TUTORIAL QUALX

The **QUALX** folder contains:

- **DEFAULT** folder
- **NO_DEFAULT folder**

- **DEFAULT** folder

It contains: **Sample_2.dat** [the file containing the experimental profile counts, in case of a four-phase mixture (**Sample 2**), laboratory data, Cu $K_{\alpha1,2}$ radiation]; **Sample_2_True_Phases.docx** [the file providing the true crystalline phases and their true weight percentages (w_T) of **Sample 2**]; **Sample_2_Round_Robin_QPA_CPD.pdf** [the paper in which the outcomes on Quantitative Phase Analysis (QPA), provided in the file **Sample_2_True_Phases.docx**, are given].

Sample 2 is a four-phase crystalline mixture (inorganic sample) belonging to a set of test mixtures prepared for the Quantitative Phase Analysis (QPA) Round Robin, organized by the International Union of Crystallography (IUCr) Commission on Powder Diffraction (CPD). This sample presents problems due to preferred orientation effects, caused by the presence of Brucite that is characterized by flat hexagonal plates inducing preferred orientation along [00l] direction.

To run QUALX on **Sample 2** to carry out the qualitative phase analysis by a default Search-Match run, inquiring the POW_COD database:

- o Click on QualX icon
- o **File** in the upper Menu
- o **Import Diffraction Data**
- o Select the experimental profile counts file 'Sample_2.dat'
- o **Open**
- o **Click** on **OK** to set the wavelength

The powder diffraction pattern is visualized

- o Select **Search** > **Search Match** in the main Menu

QUALX will automatically perform: a background estimation and subtraction from the experimental diffraction pattern; a peak-search supplying a set of peak positions ($2\theta_p$) and intensity (I_p) values; a search for the candidate database single phase pattern(s) best matching the experimental powder data. At the end of the Search-Match step on **Sample 2**, QUALX provides a list of 172 plausible phases ranked according to a decreasing value of a Figure of Merit (FoM):

No.	QM	CARD	Compound Name	Chemical Formula	Peakpos.	Intensity	Scale	FoM	S-Quant.
1	C	00-900-7060	[Fluorite]	Ca F2	0.88710	0.73952	0.39709	0.77070	4.153
2	C	00-901-1662	[Zincite]	O Zn	0.91456	0.79815	0.49310	0.76172	7.524
3	C	00-230-0112	zinc	O Zn	0.93611	0.75095	0.45667	0.74908	7.022
4	C	00-230-0450	zinc oxide [zincite]	Zn O	0.93954	0.74671	0.45816	0.74891	7.098
5	C	00-230-0449	calcium fluoride [fluorite]	Ca F2	0.87308	0.66660	0.39679	0.73676	4.409
6	C	00-152-2380		Mo Ni	0.74992	0.74645	0.53159	0.71900	12.945
7	C	00-152-3903	(Mg0.1 Mn0.9) Se	Mg0.1 Mn0.9 Se	0.87324	0.62660	0.42633	0.71220	10.974
8	C	00-153-3020	(Zn0.85 Co0.15) O	Co0.15 O Zn0.85	0.70497	0.82520	0.47660	0.67931	6.713
9	C	00-154-1462	Nd.30 Ce.70 O1.85	Ce0.7 Nd0.3 O1.85	0.88839	0.67763	0.43551	0.67414	17.313
10	C	00-154-1409	La1.6 U2.4 O8.81	La1.6 O8.81 U2.4	0.88839	0.65299	0.42554	0.66511	22.621
11	C	00-153-5864	Zr3 O	O Zr3	0.69643	0.51485	0.52683	0.65119	13.302

Matched POW_COD C   172   Selected Card   00-900-7060 Ca F2

The values of ($2\theta_p$, I_p) associated to the experimental peaks are provided on the upper right side of the QUALX window, in the columns 'Exp-2th' and 'Exp-I', respectively. Each time one of the plausible phases (identified by the POW_COD entry number in the 'CARD' column of the results list) is selected by left click of mouse, the 2θ and intensity values of the calculated reflections associated to the crystal phase are provided in the two columns labelled by the 'CARD' number on

the right of the 'Exp-I' column; the calculated reflections, belonging to the selected POW_COD entry and matching the (2θ, *I*) values of the experimental peaks, are highlighted.

To accept the selected phase
- o   Click on the ✓ button on the right side of the QUALX window (the POW_COD entry # 900-7060, concerning Fluorite, is accepted)

As soon as a possible database phase is graphically accepted, its contribution is subtracted from the list of experimental (2θ$_p$, *I*$_p$) values, obtaining an updated set of (2θ, *I*) values. The FoM is calculated again by taking into account the updated (2θ, *I*) values.

- o   Click on ✓ button to accept the next phase in the list: Zincite (POW_COD entry No. 901-1662)
- o   Click on ✓ button to accept the next phase: Corundum (POW_COD entry No. 900-8081)

   The next plausible phase in the list, not already selected, is Brucite (POW_COD entry No. 100-0054)
- o   Click on ✓ button to accept Brucite (POW_COD entry No. 100-0054).

The main experimental peaks are justified by the matched calculated reflections associated to the four identified phases (*i.e.*, Fluorite, Zincite, Corundum, Brucite): the results of the qualitative analysis are correct.

Since the POW_COD entries contain also the information on the Reference Intensity Ratio (RIR) value (*i.e.*, I/Ic), each time a crystal phase is accepted, a semi-quantitative analysis is also performed by QUALX and the weight percentage is given in the last column ('S-Quant').
The reliability of the semi-quantitative analysis strongly decreases with the increasing of the complexity of mixtures and/or, in case of strongly overlapping phases and/or preferred orientation effects and/or presence of amorphous and/or missing phases, etc…

In case of Sample 2, due to the preferred orientation effects, the estimation of the weight percentages ('S-Quant' column) is affected by not negligible errors, especially in case of Brucite:

No.	QM	CARD	Compound Name	Chemical Formula	Peakpos.	Intensity	Scale	FoM	S-Quant.
P.1	C	00-900-7060	[Fluorite]	Ca F2	0.88710	0.73952	0.39709	0.77070	19.2%
P.2	C	00-901-1662	[Zincite]	O Zn	0.91456	0.79815	0.49310	0.77511	13.2%
P.3	C	00-900-8081	[Corundum]	Al2 O3	0.82194	0.72668	0.10336	0.57613	20.8%
P.4	C	00-100-0054	Magnesium hydroxide [Brucite]	Mg (O H)2	0.63790	0.37242	0.56583	0.56565	46.8%

- **NO_DEFAULT** folder

It contains: **smrr-3.dat** [the file containing the experimental profile counts, in case of a two-phase mixture (**smrr-3**), laboratory data, Cu *K*$_a$ radiation]; **smrr-3_True_Phases.docx** [the file providing the true crystalline phases and their true weight percentages (*w*$_T$) of **smrr-3**]; **smrr-3_Round_Robin_QPA.pdf** [the paper in which the outcomes on Qualitative Phase Analysis (QPA), provided in the file **smrr-3_True_Phases.docx**, are given].

smrr-3 is a two-phase crystalline mixture (pharmaceutical sample) belonging to a set of test mixtures prepared for the 'Search/match Round Robin 2002' on Qualitative Phase Analysis.

The sample consists of a mixture of two thalidomide polymorphs [named α and b or III and I] whose true weight fraction (w_T) is 50% for both. The corresponding POW_COD entries are: No. 151-3335 (α) and No. 151-3336 (β).

A default search-match procedure by QUALX is not able to find, in the list of the results, both the thalidomide compounds but only the Thalidomide β (POW_COD entry No. 151-3336). To verify its failure, run QUALX on **smrr-3** to carry out the qualitative phase analysis by a default Search-Match run, inquiring the POW_COD database:
- o Click on QualX icon
- o **File** in the upper Menu
- o **Import Diffraction Data**
- o Select the experimental profile counts file 'smrr-3.dat'
- o **Open**
- o **Click** on **OK** to set the wavelength

The powder diffraction pattern is visualized
- o Select **Search** > **Search Match** in the main Menu

At the end of the Search-Match step, QUALX provides a list of 2902 plausible phases ranked according to a decreasing values of a Figure of Merit (FoM), among which the POW_COD entry No. 151-3336 (Thalidomide β) is second in the list, and the POW_COD entry No. 151-3335 (Thalidomide α) is missing:

| No.	QM	CARD	Compound Name	Chemical Formula	Peakpos.	Intensity	Scale	FoM	S-Quant.
1	C	00-700-3438		C32 H78 B20 K2 N2 Ni O12	0.57691	0.49678	0.54032	0.76786	0.455
2	C	00-151-3336	rac-2-(2,6-Dioxo-3-piperidinyl)-1H-isoindole-1,3(2H)-dione	C13 H10 N2 O4	0.65688	0.61454	0.80621	0.75598	0.563
3	C	00-710-0721		C45 H100 Mg N9 Na	0.56432	0.43887	0.82422	0.75178	0.636
4	C	00-703-9310		C20 H40 Li N4 P	0.53365	0.48197	0.61142	0.74900	0.529
5	C	00-407-8532		C26 H42 Fe P2	0.54020	0.38787	0.58848	0.74865	1.312
6	C	00-722-9236		C24 H45 Mg N O2 Si	0.53478	0.40308	0.50813	0.74677	0.800
7	C	00-406-9299		C60 H64 N8 Yb2	0.55190	0.39213	0.94562	0.74505	3.888
8	C	00-701-0039		C23 H29.5 Ag N6 O8.25	0.54875	0.46769	0.74882	0.74354	1.106
9	C	00-408-1996		C58 H74 F12 Ir2 O2 P4	0.55128	0.48949	0.71075	0.74256	1.793
10	C	00-412-0813		C38 H46 N2	0.55478	0.43549	0.90529	0.74249	0.696
11	C	00-721-5801		C50 H63 N3 O13 S2	0.55425	0.41749	0.44143	0.74206	0.433
12	C	00-406-4241	(C5Me4H)Y(CH2C6H4NMe2)2	C27 H37 N2 Y	0.58122	0.42017	0.53616	0.74181	1.288
13	C	00-703-5354		C29 H22 N6 O3, H2O	0.55657	0.41619	0.66227	0.74140	0.518
14	C	00-222-2520	4-Chloro-<i>N</i>-(2,6-dichlorophenyl)benzamide	C13 H8 Cl3 N O	0.56615	0.44343	0.78212	0.74093	0.545

Matched POW_COD C | 2902   Selected Card   00-700-3438   C32 H78 B20 K2 N2 Ni O12

The absence of the Thalidomide α can be due to one (or more than one) unsatisfied criterion guiding the search-match default step. One of these search-match criteria is related to the check of the strongest lines of a candidate phase. In a default search-match step this check is active, consequently a database crystal phase is considered plausible only if its three strongest lines are matched by experimental peaks. In case of very broad experimental peaks (as it is for smrr-3) it can happen that a correct phase is not considered plausible by QUALX because at least one of its three strongest calculated lines is not matched by the experimental peaks.
This drawback can be avoided by deactivating the check of the strongest lines during the search match step. It can be done by selecting:

- o **Search** > **Search-Match Option** in the main Menu
  To remove this option click on the check button 'Check strongest peaks', as in the following windows:
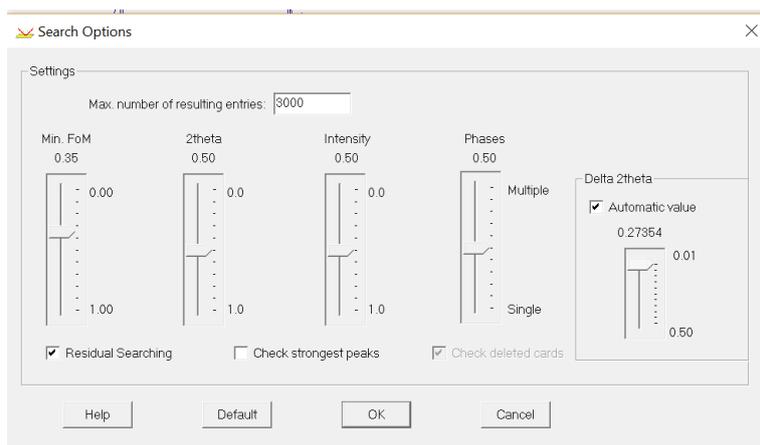
- o **OK**
- o Select **Search** > **Search Match**

The not default search-match is characterized by an increase of the matched entries and, consequently of computing time. At the end of the not default search-match step the results list contains both the Thalidomide α and β (at position n. 4 and n. 1 in the list, respectively)

No.	QM	CARD	Compound Name	Chemical Formula	Peakpos.	Intensity	Scale	FoM	S-Quant.
1	C	00-151-3336	rac-2-(2,6-Dioxo-3-piperidinyl)-1H-isoindole-1,3(2H)-dione	C13 H10 N2 O4	0.65688	0.61454	0.80621	0.75598	0.563
2	C	00-410-7387	qoxCMoP3	C17 H33 Mo N2 P3	0.55653	0.46909	0.76046	0.74995	2.065
3	C	00-202-1076	Bis[2,4-diamino-5-(4-chlorophenyl)-6-ethylpyrimidin-1-ium] naphthalene-1,5-disulfonate	2C12 H14 Cl N4 +, C10 H6 O6 S2 2-	0.56614	0.45584	0.69882	0.74305	0.501
4	C	00-151-3335	Thalidomide	C13 H10 N2 O4	0.57523	0.51178	0.88982	0.74219	0.649
5	C	00-450-5249	diaqua(15-crown-5)iron(II) nitrate	(C10 H24 O7 Fe 2+), 2(N O3 1-)	0.58167	0.26722	0.47757	0.74133	1.102
6	C	00-222-2520	4-Chloro-<i>N</i>-(2,6-dichlorophenyl)benzamide	C13 H8 Cl3 N O	0.56615	0.44343	0.78212	0.74093	0.545
7	C	00-722-4845	[HHTP.TBA.MeOSO3]n	2(C18 H12 O6), 2(C16 H36 N), 2(C H3 S O4)	0.56895	0.42522	0.70206	0.73787	0.587
8	C	00-710-9695		C102 Cl20	0.55411	0.48490	0.40864	0.73758	0.245
9	C	00-201-8924	Bis[(<i>E</i>,<i>E</i>)-3,5-bis[4-(diethylamino)benzylidene]-4-oxopiperidinium] butanedioate	2C27 H36 N3 O + , C4 H4 O4 2-	0.54498	0.39416	0.83205	0.73691	0.789
10	C	00-721-6730		C13 H10 N2 O4 S	0.53187	0.42172	0.82356	0.73685	0.547
11	C	00-151-5232		2(C23 H26 N2 Rh), 2(F6 Sb), C H2 Cl2	0.55311	0.45470	0.46279	0.73573	1.064
12	C	00-710-6509		2(C25 H40 F2 N O4 P), C H4 O	0.55067	0.35656	0.28556	0.73451	0.403
13	C	00-434-5782		C11 H19 Cl N2 P, C F3 O3 S	0.53416	0.45722	0.56938	0.73439	0.634
14	C	00-222-2250	[(<i>E</i>)-2-(2-Chloro-3,3,3-trifluoroprop-1-enyl)-<i>N</i>-(2,4- dimethylphenyl)-3,3-dimethylcyclopr	C17 H19 Cl F3 N O	0.54895	0.40627	0.37180	0.73381	0.379

Matched POW_COD C 2876   Selected Card   00-151-3336 rac-2-(2,6-Dioxo-3-piperidinyl)-1H-isoindole-1,3(2H)-dione   C13 H10 N2 O4

- o Click on ✓ button to accept the Thalidomide β (POW_COD entry No. 151-3336)
- o The next plausible phase in the list is Thalidomide α (POW_COD entry No. 151-3335)

No.	QM	CARD	Compound Name	Chemical Formula	Peakpos.	Intensity	Scale	FoM	S-Quant.
P.1	C	00-151-3336	rac-2-(2,6-Dioxo-3-piperidinyl)-1H-isoindole-1,3(2H)-dione	C13 H10 N2 O4	0.65688	0.61454	0.80621	0.75598	56.7%
P.2	C	00-151-3335	Thalidomide	C13 H10 N2 O4	0.57541	0.68127	0.70831	0.75885	43.3%