



*“Structure Solution using
Direct Methods” - EXPO*
Structure Solution Software

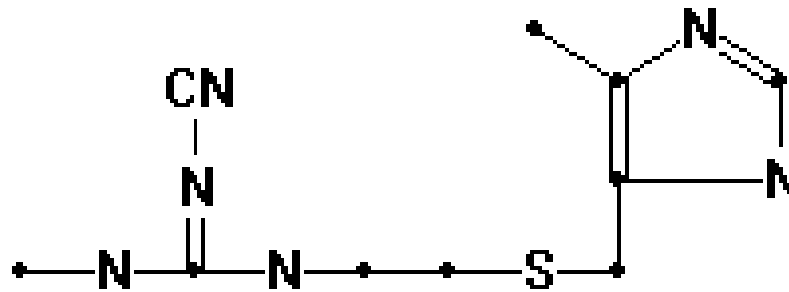
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Department of Chemistry, UMIST,
Manchester, UK**

ECM-21

24/8/03

Cimetidine: Structure



Cimetidine: **.exp file**

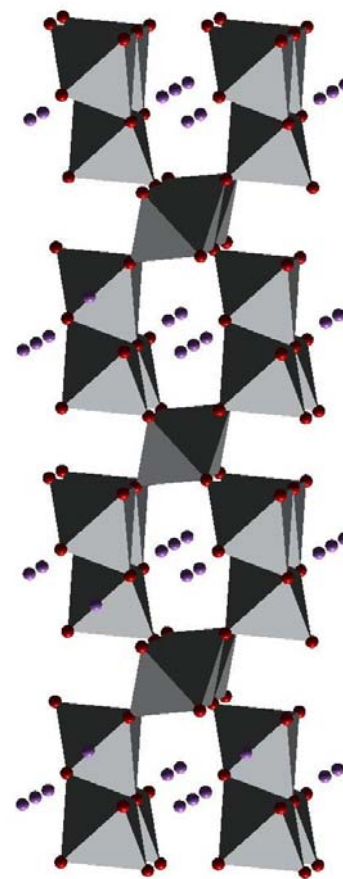
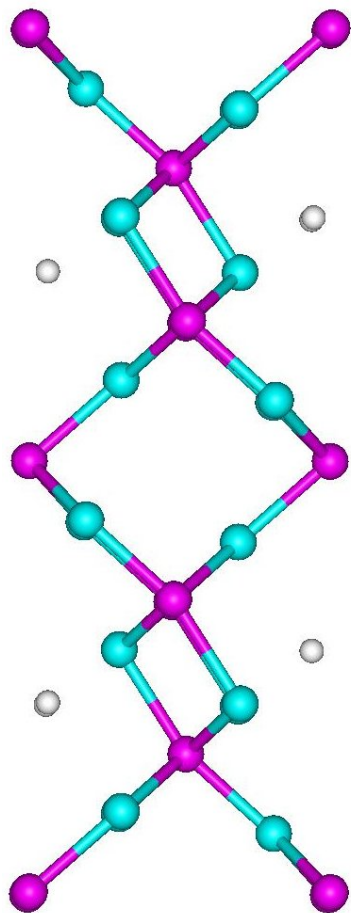
```
%struct cime
%job cimetidine -- Synchrotron data
%init
%data
range 8.01 84.99 0.01
pattern cime.pow
cont s 4 c 40 n 24 h 64
wave 1.52904
cell      10.6986  18.8181 6.8246          90.000  111.284  90.000
space p 21/n
sync
double
%extraction
%continue
```

Cimetidine:

Data file

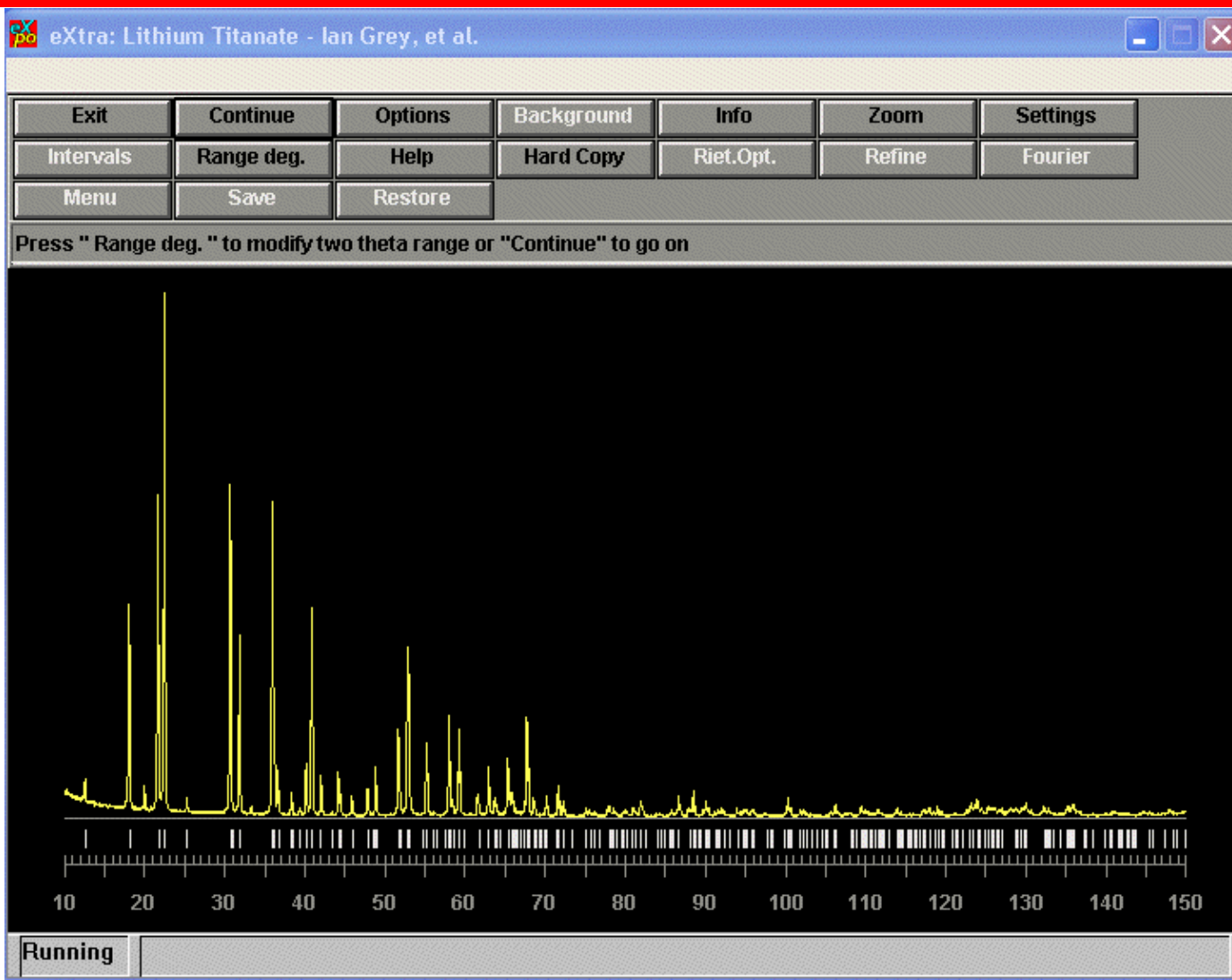
665	681	635	718	660	655	645	615	670	605
620	681	640	625	625	635	615	580	576	595
600	620	605	640	615	576	556	571	600	552
595	580	556	519	547	538	566	580	576	561
519	510	556	566	492	566	538	519	542	529
547	552	542	538	561	492	488	488	510	475
497	529	547	552	492	484	519	497	501	462
510	488	524	488	497	510	515	488	533	538
529	580	533	524	620	655	650	756	778	918
954	973	1043	942	681	595	529	462	449	436
424	428	445	412	412	462	457	412	432	462
424	416	445	392	432	424	441	420	436	388
408	408	412	404	396	441	412	404	400	388
416	388	388	416	416	376	436	396	412	334
384	400	380	388	400	368	353	384	396	353
376	361	368	349	349	368	372	376	372	368
342	338	380	342	327	364	338	376	349	338
345	368	368	302	372	368	338	372	334	334
320	327	357	364	324	353	357	338	361	361
364	345	342	342	320	361	364	327	349	306

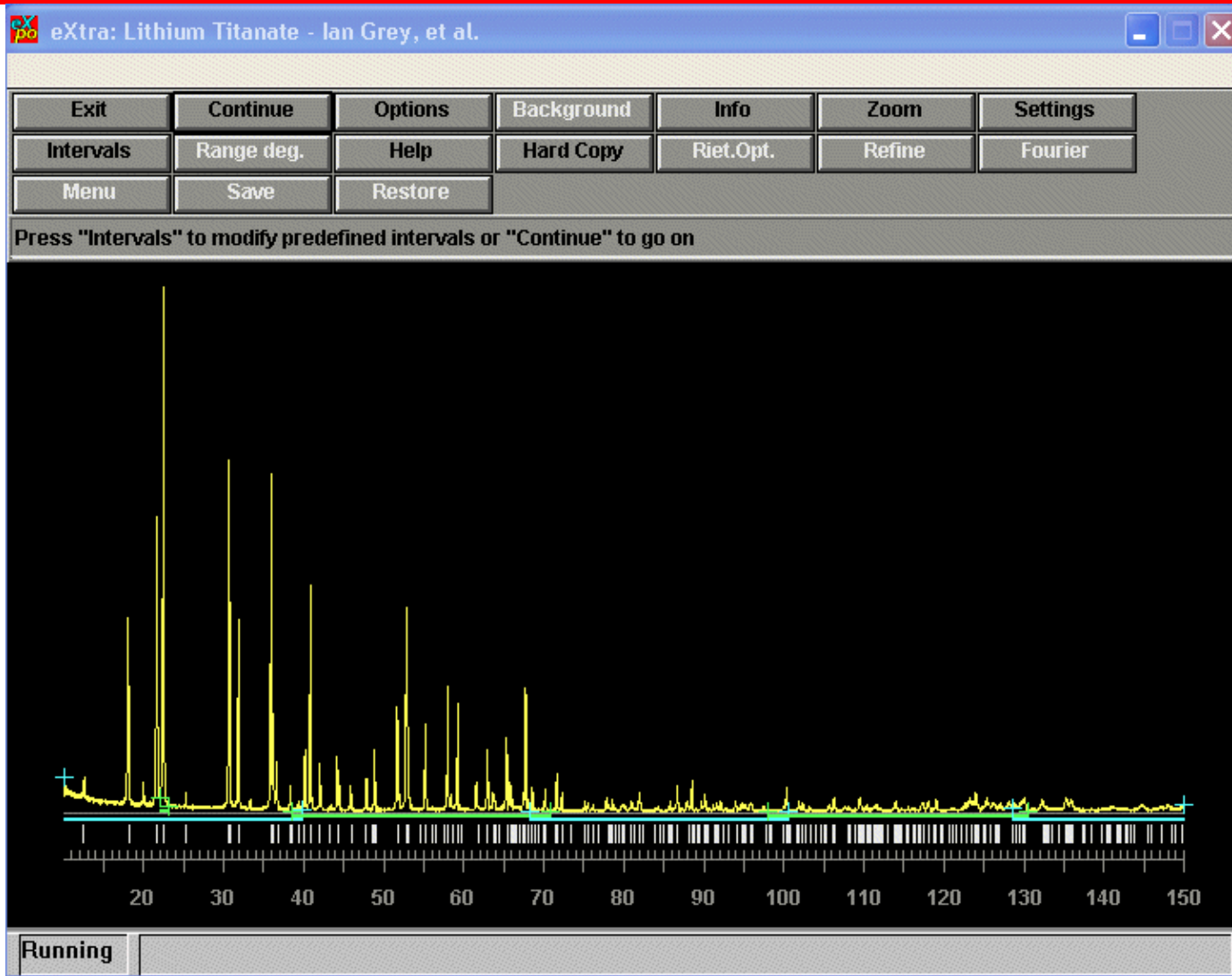
$Li_{0.98}Ti_{2.88}O_6$: Structure

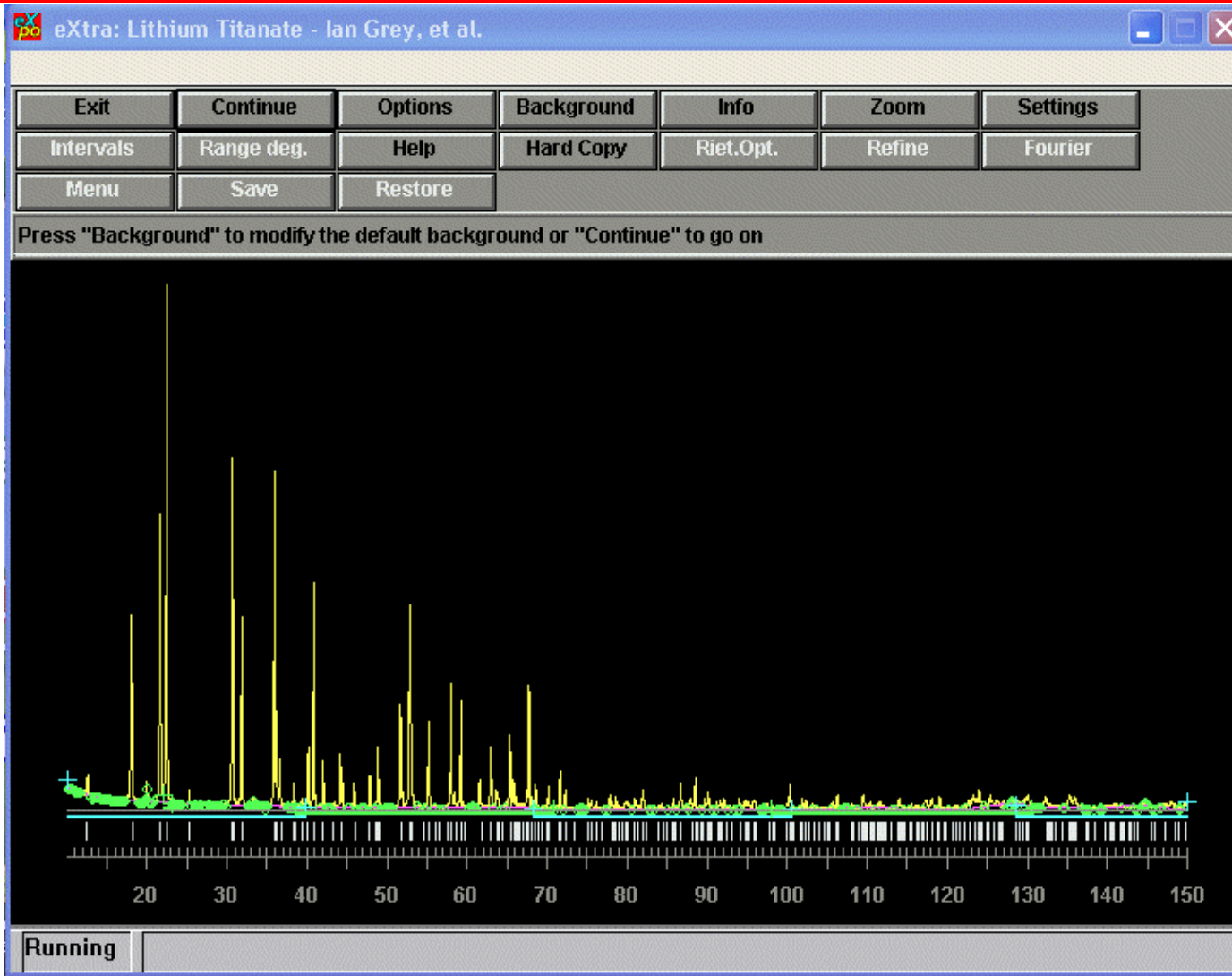


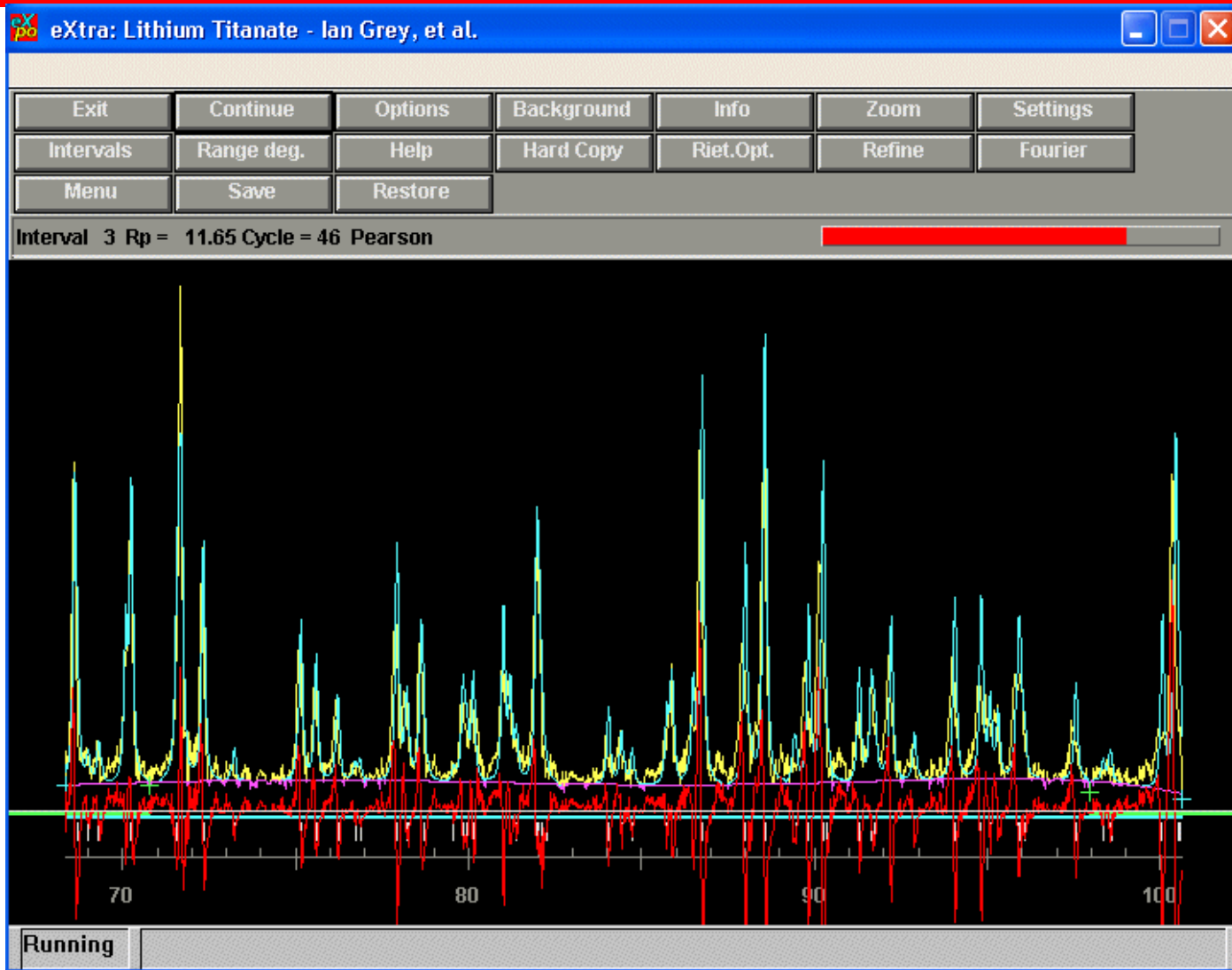
$Li_{0.98}Ti_{2.88}O_6$: .exp file

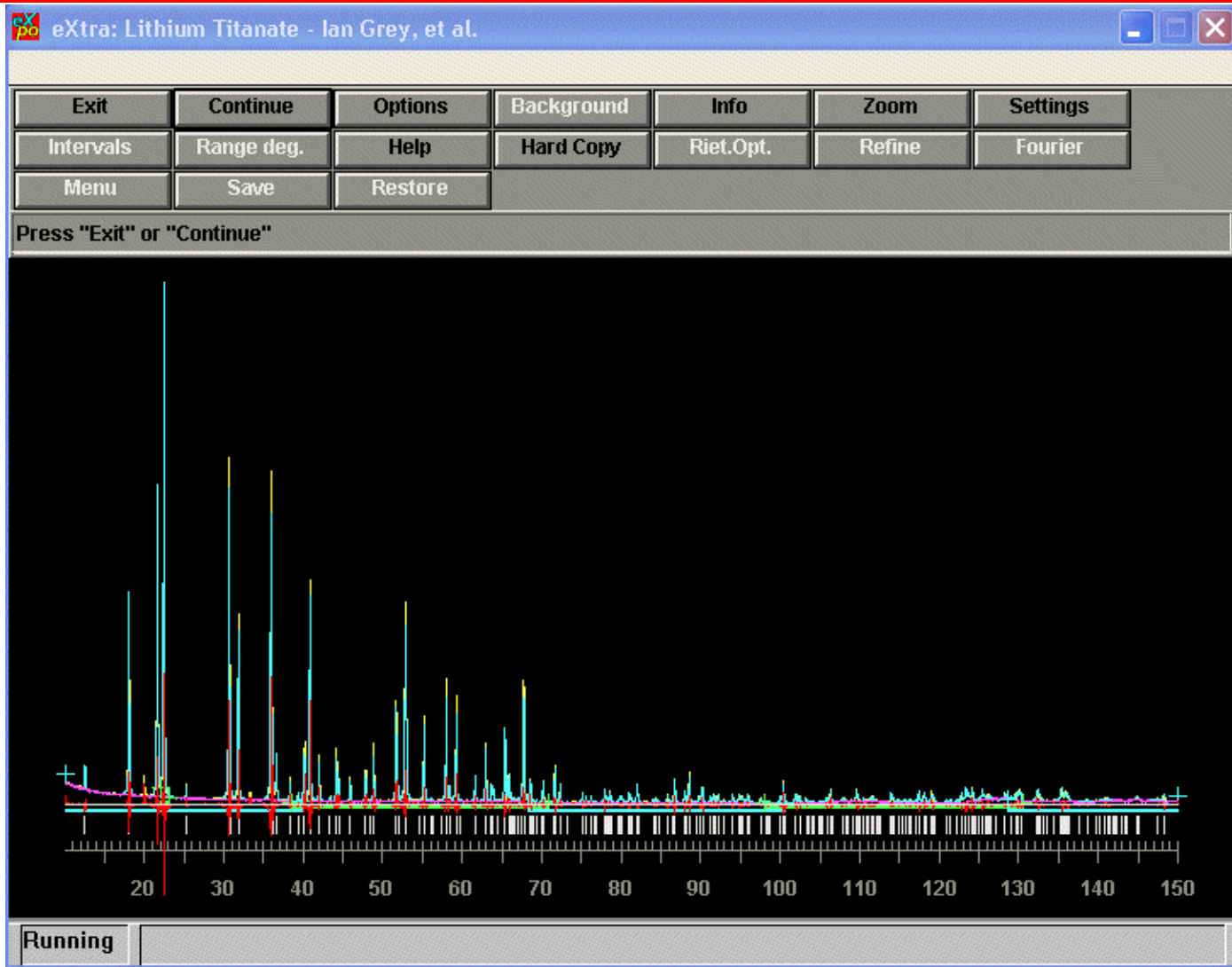
```
%struct liti
%job Lithium Titanate - Ian Grey, et al.
%init
%data
range 10.000 150.000 0.025
pattern liti.pow
cont Ti 6 O 12 Li 2
wave 1.54056
cell 14.0956 2.9524 4.8917 90.000 92.1716 90.000
space c 2/m
alpha
%extraction
%continue
```

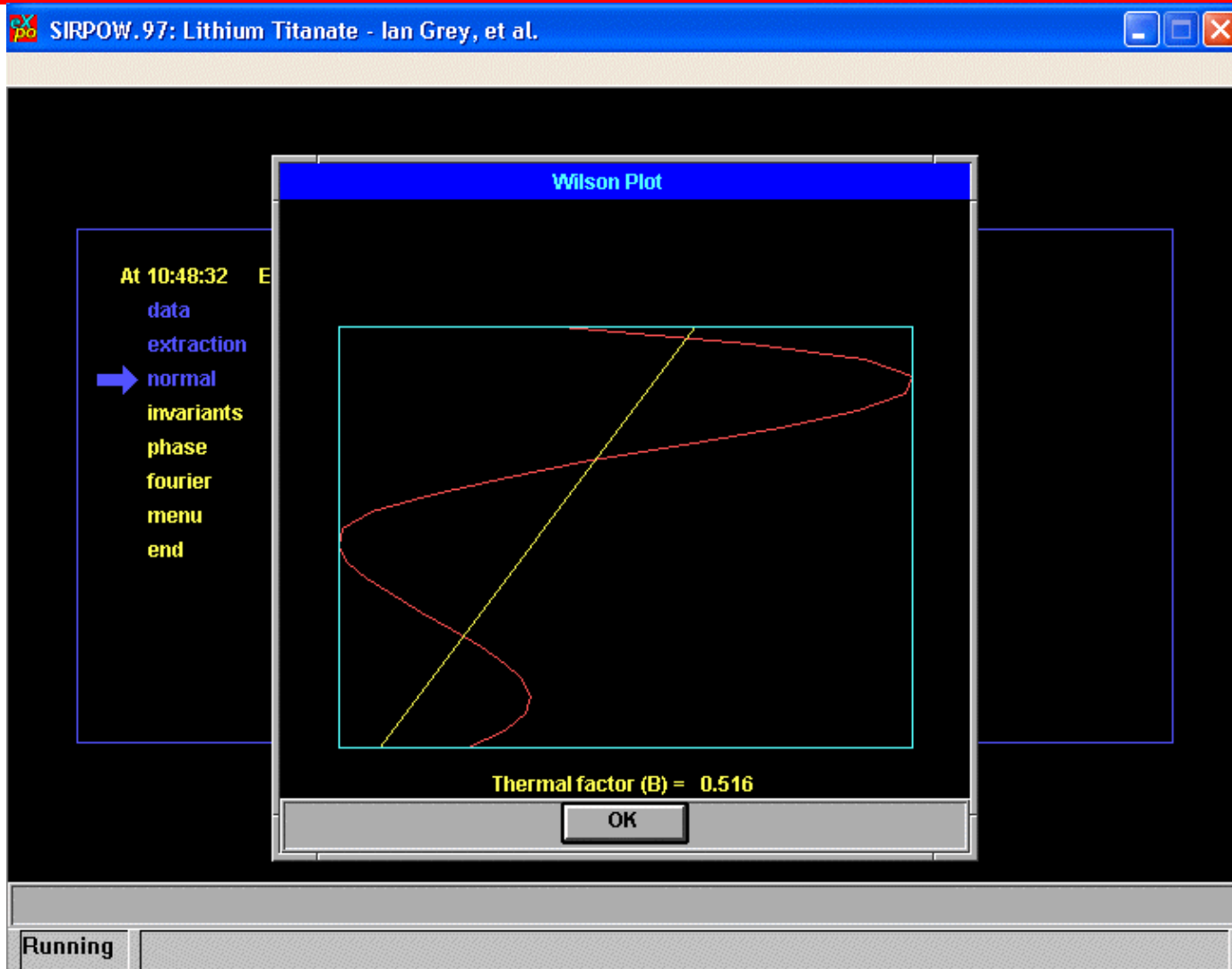














SIRPOW.97: Lithium Titanate - Ian Grey, et al.

At 10:50:50 EXPO runs on: liti

- data
- extraction
- normal
- invariants
- phase
- fourier
- menu
- end

Pseudotransl. effects are present: FSP = 16 %

Use this information in Extra

?

YES NO

Running

The image shows a screenshot of a software window titled "SIRPOW.97: Lithium Titanate - Ian Grey, et al.". The window has a blue title bar with standard Windows window controls (minimize, maximize, close). The main content area has a black background. On the left, there is a vertical menu with yellow text: "At 10:50:50 EXPO runs on: liti", "data", "extraction", "→ normal", "invariants", "phase", "fourier", "menu", and "end". A blue arrow points to the "normal" option. In the center, a dialog box is open with a red header bar containing the text "Pseudotransl. effects are present: FSP = 16 %". Below the header, the dialog box has a grey background with the text "Use this information in Extra" and a yellow question mark icon. At the bottom of the dialog box are two buttons labeled "YES" and "NO". At the bottom of the main window, there is a status bar with a tab labeled "Running".

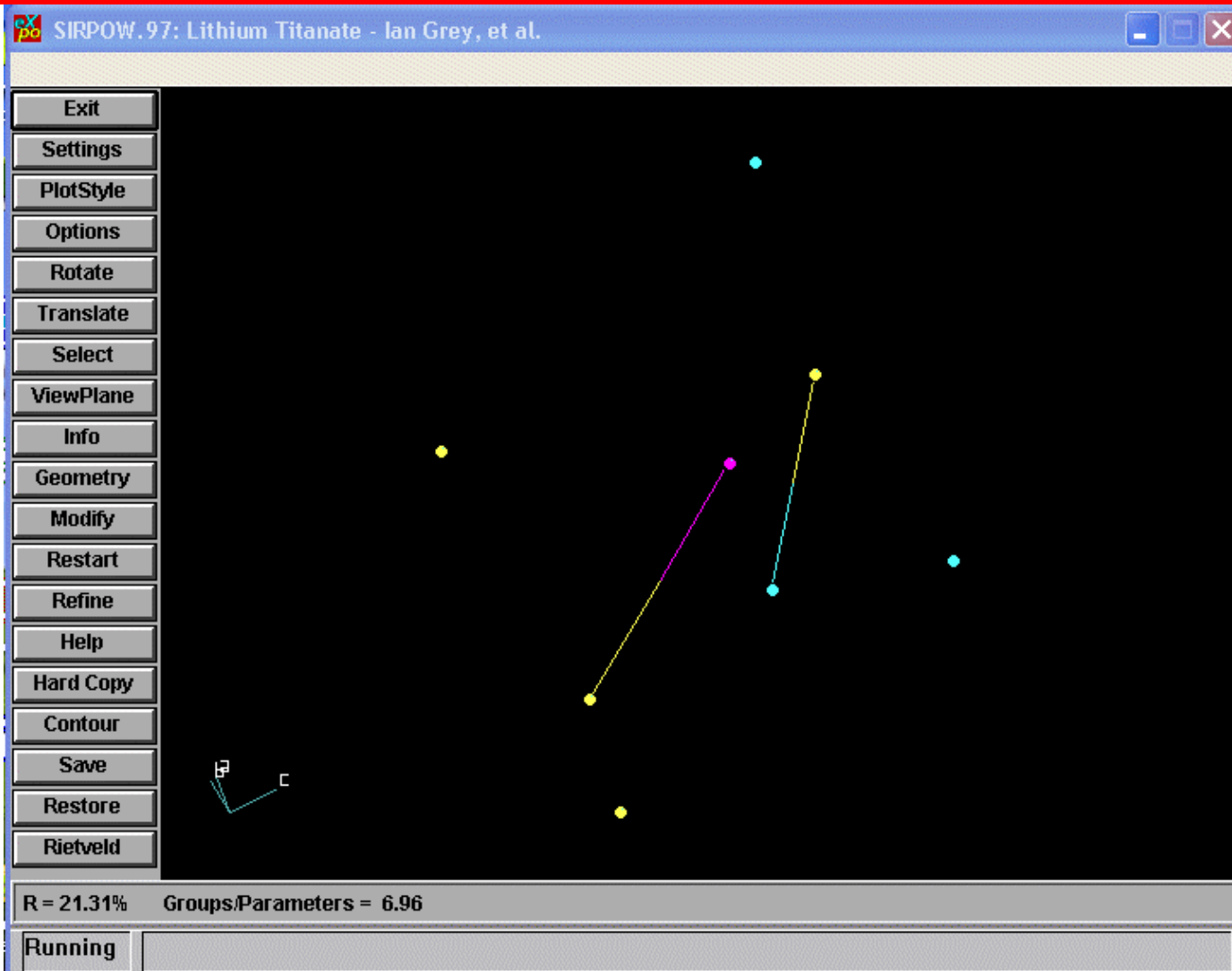


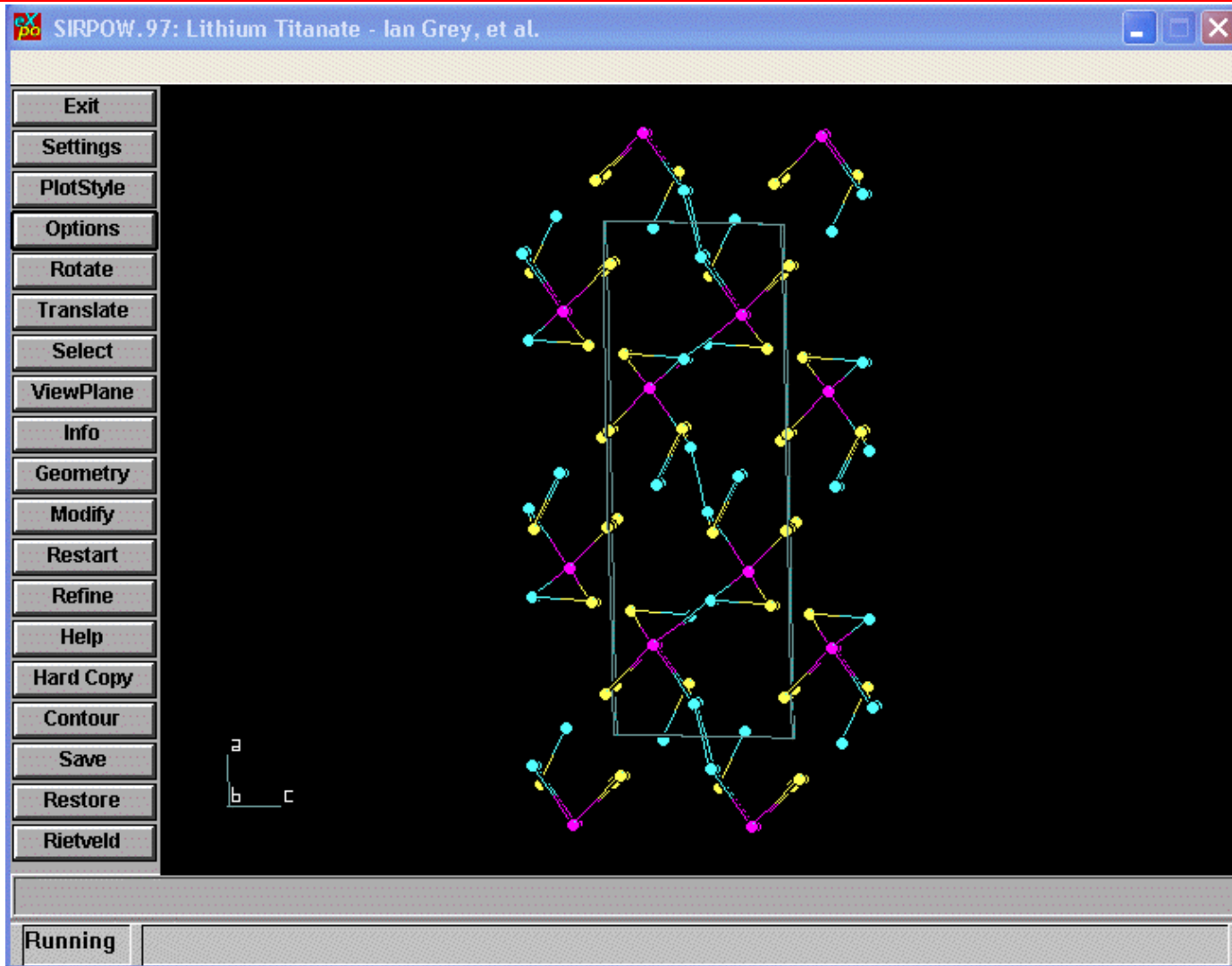
SIRPOW.97: Lithium Titanate - Ian Grey, et al.

```
At 10:55:38  EXPO runs on: liti
data
extraction
normal      B = 0.516
invariants
→ phase     Trial 13 / 32 CFom = 0.101 (max = 0.635)
fourier
menu
end
```

Assign phases to reflexions

Running

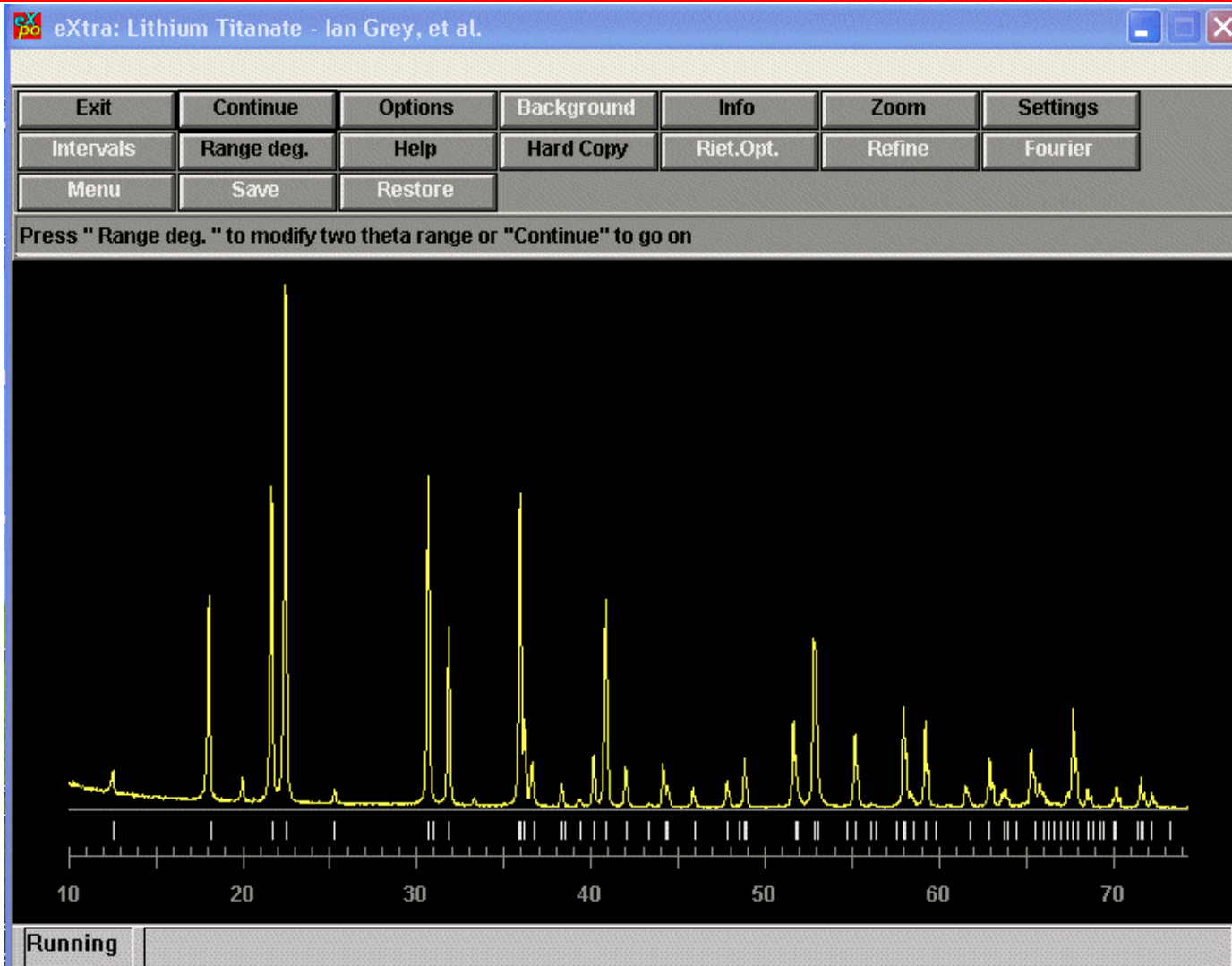




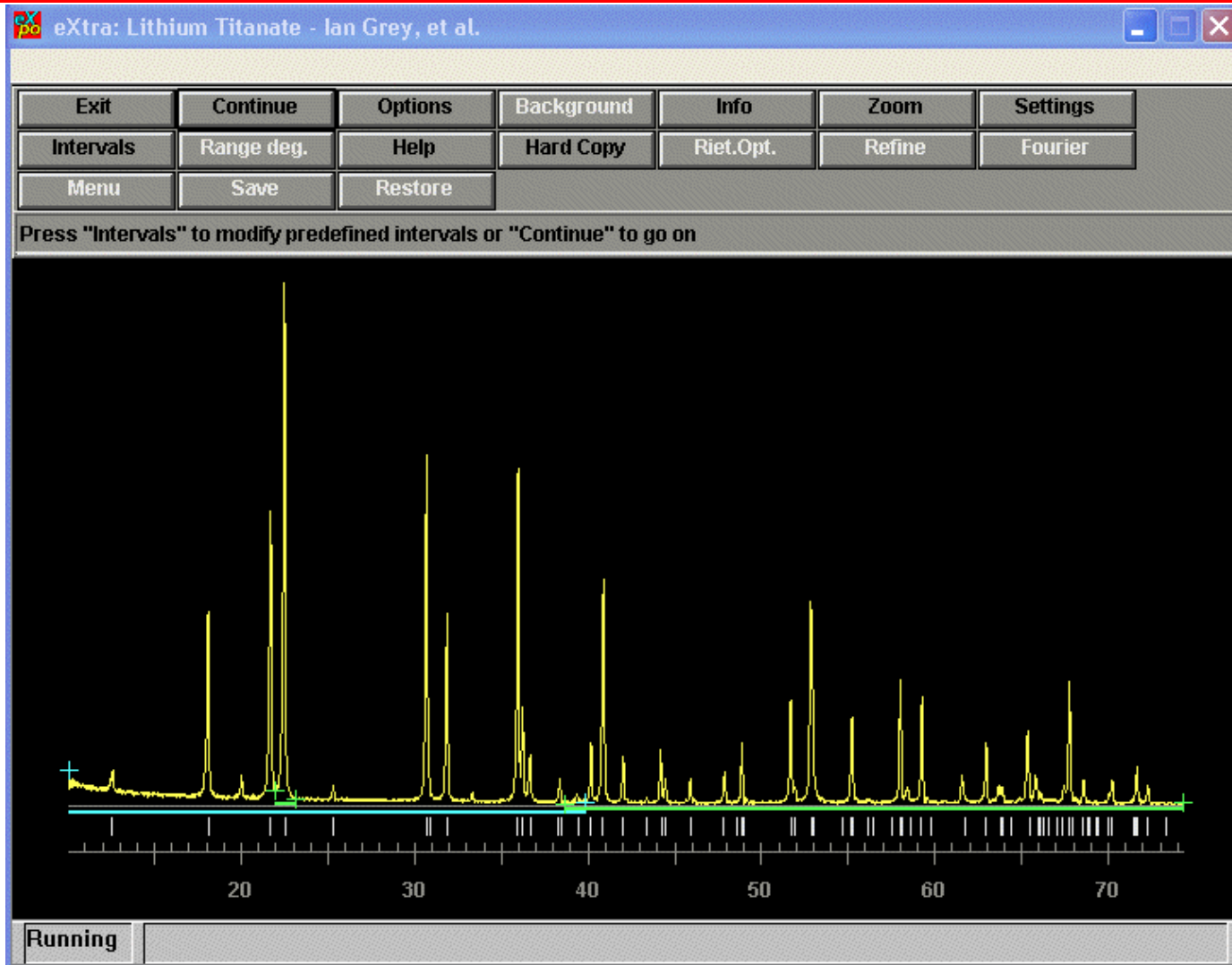
$Li_{0.98}Ti_{2.88}O_6$ - less data: .exp file

```
%struct liti
%job Lithium Titanate - Ian Grey, et al.
%init
%data
range 10.000 74.300 0.025
pattern liti.pow
cont Ti 6 O 12 Li 2
wave 1.54056
cell 14.0956 2.9524 4.8917 90.000 92.1716 90.000
space c 2/m
alpha
%extraction
%continue
```

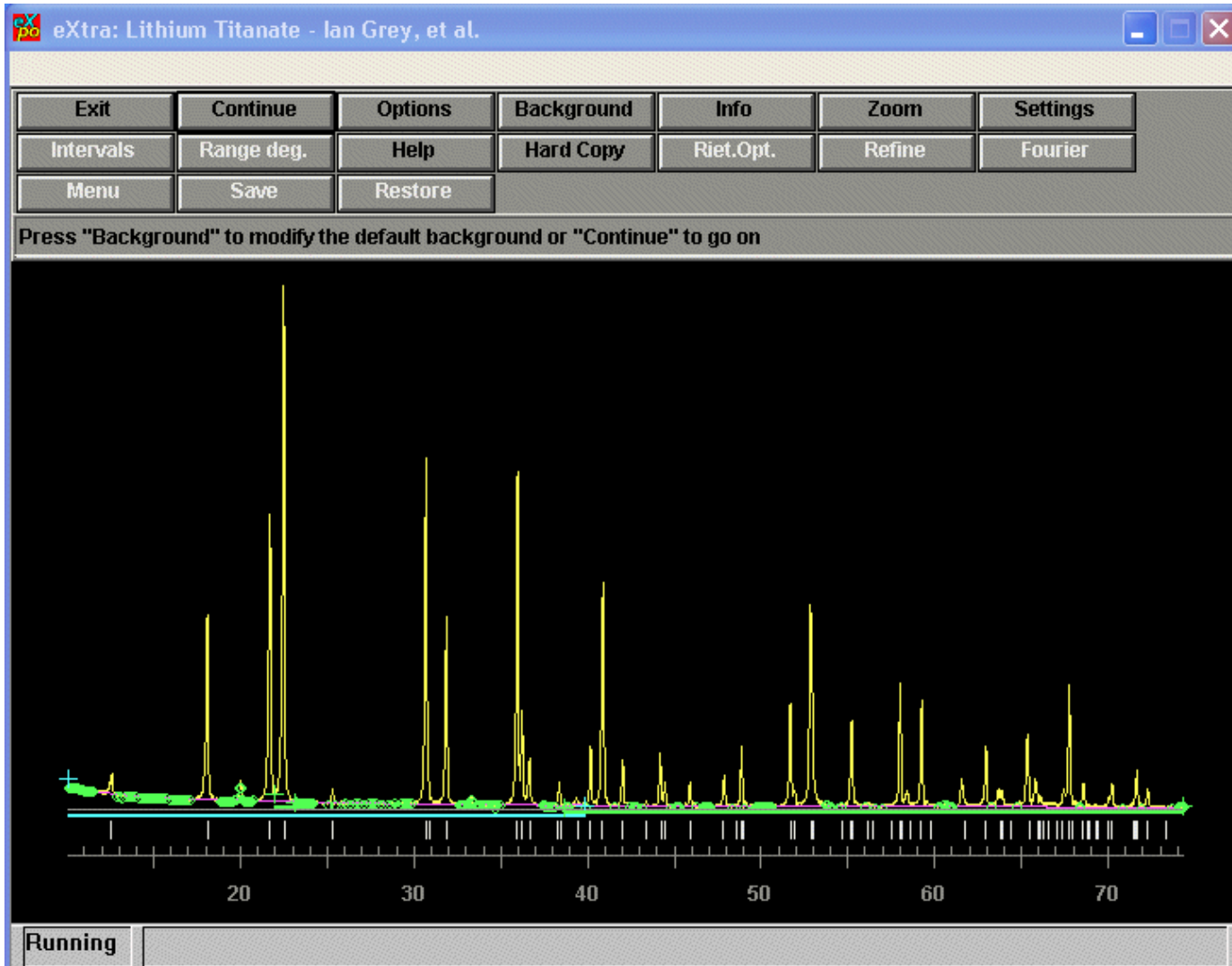
$Li_{0.98}Ti_{2.88}O_6$ - less data



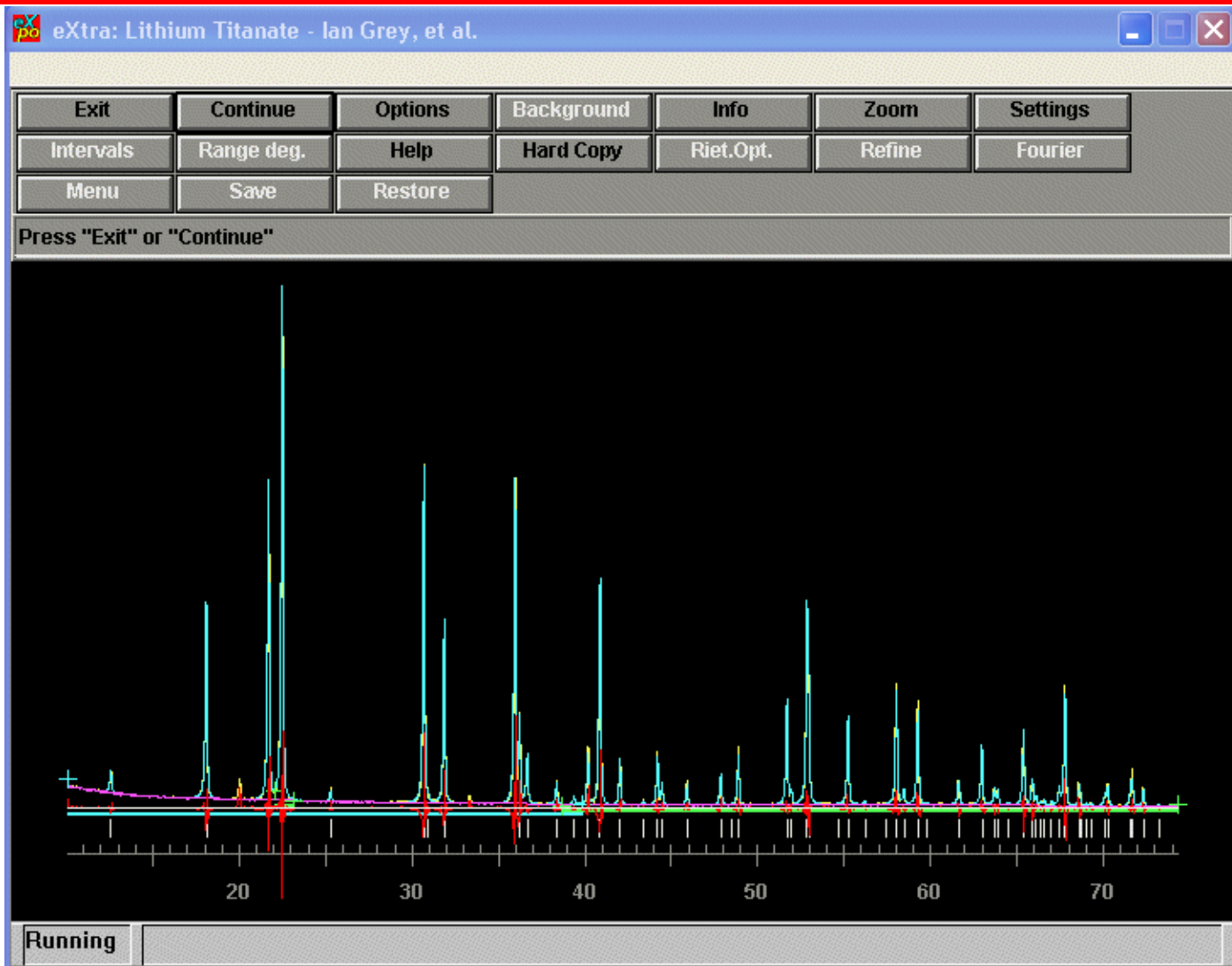
$Li_{0.98}Ti_{2.88}O_6$ - less data



$Li_{0.98}Ti_{2.88}O_6$ - less data



$Li_{0.98}Ti_{2.88}O_6$ - less data



$Li_{0.98}Ti_{2.88}O_6$ - less data



$Li_{0.98}Ti_{2.88}O_6$ - less data

The screenshot shows a window titled "SIRPOW.97: Lithium Titanate - Ian Grey, et al." with a menu on the left and a dialog box in the center.

Menu:

- At 13:49:20 EXPO runs on: liti
- data
- extraction
- normal
- invariants
- phase
- fourier
- menu
- end

Dialog Box:

Pseudotransl. effects are present: FSP = 16 %

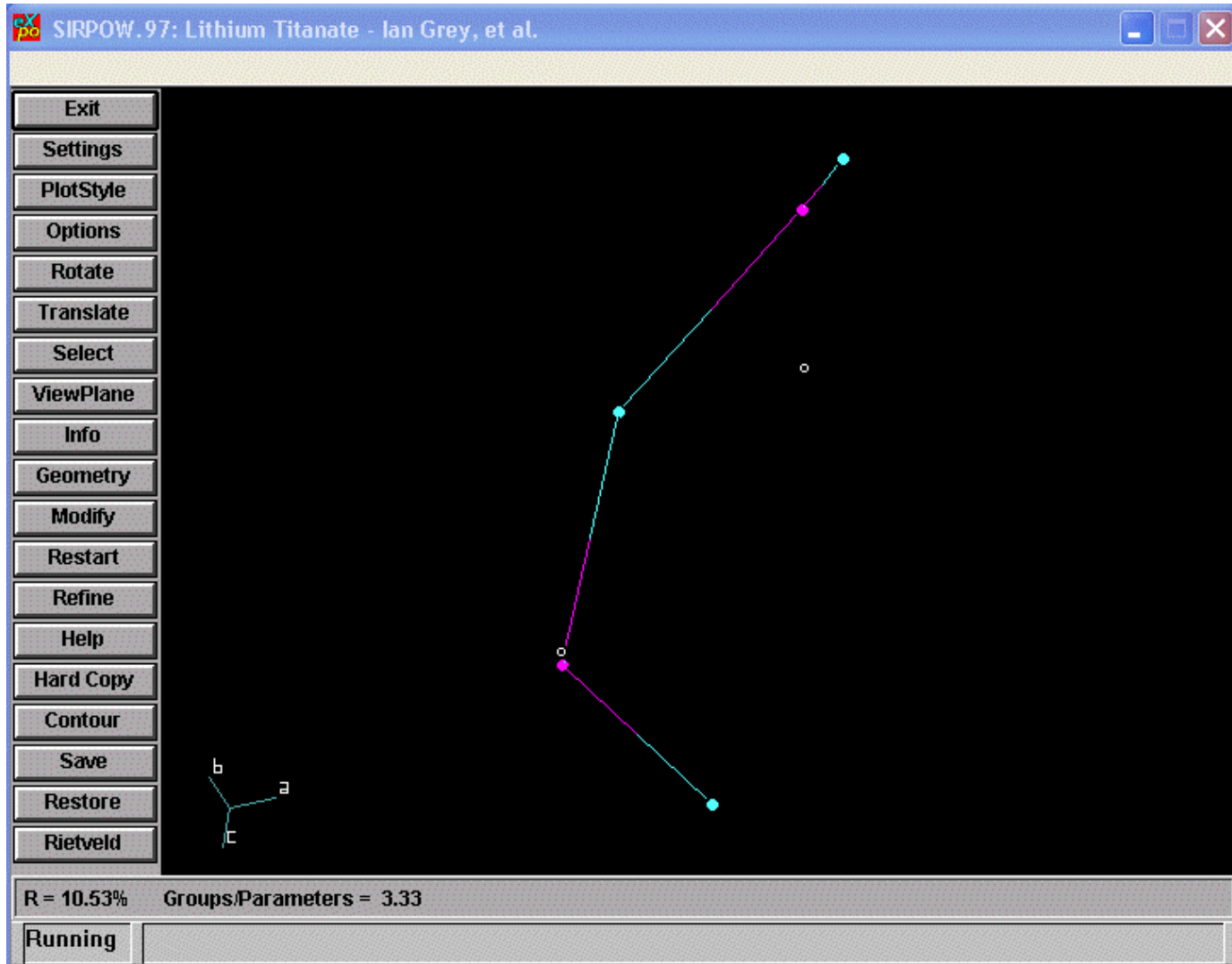
Use this information in Extra

?

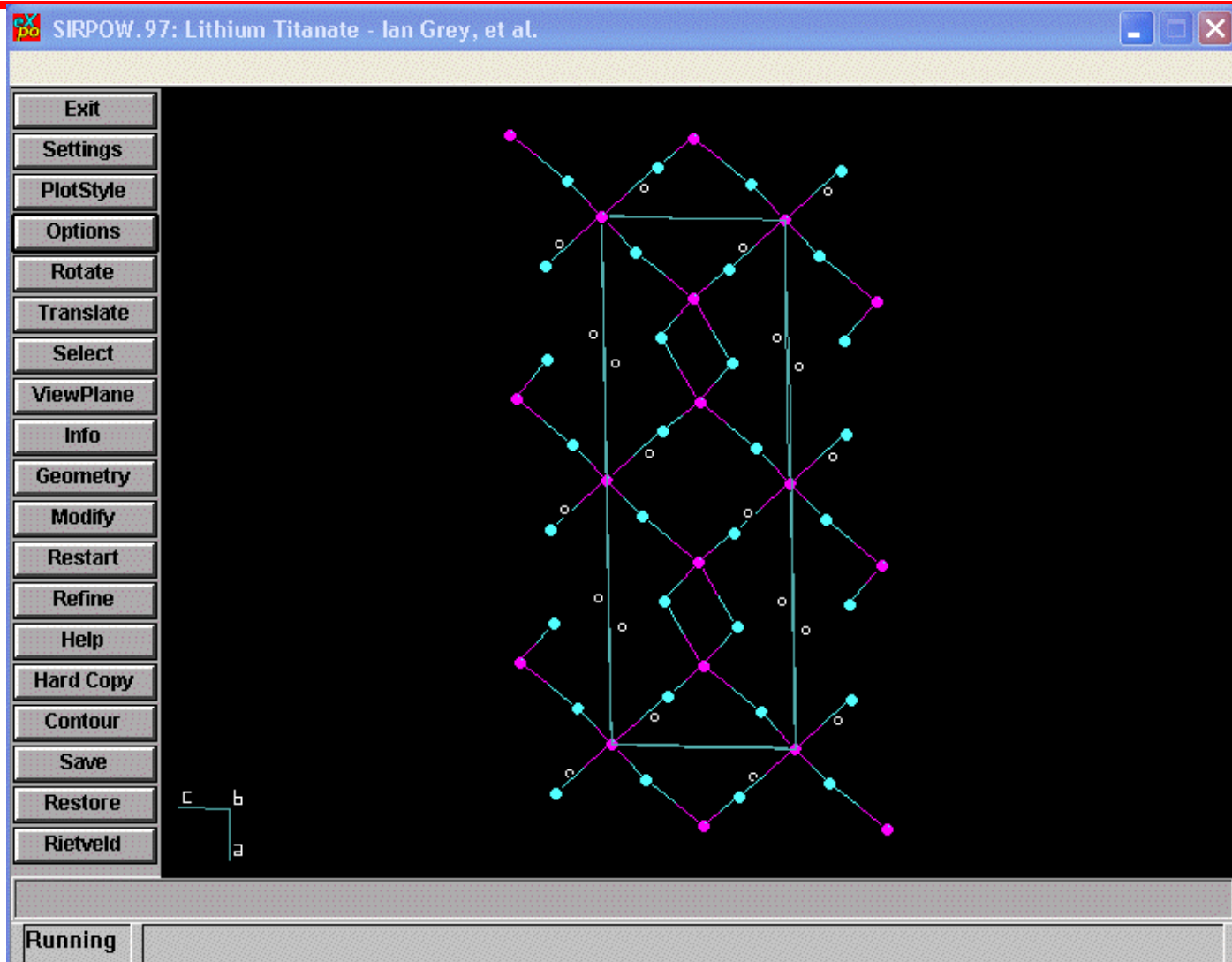
YES NO

Running

$Li_{0.98}Ti_{2.88}O_6$ - less data



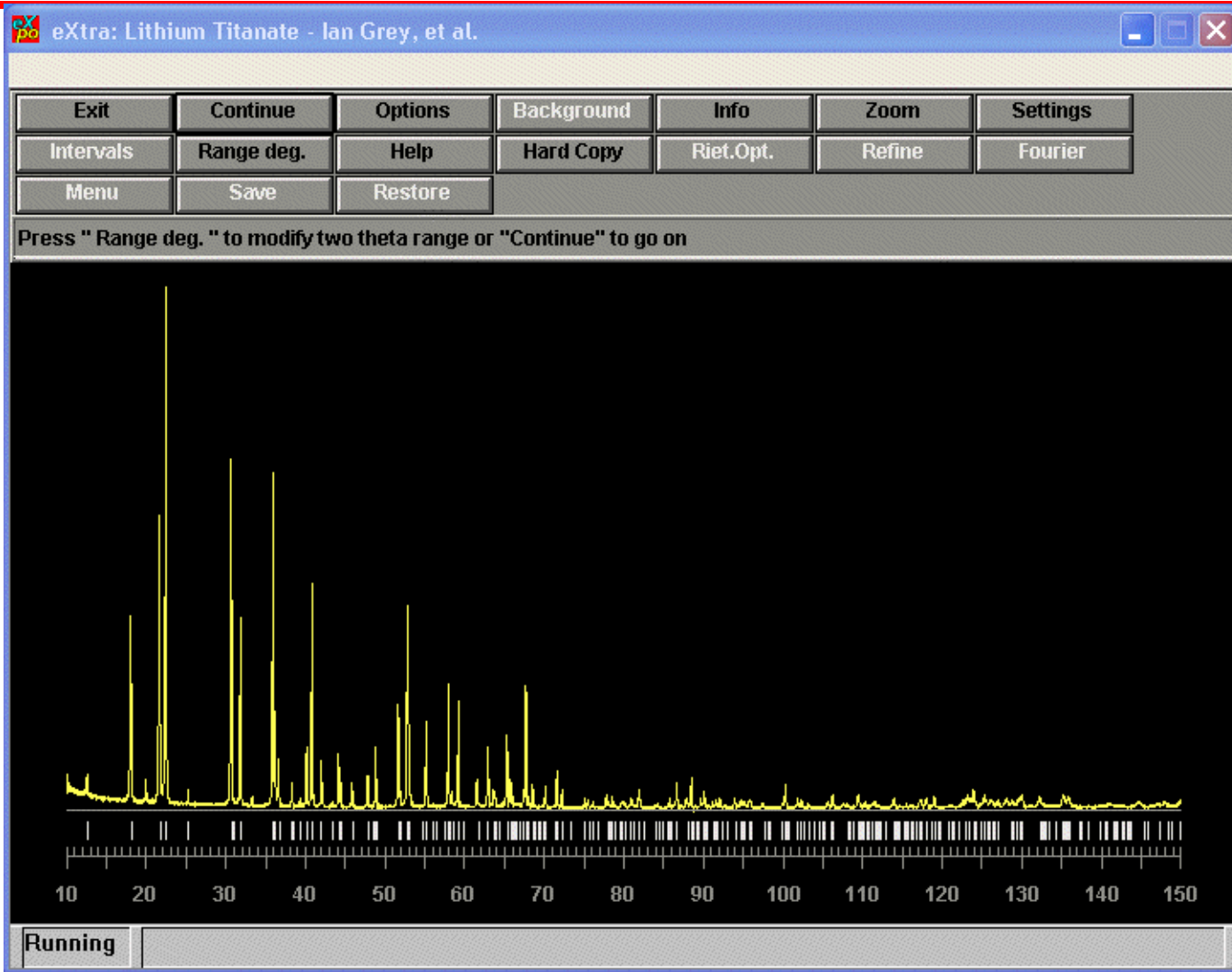
$Li_{0.98}Ti_{2.88}O_6$ - less data



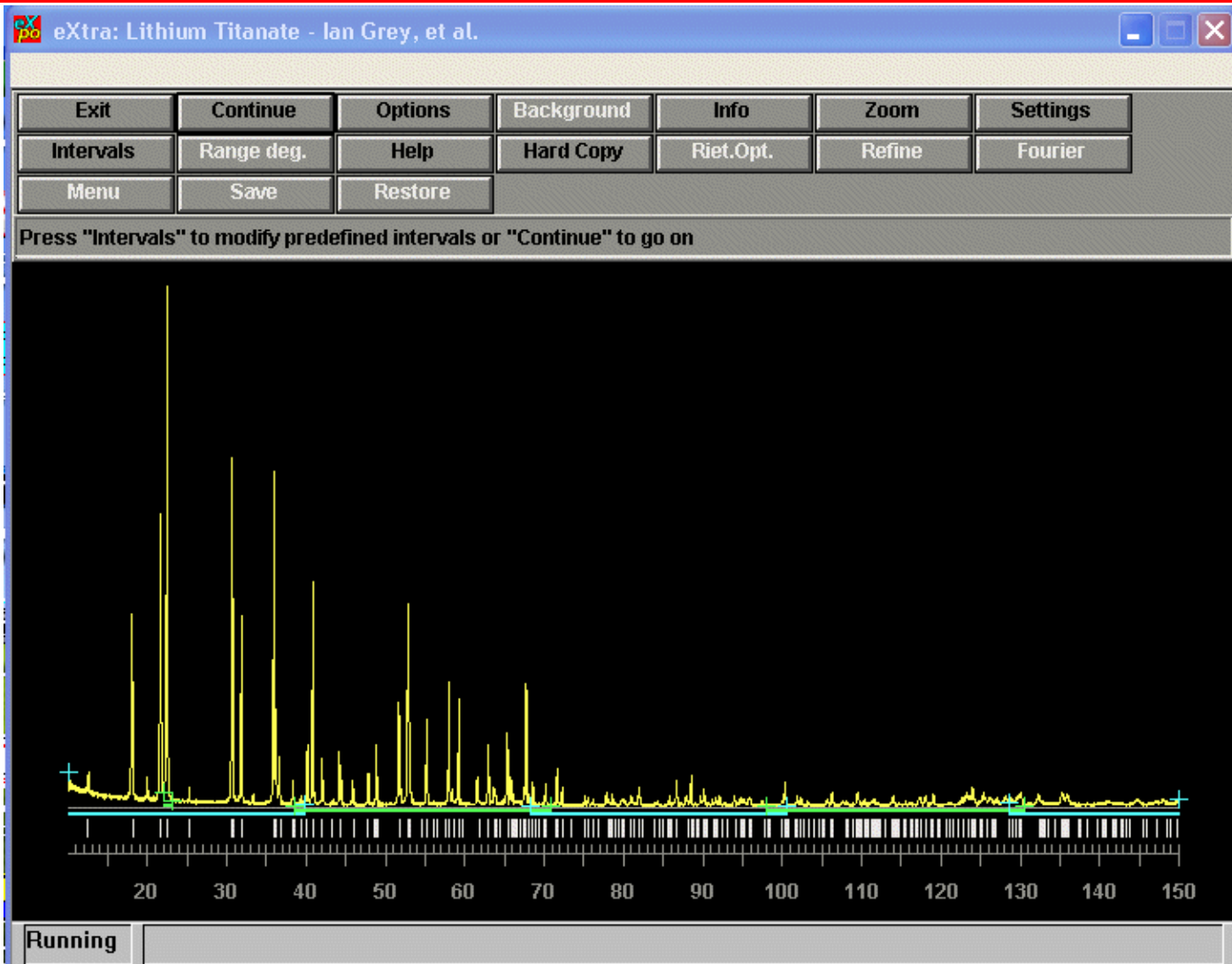
$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor: .exp file

```
%struct liti
%job Lithium Titanate - Ian Grey, et al.
%init
%data
range 10.000 150.000 0.025
pattern liti.pow
cont Ti 6 O 12 Li 2
wave 1.54056
cell 14.0956 2.9524 4.8917 90.000 92.1716 90.000
space c 2/m
alpha
%extraction
%normal
bfactor 0.4
%continue
```

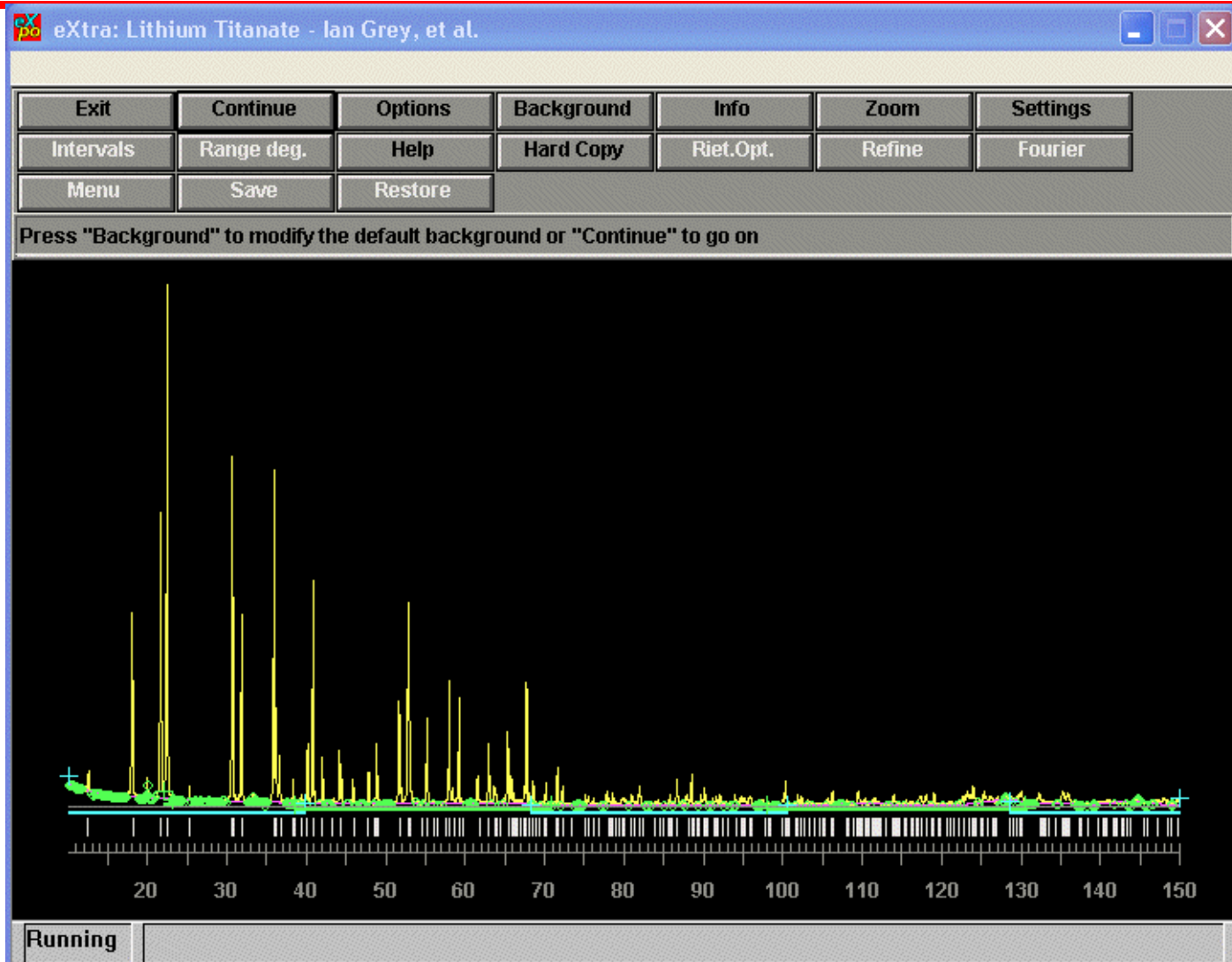
$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



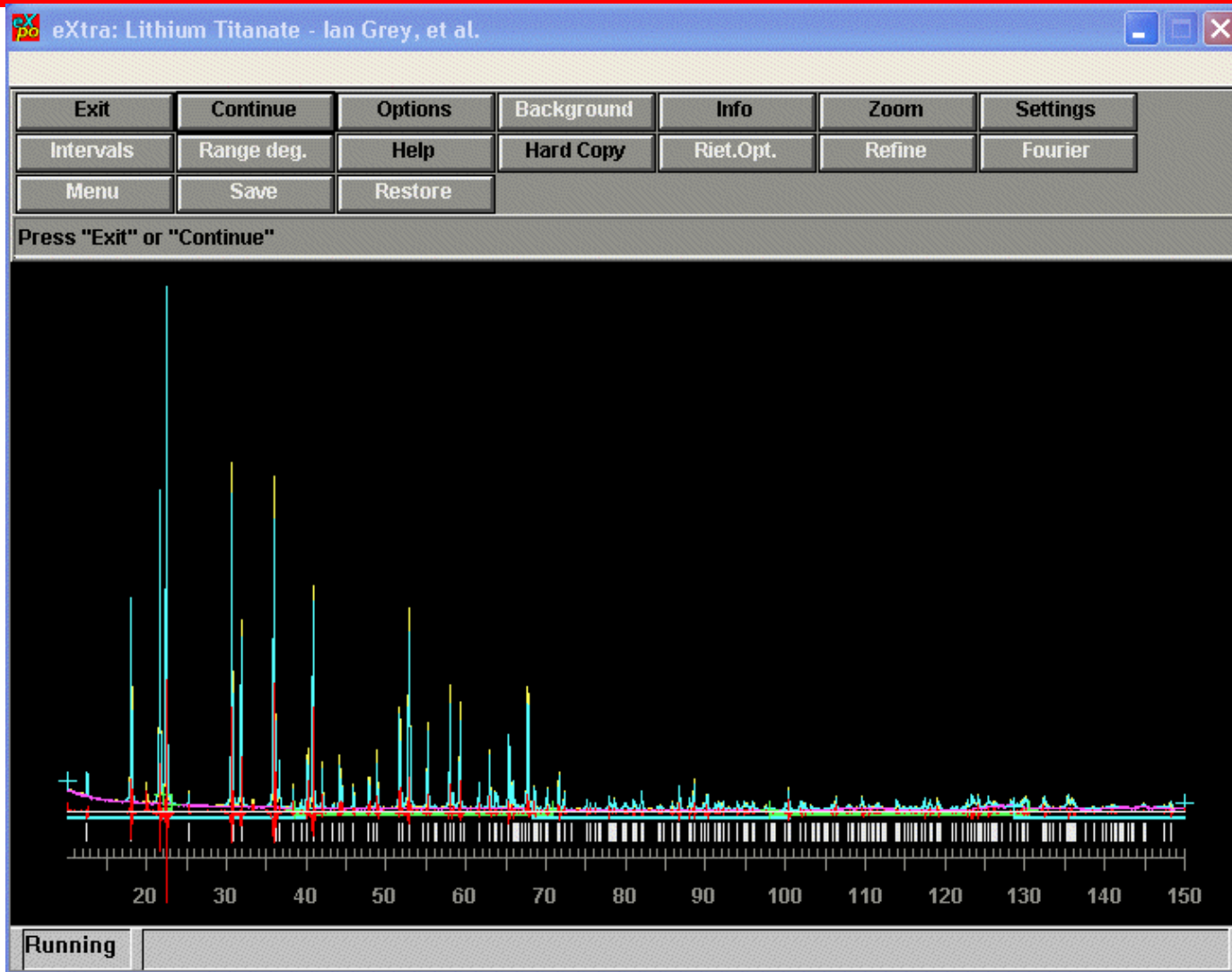
$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor

The screenshot shows the SIRPOW.97 software interface. The window title is "SIRPOW.97: Lithium Titanate - Ian Grey, et al.". The main window contains a list of options on the left side, with a blue arrow pointing to "normal". The options are: data, extraction, normal, invariants, phase, fourier, menu, and end. A dialog box is open in the center, featuring a red header bar with the text "Pseudotransl. effects are present: FSP = 16 %". Below the header, the text "Use this information in Extra" is displayed next to a yellow question mark icon. At the bottom of the dialog box are two buttons labeled "YES" and "NO".

At 14: 4:48 EXPO runs on: liti

- data
- extraction
- normal
- invariants
- phase
- fourier
- menu
- end

Pseudotransl. effects are present: FSP = 16 %

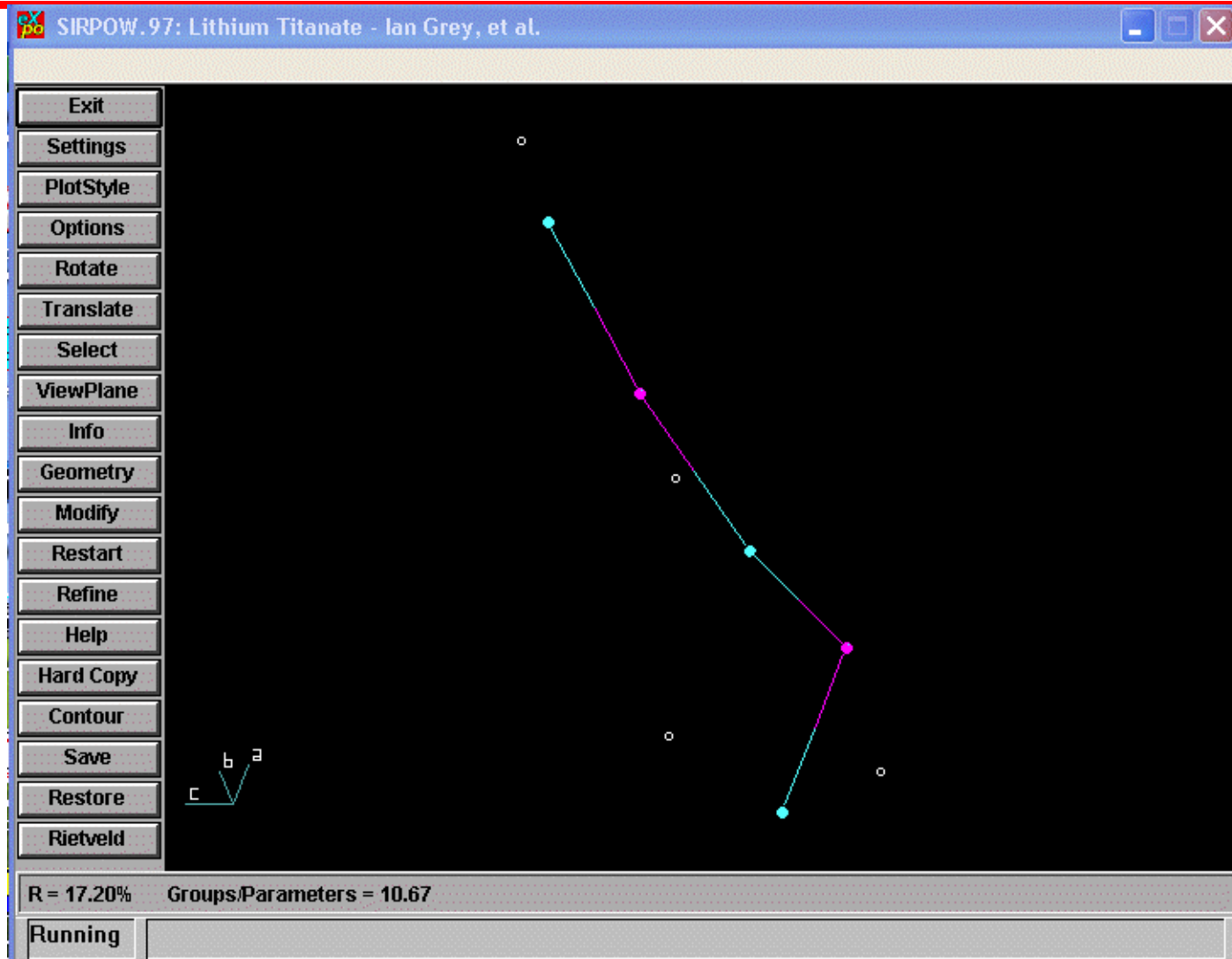
Use this information in Extra

?

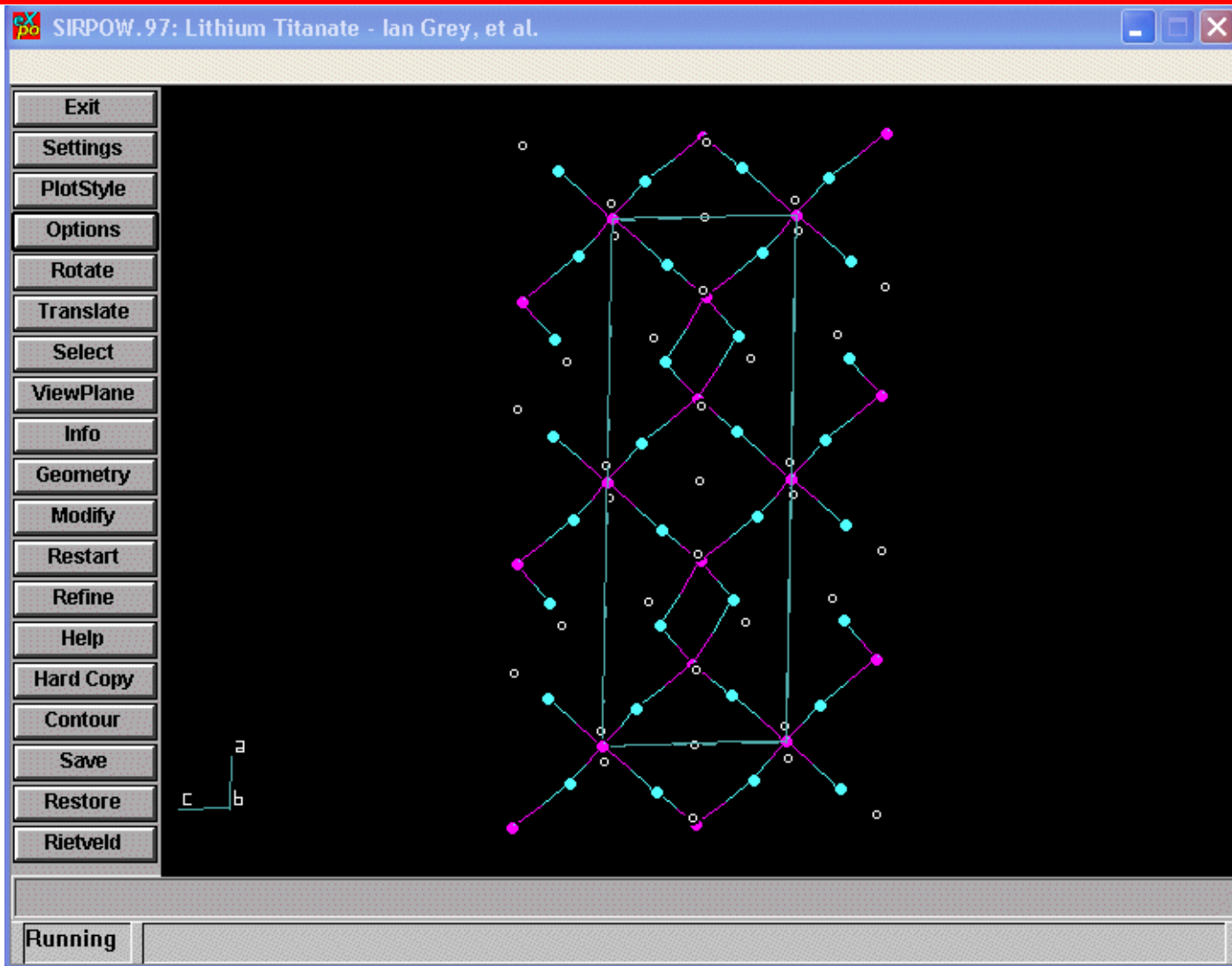
YES NO

Running

$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



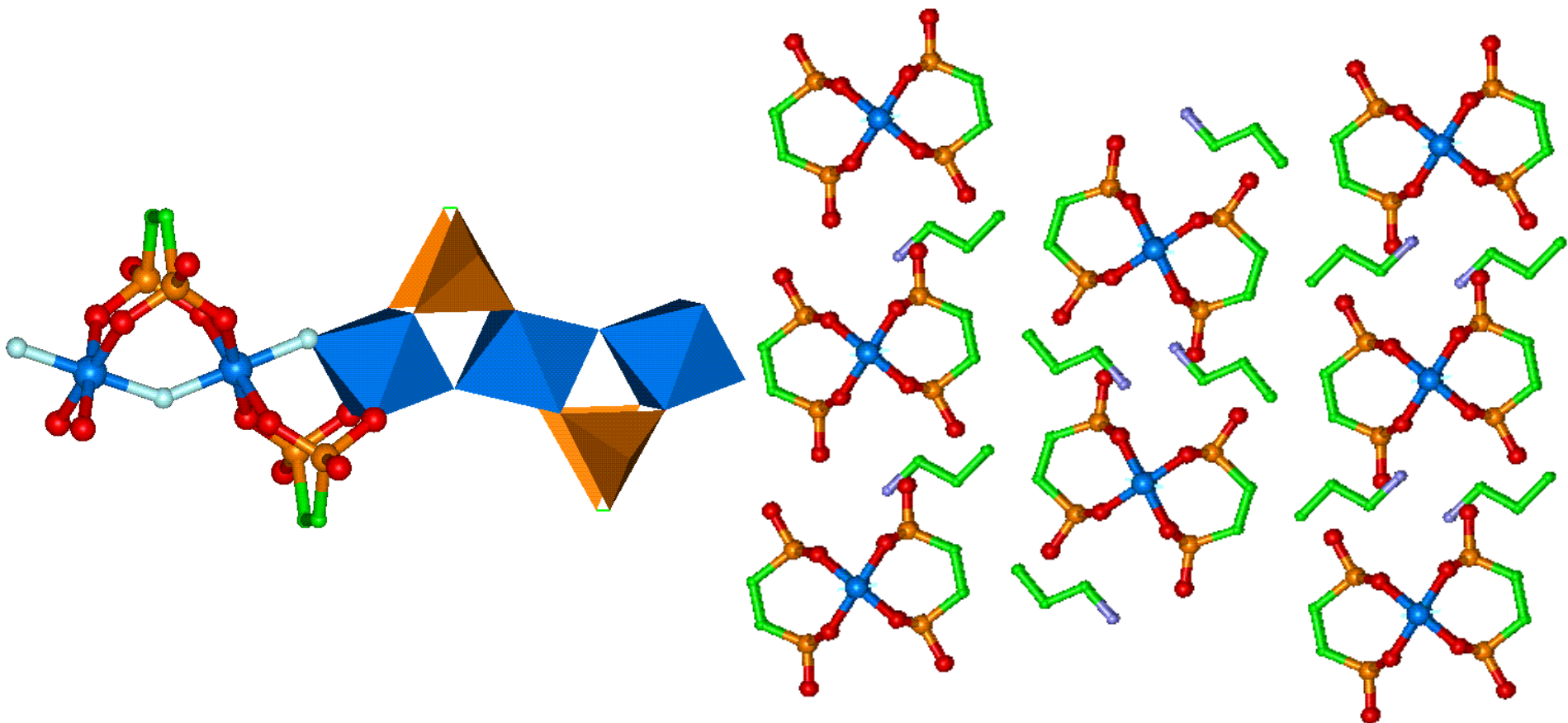
SIRPOW.97: Lithium Titanate - Ian Grey, et al.

```
At 14: 9:44  EXPO runs on: liti
data
extraction
normal  B = 0.400
invariants
phase  Trial 32 / 32 CFom = 0.026 (max = 0.631)
fourier Set# 1 Final R = 17.20%
menu
end
At 14:10:34 Expo ends ok      Click to finish !
```

Final R = 17.20% Click to finish !

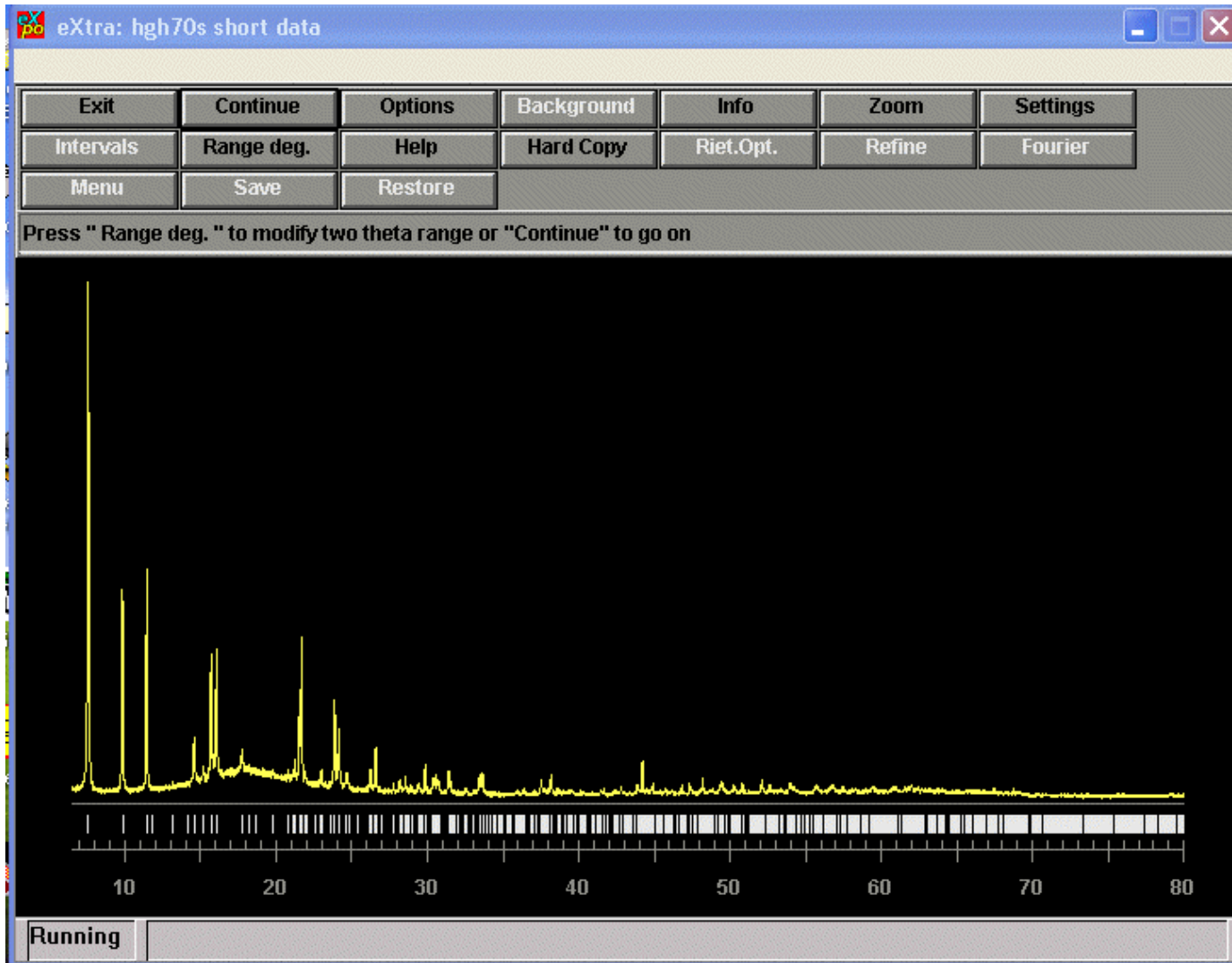
Running

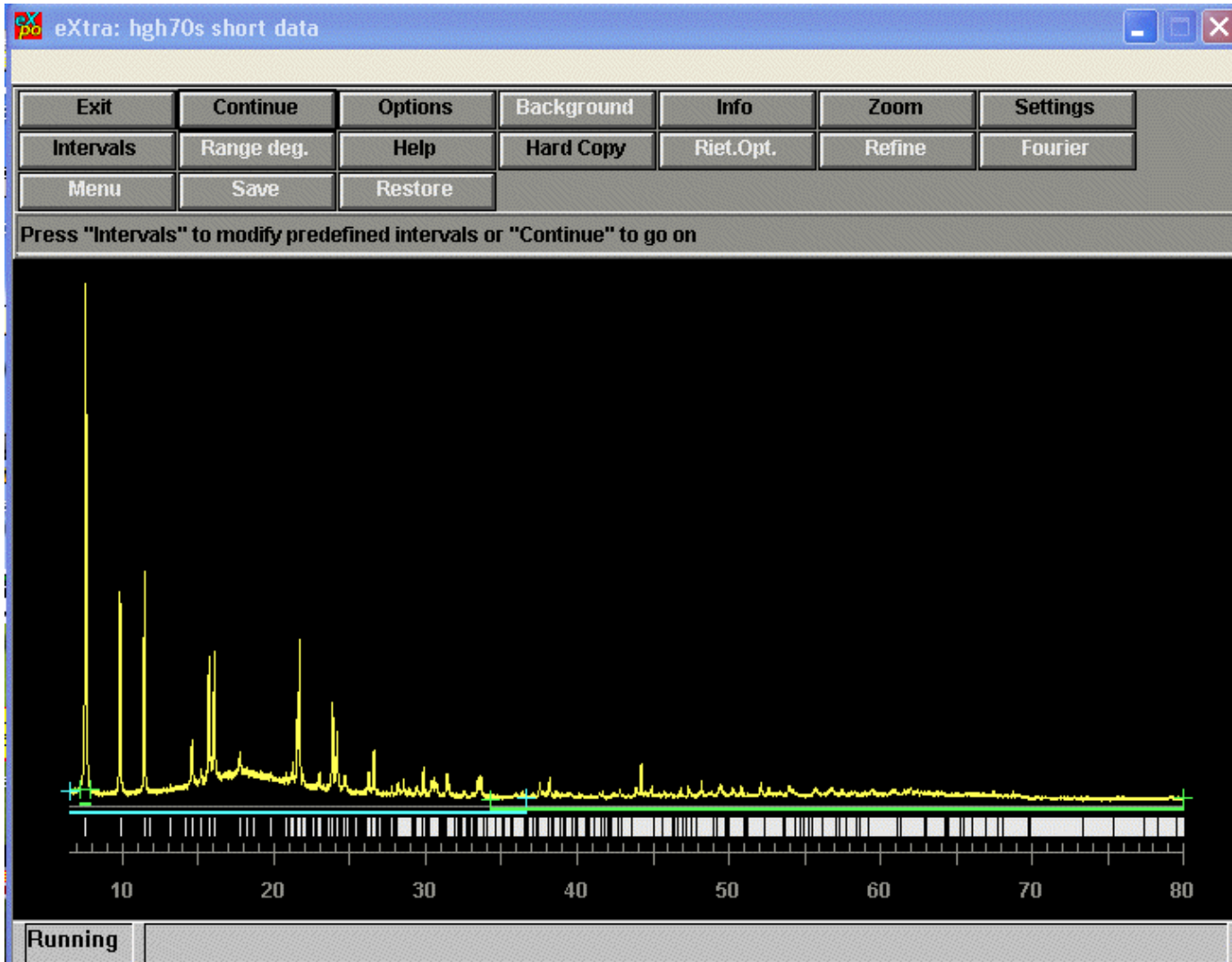
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: Structure

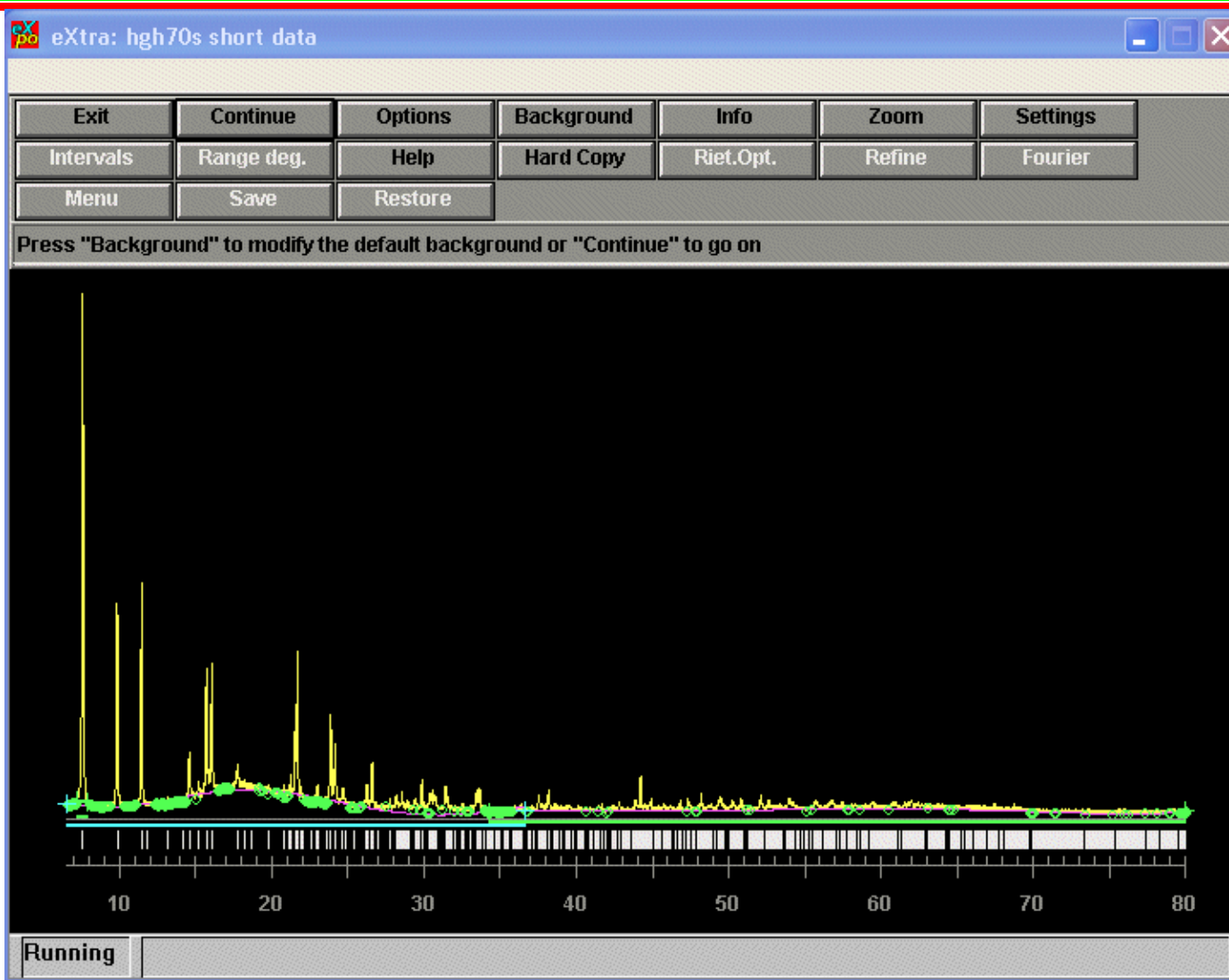


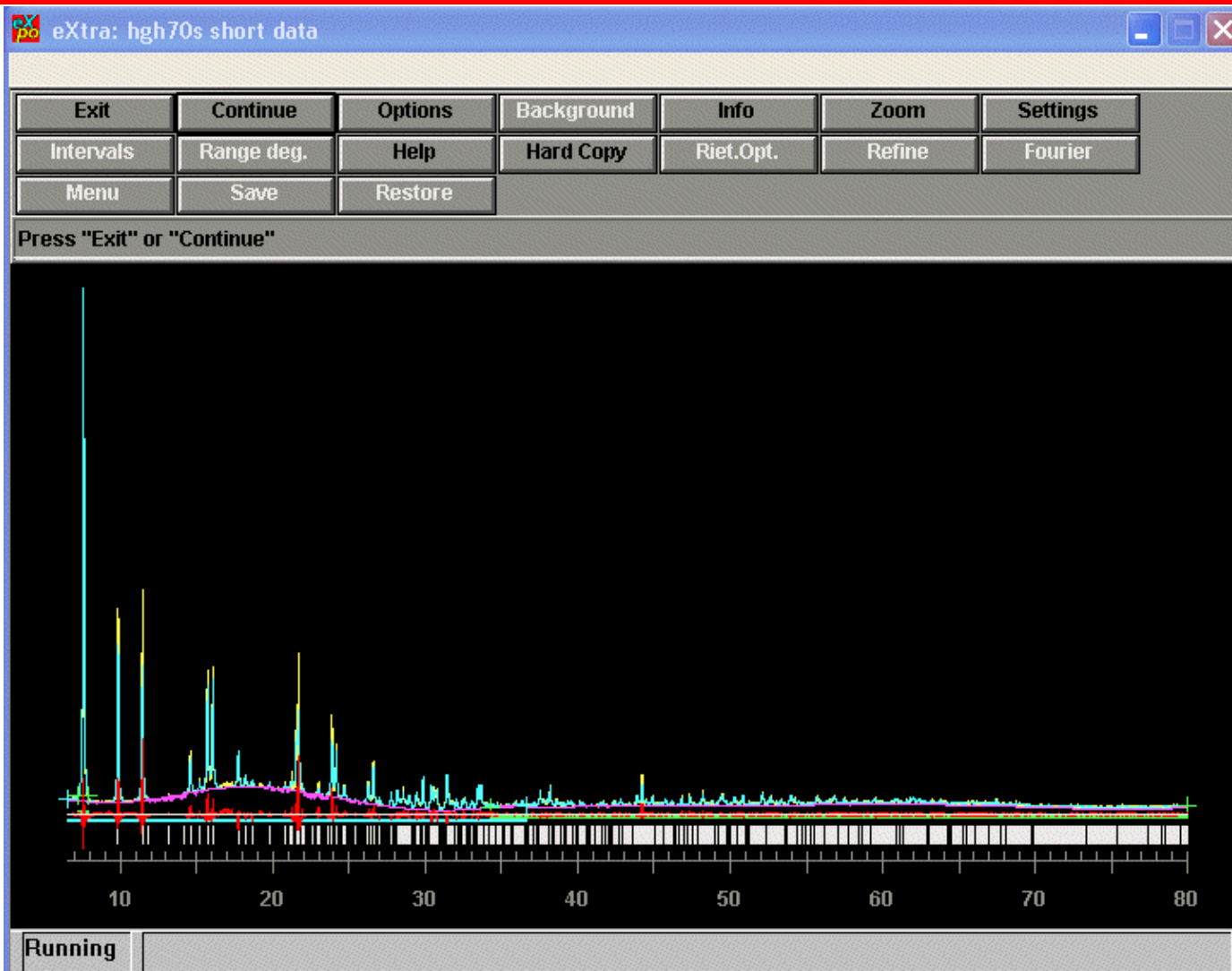
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]:$ **.exp file**

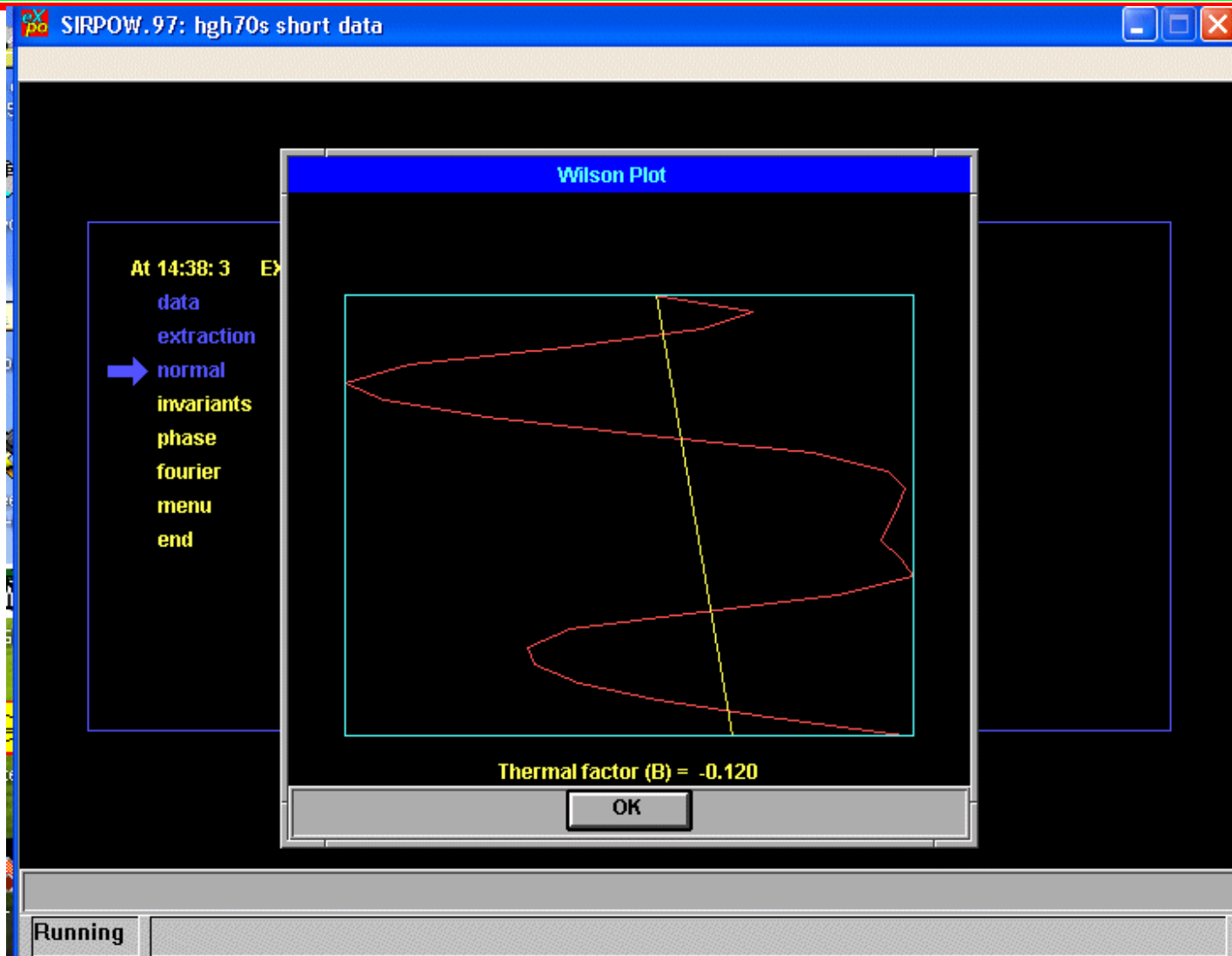
```
%struct hgh70s
%job hgh70s short data
%init
%data
range 6.5000 80.000 0.01
pattern 70s.pow
cont Al 4 P 8 F 4 O 24 C 24 N 4
wave 1.299905
cell 6.9061 8.2049 19.6604 90.000 90.000 90.000
space p 21 21 21
synchrotron
%extraction
%continue
```

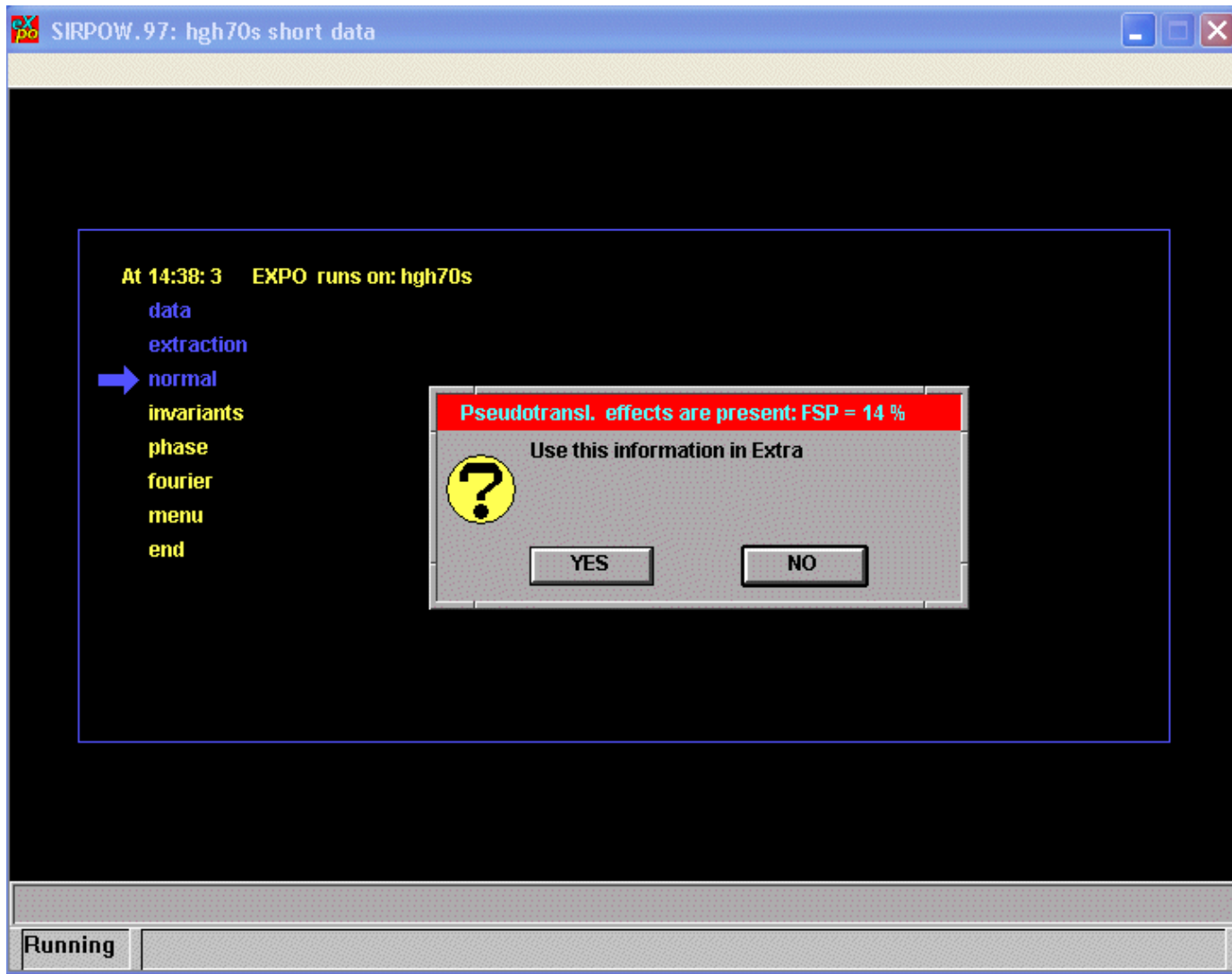


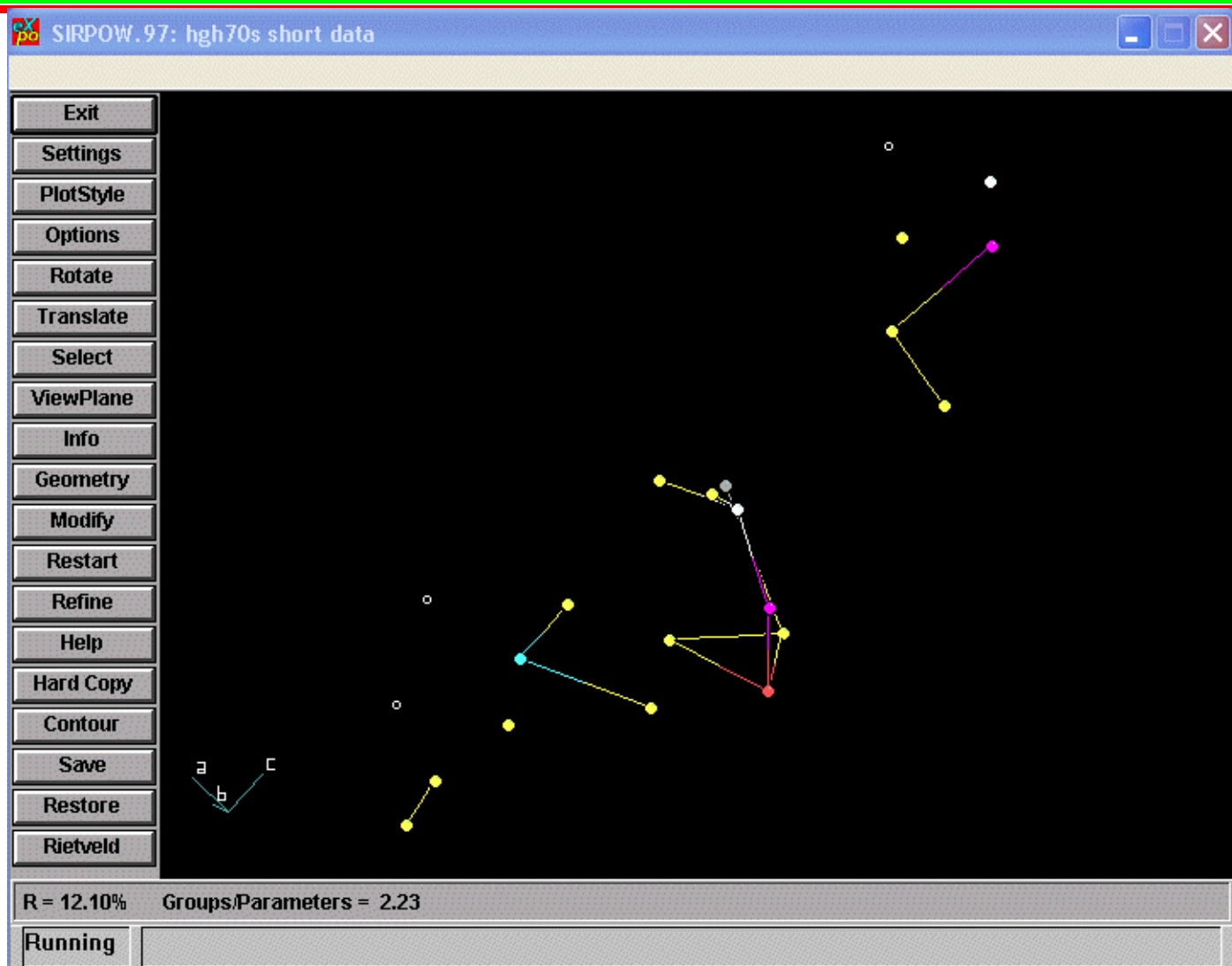


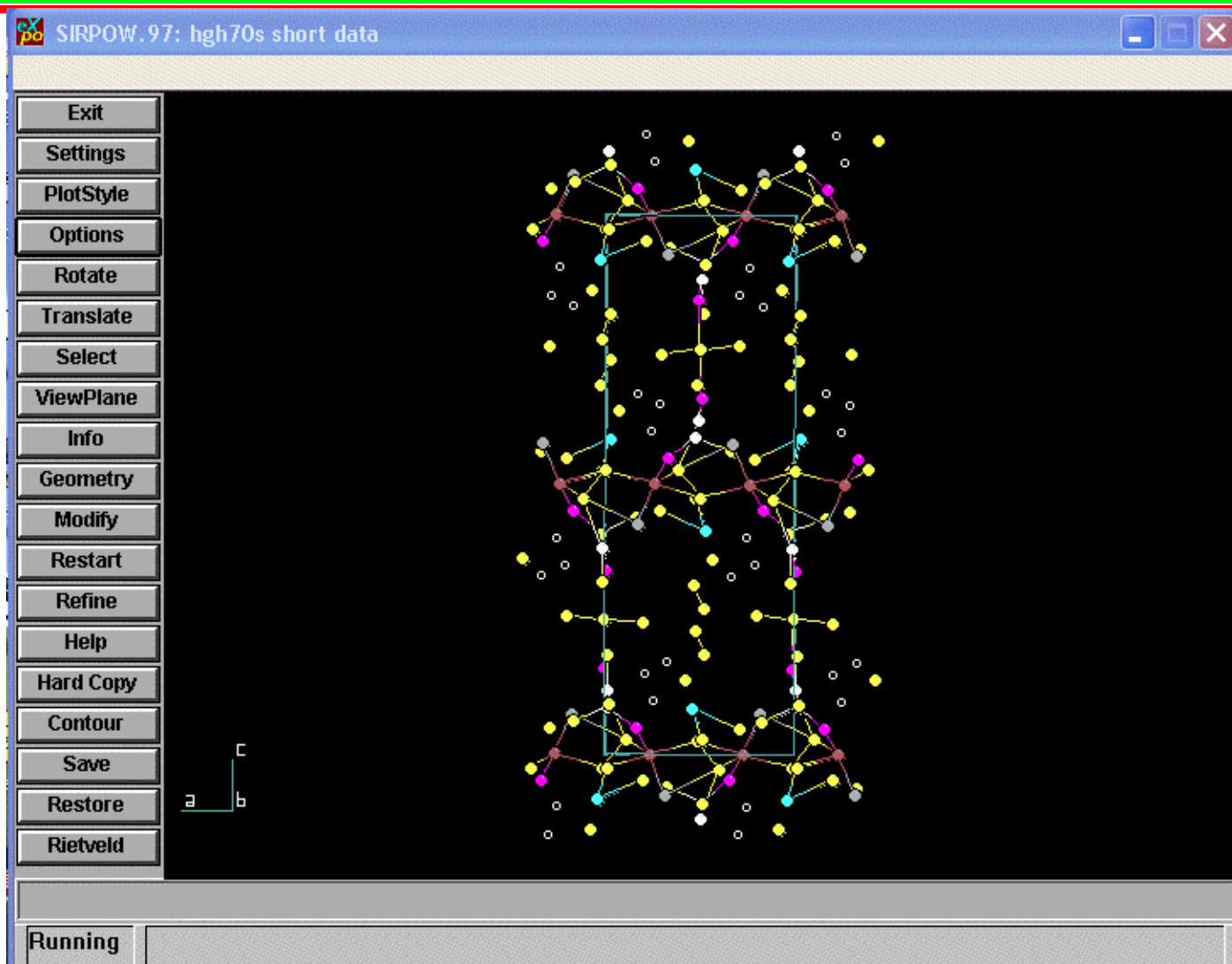


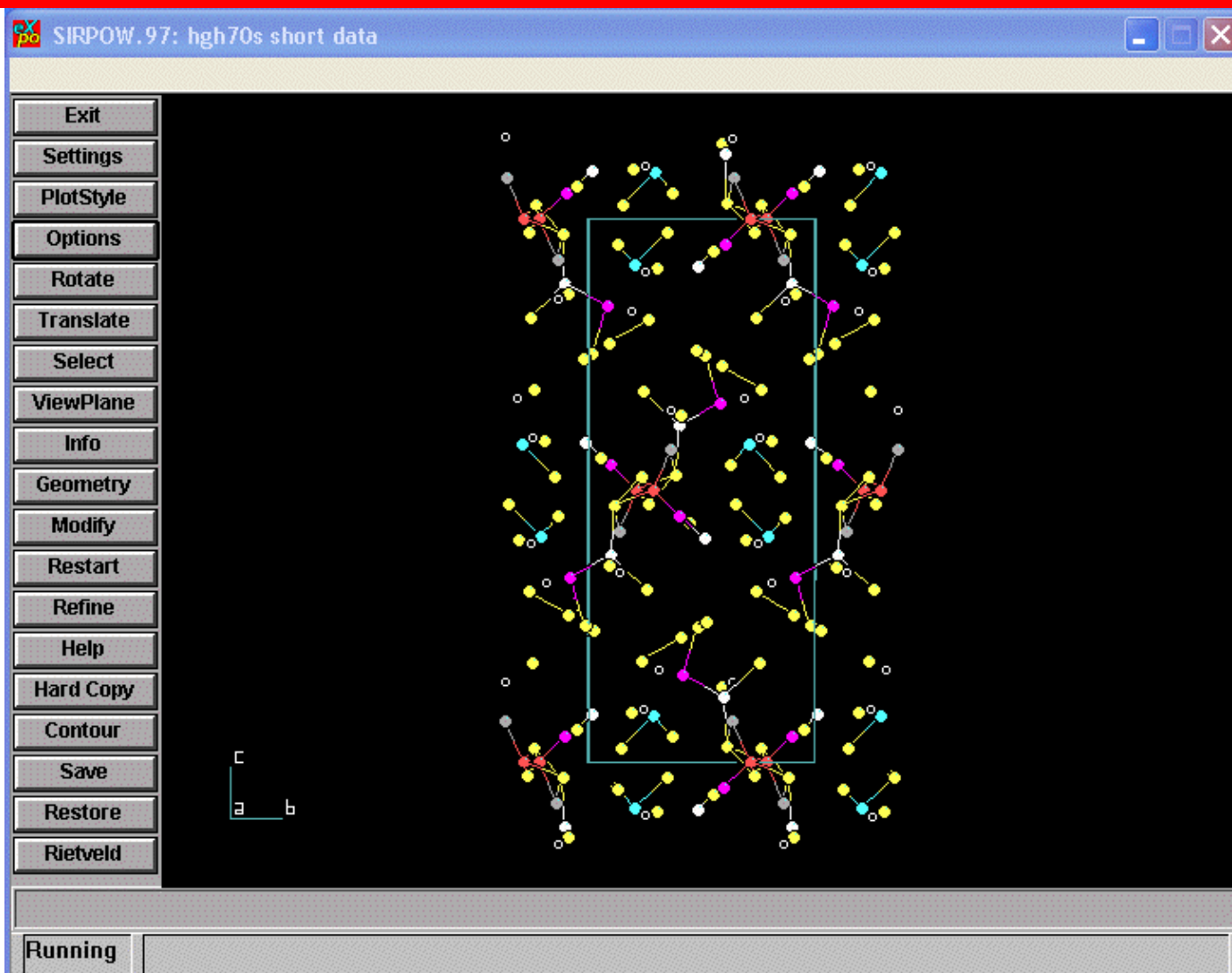














SIRPOW. 97: hgh70s short data

At 14:38: 3 EXPO runs on: hgh70s
data
extraction
normal B = -0.120
invariants
phase Trial 48 / 48 CFom = 0.358 (max = 0.450)
fourier Set# 1 Final R = 12.10%
fourier
fourier Set# 1 Final R = 12.10%
menu
end
At 14:48: 5 Expo ends ok **Click to finish !**

Final R = 12.10% **Click to finish !**

Running

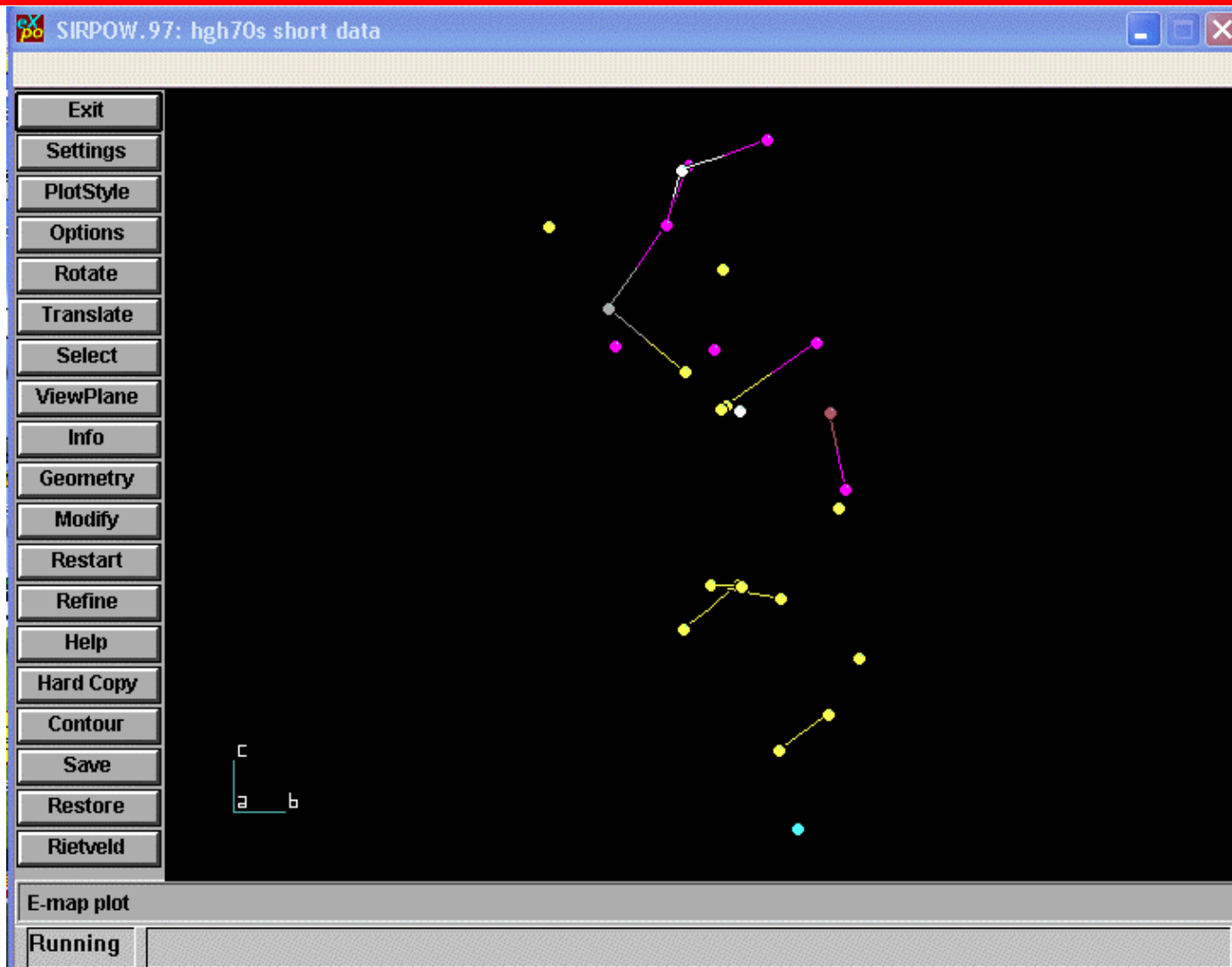
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: No Fourier recycling .exp file

```
%struct hgh70s
%job hgh70s short data
%init
%data
range 6.5000 80.000 0.01
pattern 70s.pow
cont Al 4 P 8 F 4 O 24 C 24 N 4
wave 1.299905
cell 6.9061 8.2049 19.6604 90.000 90.000 90.000
space p 21 21 21
synchrotron
%extraction
%normal
%invariants
%phase
%fourier
set 1
recyc 0
%continue
```

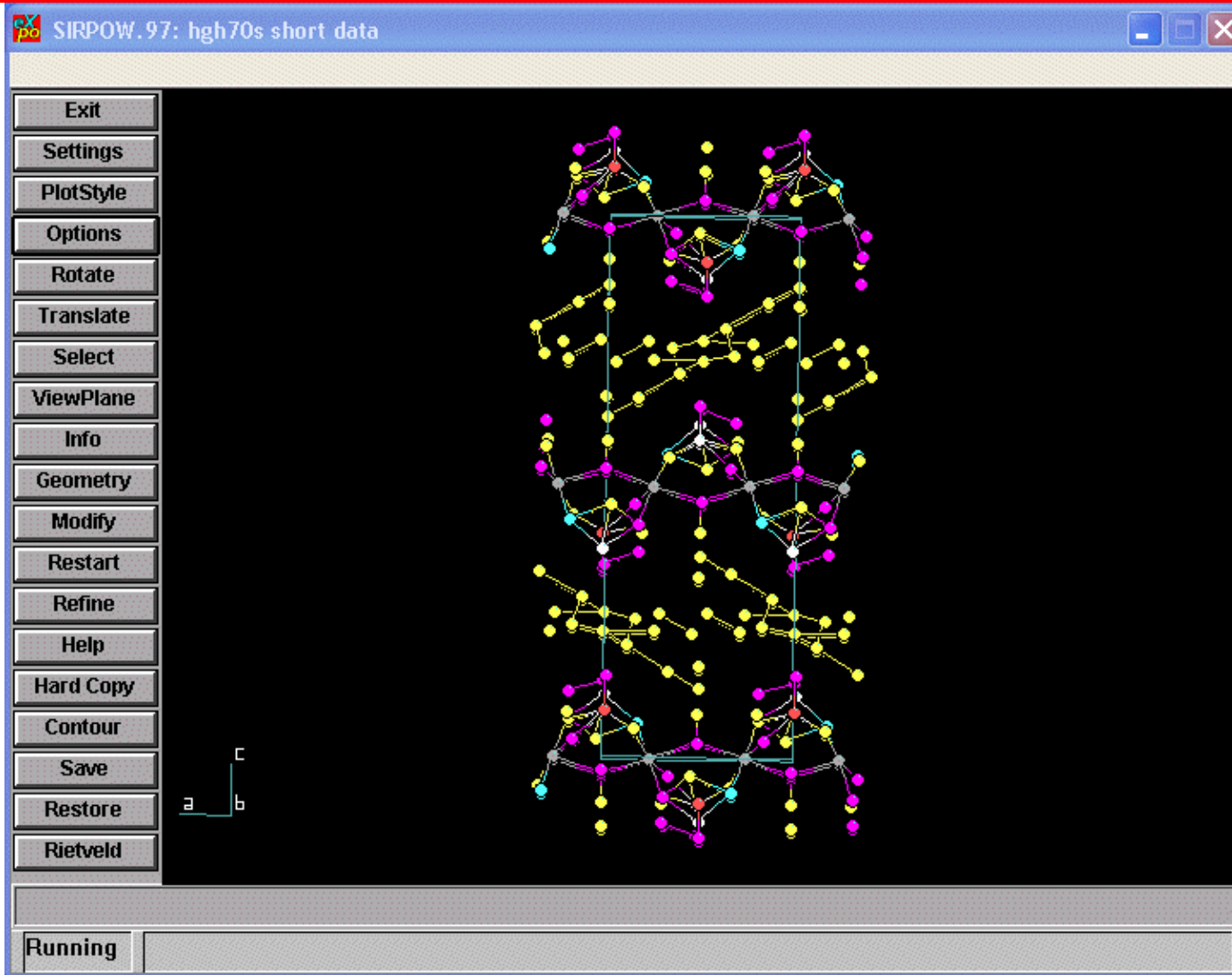
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]:$ No Fourier recycling

The image shows a screenshot of the SIRPOW.97 software interface. The window title is "SIRPOW.97: hgh70s short data". On the left side, there is a vertical menu with buttons for: Exit, Settings, PlotStyle, Options, Rotate, Translate, Select, ViewPlane, Info, Geometry, Modify, Restart, Refine, Help, Hard Copy, Contour, Save, Restore, and Rietveld. The main area displays a 3D ball-and-stick model of a crystal structure with a unit cell box. A dialog box titled "Restart Fourier/Least-Squares refinement" is open in the center. It has a red header bar. Inside the dialog, there are two radio buttons: "Default" (unselected) and "User defined" (selected). To the right, there are two spinners: "Number of cycle(s)" set to 0 and "Set #" set to 1. At the bottom of the dialog are "OK" and "Cancel" buttons. The status bar at the bottom left shows "Running".

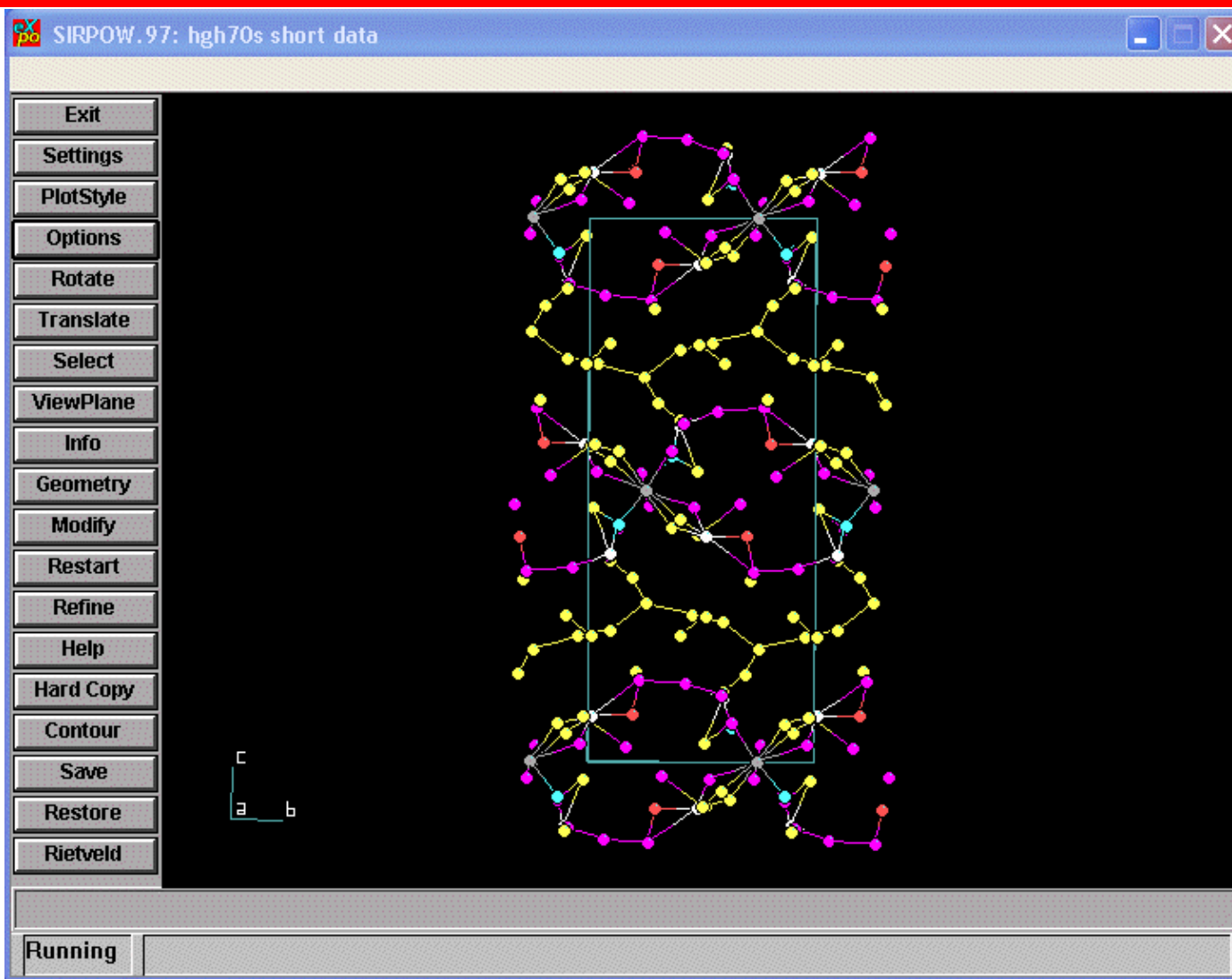
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]:$ No Fourier recycling



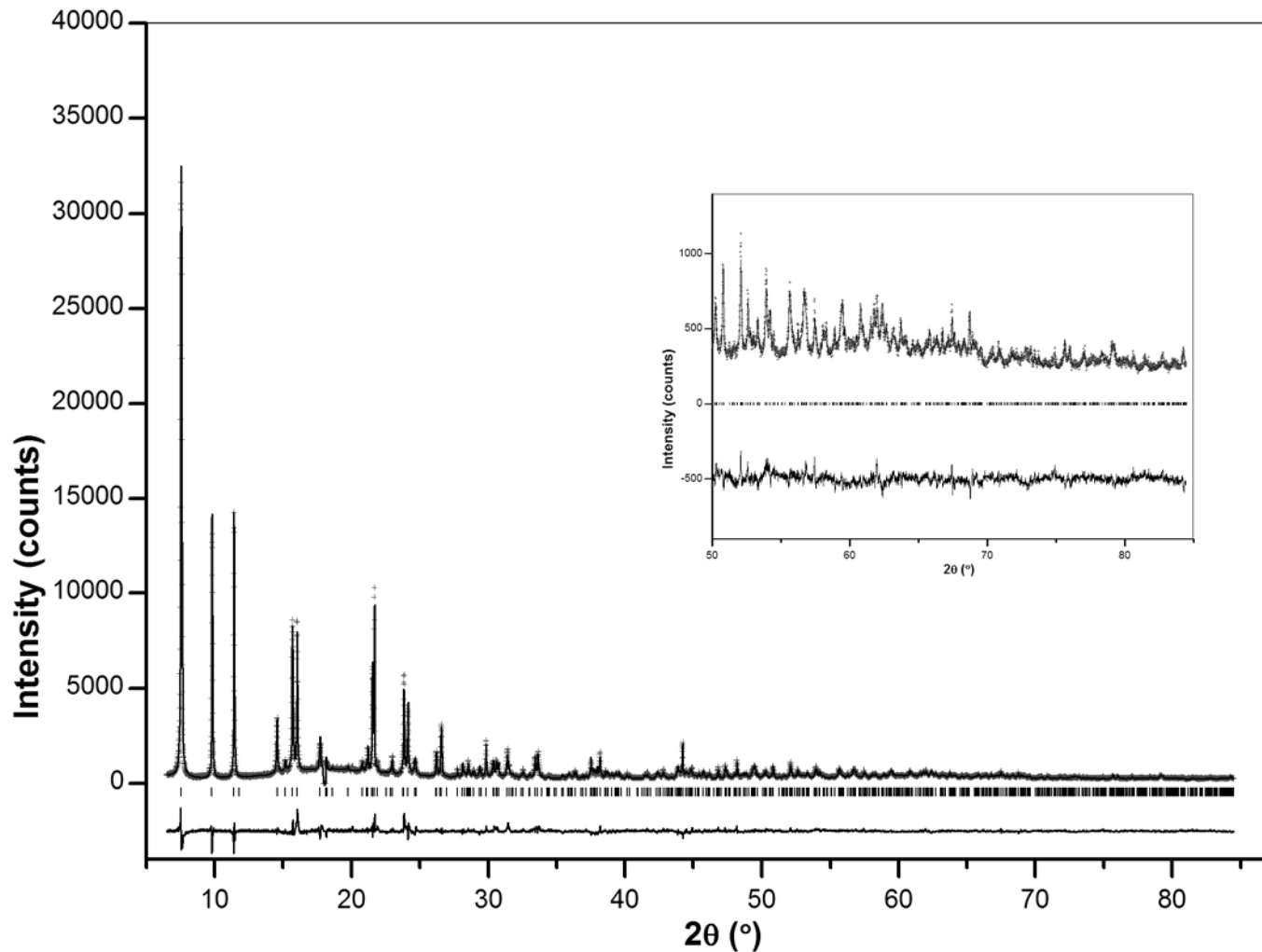
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: No Fourier recycling



$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]:$ No Fourier recycling



$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: Le Bail fit using GSAS



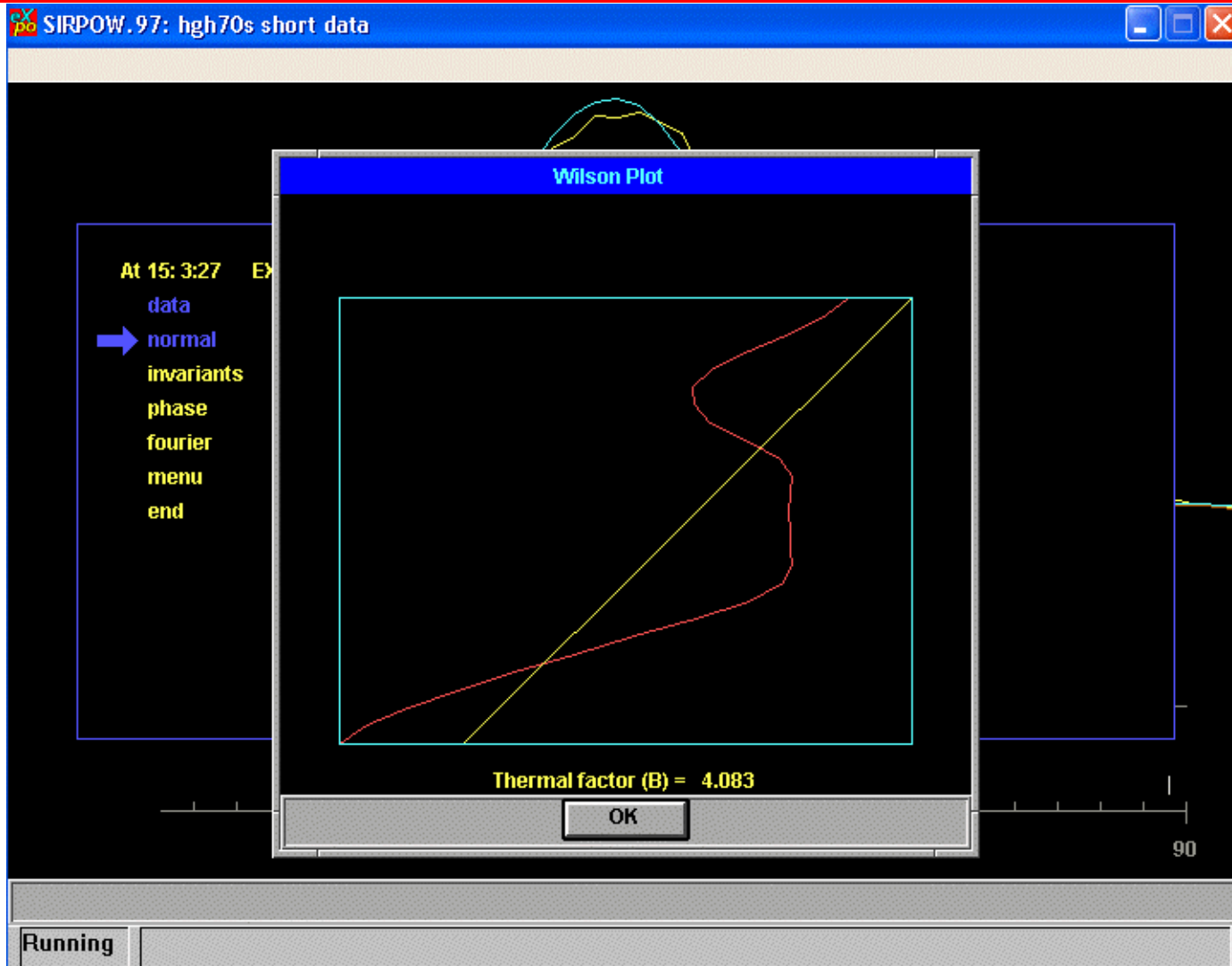
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: External Extraction hkl file

0	0	2	.090	571229.
0	1	1	.069	151954.
1	0	1	.069	190523.
0	1	2	.083	429.
1	0	2	.083	0.
1	1	0	.063	0.
0	1	3	.090	84501.
1	1	1	.069	9387.
0	0	4	.105	35472.
1	0	3	.090	269052.
1	1	2	.083	137870.
0	1	4	.098	97481.
1	1	3	.090	466.
0	2	0	.063	8530.
1	0	4	.098	0.
0	2	1	.069	0.
0	2	2	.076	20164.
1	1	4	.098	16046.
0	1	5	.105	39458.
1	2	0	.063	52715.
0	2	3	.090	190025.
1	2	1	.069	97607.
1	0	5	.105	37295.
2	0	0	.063	759254.
2	0	1	.069	373.
0	0	6	.113	23530.

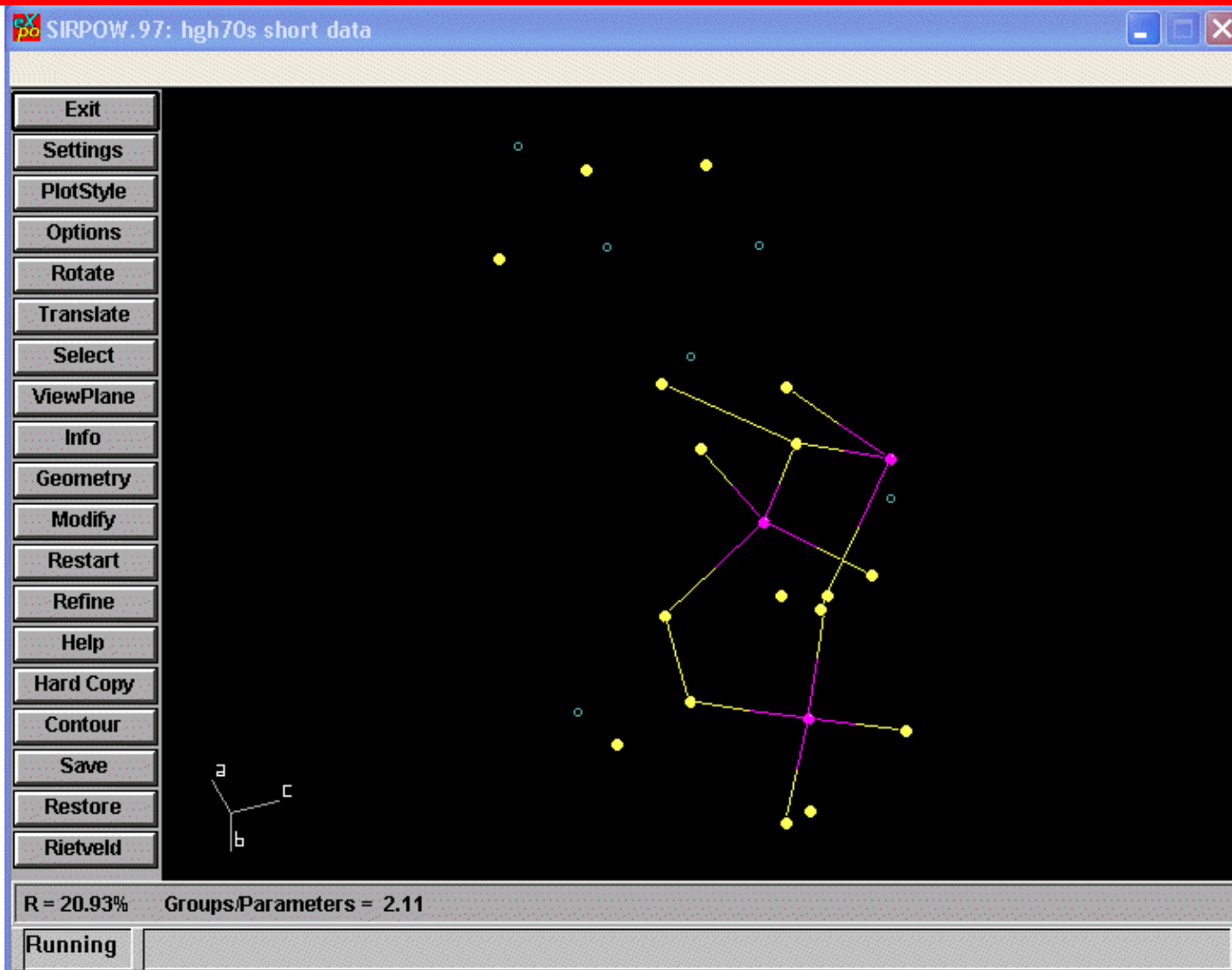
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: External Extraction .exp file

```
%window
%struct hgh70s
%init
%job hgh70s short data
%data
wavelength 1.299905
cell      6.9061  8.2049  19.6604  90.000  90.000  90.000
space P 21 21 21
ref2 70sgsa.fou
content  Al    4  P    8  F    4  O    24  C    24  N    4
%normal
%continue
```

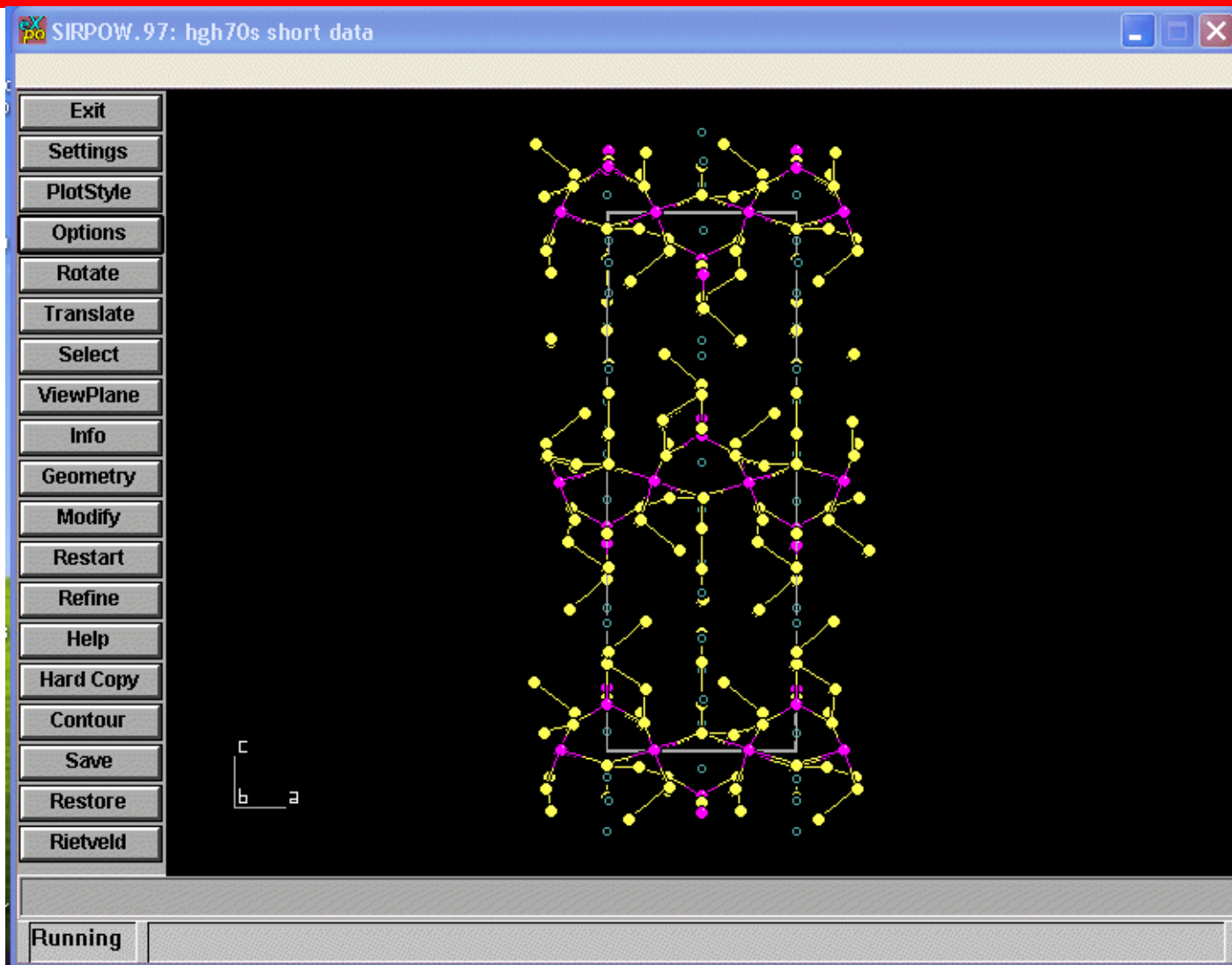
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: External Extraction



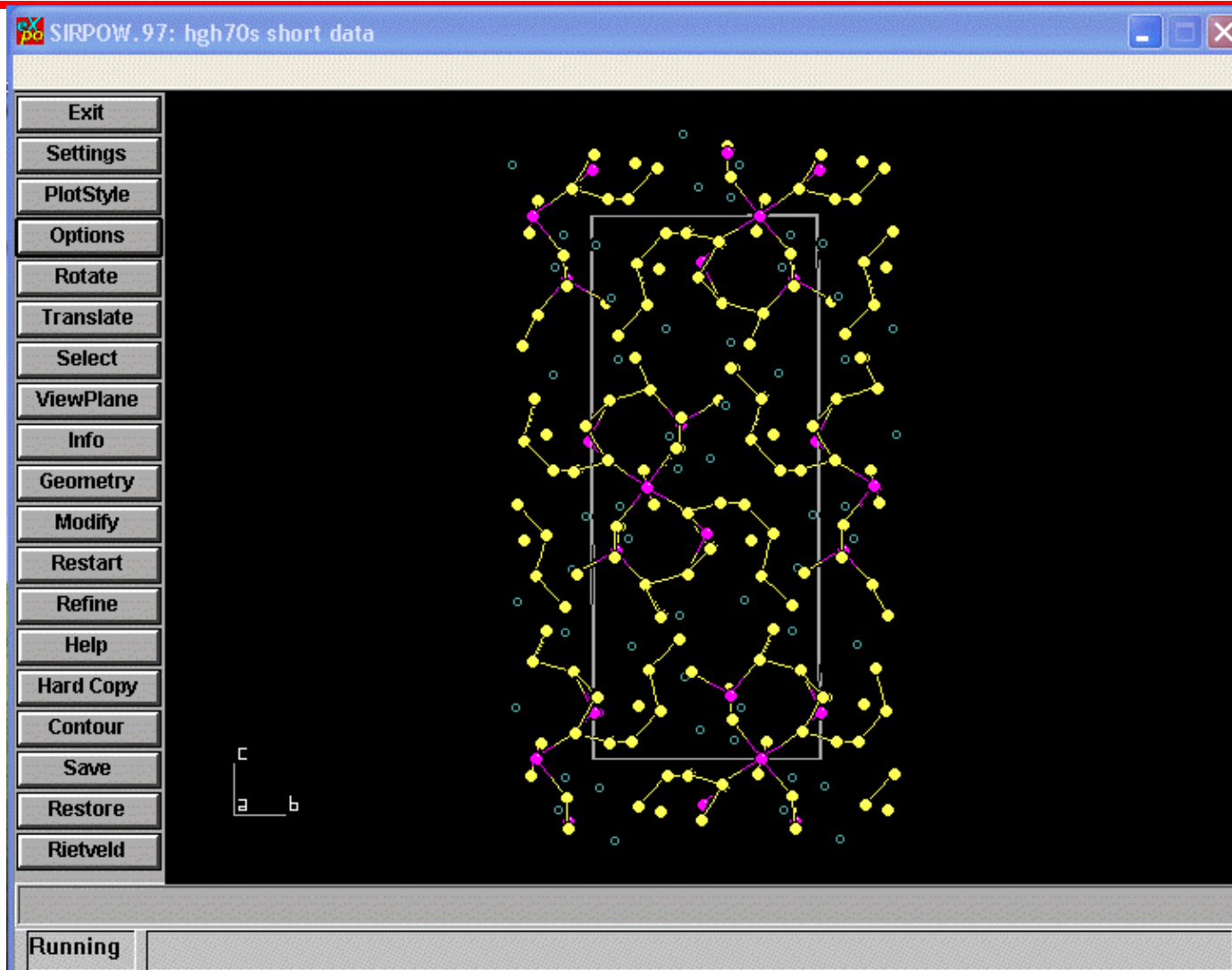
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]:$ External Extraction



$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]:$ External Extraction



$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: External Extraction



Summary

- **Adjust emphasis of high angle data**
 - » **Cut data range**
 - » **Varying overall thermal factor**
- **Look at starting (un-recycled) solution**
- **Intensity extraction**
- **Other:**
 - » **Increase number of reflection used in phasing**
 - » **Look at other trial solutions with similar CFOM**
 - » **Other strategies used in direct methods**
 - » **<http://www.ba.cnr.it/IRMEC/SirWare.html>**