

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

T. Roisnel

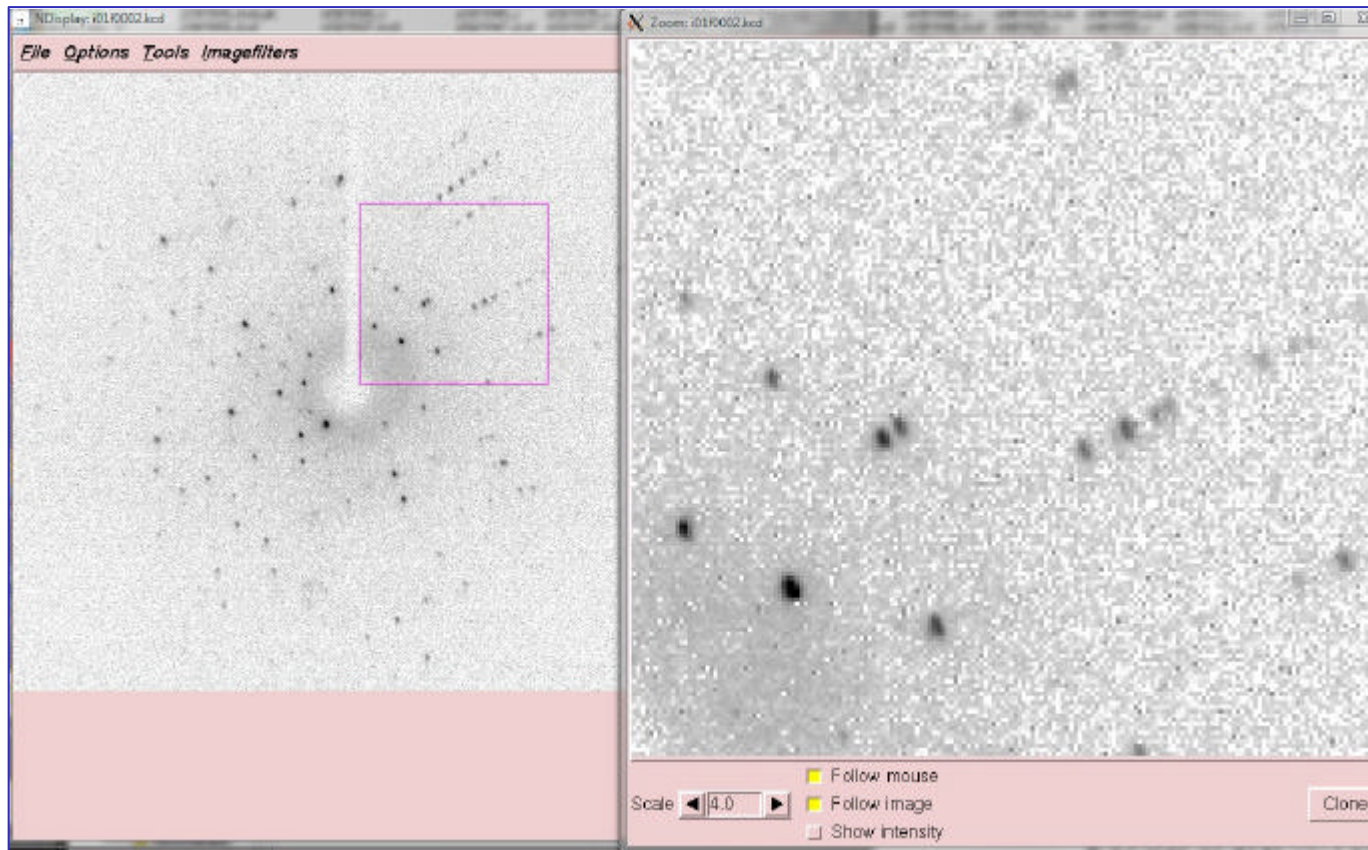
Centre de Diffractométrie X (**CDIFX**)
Sciences Chimiques de Rennes
www.cdifx.univ-rennes1.fr

Maclage par non-mérohédrie

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

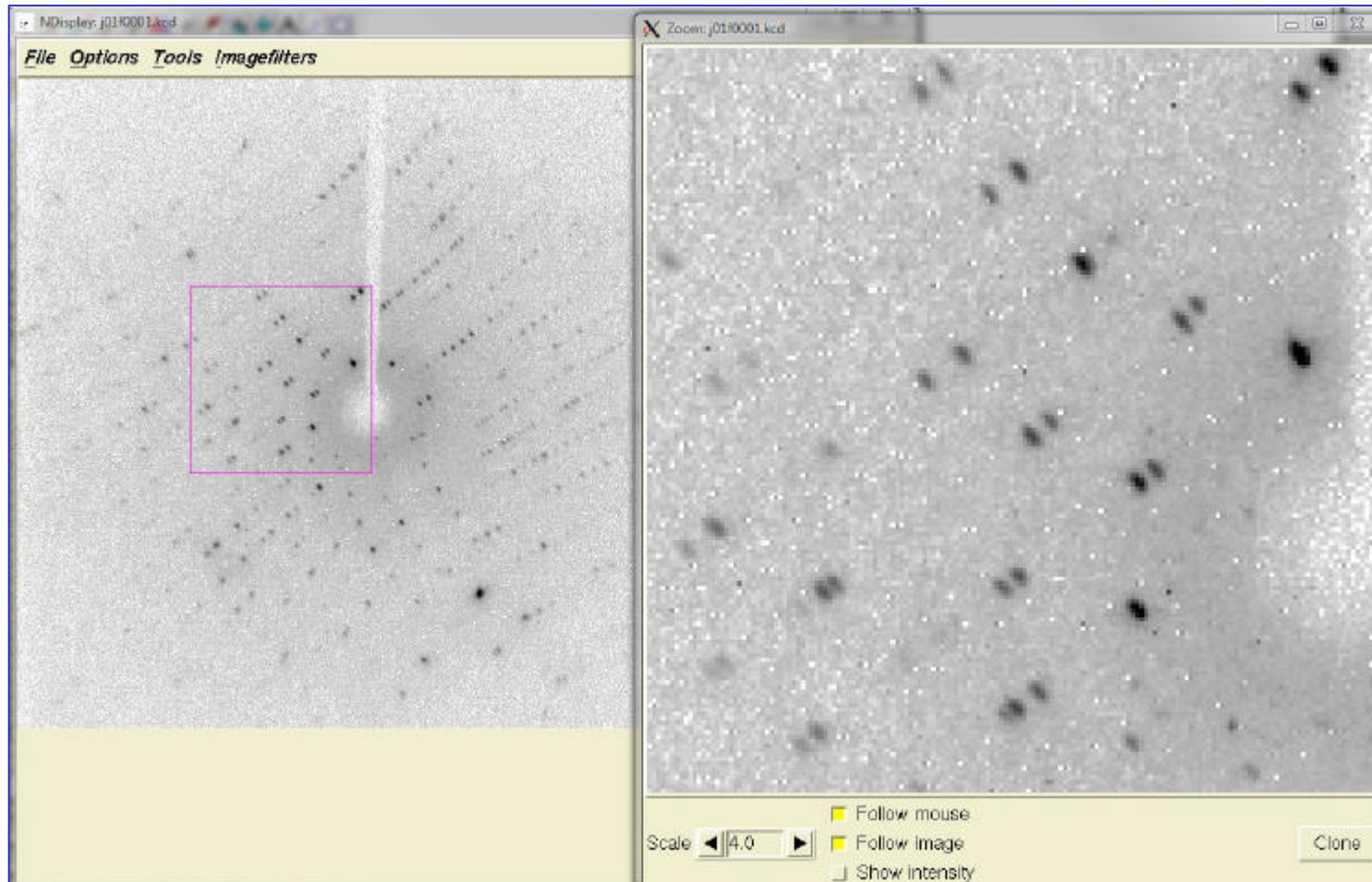
- . réflexions propres à #1
 - . réflexions propres à #2
 - . réflexions communes à #1 et #2 (superposition partielle ou complète)
-
- . Résolution structurale à partir des réflexions de l'individu majoritaire
 - . Affinement final à partir d'un jeu de données contenant les 2 contributions

Omega scan



Essai d'indexation avec DENZO : pas de succès

Phi-chi scan



Indexation avec NDIRAX

Phi-chi scan / NDIRAX : indexation #1

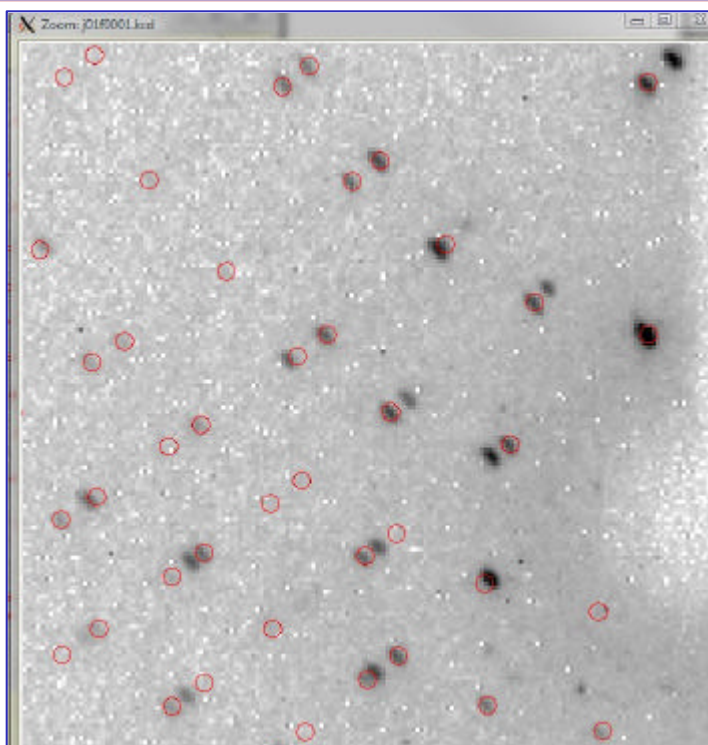
Chosen solution

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

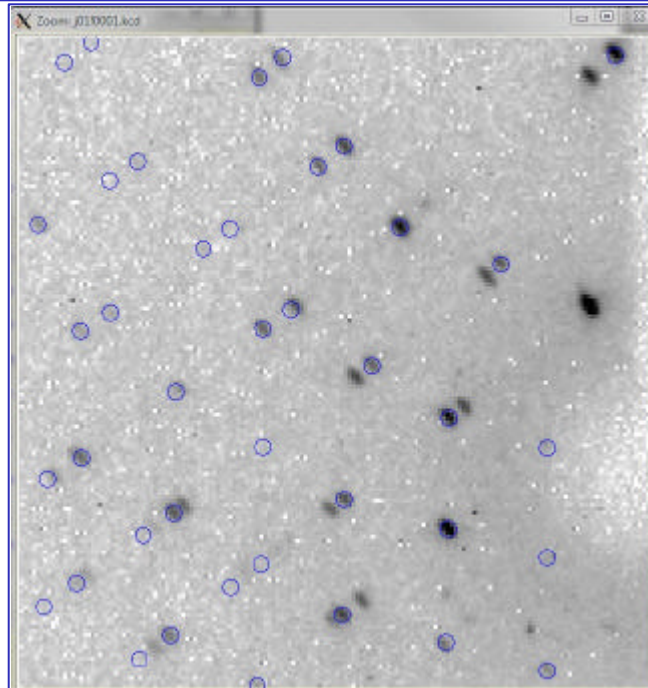
Chosen solution

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

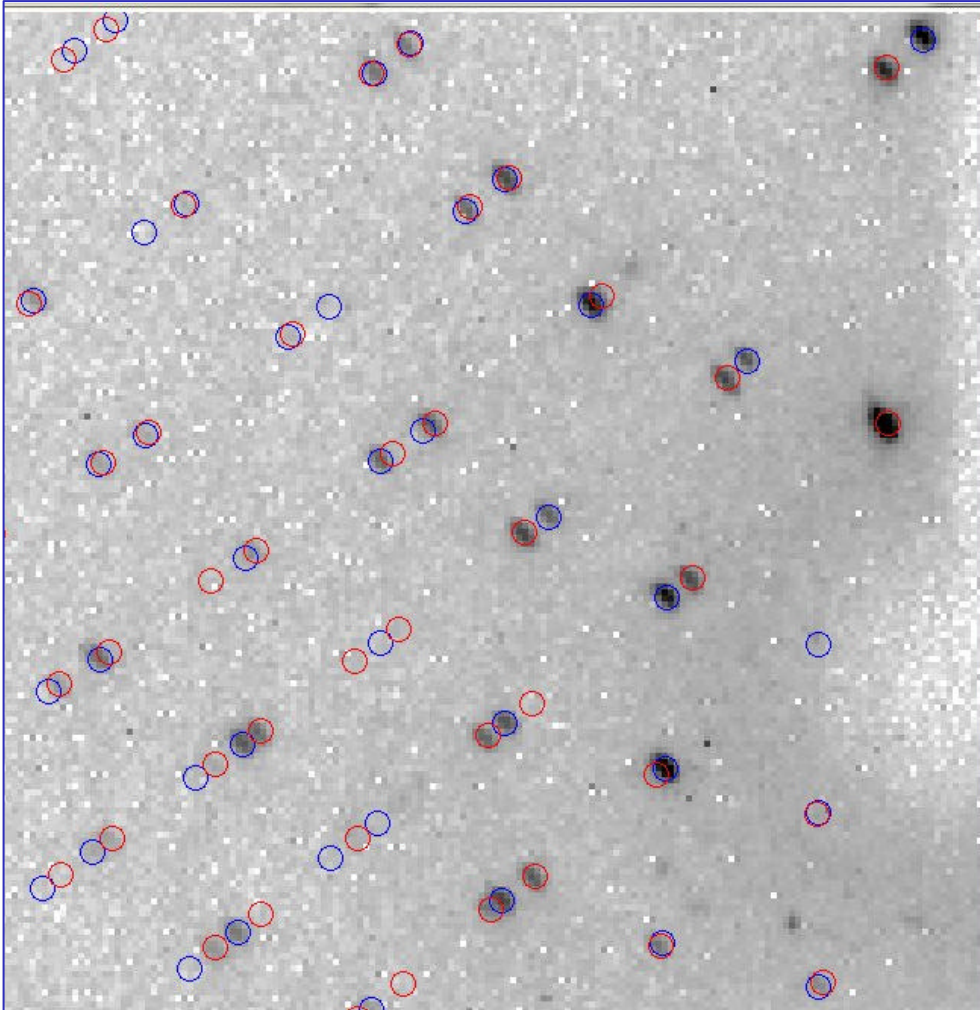


Diagramme de diffraction =
superposition de diverses
contributions:

- . 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
```

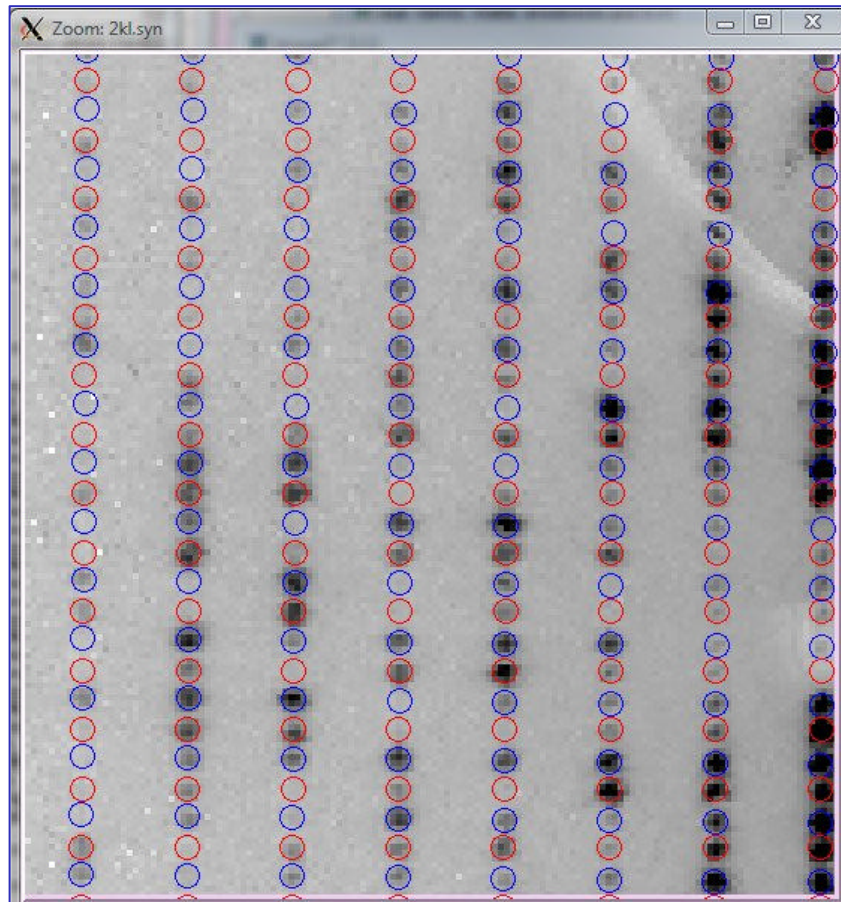


Diagramme de diffraction =
superposition de diverses
contributions:

- . 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

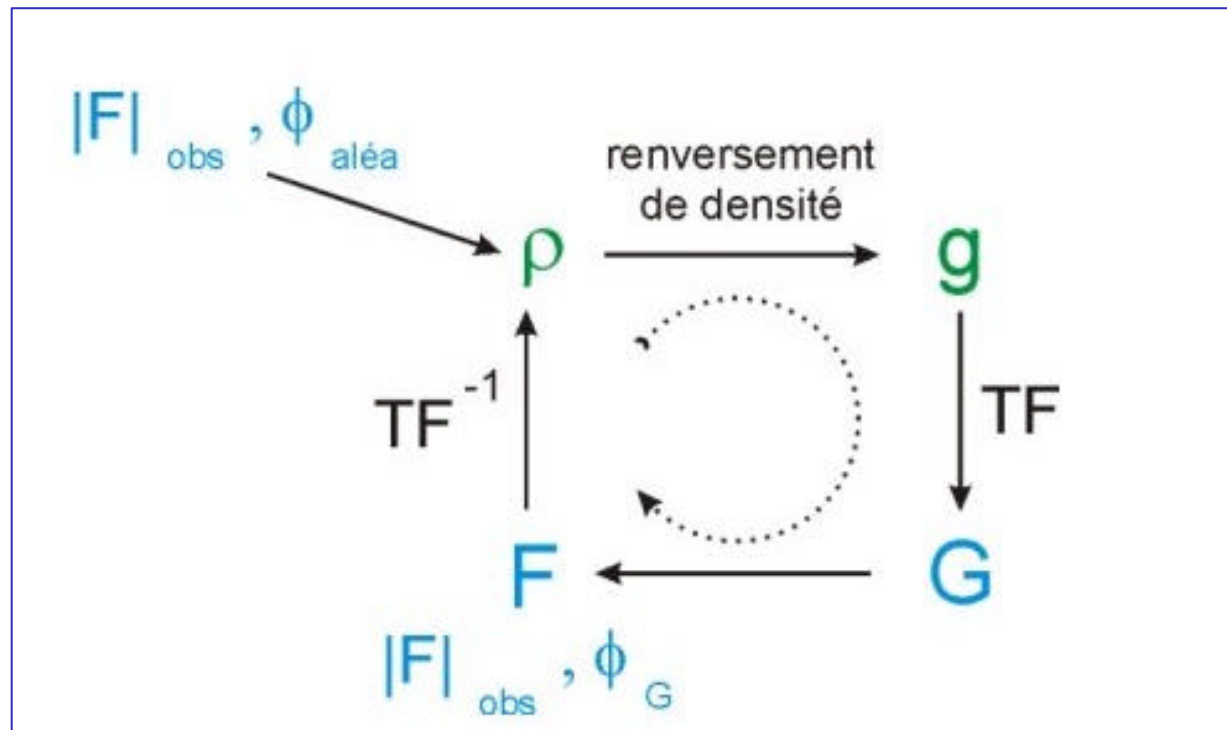
Superflip (charge flipping algorithm)

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

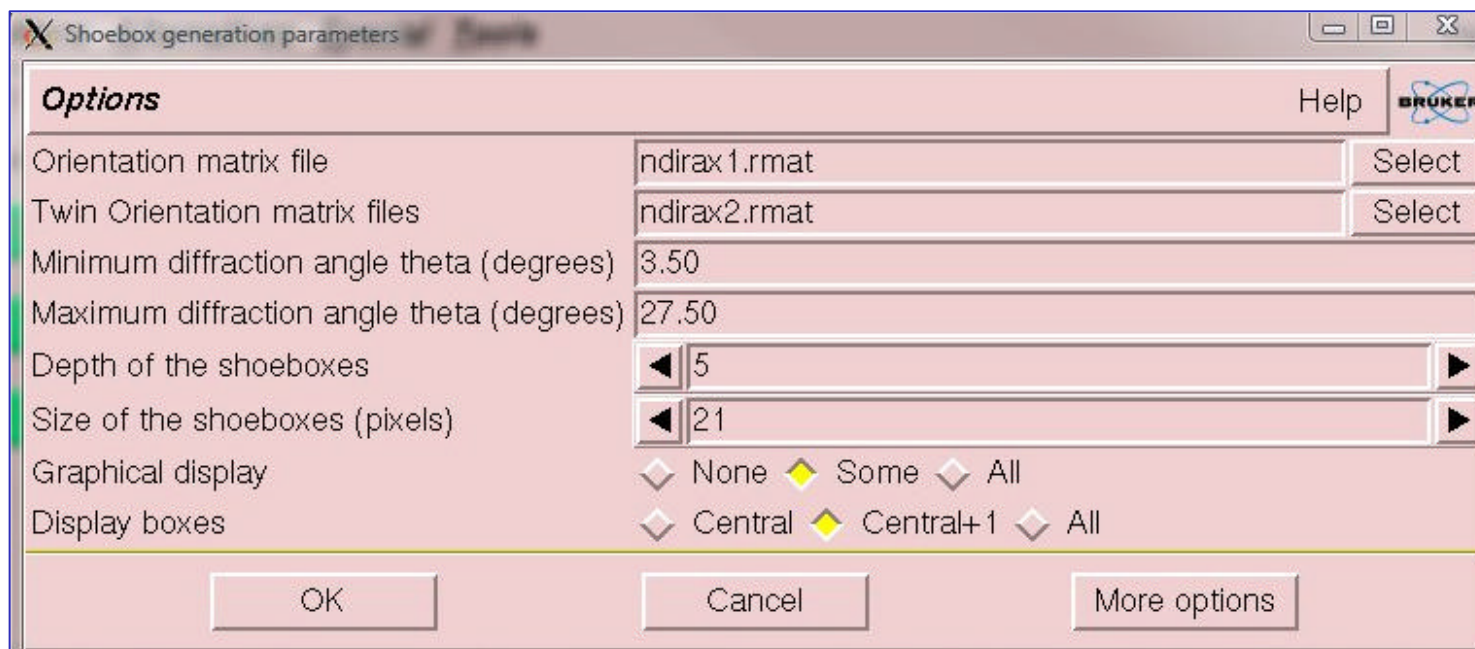
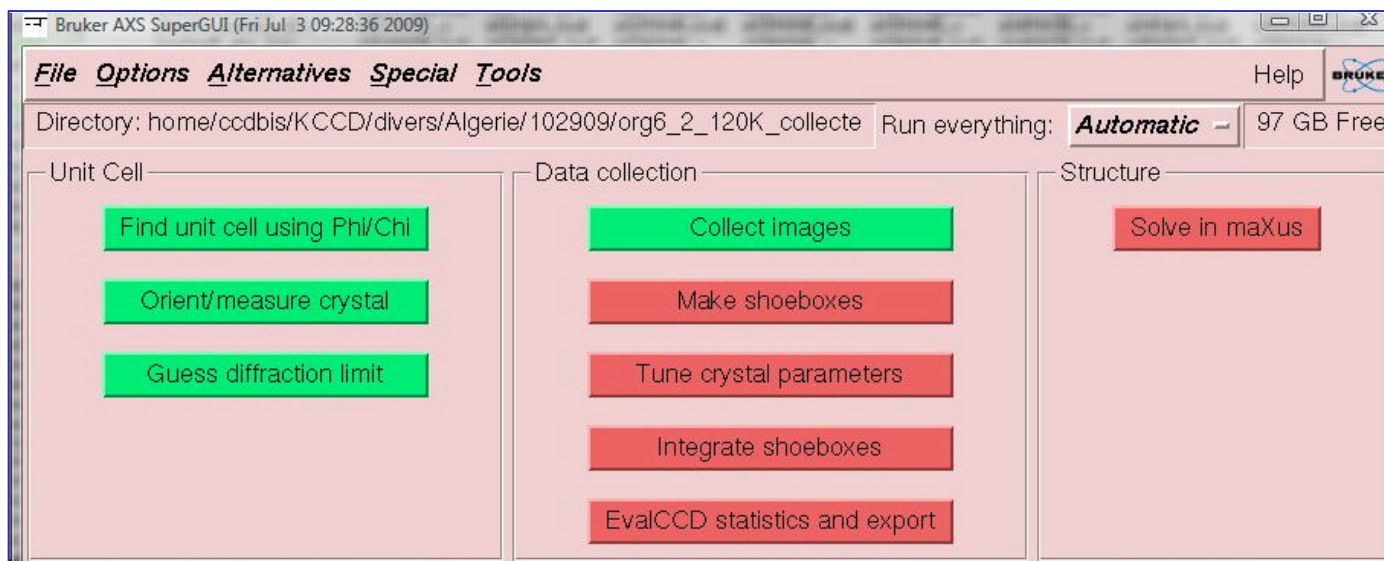
Affinement structural (SHELXL)

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 shows two windows from the Bruker AXS SuperGUI software. The top window is titled "Bruker AXS SuperGUI (Fri Jul 3 09:28:36 2009)" and has a menu bar with "File", "Options", "Alternatives", "Special", and "Tools". The directory is "home/ccdbis/KCCCD/divers/Algerie/102909/org6_2_120K_collecte" and the run mode is "Automatic". It features three main sections: "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 titled "Nanny (ndirax1-ndirax2/final.y)" and has a menu bar with "File", "Options", "Filters", and "Graphics". It displays a list of file operations: "Load .y file...", "Write nanny.hkl", "Write nanny.hklf5", "Write jana.hkl", "Write shex.sad", "Write shex.sam", and "Quit". Below this list are three sliders with values 0.025, 2.5, and 1000.0, each with associated text: "6 too negative reflections (allowed) 1588 weak reflections (allowed) 70 bad background reflections (70 set on quality. 0 with fixed badbg flag) (forbidden)", "6 too negative reflections 1588 weak reflections 70 bad background reflections (70 set on quality. 0 with fixed badbg flag)", and "no reflections rejected". At the bottom, it shows statistics: "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 tree view on the left shows a hierarchy of data points: "Total", "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

