"Structure Solution using Direct Methods" - EXPO Structure Solution Software

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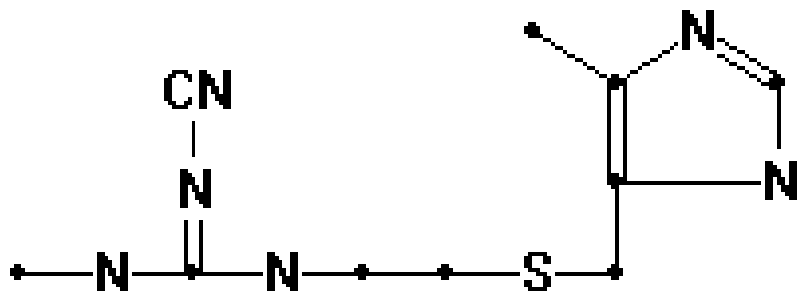
ECM-21

24/8/03

Cimetidine:
Structure



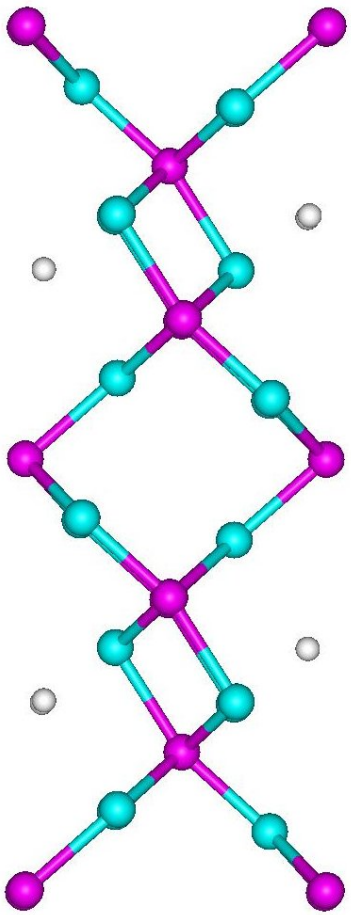
Cimetidine:
.exp file

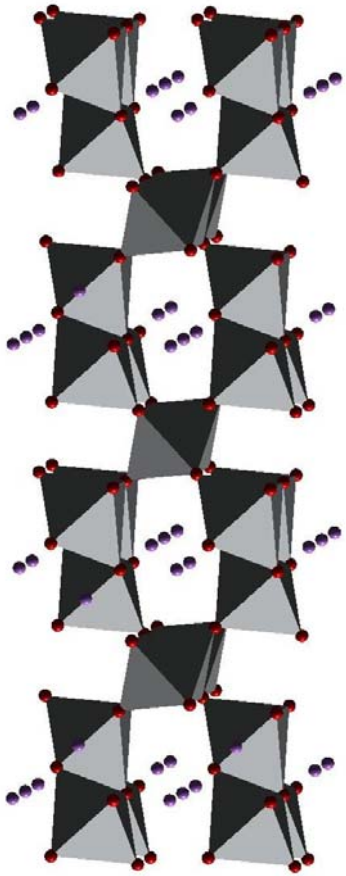
```
%struct cime
%job cimetidine -- Synchrotron data
%init
%data
range  8.01 84.99 0.01
pattern cime.pow
cont s 4 c 40 n 24 h 64
wave 1.52904
cell      10.6986  18.8181 6.8246       90.000   111.284    90.000
space p 21/n
sync
double
%extraction
%continue
```

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



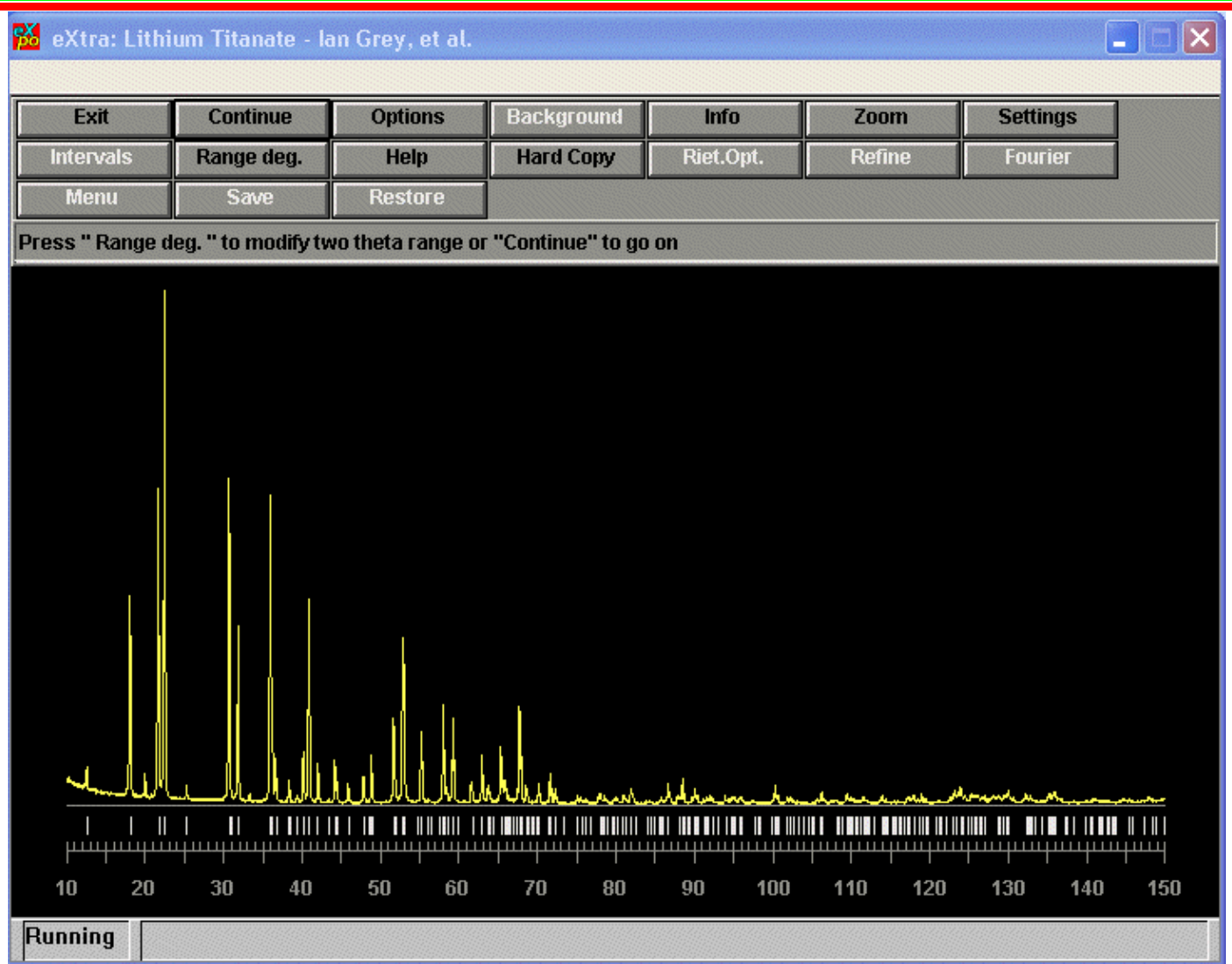


I. E. Grey, L. M. D. Cranswick, C. Li, L. A. Bursill, and J. L. Peng, *J. Solid State Chem.*, 1998, *138*, 74.

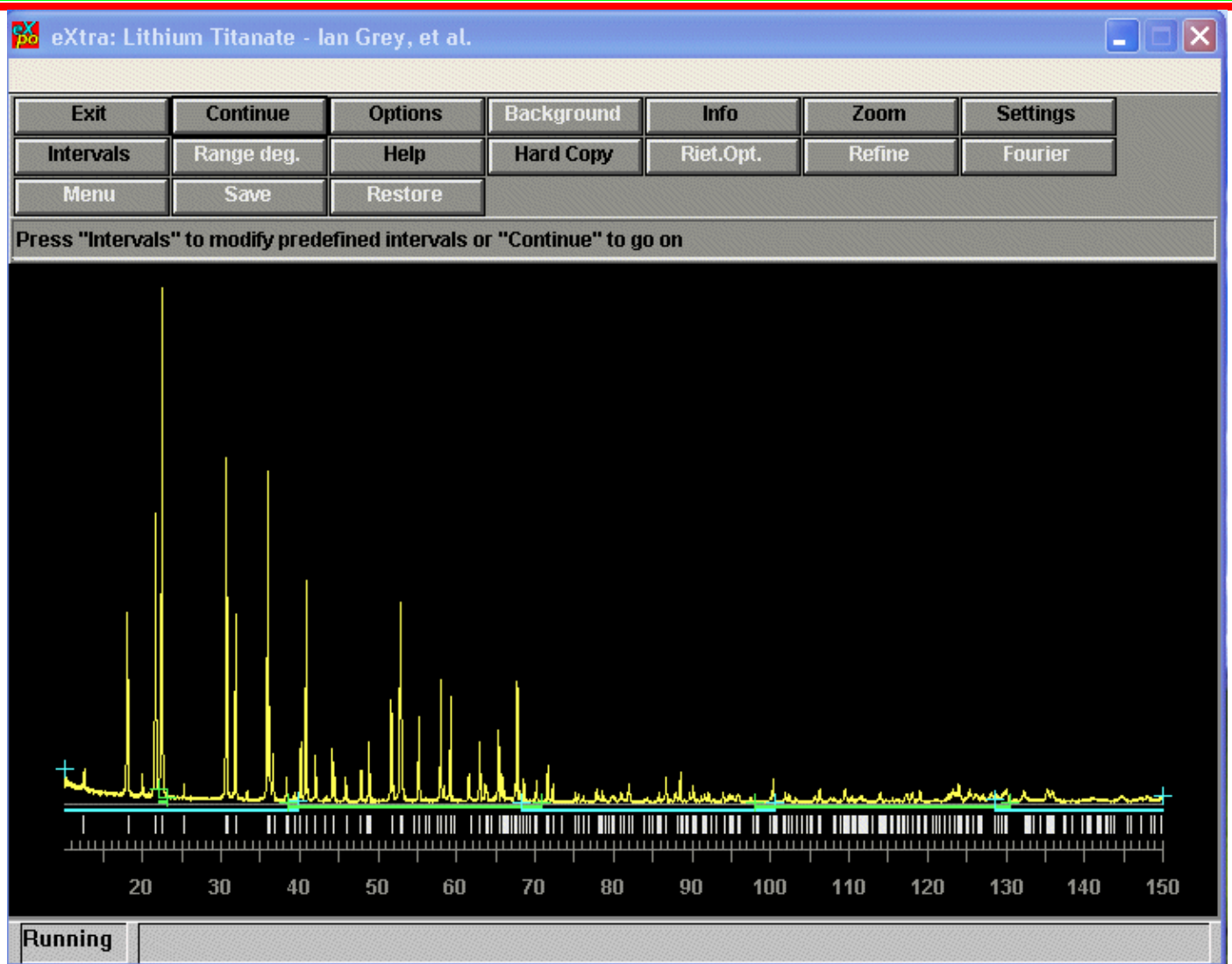
$Li_{0.98}Ti_{2.88}O_6$: .exp file

```
%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
```

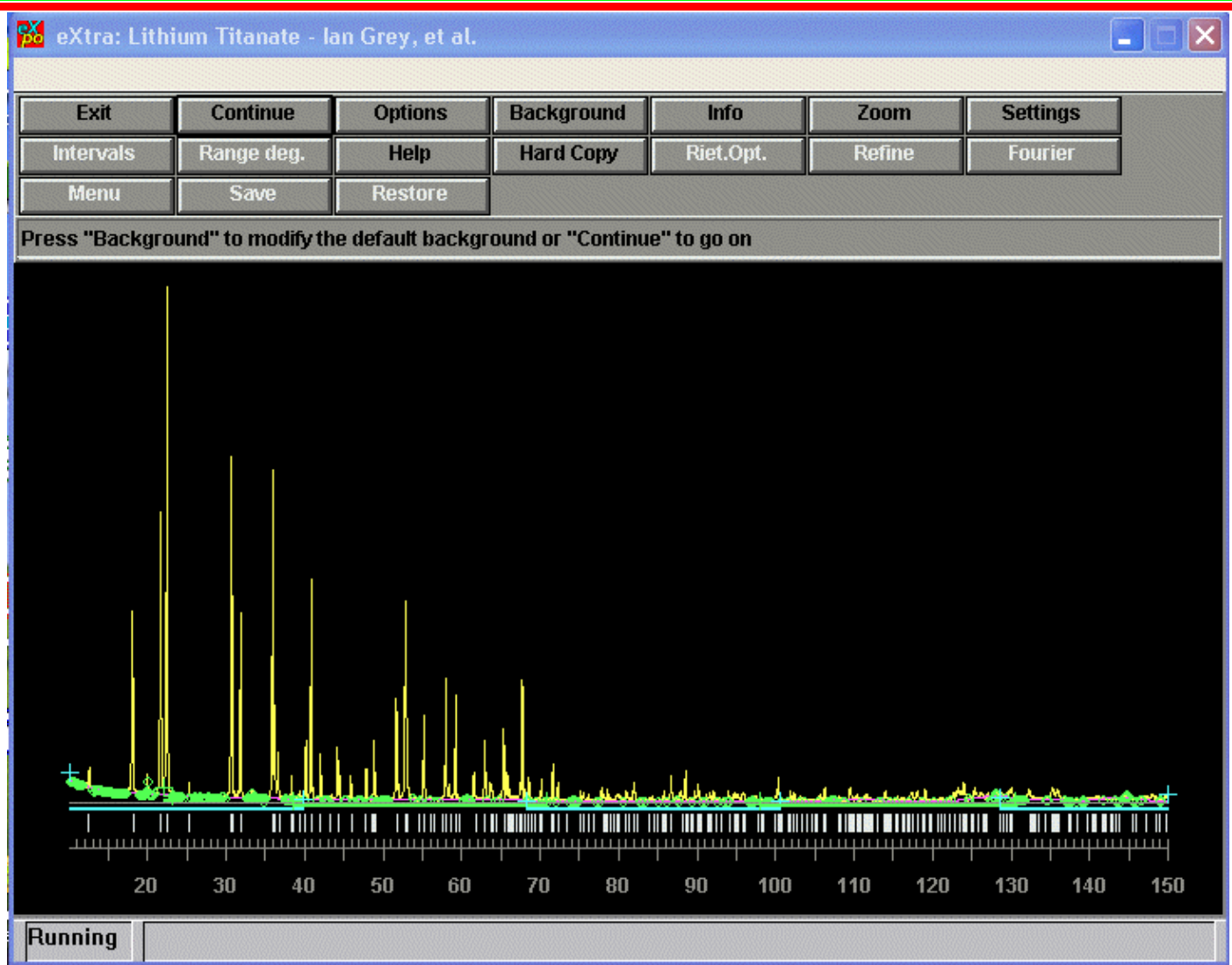
$Li_{0.98}Ti_{2.88}O_6$



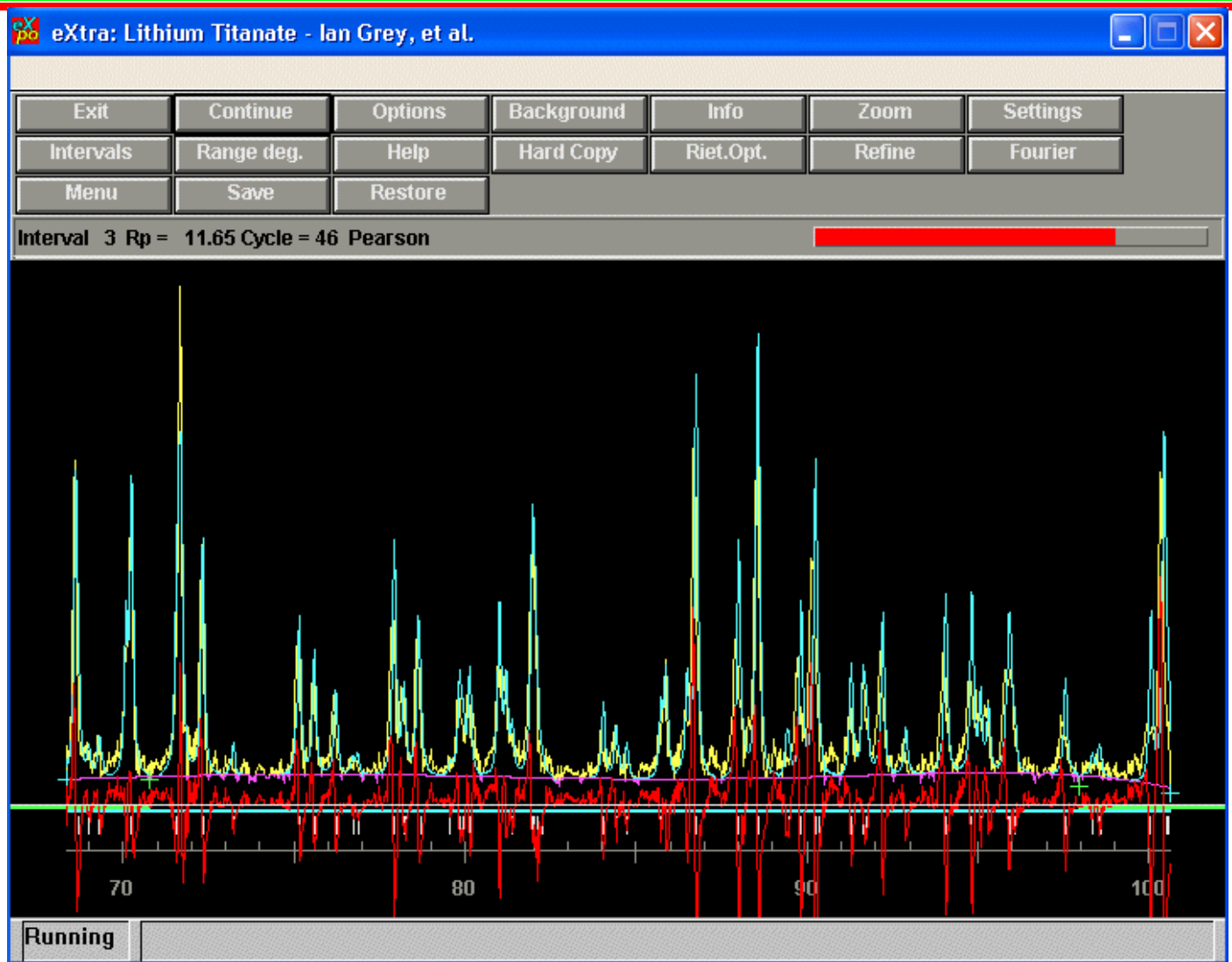
$Li_{0.98}Ti_{2.88}O_6$



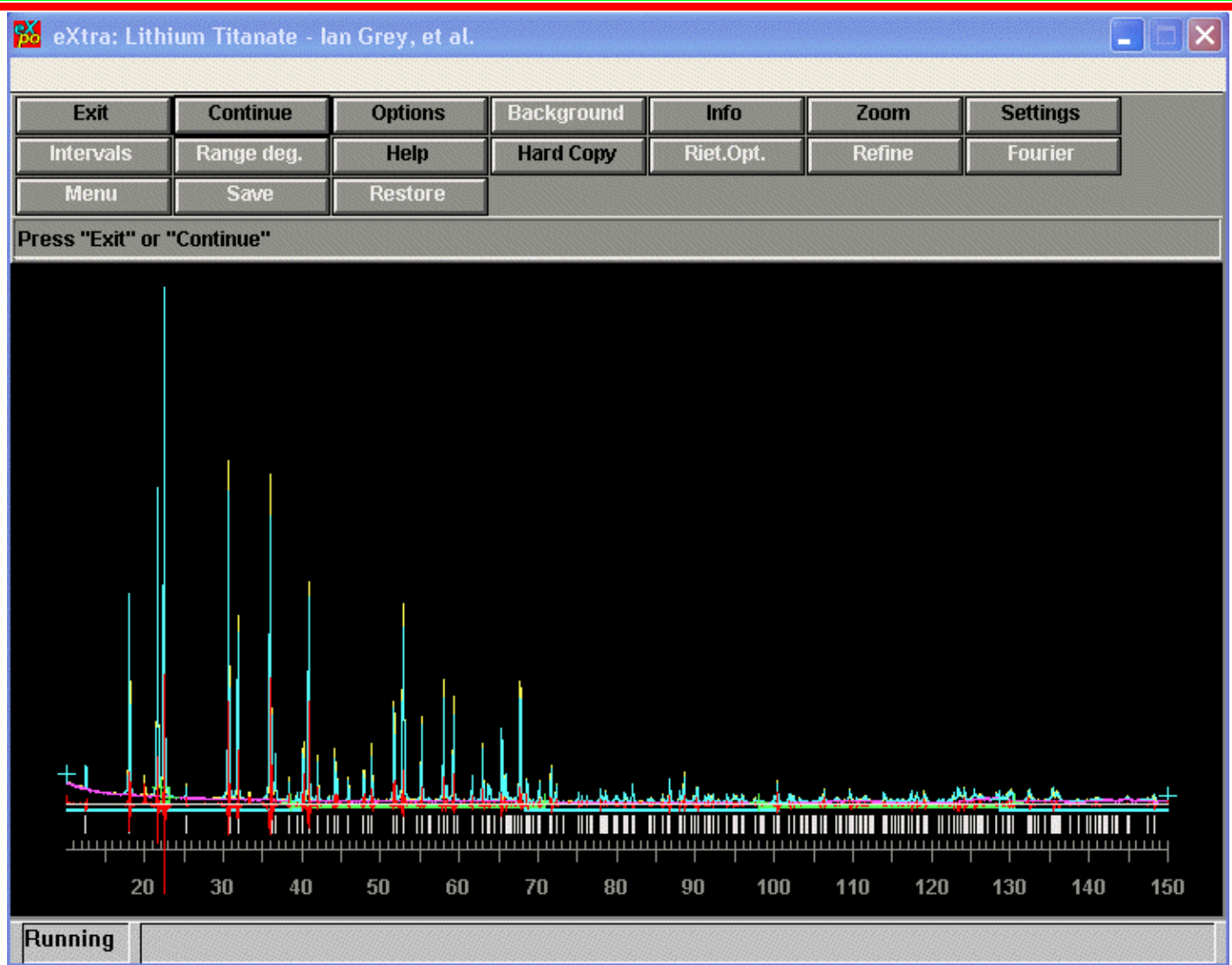
$Li_{0.98}Ti_{2.88}O_6$



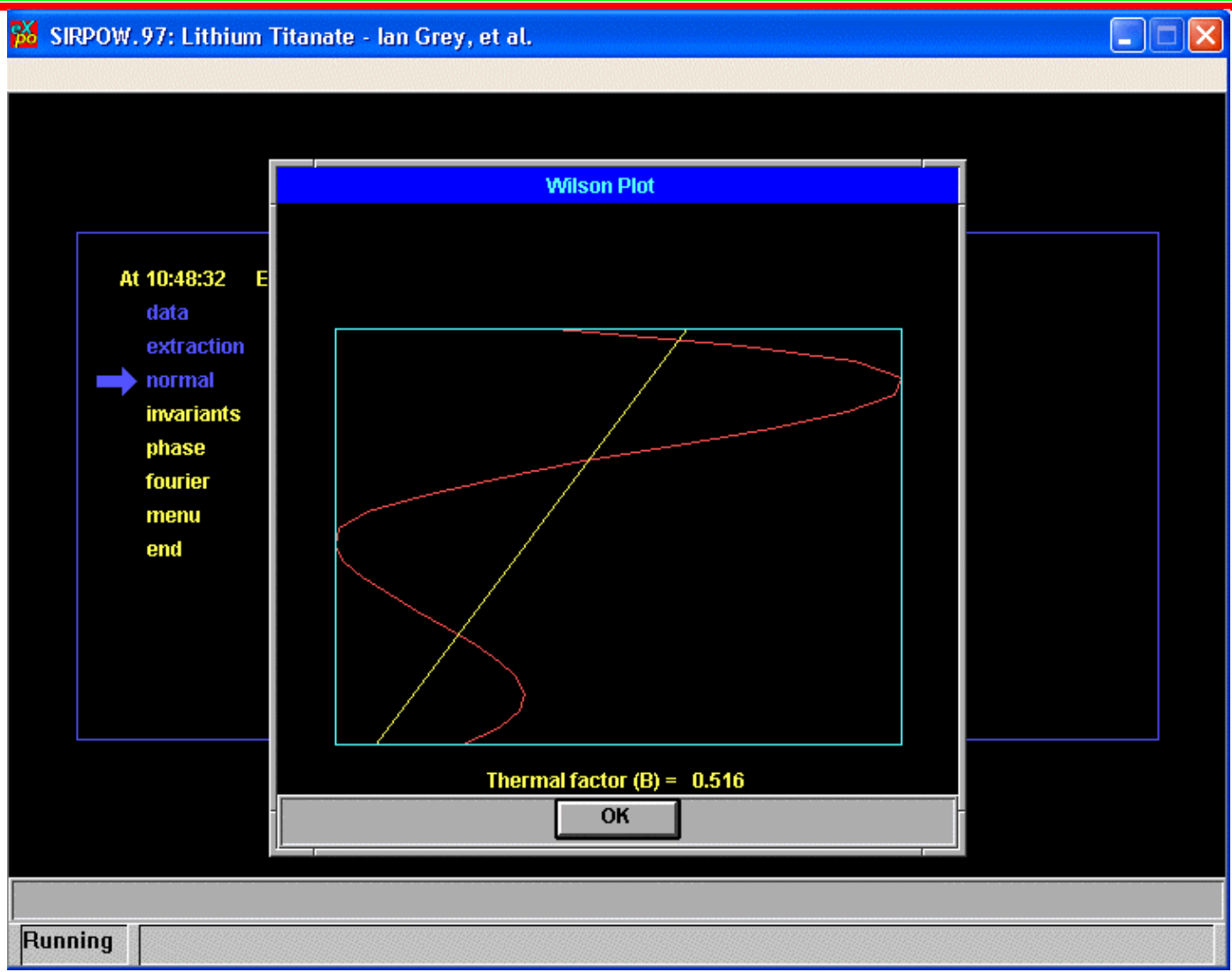
$Li_{0.98}Ti_{2.88}O_6$



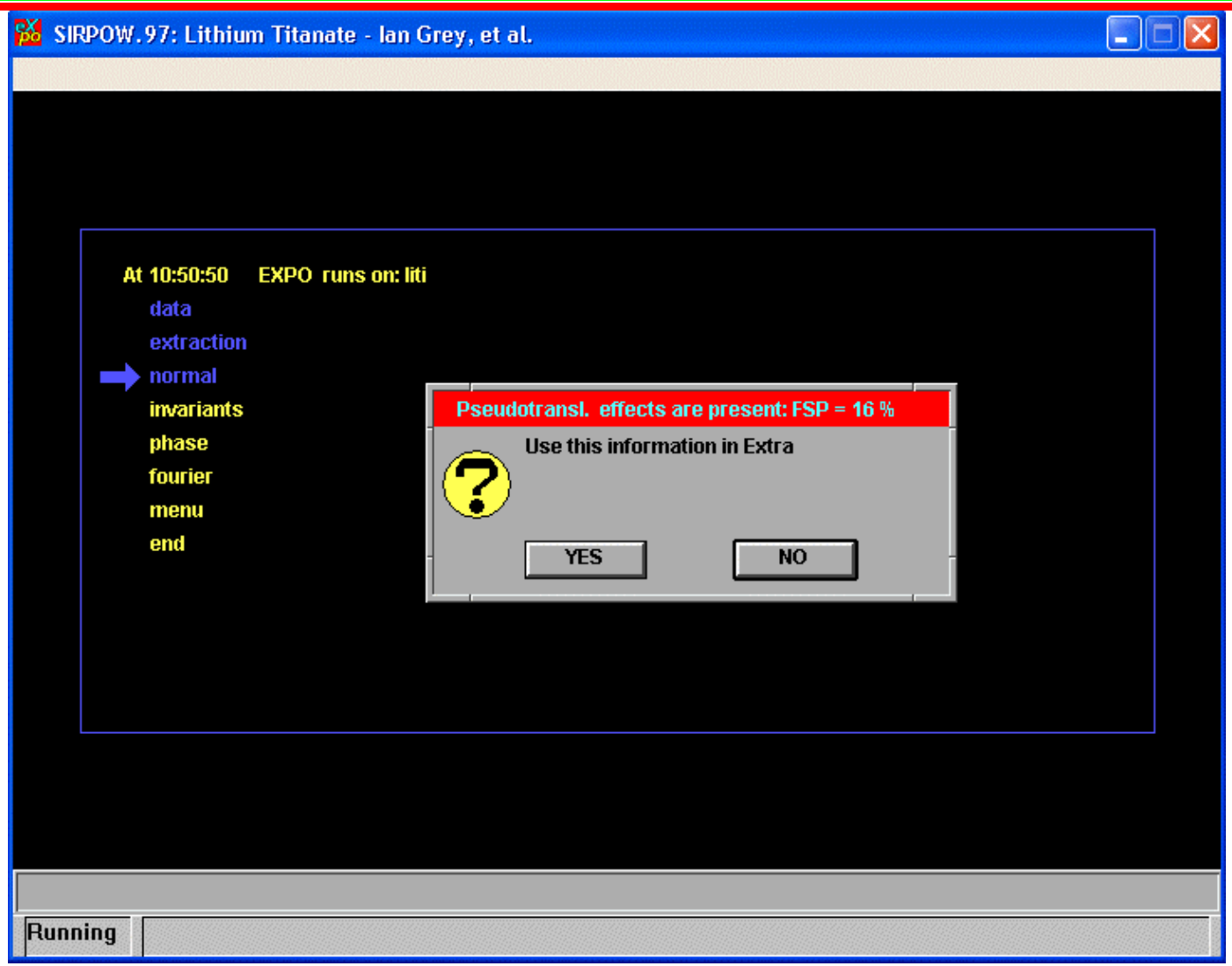
$Li_{0.98}Ti_{2.88}O_6$



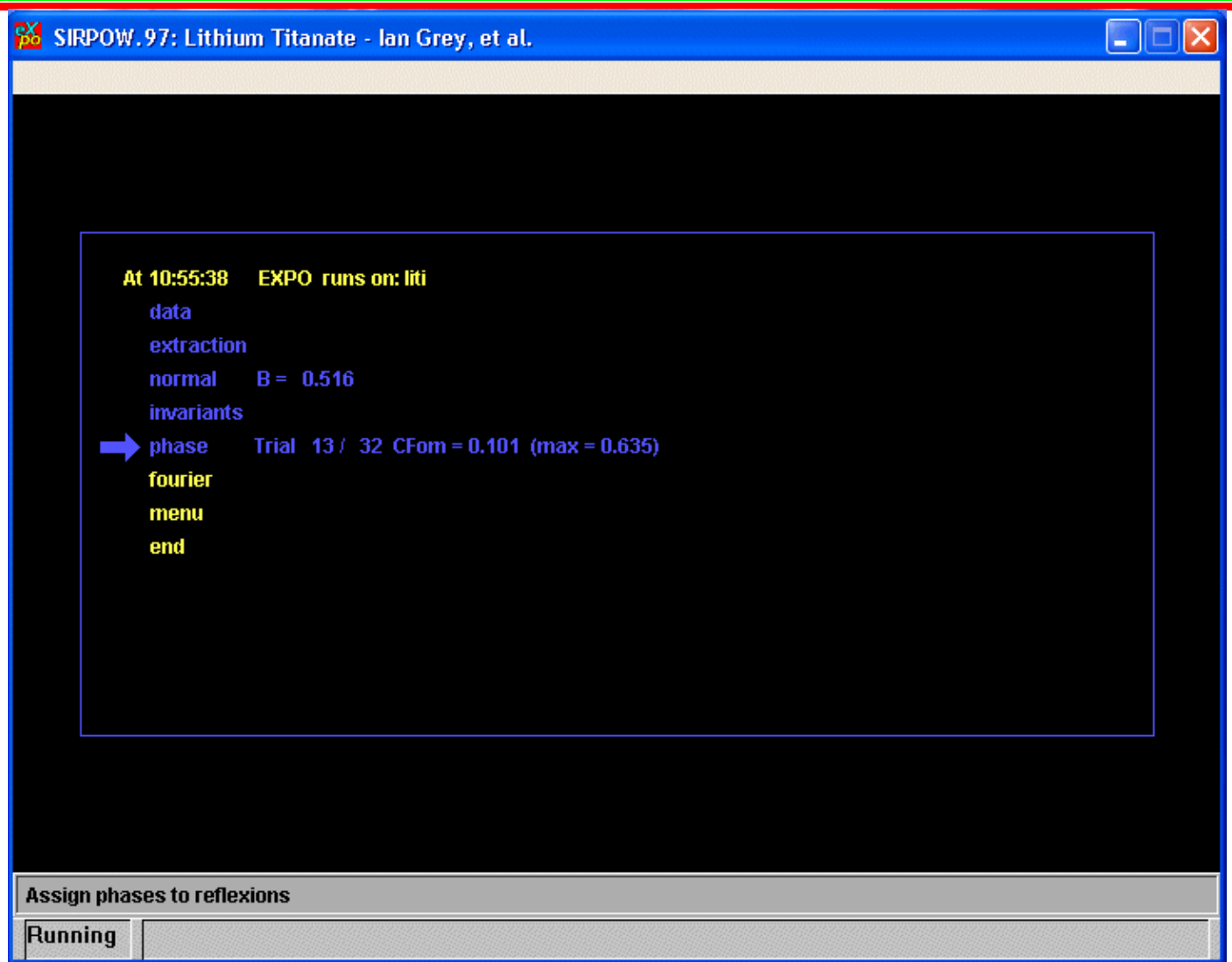
$Li_{0.98}Ti_{2.88}O_6$



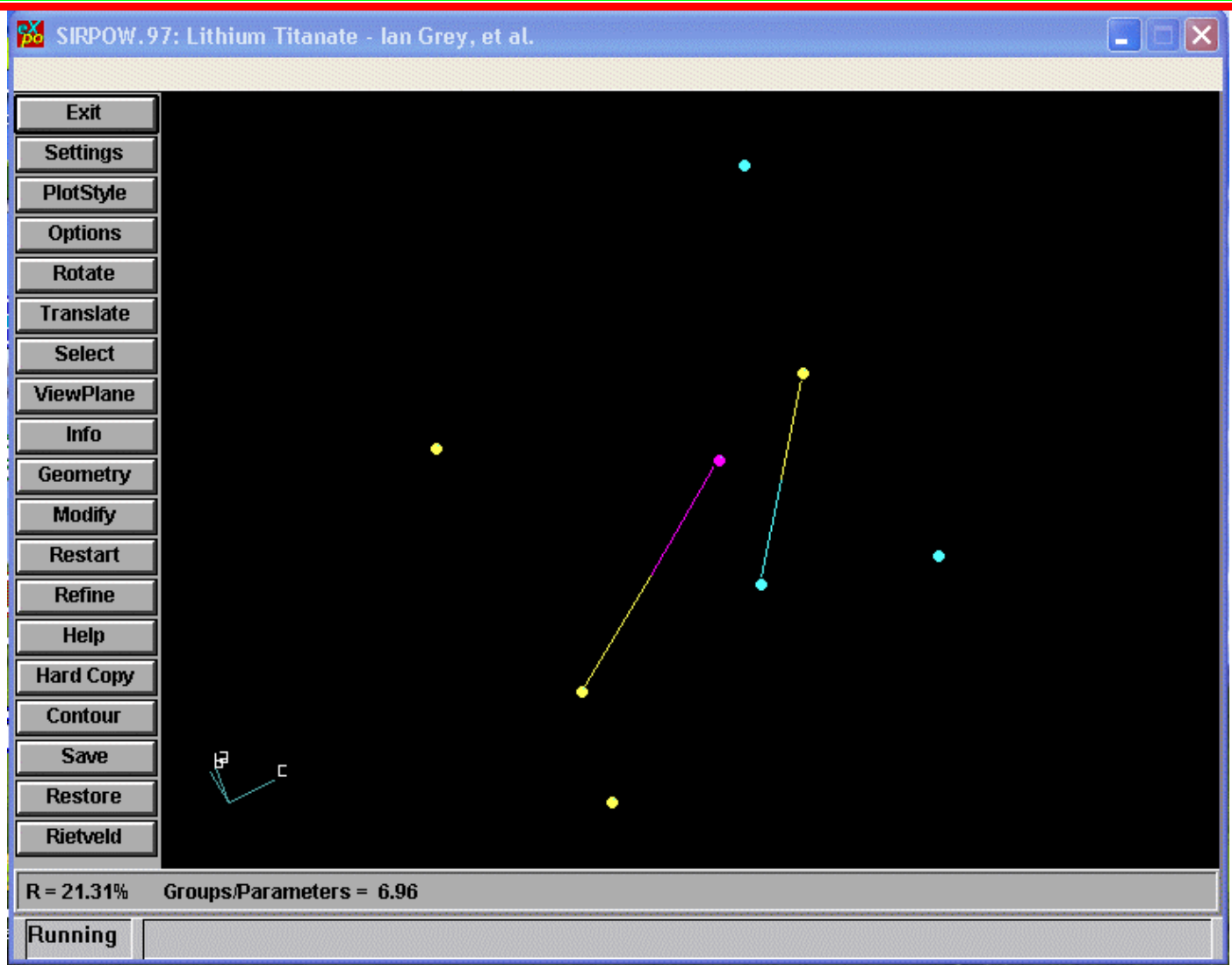
$Li_{0.98}Ti_{2.88}O_6$



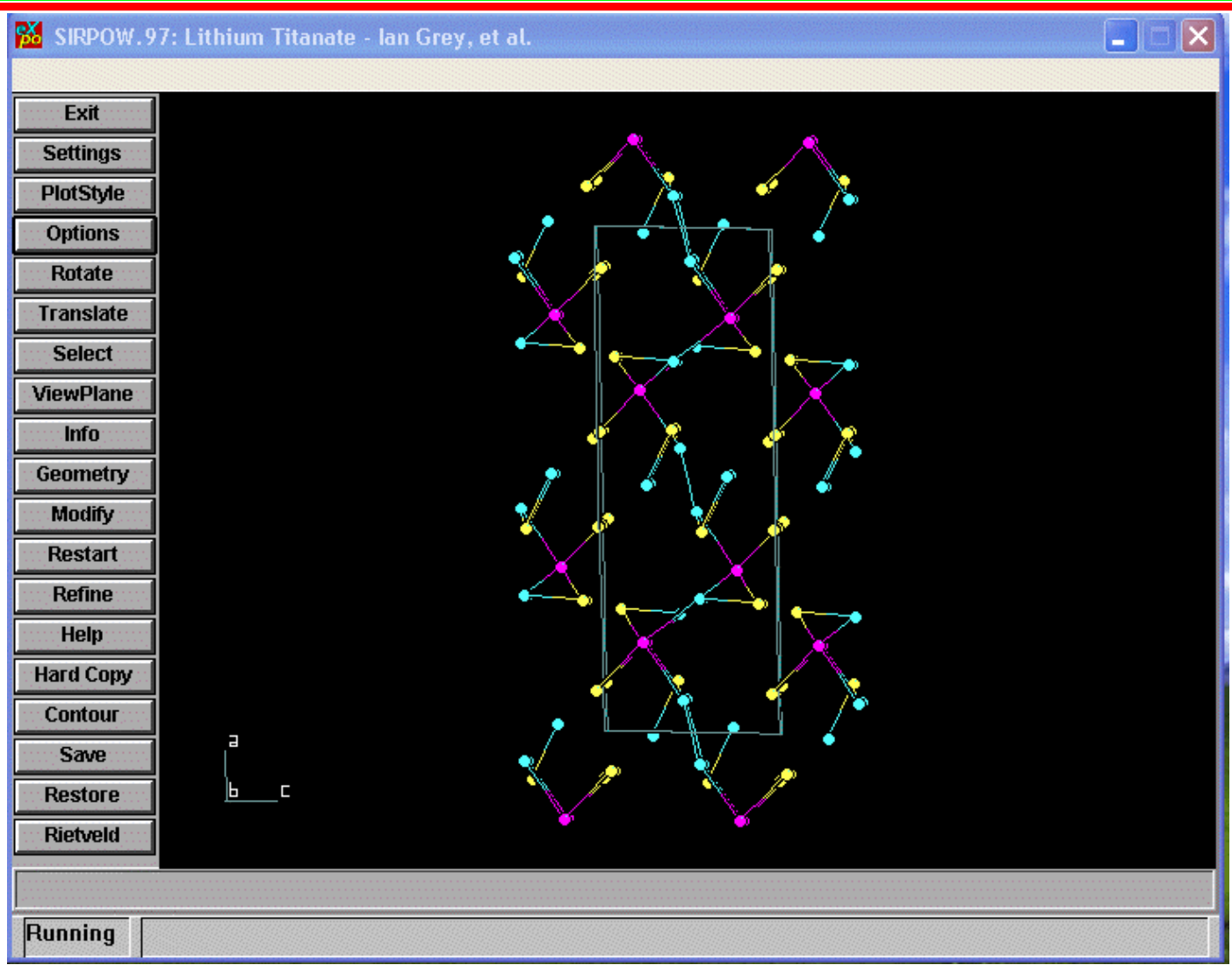
$Li_{0.98}Ti_{2.88}O_6$



$Li_{0.98}Ti_{2.88}O_6$



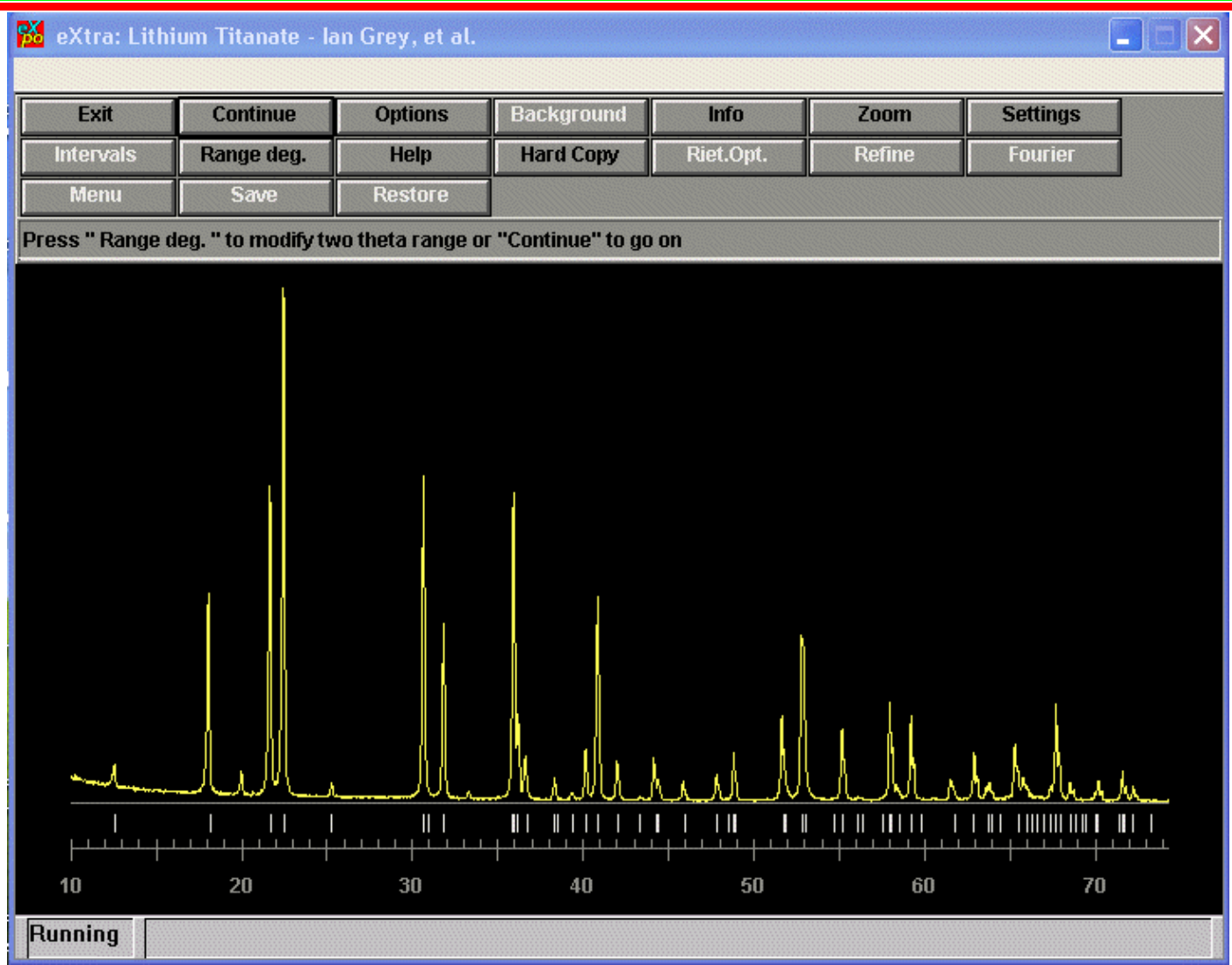
$Li_{0.98}Ti_{2.88}O_6$



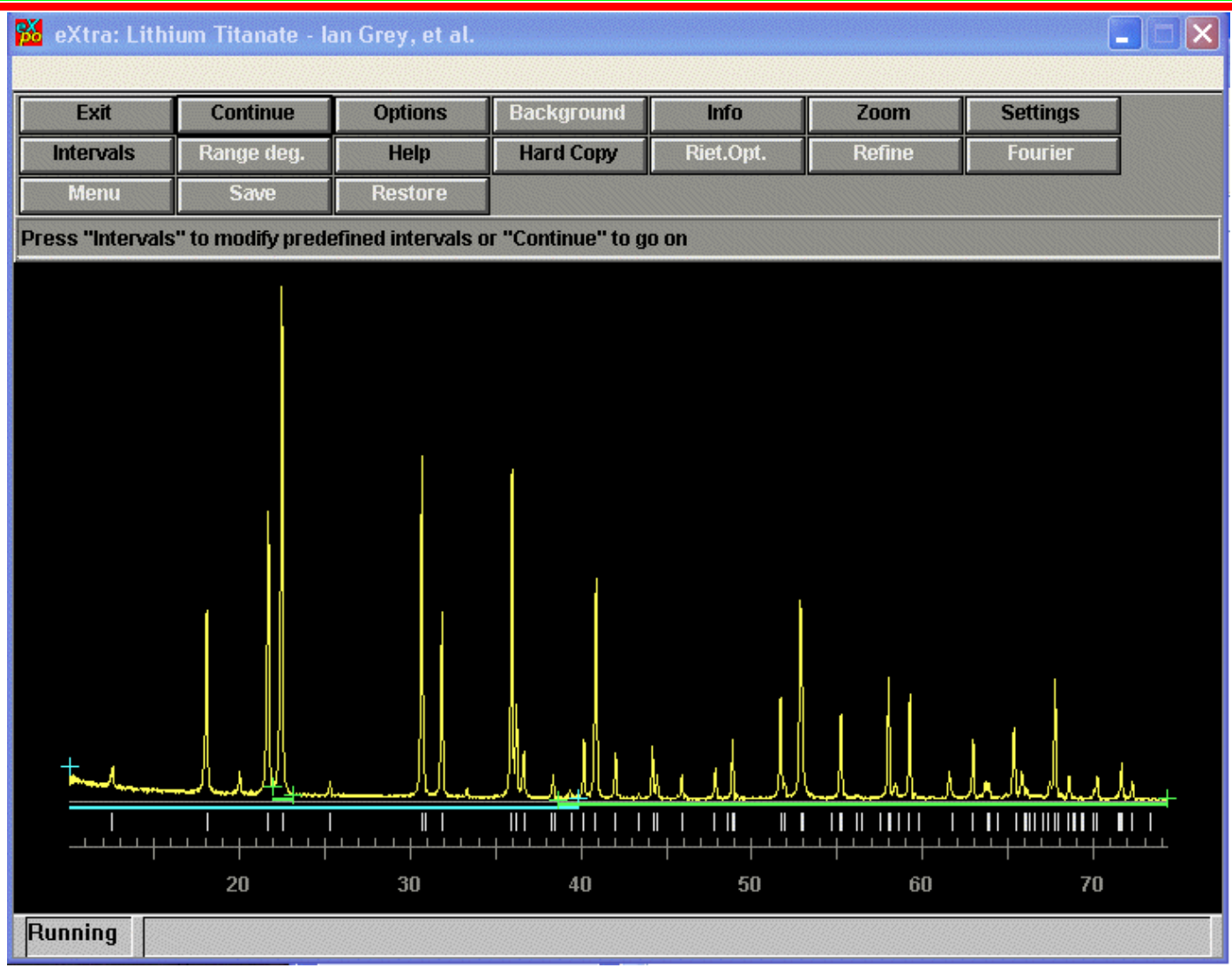
$Li_{0.98}Ti_{2.88}O_6$ - less data: .exp file

```
%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
```

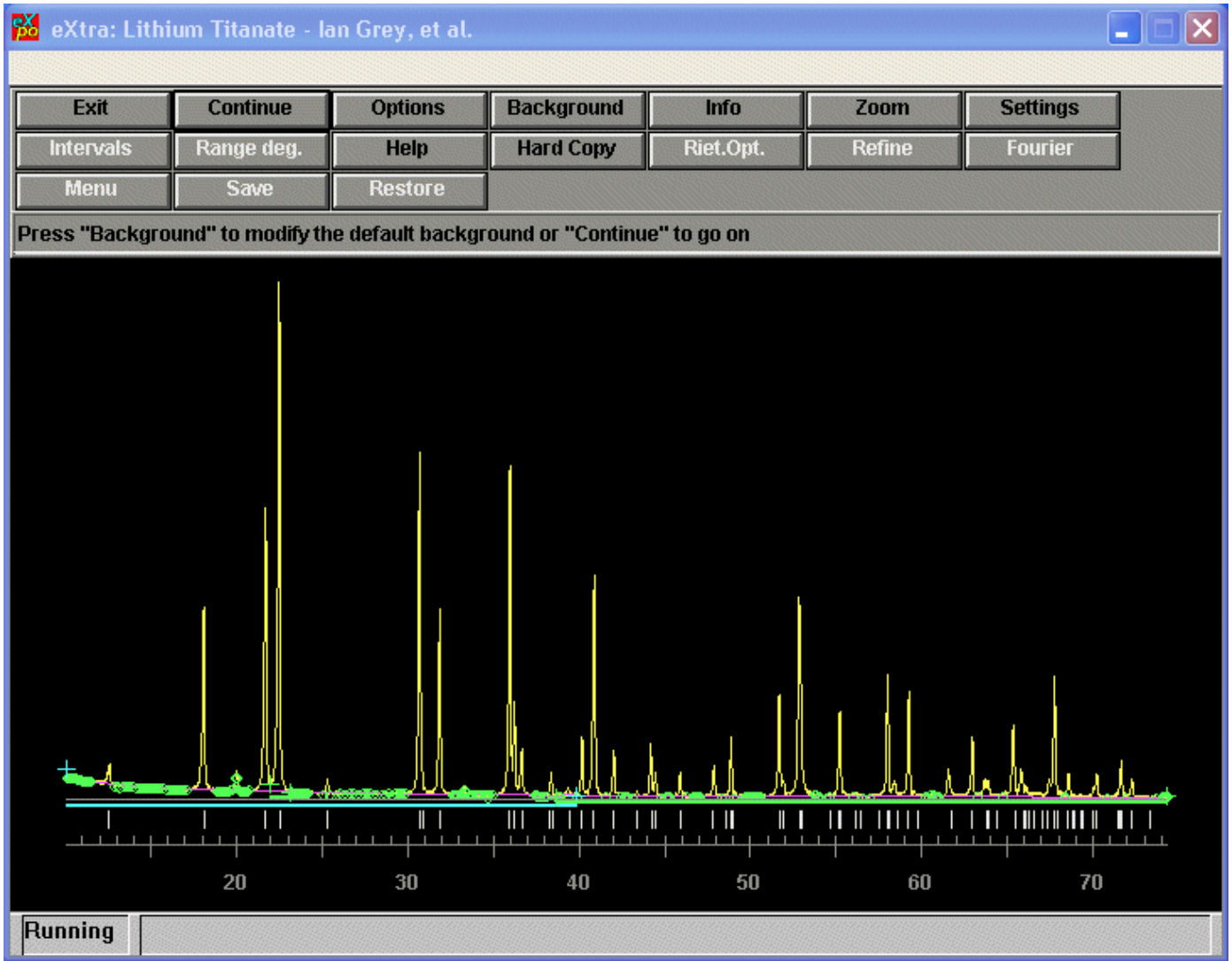
$Li_{0.98}Ti_{2.88}O_6$ - less data



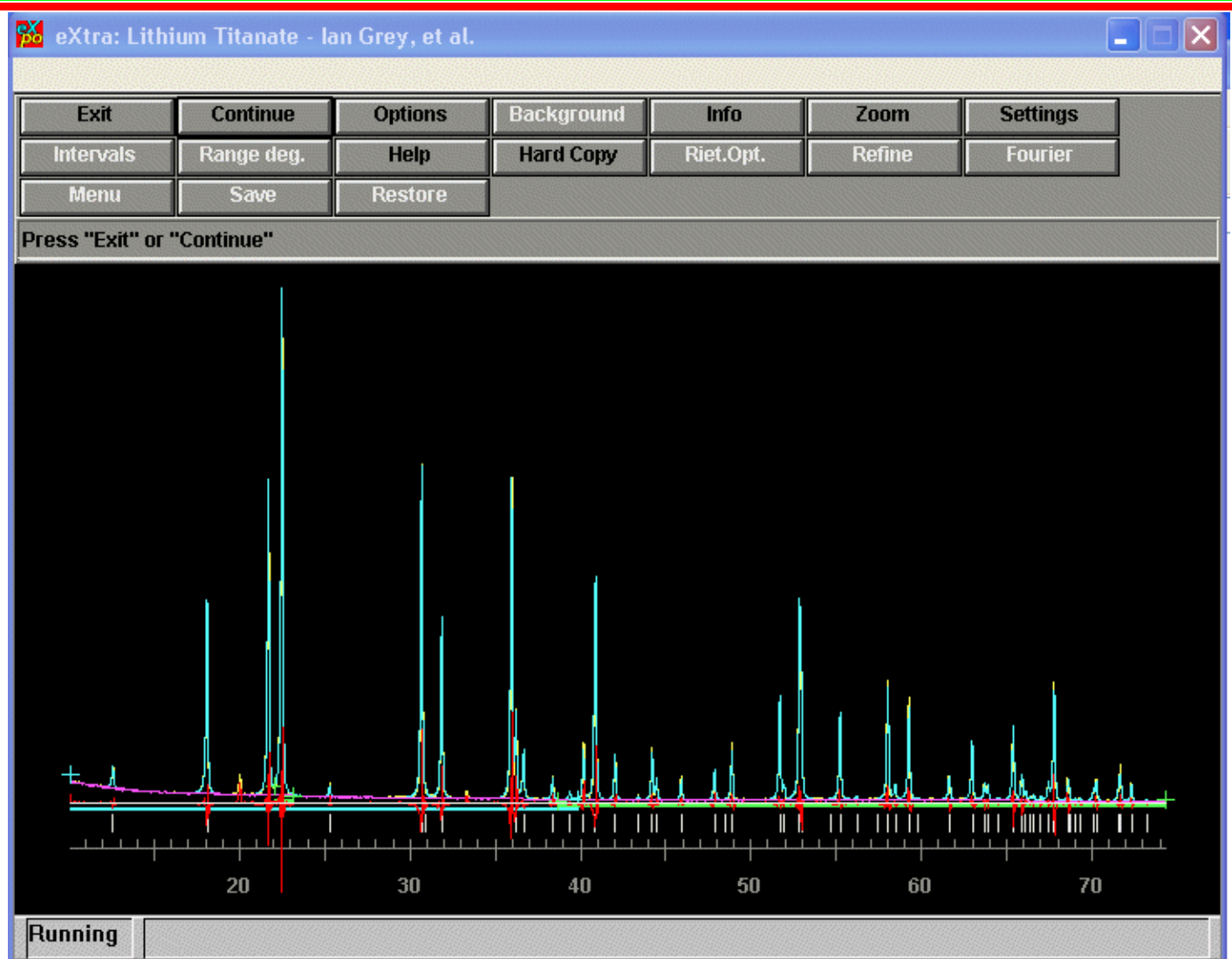
$Li_{0.98}Ti_{2.88}O_6$ - less data



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

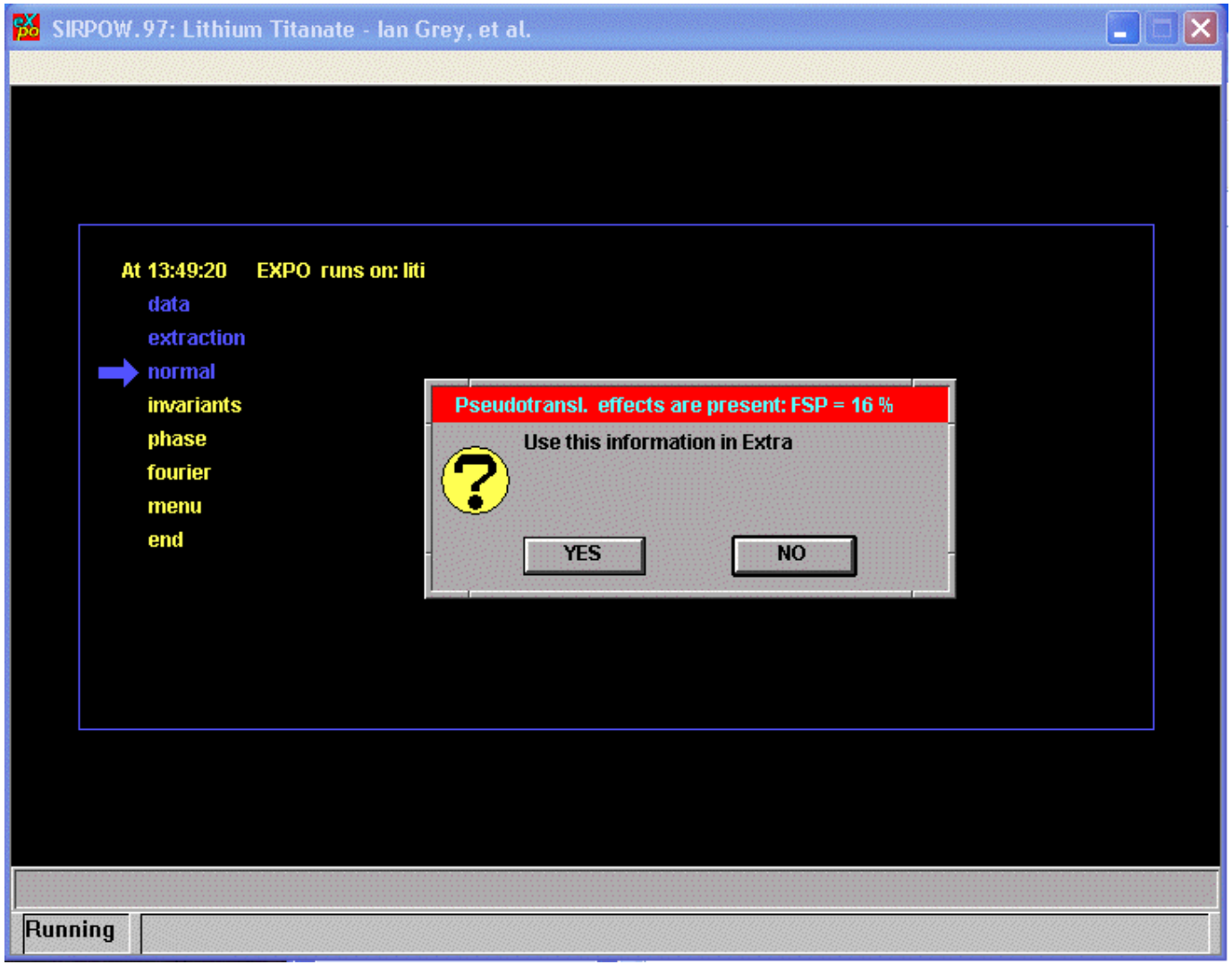


$Li_{0.98}Ti_{2.88}O_6$ - less data

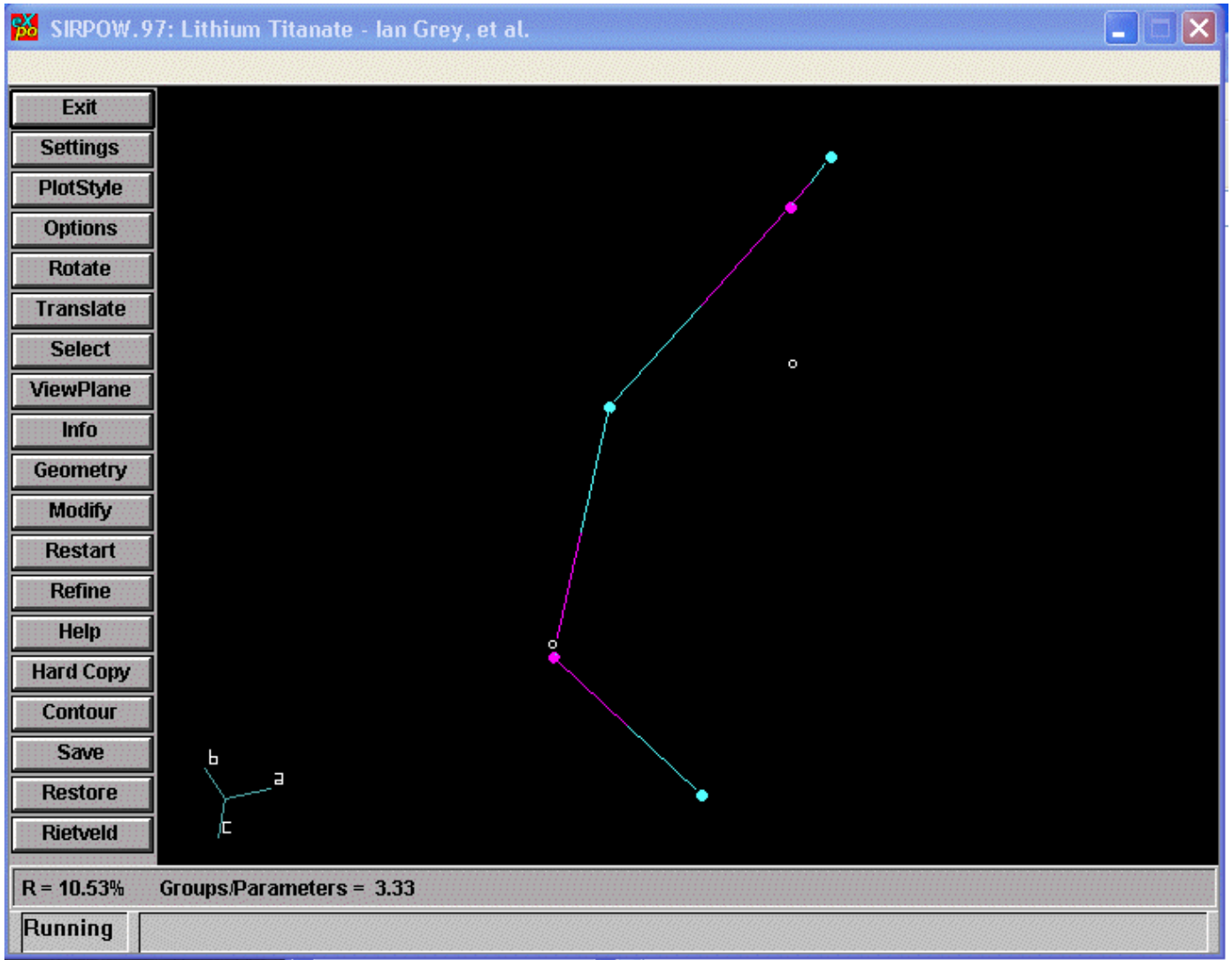


$Li_{0.98}Ti_{2.88}O_6$ - less data

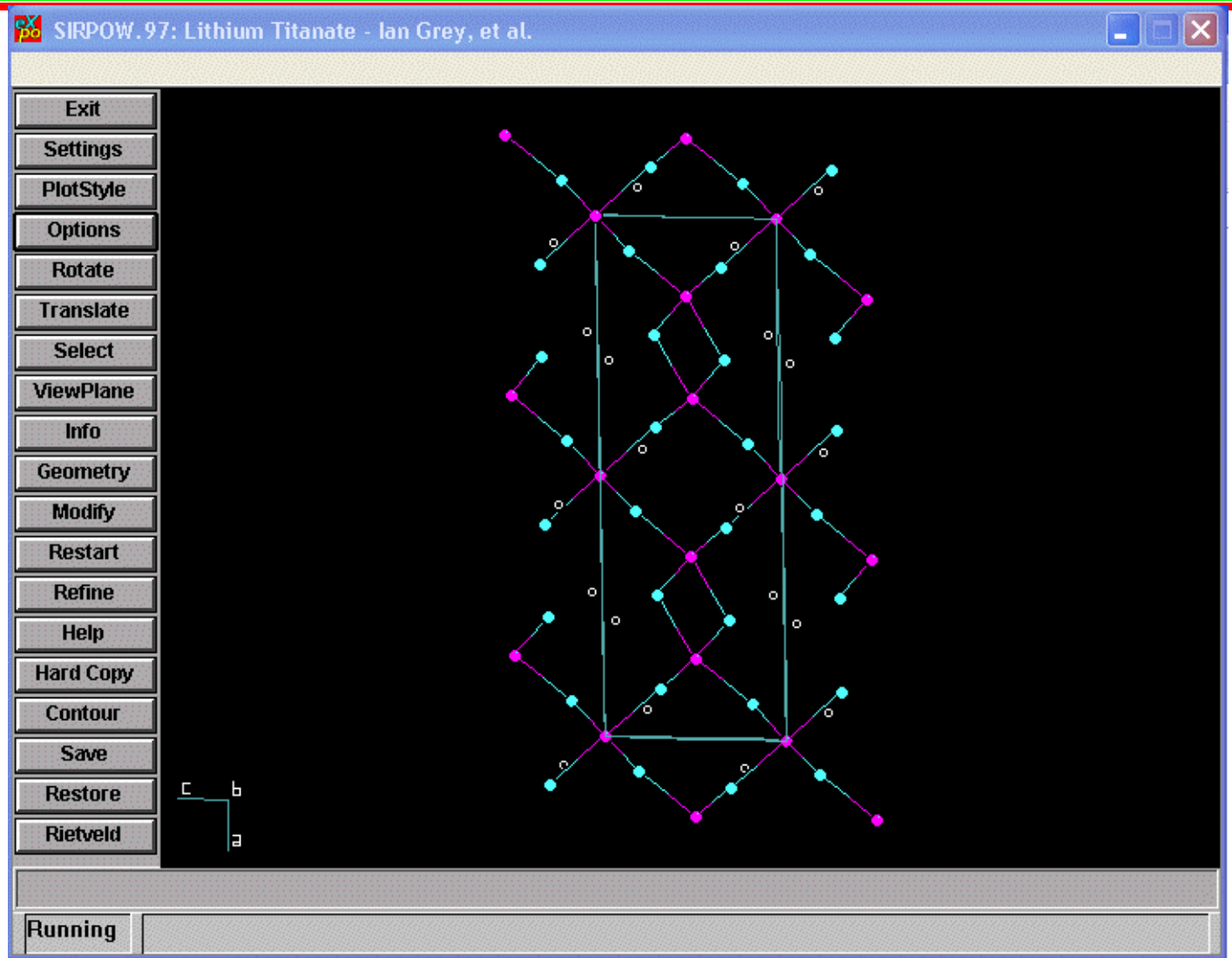
$Li_{0.98}Ti_{2.88}O_6$ - less data



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



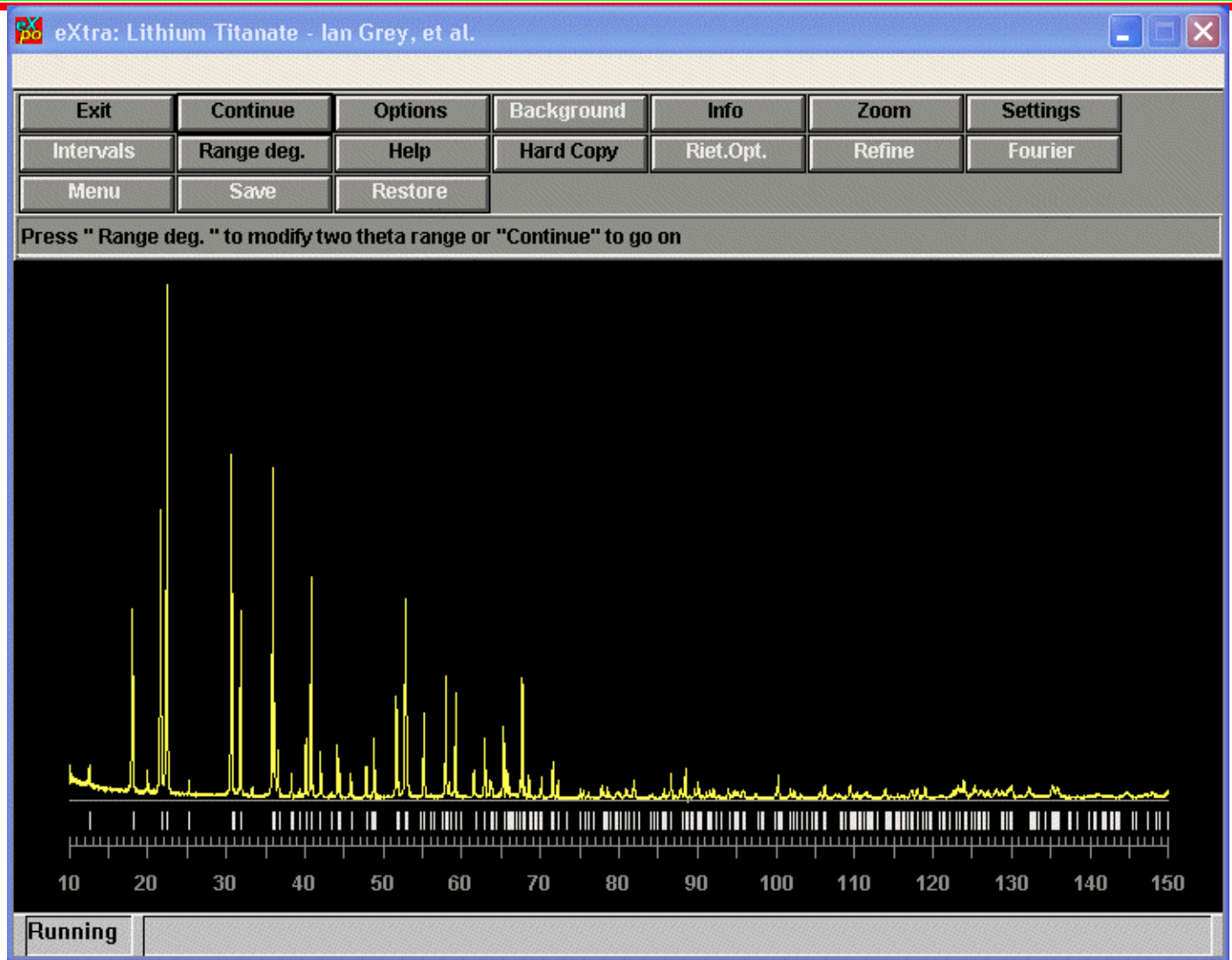
$Li_{0.98}Ti_{2.88}O_6$ - less data



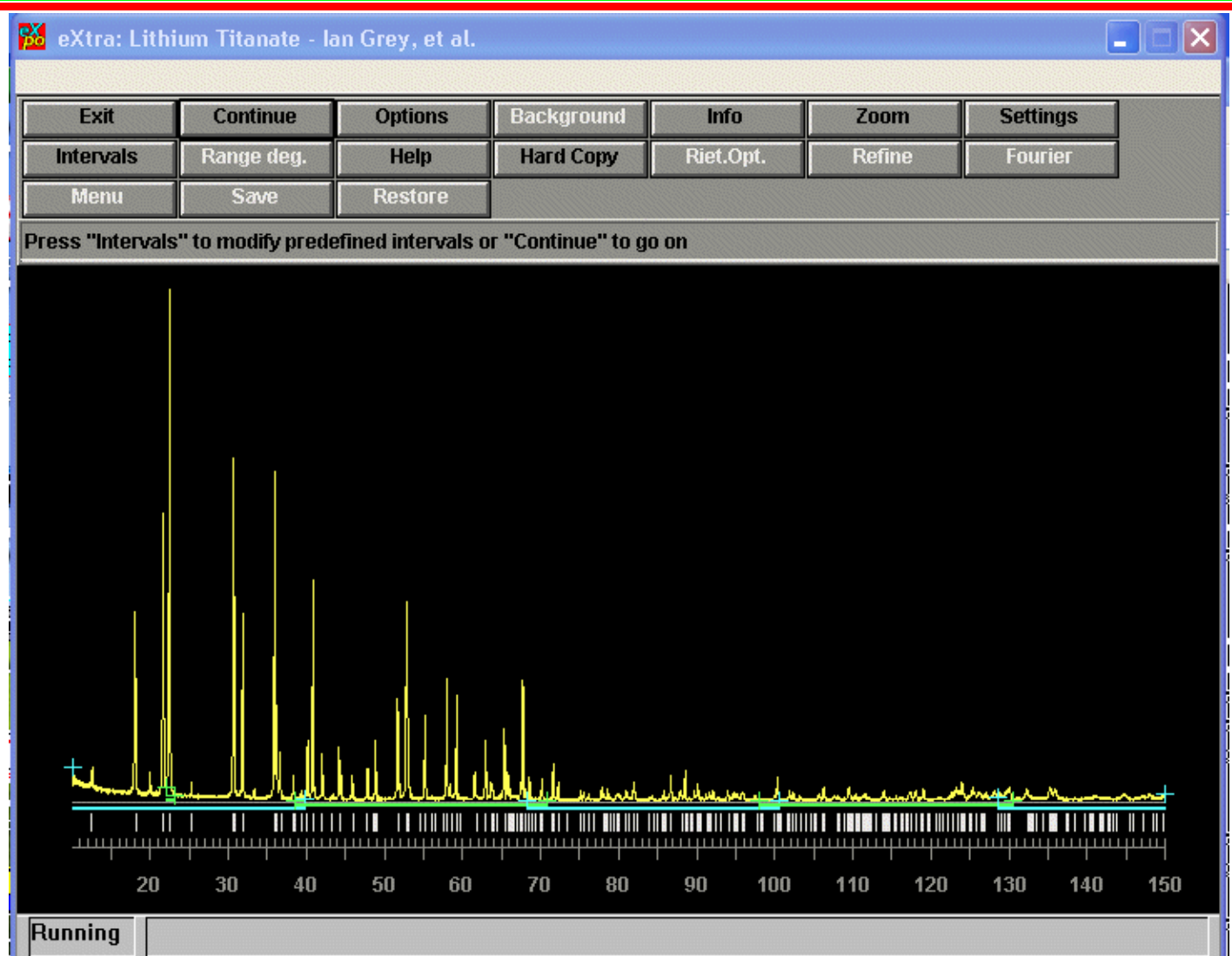
$Li_{0.98}Ti_{2.88}O_6$ – *decrease B factor:* .exp file

```
%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
```

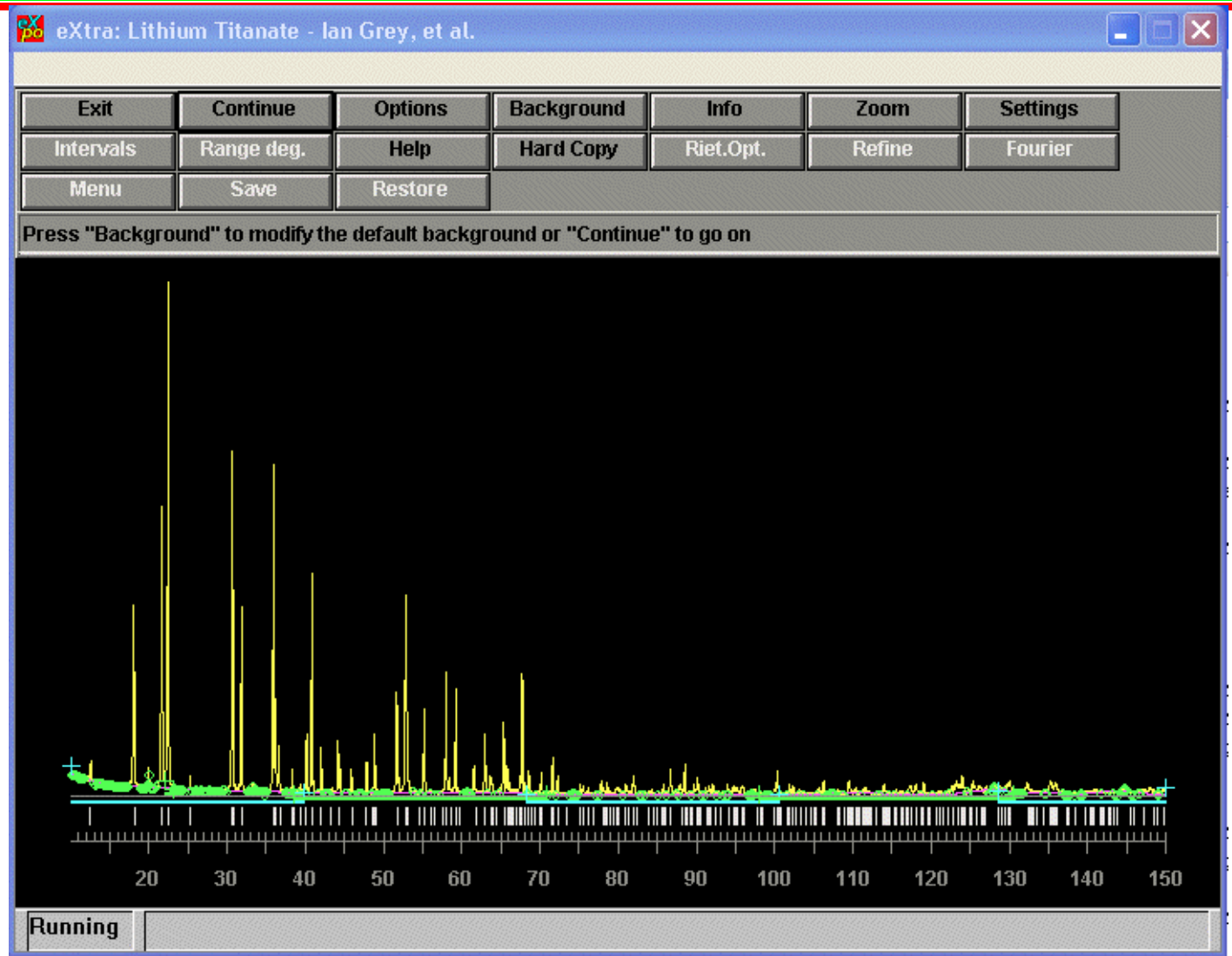
$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



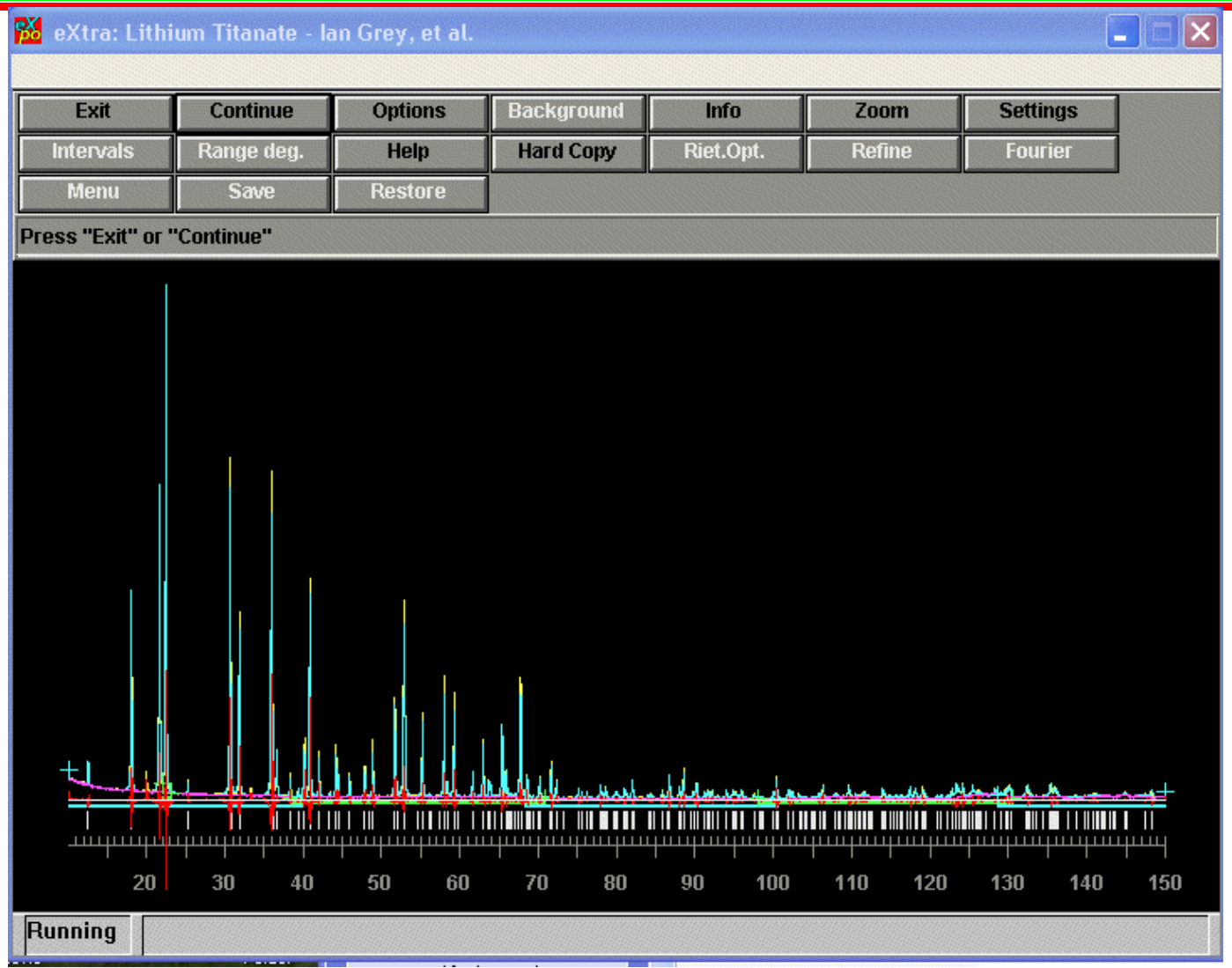
$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



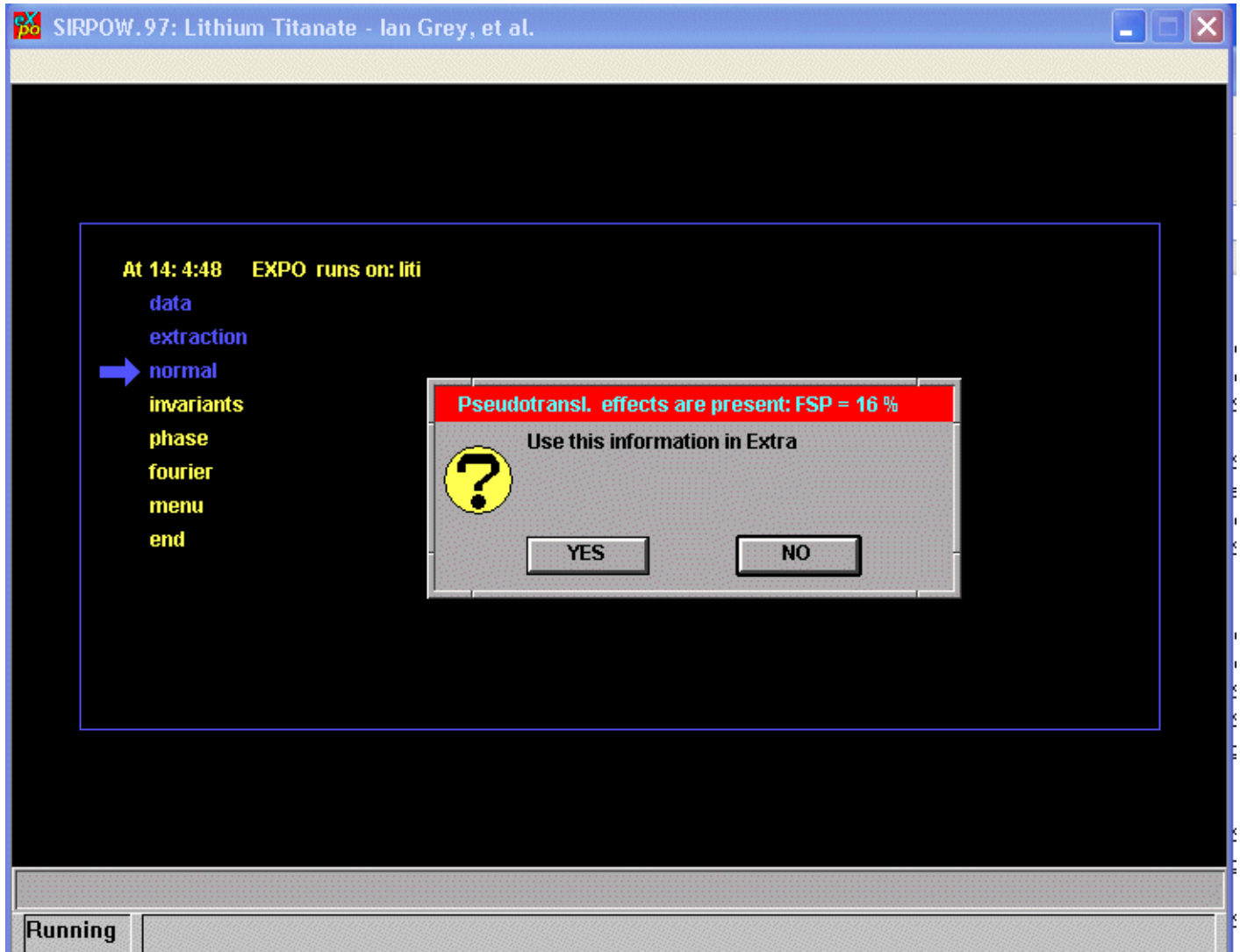
$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



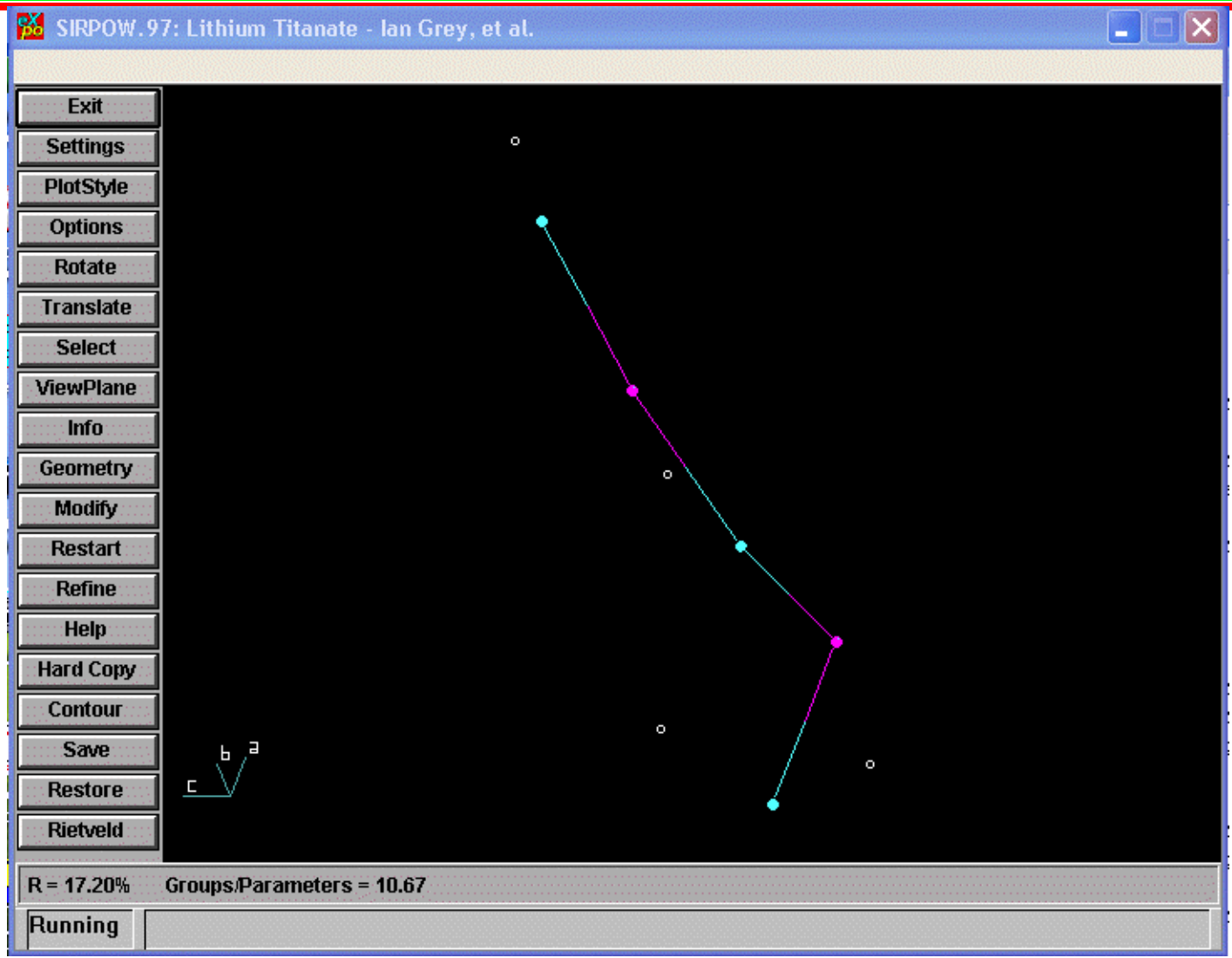
$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



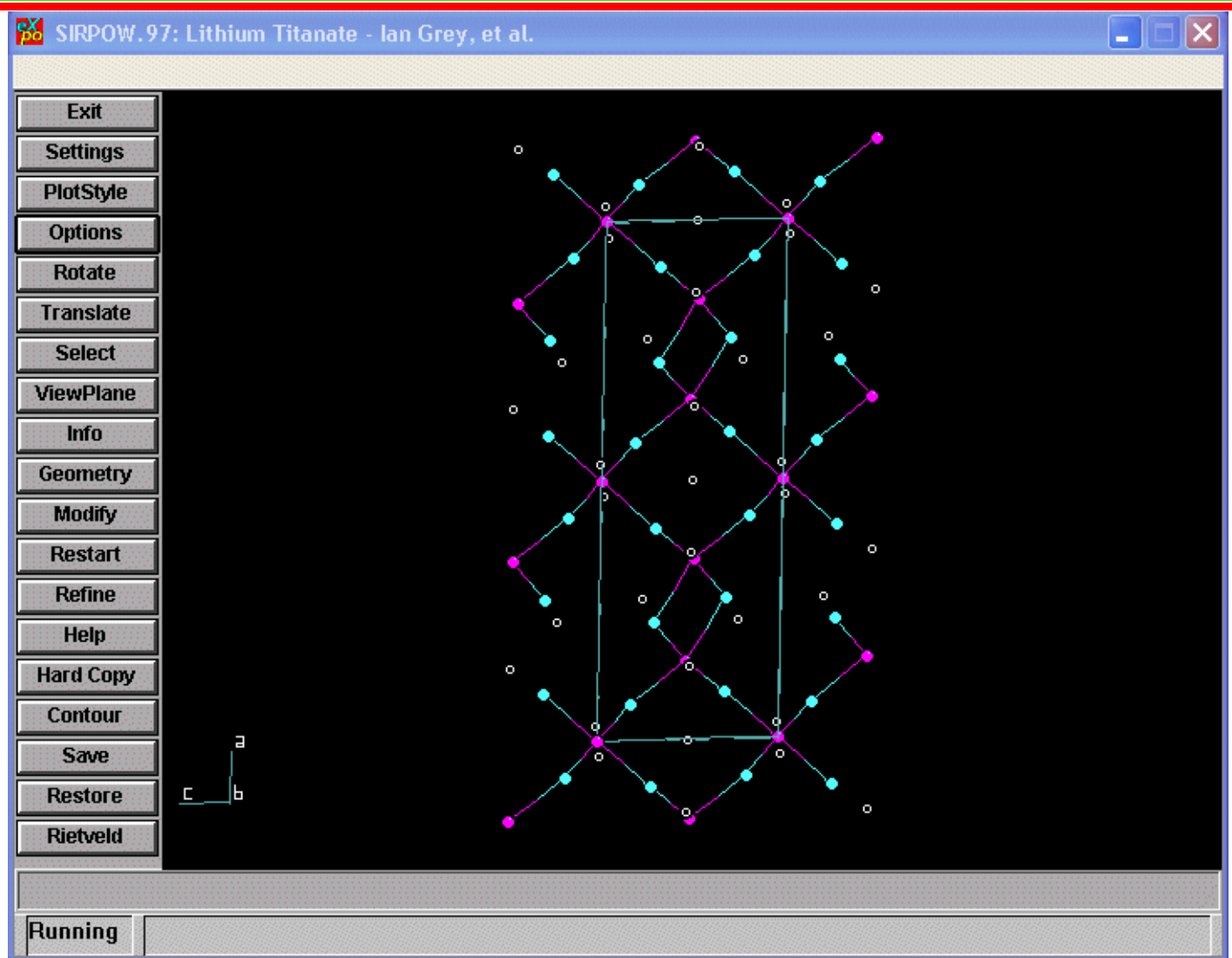
$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



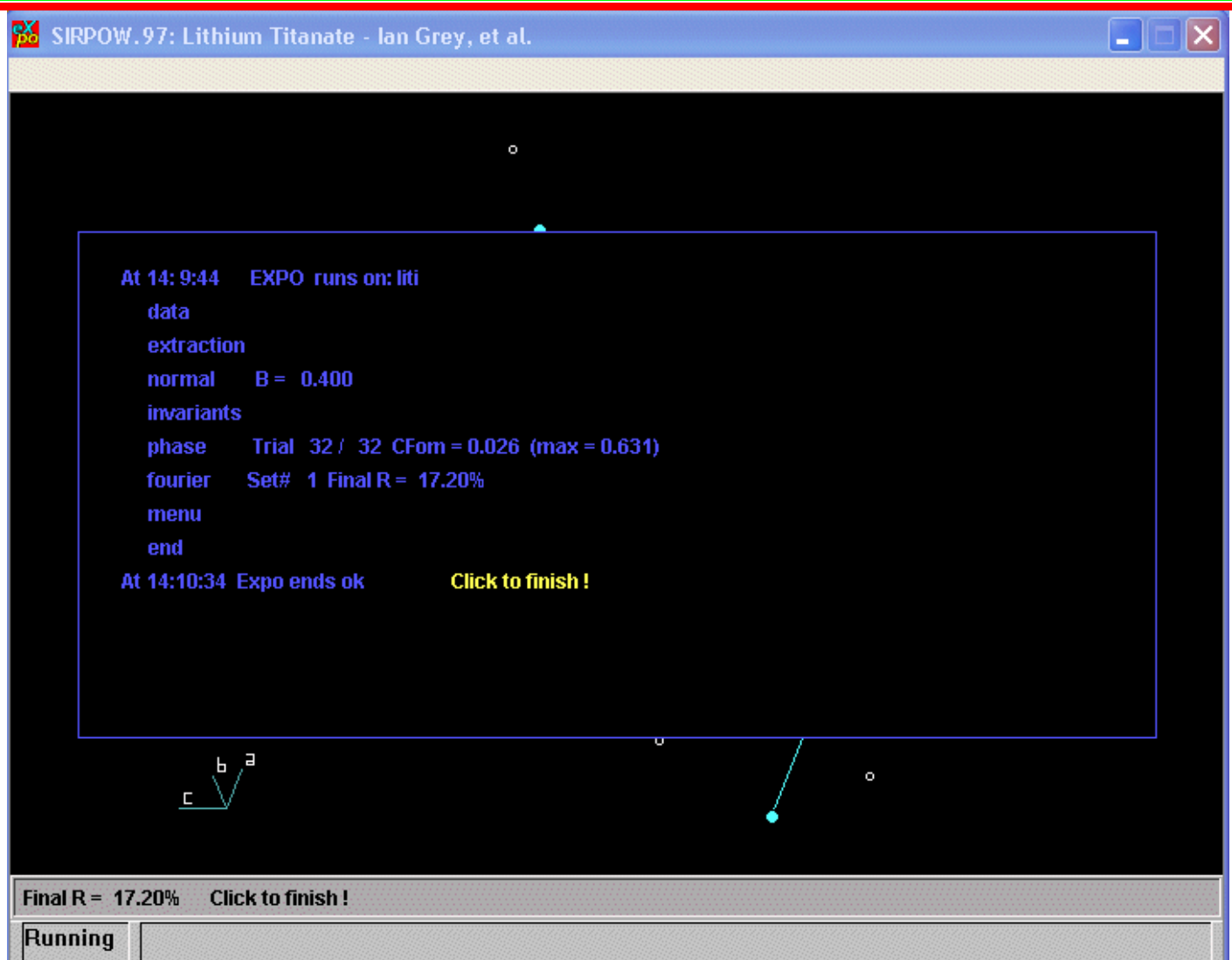
$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



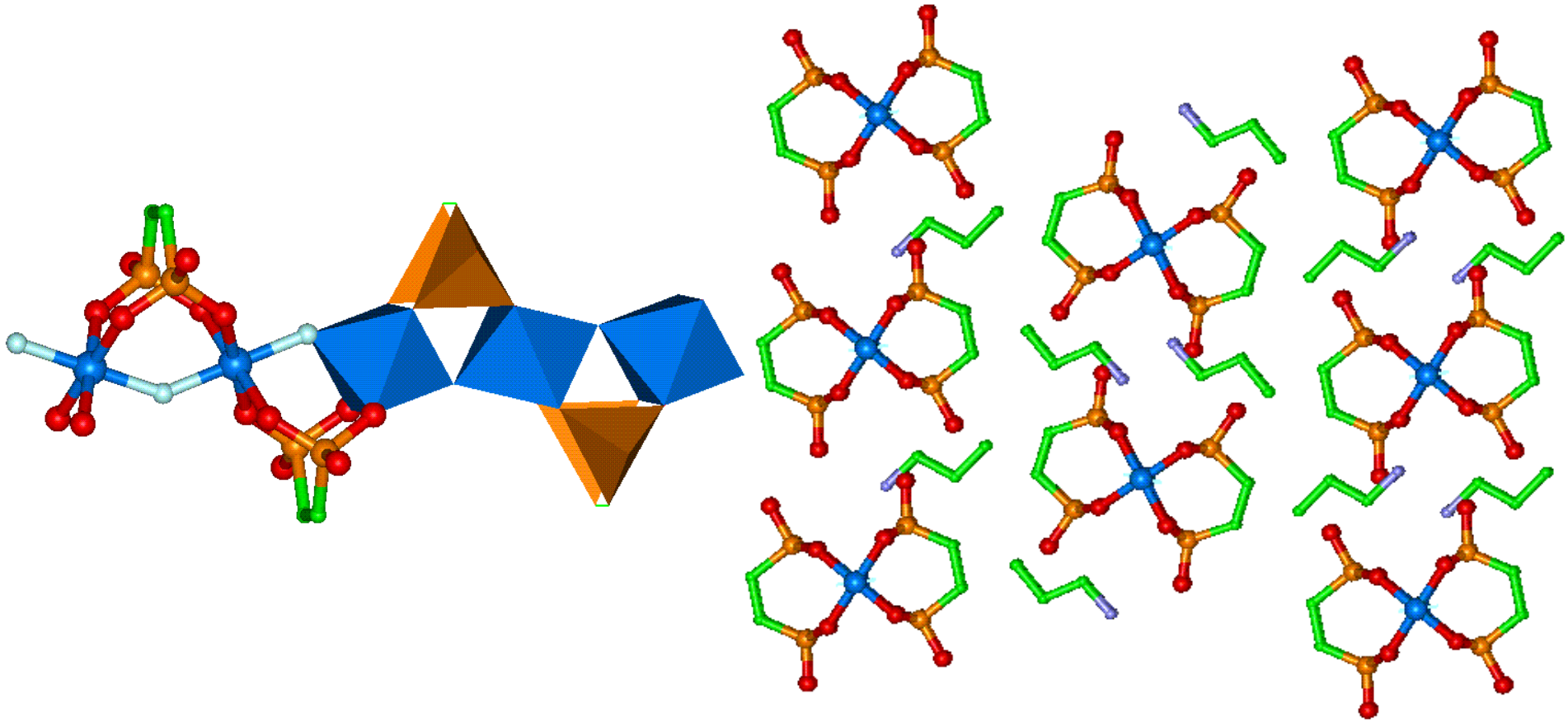
$Li_{0.98}Ti_{2.88}O_6$ – decrease B factor



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



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

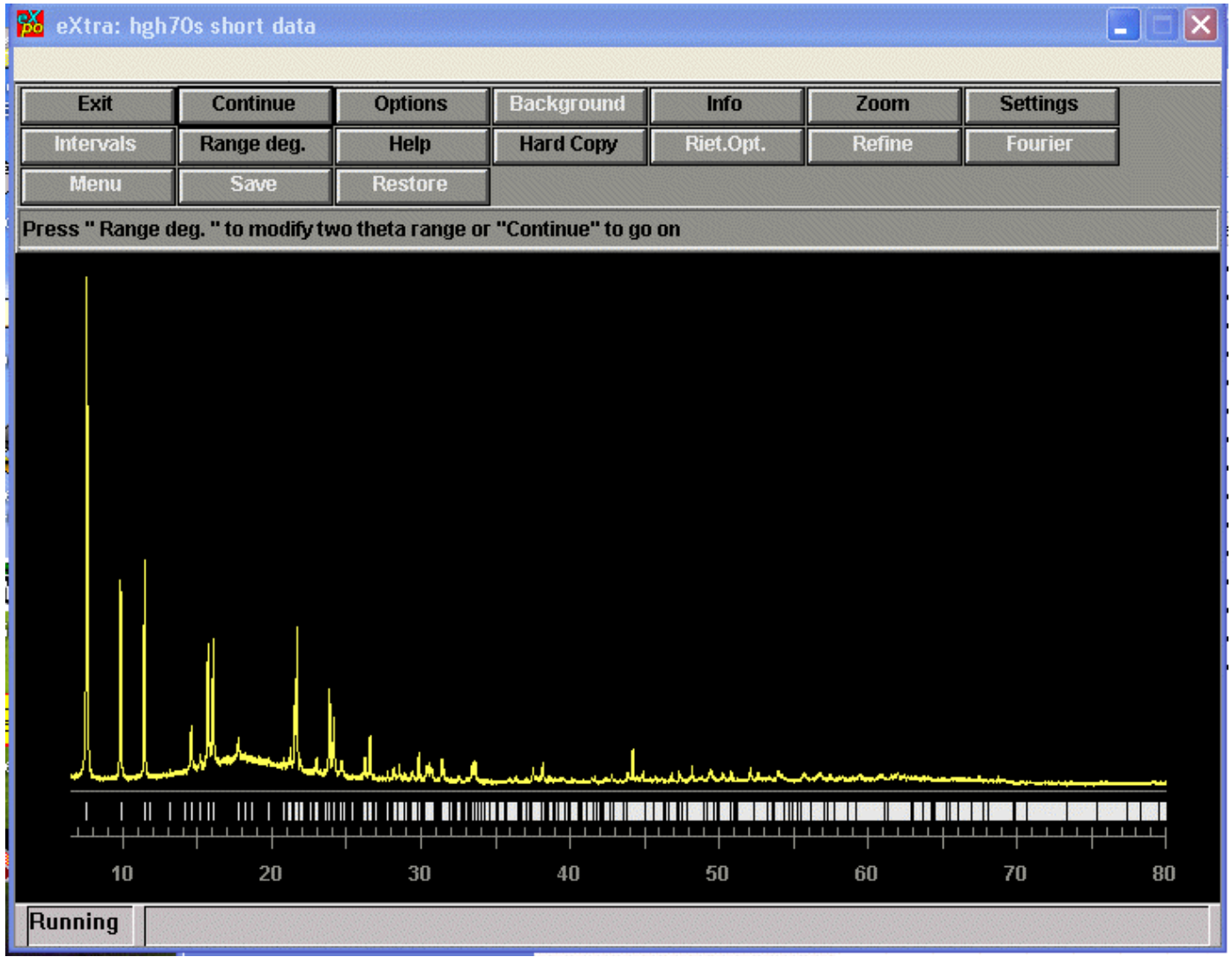


H. G. Harvey, S. J. Teat, C. C. Tang, L. M. Cranswick and M. P. Attfield, *Inorg. Chem.*, 2003, *42*, 2428.

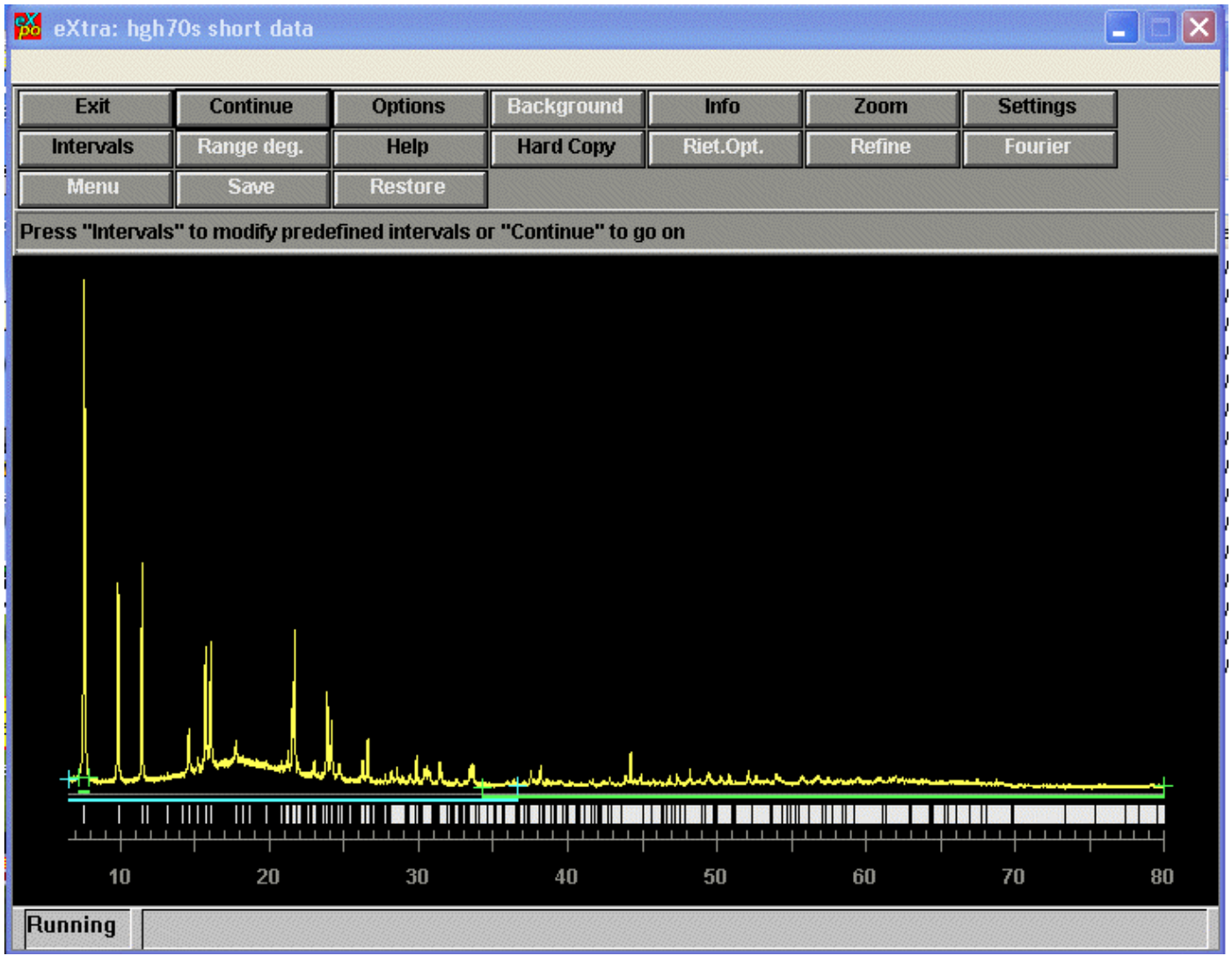
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: .exp file

```
%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
```

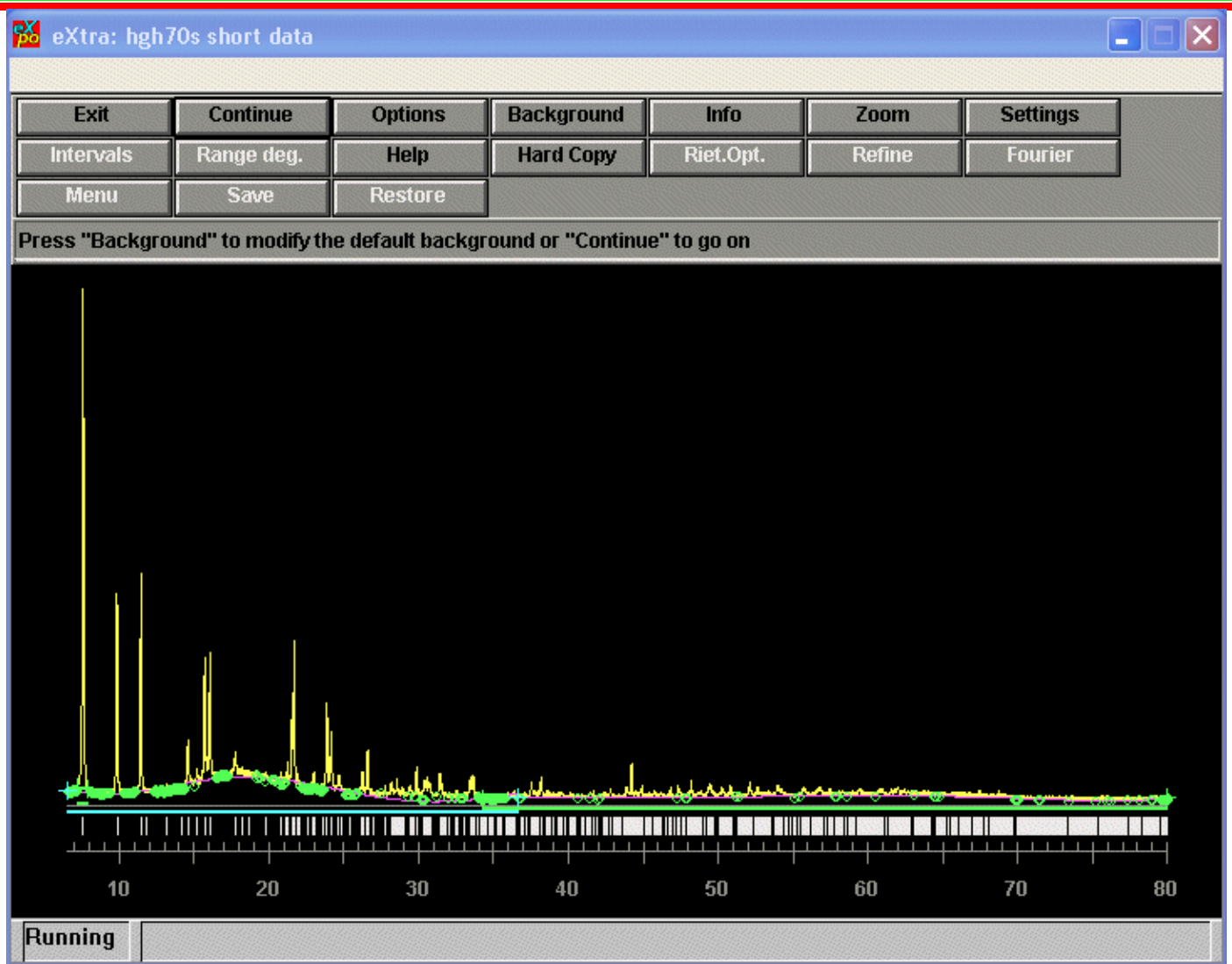
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$



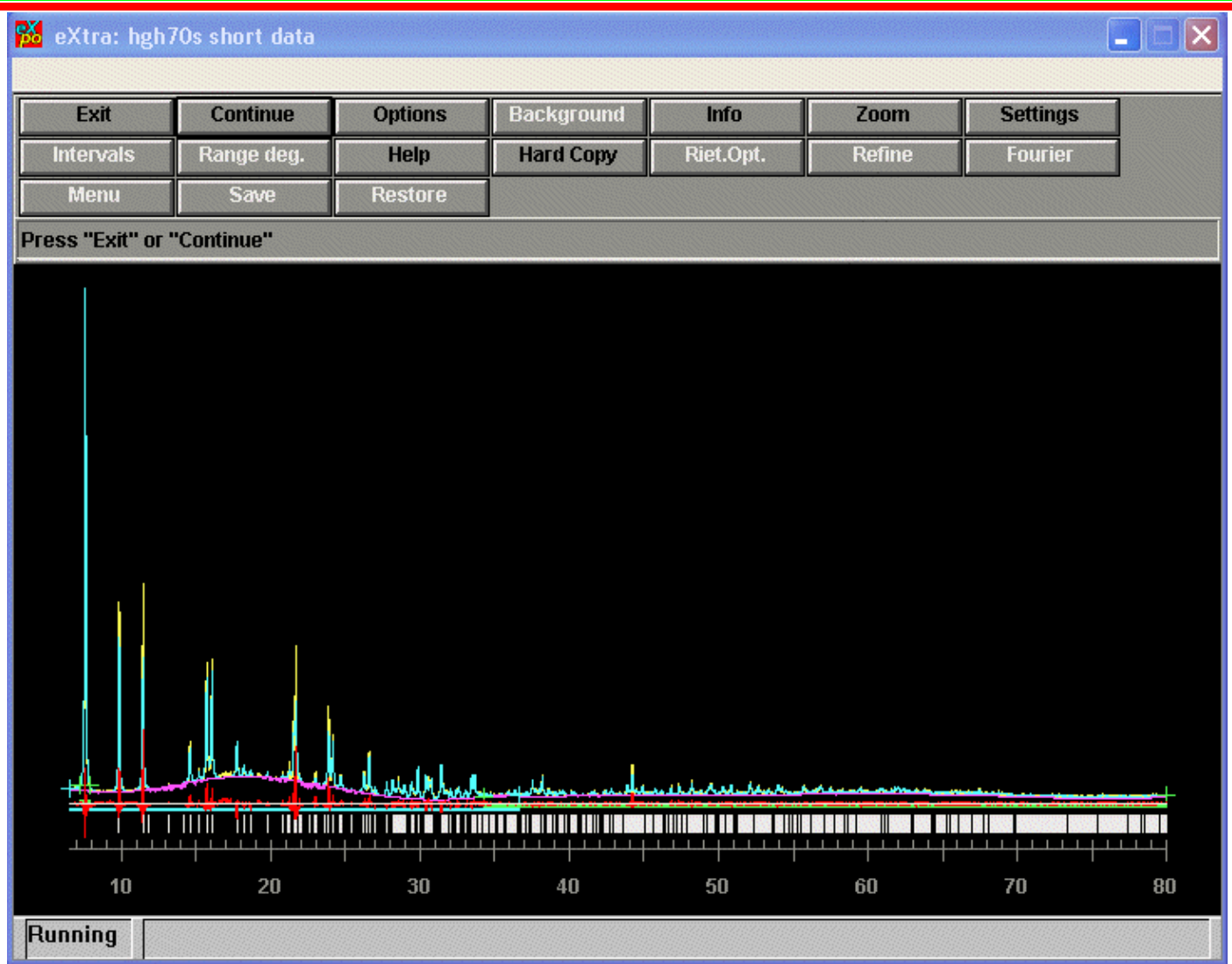
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$



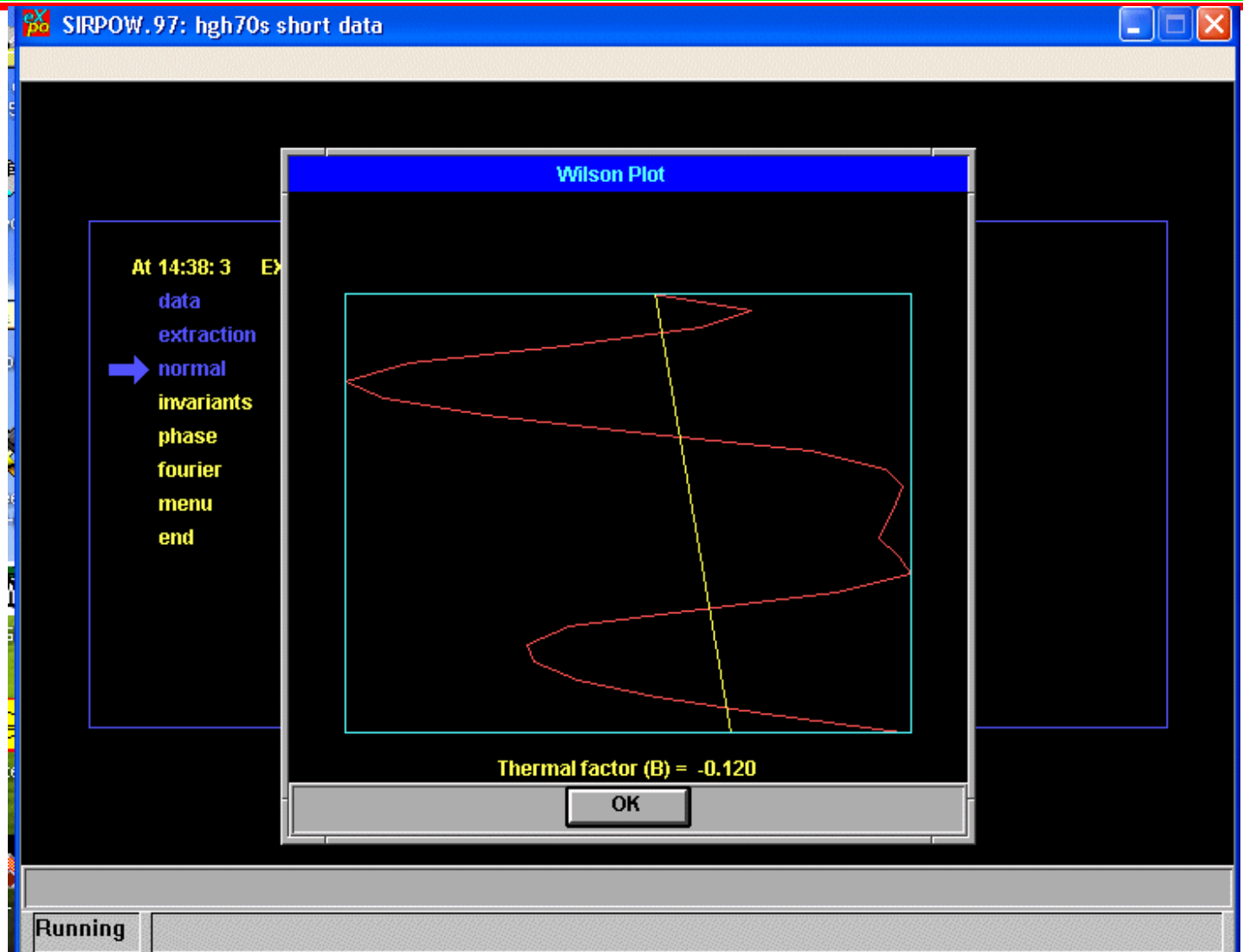
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$



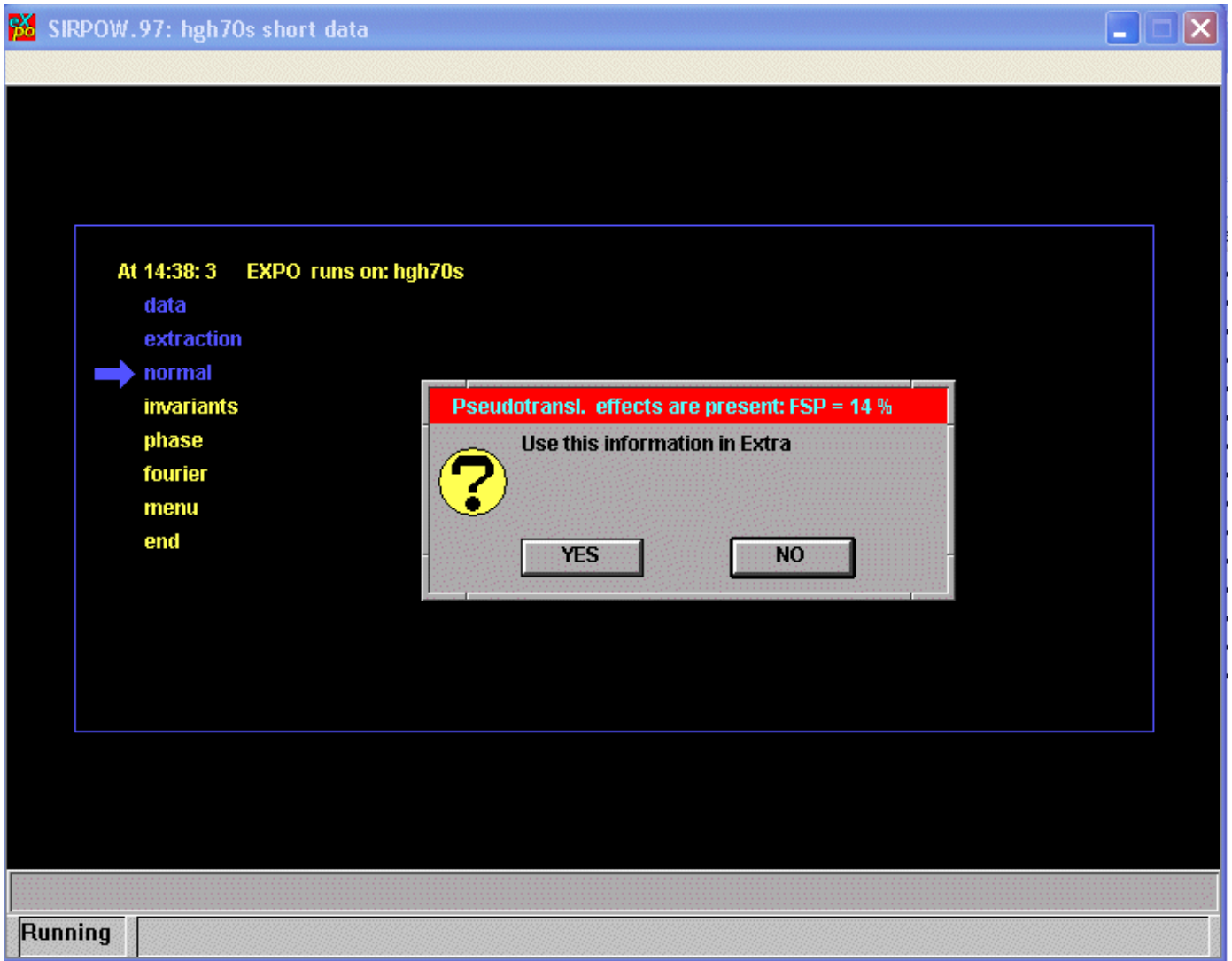
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$



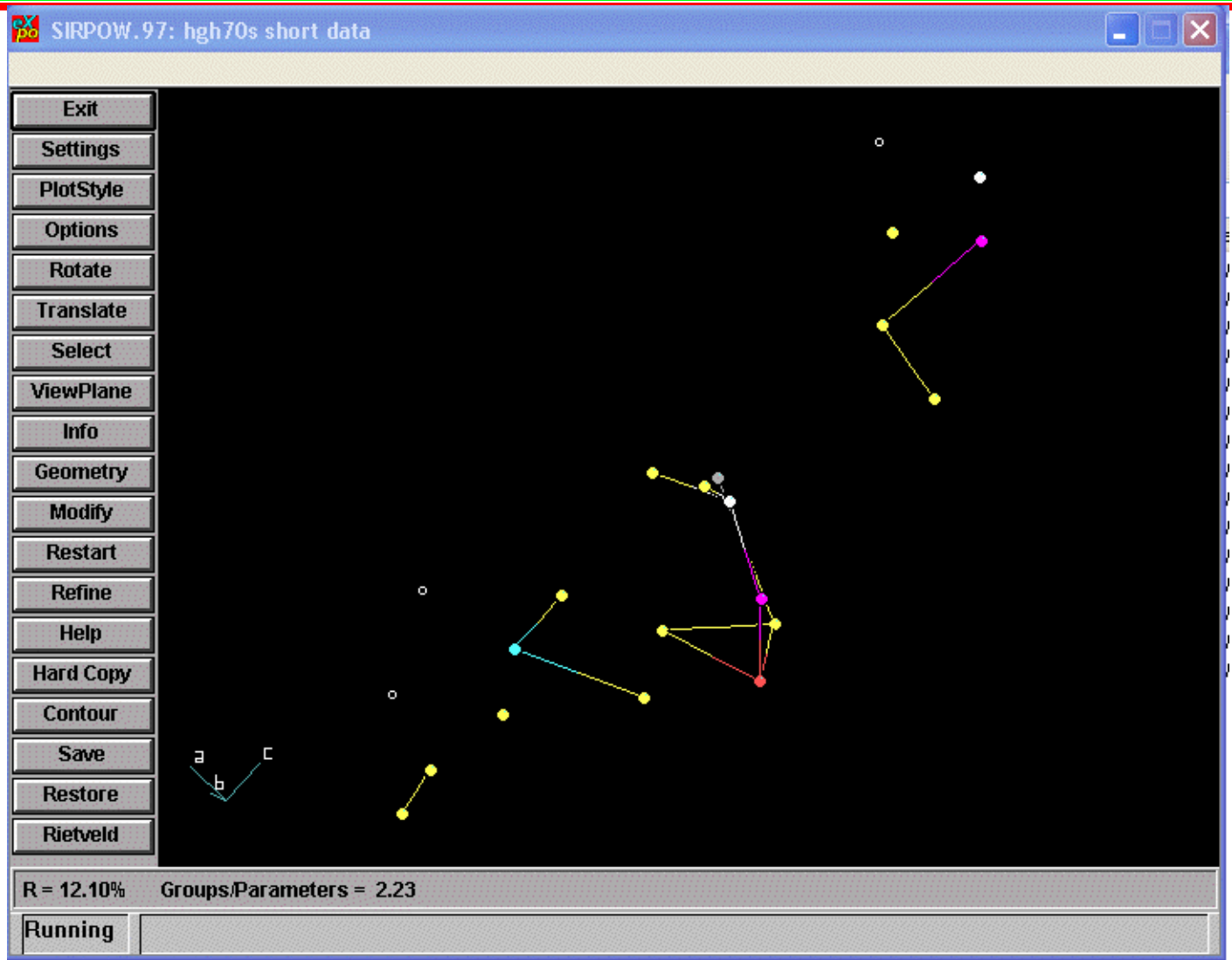
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$



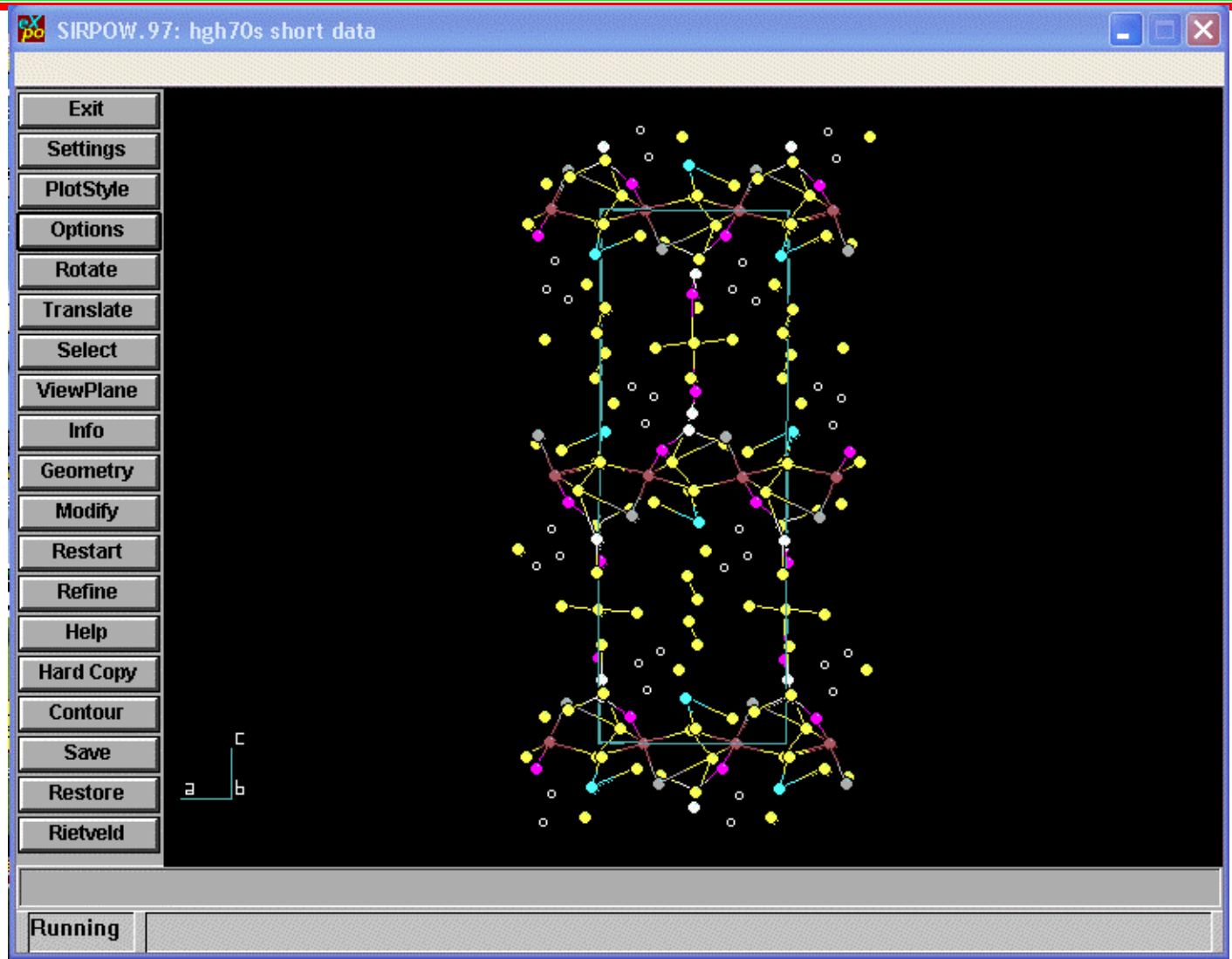
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$



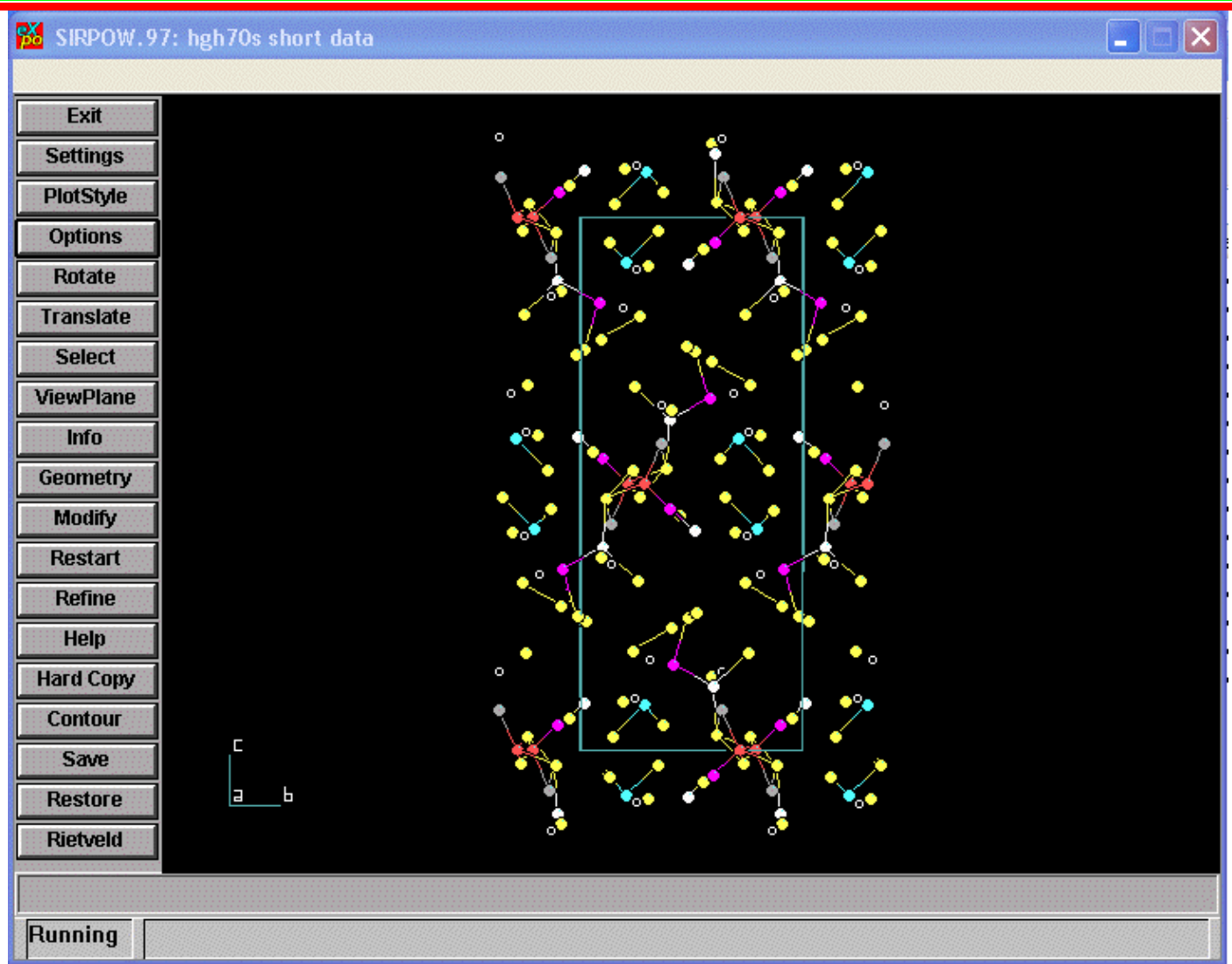
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$



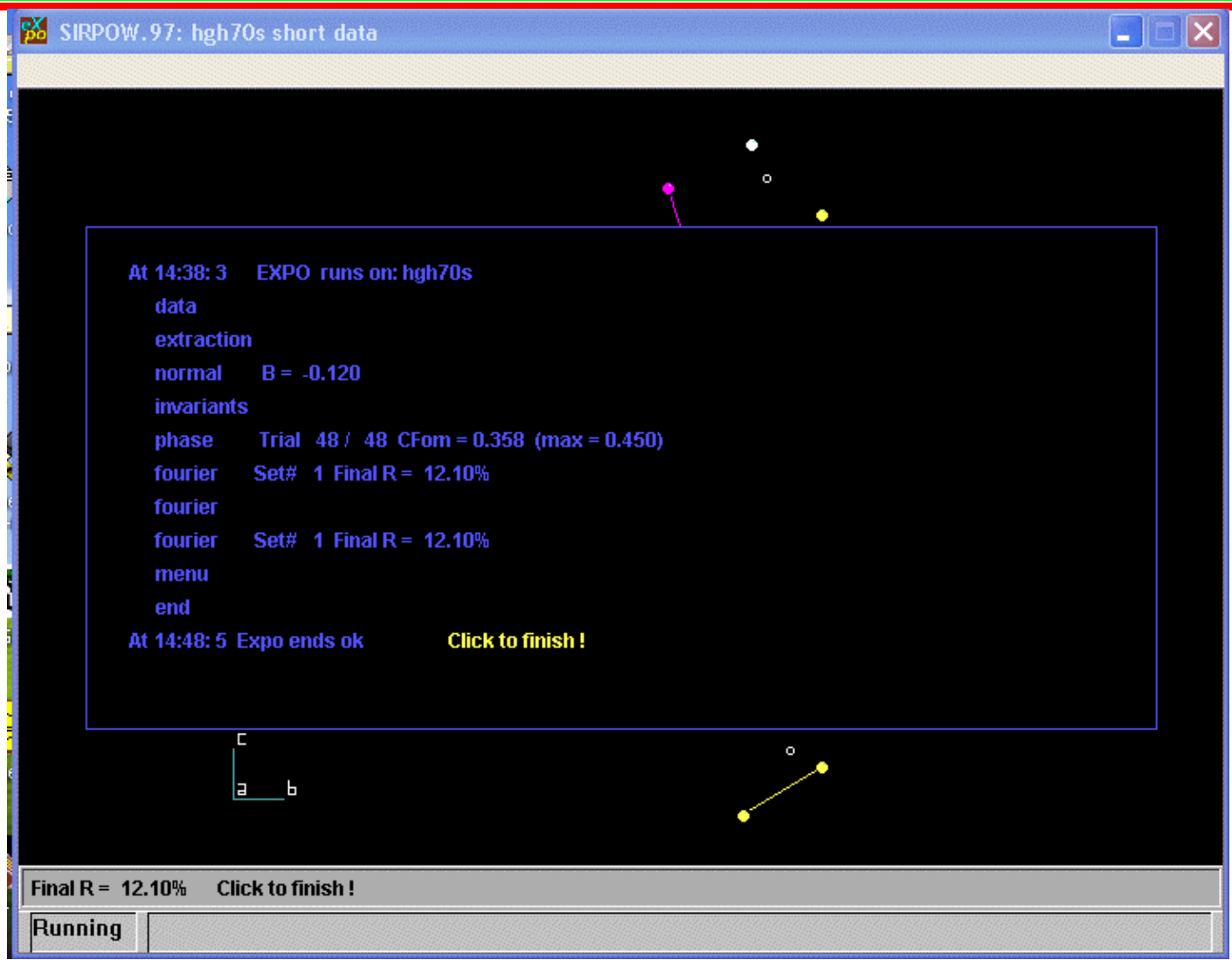
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$



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



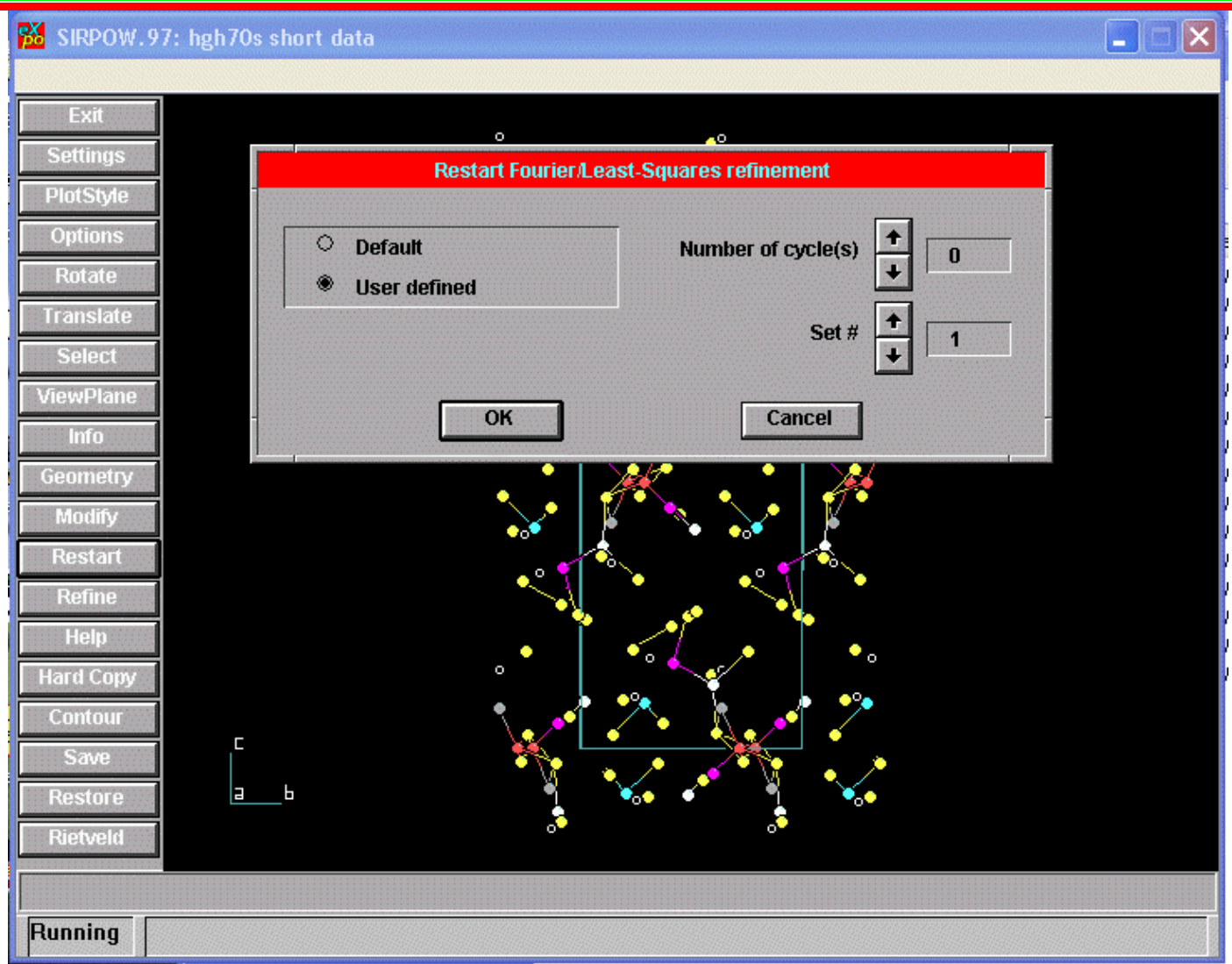
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$



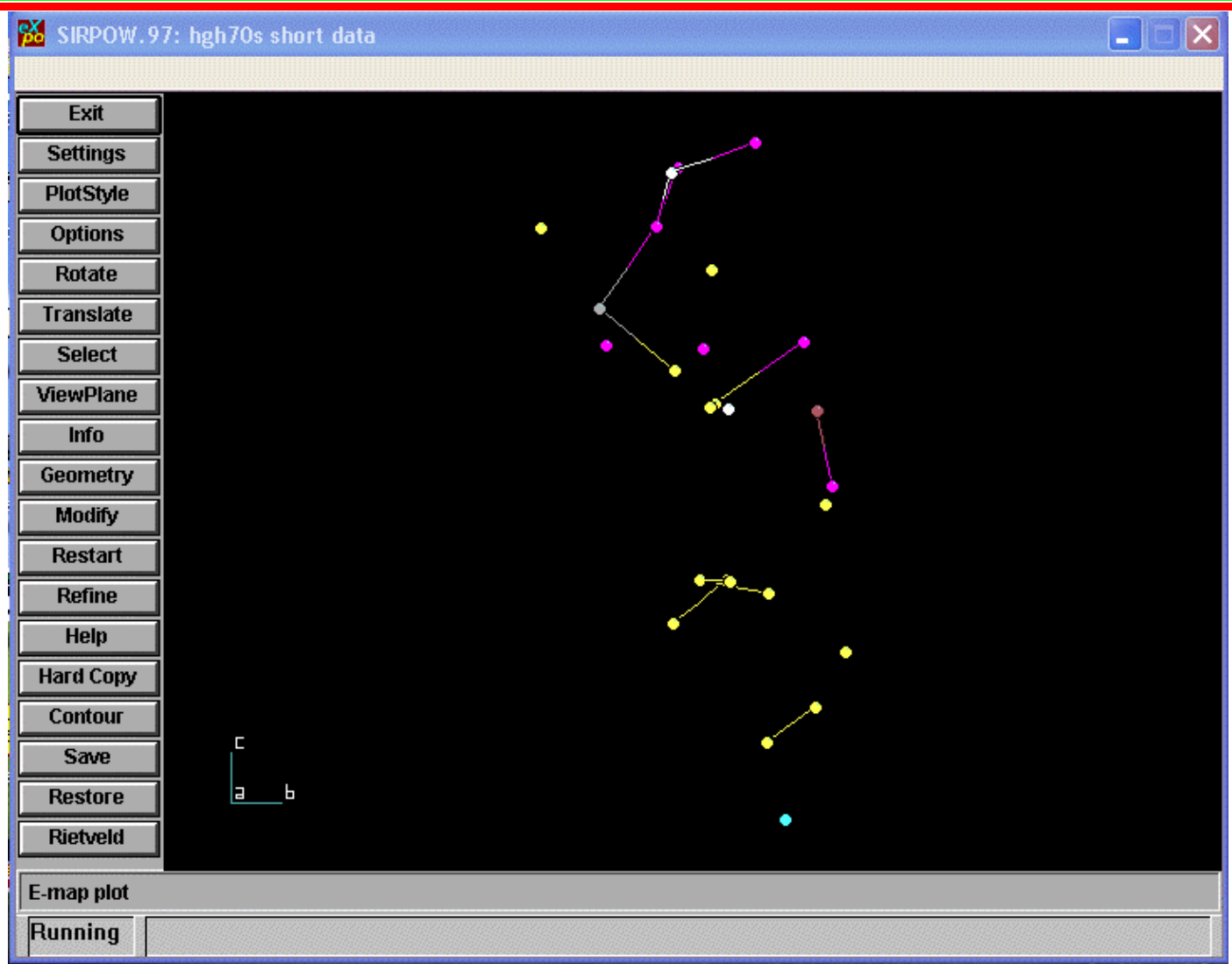
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: No Fourier recycling .exp file

```
%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
```

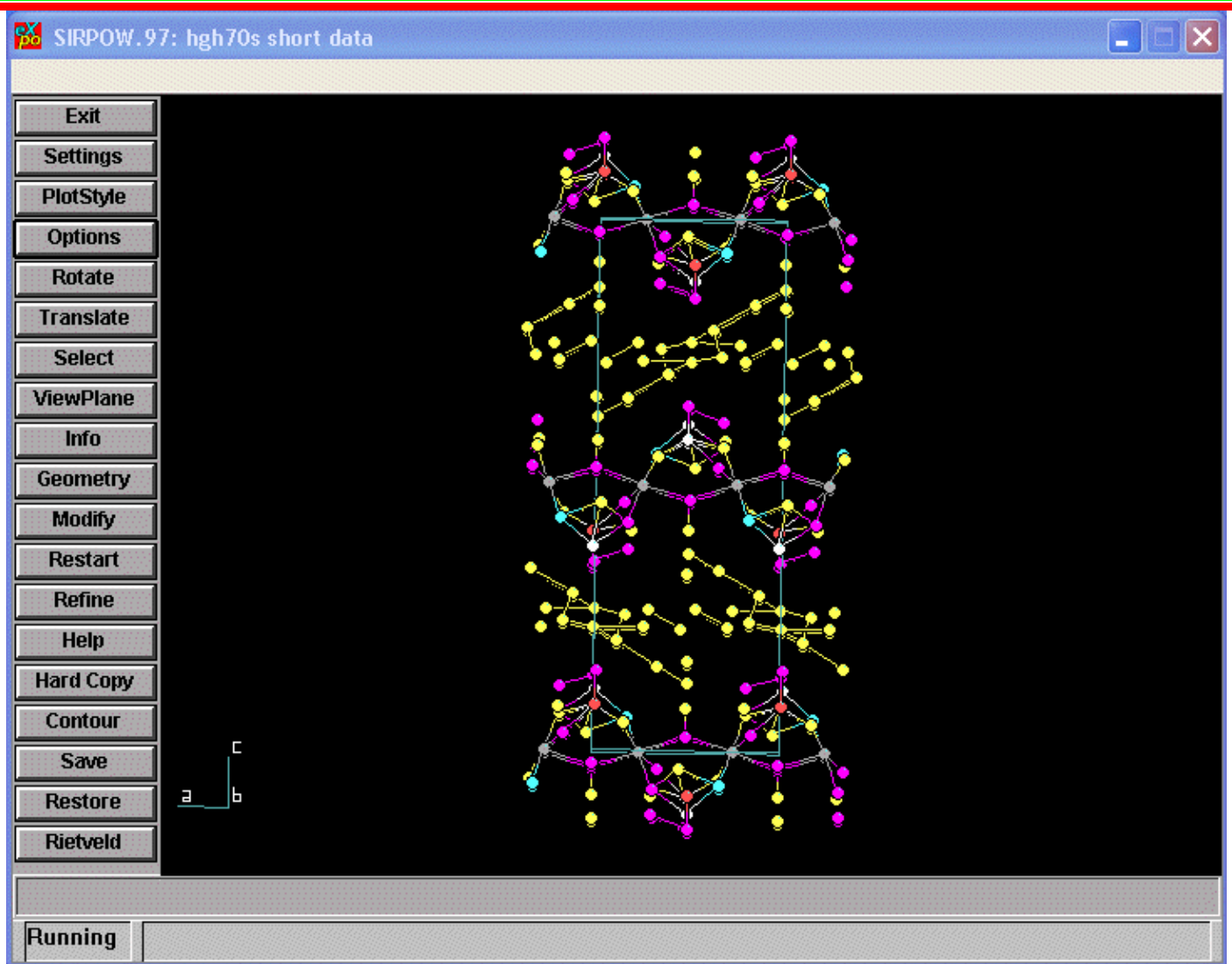
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: No Fourier recycling



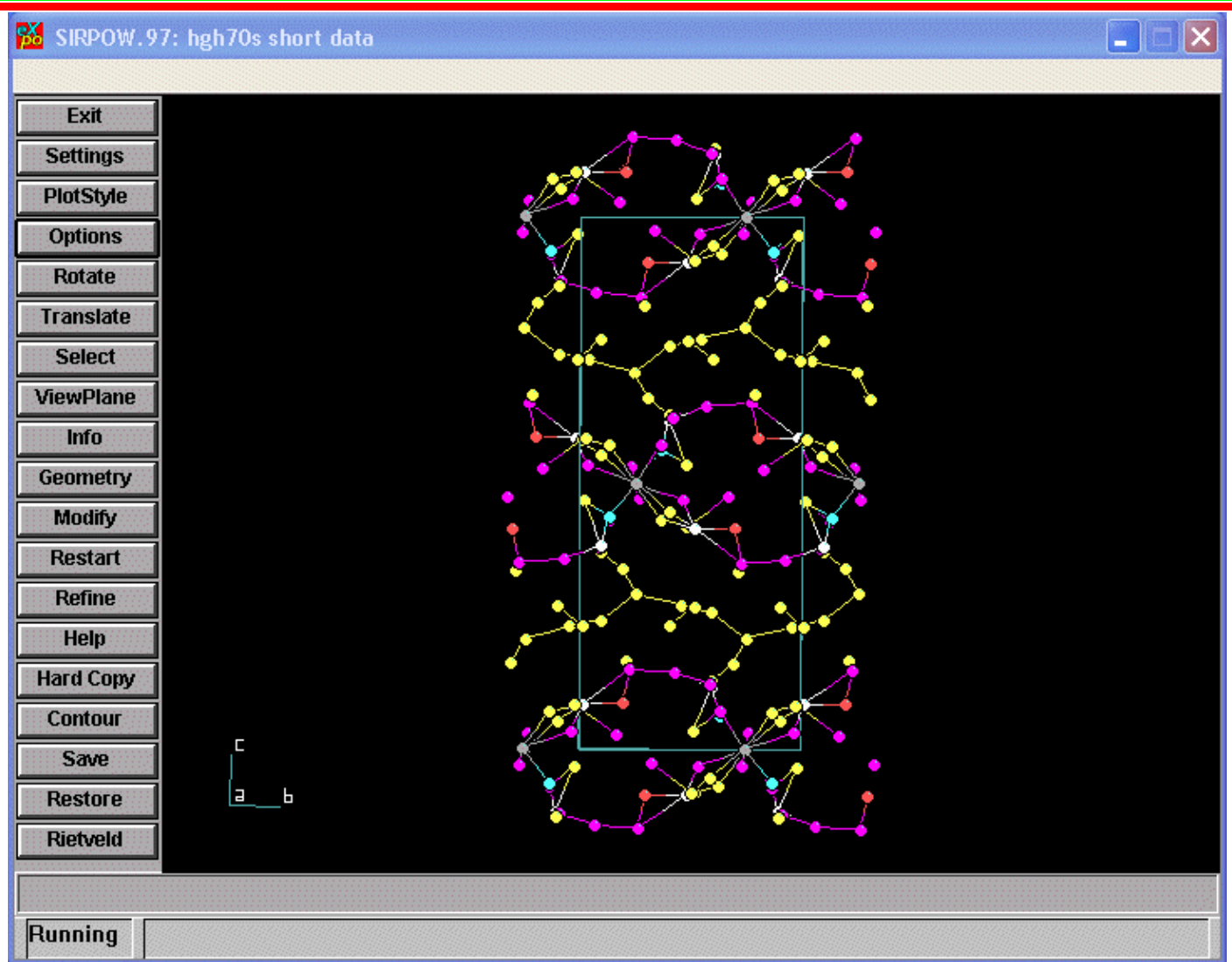
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: No Fourier recycling



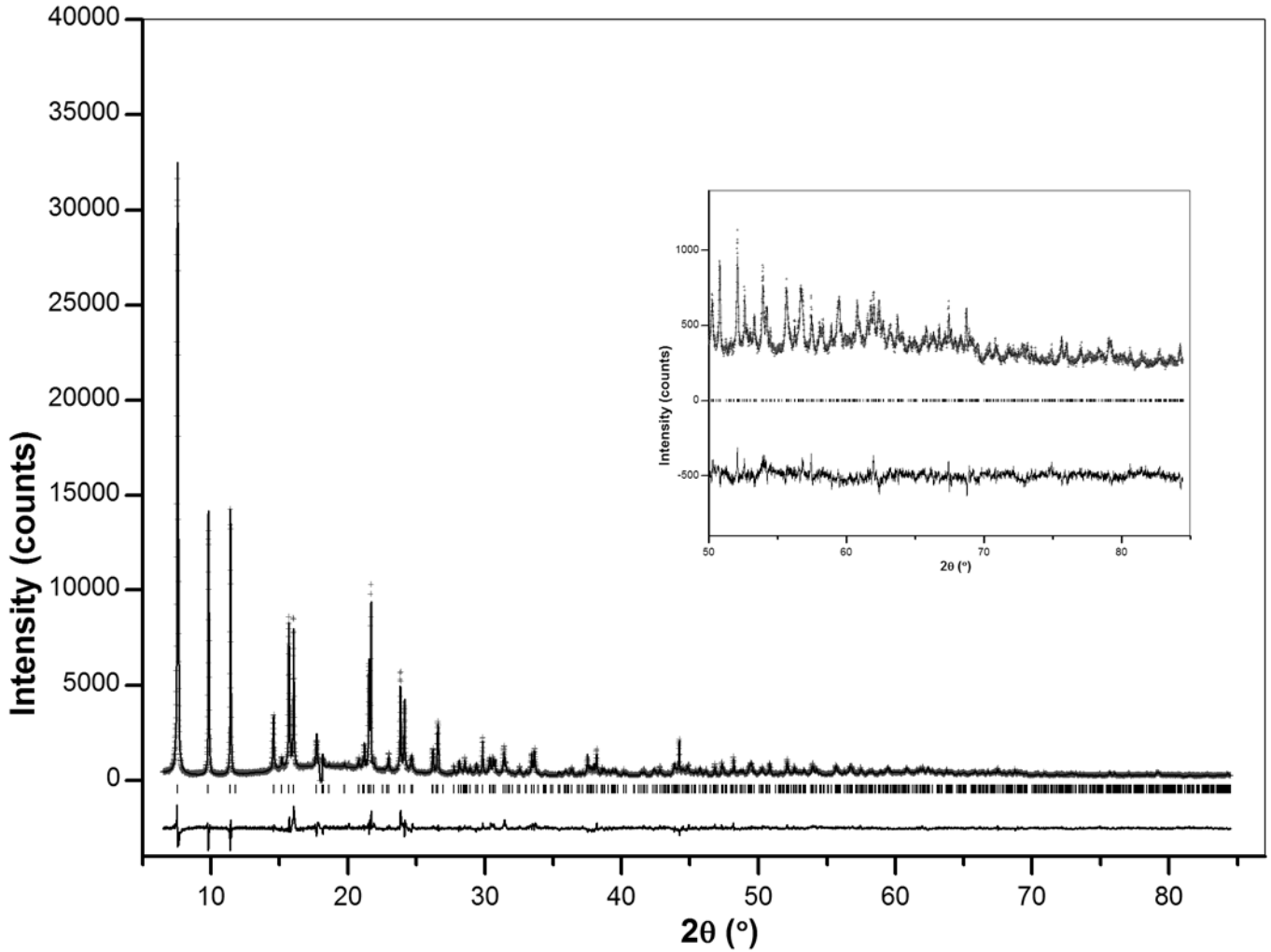
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: No Fourier recycling



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



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



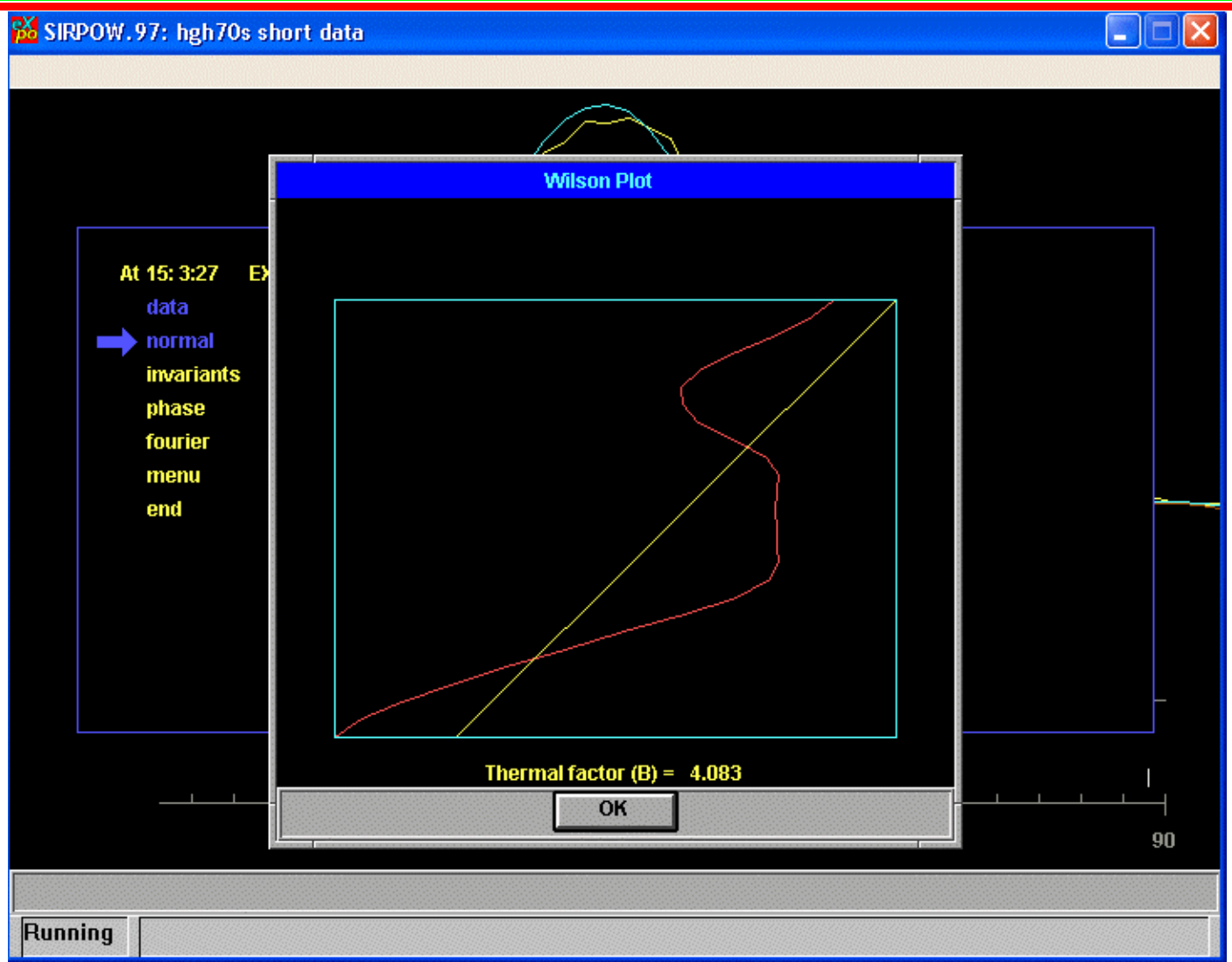
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: External Extraction hkl file

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.

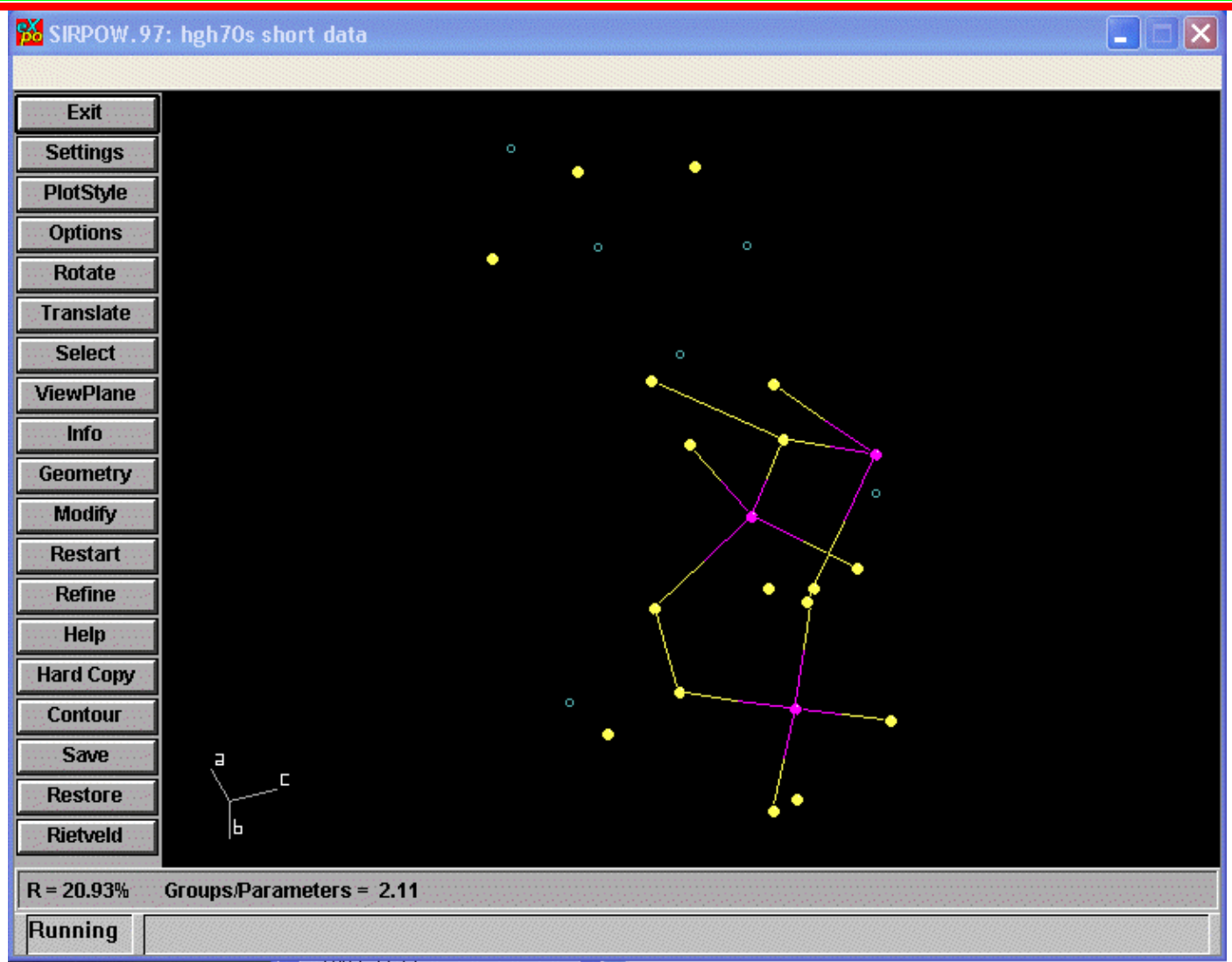
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: External Extraction .exp file

```
%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
```

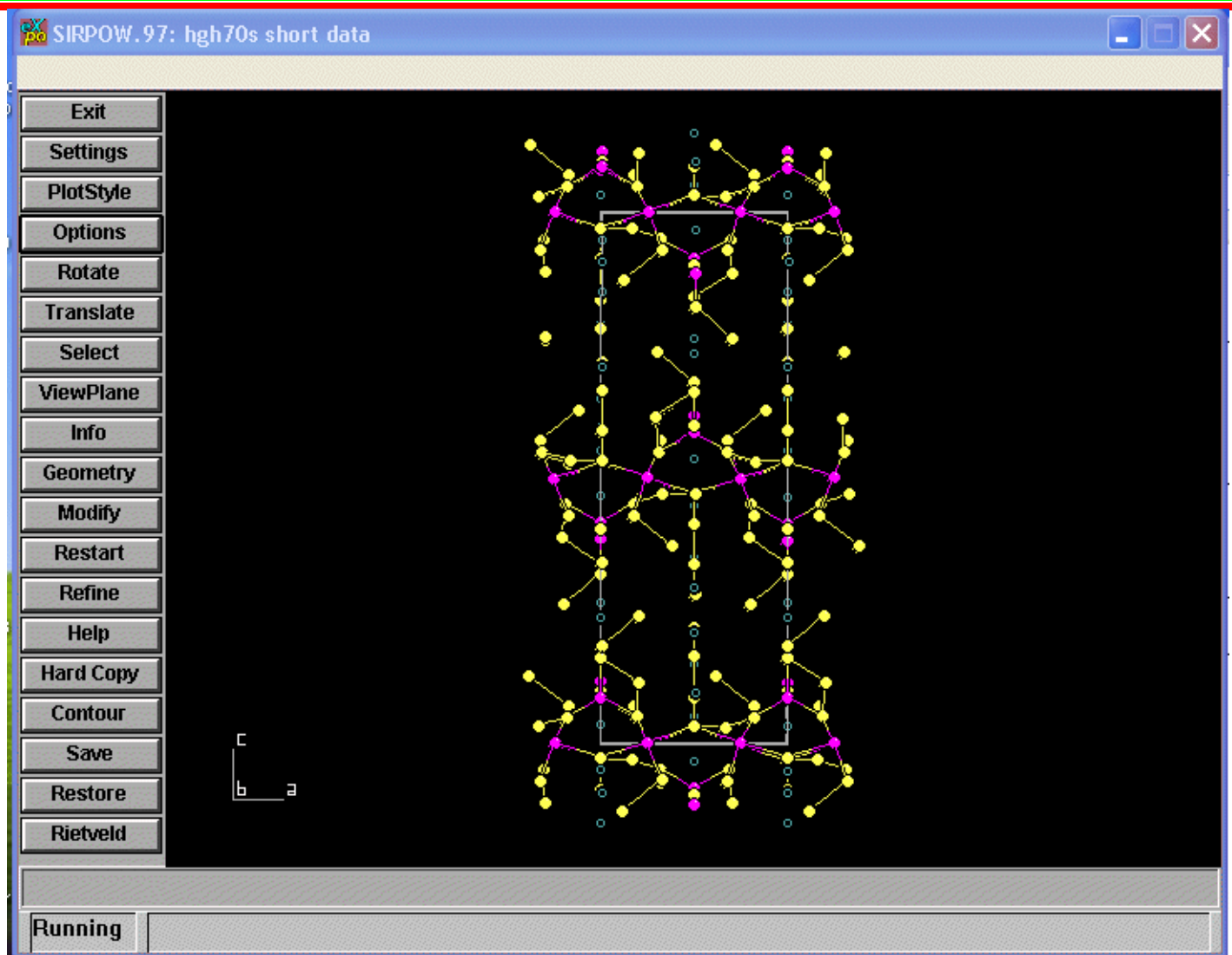
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: External Extraction



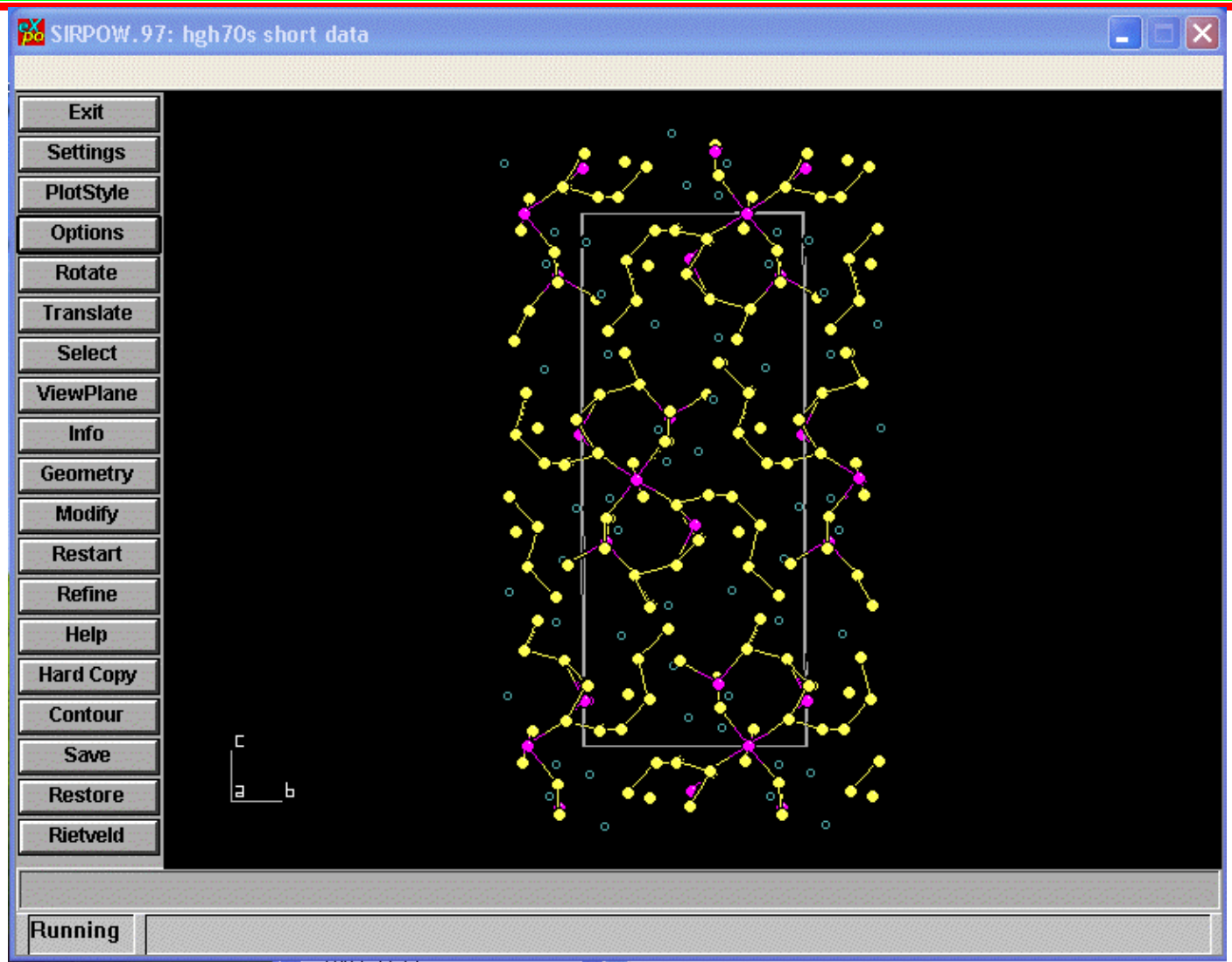
$[C_3H_7NH_3][AlF((HO)O_2PC_2H_4PO_3)]$: External Extraction



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