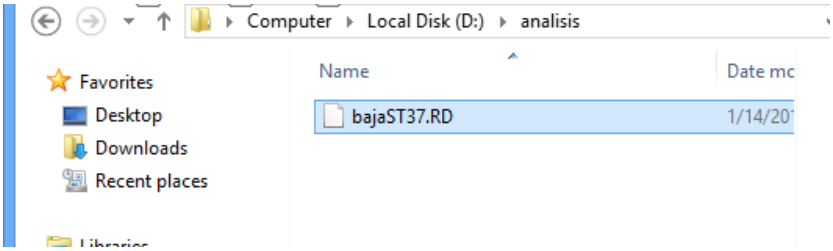


Berikut contoh langkah-langkah menganalisis pola XRD dengan nama file "bajaST37.RD". Ekstensi RD, merupakan file pola XRD dari mesin PANalytical. Tiap mesin XRD mempunyai tipe file tersendiri.

NOTE:

Untuk mengkonversi file pola XRD ke tipe yang lainnya dapat menggunakan aplikasi POWDLL.

Berikut langkah-langkah untuk menganalisis pola XRD menggunakan metode Rietveld dengan aplikasi X'PERT HIGHSCORE PLUS.

1. SEARCH-MATCH

Pada langkah ini, akan dilakukan pencocokan pola XRD dari sampel dengan pola XRD dari database. Database pola XRD ada beberapa, yaitu JCPDS/ICDD, COD dan lain-lain. Saat pencocokan pola XRD yang menjadi acuan adalah sudut/ d spacing puncak dengan intensitas tertinggi dan komposisi yang sebelumnya diperoleh dari XRF atau EDS (bila sampel batuan/ bahan yang tidak dikenal)

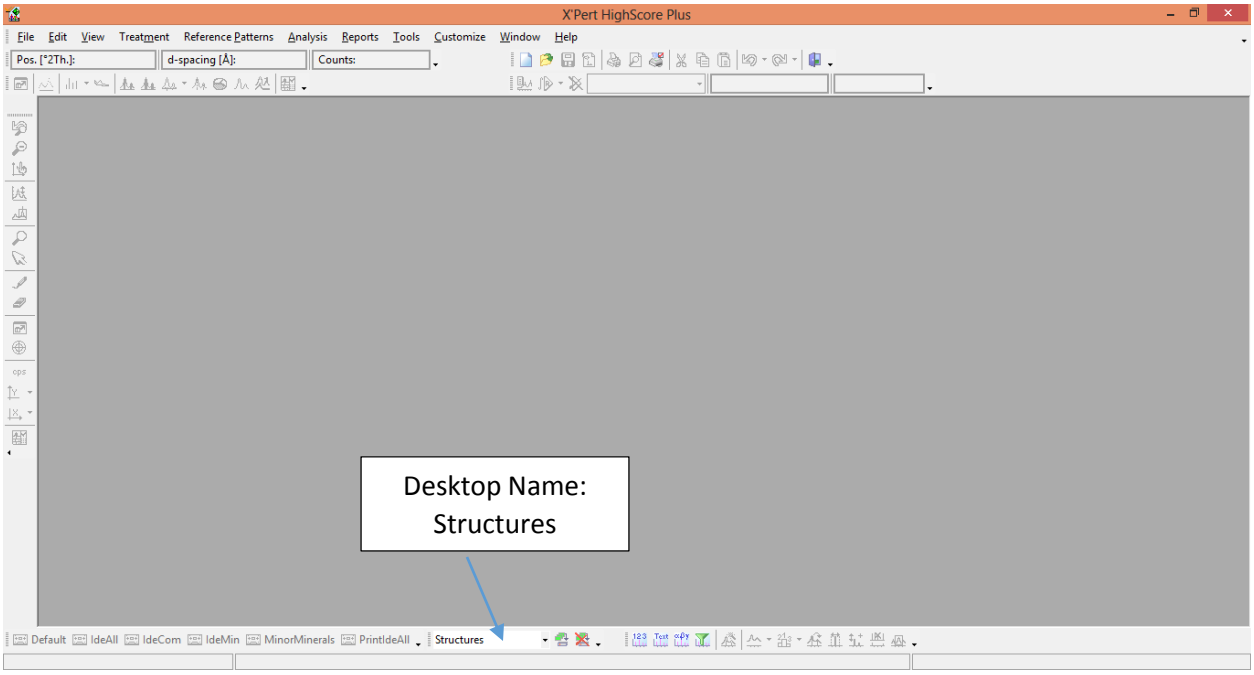
Dari database diperoleh data sistem kristal, space group, parameter kisi, posisi atom dan hkl dari puncak-puncak pola difraksi. Database ICDD PDF2 tidak menyediakan data posisi atom, sehingga diperlukan sumber lain, sedangkan COD sudah disertakan namun jumlah kartu pola difraksi lebih sedikit dibandingkan ICDD. Database ICDD yang menyediakan data posisi atom adalah PDF4, namun lisensinya hanya berlaku satu tahun saja, yang berbeda dengan PDF2.

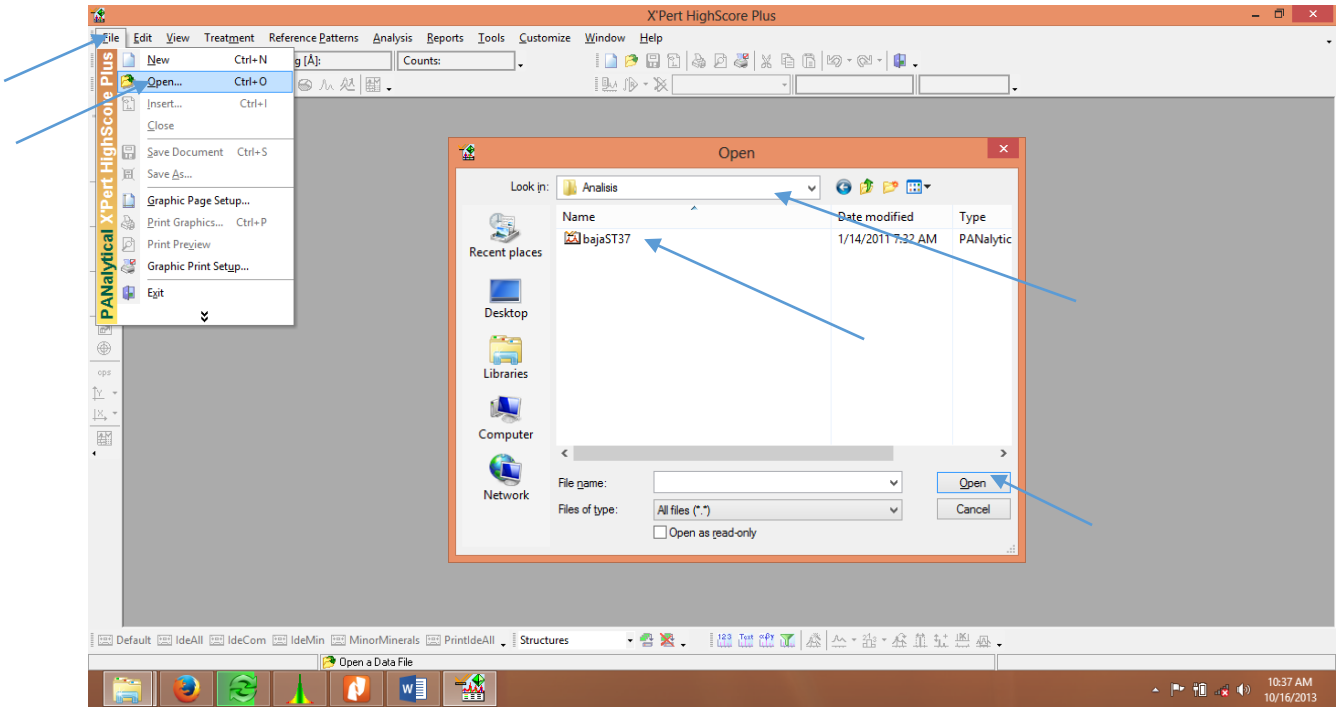
Program yang digunakan adalah X'PERT HIGHSCORE PLUS berikut langkah-langkahnya.

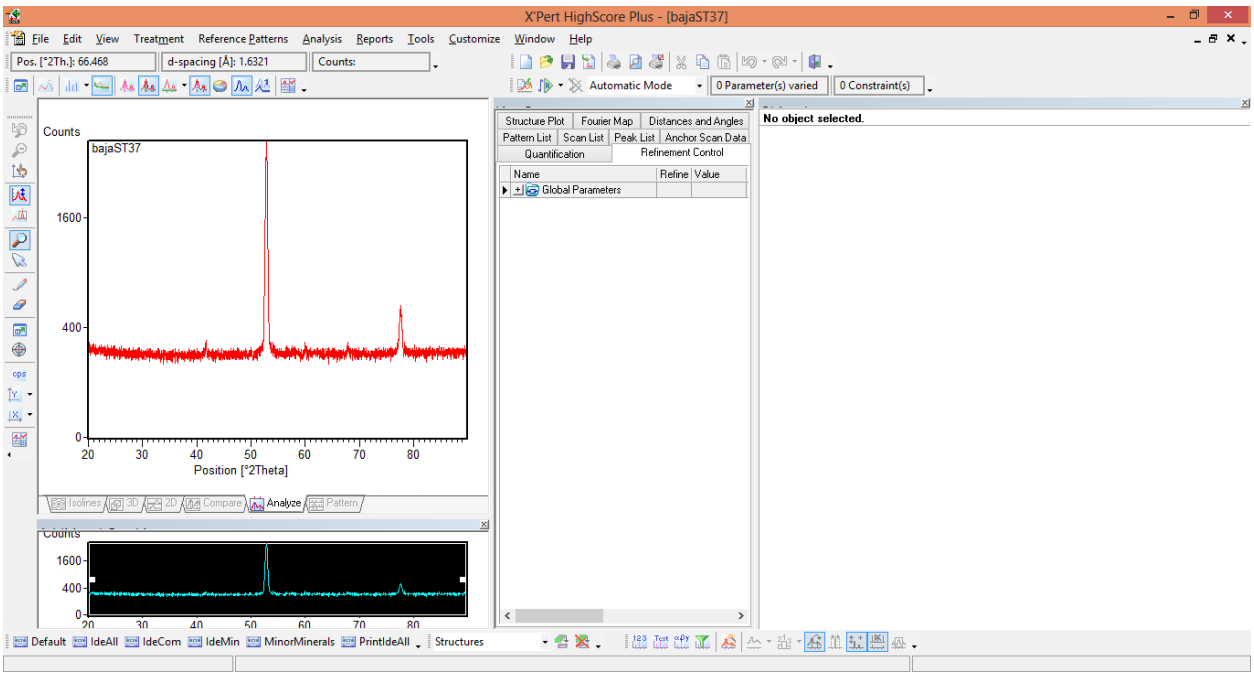
a. Menajalankan program XHP

Klik dua kali pada icon tesebut pada desktop untuk menjalankan XHP

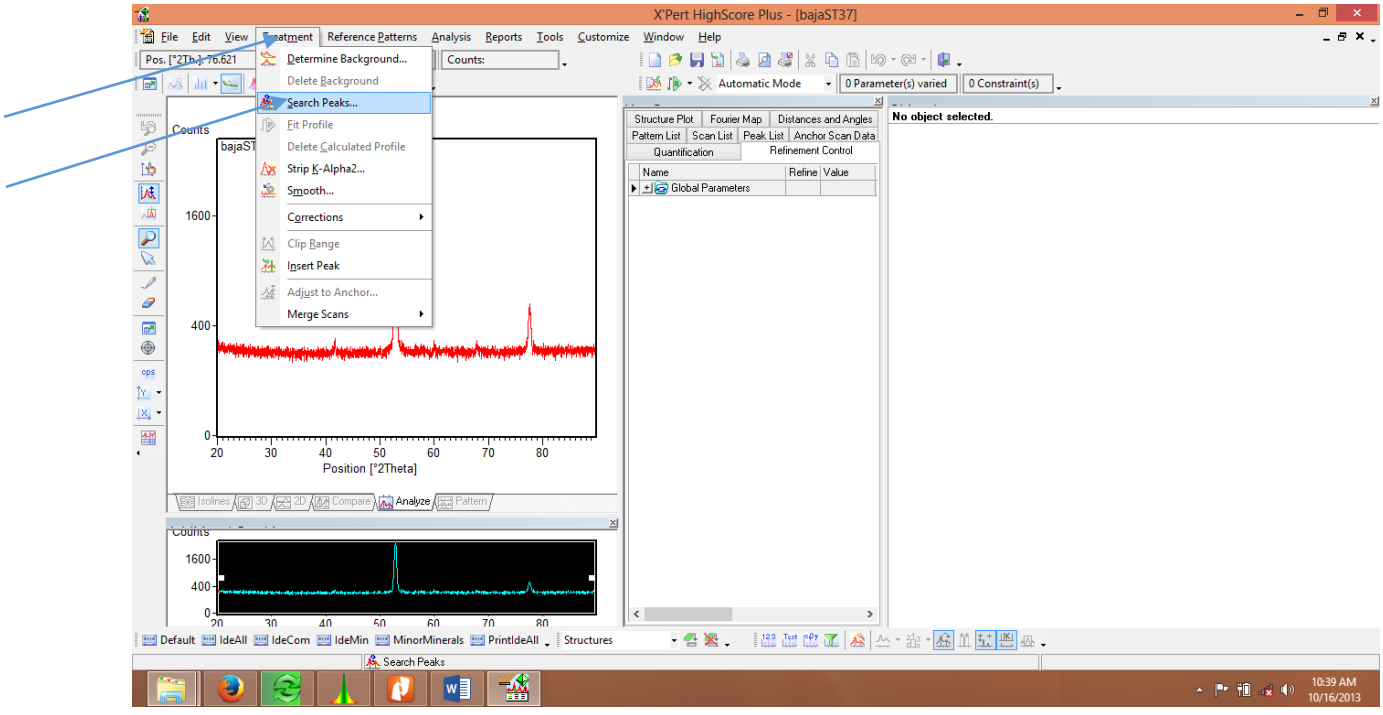
b. Membuka file pola XRD

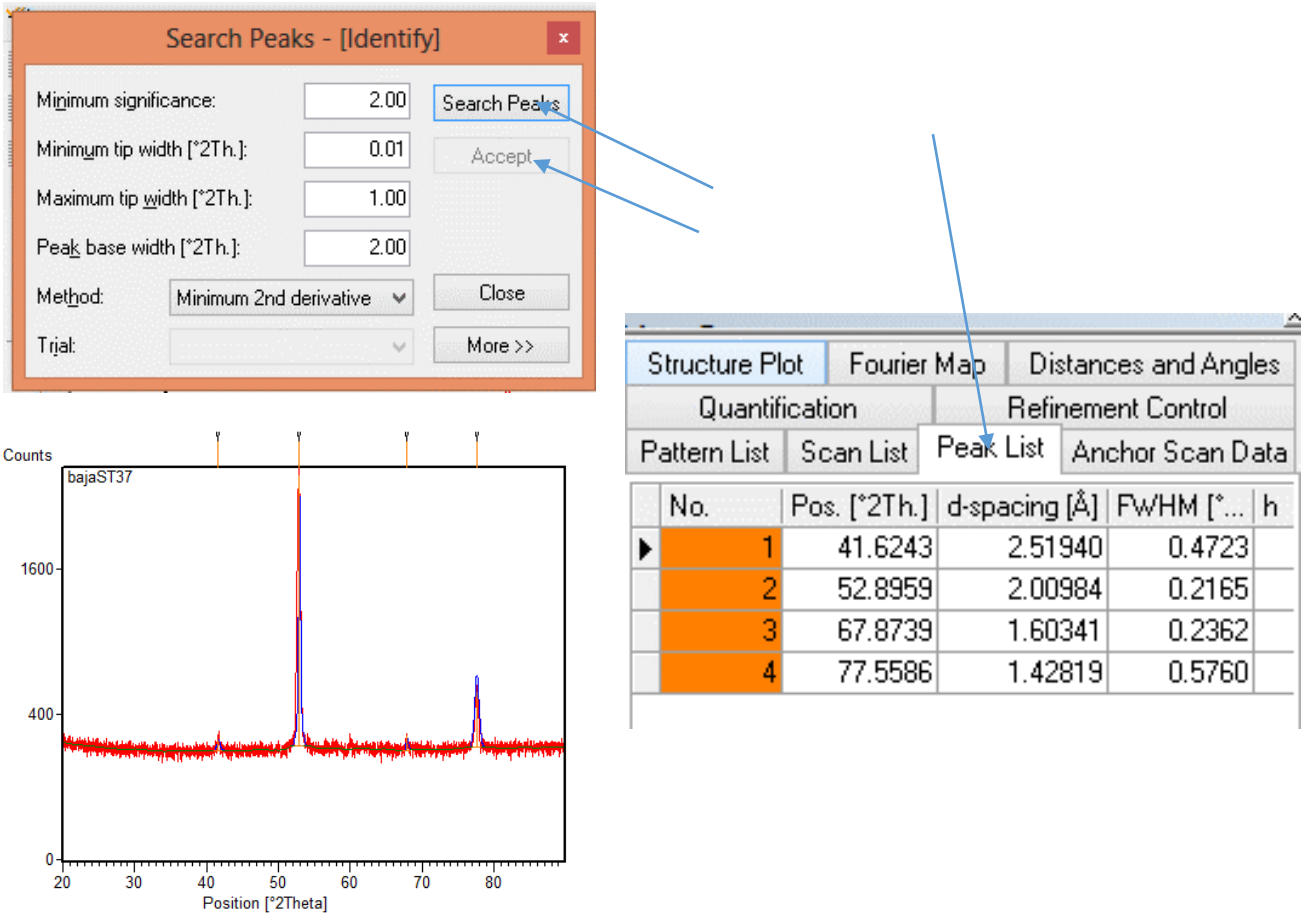

X'Pert HighScore Plus
File Edit View Treatment Reference Patterns Analysis Reports Tools Customize Window Help
Pos. [°2Th.]:
d-spacing [Å]:
Counts:
Desktop Name:
Structures
Default IdeAll IdeCom IdeMin MinorMinerals PrintIdeAll Structures


X'Pert HighScore Plus
File Edit View Treatment Reference Patterns Analysis Reports Tools Customize Window Help
PANalytical X'Pert HighScore Plus
New Ctrl+N
Open... Ctrl+O
Insert... Ctrl+I
Close
Save Document Ctrl+S
Save As...
Graphic Page Setup...
Print Graphics... Ctrl+P
Print Preview
Graphic Print Setup...
Exit
Open
Look in: Analisis
Name
Date modified
Type
bajaST37
1/14/2011 7:32 AM
PANalytic
Recent places
Desktop
Libraries
Computer
Network
File name:
Files of type: All files (*.*)
Open
Cancel
Open as read-only
Default IdeAll IdeCom IdeMin MinorMinerals PrintIdeAll Structures
Open a Data File
10:37 AM
10/16/2013

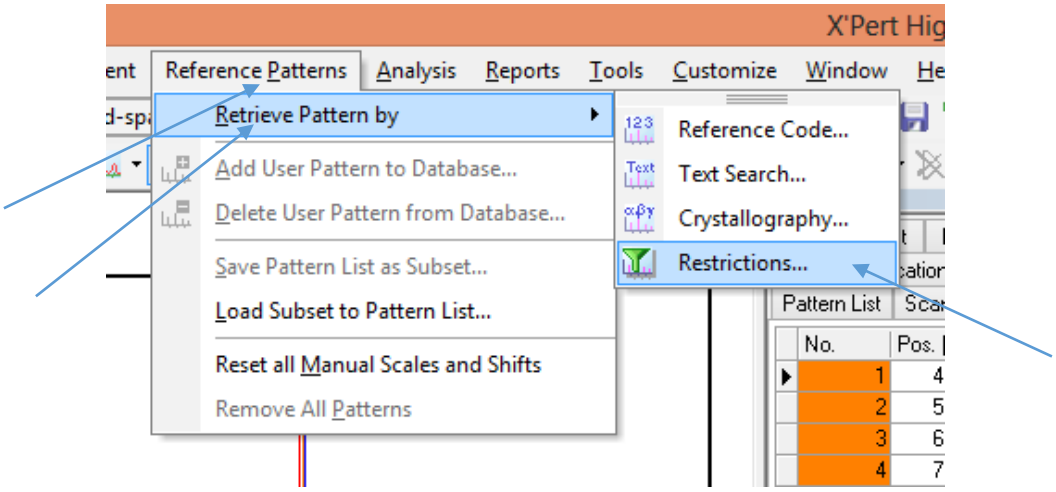


c. Menentukan puncak-puncak pola XRD sampel.

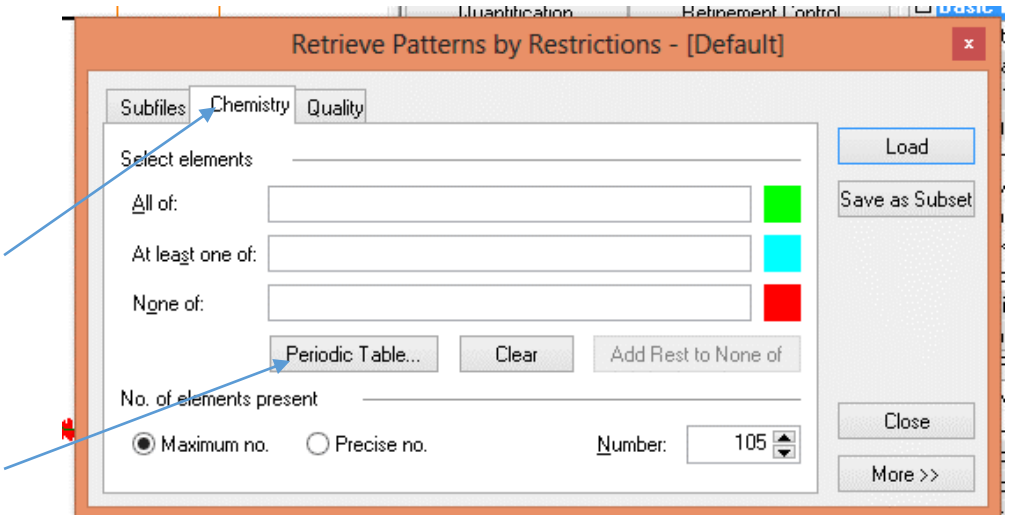


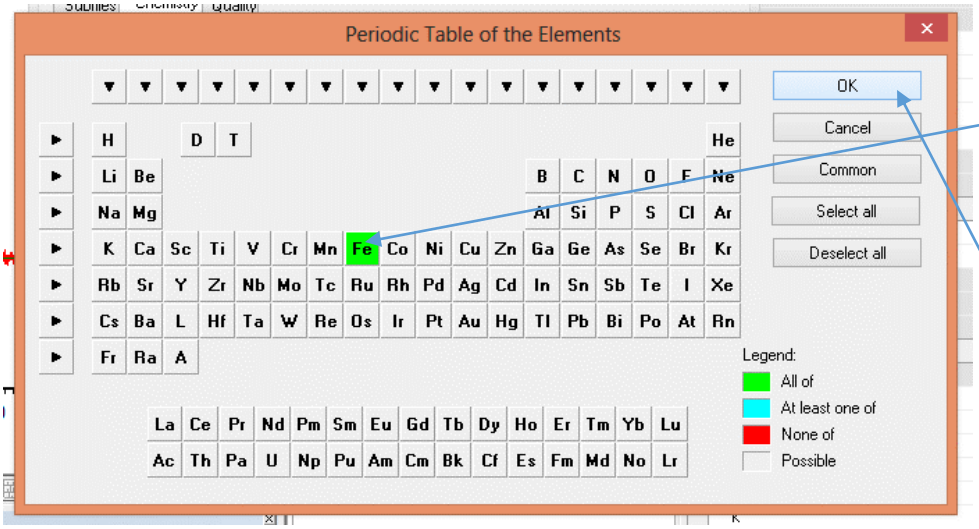


d. Dari data XRF diketahui baja yang dianalisis unsur utamanya adalah Fe sehingga pencarian yang dilakukan hanya pada elemen Fe saja. Berikut langkah-langkahnya,

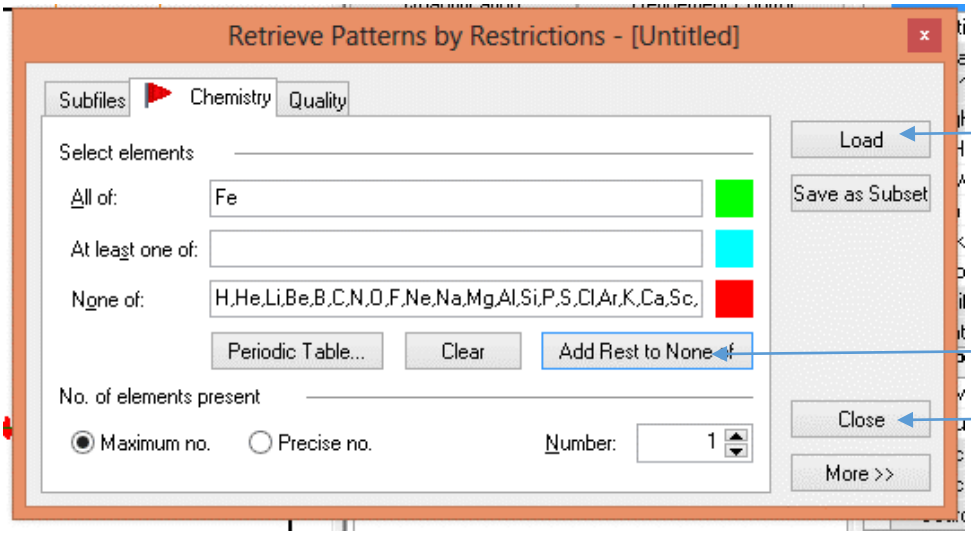


Klik menu Reference Patterns → Retrieve Pattern by → Restrictions…., pencocokan ini akan memakan waktu dan mungkin tidak akan memperoleh hasil.



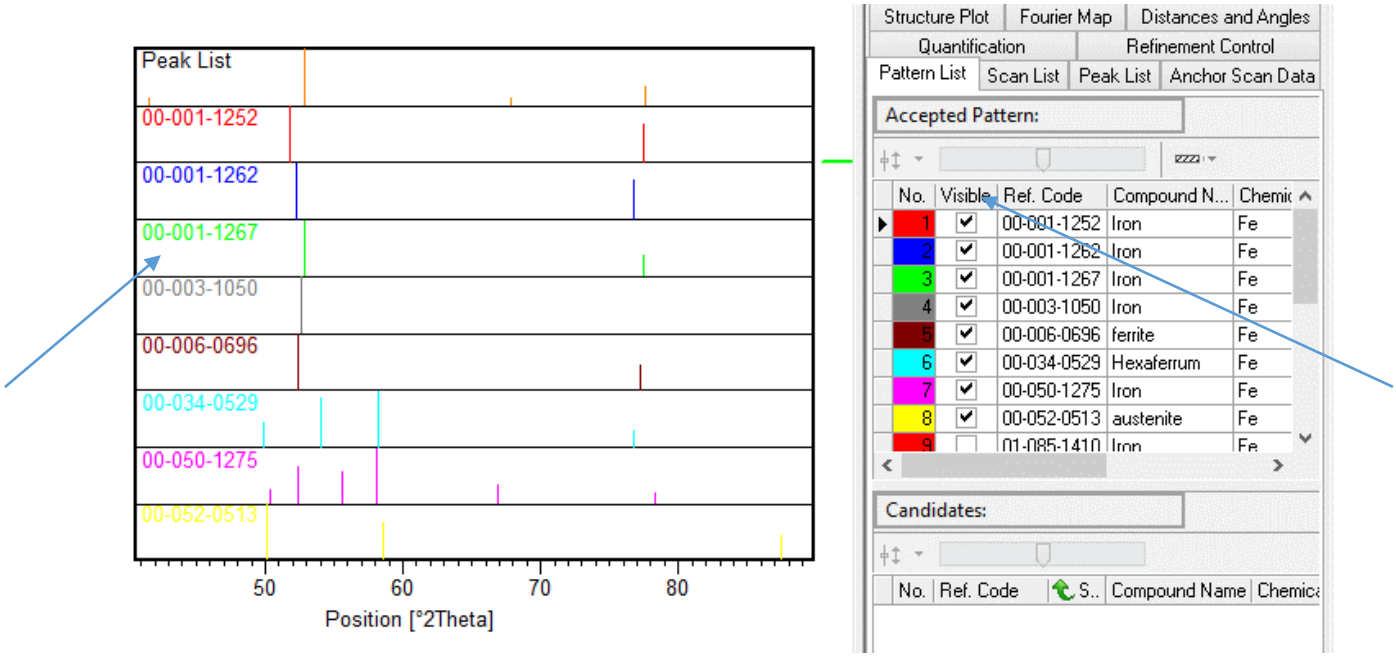


Klik elemen yang diinginkan, bila di klik lebih dari satu kali warna pilihannya akan berubah sesui urutan LEGEND

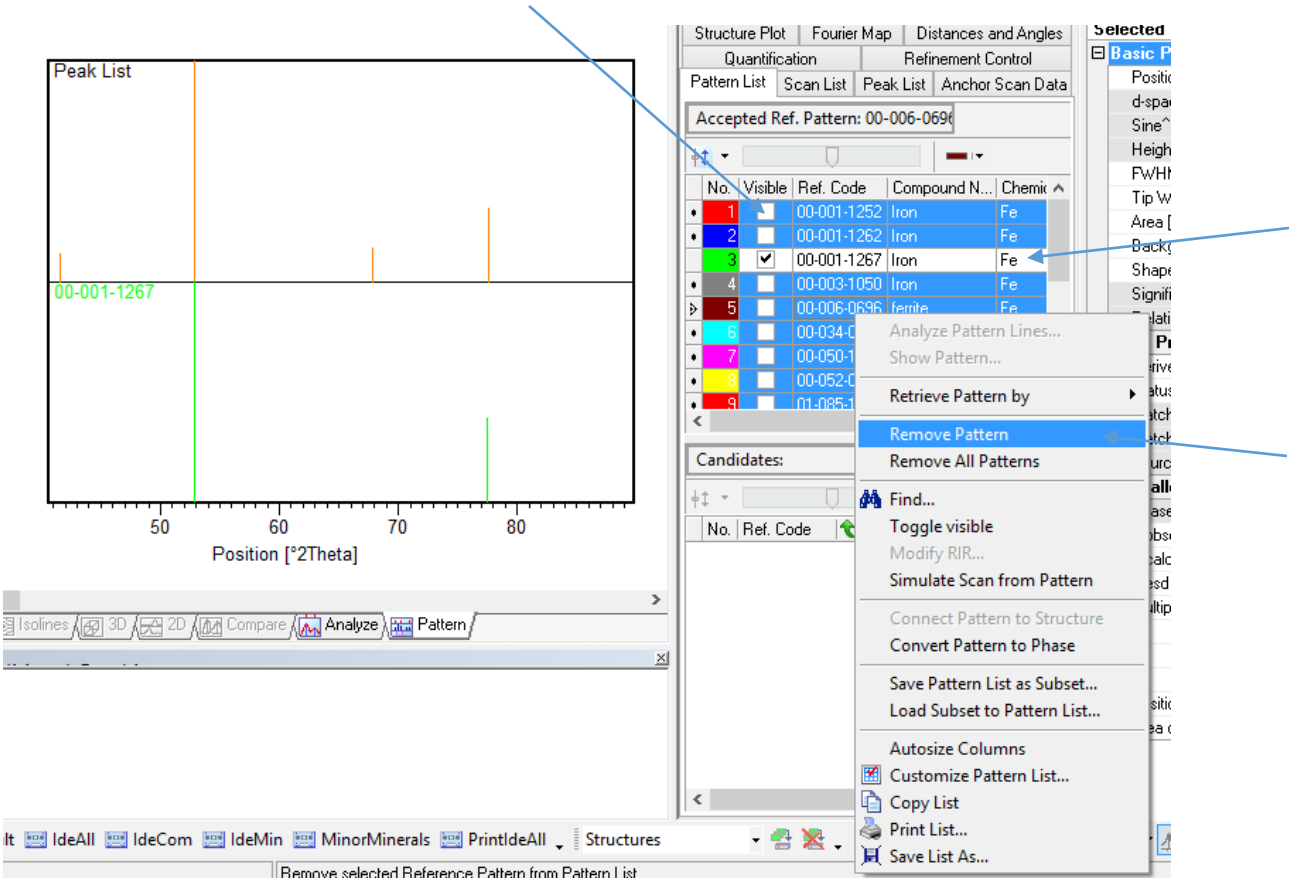


Menunggu.....

ditampilkan entries pada “Pattern List”

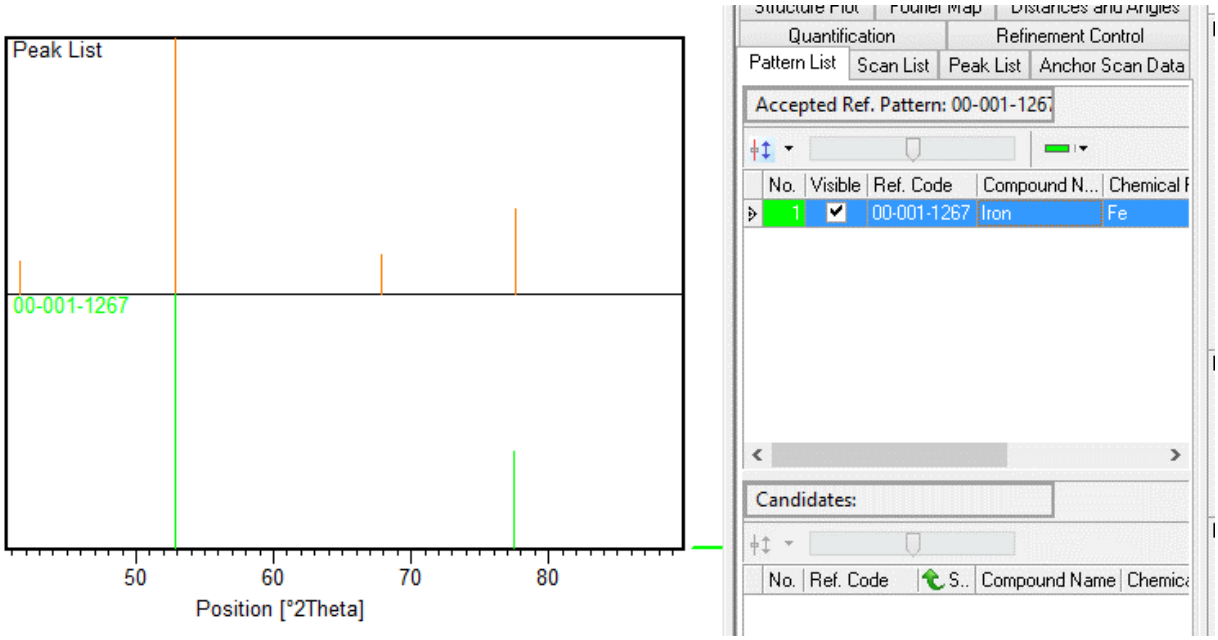


Berikutnya menyisihkan pola difraksi yang tidak sesuai. Dari gambar di atas, yang cocok adalah pola difraksi 00-001-1267.



Pada Patten List, hilangkan tanda check list pada kolom Visible untuk pola difraksi yang tidak cocok, kemudian tekan CTRL+A untuk memilih semua pola difraksi, tahan tombol CTRL dan klik kiri pada pola difraksi 00-001-1267 untuk membatalkan pemilihan pada pola difraksi tersebut.

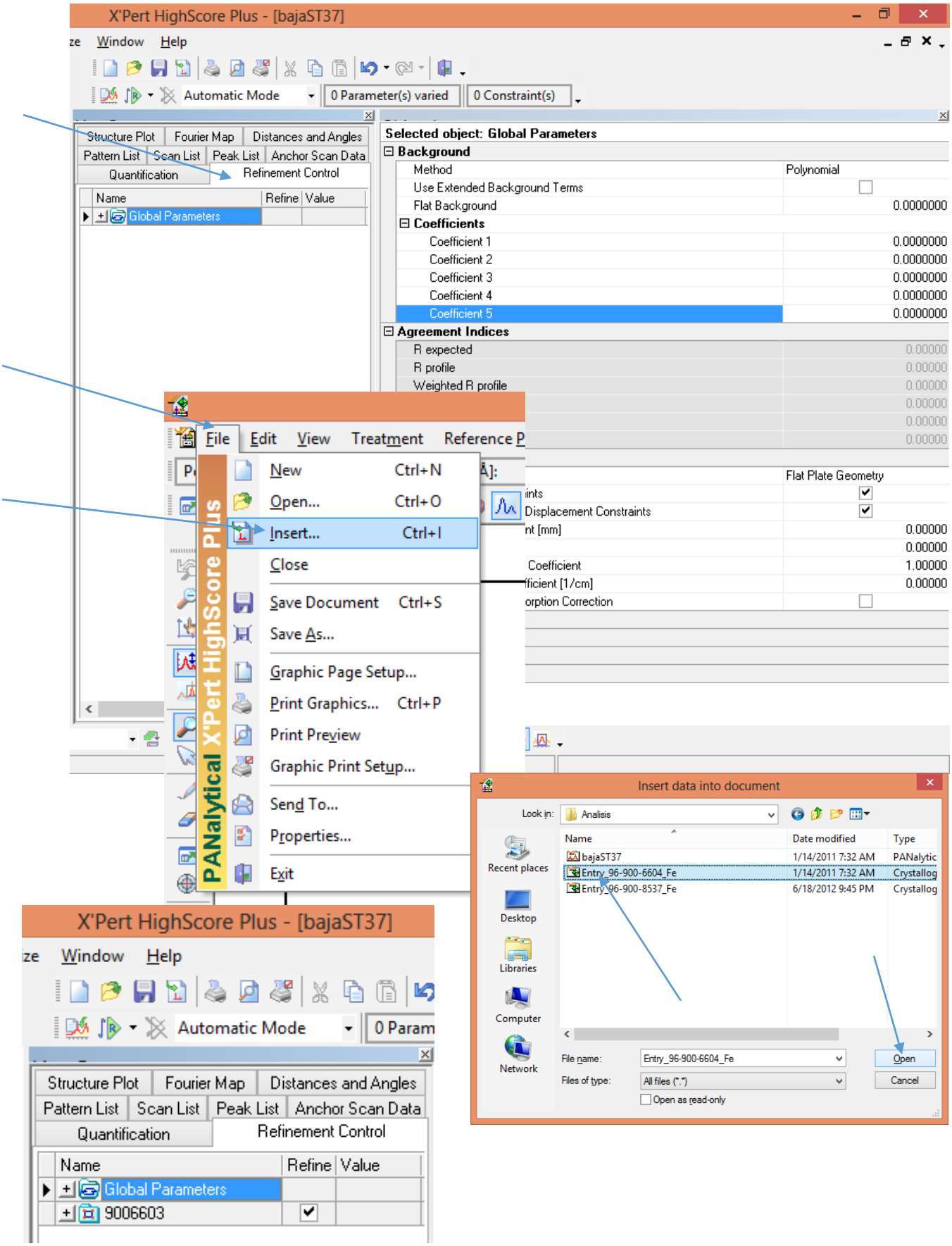
Selanjutnya klik kanan pada pola difraksi yang terpilih, klik pada pilihan Remove Pattern.



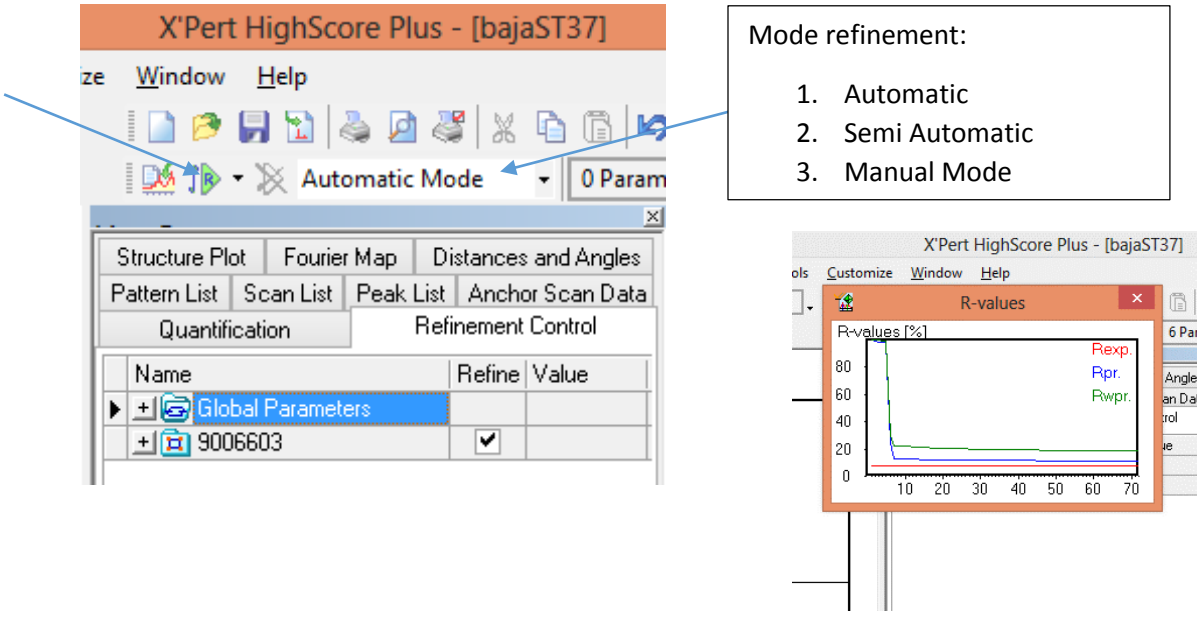
e. Memulai Analisis Rietveld

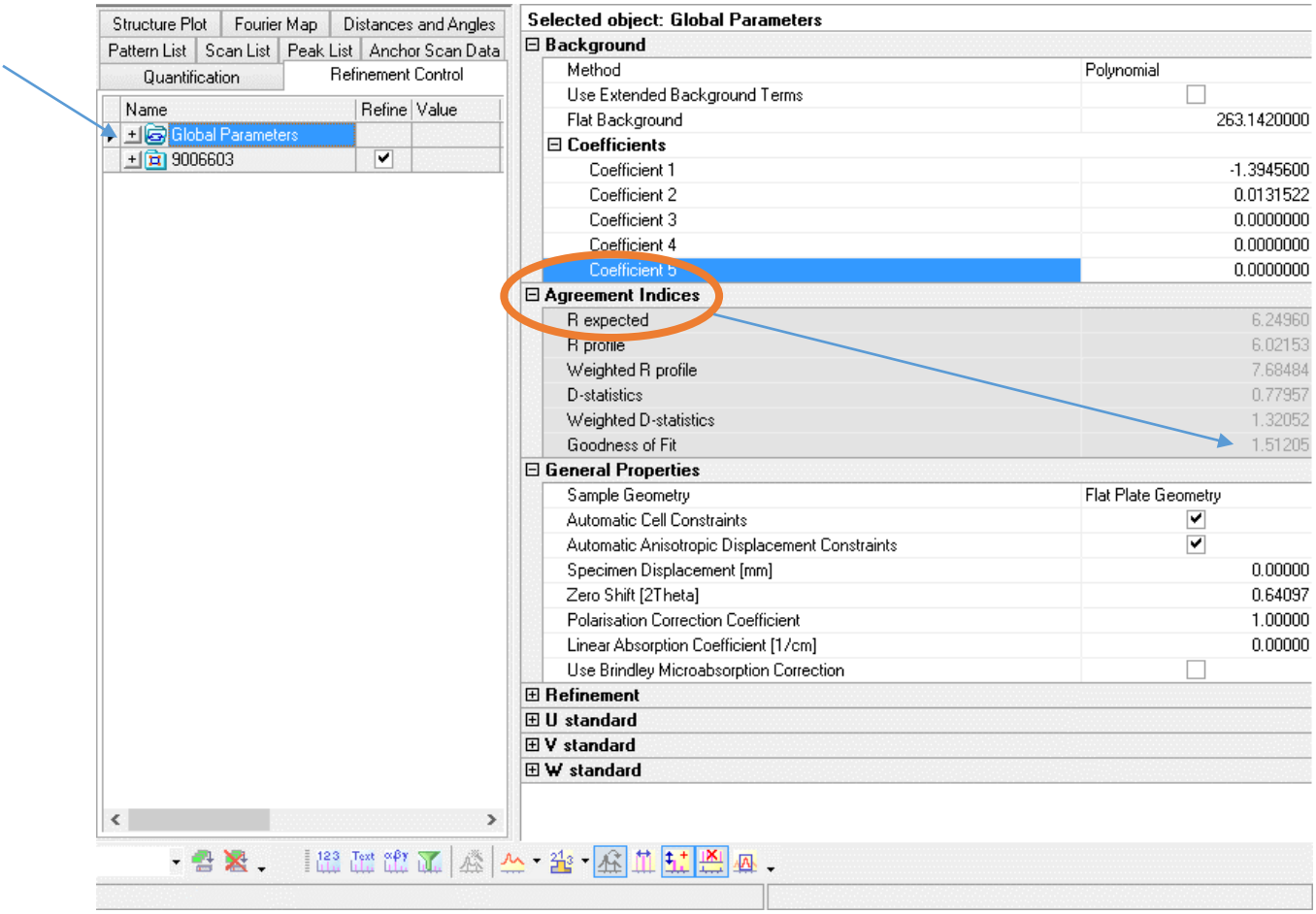
Bila database PDF-4 yang digunakan, pola difraksi pada Pattern List dapat langsung dijadikan acuan untuk analisis Rietveld.

Oleh karena itu diperlukan data kristalografi dari database COD yang sudah berisi data posisi atom. Untuk keperluan analisis diperoleh data CIF dari COD (bisa melihat penggunaan aplikasi MATCH!) dengan nomor kartu 96-900-8537 dan 96-900-6604.


X'Pert HighScore Plus - [bajaST37]
Window
Help
Automatic Mode
0 Parameter(s) varied
0 Constraint(s)
Structure Plot
Fourier Map
Distances and Angles
Pattern List
Scan List
Peak List
Anchor Scan Data
Quantification
Refinement Control
Name
Refine
Value
Global Parameters
Selected object: Global Parameters
Background
Method
Polynomial
Use Extended Background Terms
Flat Background
0.0000000
Coefficients
Coefficient 1
Coefficient 2
Coefficient 3
Coefficient 4
Coefficient 5
Agreement Indices
R expected
R profile
Weighted R profile
Flat Plate Geometry
Displacement Constraints
Coefficient
1.00000
File
Edit
View
Treatment
Reference P
New Ctrl+N
Open... Ctrl+O
Insert... Ctrl+I
Close
Save Document Ctrl+S
Save As...
Graphic Page Setup...
Print Graphics... Ctrl+P
Print Preview
Graphic Print Setup...
Send To...
Properties...
Exit
PANalytical X'Pert HighScore Plus
Insert data into document
Look in:
Analisis
Recent places
Desktop
Libraries
Computer
Network
Name
Date modified
Type
bajaST37
1/14/2011 7:32 AM
PANalytic
Entry_96-900-6604_Fe
1/14/2011 7:32 AM
Crystallog
Entry_96-900-8537_Fe
6/18/2012 9:45 PM
Crystallog
File name:
Entry_96-900-6604_Fe
Open
Files of type:
All files (*.*)
Cancel
Open as read-only
9006603

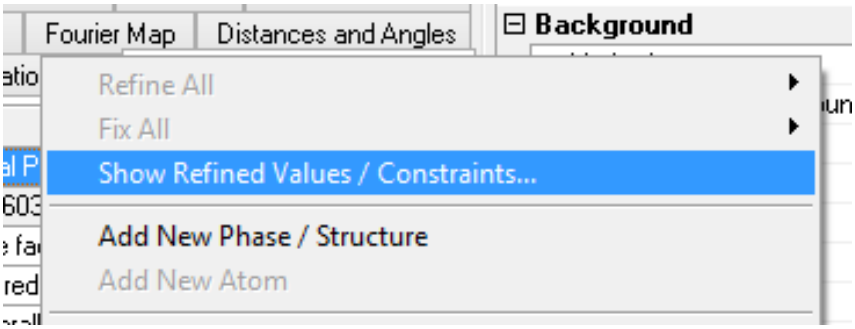
f. Selanjutnya pertama kali “Refine”

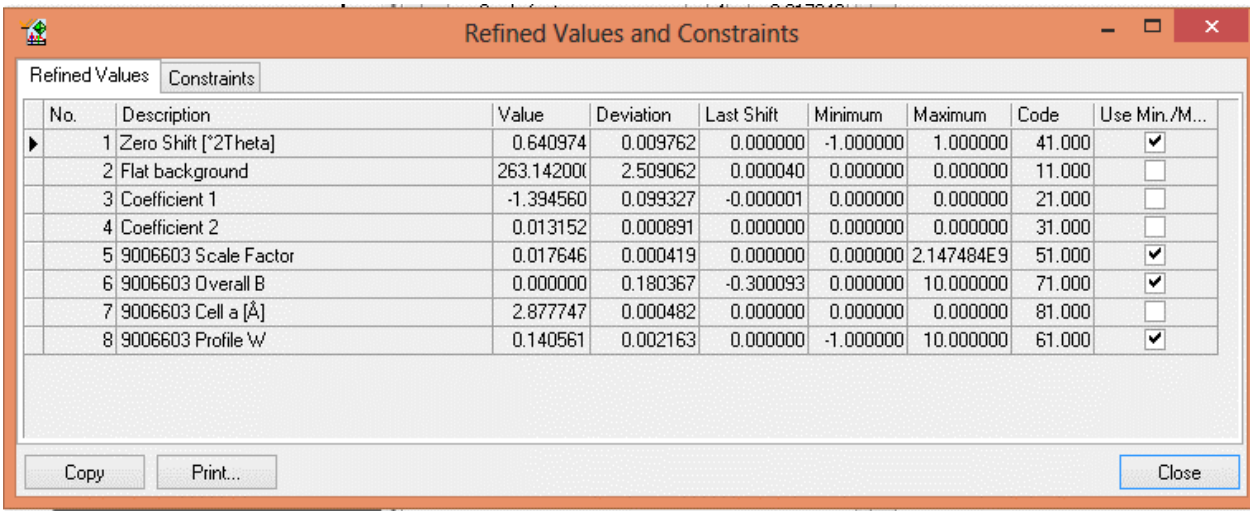




Nilai Goodness of Fit yang diperoleh 1.51205

Untuk melihat parameter apa saja yang di “Refine” klik kanan pada area “Global Parameter” lalu klik “Show Refined Values/Constrains……”





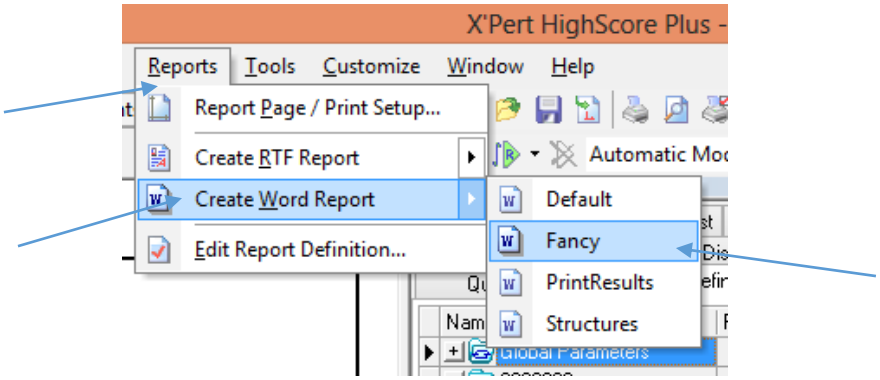
No.	Description	Value	Deviation	Last Shift	Minimum	Maximum	Code	Use Min./M...
1	Zero Shift [°2Theta]	0.640974	0.009762	0.000000	-1.000000	1.000000	41.000	☑
2	Flat background	263.14200(	2.509062	0.000040	0.000000	0.000000	11.000	☐
3	Coefficient 1	-1.394560	0.099327	-0.000001	0.000000	0.000000	21.000	☐
4	Coefficient 2	0.013152	0.000891	0.000000	0.000000	0.000000	31.000	☐
5	9006603 Scale Factor	0.017646	0.000419	0.000000	0.000000	2.147484E9	51.000	☑
6	9006603 Overall B	0.000000	0.180367	-0.300093	0.000000	10.000000	71.000	☑
7	9006603 Cell a [Å]	2.877747	0.000482	0.000000	0.000000	0.000000	81.000	☐
8	9006603 Profile W	0.140561	0.002163	0.000000	-1.000000	10.000000	61.000	☑

g. Proses “Refine” lanjutan.

Untuk melakukan proses “Refine” lanjutan pilih mode refinement menjadi semi automatic. Selanjutnya lakukan penambahan checklist pada parameter global ataupun pada parameter phase.

2. MEMBUAT LAPORAN

Pada langkah ini akan membuat laporan dari ditampilkan dari XHP.



Berikut lampiran file yang terbentuk

X'Pert HighScore **PANalytical**	**X'Pert HighScore Report** 	date: 10/16/13 for internal use only

Fantastic report on Document1

Distribution:

Name	Function	0.1	0.5	1.0
N.N.-999	Masterking Group Manager (MGM)	✓		
N.N.-998	Product Ontwikkelt Manager (POM)	✓		
N.N.-1	(AS)			✓
N.N.-2	(DS)			✓
N.N.-3	(BS)			✓
T.P.	Supervisor All Coding			✓

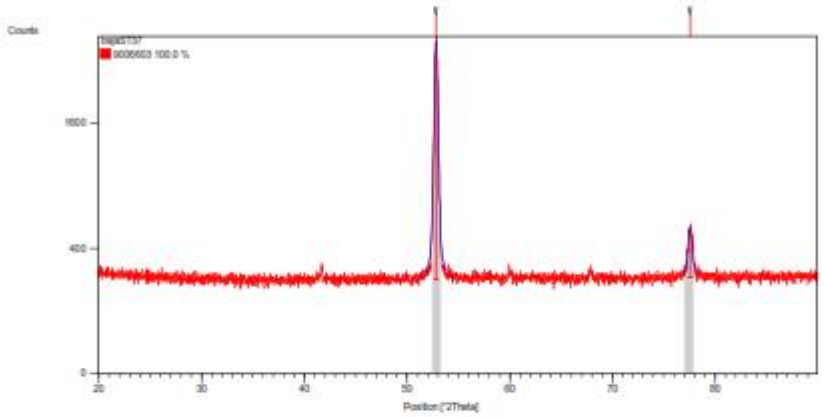
Last saved by Jan Setiawan	Draft	file: bajaST37	rev: 1
			No. of pages: 1/9

Table of Contents:

Anchor Scan Parameters: (Bookmark1)

Sample Identification	ST370
Comment	Material Science
Comment	PC-APD, Diffraction software
Measurement Date / Time	9/5/2009 1:20:00 PM
Raw Data Origin	PHILIPS-binary (scan) (.RD)
Scan Axis	Gonio
Start Position [°2Th.]	20.0000
End Position [°2Th.]	89.9800
Step Size [°2Th.]	0.0200
Scan Step Time [s]	1.0000
Scan Type	Continuous
Offset [°2Th.]	0.0000
Divergence Slit Type	Fixed
Divergence Slit Size [°]	0.2500
Specimen Length [mm]	10.00
Receiving Slit Size [mm]	0.2000
Measurement Temperature [°C]	0.00
Anode Material	Co
K-Alpha1 [Å]	1.78901
K-Alpha2 [Å]	1.79290
K-Beta [Å]	1.62083
K-A2 / K-A1 Ratio	0.50000
Generator Settings	30 mA, 40 kV
Diffractometer Type	PW3710
Diffractometer Number	1
Goniometer Radius [mm]	173.00
Dist. Focus-Diverg. Slit [mm]	91.00
Incident Beam Monochromator	No
Spinning	No

Graphics: (Bookmark2)



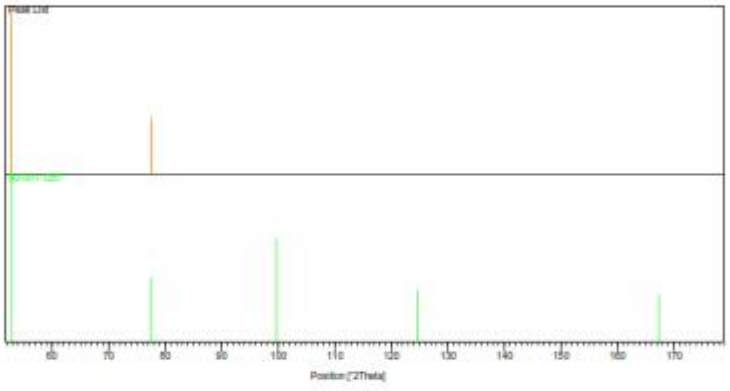
Peak List: (Bookmark 3)

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip width [°2Th.]	Matched by
52.7961	2537.10	0.3749	2.01191	100.00	0.4499	00-001-1267
52.9180	2279.90	0.3749	2.01197	89.86	0.4499	
77.5174	301.83	0.3749	1.42883	11.90	0.4499	00-001-1267
77.7153	236.54	0.3749	1.42886	9.32	0.4499	

Identified Patterns List: (Bookmark4)

Visible	Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
*	00-001-1267	29	Iron	0.000	0.969	Fe

Plot of Identified Phases: (Bookmark 5)



Document History: (Bookmark 6)

Insert Measurement:
- File name = "bajaST37.RD"
- Modification time = "10/16/2013 10:38:43 AM"
- Modification editor = "Jan Setiawan"

Search Peaks:
- Minimum significance = "2.00"
- Minimum tip width = "0.01"
- Maximum tip width = "1.00"
- Peak base width = "2.00"
- Method = "Minimum 2nd derivative"
- Modification time = "2/20/2001 11:55:18 AM"
- Modification editor = "PANalytical"

Insert Structure(s):
- File name = "Entry_96-900-6604_Fe.cif"
- Modification time = "10/16/2013 11:09:08 AM"
- Modification editor = "Jan Setiawan"

Delete Selected Phase:
- Modification time = "10/16/2013 11:09:17 AM"
- Modification editor = "Jan Setiawan"

Insert Structure(s):
- File name = "Entry_96-900-6604_Fe.cif"
- Modification time = "10/16/2013 11:09:25 AM"
- Modification editor = "Jan Setiawan"

Change 9006603 Use Flag:
- Modification time = "10/16/2013 11:09:54 AM"
- Modification editor = "Jan Setiawan"

Delete All Phases:
- Modification time = "10/16/2013 11:10:01 AM"
- Modification editor = "Jan Setiawan"

Insert Structure(s):
- File name = "Entry_96-900-6604_Fe.cif"
- Modification time = "10/16/2013 11:16:09 AM"
- Modification editor = "Jan Setiawan"

Rietveld Refinement:
- Step No. 1
- Title = "Scale factor"
- Min. Shift/ESD = "0.1"
- Switch off after usage = "False"
- Step No. 2
- Title = "Flat background"
- Min. Shift/ESD = "0.1"

- Switch off after usage = "False"
- Step No. 3
- Title = "Zero shift"
- Min. Shift/ESD = "0.1"
- Switch off after usage = "False"
- Refine spec. displ. = "False"
- Step No. 4
- Title = "Lattice parameters"
- Min. Shift/ESD = "0.8"
- Switch off after usage = "False"
- Minimum weight % = "-1"
- Step No. 5
- Title = "More background"
- Min. Shift/ESD = "0.1"
- Switch off after usage = "False"
- No. additional parameters = "2"
- Step No. 6
- Title = "W (Halfwidth)"
- Min. Shift/ESD = "0.1"
- Switch off after usage = "False"
- Minimum weight % = "1.5"
- Step No. 7
- Title = "Site ocupancy factor and B isotropic"
- Min. Shift/ESD = "0.1"
- Switch off after usage = "False"
- Minimum atomic number = "10"
- Use overall B = "True"
- Refine mixed sites = "False"
- Minimum weight % = "-1"
- Modification time = "10/16/2013 11:20:35 AM"
- Modification editor = "Jan Setiawan"

Rietveld Refinement:
- Step No. 1
- Title = "Scale factor"
- Min. Shift/ESD = "0.1"
- Switch off after usage = "False"
- Step No. 2
- Title = "Flat background"
- Min. Shift/ESD = "0.1"
- Switch off after usage = "False"
- Step No. 3
- Title = "Zero shift"
- Min. Shift/ESD = "0.1"
- Switch off after usage = "False"
- Refine spec. displ. = "False"
- Step No. 4
- Title = "Lattice parameters"
- Min. Shift/ESD = "0.8"
- Switch off after usage = "False"
- Minimum weight % = "-1"
- Step No. 5
- Title = "More background"

- Min. Shift/ESD = "0.1"
- Switch off after usage = "False"
- No. additional parameters = "2"
- Step No. 6
- Title = "W (Halfwidth)"
- Min. Shift/ESD = "0.1"
- Switch off after usage = "False"
- Minimum weight % = "1.5"
- Step No. 7
- Title = "Site ocupancy factor and B isotropic"
- Min. Shift/ESD = "0.1"
- Switch off after usage = "False"
- Minimum atomic number = "10"
- Use overall B = "True"
- Refine mixed sites = "False"
- Minimum weight % = "-1"
- Modification time = "10/16/2013 11:45:02 AM"
- Modification editor = "Jan Setiawan"

Untuk mengubah penampilan Report sesuai dengan kebutuhan,