3.75   0.085   1
   6   4   0    3.75   0.092   1
   5   5   2    3.34   0.070   1
   6   3   3    4.15   0.166   1
   7   2   1    5.81   0.106   1
   6   4   2    1.79   0.162   1
   7   3   0    0.92   0.185   1
   6   5   1    1.77   0.212   1
   7   3   2    2.56   0.123   1
   8   0   0    1.04   0.115   1
   5   5   4    3.46   0.104   1
   7   4   1    1.78   0.143   1
   8   1   1    4.74   0.097   1
   6   4   4    2.63   0.152   1
   8   2   0    2.65   0.077   1
   6   5   3    3.91   0.131   1
   6   6   0    1.34   0.190   1
   8   2   2    3.42   0.137   1
   7   4   3    2.22   0.147   1
   7   5   0    1.51   0.155   1
```

Data for ESPOIR-2 – special version

```
Test on C61Br2 I23 Twinned ?
18.87863  18.87863  18.87863  90.0  90.0  90.0
I 2 3
0.7073 4 4 -3 2
0 1 0                    !!! twinning law
1 0 0
0 0 -1
C60 Br  C
57 2000. 360.            !!! C60 scattering factor
   0.000      1.00000
  22.599      0.95840
  45.198      0.83990
  67.797      0.66190
  90.396      0.45010
 112.994      0.23400
 135.593      0.04170
 158.192     -0.10450
 180.791     -0.19150
 203.390     -0.21710
 225.989     -0.18930
 248.588     -0.12390
 271.186     -0.04140
 293.785      0.03790
 316.384      0.09720
 338.983      0.12610
 361.582      0.12220
```

```
 384.181      0.09030
 406.780      0.04080
 429.379     -0.01300
 451.977     -0.05840
 474.576     -0.08570
 497.175     -0.09030
 519.774     -0.07310
 542.373     -0.04010
 564.972     -0.00010
 587.571      0.03670
 610.169      0.06210
 632.768      0.07090
 655.367      0.06210
 677.966      0.03910
 700.565      0.00820
 723.164     -0.02280
 745.763     -0.04640
 768.362     -0.05750
 790.961     -0.05410
 813.559     -0.03790
 836.158     -0.01350
 858.757      0.01290
 881.356      0.03480
 903.955      0.04730
 926.554      0.04770
 949.153      0.03660
 971.751      0.01720
 994.350     -0.00550
1016.949     -0.02590
1039.548     -0.03910
1062.147     -0.04230
1084.746     -0.03510
1107.345     -0.01970
1129.943     -0.00010
1152.542      0.01870
1175.141      0.03220
1197.740      0.03750
1220.339      0.03340
1242.938      0.02140
1265.537      0.00460
9 1. 1.                  !!!   Br scattering factor
0.00 35.000
0.05 34.291
0.10 32.450
0.15 30.095
0.20 27.749
0.30 23.857
0.40 20.874
0.50 18.307
0.60 15.958
23 1. 1.                 !!!    C scattering factor
0.00 6.000
0.03 5.907
0.05 5.749
0.08 5.396
0.10 5.107
0.12 4.794
0.15 4.311
0.18 3.847
0.20 3.560
0.22 3.297
```

```
0.25 2.949
0.28 2.658
0.30 2.494
0.32 2.351
0.35 2.171
0.38 2.028
0.40 1.948
0.42 1.880
0.45 1.794
0.48 1.725
0.50 1.685
0.55 1.603
0.60 1.537
1 2 1
5. 1 0 1 1
1 1 1 1
2 1 1 1
1.0 0.001
9. 9. 9.
2
5000
100000 20000
100000 0.40 2
10
10
```

Similar results in I-43m space group :

```
    265 moves acc.   90000 tested; Chi**2=0.128    , R=0.128
      3 perm. acc.    9999 tested
     29 events did not improved the fit, dump = 0.000000

 Final coordinates x,y,z and occupation numbers

C60      0.18700    0.18700    0.18700      1.000
Br1      0.58072    0.92454    0.94801      1.000
Br2      0.99083    0.50888    0.77983      1.000
C1       0.51759    0.89181    0.92873      1.000
 14-Dec-1999   Hour: 10 Min: 34 Sec: 15
```

Data for ESPOIR-2 – no twinning

```
Test on C61Br2 I-43m
18.8789   18.8789   18.8789   90.0000   90.0000   90.0000
I -4 3 M
0.7073 4 4 -3 0
c   br  c
57 2000. 360.
   0.000      1.00000
  22.599      0.95840
  45.198      0.83990
  67.797      0.66190
  90.396      0.45010
 112.994      0.23400
 135.593      0.04170
 158.192     -0.10450
 180.791     -0.19150
 203.390     -0.21710
```

```
 225.989     -0.18930
 248.588     -0.12390
 271.186     -0.04140
 293.785      0.03790
 316.384      0.09720
 338.983      0.12610
 361.582      0.12220
 384.181      0.09030
 406.780      0.04080
 429.379     -0.01300
 451.977     -0.05840
 474.576     -0.08570
 497.175     -0.09030
 519.774     -0.07310
 542.373     -0.04010
 564.972     -0.00010
 587.571      0.03670
 610.169      0.06210
 632.768      0.07090
 655.367      0.06210
 677.966      0.03910
 700.565      0.00820
 723.164     -0.02280
 745.763     -0.04640
 768.362     -0.05750
 790.961     -0.05410
 813.559     -0.03790
 836.158     -0.01350
 858.757      0.01290
 881.356      0.03480
 903.955      0.04730
 926.554      0.04770
 949.153      0.03660
 971.751      0.01720
 994.350     -0.00550
1016.949     -0.02590
1039.548     -0.03910
1062.147     -0.04230
1084.746     -0.03510
1107.345     -0.01970
1129.943     -0.00010
1152.542      0.01870
1175.141      0.03220
1197.740      0.03750
1220.339      0.03340
1242.938      0.02140
1265.537      0.00460
23 1. 1.
0.00 35.000
0.03 34.739
0.05 34.291
0.08 33.284
0.10 32.450
0.12 31.535
0.15 30.095
0.18 28.664
0.20 27.749
0.22 26.876
0.25 25.658
0.28 24.545
0.30 23.857
```

```
0.32 23.206
0.35 22.288
0.38 21.425
0.40 20.874
0.42 20.338
0.45 19.558
0.48 18.801
0.50 18.307
0.55 17.107
0.60 15.958
23 1. 1.
0.00 6.000
0.03 5.907
0.05 5.749
0.08 5.396
0.10 5.107
0.12 4.794
0.15 4.311
0.18 3.847
0.20 3.560
0.22 3.297
0.25 2.949
0.28 2.658
0.30 2.494
0.32 2.351
0.35 2.171
0.38 2.028
0.40 1.948
0.42 1.880
0.45 1.794
0.48 1.725
0.50 1.685
0.55 1.603
0.60 1.537
1 2 1
5. 1 0 1 1
1 1 1 1
2 1 1 1
1.0 0.001
9. 9. 9.
2
1000
100000 50000
100000 0.40 2
10
10
```

I am not a specialist of fullerene derivatives and don't know if these results are quite convincing. When trying to add more data than the first 50hkl, the R_F value increases, which is not really surprising, if one considers the approximation applied with this C_{60} sphere description.

About interatomic distances, this is not something very clear in disordered compounds (!), but at least among them, one should recognize the usual ones. You cannot expect a C_{60} highly disordered and Br atoms at precise places. Br atoms are necessarily disordered too. And if some Br-Br distances are not good, you may just think that the Br atoms are never really there together. Well, the theoretical distances between Br and the C_{60} center should be of the order

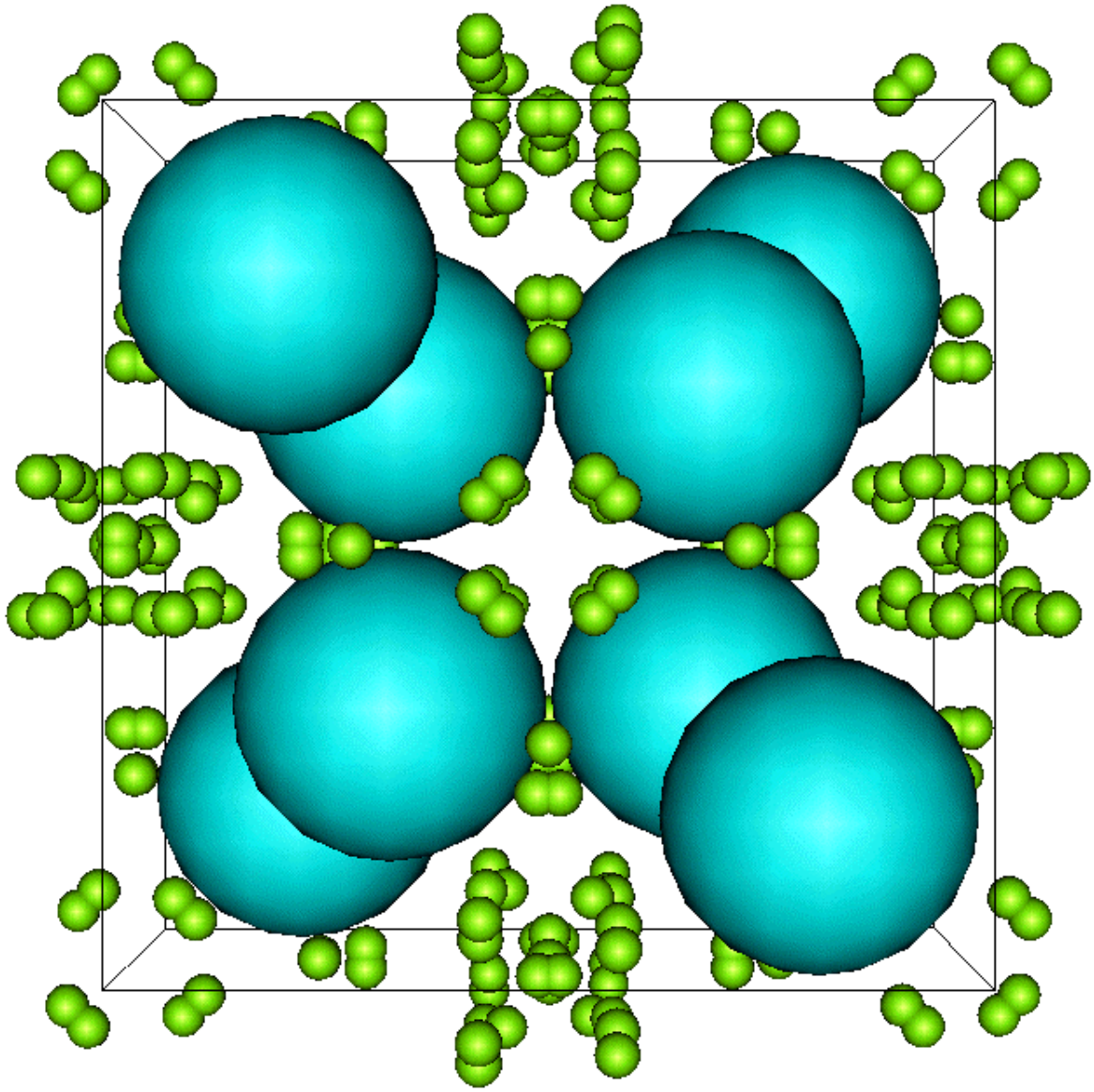
of 6.60-6.75 A. And the Br-Br distances are estimated to be near of 3.35. The current model gives first distances (where Cm is the center of the C_{60} molecule) :

Cm- Br(1)	6.67487
Cm- Br (2)	6.71488

Br(1)-Br(2)	3.16880
-Br(2)	3.37673
- Br(1)	3.43917

The 3.38 A distances are what is needed... But the shortest Br-Br distances are excluded from the list.

Below is to what looks like the structure. The big blue spheres are the disordered C_{60} molecules, given here 3.5 A radius. The green spheres are the Br atoms distributed on several places which they could occupy…



Now, about the fits on the powder data. I recognize a problem due to the bad calculated intensities for the 3 first reflections of the pattern (excluded). However, this could be explained by the global scattering factor which is very high in that part and could be inadequate. The refinements were made by using FULLPROF which allows to take account of at C_{60} disoriented molecule. It is even possible to refine the sphere radius.

	x	y	z	B	Occup
C60	0.1877(8)	0.1877(8)	0.1877(8)	12.40(99)	360.0
Br1	0.5853(39)	0.9238(68)	0.9476(64)	12.40(99)	1.0
Br2	0.9965(447)	0.4964(223)	0.7797(27)	12.40(99)	1.0
C1	0.5161(252)	0.8829(***)	0.9633(***)	12.40(99)	1.0

The "occupancy" factor set to 360, if compared to 1 for Br is because the in-built global scattering factor for a sphere is scaled to f(000)=1., so that for one C it should be multiplied by 6, and again by 60 for C_{60}. So that the ratio 360/1 is here respected.

Such global scattering factor was used in a recent publication (J. Solid State Chem, 145, 1999, 471-478), very probably by using Fullprof (?? not clear in the text !).

The problem of the powder data fits is that they look good mainly because a few very intense reflections are well fitted. If you zoom on the large angle part (30-60° 2-theta on the rotating anode data), the fit appears not that good. The above paper in JSSC presents fits on powder data in very small angular ranges, if I remember well

I trust more the single crystal data fit at RF=11% on the first 50 hkl in I-43m than the fit at RB=7.5% in the 12-60° 2theta range of the Rigaku powder data.

You may want to know how is calculated the global scattering factor for a sphere in Fullprof. This is in clear in the Fdum1.for part of the source : simply SinQr/Qr as below. There are other possibilities which I did not tested.

```
c-----------------------------------Simple 0-order Bessel function
      IF(LABELFF.EQ.'SPHS') THEN
c-----------------------------------Spherical surface
      r= XL(Ipiof,19)
      scatt= XL(Ipiof,20)
      qr=q*r
      FFX=scatt*sin(qr)/qr
      if(MAXS.eq.0) return
      if(LP(ipiof,19).ne.0) DXIr(19)=(cos(qr)/qr-ffx/r)*scatt
      if(LP(ipiof,20).ne.0) DXIr(20)=sin(qr)/qr
c-
```

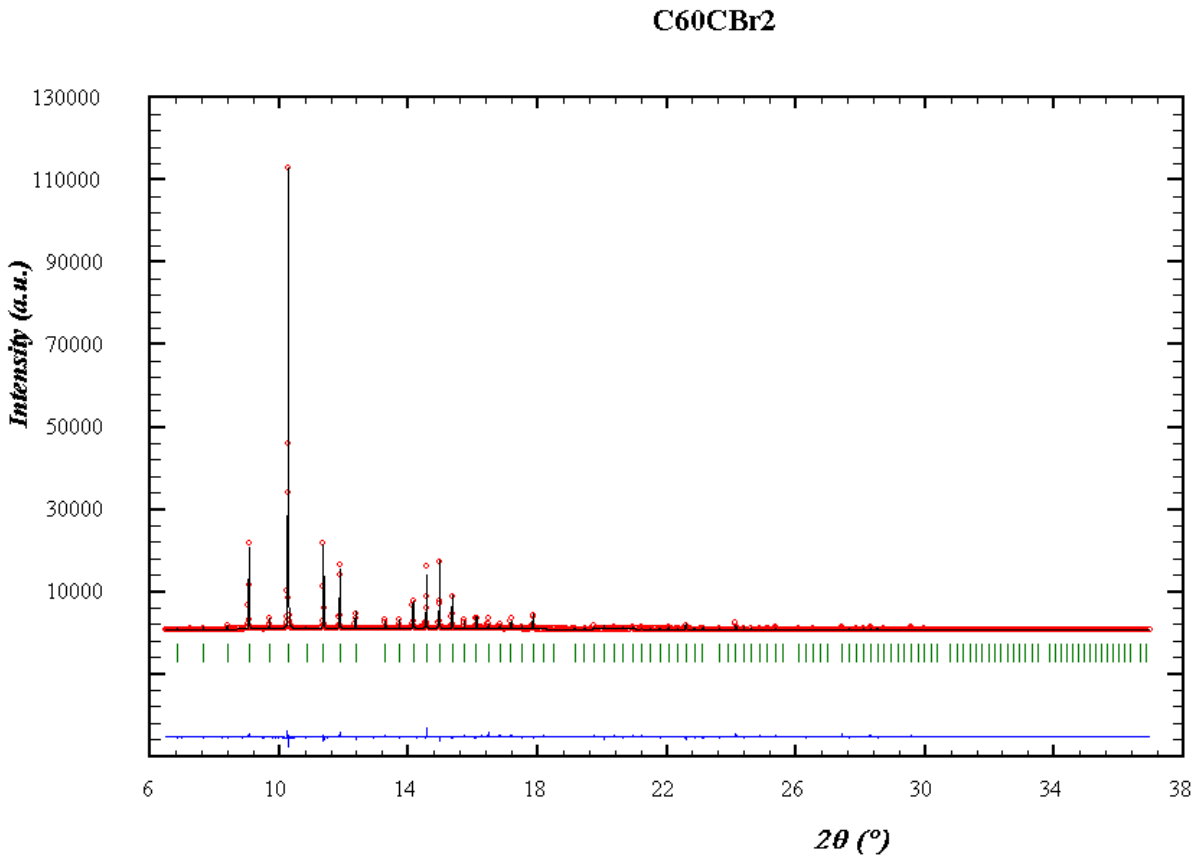
FULLPROF refinement results :

Synchrotron data :

```
        ******************************************************
        ** PROGRAM FULLPROF.98 (Version 0.2  - Mar98-LLB JRC) **
        ******************************************************
           Rietveld, Profile Matching & Integrated Intensity
                Refinement of X-ray and/or Neutron Data


    Date: 15/12/1999  Time: 16:15:12.180

 => PCR file code: I-43mS
 => DAT file code: I-43mS
 => Title:   C60CBr2

 ==> CONDITIONS OF THIS RUN:

 => Global Refinement of X-ray powder diffraction data
    Bragg-Brentano or Debye-Scherrer geometry
 => The    5th default profile function was selected
 => Data supplied in free format
 => Wavelengths:  0.79764 0.79764
 => Cos(Monochromator angle)=   0.0000
 => Absorption correction (muR-eff):   0.0000
 => Base of peaks: 2.0*HW*    9.00
 ==> Angular range, step and number of points:
     2Thmin:   1.5000  2Thmax:  36.9700  Step:   0.0100  No. of points:   3548
 => Crystal Structure Refinement for phase: 1
 => Scor: 1.7292

 ==> RESULTS OF REFINEMENT:


 => No. of fitted parameters:   17


------------------------------------------------------------------------------
 => Phase No.  1 C60CBr2                                  I -4 3 M
------------------------------------------------------------------------------

 =>  No. of reflections:    205


 ==> ATOM PARAMETERS:

  Name    x      sx       y      sy       z       sz       B    sB  occ.  socc.

 C1    0.18806(  12)  0.18806(  12)  0.18806(  12) 16.735(306)  3.600(  0)
 BR1   0.56199(  79)  0.92729(  61)  0.92729(  61) 16.735(306)  0.010(  0)
 BR2   0.00000(   0)  0.50000(   0)  0.78222(  59) 16.735(306)  0.010(  0)
 C2    0.59314( 636)  0.89950( 565)  0.96153( 659) 16.735(306)  0.010(  0)

 =>Form-Factor Parameters:

Atom        f1        f2        f3        f4        f5        f6        f7
           sf1       sf2       sf3       sf4       sf5       sf6       sf7
            f8        f9       f10       f11       f12       f13       f14
           sf8       sf9      sf10      sf11      sf12      sf13      sf14
 C1     3.51906   1.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00309   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000

 BR1    0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000

 BR2    0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000

 C2     0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
```

```
==> PROFILE PARAMETERS:

=> Cell parameters      :
                           18.88342   0.00049
                           18.88342   0.00049
                           18.88342   0.00049
                           90.00000   0.00000
                           90.00000   0.00000
                           90.00000   0.00000

=> overall scale factor :    0.001697654   0.000031420
=> Eta(p-v) or m(p-vii) :    0.41267   0.01282
=> Overall tem. factor  :    0.00000   0.00000
=> Halfwidth parameters :    0.00200   0.00000
                           -0.00001   0.00016
                            0.00035   0.00002
=> Preferred orientation:    1.00000   0.00000
                            0.00000   0.00000
=> Asymmetry parameters :    0.05989   0.00423
                           -0.00371   0.00054
                            0.00000   0.00000
                            0.00000   0.00000
=> X and y parameters   :    0.00000   0.00000
                            0.00000   0.00000
=> Strain parameters    :    0.00000   0.00000
                            0.00000   0.00000
                            0.00000   0.00000
=> Size   parameters    :    0.00000   0.00000
                            0.00000   0.00000

==> GLOBAL PARAMETERS:

=> Zero-point:   0.0012  0.0005
=> Cos( theta)-shift parameter :   0.0000  0.0000
=> Sin(2theta)-shift parameter :   0.0000  0.0000

==> RELIABILITY FACTORS WITH ALL NON-EXCLUDED POINTS:

=> Cycle: 20 => MaxCycle: 20
=> N-P+C:  3029
=> Rp: 4.13     Rwp: 6.25     Rexp:    3.07 Chi2:  4.14      L.S. refinement
=> Conventional Rietveld R-factors ==>
=> Rp: 24.7     Rwp: 16.9     Rexp:    8.32 Chi2:  4.14
=> Deviance: 0.134E+05     Dev*  :  4.434
=> DW-Stat.:    1.0060     DW-exp:     1.8986
=> N-sigma of the GoF:  122.007

==> RELIABILITY FACTORS FOR POINTS WITH BRAGG CONTRIBUTIONS:

=> N-P+C:  2637
=> Rp: 4.23     Rwp: 6.46     Rexp:    3.05 Chi2:  4.47      L.S. refinement
=> Conventional Rietveld R-factors ==>
=> Rp: 22.2     Rwp: 16.4     Rexp:    7.77 Chi2:  4.47
=> Deviance: 0.127E+05     Dev*  :  4.818
=> DW-Stat.:    1.0688     DW-exp:     1.8922
=> N-sigma of the GoF:      126.031
=> Phase:  1
=> Bragg R-factor:  10.1       Vol: 6733.520( 0.304)  Fract(%):    0.00( 0.00)
=> Rf-factor= 29.1             ATZ:    0.000          Brindley:  1.0000

=> Run finished at:     Date: 15/12/1999  Time: 16:15:21.520
```

Plot of the fit :



Rigaku data – I-43m :

```
        ******************************************************
        ** PROGRAM FULLPROF.98 (Version 0.2  - Mar98-LLB JRC) **
        ******************************************************
           Rietveld, Profile Matching & Integrated Intensity
                Refinement of X-ray and/or Neutron Data


   Date: 15/12/1999  Time: 16:39:39.290

=> PCR file code: I-43m
=> DAT file code: I-43m
=> Title:   C60CBr2

==> CONDITIONS OF THIS RUN:

=> Global Refinement of X-ray powder diffraction data
   Bragg-Brentano or Debye-Scherrer geometry
=> The    5th default profile function was selected
=> Data supplied in free format
=> Wavelengths:  1.54056 1.54439
=> Cos(Monochromator angle)=   0.7998
=> Absorption correction (muR-eff):   0.0000
=> Base of peaks: 2.0*HW*   12.00
==> Angular range, step and number of points:
    2Thmin:   5.0200  2Thmax:  89.9800  Step:   0.0200  No. of points:   4249
=> Crystal Structure Refinement for phase: 1
=> Scor: 3.2349

==> RESULTS OF REFINEMENT:


=> No. of fitted parameters:   18
```

=> Phase No. 1 C60CBr2 I -4 3 M

=> No. of reflections: 610

==> ATOM PARAMETERS:

Name	x	sx	y	sy	z	sz	B	sB	occ.	socc.
C1	0.18783	11	0.18783	11	0.18783	11	16.637	220	3.600	0
BR1	0.56297	62	0.92627	47	0.92627	47	16.637	220	0.010	0
BR2	0.00000	0	0.50000	0	0.78381	65	16.637	220	0.010	0
C2	0.60861	543	0.86365	499	0.97673	564	16.637	220	0.010	0

=>Form-Factor Parameters:

Atom	f1	f2	f3	f4	f5	f6	f7
	sf1	sf2	sf3	sf4	sf5	sf6	sf7
	f8	f9	f10	f11	f12	f13	f14
	sf8	sf9	sf10	sf11	sf12	sf13	sf14
C1	3.49866	1.00000	0.00000	0.00000	0.00000	0.00000	0.00000
	0.00301	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
BR1	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
BR2	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
C2	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000

==> PROFILE PARAMETERS:

=> Cell parameters	:	18.87332 0.00127
		18.87332 0.00127
		18.87332 0.00127
		90.00000 0.00000
		90.00000 0.00000
		90.00000 0.00000
=> overall scale factor	:	0.004435983 0.000036678
=> Eta(p-v) or m(p-vii)	:	0.26108 0.00721
=> Overall tem. factor	:	0.00000 0.00000
=> Halfwidth parameters	:	0.08000 0.00000
		-0.05742 0.00435
		0.03080 0.00085
=> Preferred orientation	:	1.00000 0.00000
		0.00000 0.00000
=> Asymmetry parameters	:	0.05160 0.00590
		0.06331 0.00111
		0.00000 0.00000
		0.00000 0.00000
=> X and y parameters	:	0.00000 0.00000
		0.00000 0.00000
=> Strain parameters	:	0.00000 0.00000
		0.00000 0.00000
		0.00000 0.00000
=> Size parameters	:	0.00000 0.00000
		0.00000 0.00000

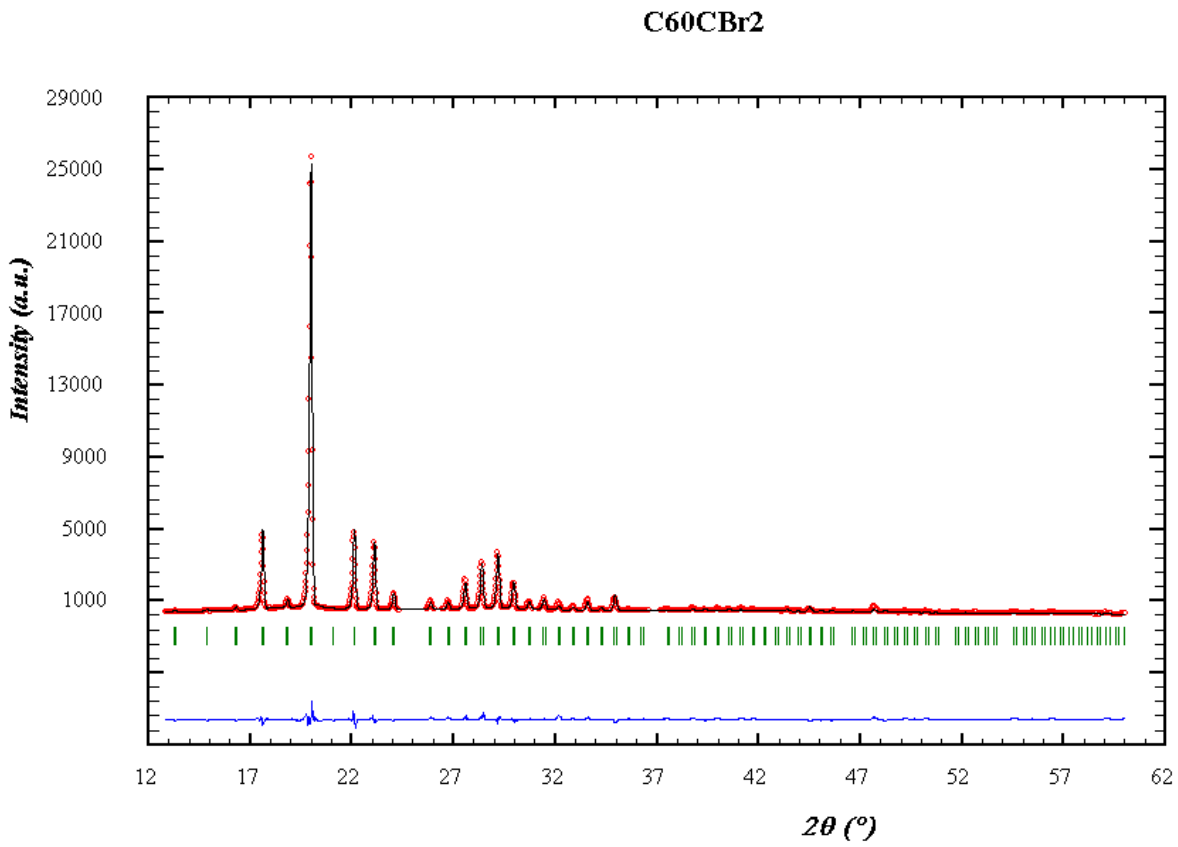
==> GLOBAL PARAMETERS:

=> Zero-point: -0.0449 0.0024

=> Cos(theta)-shift parameter : 0.0000 0.0000

```
=> Sin(2theta)-shift parameter :   0.0000  0.0000

==> RELIABILITY FACTORS WITH ALL NON-EXCLUDED POINTS:

=> Cycle:  8 => MaxCycle: 20
=> N-P+C:  2217
=> Rp: 4.13     Rwp: 5.83     Rexp:    3.89 Chi2:  2.24      L.S. refinement
=> Conventional Rietveld R-factors ==>
=> Rp: 12.8     Rwp: 11.9     Rexp:    7.93 Chi2:  2.24
=> Deviance: 0.510E+04     Dev*  :  2.297
=> DW-Stat.:    0.1638     DW-exp:     1.8847
=> N-sigma of the GoF:   41.356

==> RELIABILITY FACTORS FOR POINTS WITH BRAGG CONTRIBUTIONS:

=> N-P+C:  2217
=> Rp: 4.13     Rwp: 5.83     Rexp:    3.89 Chi2:  2.24      L.S. refinement
=> Conventional Rietveld R-factors ==>
=> Rp: 12.8     Rwp: 11.9     Rexp:    7.93 Chi2:  2.24
=> Deviance: 0.510E+04     Dev*  :  2.297
=> DW-Stat.:    0.1638     DW-exp:     1.8847
=> N-sigma of the GoF:      41.356
=> Phase:  1
=> Bragg R-factor:  7.53       Vol: 6722.720( 0.786)  Fract(%):    0.00( 0.00)
=> Rf-factor= 19.8             ATZ:    0.000          Brindley:  1.0000

=> Run finished at:     Date: 15/12/1999  Time: 16:39:53.410
```

Plot (note - more impurities in that sample, excluded zones) :

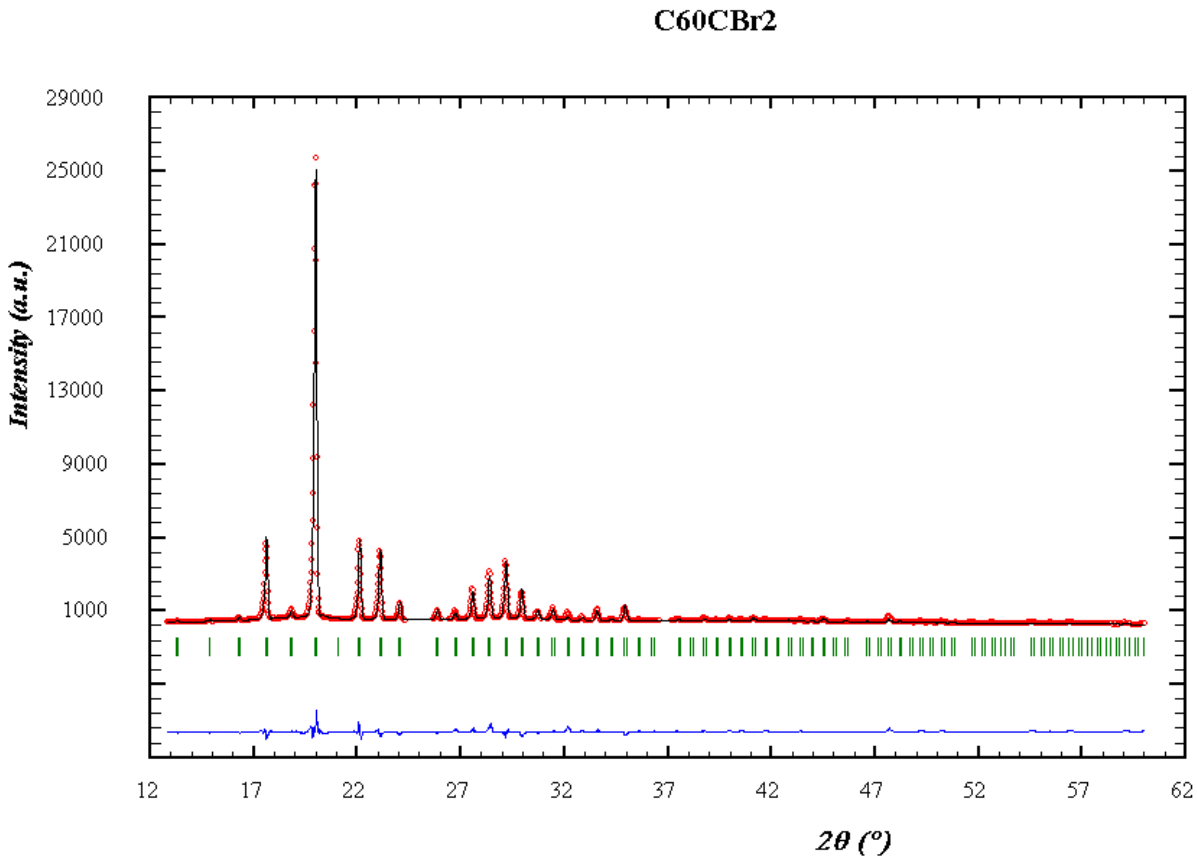


Rigaku data – I23 space group :

```
        ******************************************************
        ** PROGRAM FULLPROF.98 (Version 0.2  - Mar98-LLB JRC) **
        ******************************************************
           Rietveld, Profile Matching & Integrated Intensity
                Refinement of X-ray and/or Neutron Data


    Date: 15/12/1999  Time: 16:36:36.560

 => PCR file code: I23
 => DAT file code: I23
 => Title:   C60CBr2

 ==> CONDITIONS OF THIS RUN:

 => Global Refinement of X-ray powder diffraction data
    Bragg-Brentano or Debye-Scherrer geometry
 => The    5th default profile function was selected
 => Data supplied in free format
 => Wavelengths:  1.54056 1.54439
 => Cos(Monochromator angle)=   0.7998
 => Absorption correction (muR-eff):   0.0000
 => Base of peaks: 2.0*HW*   12.00
 ==> Angular range, step and number of points:
     2Thmin:   5.0200  2Thmax:  89.9800  Step:   0.0200  No. of points:   4249
 => Crystal Structure Refinement for phase: 1
 => Scor: 3.2946

 ==> RESULTS OF REFINEMENT:


 => No. of fitted parameters:   19


------------------------------------------------------------------------------
 => Phase No.  1 C60CBr2                                  I 2 3
------------------------------------------------------------------------------

 =>  No. of reflections:   1035


 ==> ATOM PARAMETERS:

  Name    x     sx       y     sy       z      sz      B   sB  occ.  socc.

 C1    0.18734(  14)  0.18734(  14)  0.18734(  14) 19.367(218)  3.600(  0)
 BR1   0.56189(  61)  0.93729(  84)  0.90828( 102) 19.367(218)  0.012(  0)
 BR2   0.00000(   0)  0.50000(   0)  0.76552( 183) 19.367(218)  0.008(  0)
 C2    0.55391( 605)  0.84598( 754)  0.99141( 854) 19.367(218)  0.010(  0)

 =>Form-Factor Parameters:

Atom        f1        f2        f3        f4        f5        f6        f7
           sf1       sf2       sf3       sf4       sf5       sf6       sf7
            f8        f9       f10       f11       f12       f13       f14
           sf8       sf9      sf10      sf11      sf12      sf13      sf14
 C1     3.50534   1.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00331   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000

 BR1    0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000

 BR2    0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000

 C2     0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
```

```
==> PROFILE PARAMETERS:

=> Cell parameters      :
                           18.87405   0.00134
                           18.87405   0.00134
                           18.87405   0.00134
                           90.00000   0.00000
                           90.00000   0.00000
                           90.00000   0.00000

=> overall scale factor :    0.018921617   0.000164389
=> Eta(p-v) or m(p-vii) :    0.26532   0.00745
=> Overall tem. factor  :    0.00000   0.00000
=> Halfwidth parameters :    0.08000   0.00000
                            -0.05556   0.00455
                             0.03033   0.00089
=> Preferred orientation:    1.00000   0.00000
                             0.00000   0.00000
=> Asymmetry parameters :    0.05327   0.00611
                             0.06335   0.00114
                             0.00000   0.00000
                             0.00000   0.00000
=> X and y parameters   :    0.00000   0.00000
                             0.00000   0.00000
=> Strain parameters    :    0.00000   0.00000
                             0.00000   0.00000
                             0.00000   0.00000
=> Size   parameters    :    0.00000   0.00000
                             0.00000   0.00000

==> GLOBAL PARAMETERS:

=> Zero-point:  -0.0436  0.0025
=> Cos( theta)-shift parameter :   0.0000  0.0000
=> Sin(2theta)-shift parameter :   0.0000  0.0000

==> RELIABILITY FACTORS WITH ALL NON-EXCLUDED POINTS:

=> Cycle: 20 => MaxCycle: 20
=> N-P+C:  2216
=> Rp: 4.26     Rwp: 5.99     Rexp:    3.89 Chi2:  2.37      L.S. refinement
=> Conventional Rietveld R-factors ==>
=> Rp: 13.2     Rwp: 12.2     Rexp:    7.92 Chi2:  2.37
=> Deviance: 0.546E+04     Dev*  :  2.464
=> DW-Stat.:    0.1549     DW-exp:     1.8856
=> N-sigma of the GoF:   45.508

==> RELIABILITY FACTORS FOR POINTS WITH BRAGG CONTRIBUTIONS:

=> N-P+C:  2216
=> Rp: 4.26     Rwp: 5.99     Rexp:    3.89 Chi2:  2.37      L.S. refinement
=> Conventional Rietveld R-factors ==>
=> Rp: 13.2     Rwp: 12.2     Rexp:    7.92 Chi2:  2.37
=> Deviance: 0.546E+04     Dev*  :  2.464
=> DW-Stat.:    0.1549     DW-exp:     1.8856
=> N-sigma of the GoF:       45.508
=> Phase:  1
=> Bragg R-factor:  8.23       Vol: 6723.495( 0.824)  Fract(%):    0.00( 0.00)
=> Rf-factor= 22.1             ATZ:    0.000          Brindley:  1.0000

=> Run finished at:     Date: 15/12/1999  Time: 16:37:31.370
```

Plot :



Single crystal data – I-43m with FULLPROF:

```
         ******************************************************
         ** PROGRAM FULLPROF.98 (Version 0.2  - Mar98-LLB JRC) **
         ******************************************************
            Rietveld, Profile Matching & Integrated Intensity
                 Refinement of X-ray and/or Neutron Data


    Date: 14/12/1999  Time: 23:15:24.850

 => PCR file code: C61Br2
 => DAT file code: C61Br2
 => Title: Single crystal data of C61Br2 (X-ray, MoKa)

 ==> CONDITIONS OF THIS RUN:

 => Refinement of X-ray integrated intensity data
 => Integrated intensities as observations for phase: 1
 => Crystal Structure Refinement for phase: 1

 ==> RESULTS OF REFINEMENT:


 => No. of fitted parameters:   13


------------------------------------------------------------------------------
 => Phase No.  1  C61Br2                                 I -4 3 m
------------------------------------------------------------------------------
```

```
 =>  No. of reflections:     47


 ==> ATOM PARAMETERS:

  Name    x      sx       y      sy       z      sz      B    sB  occ.  socc.

 C1    0.18768(  78)  0.18768(  78)  0.18768(  78) 12.404(999)  3.600(  0)
 BR1   0.58535( 391)  0.92376( 687)  0.94756( 642) 12.404(999)  0.010(  0)
 BR2   0.99652(4476)  0.49639(2231)  0.77973( 267) 12.404(999)  0.010(  0)
 C2    0.51615(2526)  0.88287(****)  0.96335(****) 12.404(999)  0.010(  0)

 =>Form-Factor Parameters:

Atom        f1        f2        f3        f4        f5        f6        f7
           sf1       sf2       sf3       sf4       sf5       sf6       sf7
            f8        f9       f10       f11       f12       f13       f14
           sf8       sf9      sf10      sf11      sf12      sf13      sf14
 C1     3.50253   1.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.01681   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000

 BR1    0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000

 BR2    0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000

 C2     0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
        0.00000   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000


 ==> OVERALL PARAMETERS:

 => Scale factors        :
                            3.022934680   0.316073149
                            0.000000000   0.000000000
                            0.000000000   0.000000000
                            0.000000000   0.000000000
                            0.000000000   0.000000000
                            0.000000000   0.000000000

 => Extinction parameters:
                            0.000000   0.000000
                            0.000000   0.000000
                            0.000000   0.000000
                            0.000000   0.000000
                            0.000000   0.000000
                            0.000000   0.000000
                            0.000000   0.000000


 => Phase:  1
 =>    RF2 -factor :  12.4
 =>    RF2w-factor :  19.4
 =>    RF  -factor :  11.0
 =>    Chi2(Intens):  67.0

 => Run finished at:     Date: 14/12/1999  Time: 23:15:26.110
```

Final attempt with an ordered model with SHELX :

Below is the output file from SHELXL93, with an hypothesis of merohedral twinning in I23 by hkl and kh-l overlapping which would simulate the m3m Laue class R1=16% with an OMIT 6 (226 hkl) for 92 refined parameters (!!) and refinement of the C_{60} radius (an H atom

is placed at xxx and C-H distances are constrained to be near of 3.55 Angstroms). You may try to play with this model. Many C-C distances are not good. Of course, Fourier difference in case of twinning in quite meaningless. I have similar solution at R1=22% in I-43m. Several supplementary atoms are proposed (is $C_{60}CBr_2$ the right formula after all ??).

```
++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
+  SHELXL-93 - CRYSTAL STRUCTURE REFINEMENT - MSDOS 32-BIT VERSION  +
+  wini23                       started at 16:41:56 on 16-Dec-1999  +
++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++

TITL C60CBr2 Twin ??  I 2 3
CELL   0.7107  18.90 18.90 18.90 90.000  90.000  90.000
ZERR 2 0.009 0.009 0.009 0.0 0.0 0.0
LATT  -2
SYMM  X, -Y, -Z
SYMM  -X, +Y, -Z
SYMM  -X, -Y, +Z
SYMM  +Y, +Z, X
SYMM  -Y, -Z, X
SYMM  +Y, -Z, -X
SYMM  -Y, +Z, -X
SYMM  +Z, X, +Y
SYMM  -Z, X, -Y
SYMM  -Z, -X, +Y
SYMM  +Z, -X, -Y
SFAC C BR O H
UNIT 480 16 24 8

V =     6751.27     F(000) =    3640.0     Mu =   2.46 mm-1      Cell Wt =     7435.42    Rho =  1.829

TWIN 0 1 0  1 0 0  0 0 -1
OMIT 6
EQIV $1   x, -y, -z+1
EQIV $2   -x, y, -z+1
EQIV $3   y, -z+1, -x+1
EQIV $4   -z+1, x, -y+1
L.S. 10
BOND
FMAP 2
PLAN 20
WGHT    0.200000
FVAR       0.03917   0.02576   0.22804   0.47565   3.53898
H1      4   0.31192   0.31192   0.68808  10.33333  10.05000
C2      1   0.23255   0.14653   0.72470  11.00000  21.00000
C3      1   0.18343   0.18889   0.74305  11.00000  21.00000
C4      1   0.44697   0.26099   0.57115  11.00000  21.00000
C5      1   0.40607   0.27138   0.53033  11.00000  21.00000
C6      1   0.44159   0.17545   0.67876  11.00000  21.00000
C7      1   0.32797   0.15846   0.58208  11.00000  21.00000
C8      1   0.37905   0.19762   0.55396  11.00000  21.00000
C9      1   0.21095   0.22167   0.55768  11.00000  21.00000
C10     1   0.28444   0.12894   0.65193  11.00000  21.00000
C11     1   0.22800   0.18093   0.58589  11.00000  21.00000
C12     1   0.12791   0.29720   0.65219  11.00000  21.00000
C13     1   0.49330   0.29356   0.65191  11.00000  21.00000
C14     1   0.32649   0.39630   0.52014  11.00000  21.00000
C15     1   0.15316   0.21903   0.66264  11.00000  21.00000
C16     1   0.40781   0.16286   0.62653  11.00000  21.00000
C17     1   0.39925   0.35760   0.52887  11.00000  21.00000
C18     1   0.21812   0.26807   0.53182  11.00000  21.00000
C19     1   0.32925   0.27471   0.50742  11.00000  21.00000
C20     1   0.26927   0.23345   0.52294  11.00000  21.00000
C21     1   0.22909   0.14844   0.64728  11.00000  21.00000
BR2     2   0.59353   0.09258   0.57930  40.66667  31.00000
BR1     2   0.22652   0.00000   0.50000 -40.66667  31.00000
C22     1   0.62576   0.03032   0.55739  11.00000   0.03150
C23     1   0.41981   0.41981   0.58019  10.33333   0.06222
C24     1   0.54515   0.08670   0.55904  11.00000   0.03524
C25     1   0.19233   0.22564   0.62028  11.00000   0.04319
C26     1   0.55615   0.02885   0.56168  11.00000   0.21169
C27     1   0.25305   0.08102   0.60765  11.00000   0.16310
DFIX 51 H1 C2 H1 C3 H1 C4 H1 C5 H1 C6 H1 C7 H1 C8 H1 C9 H1 C10
DFIX 51 H1 C11 H1 C12 H1 C13 H1 C14 H1 C15
DFIX 51 H1 C16 H1 C17 H1 C18 H1 C19 H1 C20 H1 C21 H1 C23
LIST 1 1
HKLF 4


Covalent radii and connectivity table for  C60CBr2 Twin ??  I 2 3

C    0.770
BR   1.140
```

```
O    0.660
H    0.320

C2 - C3 C21 C10 C3_$2
C3 - C2 C15 C2_$1 C3_$2 C3_$1
C4 - C5 C14_$2 C8 C13
C5 - C4 C19 C8 C17
C6 - C16 C18_$2 C9_$2 C12_$2
C7 - C8 C10 C16 C11
C8 - C7 C5 C16 C4 C19
C9 - C11 C18 C25 C20 C6_$1
C10 - C21 C27 C12_$2 C7 C2 C11
C11 - C9 C25 C21 C20 C10 C7 C18 C27
C12 - C10_$1 C15 C25 C16_$1 C6_$1
C13 - C14_$2 C19_$2 C4
C14 - C13_$1 C4_$1 C17
C15 - C25 C12 C3 C21
C16 - C6 C8 C7 C12_$2
C17 - C14 C23 C5
C18 - C9 C20 C6_$1 C25 C11
C19 - C20 C5 C13_$1 C8
C20 - C18 C9 C19 C11
C21 - C10 C11 C2 C27 C25 C15
Br2 - C24 C22 C26
Br1 - no bonds found
C22 - C26 Br2 C24
C23 - C17_$2 C17 C17_$1
C24 - Br2 C26 C22
C25 - C15 C9 C11 C21 C12 C18
C26 - C24 C22 Br2
C27 - C10 C21 C11


Operators for generating equivalent atoms:

$1   y, -z+1, -x+1
$2   -z+1, x, -y+1



Summary of restraints for  C60CBr2 Twin ??  I 2 3


Distance, sigma and atom pairs for DFIX

   51.0000  0.030   H1 - C2   H1 - C3   H1 - C4   H1 - C5   H1 - C6   H1 - C7   H1 - C8   H1 - C9   H1 -
C10   H1 - C11
                    H1 - C12   H1 - C13   H1 - C14   H1 - C15   H1 - C16   H1 - C17   H1 - C18   H1 - C19
H1 - C20
                    H1 - C21   H1 - C23


    614  Reflections read, of which     0  rejected

  1 =< h =< 22,      0 =< k =< 15,      0 =< l =< 12,   Max. 2-theta =   49.62

      0  Systematic absence violations

      0  Inconsistent equivalents

    614  Unique reflections, of which    388  suppressed

R(int) = 0.0000     R(sigma) = 0.1005      Friedel opposites not merged

Maximum memory for data reduction =  2613 /   10323


Special position constraints for H1
y =  1.0 * x + 0.0000    z = -1.0 * x + 1.0000    sof = 0.33333
Input constraints retained (at least in part) for  sof and Uij

Special position constraints for Br1
y =  0.0000              z =  0.5000              sof = 0.50000
Input constraints retained (at least in part) for  sof and Uij

Special position constraints for C23
y =  1.0 * x + 0.0000    z = -1.0 * x + 1.0000    sof = 0.33333
Input constraints retained (at least in part) for  sof


  ** Cell contents from UNIT instruction and atom list do not agree **


Unit-cell contents from UNIT instruction and atom list resp.

C       480.00    608.00
BR       16.00     16.00
O        24.00      0.00
```

```
H         8.00      8.00

      0.7 seconds elapsed time


Least-squares cycle   1      Maximum vector length =  511      Memory required =   2850 /  120970

wR2 =  0.3832 before cycle   1 for    226 data and    92 /    92 parameters


Summary of restraints applied in cycle    1

                  DFIX-     DFIX+   SAME/SADI    CHIV      FLAT      DELU      SIMU      ISOR      SUMP

       Number        0.       21.        0.        0.        0.        0.        0.        0.        0.

Mean(w*del^2)     0.000     0.421     0.000     0.000     0.000     0.000     0.000     0.000     0.000

Mean sigma        0.000     0.030     0.000     0.000     0.000     0.000     0.000     0.000     0.000

Mean deviation    0.000     0.016     0.000     0.000     0.000     0.000     0.000     0.000     0.000


GooF = S =     2.071;     Restrained GooF =      1.963  for     21 restraints

Weight = 1 / [ sigma^2(Fo^2) + ( 0.2000 * P )^2 +   0.00 * P ]   where  P = ( Max ( Fo^2, 0 ) + 2 * Fc^2 )
/ 3


    N      value        esd    shift/esd  parameter

    1     0.03927     0.00156     0.065    OSF
    2     0.02623     0.00416     0.113   FVAR  2
    3     0.22831     0.02175     0.012   FVAR  3
    4     0.45862     0.02663    -0.640   FVAR  4
    5     3.54565     0.01584     0.421   FVAR  5

Mean shift/esd =   0.135    Maximum =   0.677 for   y  C26

Max. shift = 0.155 A for C26      Max. dU =-0.011 for C23

      0.4 seconds elapsed time

Least-squares cycle  10      Maximum vector length =  511      Memory required =   2850 /  120970

wR2 =  0.3746 before cycle  10 for    226 data and    92 /    92 parameters


Summary of restraints applied in cycle   10

                  DFIX-     DFIX+   SAME/SADI    CHIV      FLAT      DELU      SIMU      ISOR      SUMP

       Number        0.       21.        0.        0.        0.        0.        0.        0.        0.

Mean(w*del^2)     0.000     0.499     0.000     0.000     0.000     0.000     0.000     0.000     0.000

Mean sigma        0.000     0.030     0.000     0.000     0.000     0.000     0.000     0.000     0.000

Mean deviation    0.000     0.018     0.000     0.000     0.000     0.000     0.000     0.000     0.000


GooF = S =     2.009;     Restrained GooF =      1.911  for     21 restraints

Weight = 1 / [ sigma^2(Fo^2) + ( 0.2000 * P )^2 +   0.00 * P ]   where  P = ( Max ( Fo^2, 0 ) + 2 * Fc^2 )
/ 3


    N      value        esd    shift/esd  parameter

    1     0.03963     0.00155     0.032    OSF
    2     0.02680     0.00407     0.025   FVAR  2
    3     0.23980     0.02235    -0.009   FVAR  3
    4     0.45214     0.02999     0.092   FVAR  4
    5     3.54533     0.01594     0.001   FVAR  5

Mean shift/esd =   0.025    Maximum =   0.223 for   z  C26

Max. shift = 0.074 A for C26      Max. dU =-0.001 for C26


Largest correlation matrix elements

    0.825 FVAR 2 / OSF                    0.593 z C7 / y C7                     0.526 x C23 / x H1
    0.712 y C3 / x C3                     0.574 y C6 / x C6

      0.5 seconds elapsed time
```

C60CBr2 Twin ?? I 2 3

ATOM	x	y	z	sof	U11	U22	U33	U23	U13
U12	Ueq								
H1	0.31236	0.31236	0.68764	0.33333	0.05000				
	0.00079	0.00079	0.00079	0.00000	0.00000				
C2	0.23331	0.14663	0.72432	1.00000	0.02680				
	0.00311	0.00228	0.00351	0.00000	0.00407				
C3	0.18358	0.18914	0.74165	1.00000	0.02680				
	0.00383	0.00384	0.00305	0.00000	0.00407				
C4	0.44754	0.26079	0.57039	1.00000	0.02680				
	0.00270	0.00343	0.00278	0.00000	0.00407				
C5	0.40734	0.27101	0.53049	1.00000	0.02680				
	0.00329	0.00360	0.00243	0.00000	0.00407				
C6	0.44381	0.17656	0.67912	1.00000	0.02680				
	0.00283	0.00283	0.00359	0.00000	0.00407				
C7	0.32808	0.15945	0.58016	1.00000	0.02680				
	0.00347	0.00271	0.00331	0.00000	0.00407				
C8	0.37949	0.19784	0.55312	1.00000	0.02680				
	0.00340	0.00301	0.00301	0.00000	0.00407				
C9	0.21090	0.22177	0.55731	1.00000	0.02680				
	0.00315	0.00382	0.00285	0.00000	0.00407				
C10	0.28395	0.12896	0.65241	1.00000	0.02680				
	0.00352	0.00196	0.00396	0.00000	0.00407				
C11	0.22732	0.18131	0.58559	1.00000	0.02680				
	0.00365	0.00316	0.00303	0.00000	0.00407				
C12	0.12790	0.29701	0.65226	1.00000	0.02680				
	0.00198	0.00323	0.00408	0.00000	0.00407				
C13	0.49386	0.29442	0.65078	1.00000	0.02680				
	0.00217	0.00343	0.00426	0.00000	0.00407				
C14	0.32642	0.39749	0.51938	1.00000	0.02680				
	0.00329	0.00301	0.00218	0.00000	0.00407				
C15	0.15379	0.21846	0.66134	1.00000	0.02680				
	0.00264	0.00313	0.00387	0.00000	0.00407				
C16	0.40682	0.16188	0.62657	1.00000	0.02680				
	0.00286	0.00251	0.00318	0.00000	0.00407				
C17	0.40010	0.36068	0.52903	1.00000	0.02680				
	0.00294	0.00335	0.00245	0.00000	0.00407				
C18	0.21804	0.26867	0.53074	1.00000	0.02680				
	0.00334	0.00432	0.00259	0.00000	0.00407				
C19	0.32916	0.27399	0.50731	1.00000	0.02680				
	0.00354	0.00327	0.00199	0.00000	0.00407				
C20	0.26983	0.23260	0.52248	1.00000	0.02680				
	0.00362	0.00346	0.00230	0.00000	0.00407				
C21	0.22879	0.14886	0.64766	1.00000	0.02680				
	0.00327	0.00248	0.00358	0.00000	0.00407				
Br2	0.59302	0.09321	0.57745	0.30143	0.23980				
	0.00715	0.00526	0.00652	0.01999	0.02235				
Br1	0.22774	0.00000	0.50000	0.36524	0.23980				
	0.00319	0.00000	0.00000	0.01999	0.02235				
C22	0.62496	0.03112	0.55631	1.00000	0.03424				
	0.00420	0.00379	0.00395	0.00000	0.02189				
C23	0.42029	0.42029	0.57971	0.33333	0.04919				
	0.00140	0.00140	0.00140	0.00000	0.03342				
C24	0.54689	0.08627	0.55844	1.00000	0.04239				
	0.00471	0.00455	0.00403	0.00000	0.02103				
C25	0.19283	0.22562	0.62156	1.00000	0.04909				
	0.00415	0.00430	0.00444	0.00000	0.02378				
C26	0.55333	0.03193	0.58252	1.00000	0.26083				

	0.01395	0.01354	0.01632	0.00000	0.13165
C27	0.25432	0.08182	0.60856	1.00000	0.17181
	0.00883	0.00906	0.01017	0.00000	0.06637

Final Structure Factor Calculation for C60CBr2 Twin ?? I 2 3

Total number of l.s. parameters = 92 Maximum vector length = 511 Memory required = 2760 / 24535

wR2 = 0.3741 before cycle 11 for 226 data and 2 / 92 parameters

Summary of restraints applied in cycle 11

	DFIX-	DFIX+	SAME/SADI	CHIV	FLAT	DELU	SIMU	ISOR	SUMP
Number	0.	21.	0.	0.	0.	0.	0.	0.	0.
Mean(w*del^2)	0.000	0.509	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Mean sigma	0.000	0.030	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Mean deviation	0.000	0.018	0.000	0.000	0.000	0.000	0.000	0.000	0.000

GooF = S = 2.006; Restrained GooF = 1.909 for 21 restraints

Weight = 1 / [sigma^2(Fo^2) + (0.2000 * P)^2 + 0.00 * P] where P = (Max (Fo^2, 0) + 2 * Fc^2) / 3

R1 = 0.1622 for 226 Fo > 4.sigma(Fo) and 0.2654 for all 614 data
wR2 = 0.6502, GooF = S = 2.074, Restrained GooF = 2.038 for all data

Flack x parameter = 0.1080 with esd 0.8312
(expected values are 0 (within 3 esd's) for correct and +1 for inverted absolute structure)

Absolute structure cannot be determined reliably

0.8 seconds elapsed time

Analysis of variance for reflections employed in refinement K = Mean[Fo^2] / Mean[Fc^2] for group

Fc/Fc(max)	0.000	0.068	0.080	0.091	0.107	0.124	0.144	0.173	0.211	0.296	1.000
Number in group	25.	21.	22.	25.	21.	22.	23.	23.	22.	22.	
GooF	1.548	0.906	1.255	1.454	1.408	1.799	2.059	1.970	1.863	2.062	
K	1.766	1.240	1.176	1.064	0.938	1.044	1.153	1.062	0.957	1.468	

Resolution(A)	0.85	1.05	1.11	1.22	1.33	1.49	1.64	1.89	2.22	2.98	inf
Number in group	23.	23.	22.	24.	21.	23.	24.	21.	23.	22.	
GooF	1.367	1.388	1.184	1.313	1.516	2.330	1.555	1.585	1.945	2.172	
K	1.280	1.062	1.178	0.962	1.172	1.166	0.937	1.067	1.031	1.535	
R1	0.188	0.168	0.140	0.145	0.155	0.189	0.126	0.146	0.149	0.183	

Recommended weighting scheme: WGHT 0.3190 0.0000

Most Disagreeable Reflections (* if suppressed)

	h	k	l	Fo^2	Fc^2	Delta(F^2)/esd	Fc/Fc(max)	Resolution(A)
*	13	0	17	6.37	612.22	4.77	0.058	0.88
	5	0	11	20696.70	4377.57	4.65	0.154	1.56
*	12	12	12	375.72	11313.48	4.23	0.247	0.91
*	2	0	4	292.94	4248.55	4.22	0.152	4.23
*	8	2	10	566.77	8080.66	4.21	0.209	1.46
*	11	0	15	6.37	107.25	4.07	0.024	1.02
*	0	1	1	617.72	6150.51	3.87	0.182	13.36
*	7	5	10	764.19	6737.10	3.83	0.191	1.43
*	0	0	14	662.29	5122.96	3.66	0.166	1.35
*	4	6	6	649.56	4585.34	3.64	0.157	2.01
*	9	2	11	878.81	5904.75	3.63	0.179	1.32

*	7	0	11	910.65	5938.64	3.56	0.179	1.45
*	8	5	9	636.82	4156.94	3.51	0.150	1.45
*	3	2	13	592.24	3947.51	3.47	0.146	1.40
*	6	5	11	464.88	3488.78	3.44	0.137	1.40
*	5	3	12	770.55	4973.35	3.40	0.164	1.42
*	8	4	10	789.66	4493.00	3.31	0.156	1.41
*	5	1	12	611.35	3441.00	3.22	0.136	1.45
*	2	9	9	821.50	4098.61	3.21	0.149	1.47
*	6	2	12	796.03	3855.79	3.13	0.144	1.39
*	4	3	11	706.87	3606.84	3.06	0.140	1.56
*	6	6	10	528.56	2955.18	3.00	0.126	1.44
*	9	3	10	738.71	3312.40	3.00	0.134	1.37
*	8	3	11	706.87	3094.92	2.98	0.129	1.36
*	2	1	13	592.24	2919.95	2.96	0.126	1.43
*	10	8	14	522.19	3541.32	2.90	0.138	1.00
*	5	3	14	1381.90	5675.98	2.86	0.175	1.25
*	9	0	13	917.02	4141.65	2.85	0.150	1.20
*	3	3	14	1018.91	4082.40	2.82	0.149	1.29
	4	1	7	2012.36	6729.81	2.82	0.191	2.33
*	9	5	10	713.24	3302.22	2.79	0.134	1.32
*	9	8	11	668.66	3114.08	2.74	0.130	1.16
*	11	0	17	668.66	2906.97	2.74	0.125	0.93
*	8	6	14	585.88	2426.04	2.69	0.114	1.10
*	3	2	15	757.82	2845.04	2.67	0.124	1.23
*	7	5	12	993.44	3668.21	2.66	0.141	1.28
	0	3	3	390658.16	185082.81	2.62	1.000	4.45
*	5	4	7	509.46	1958.91	2.54	0.103	1.99
	7	2	9	8546.14	3789.61	2.53	0.143	1.63
*	7	6	11	783.29	2789.07	2.48	0.123	1.32
*	10	7	11	745.08	2689.97	2.45	0.121	1.15
	2	1	7	21486.36	10507.41	2.42	0.238	2.57
	2	2	2	6088.01	14935.60	2.38	0.284	5.46
*	10	5	11	897.92	3146.16	2.33	0.130	1.21
*	10	5	15	1063.49	3641.92	2.26	0.140	1.01
*	6	4	8	433.04	1632.82	2.25	0.094	1.75
*	8	5	15	592.24	2208.84	2.25	0.109	1.07
*	3	1	10	522.19	1742.87	2.24	0.097	1.80
	7	3	8	433.04	1185.34	2.15	0.080	1.71
*	8	6	12	566.77	2192.30	2.12	0.109	1.21

Bond lengths and angles

C2 -	Distance	Angles						
C3	1.279 (0.107)							
C21	1.452 (0.098)	101.19 (5.83)						
C10	1.695 (0.096)	138.06 (5.26)	40.45 (3.53)					
C3_$2	1.840 (0.113)	73.92 (5.61)	153.00 (2.84)	129.95 (4.04)				
	C2 -	C3	C21	C10				

C3 -	Distance	Angles						
C2	1.279 (0.107)							
C15	1.711 (0.088)	102.59 (5.30)						
C2_$1	1.840 (0.113)	154.53 (3.49)	79.36 (4.79)					
C3_$2	1.929 (0.098)	66.49 (6.79)	149.37 (1.99)	98.74 (4.32)				
C3_$1	1.929 (0.098)	125.00 (6.47)	116.63 (6.72)	39.59 (4.36)	60.00 (0.01)			
	C3 -	C2	C15	C2_$1	C3_$2			

C4 -	Distance	Angles						
C5	1.088 (0.088)							
C14_$2	1.516 (0.090)	114.85 (6.93)						
C8	1.782 (0.089)	59.17 (5.12)	156.53 (2.82)					
C13	1.865 (0.102)	146.31 (5.89)	37.00 (2.92)	135.68 (3.99)				
	C4 -	C5	C14_$2	C8				

C5 -	Distance	Angles						
C4	1.088 (0.088)							
C19	1.542 (0.098)	150.76 (4.78)						
C8	1.540 (0.090)	83.49 (6.29)	77.54 (4.31)					
C17	1.700 (0.097)	104.15 (6.30)	83.21 (4.78)	150.66 (3.78)				
	C5 -	C4	C19	C8				

C6 -	Distance	Angles						
C16	1.246 (0.087)							
C18_$2	1.349 (0.102)	155.87 (4.27)						
C9_$2	1.983 (0.103)	145.10 (4.35)	28.64 (3.72)					
C12_$2	2.085 (0.101)	65.52 (4.35)	114.12 (5.43)	86.02 (3.61)				
	C6 -	C16	C18_$2	C9_$2				

C7 -	Distance	Angles						
C8	1.316 (0.092)							
C10	1.701 (0.106)	149.40 (4.82)						
C16	1.728 (0.088)	63.04 (4.13)	91.36 (4.71)					
C11	1.951 (0.093)	128.62 (5.45)	63.32 (3.44)	143.91 (3.62)				
	C7 -	C8	C10	C16				

C8 -	Distance	Angles

```
C7          1.316 (0.092)
C5          1.540 (0.090)   148.79 (4.79)
C16         1.630 (0.086)    70.93 (4.38) 120.17 (5.07)
C4          1.782 (0.089)   146.07 (4.43)  37.33 (3.43)  83.89 (4.34)
C19         1.930 (0.091)   102.80 (4.79)  51.28 (3.85) 148.13 (2.63)  86.56 (3.74)
               C8 -          C7            C5            C16           C4

C9 -         Distance       Angles
C11         0.983 (0.078)
C18         1.028 (0.085)   153.52 (7.67)
C25         1.264 (0.099)    66.90 (5.90) 117.09 (7.42)
C20         1.310 (0.086)    97.27 (7.46)  60.53 (4.95) 134.74 (4.98)
C6_$1       1.983 (0.103)   147.56 (5.16)  38.99 (5.55)  82.38 (5.23)  97.20 (4.89)
               C9 -          C11           C18           C25           C20

C10 -        Distance       Angles
C21         1.112 (0.081)
C27         1.339 (0.183)    77.46 (8.17)
C12_$2      1.539 (0.080)   142.11 (7.32) 134.70 (8.47)
C7          1.701 (0.107)   106.28 (6.72)  86.20 (9.08)  96.83 (5.06)
C2          1.695 (0.096)    57.92 (5.19) 113.02 (8.78)  86.95 (5.42) 148.61 (3.16)
C11         1.929 (0.100)    41.69 (4.53)  72.79 (8.47) 148.02 (3.55)  64.69 (3.82)  96.35 (3.95)
               C10 -         C21           C27           C12_$2        C7            C2

C11 -        Distance       Angles
C9          0.983 (0.078)
C25         1.260 (0.086)    67.25 (6.68)
C21         1.324 (0.092)   148.06 (7.47)  80.84 (5.60)
C20         1.734 (0.092)    48.52 (5.30) 103.84 (5.94) 149.15 (4.95)
C10         1.929 (0.100)   158.46 (4.10) 105.94 (5.58)  33.95 (3.61) 118.69 (4.53)
C7          1.951 (0.093)   116.36 (6.30) 132.36 (4.53)  85.87 (5.28)  68.29 (3.72)  51.99 (3.75)
C18         1.957 (0.100)    13.54 (3.87)  71.27 (5.77) 149.53 (4.24)  37.42 (2.77) 146.03 (3.12) 103.78
(3.77)
C27         1.996 (0.191)   158.68 (7.17) 129.85 (7.76)  50.62 (6.44) 123.90 (6.32)  39.86 (5.83)  64.06
(5.67) 158.86 (6.57)
               C11 -         C9            C25           C21           C20           C10           C7
C18

C12 -        Distance       Angles
C10_$1      1.539 (0.079)
C15         1.573 (0.087)   119.80 (6.44)
C25         1.914 (0.094)   131.85 (4.38)  33.70 (3.48)
C16_$1      1.935 (0.090)    89.09 (4.65) 131.63 (4.87)  97.96 (4.74)
C6_$1       2.085 (0.100)   122.84 (4.79)  99.33 (4.85)  66.78 (4.14)  35.86 (2.79)
               C12 -         C10_$1        C15           C25           C16_$1

C13 -        Distance       Angles
C14_$2      1.123 (0.081)
C19_$2      1.566 (0.088)   120.57 (5.61)
C4          1.865 (0.102)    54.35 (4.71) 151.13 (2.78)
               C13 -         C14_$2        C19_$2

C14 -        Distance       Angles
C13_$1      1.123 (0.082)
C4_$1       1.516 (0.090)    88.65 (5.38)
C17         1.567 (0.080)    98.27 (5.44) 148.95 (3.47)
               C14 -         C13_$1        C4_$1

C15 -        Distance       Angles
C25         1.062 (0.097)
C12         1.573 (0.087)    91.06 (6.06)
C3          1.711 (0.088)   116.15 (6.11) 120.33 (5.70)
C21         1.951 (0.079)    59.16 (5.88) 148.00 (4.27)  70.13 (4.16)
               C15 -         C25           C12           C3

C16 -        Distance       Angles
C6          1.246 (0.087)
C8          1.630 (0.086)   139.83 (5.80)
C7          1.728 (0.088)   153.36 (4.00)  46.03 (3.44)
C12_$2      1.935 (0.090)    78.62 (4.91) 126.24 (4.25)  82.71 (4.10)
               C16 -         C6            C8            C7

C17 -        Distance       Angles
C14         1.567 (0.080)
C23         1.527 (0.065)    88.15 (3.57)
C5          1.700 (0.097)   121.05 (4.76) 134.79 (4.33)
               C17 -         C14           C23

C18 -        Distance       Angles
C9          1.028 (0.085)
C20         1.203 (0.077)    71.42 (6.56)
C6_$1       1.349 (0.101)   112.37 (7.86) 159.02 (4.77)
C25         1.958 (0.113)    35.06 (4.37)  94.38 (5.28)  81.38 (4.84)
C11         1.957 (0.100)    12.94 (3.97)  61.18 (4.90) 118.71 (5.47)  37.55 (2.89)
               C18 -         C9            C20           C6_$1         C25

C19 -        Distance       Angles
C20         1.397 (0.084)
```

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C5          1.542 (0.098)   133.66 (5.83)
C13_$1      1.566 (0.088)   100.06 (5.41) 116.03 (4.26)
C8          1.930 (0.091)    83.45 (4.67)  51.19 (3.76) 152.85 (3.03)
               C19 -         C20           C5           C13_$1

C20 -        Distance       Angles
C18         1.203 (0.077)
C9          1.310 (0.086)    48.05 (4.84)
C19         1.397 (0.084)   111.26 (6.25) 150.84 (4.72)
C11         1.734 (0.092)    81.40 (5.72)  34.21 (3.84) 145.69 (4.07)
               C20 -         C18           C9           C19

C21 -        Distance       Angles
C10         1.112 (0.081)
C11         1.324 (0.092)   104.36 (6.59)
C2          1.452 (0.098)    81.63 (6.55) 153.99 (4.08)
C27         1.544 (0.172)    57.87 (7.37)  87.87 (8.33) 115.81 (8.18)
C25         1.676 (0.102)   134.16 (5.26)  47.92 (4.23) 110.03 (5.04) 134.11 (8.83)
C15         1.951 (0.079)   153.96 (4.08)  77.87 (5.19)  85.98 (4.64) 147.53 (7.91)  32.95 (3.37)
               C21 -         C10           C11          C2            C27          C25

Br2 -        Distance       Angles
C24         0.952 (0.119)
C22         1.379 (0.118)   100.04 (9.99)
C26         1.383 (0.268)    54.14 (9.99)  62.93 (9.99)
               Br2 -         C24           C22

C22 -        Distance       Angles
C26         1.442 (0.288)
Br2         1.379 (0.118)    58.69 (9.99)
C24         1.807 (0.119)    38.69 (9.99)  31.25 (4.65)
               C22 -         C26           Br2

C23 -        Distance       Angles
C17_$2      1.527 (0.066)
C17         1.527 (0.066)   115.82 (1.26)
C17_$1      1.527 (0.065)   115.81 (1.26) 115.79 (1.26)
               C23 -         C17_$2        C17

C24 -        Distance       Angles
Br2         0.952 (0.119)
C26         1.130 (0.275)    82.80 (9.99)
C22         1.807 (0.119)    48.71 (8.20)  52.90 (9.99)
               C24 -         Br2           C26

C25 -        Distance       Angles
C15         1.062 (0.097)
C9          1.264 (0.099)   149.42 (8.67)
C11         1.260 (0.086)   131.04 (8.98)  45.85 (4.87)
C21         1.676 (0.102)    87.89 (6.81)  97.08 (5.72)  51.24 (4.35)
C12         1.914 (0.094)    55.24 (5.12) 120.35 (6.57) 164.49 (7.47) 141.35 (6.57)
C18         1.958 (0.113)   147.40 (7.38)  27.85 (4.06)  71.18 (5.77) 121.25 (5.25)  97.40 (4.85)
               C25 -         C15           C9           C11          C21          C12

C26 -        Distance       Angles
C24         1.130 (0.275)
C22         1.442 (0.288)    88.41 (9.99)
Br2         1.383 (0.268)    43.07 (9.99)  58.38 (9.99)
               C26 -         C24           C22

C27 -        Distance       Angles
C10         1.339 (0.182)
C21         1.544 (0.171)    44.67 (6.45)
C11         1.996 (0.191)    67.35 (7.67)  41.52 (4.99)
               C27 -         C10           C21

      0.1 seconds elapsed time


FMAP and GRID set by program

FMAP   2   3  21
GRID    -1.389  -2  -1     1.389   2   1

R1 =  0.1636 for    226 unique reflections after merging


Electron density synthesis with coefficients Fo-Fc

Maximum =    0.66,     Minimum =   -0.64 e/A^3,     Highest memory used =  1348 / 25059
Mean =    0.00,    Rms deviation from mean =    0.20 e/A^3


Fourier peaks appended to .res file

            x       y       z       sof     U      Peak   Distances to nearest atoms (including symmetry
equivalents)
```

```
Q1    1   0.3188  0.2500  0.5281   1.00000  0.05    0.66   0.63 C19  0.99 C20  1.58 C8  1.72 C5
Q2    1   0.3284  0.3036  0.5183   1.00000  0.05    0.58   0.60 C19  1.10 C13  1.63 C5  1.74 C20
Q3    1   0.5823  0.0805  0.5706   1.00000  0.05    0.58   0.34 BR2  0.72 C24  1.09 C26  1.26 C22
Q4    1   0.1719  0.2422  0.6058   1.00000  0.05    0.52   0.59 C25  1.19 C15  1.24 C9  1.59 C12
Q5    1   0.4518  0.1973  0.6257   1.00000  0.05    0.52   1.08 C16  1.09 C6  1.59 C4  1.94 C8
Q6    1   0.1784  0.1784  0.6670   1.00000  0.05    0.52   0.89 C15  1.16 C21  1.27 C25  1.43 C3
Q7    1   0.3849  0.3974  0.5309   1.00000  0.05    0.51   0.75 C17  1.13 C14  1.22 C23  1.96 C17
Q8    1   0.3200  0.5000  0.5000   0.50000  0.05    0.51   1.98 C14  2.00 C4  2.06 C5  2.11 C17
Q9    1   0.2705  0.0542  0.5184   1.00000  0.05    0.49   1.35 BR1  1.81 C27  2.47 C22  2.55 C7
Q10   1   0.2893  0.0204  0.4882   1.00000  0.05    0.48   1.25 BR1  1.84 C22  2.29 C22  2.64 C27
Q11   1   0.3516  0.1854  0.5372   1.00000  0.05    0.48   0.65 C8  1.05 C7  1.81 C20  1.82 C19
Q12   1   0.4240  0.3300  0.5317   1.00000  0.05    0.47   0.74 C17  1.16 C5  1.56 C4  1.72 C14
Q13   1   0.3809  0.1243  0.6395   1.00000  0.05    0.47   0.90 C16  1.36 C12  1.64 C7  1.72 C6
Q14   1   0.2821  0.0396  0.4982   1.00000  0.05    0.47   1.27 BR1  2.04 C22  2.29 C27  2.47 C22
Q15   1   0.3000  0.0000  0.5000   0.50000  0.05    0.46   1.37 BR1  1.87 C22  2.71 C27  3.06 BR2
Q16   1   0.1911  0.1981  0.7243   1.00000  0.05    0.45   0.40 C3  1.26 C2  1.44 C15  1.86 C21
Q17   1   0.2253  0.1239  0.6281   1.00000  0.05    0.43   0.60 C21  1.03 C27  1.20 C10  1.35 C11
Q18   1   0.4720  0.2477  0.6072   1.00000  0.05    0.42   0.87 C4  1.28 C13  1.50 C14  1.95 C5
Q19   1   0.2703  0.2361  0.4981   1.00000  0.05    0.40   0.47 C20  1.32 C18  1.33 C19  1.61 C9
Q20   1   0.1382  0.2863  0.6188   1.00000  0.05    0.40   0.69 C12  1.53 C6  1.54 C15  1.54 C25

Shortest distances between peaks (including symmetry equivalents)

   10  14  0.43     10  15  0.49      9  14  0.52     14  15  0.82     10  10  0.89      9  10  0.93      1   2  1.05
    4  20  1.08      5  18  1.08      1  19  1.11      6  16  1.17     10  14  1.17      9  15  1.22      1  11  1.38
   13  20  1.43      9  10  1.46      7  12  1.47     14  14  1.50      6  17  1.54      8  12  1.57      4   6  1.68
    2  19  1.73      9  14  1.81      2  12  1.89      7  12  1.90      5  13  1.94     11  19  1.96      2   7  2.08
    7   7  2.10      9   9  2.16     16  16  2.17      2  18  2.28     12  18  2.30      2  11  2.31     11  13  2.32
    6  20  2.36      7   8  2.37     16  17  2.39      5  20  2.41      4  16  2.42      4  13  2.42      4  17  2.49
    1  12  2.50      8  18  2.50      5  11  2.54      7  18  2.60      9  17  2.60      7   8  2.68      4  19  2.76
    4   5  2.76     16  20  2.78      2   5  2.87     11  18  2.88      3  10  2.90      9  11  2.94     13  17  2.95
   13  18  2.96      3  14  2.98

      0.4 seconds elapsed time

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+  wini23        finished at 16:42:03   Total elapsed time:       6.4 secs  +
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CONCLUSION :

Routine sample ? Probably yes for people working on fullerides. And to be considered always a possibility that a high resolution powder pattern is the image of disorder (though some diffusion should occur in the background, which is the case here).

Ordered ? Disordered ? So sad that no answer different from that of the organizers was proposed.

If someone wants to work more on that problem, all data are available (synchrotron, conventional X-ray powder and single crystal). Just ask for them (alb@cristal.org).