

Maclage par non-mérohédrie : utilisation de la suite EVALCCD (Nonius)

T. Roisnel

Centre de Diffractométrie X (**CDIFX**)
Sciences Chimiques de Rennes
www.cdifx.univ-rennes1.fr

Maclage par non-mérohédrie

Cas d'un cristal multiple : superposition de 2 sous-réseaux

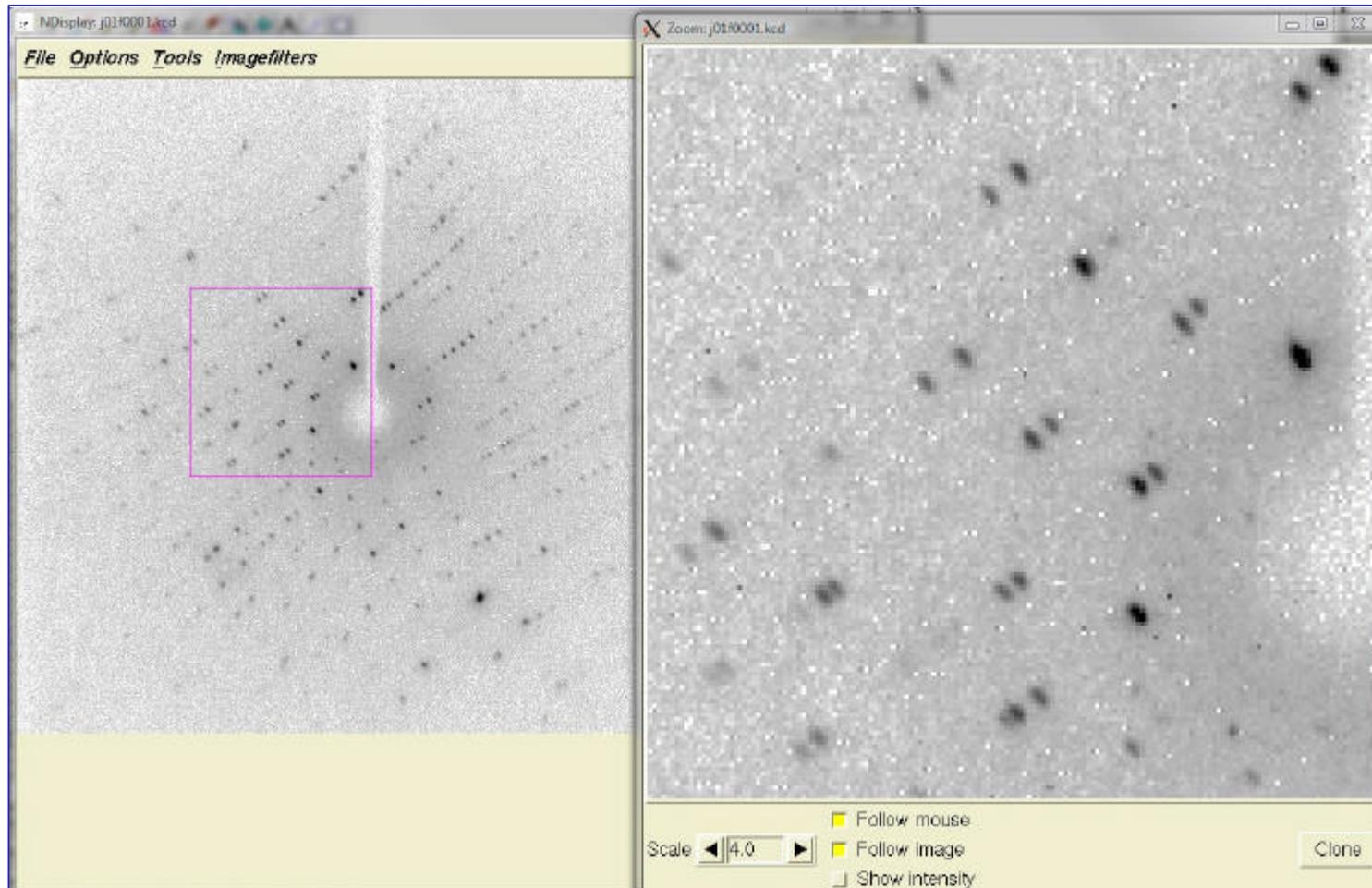
- . réflexions propres à #1
 - . réflexions propres à #2
 - . réflexions communes à #1 et #2 (superposition partielle ou complète)
-
- . Résolution structurale à partir des réflexions de l'individu majoritaire
 - . Affinement final à partir d'un jeu de données contenant les 2 contributions

Omega scan



Essai d'indexation avec DENZO : pas de succès

Phi-chi scan



Indexation avec NDIRAX

Phi-chi scan / NDIRAX : indexation #1

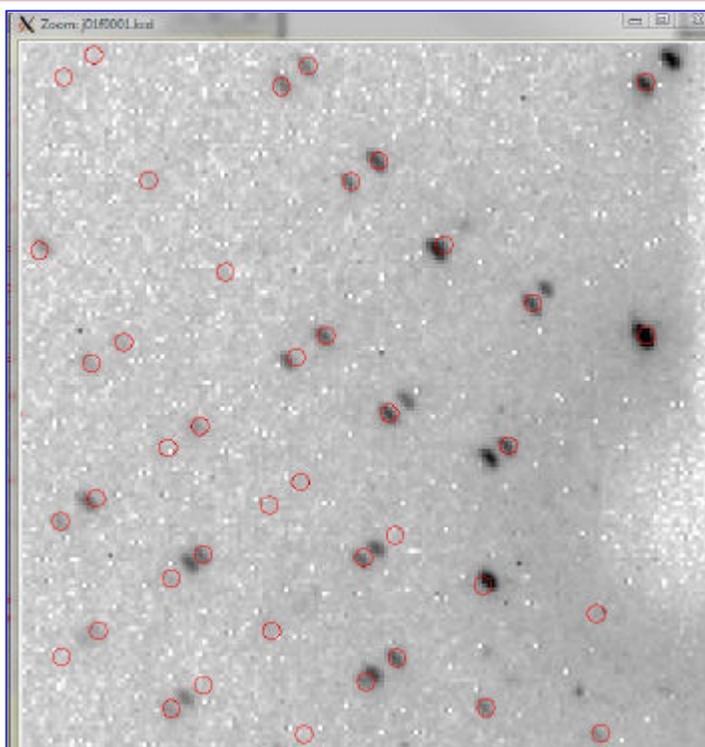
Chosen solution

Acceptance level 63; solution #2
Goodness of fit for 83 accepted reflections between 273 and 3089
for 52 rejected reflections between 44 and 254

Cell Reduction

Coinciding axes criterion (degrees)

```
{Input cell   : a=9.1414 b=12.5009 c=21.8650 alpha=86.961 beta=80.025 gamma=73.803 P
Reduced cell  : a=9.1414 b=12.5009 c=21.8650 alpha=86.961 beta=80.025 gamma=73.803
Conventional : a=9.1414 b=12.5009 c=21.8650 alpha=86.961 beta=80.025 gamma=73.803 P
Volume       : 2363.17; System: triclinic; Point group: -1}
```



save as ndirax1.rmat

Phi-chi scan / NDIRAX : indexation #2

Restart using nonfitting spots

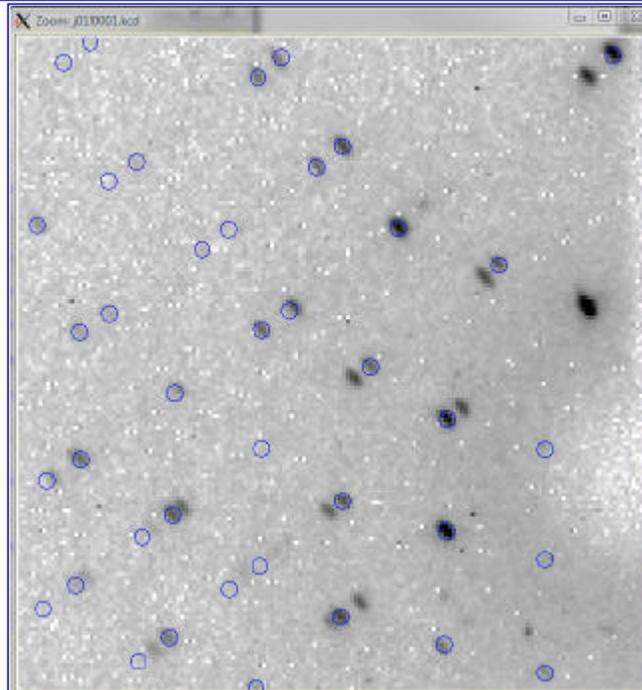
Chosen solution

Acceptance level 34; solution #10
Goodness of fit for 81 accepted reflections between 267 and 2627
for 54 rejected reflections between 45 and 258

Cell Reduction

Coinciding axes criterion (degrees) ◀ 0.20 ▶

```
{Input cell : a=9.1408 b=12.5017 c=21.7958 alpha=86.886 beta=80.092 gamma=73.812 P
Reduced cell : a=9.1408 b=12.5017 c=21.7958 alpha=86.886 beta=80.092 gamma=73.812
Conventional : a=9.1408 b=12.5017 c=21.7958 alpha=86.886 beta=80.092 gamma=73.812 P
Volume : 2356.24; System: triclinic; Point group: -1}
```



save as ndirax2.rmat

Phi-chi scan / NDIRAX : indexation #1 + #2

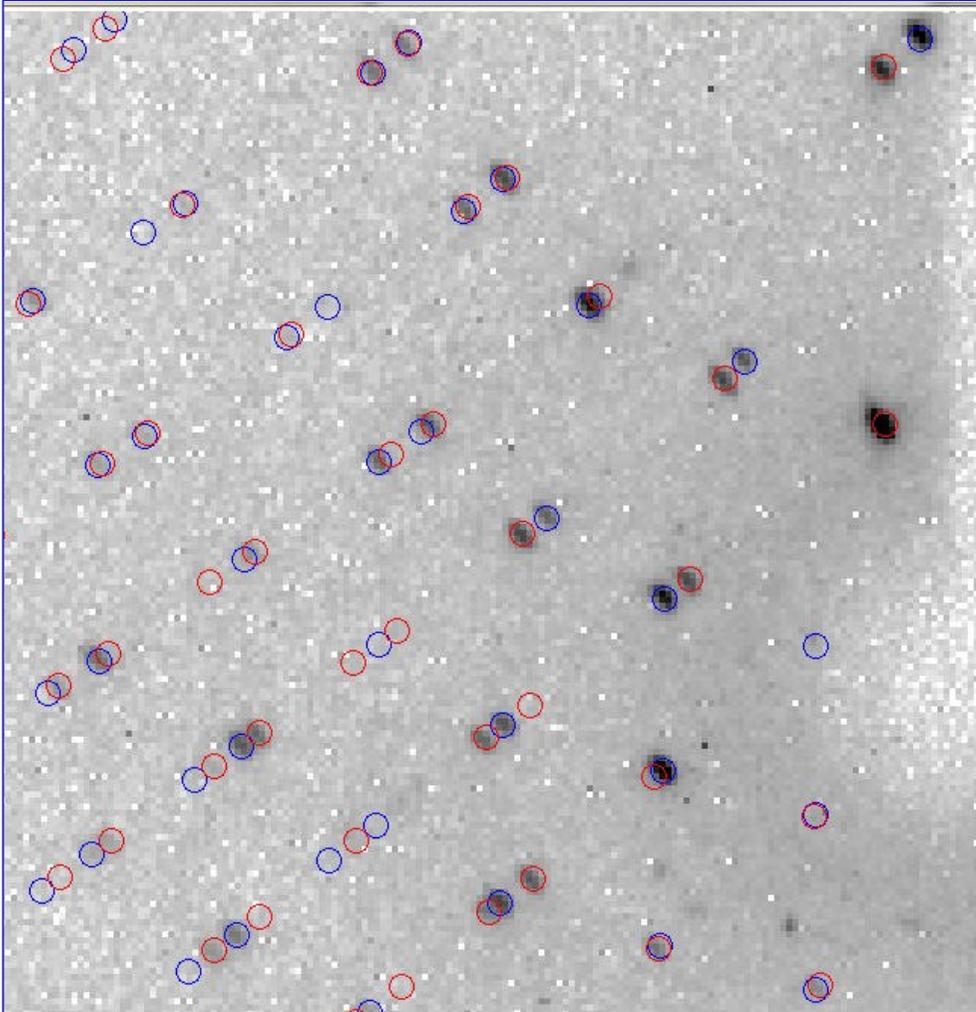


Diagramme de diffraction =
superposition de diverses
contributions:

- . réflexions individu **bleu**
- . réflexions individu **rouge**
- . taches communes

Programme PRECESSION : reconstruction de plans de l'espace réciproque

```
> precession hk0 hk1 hk2 hk3 ... s*.kcd ndirax1.rmat
```

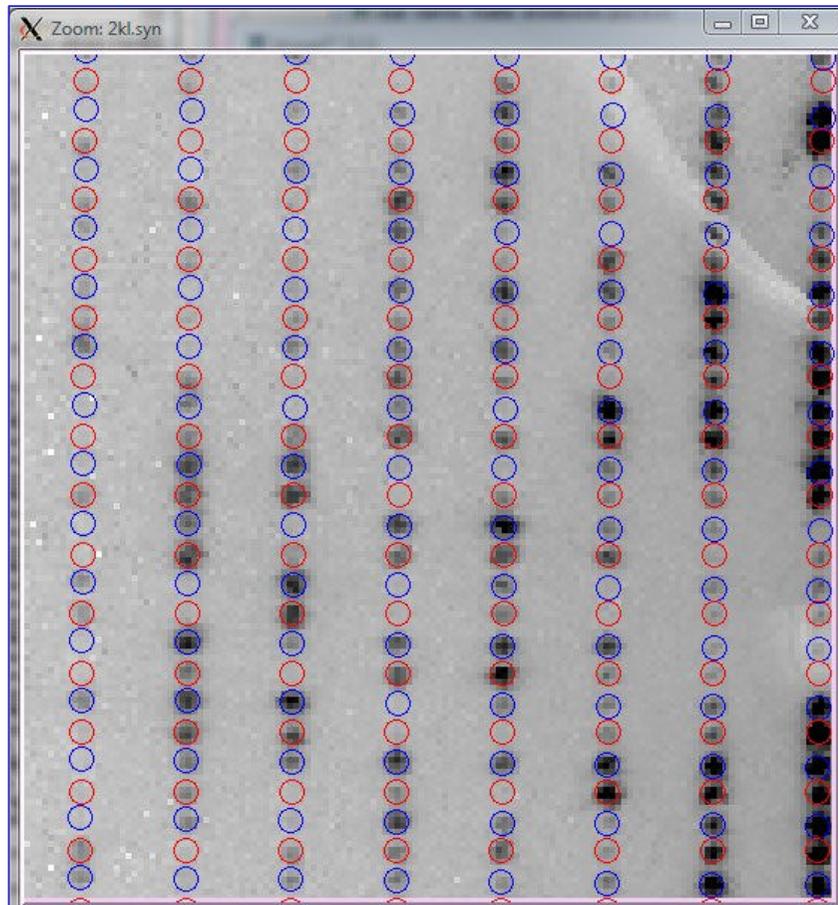


Diagramme de diffraction =
superposition de diverses
contributions:

- . réflexions individu **bleu**
- . réflexions individu **rouge**
- . taches communes

EvalCCD : intégration individu #1

Fichier.hkl (format HKLF 4)

Rint ~ 6%

Formule brute : C₂₆ H₂₆ Cl N₃ O₄

Groupe spatial : P -1

Résolution structurale : ~~SIR92~~
~~SIR97~~
~~SIR2002/SIR2004~~
~~SHELXS~~

Superflip

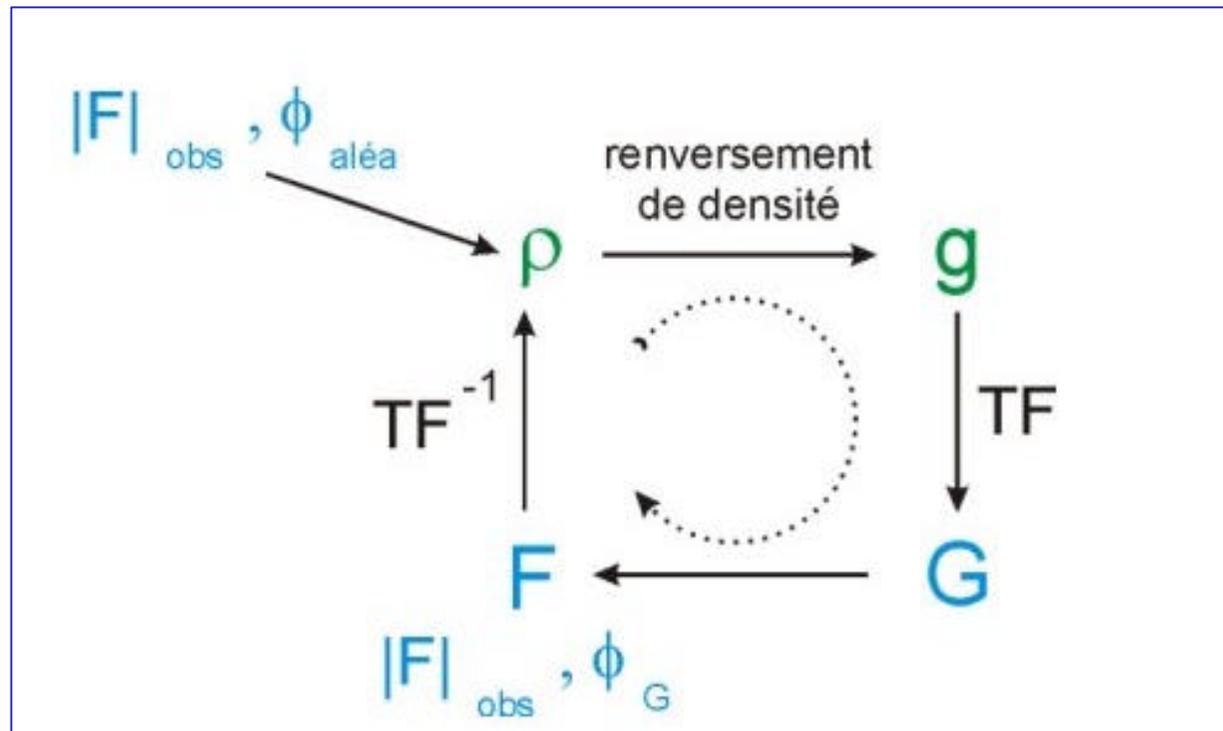
Superflip (charge flipping algorithm)

L. Palatinus, G. Chapuis (Lausanne)

<http://superspace.epfl.ch/superflip/>

http://www.crm2.uhp-nancy.fr/crm2/fr/labo/pages_perso/Aubert/ChargeFl/chargefl.html

$$F_H = T.F.(\text{densité électronique ?})$$



EDMA : analyse de la densité électronique finale

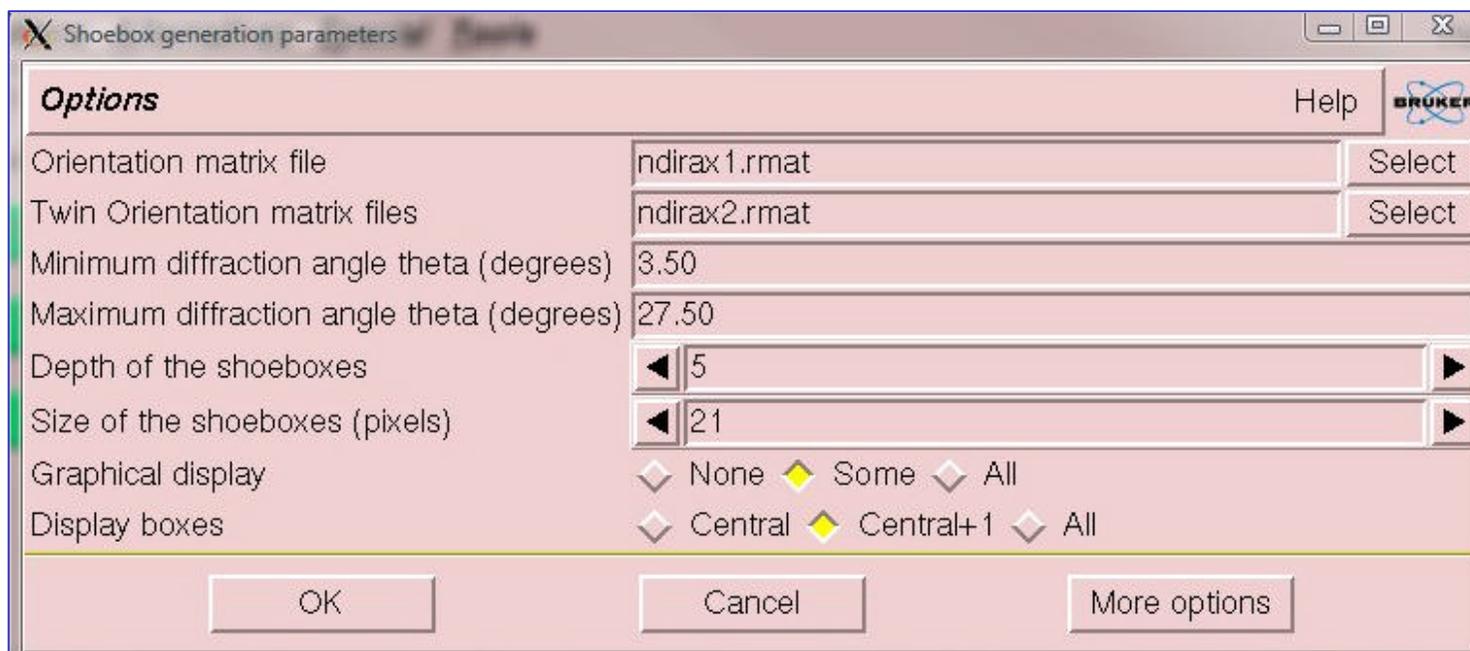
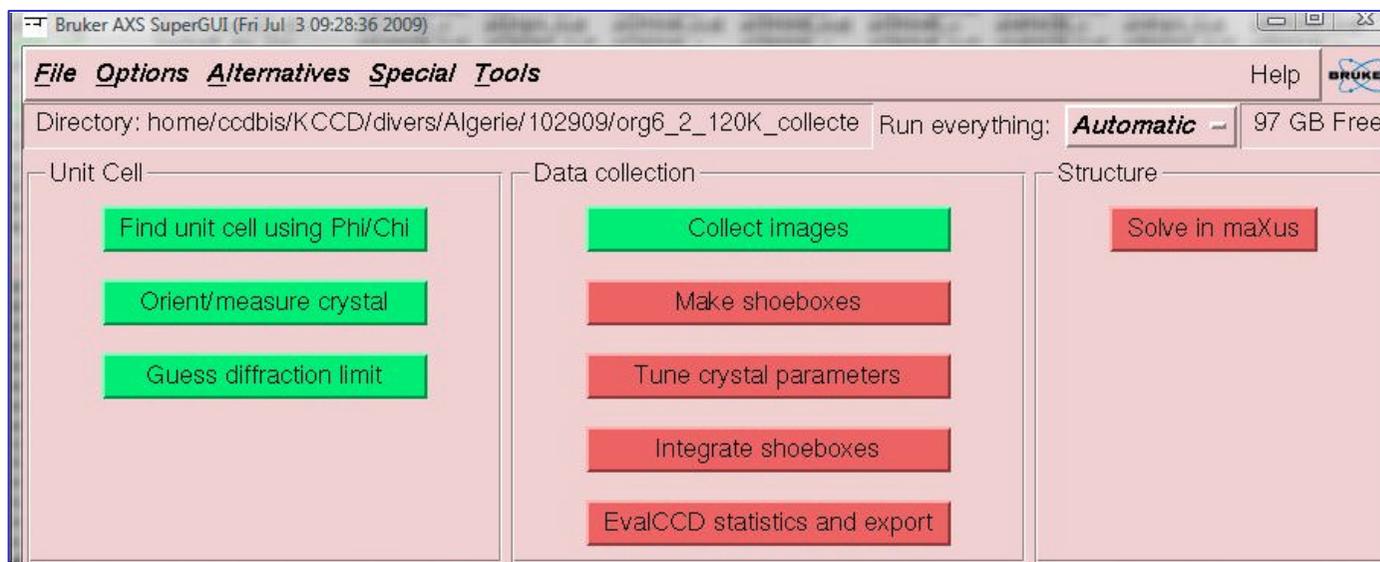
Affinement structural (SHELXL)

R1 ~28 %

Most Disagreeable Reflections (* if suppressed or used for Rfree)

h	k	l	Fo ²	Fc ²	Delta(F ²)/esd	Fc/Fc(max)	Resolution(Å)
-1	-2	6	1662.81	9.32	4.18	0.014	2.89
1	6	12	3028.58	0.03	4.17	0.001	1.39
-6	-3	13	509.20	0.03	4.17	0.001	1.02
1	5	12	1347.07	0.44	4.16	0.003	1.49
-6	-4	11	997.76	0.13	4.15	0.002	1.09
0	7	12	882.17	0.19	4.13	0.002	1.24
1	7	14	10169.39	55.38	4.12	0.035	1.19
4	-4	8	1099.65	4.66	4.10	0.010	1.43
-6	4	2	286.35	0.65	4.09	0.004	1.16
1	6	8	3945.05	3.39	4.08	0.009	1.68
-6	-7	1	2087.82	0.20	4.07	0.002	1.28
6	0	3	778.18	5.18	4.07	0.011	1.46
0	3	9	605.76	0.24	4.07	0.002	2.06
6	8	0	1661.66	2.75	4.06	0.008	1.22

EvalCCD : intégration individus #1 + #2



EvalCCD / export : création d'un fichier .hklf5

The image displays two software windows from Bruker AXS. The top window is 'Bruker AXS SuperGUI' (Fri Jul 3 09:28:36 2009) with a menu bar containing 'File', 'Options', 'Alternatives', 'Special', and 'Tools'. The directory is 'home/ccdbis/KCCCD/divers/Algerie/102909/org6_2_120K_collecte' and 'Run everything:' is set to 'Automatic'. It features three panels: 'Unit Cell' with buttons for 'Find unit cell using Phi/Chi', 'Orient/measure crystal', and 'Guess diffraction limit'; 'Data collection' with buttons for 'Collect images', 'Make shoeboxes', 'Tune crystal parameters', 'Integrate shoeboxes', and 'EvalCCD statistics and export'; and 'Structure' with a 'Solve in maXus' button.

The bottom window is 'Nanny (ndirax1-ndirax2/final.y)' with a menu bar containing 'File', 'Options', 'Filters', and 'Graphics'. It shows file operations: 'Load .y file...', 'Write nanny.hkl', 'Write nanny.hklf5', 'Write jana.hkl', 'Write shelx.sad', 'Write shelx.sam', and 'Quit'. Three sliders are visible: the first is at 0.025 (6 too negative reflections, 1588 weak reflections, 70 bad background reflections); the second is at 2.5 (6 too negative reflections, 1588 weak reflections, 70 bad background reflections); the third is at 1000.0 (no reflections rejected). Statistics are displayed: Rsym=0.02, Rmeas=0.028, Rpim=0.02, Chi2=3.791, nRsym=226, Unique=113, <I>=56.487, <s>=2.317, <I/s>=15.572, <I/<s>=24.377, nMean=1230. A list of filters is shown on the left: Total (checked), by Shell, by Intensity, by # Equivs, by Scan, by Shoebox, and by Point group.

Nanny : création d'un fichier hklf 5

Format : h k l F2 sig m cos_dir(1:6)

m : indicateur d'appartenance de la réflexion

- m = -2 : réflexion commune à #1 + # 2 (2 indexations, 1 seul F2)
- m = 1 : réflexion individu #1
- m = 2 : réflexion individu #2

```
====>
*** Top of File ***
-5 -1 20 189.23 18.01 -2 0.41534-0.15320-0.93621 0.87837 0.28052 0.44257
 5  1 24 189.23 18.01  1 0.41534-0.15320-0.93621 0.87837 0.28052 0.44257
 3  2 15 471.09 16.17  1 0.41534-0.28398-0.93621 0.98410 0.28052 0.17304
 1 -4 -13 120.90  5.97  1 0.41556-0.19685-0.93596 0.67874 0.28105-0.72366
 1 -7 -17 258.97 11.68  1 0.41566-0.12612-0.93584 0.50158 0.28131-0.85572
-4 -7 -11  19.02  5.02  1 0.41572-0.57034-0.93578 0.61364 0.28144-0.58888
 9  2 19  5.20 13.01  1 0.41598 0.18545-0.93547 0.84729 0.28209 0.22110
-10 -2  7 347.57 19.87 -2 0.41598 0.28950-0.93547 0.82535 0.28209 0.07551
 10  2 15 347.57 19.87  1 0.41598 0.28950-0.93547 0.82535 0.28209 0.07551
-4 -1 20  36.67 17.19 -2 0.41598-0.23033-0.93547 0.90029 0.28209 0.42173
 4  1 23  36.67 17.19  1 0.41598-0.23033-0.93547 0.90029 0.28209 0.42173
-1 -1 14 417.68 14.37 -2 0.41614-0.43267-0.93528 0.96924 0.28249 0.19875
 1  1 15 417.68 14.37  1 0.41614-0.43267-0.93528 0.96924 0.28249 0.19875
 3  0 25 148.63 13.48  1 0.41614-0.30728-0.93528 0.86327 0.28249 0.50122
-7 -1 19 141.97 30.24 -2 0.41625 0.00438-0.93516 0.83205 0.28275 0.44586
 7  1 25 141.97 30.24  1 0.41625 0.00438-0.93516 0.83205 0.28275 0.44586
-1  2 26  73.72 33.67 -2 0.41646-0.45022-0.93491 0.78958 0.28327 0.59433
 1 -2 27  73.72 33.67  1 0.41646-0.45022-0.93491 0.78958 0.28327 0.59433
 3  7 -10  69.49 15.43 -2 0.41652-0.47791-0.93485 0.59043 0.28340-0.67073
-3 -7 -13  69.49 15.43  1 0.41652-0.47791-0.93485 0.59043 0.28340-0.67073
-3 -3 -3 237.39  7.92  1 0.41657-0.59786-0.93479 0.82558 0.28353-0.34092
-2 -1 17 468.39 17.75 -2 0.41657-0.37301-0.93479 0.94548 0.28353 0.31590
 2  1 19 468.39 17.75  1 0.41657-0.37301-0.93479 0.94548 0.28353 0.31590
```

Affinement structural (SHELXL) en tenant compte des 2 individus

- . Modèle structural déterminé au préalable avec #1
- . Affinement de la proportion de #2 (BASF)
- . HKLF 5

R1 ~8.2 %

BASF ~0.43

