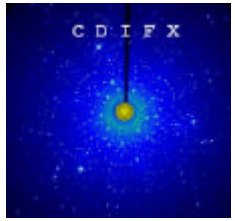

Maclage par non-mérohédrie :

utilisation de la suite EVALCCD (Nonius)

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Sciences Chimiques de Rennes
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Maclage par non-mérohédrie

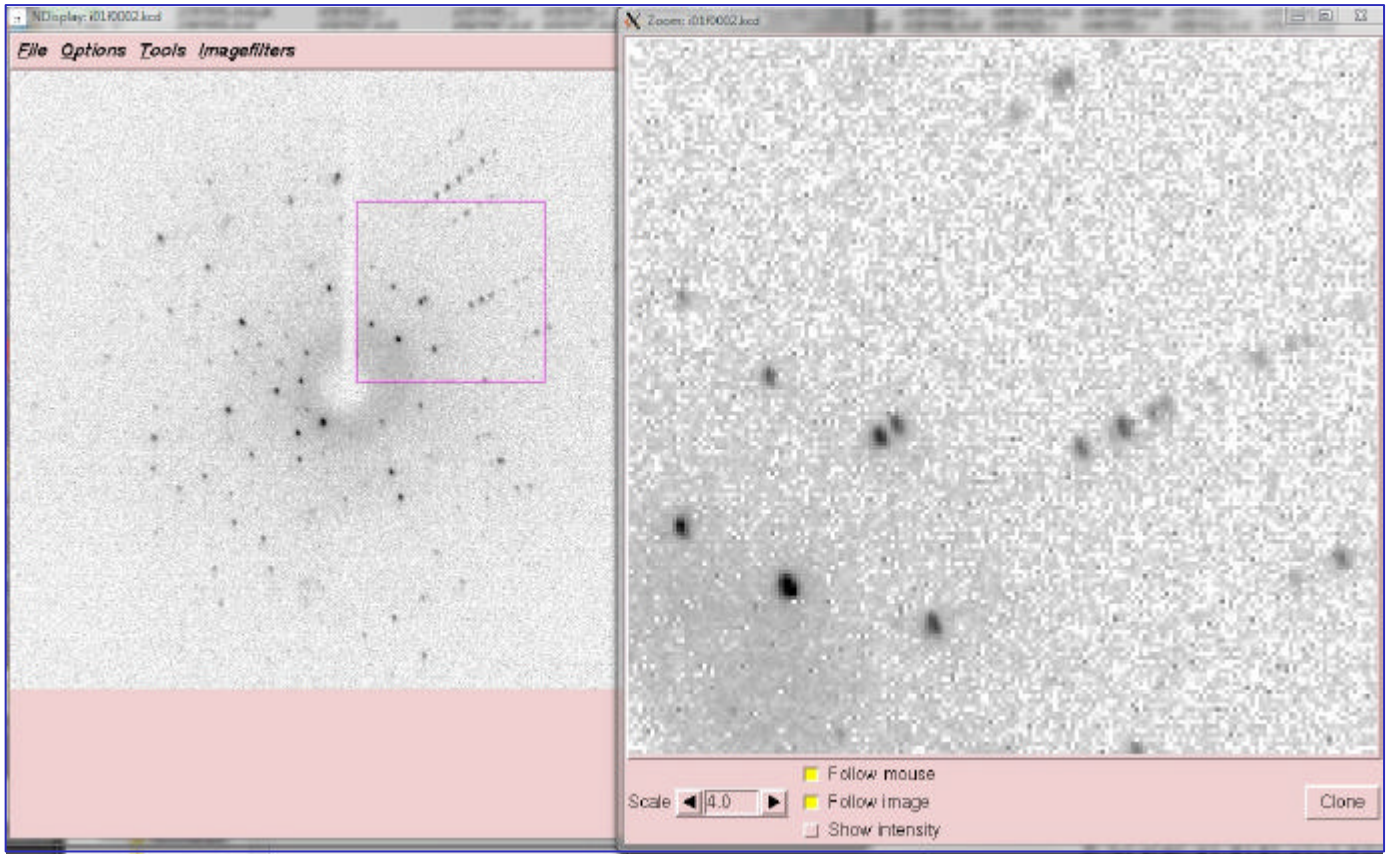
Cas d'un cristal multiple : superposition de 2 sous-réseaux

. 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

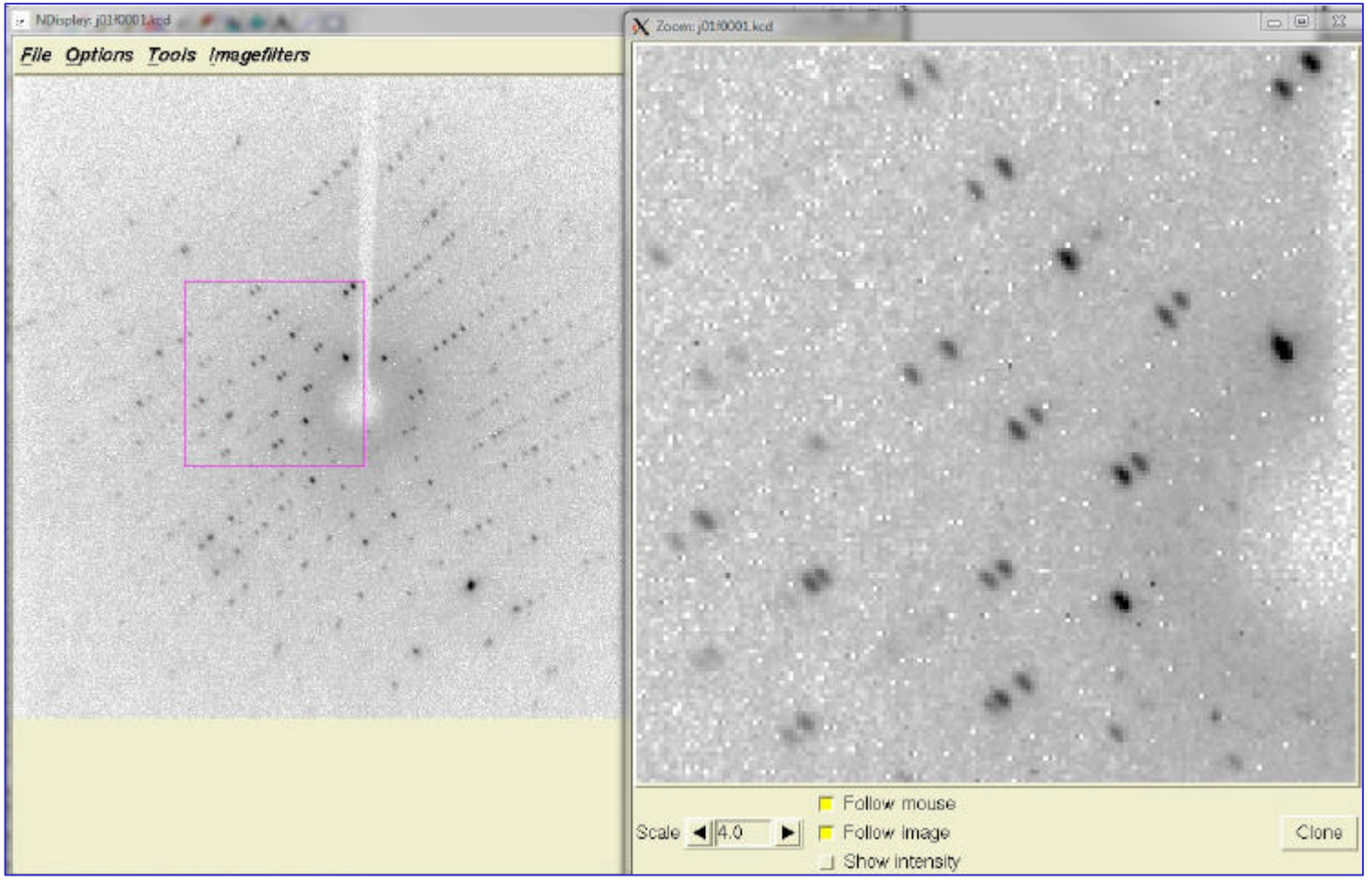
. Affinement final à partir d'un jeu de données contenant les 2 contributions

Omega scan



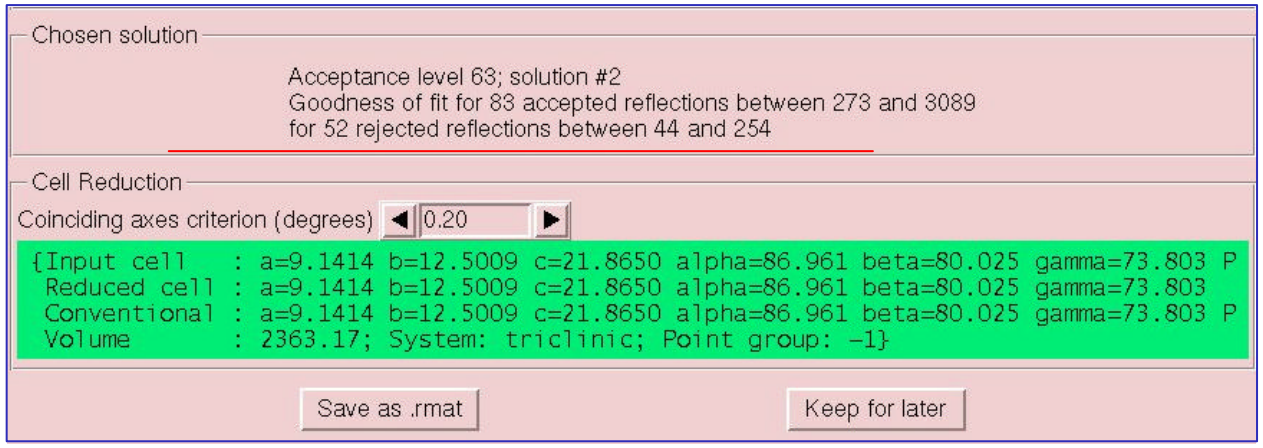
Essai d'indexation avec DENZO : pas de succès

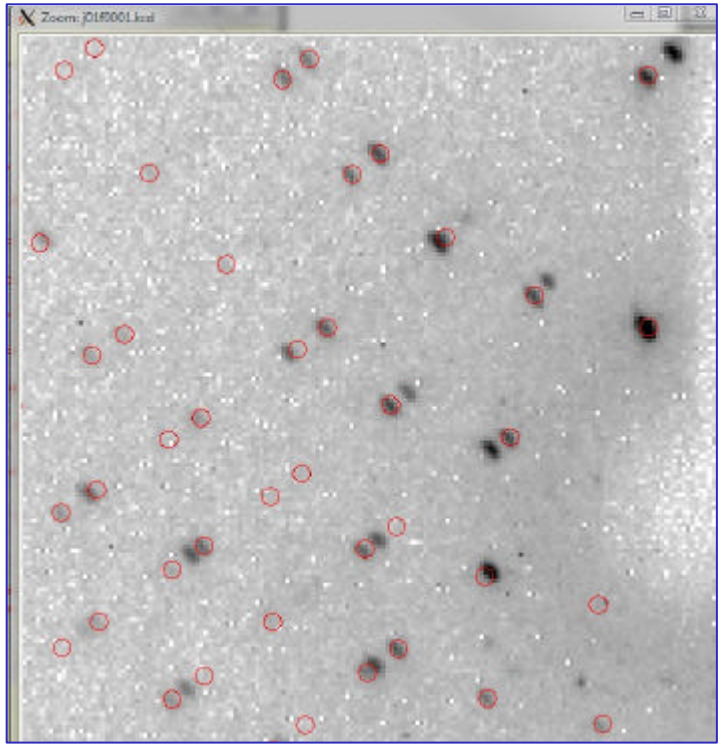
Phi-chi scan



Indexation avec NDIRAX

Phi-chi scan / NDIRAX : indexation #1





save as `ndirax1.rmat`

Phi-chi scan / NDIRAX : indexation #2

Restart using nonfitting spots

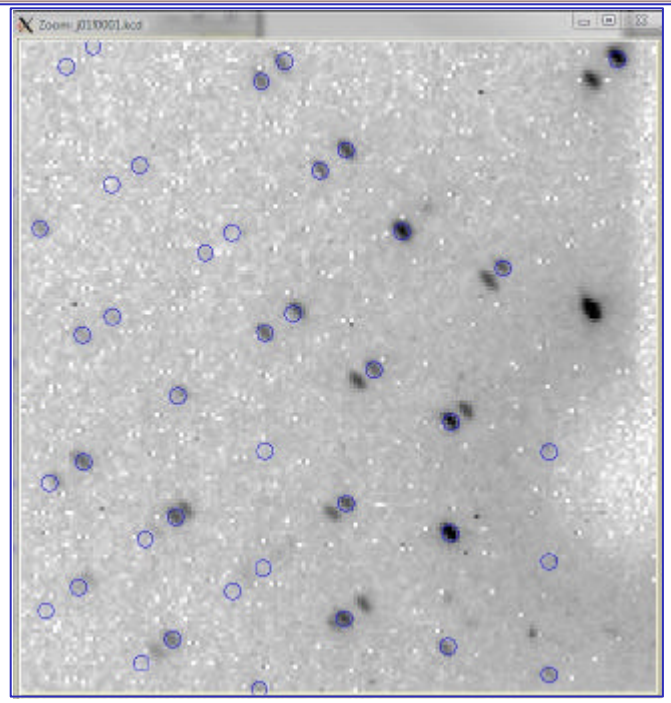
Chosen solution

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

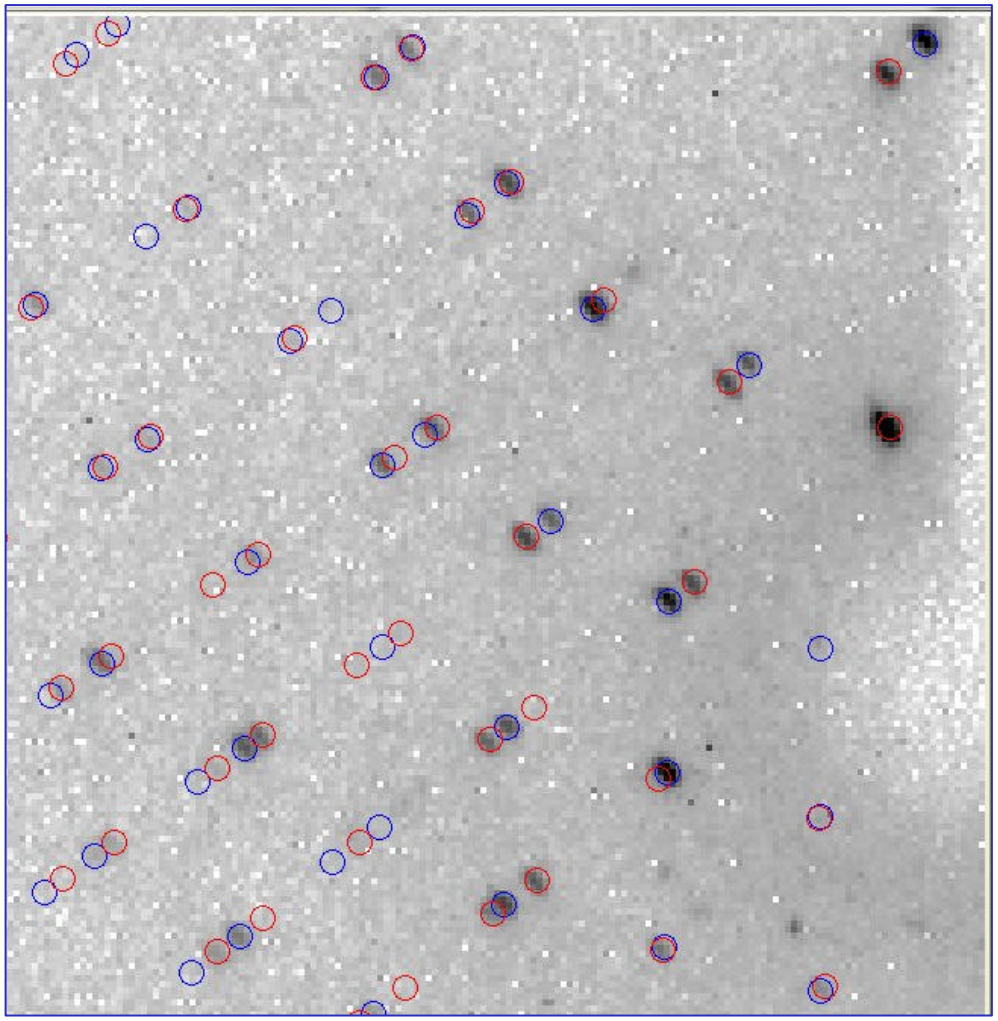
Coinciding axes criterion (degrees) ◀ 0.20 ▶

```
{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

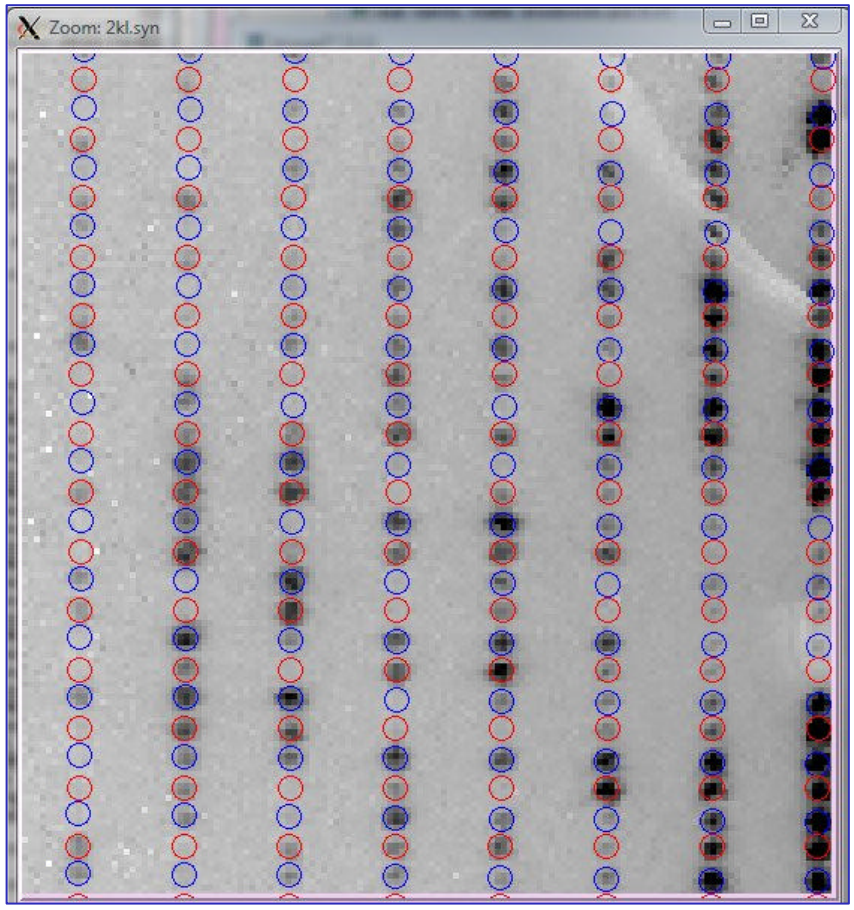


Diagrammme de diffraction = superposition de diverses contributions:

. 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
```



Diagrammme 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 : C_{26} H_{26} Cl N_3 O_4

Groupe spatial : `P -1`

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

Superflip

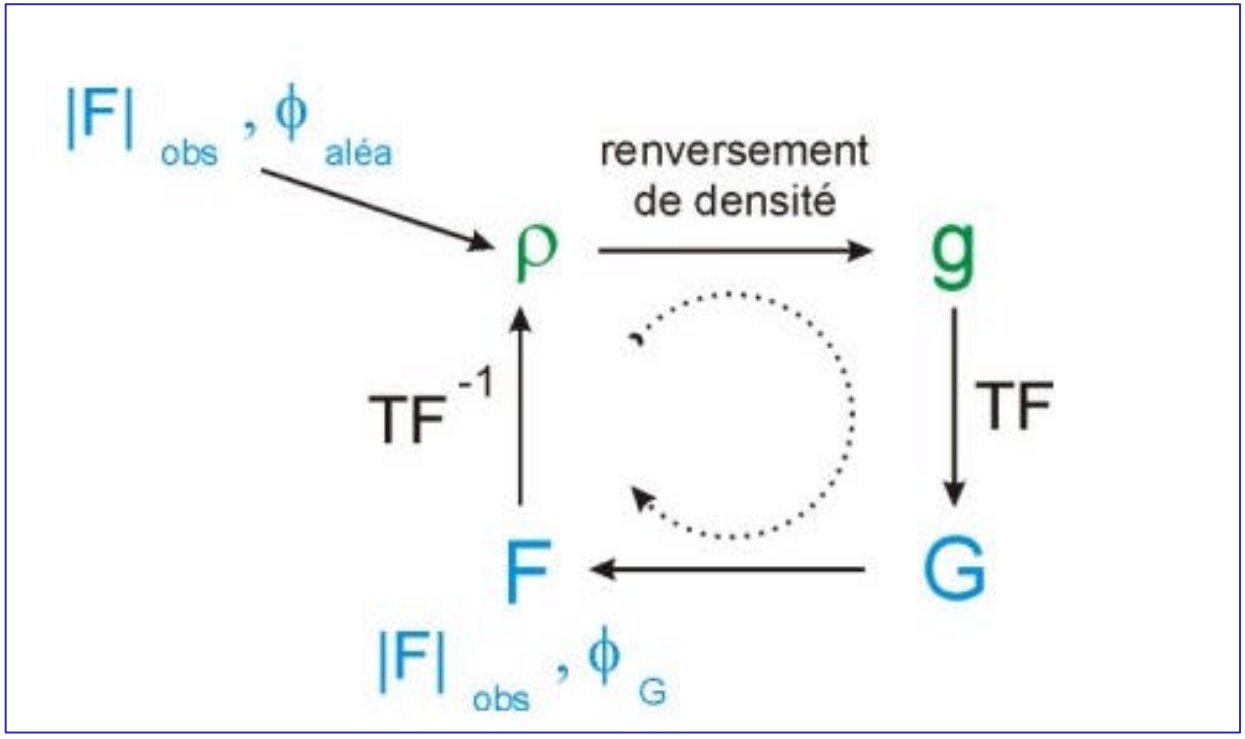
Superflip (charge flipping algorithm)

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.(densité électronique ?)



EDMA : analyse de la densité électronique finale

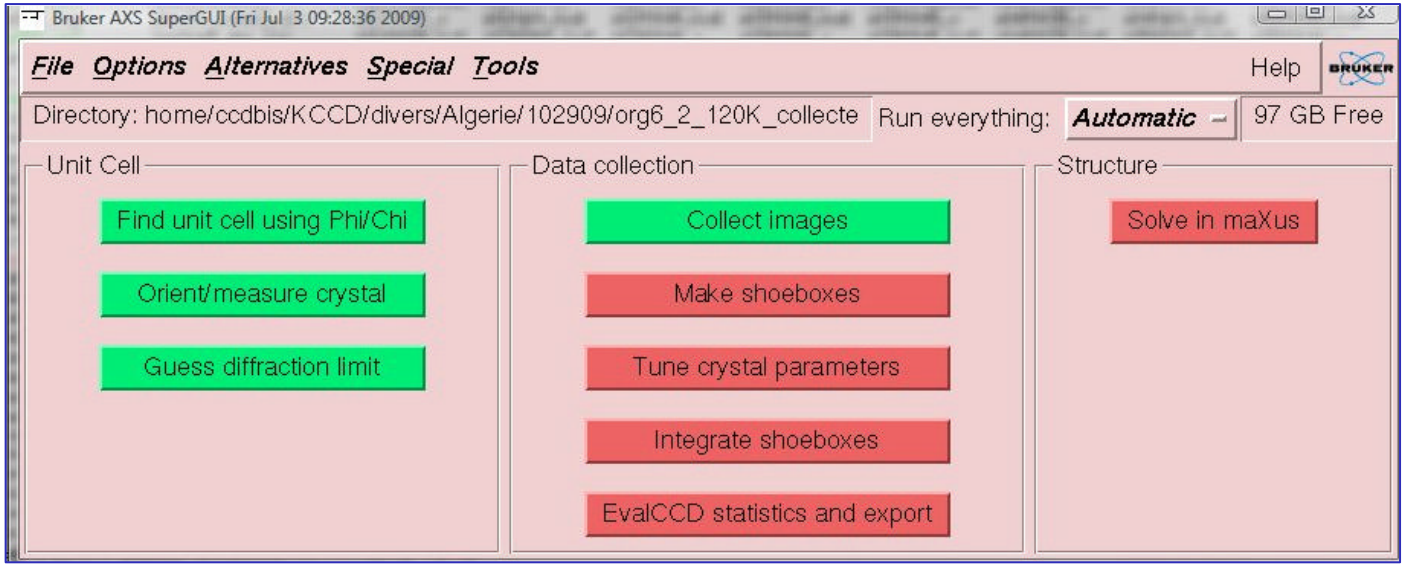
Affinement structural (SHELXL)

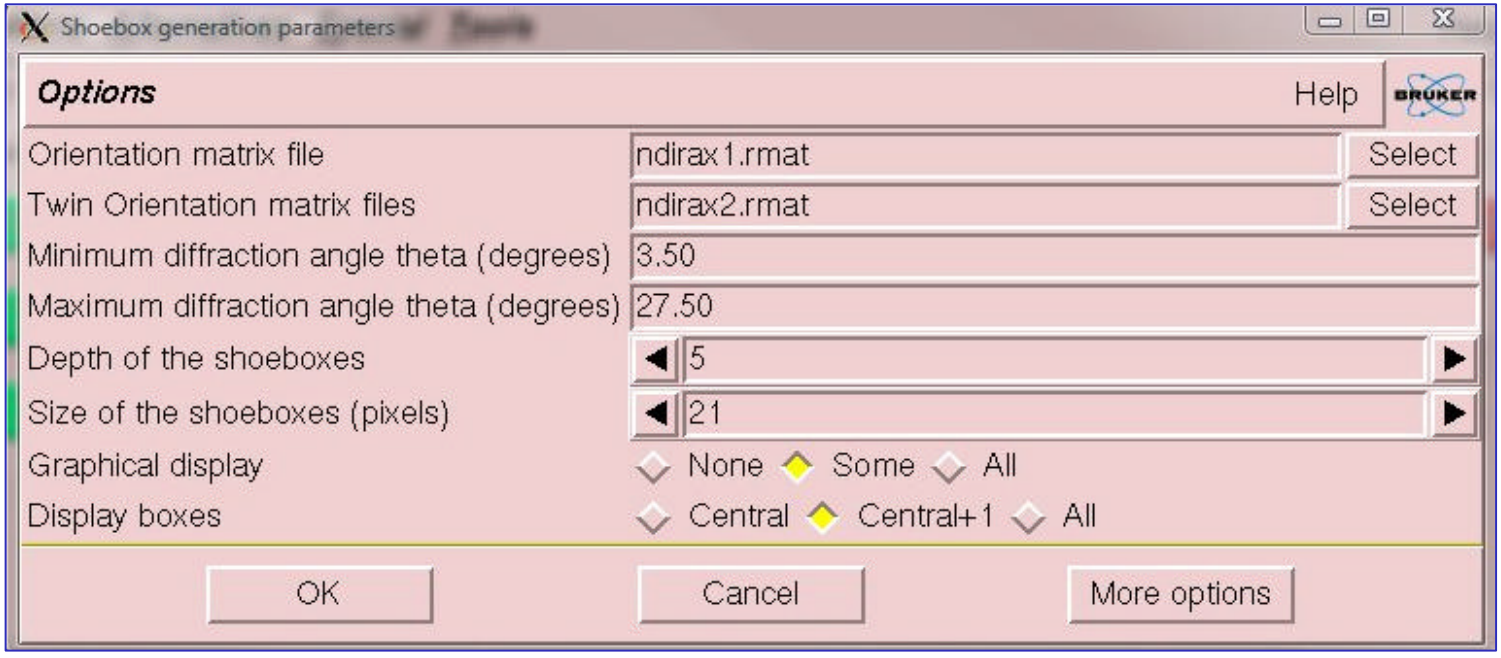
R1 ~28 %

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

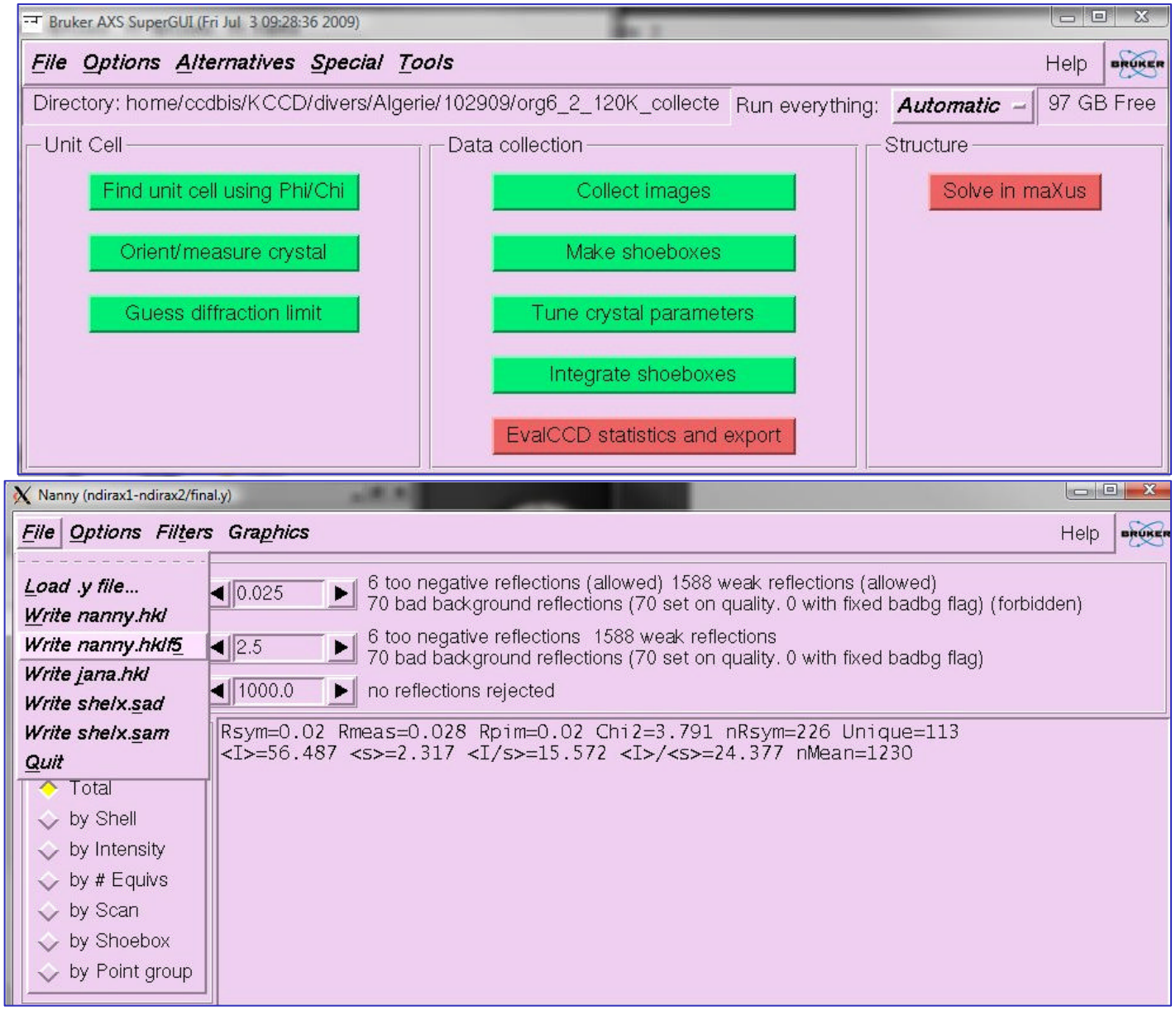
h	k	l	Fo^2	Fc^2	Delta(F^2)/esd	Fc/Fc(max)	Resolution(A)
-1	-2	6	1662.81	9.32	4.18	0.014	2.89
1	6	12	3028.58	0.03	4.17	0.001	1.39
-6	-3	13	509.20	0.03	4.17	0.001	1.02
1	5	12	1347.07	0.44	4.16	0.003	1.49
-6	-4	11	997.76	0.13	4.15	0.002	1.09
0	7	12	882.17	0.19	4.13	0.002	1.24
1	7	14	10169.39	55.38	4.12	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`

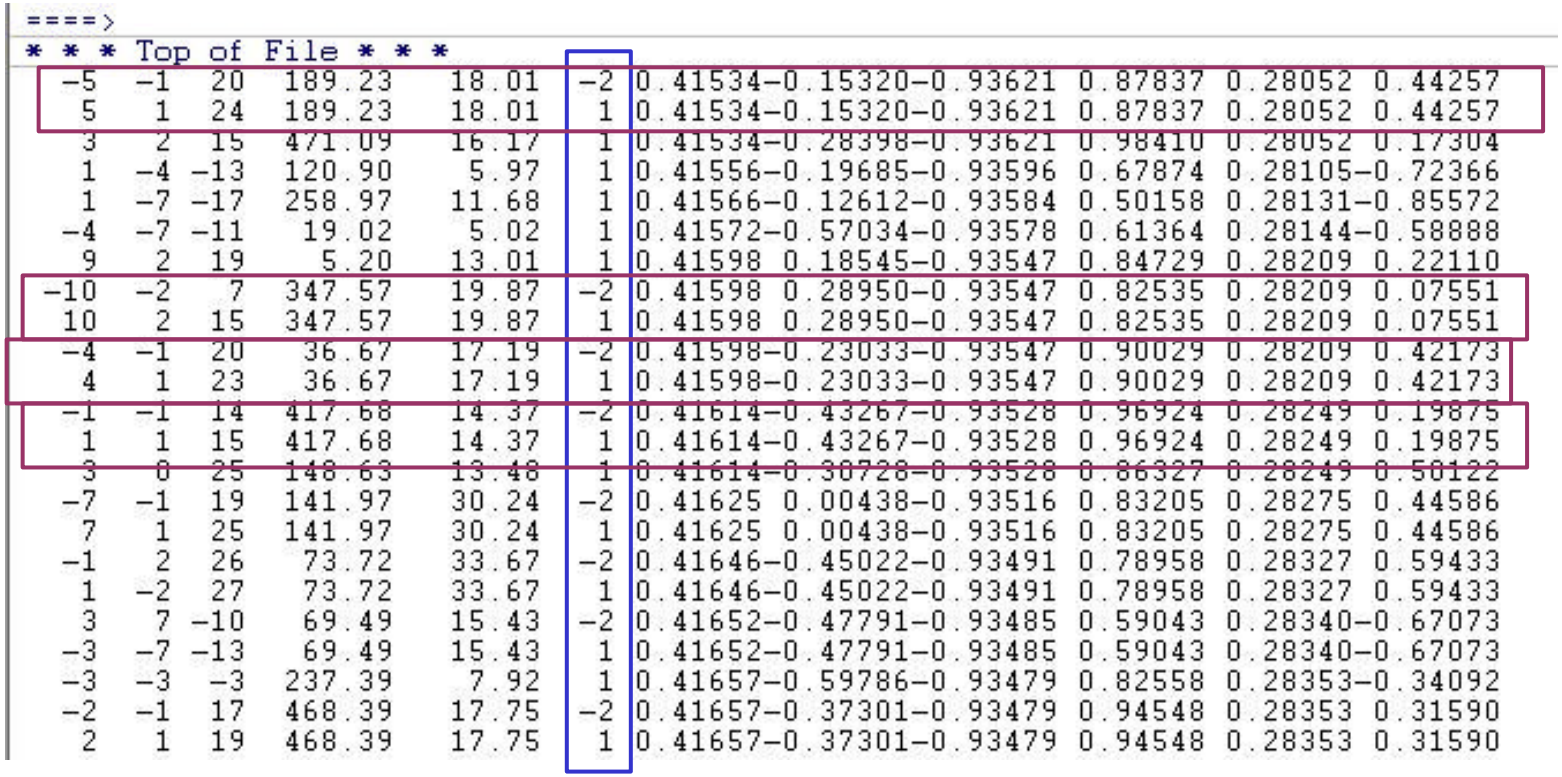


Nanny : création d'un fichier `hklf 5`

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`