Indexing XRD-patterns, getting cell parameters

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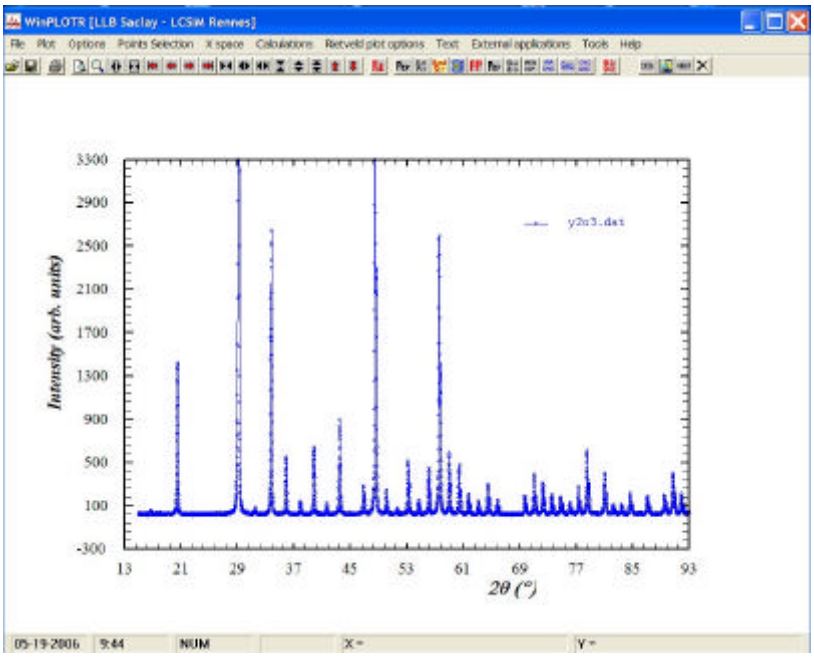
Abstract
Some keys, hints and tips for the successful indexing of a powder diffraction pattern are given through examples, by using exclusively free access programs for academic research. Essentially, the sample has to correspond to a single phase; the powder pattern resolution should be maximal, avoiding preferred orientation; the tools used for peak position hunting and indexing should be the best on the market; the figures of merit have to be understood so as to be able to save time by discarding the poor quality suggestions of the indexing programs; further checking by post-indexing visualization is recommended in case of good quality multiple equivalent propositions; finally, whole pattern fitting has to be the ultimate test in order to be convinced enough for attempting the structure solution.

Tip-1 : Indexing powder diffraction data is probably the crystallography topic where the traditional way of sharing openly computer programs remains the more respected (ITO, TREOR, DICVOL, CRYSFIRE, McMaille, etc).

Exercise 1 – starting with a simple case : Y_2O_3

The powder pattern for that exercise is available at http://www.cristal.org/powbase/ (Powbase is a small database of powder pattern), as well as the others.

Start WinPLOTR, select “File”, then select “Open pattern file”, select INSTRM=0 in the list of “Format of data file”, a windows allows you to select the data, find y2o3.dat (inside of 81.zip in PowBase) and open it.

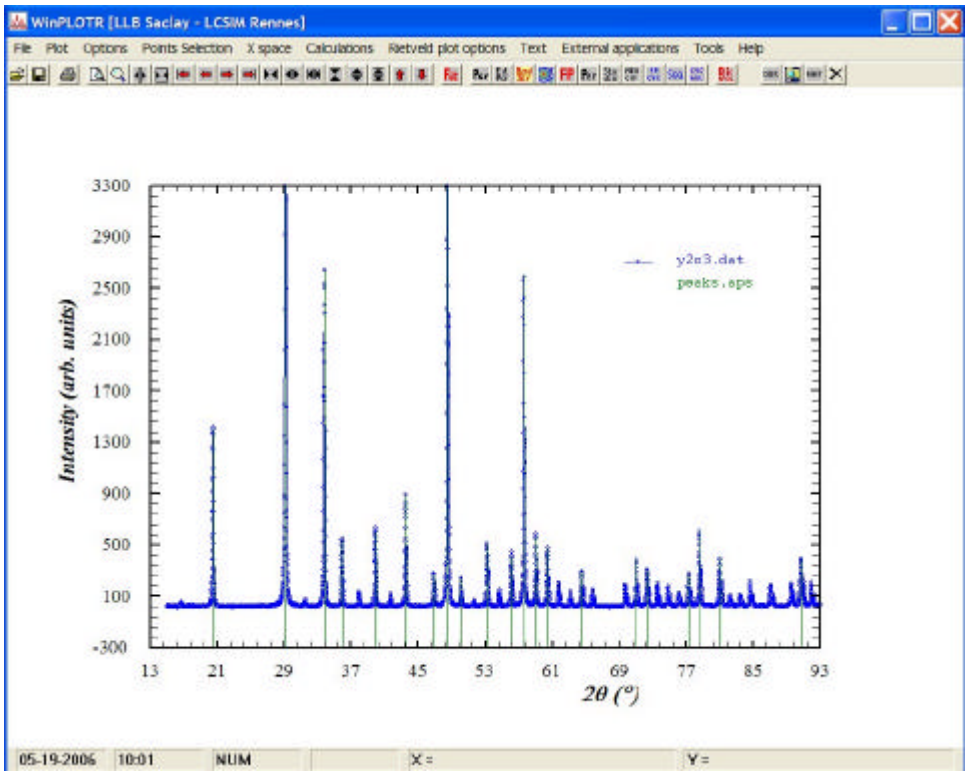


Tip-2 : The file y2o3.dat is a text file. Lines starting by ! or # are ignored by WinPLOTR. The data format corresponding to INSTRM=0 corresponds to a line with starting angle, step and last angle in degrees 2θ, then follow the intensities step by step in free format :

```
15.00 0.01 145.90
   25       22       19       21       19       14       23       25
   26       21       24       22       33       26       15       21
   19       20       20       16       14       17       22       21
   17       27       18       30       21       21       21       18
   19       22       22       28       19       19       13       30
.....etc
   37       33       44       48       55       46       61       48
   64       53       72       80       87      122      150      142
  150      203      238      256      316      386      453      557
  695      762      918     1005     1263     1372     1416     1398
 1363     1242     1095     1027      837      761      587      506
  384      300      205      199      158      121      123      109
  107       82       64       67       42       68       62       59
   55       51       45       47       36       31       31       43
   32       28       39       27       22       35       25       29
   29       17       23       24       18       23       26       29
```

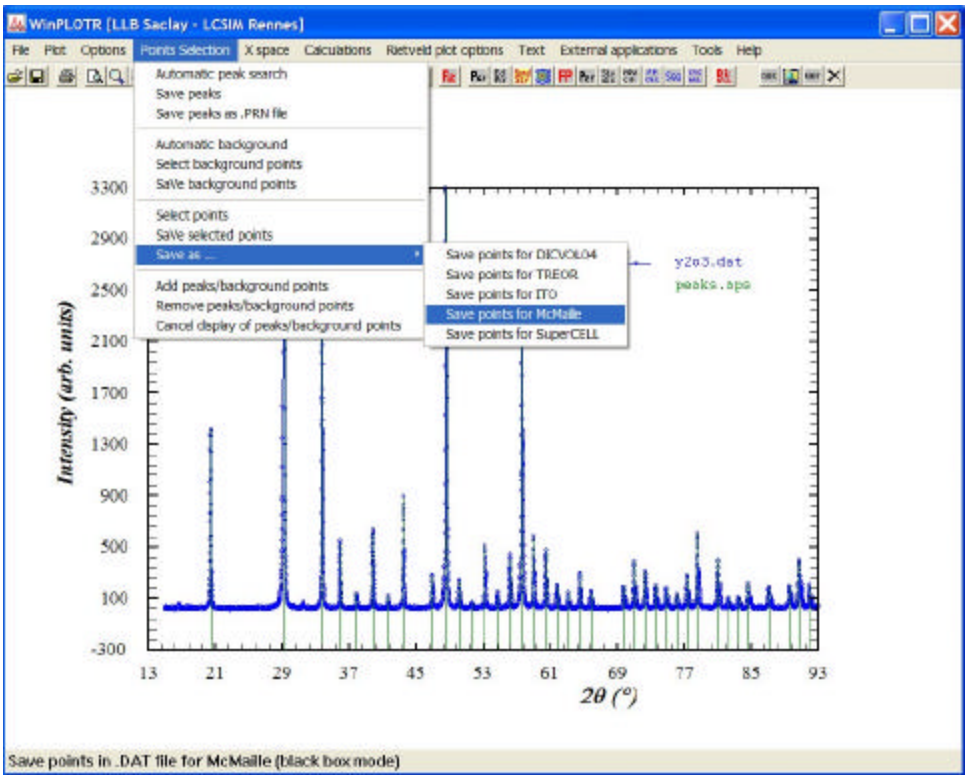
At this stage you are expected to make use of your crystallography expertise already. You must answer to the question : high or low symmetry ? Clearly, just by looking at the pattern, there are evidences for a high symmetry because of the quasi constant angular interval between the peak positions. Keep that possibility in mind.

Then click on “Points selection”, and “Automatic peak search”, select “Search Cu Kα1 Kα2 doublets” (yes, this is conventional laboratory X-ray data without any monochromator in the incident beam). Click on “OK”, the result is 21 peaks found if the range goes up to 93°(2θ). Clearly, many peaks with small intensity were not identified.



Go back to “Points selection”, and “Automatic peak search”, and change the “peak threshold” to 0.005 instead of 0.02, click on OK, that time 38 peaks are detected, click OK. Even if some very small peaks are not taken into account, we can try now to index.

Click again on “Points selection”, then on “save as”, then on “Save points for McMaille” (what were you expecting, I am the author of McMaille ;-):



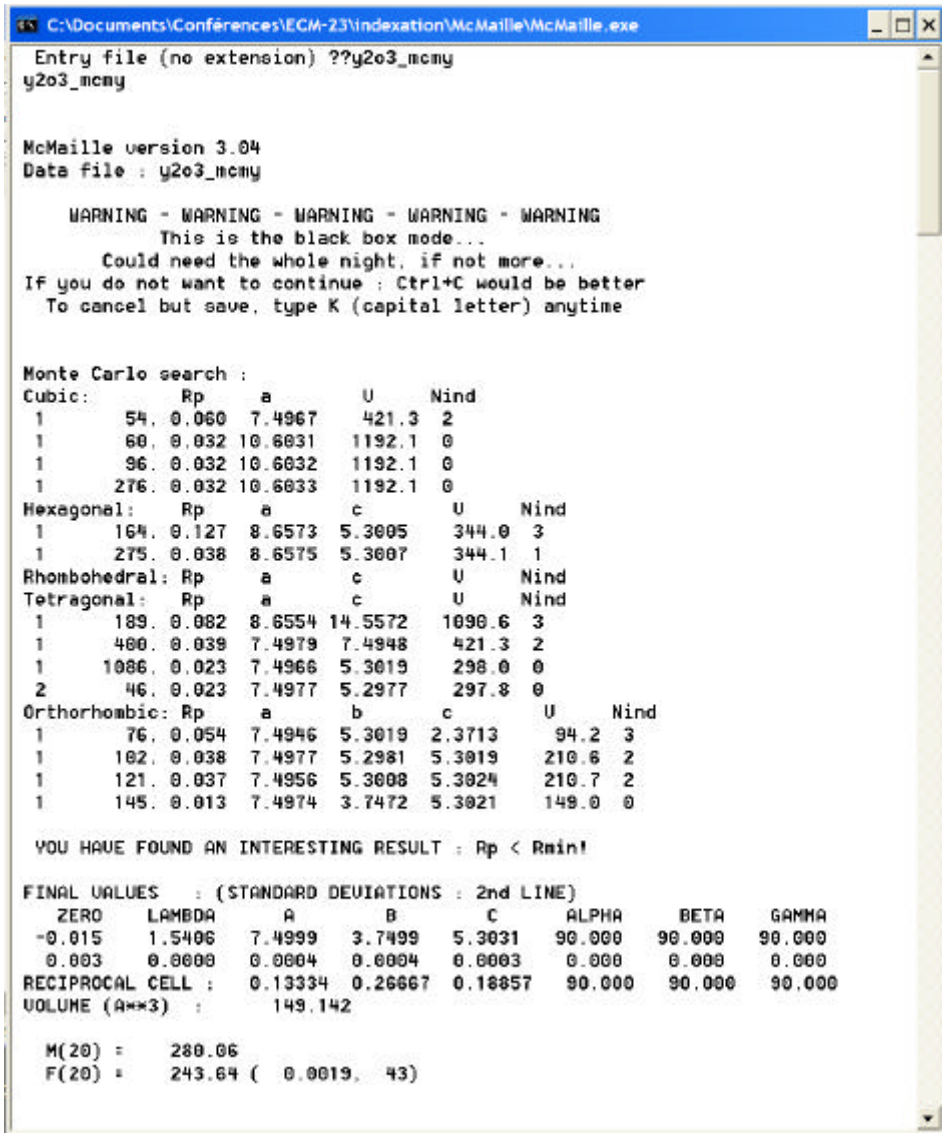
Then a box is opened, type a title (why not Y2O3), give the wavelength (1.54056, yes this is Kα1 only now) and let the zeroshift to be 0, click OK, the program says that a file named y2o3_mcmy.dat has been created, click OK and quit WinPLOTR.

Here is the y2o3_mcmy.dat file content :

```
Y2O3
! Wavelength, zeropoint and NGRID (NGRID=3: black box mode)
  1.540560   0.000  3
! List of 2theta positions, intensity (min.=20)
      20.50481    1411.71155
      29.15784   11198.95700
      33.79123    2632.66553
      35.91046     531.02966
      37.92135     135.21042
      39.84880     606.23480
      41.70092     118.49068
      43.48877     881.15845
      46.88918     271.40564
      48.52835    4213.47021
      50.12016     230.08562
      51.68559      66.62399
      53.20726     506.43854
      54.69715     117.27492
```

```
56.16987     404.76215
57.61465    2603.39868
59.03540     563.80334
60.43375     471.21359
61.81146     194.23712
Etc
```

Copy that file inside of the directory where McMaille is installed and start this indexing program (click on “McMaille.exe, and give the file name without extension: y2o3_mcmy) :



Tip-3 : You will probably not obtain exactly this result, because McMaille uses a Monte Carlo algorithm, implying a sequence of pseudo-random number. You would obtain the same result if you start exactly at the same point in that sequence. The starting point is defined in McMaille by the time (expressed in seconds since the beginning of the day) at the instant when you start the program. At least you should obtain a similar result in a different order.

Let us comment these results. McMaille is working in the so-called “black box mode”. We have given only very few informations : peak positions, intensities, wavelength, a null zeropoint (this is a constant – an approximation - angular shift due to both instrumental – if misaligned - and sample – if not in the diffracting plane – effects that we should not neglect in

the next exercises). In that black box mode, McMaille follows its own strategy : testing first the highest symmetries, down to triclinic, inside of some predetermined limits of cell parameters and volumes.

Tip-4 : All indexing programs have such maximal default values which the user has to know – see the manuals - and should modify if no result is obtained.

The essential things to see in the output file of any indexing program are the FoMs (Figures of Merit), generally noted M20 and F20 or F30. The higher are these FoMs, the more the corresponding cells are plausible. For the definitions of the FoMs, see the excellent chapter 7, autoindexing, written by Per-Erik Wernet in the book "Structure Determination from Powder Diffraction Data.

Tip-5 : References are at the end of the tutorial

McMaille provides also these FoMs for selected good cells, but the solutions are sorted according to a somewhat different criterion noted Rp which is equivalent to a Rietveld profile reliability. The program will not always stop like it has done here, it may well examine all symmetries down to triclinic. In black box mode, it will stop if a solution with Rp < 0.02 is found. In that case, it has found an orthorhombic solution with Rp = 0.013 with a very small cell volume (149 $Å^3$) and very large conventional FoMs (M20 = 280). A very small Rp value (< 0.05) means that the powder pattern is well fitted, the smaller will be Rp, the larger will be the conventional FoMs M20 and F20.

But is it the good cell ?

Tip-6 : The ultimate decision has to be made by the user, always…

Having a good formation in crystallography will help a lot to make the decision. In that case, solutions with small Rp are also proposed in cubic (R = 0.032), hexagonal (R = 0.038) and tetragonal (R = 0.023).

Tip-7 : First important point to keep in mind for becoming an expert in indexing : any cell with a high symmetry can also be indexed in all the lower symmetries.

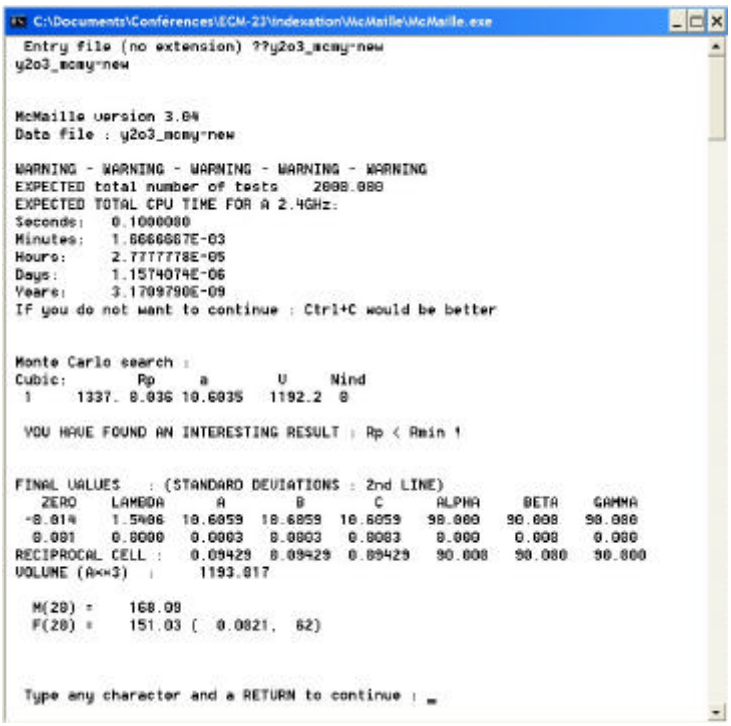
In that case, if McMaille had not stopped at the orthorhombic symmetry, it would have provided cells also in monoclinic and triclinic symmetries, possibly with even lower Rp values.

Are all lines indexed ? In black box mode, whatever the number of lines given, McMaille will work only by using the first 20 lines, and has a tolerancy of 3 unindexed lines. The number of not indexed lines is provided (parameter Nind in the figure above). All lines are indexed in cubic, tetragonal and orthorhombic (Nind = 0). Making the ratio Vcubic/Vortho gives 1192/148 = 8… So, very probably, the cell is cubic. McMaille prepares a new .dat file for further investigations in manual mode. That file name is y2o3_mcmy-new.dat. Edit that file :

```
Y2O3
! Wavelength, zeropoint, Ngrid
 1.540560 0.0000 0
! Codes for symmetry
```

```
 1 0 0 0 0 0                                          Cubic only
! W, Nind
0.300  3
!Pmin, Pmax, Vmin, Vmax, Rmin, Rmax, Rmaxref
  2. 20. 8. 2000. 0.05 0.25 0.50                      Decrease some values here
! Ntests, Nruns
 2000 1
!  2-theta   Intensity
   20.50481       1411.712
   29.15784       11198.96
   33.79123       2632.666
   35.91046       531.0297
   37.92135       135.2104
   39.84880       606.2348
   41.70092       118.4907
   43.48877       881.1584
   46.88918       271.4056
   48.52835       4213.470
   50.12016       230.0856
   51.68559       66.62399
   53.20726       506.4385
   54.69715       117.2749
   56.16987       404.7621
   57.61465       2603.399
   59.03540       563.8033
   60.43375       471.2136
   61.81146       194.2371
   63.17843       149.1199
   64.53022       288.8621
   65.85427       145.6454
   69.78623       190.6706
```

If you read the manual (don't believe you can become an expert in 2 hours…) you will see that that file is configured for a search only in cubic, just fine, this is what we want to do. Apply again McMaille to that file :



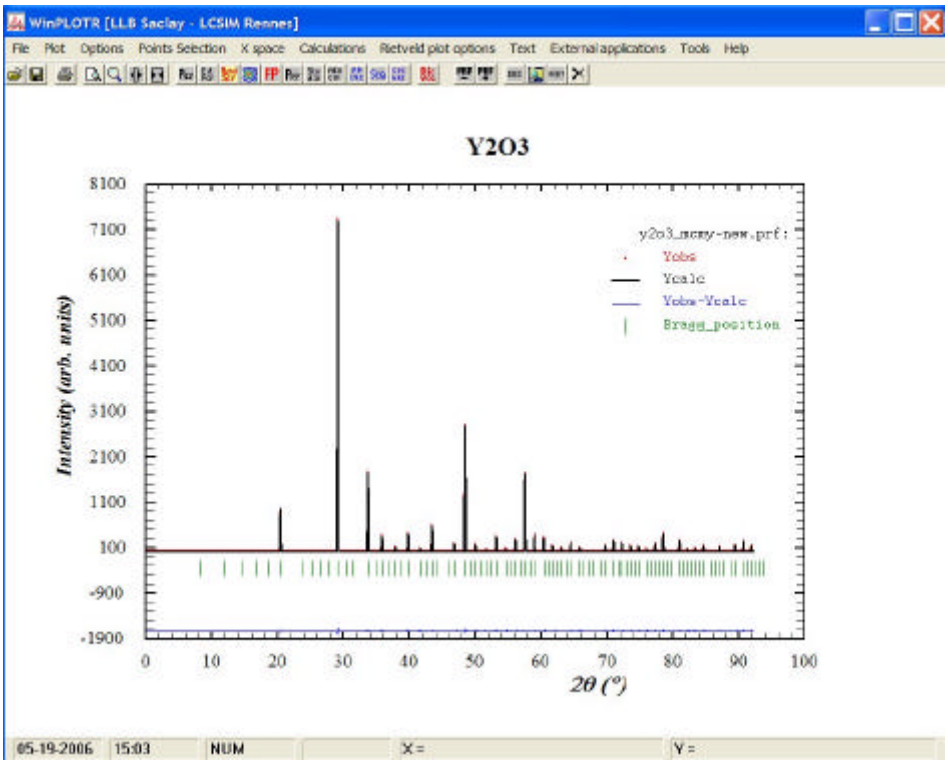
Have a look inside of the y2o3_mcmy-new.imp file containing the detailed results :

```
FINAL VALUES   : (STANDARD DEVIATIONS : 2nd LINE)

    ZERO     LAMBDA       A         B         C       ALPHA      BETA     GAMMA
  -0.014     1.5406   10.6059   10.6059   10.6059    90.000    90.000    90.000
   0.001     0.0000    0.0003    0.0003    0.0003     0.000     0.000     0.000
 RECIPROCAL CELL :    0.09429   0.09429   0.09429    90.000    90.000    90.000
 VOLUME (A**3)  :      1193.017

    H      K      L   TH(OBS)    TH-ZERO    TH(CALC)     DIFF.

    2      1      1    20.505     20.491     20.495     -0.004
    2      2      2    29.158     29.144     29.143      0.001
    4      0      0    33.791     33.777     33.777      0.000
    4      1      1    35.910     35.897     35.893      0.003
    4      2      0    37.921     37.907     37.907      0.001
    3      3      2    39.849     39.835     39.833      0.002
    4      2      2    41.701     41.687     41.685      0.002
    4      3      1    43.489     43.475     43.472      0.003
    5      2      1    46.889     46.875     46.881     -0.006
    4      4      0    48.528     48.514     48.515     -0.001
    4      3      3    50.120     50.106     50.110     -0.003
    4      4      2    51.686     51.672     51.668      0.004
    6      1      1    53.207     53.193     53.193      0.000
    6      2      0    54.697     54.683     54.688     -0.005
    5      4      1    56.170     56.156     56.157     -0.001
    6      2      2    57.615     57.601     57.600      0.001
    6      3      1    59.035     59.022     59.021      0.001
    4      4      4    60.434     60.420     60.421     -0.001
    7      1      0    61.811     61.798     61.801     -0.004
    6      4      0    63.178     63.165     63.165      0.000
    6      3      3    64.530     64.516     64.511      0.005
    6      4      2    65.854     65.840     65.843     -0.003
    7      3      2    69.786     69.772     69.761      0.011
    8      0      0    71.059     71.045     71.044      0.001
    5      5      4    72.331     72.318     72.318      0.000
    8      2      0    73.591     73.577     73.582     -0.006
    6      5      3    74.859     74.845     74.839      0.006
    6      6      0    76.097     76.083     76.087     -0.004
    7      4      3    77.347     77.333     77.330      0.004
    6      6      2    78.580     78.566     78.566      0.000
    8      4      0    81.036     81.022     81.023      0.000
    8      3      3    82.255     82.241     82.244     -0.003
    8      4      2    83.480     83.466     83.463      0.003
    6      5      5    84.689     84.675     84.678     -0.004
    7      5      4    87.115     87.101     87.102     -0.001
    7      6      3    89.529     89.515     89.521     -0.006
    8      4      4    90.747     90.733     90.730      0.003
    9      4      1    91.955     91.941     91.940      0.001

  M(20) =    168.09
  F(20) =    151.03 (  0.0021,  62)
```

This looks extremely good.

Will any attempt be that easy ? Certainly not… this was the first exercise and you are lucky.

McMaille prepares a file for a Rietveld-like fit (a .prf file) for the best solution which you can see by using WinPLOTR. Let us see that y2o3_mcmy-new.prf file. Start WinPLOTR, click on "File", then on "Open Rietveld/profile file", select "101 : Fullprof PRF file", click on OK, and select the file in the Windows box.



The vertical bars note all lines that should be there. A lot are lacking. You may realize, by examining the hkl indices of the indexed lines that there is a systematic relation h + k + l = 2n, and thus the Bravais lattice is very probably I – centered. McMaille will not do that automatically for you.

Tip-8 : Some programs perform automatically everything (indexing, space group suggestion, solving the structure, refining, etc), like EXPO2006 : use these programs first, and if they fail, go back to some standalone programs.

Tip-9 : Only solving the structure confirm the indexing… but this is not the scope of this tutorial.

Tip-10 : you should never try to index without having defined the zeropoint…

Exercise 2 – A bit more difficult : t-AlF_3

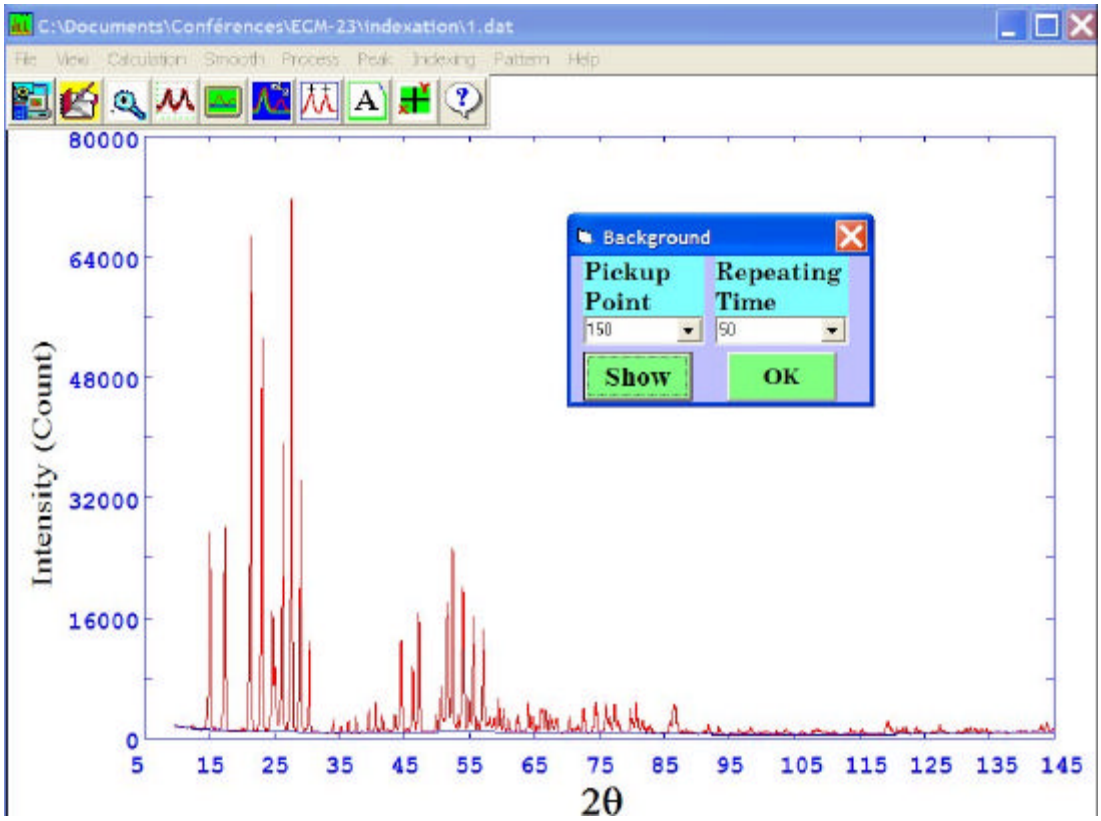
Software for hunting the peak positions are numerous (see session 2 of the SDPD Internet Course : http://sdpd.univ-lemans.fr/DU-SDPD/), free access (WinPLOTR, PowderX, EXPO2004, etc) or commercial (EVA, Highscore, Jade, etc). Let us try PowderX on the τ-AlF_3 powder pattern. In fact the pattern is corrected for a large zeropoint error (0.3° !) due to the fact that the sample was dusted on the sample holder for avoiding preferred orientation.

Tip–11 : Dusting a sample on the holder through a sieve allows for reducing preferred orientation effects. But since this enlarges the peak width, it is not recommended at the indexing stage : press the sample for a better resolution.

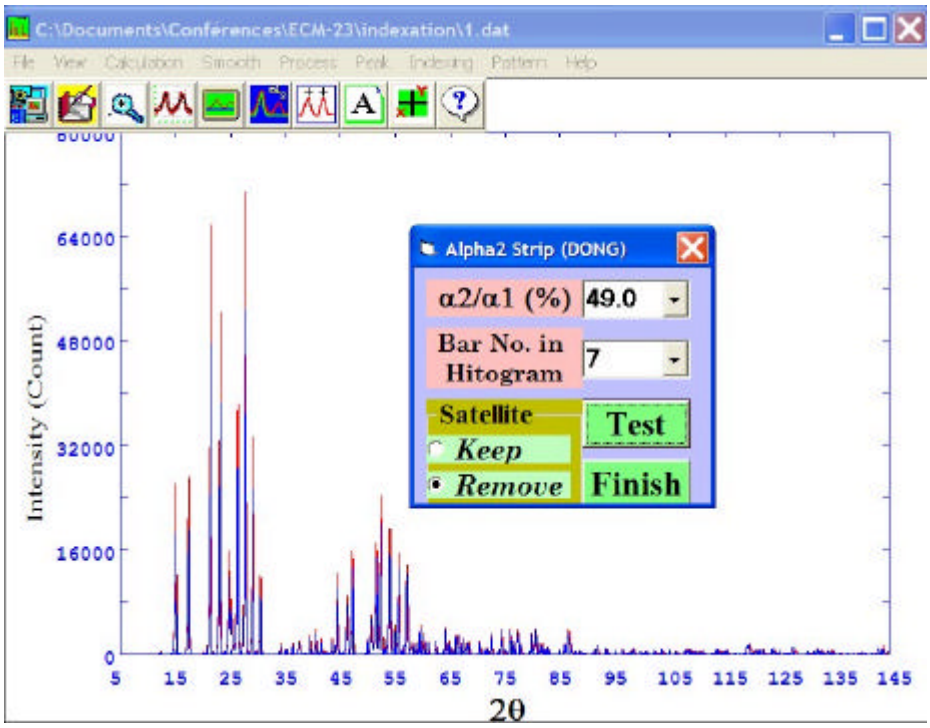
Tip-12 : Typical excellent minimal FWHMs (Full Width at Half Maximum) are (for well crystallized compounds) : 0.04 to 0.06°(2θ) for conventional laboratory instrument and 0.005 to 0.01°(2θ) for third generation synchrotron source. Note that a 0.02° FWHM at a synchrotron source with a 0.7 Å wavelength is equivalent to 0.04° in your lab with a copper target…

Tip-13 : The zeropoint correction can be done before indexing by two methods : mixing a reference compound with the sample or using the harmonics technique. Let us consider that you know how doing these "simple" things. If not, see sessions 2 and 3 of the SDPD Internet Course.

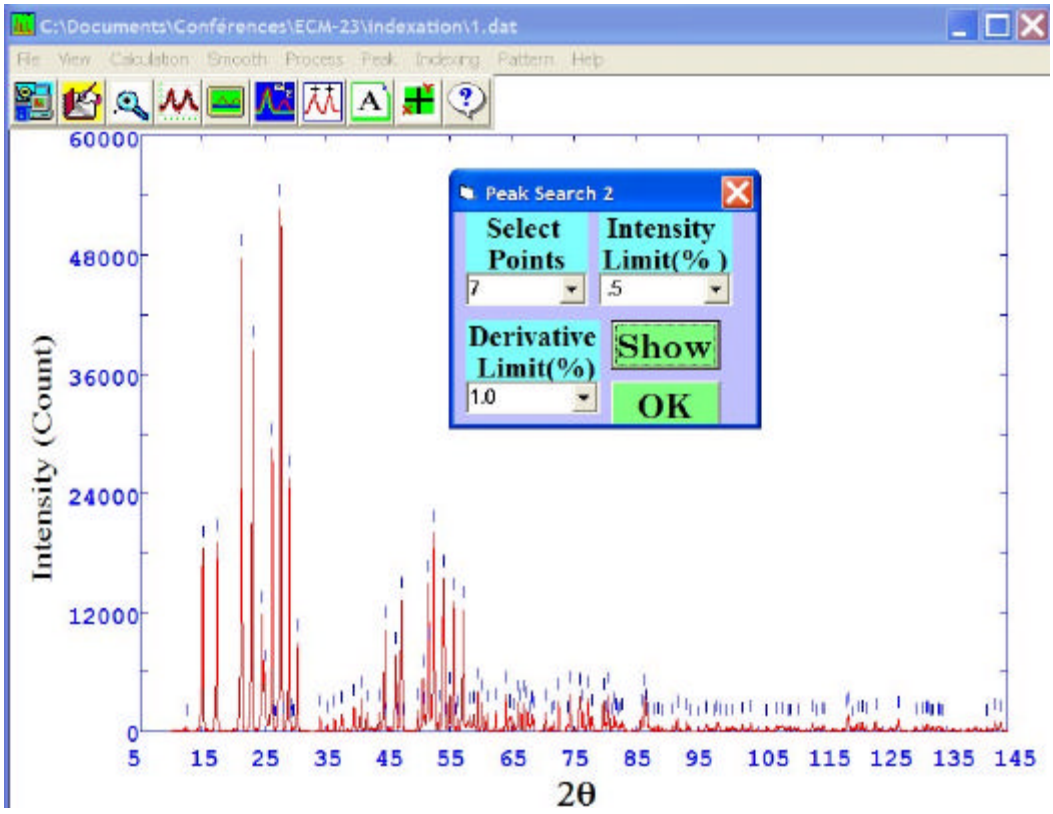
The τ-AlF_3 powder pattern is also available in PowBase (1.dat inside of 1.zip), and can be read with PowderX by clicking on "File", then "Import Data", then "RIET7" for the format. This is a format analogous to the one read by WinPLOTR (INSTRM=0), but you have to add 4 header lines (blank if you wish). First remove the background : click on "Substract Background" then on "Show" in the small box and the on "OK" if you are satisfied (otherwise play with the options, and zoom to see the effect).



Click on "Alpha2 Elimination", click on "Test", click on "Finish".



Click on “Peak Search”, then on “Show”, modify the intensity limit for the detection of the weakest peaks if you wish, then click on OK.



Save the file after confirmation of the wavelength :

2Theta	d (A)	Height	Area	FWHM
12,292	7,19449	336,0	1262,2	0,0800
15,040	5,88588	18391,1	147126,3	0,1600
17,358	5,10448	19047,3	144454,3	0,1600
21,324	4,16331	47804,6	323420,4	0,1400
23,056	3,85441	38529,0	268961,3	0,1400
24,656	3,60768	11816,5	82581,7	0,1400
25,039	3,55334	5890,4	54511,0	0,2000
26,272	3,38940	28732,0	227980,7	0,1600
26,632	3,34432	408,5	1447,7	0,0800
26,748	3,33017	290,9	1085,7	0,0800
27,634	3,22529	52845,1	474146,2	0,1800
27,936	3,19114	644,3	2064,0	0,0800
29,023	3,07407	25452,0	203382,7	0,1600
29,240	3,05179	943,2	3265,4	0,0800
29,401	3,03544	284,2	1420,5	0,1000
29,729	3,00260	337,7	1909,7	0,1200
30,394	2,93842	8950,3	71150,3	0,1600
34,038	2,63177	1399,2	11181,9	0,1600
35,239	2,54473	698,6	4946,1	0,1600
36,355	2,46914	1346,2	10729,8	0,1600
37,523	2,39493	1679,4	12700,5	0,1600
39,501	2,27942	2415,3	16903,5	0,1400
40,567	2,22199	3282,1	22194,2	0,1400
41,526	2,17285	1868,9	14926,1	0,1600

Tip-14 : You may also play with the zeropoint correction system inside of PowderX. Then save the file for TREOR and transform it into a file for McMaille ;-). You have to change the comma into points and remove the stuff at the end of the file, rename it as AlF3.dat in the McMaille directory.

Tip-15 : When using PowderX, be carefull that the wavelength was taken into account, otherwise use only 2θ values, but not d(Å) values.

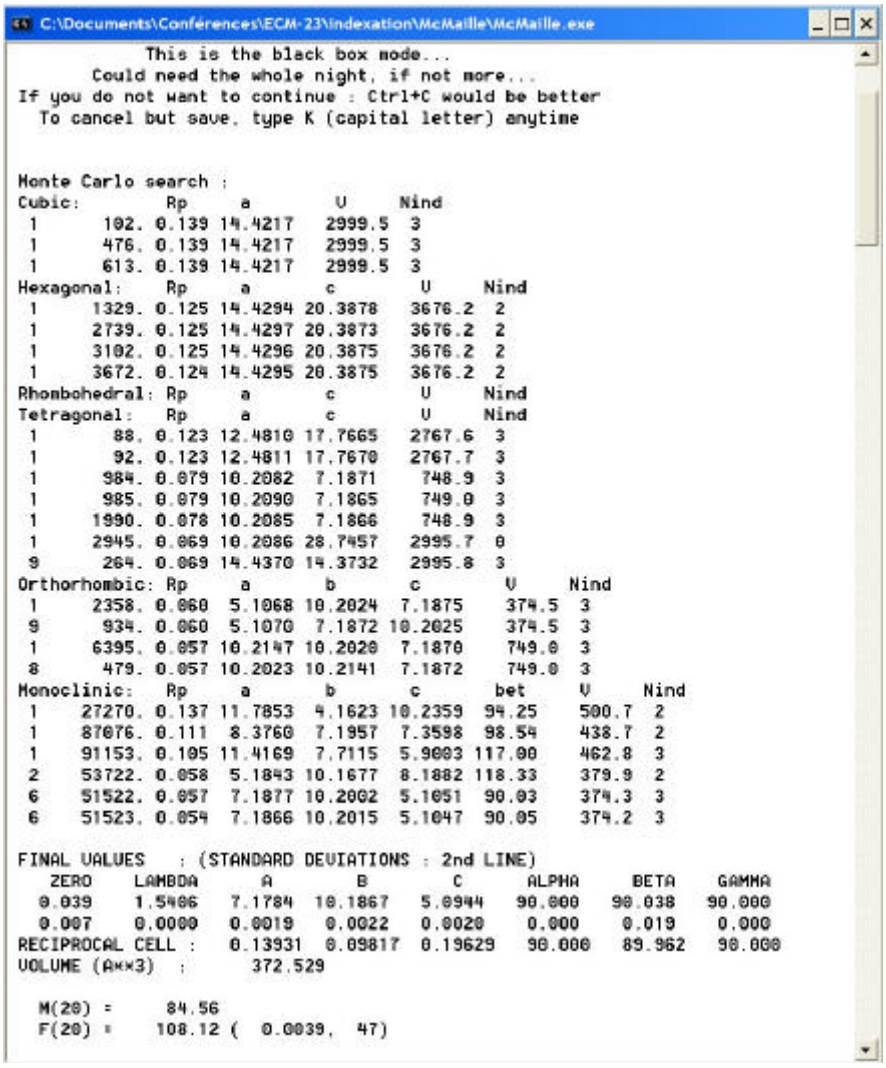
```
AlF3
1.54056  0  3
       12.292          6
       15.040        348
       17.358        360
       21.324        905
       23.056        729
       24.656        224
       25.039        111
       26.272        544
       26.632          8   Remove
       26.748          6   Remove
       27.634       1000
       27.936         12
       29.023        482
       29.240         18
       29.401          5   Remove
       29.729          6   Remove
       30.394        169
```

```
34.038          26
35.239          13
36.355          25
37.523          32
39.501          46
40.567          62
41.526          35
43.515          36
44.450         195
44.727           8    Remove
46.241         145
46.523          10
47.176         251
47.480           9    Remove
49.878          38
```

Remove those weak peaks with intensity < 1% of the most intense one (maybe leaving the first at low angle).

Tip-16 : Peaks at low angles are essential even if they are weak.

Start McMaille and see the results. Cells with R close to 5% appear in tetragonal, orthorhombic, monoclinic, you may stop the calculation by typing K (large character), and then think…



Which cell is the good one ? Again there are several possibilities. Remember that any high symmetry cell can be proposed in a lower symmetry. In that case, the tetragonal cell with smallest volume seems to have chances to be the correct one. McMaille produces files with .ckm extension which can be read by the Chekcell program and may help to decide.

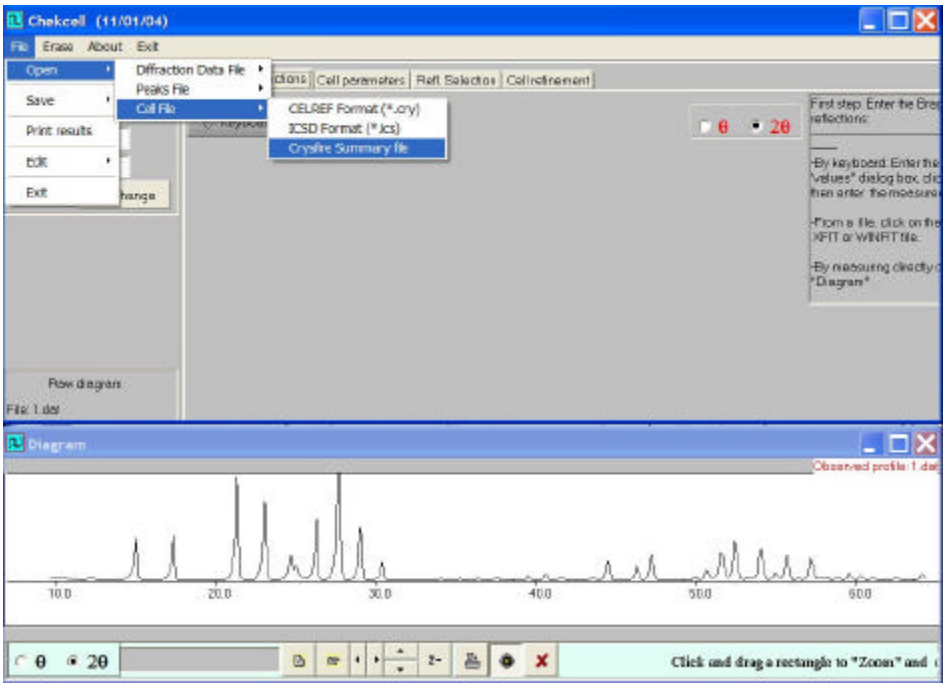
The AlF3.ckm file :

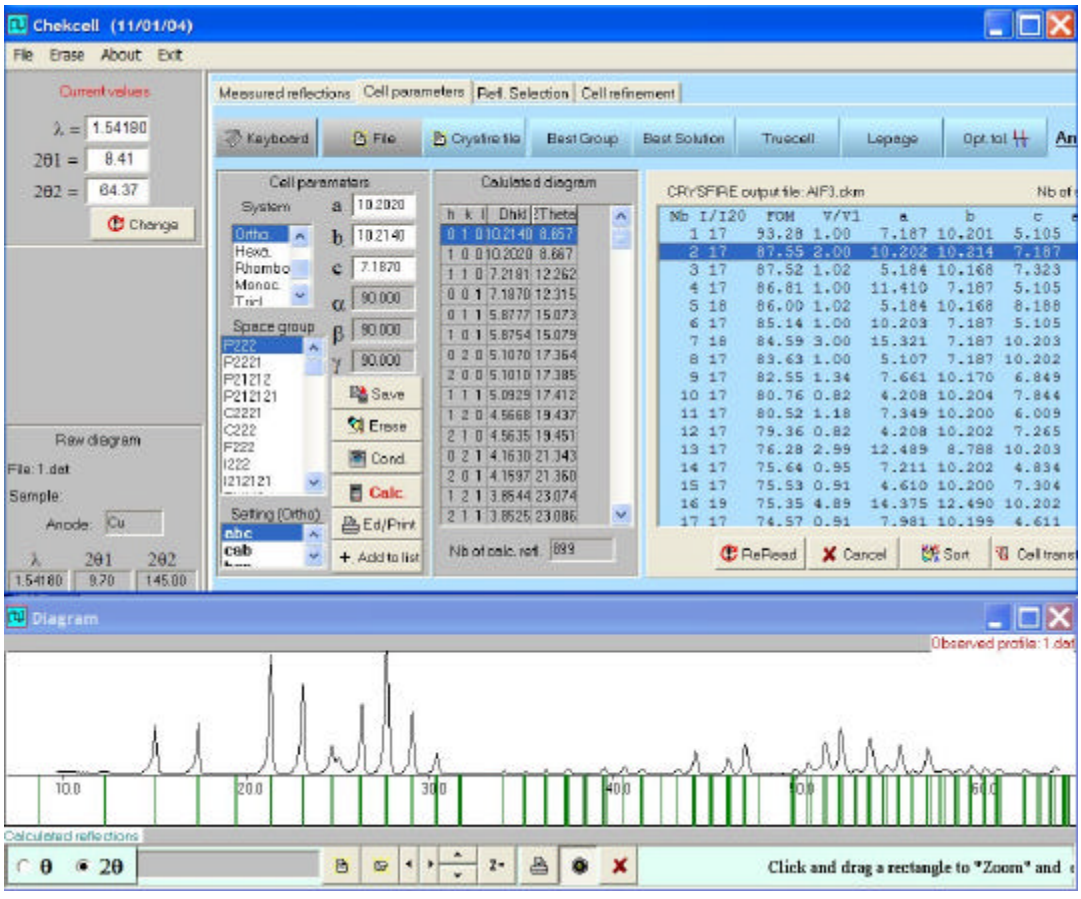
N	FoM	V	V/V1						
17	93.28	374.247	1.00	7.1866	10.2015	5.1047	90.000	90.047	90.000
17	**87.55**	**748.954**	**2.00**	**10.2023**	**10.2141**	**7.1872**	**90.000**	**90.000**	**90.000**
17	87.52	379.961	1.02	5.1844	10.1684	7.3235	90.000	100.212	90.000
17	86.81	374.340	1.00	11.4104	7.1869	5.1053	90.000	116.603	90.000
18	86.00	379.919	1.02	5.1843	10.1677	8.1882	90.000	118.331	90.000
17	85.14	374.347	1.00	10.2026	7.1867	5.1054	90.000	90.030	90.000
18	84.59	1123.379	3.00	15.3210	7.1867	10.2026	90.000	90.000	90.000
17	83.63	374.484	1.00	5.1070	7.1872	10.2025	90.000	90.000	90.000
17	82.55	502.511	1.34	7.6607	10.1696	6.8487	90.000	109.642	90.000
17	80.76	308.628	0.82	4.2084	10.2036	7.8445	90.000	113.620	90.000
17	80.52	441.141	1.18	7.3487	10.1996	6.0087	90.000	101.627	90.000
17	79.36	308.588	0.82	4.2083	10.2022	7.2651	90.000	98.381	90.000
17	76.28	1119.785	2.99	12.4887	8.7879	10.2032	90.000	90.000	90.000
17	75.64	354.395	0.95	7.2113	10.2017	4.8337	90.000	94.722	90.000
17	75.53	338.952	0.91	4.6103	10.2002	7.3041	90.000	99.319	90.000
19	75.35	1831.669	4.89	14.3750	12.4897	10.2020	90.000	90.000	90.000
17	74.57	338.922	0.91	7.9813	10.1991	4.6105	90.000	115.436	90.000
17	73.60	502.352	1.34	7.6535	10.1970	6.8273	90.000	109.471	90.000
17	72.48	2995.752	8.00	14.4370	14.4370	14.3732	90.000	90.000	90.000
20	72.46	2995.731	8.00	10.2086	10.2086	28.7457	90.000	90.000	90.000
18	68.96	336.200	0.90	7.9158	10.2015	4.5834	90.000	114.722	90.000
18	68.34	371.538	0.99	10.1685	5.1044	7.1584	90.000	90.522	90.000
19	67.56	1208.311	3.23	14.2227	10.2029	8.3267	90.000	90.000	90.000
18	67.31	470.527	1.26	6.4170	10.2025	7.4635	90.000	105.645	90.000
17	64.36	429.930	1.15	7.2975	10.2033	5.8628	90.000	99.978	90.000
17	63.96	362.825	0.97	4.9471	10.2019	7.1966	90.000	92.649	90.000
17	**63.92**	**748.935**	**2.00**	**10.2085**	**10.2085**	**7.1865**	**90.000**	**90.000**	**90.000**
17	63.25	338.099	0.90	7.3083	10.1965	4.6008	90.000	99.549	90.000

Most of the cells with the highest FoMs have volumes multiple of 374 $Å^3$.

Start Chekcell. Click on “File”, then “Open”, then “Diffraction Data File”, then “Rietveld Format”, specify the wavelength when asked by the program, the pattern should appear (the data file must have 4 header lines), zoom eventually.

Then, open the .ckm file : click on “File”, “Open”, “Cell File”, “Crysfire Summary File”, search for the .ckm option and select it, find AlF3.ckm (note that sub-files for different symmetries are also built up by McMaille) :





There is a chance that the cell would be tetragonal with cell parameters a = 10.2 and c = 7.2 Å. One intense impurity line is obvious (this is α-AlF_3).

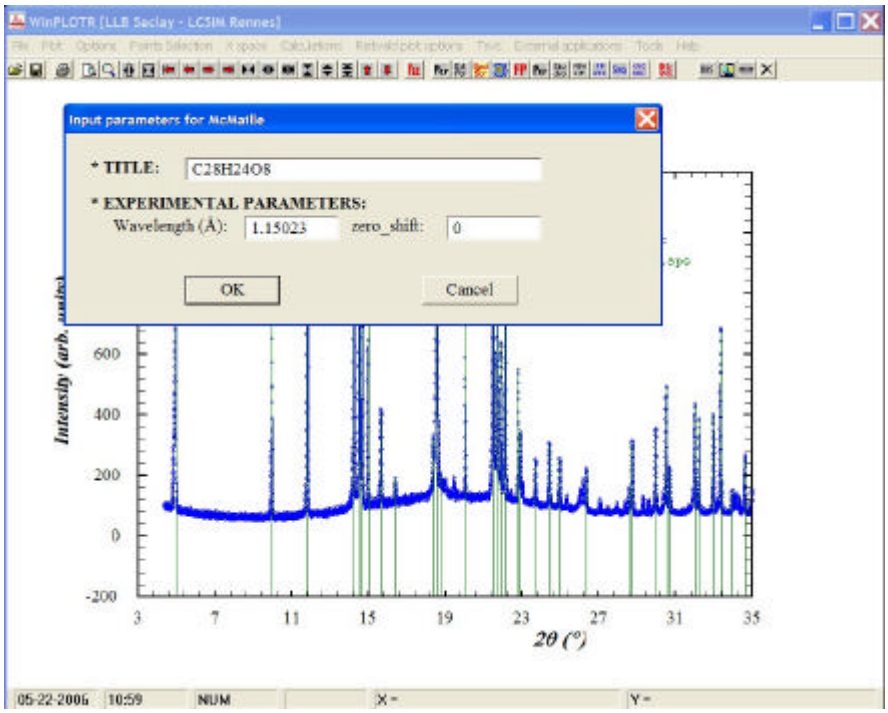
Tip-17 : Whole profile fitting (Pawley of Le Bail methods) would provide the ultimate convincing arguments (a perfect fit…) and allow to decide for some space group possibilities.

Exercise 3 – $C_{28}H_{24}O_8$ synchrotron data - Even more difficult ?

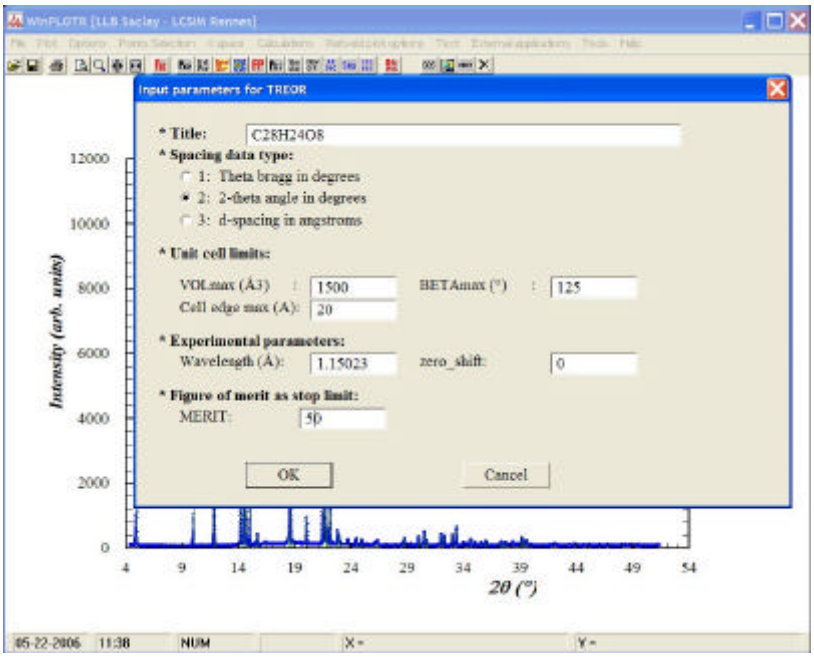
The powder pattern for that sample is also inside of PowBase (see 89.dat in 89.zip). Let us come back to WinPLOTR for the peak position extraction. Wavelength : λ = 1.15023 Å.

Tip-18 : in principle, synchrotron data corresponds to parallel beamline so that there is no zeropoint. In practice, but verify…

Use the automatic peak search option as in the exercise 1, decrease the peak threshold to 0.005 in order to locate some of the weakest peaks and save the file for McMaille :



Start McMaille, and during the calculations, save also the results for TREOR :



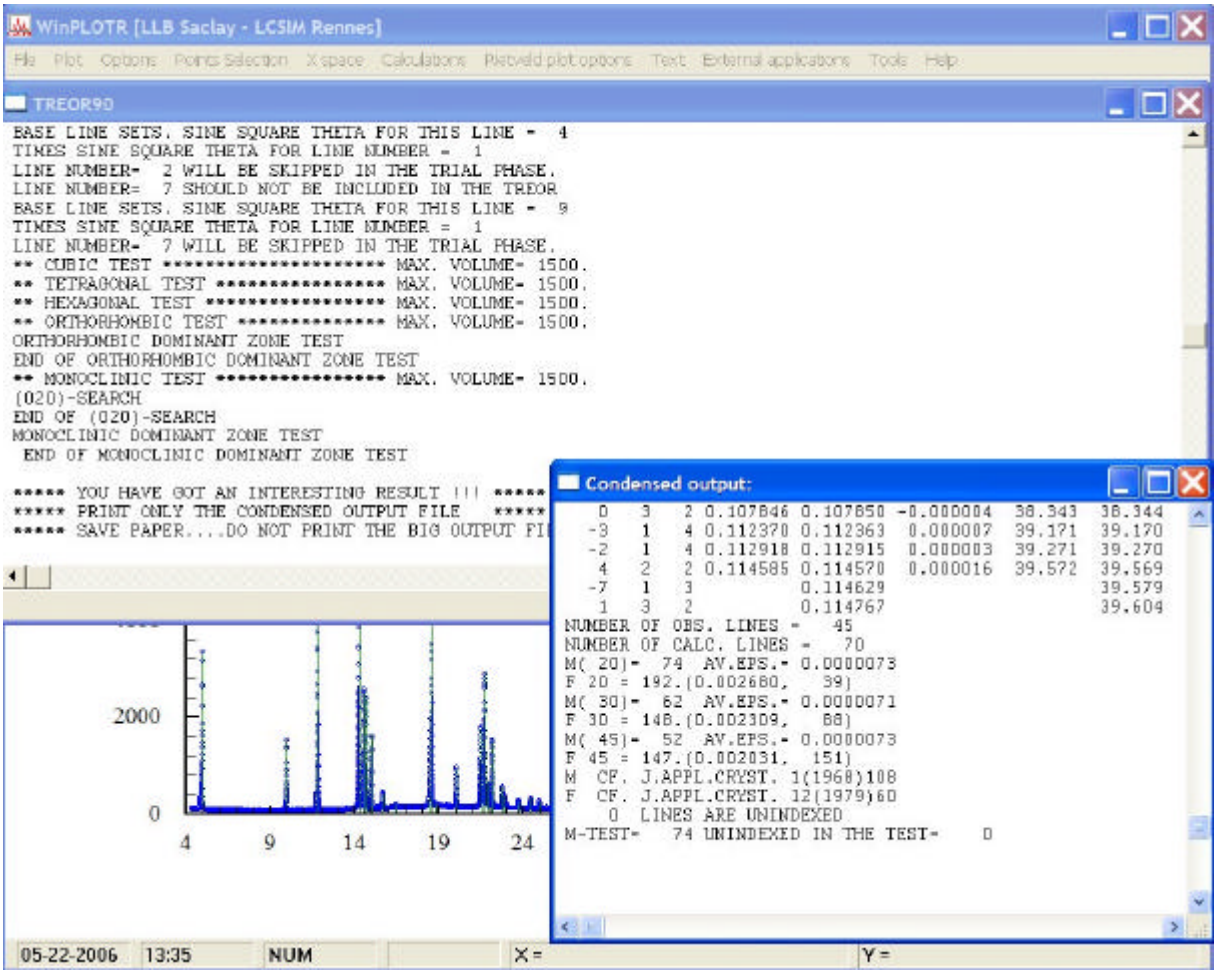
Do not forget to change the wavelength and increase the limit of the FoM to 50 : this is synchrotron data, you may expect high FoMs.

```
! DATA FILE: 89.dat
! INSTRM:  0
 C28H24O8
      4.98193   3286.80762     83.30889
      9.99003   1472.15564     57.64410
     11.83465   4172.48730     66.85038
     14.26077   5120.27832    132.29897
     14.54244   2541.02197    127.71822
     14.68000   2377.54810    114.03048
     15.01395   1603.88196     96.83777
     15.67321    402.99741    105.96571
     16.43242    187.91476    106.60686
     18.40516    320.04770    157.74744
     18.57075  10253.10740    161.52725
     18.81269    205.76633    155.59302
     20.06825    933.78021    125.71569
     21.49577   1768.68835    149.90627
     21.70489   2902.25244    157.81589
     21.89608    637.32843    155.65938
     22.13791   1497.10925    137.45313
     22.80752    548.14728    116.36189
     22.90763    345.16406    117.80325
     23.72424    243.58403    108.58457
     24.44472    301.27338     99.54028
     24.98247    252.92178     90.35323
     26.37432    219.01628     82.18118
     28.60269    157.93480     75.28192
     28.76217    303.62329     73.10251
     29.97874    350.80719     71.17887
     30.52339    490.87183     83.80813
     30.69416    222.56485     81.57237
     32.01038    431.36816     96.65090
     32.23336    385.18842     92.53907
     32.98502    394.52618     82.19423
     33.36611    675.33411     80.25625
     33.97608    144.84073     73.54704

 CHOICE=3,
 WAVE= 1.15023,
 MONO=125.00,
 VOL=  1500.,
 CEM=  20.,
 MERIT= 50.,
 END*
```

Maybe there are too much data.

Tip-19 : generally, the first 20 lines of a powder pattern are sufficient for indexing. Using more may even obscure the results…

And run TREOR inside of WinPLOTR :



Inside of WinPLOTR, TREOR produces also files for CHEKCELL.

Tip-20 : the statement ***** YOU HAVE GOT AN INTERESTING RESULT !!! ***** is given only if M(20) is better than your specifications, generally it announces a correct solution.

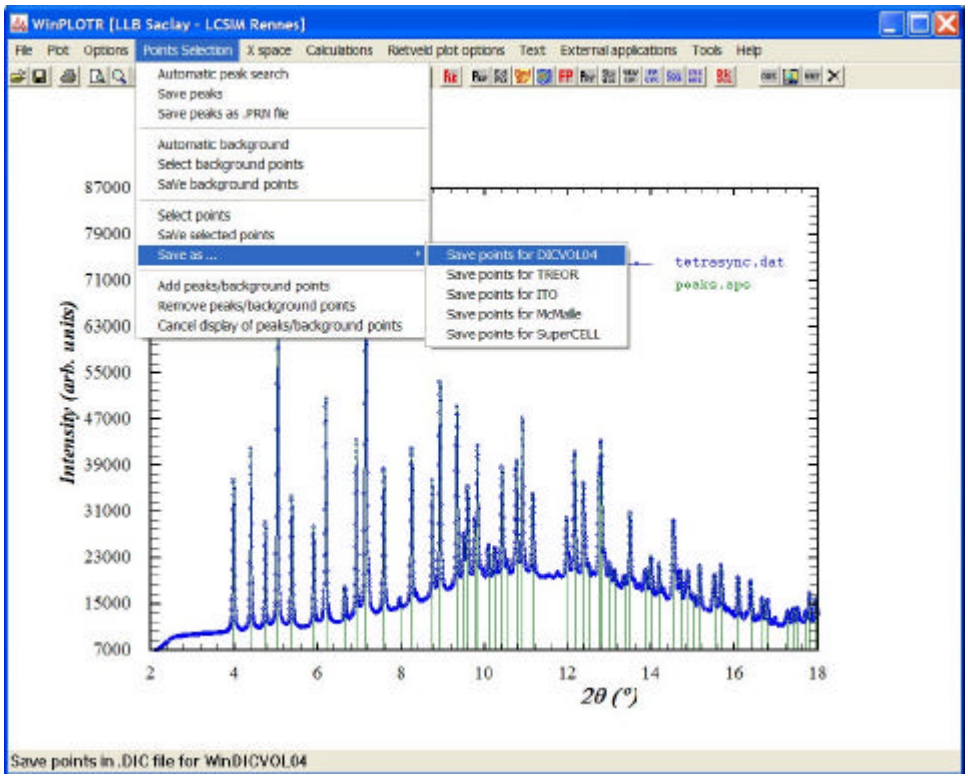
Tip-21 : TREOR runs much faster than McMaille… It is better to apply it first.

Now go back to the McMaille screen, and stop the calculations by typing K (capital letter). Maybe it had enough time to find that monoclinic solution as well ?

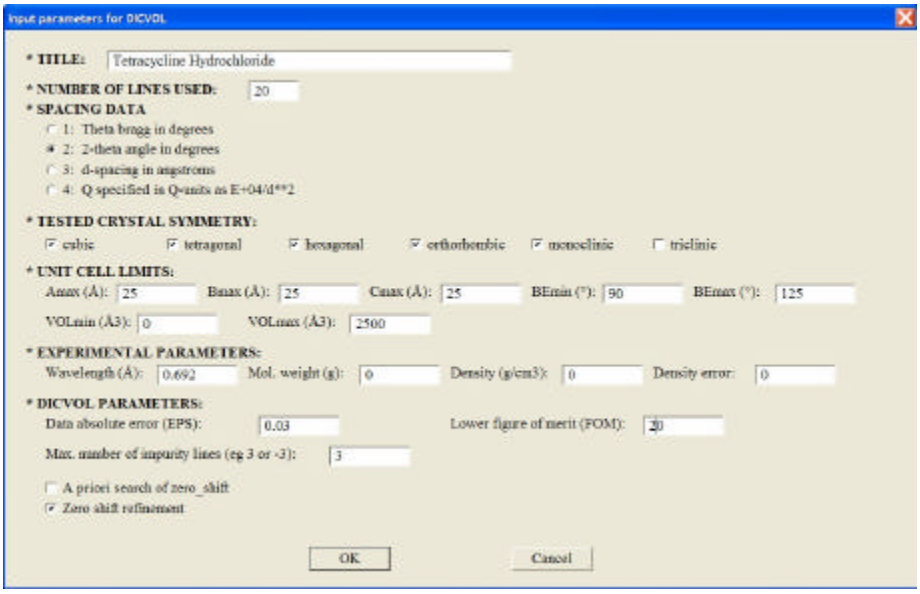
Exercise 4 – Tetracycline Hydrochloride – Synchrotron data

Use the file tetrasync.dat in PowBase (download 17.zip) with wavelength : λ = 0.692 Å. This is a capillary measurement (explaining the amorphous component).

Let us try again WinPLOTR :



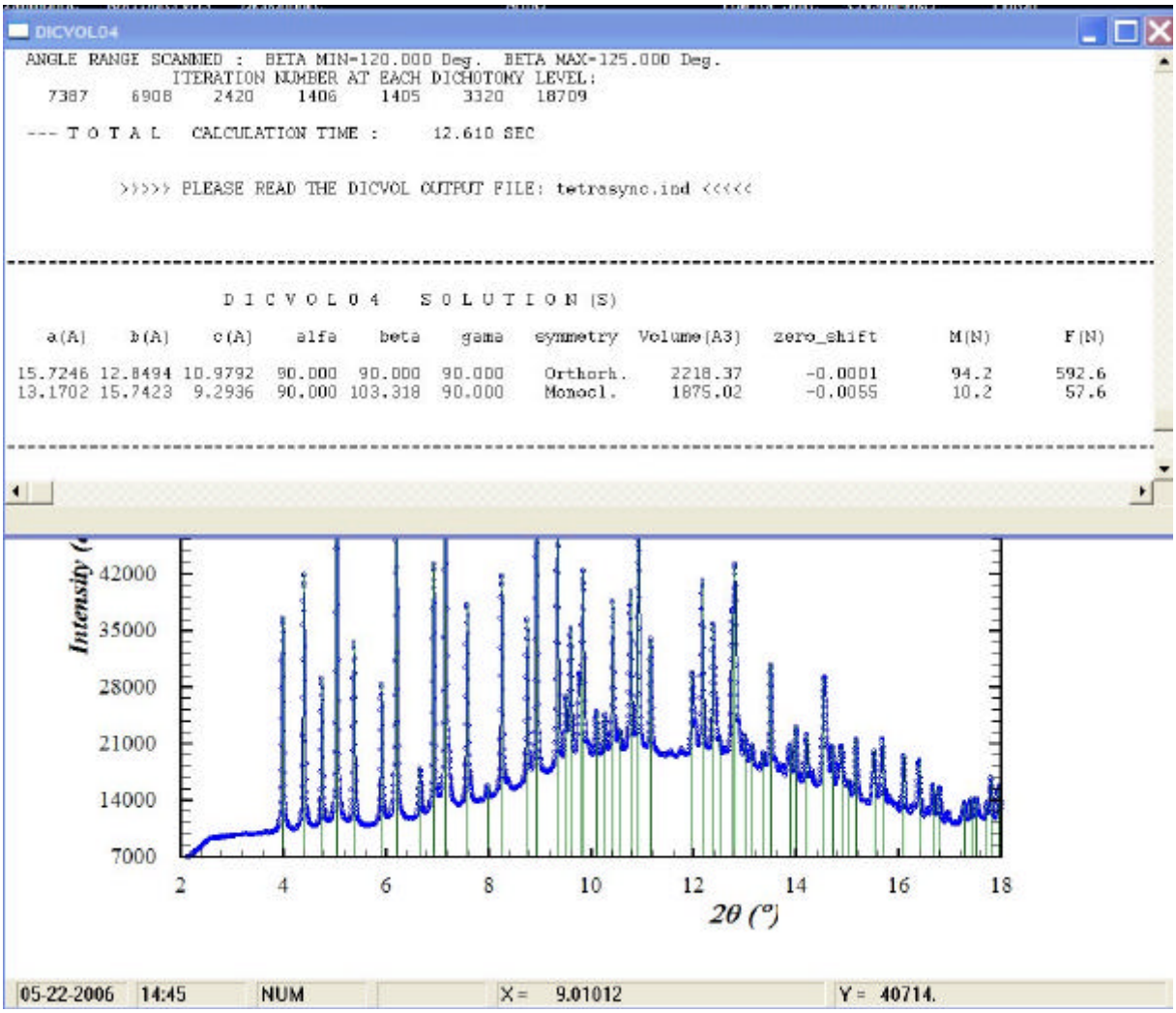
Let us save the data for DICVOL04 :



Tip-22 : DICVOL04 is generally much faster than McMaille but is a bit longer than TREOR for low symmetries (monoclinic and triclinic), in general, so, start with TREOR, then DICVOL04, then McMaille, then CRYSFIRE.

Tip-23 : Using first TREOR and DICVOL can be sufficient and convincing enough for not applying any other indexing software.

```
# Input file for WDICVOL04 (created by WinPLOTR)
# created by WinPLOTR: 22-05-2006  at 14:40:39
! DATA FILE: tetrasync.dat
! INSTRM:  0
 Tetracycline Hydrochloride
  20   2   1   1   1   1   1   0                  ! N,ITYPE,JC,JT,JH,JO,JM,JTR
  25.0  25.0  25.0 0.0   2500.0  90.0 125.0  ! AMAX, BMAX, CMAX, VOLMIN, VOLMAX,
                                                  BEMIN, BEMAX
 0.69200    0.000    0.000    0.000            ! WAVE,POIMOL,DENS,DELDEN
   0.030   20.000   3   0   1   1    ! EPS,FOM, N_IMP, ZERO_search, ZERO_refinement, ISUP
     3.98593   36431.48050   10162.20610
     4.40658   41888.94530   10565.47270
     4.75146   29037.62700   11002.17770
     5.04268   65646.02340   11368.10350
     5.38045   33498.84770   11182.45120
     5.91553   28324.10940   11602.85940
     6.20306   50488.21090   12065.03910
     6.67000   17883.95510   12214.81350
     6.93070   43182.59770   13354.95020
     7.15491   77665.66410   13321.50490
     7.58623   38302.71090   13787.45120
     7.97693   15808.31350   14298.10740
     8.25668   41806.14060   14822.07620
     8.75711   36411.66410   17468.19530
     8.93881   53459.96480   17769.21090
     9.34233   49226.54300   18679.95310
     9.51136   26989.77340   19966.95310
     9.60437   35311.25780   20394.47270
     9.77657   29438.52150   20606.06640
     9.84160   42509.96090   20488.37500
    10.09644   24946.23630   19272.28520
    10.26473   24402.79490   19494.67380
    10.42091   38604.35940   20171.22270
    10.55440   22594.34180   20487.28520
    10.76932   39757.06250   20055.43360
    10.91930   46911.87110   20154.14650
    11.16057   33845.78910   20396.83400
    11.97332   29695.01560   19972.38870
    12.16527   41121.92190   20340.83790
```



Try also with TREOR and McMaille.

Go back to previous exercises and try the programs that were not already applied. Paly with Chekcell.

Tip-24 : A simple way to deal with large cells without changing the default values of the indexing program is to give a fictitious wavelength being 2, 5 or 10 times (proteins) smaller than the real one. Then, the obtained cell parameters will be 2, 5 or 10 times smaller that the effective ones.

Additional notes

Not obtaining results with TREOR, DICVOL04 or McMaille, you may consider trying with CRYSFIRE (see session 3 of the SDPD Internet Course, or the CCP14 Web site). Consider also to use N-TREOR (either standalone or inside of EXPO2004) instead of TREOR. Visit the Indexing Benchmarks Web pages comparing the performances of many indexing programs, and see why using ITO may not be very interesting.

References

Useful Web address :

CCP14 : http://www.ccp14.ac.uk/

Indexing Benchmarks : http://sdpd.univ-lemans.fr/uppw/benchmarks/

SDPD Internet Course : http://sdpd.univ-lemans.fr/DU-SDPD/

Most recent review papers about indexing :

P.-E. Werner in "Structure Determination from Powder Diffraction Data", Edited by W.I.F David, K. Shankland, L.B. McCusker and Ch. Baerlocher, Oxford Science Publications, Chapter 7 : "Autoindexing", 2002, 118-135.

R. Shirley, "Overview of powder-indexing program algorithms (history and strenghts and weaknesses). IUCr Computing Commission Newsletter 2 (2003) 48-54.

J. Bergmann, A. Le Bail, R. Shirley and V. Zlokazov, "Renewed interest in powder diffraction data indexing", Z. Kristallogr. 219 (2004) 783-790.

Programs :

Chekcell : J. Laugier & B. Bochu, http://www.inpg.fr/LMGP (and CCP14)

CRYSFIRE : R. Shirley, "The Crysfire 2002 system for automatic powder indexing: user's manual. Lattice Press: Guilford, UK (2002).

DICVOL04: A. Boultif & D. Louër, "Powder pattern indexing with the dichotomy method", J. Appl. Crystallogr. 37 (2004) 724-732.

McMaille : A. Le Bail, "Monte Carlo indexing with McMaille", Powder Diffraction 19, 2004, 249-254. http://www.cristal.org/McMaille/

N-TREOR : A. Altomare, C. Giacovazzo, A. Guagliardi, A.G.G. Moliterni, R. Rizzi & P.-E. Werner,"New techniques for indexing : N-TREOR in EXPO", J. Appl. Crystallogr. 33 (2000) 1180-1186.

PowderX : C. Dong, "PowderX: Windows-95-based program for powder X-ray diffraction data processing", J. Appl. Crystallogr. 32 (1999) 838.

TREOR: P.-E. Werner, L. Eriksson & M. Westdahl, "TREOR, a semi-exhaustive trial-and-error powder indexing program for all symmetries", J. Appl. Cryst. 18 (1985) 367-370.

WinPLOTR : T. Roisnel & J. Rodriguez-Carvajal,
http://www-llb.cea.fr/fullweb/winplotr/winplotr.htm