New PXRD Software and Hardware Tools for Minerals

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INTRODUCCIÓN

X-Ray Diffraction is a well established method for minerals characterization. Software and hardware tools are constantly being added. In this article a review of recent developments is presented.

LARGE AMOUNTS OF DATA

Researchers frequently face the need of analysing large amounts of X-Ray diffraction data. Sampling campaigns can quickly generate hundreds of diffractograms. Also when many data sets are obtained during a non-ambient experimento on just one sample (at various temperatures, pressures...), quick and effective tools are needed for handling and analyzing this data. Tools such as isolines plot, cluster analysis, and fully automated refinements are available and easy to use in HighScore Plus (Degen et al., 2014).

Visualization

Isolines plot is the preferred visualization option when large amount of datasets have to be presented. Usually 2Theta range is presented in the horizontal axis. Trends, changes in the patterns and phase transitions, can immediately be tracked. Intensity is represented by a color pallet (e.g. Fig. 1)

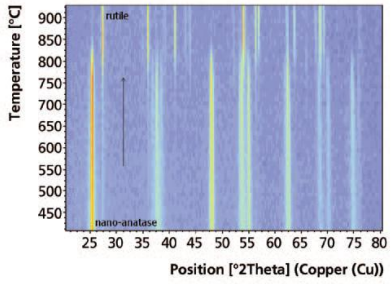


fig 1. *Isolines plot showing the thermal evolution of XRD patters with an increasing intensity from blue (background) to yellow and orange (Bragg Peaks)*

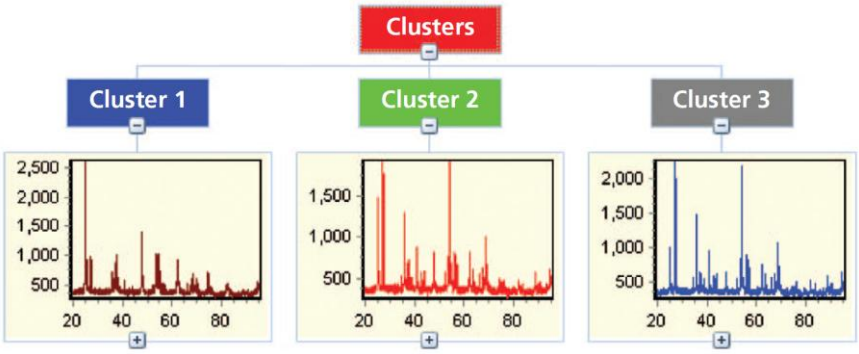


fig 2. *Pattern classification in groups performed with cluster analysis*

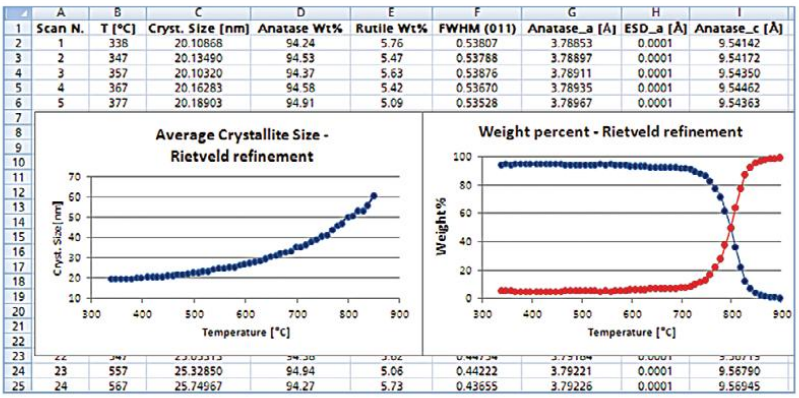
Cluster Analysis

With cluster analysis all collected scans are compared, reduced to probability curves, and similarities and differences are calculated. This allows for a preliminary discrimination of the patterns, dividing them into major groups with similar characteristics (Fig.2). Moreover, the most representative dataset is defined, so manual analysis could be performed only on these flaged datasets, and then using automated routines for the rest.

HighScore also shorts all candidates for phase identification into groups depending on their similarity, using hierarchical agglomerative clustering. The idea is to group together all identical, very similar and isotypical patterns. This provides a better overview on the different candidates.

Automation

A user batch program is a collection of data treatment and analysis steps. It can include reporting and storage of the results. User batches execute exactly the same analysis again and again on different input data, allowing for automation and ensuring fully comparable analytical results (Fig. 3).



	A	B	C	D	E	F	G	H	I
1	Scan N.	T [°C]	Cryst. Size [nm]	Anatase Wt%	Rutile Wt%	FWHM (011)	Anatase_a [Å]	ESD_a [Å]	Anatase_c [Å]
2	1	338	20.10868	94.24	5.76	0.53807	3.78853	0.0001	9.54142
3	2	347	20.13490	94.53	5.47	0.53788	3.78897	0.0001	9.54172
4	3	357	20.10320	94.37	5.63	0.53876	3.78911	0.0001	9.54350
5	4	367	20.16283	94.58	5.42	0.53670	3.78935	0.0001	9.54462
6	5	377	20.18903	94.91	5.09	0.53528	3.78967	0.0001	9.54363
24	23	557	25.32850	94.94	5.06	0.44222	3.79221	0.0001	9.56790
25	24	567	25.74967	94.27	5.73	0.43655	3.79226	0.0001	9.56945

fig 3. *Output of automatic analysis with refined parameters and corresponding plots*

palabras clave: Cluster Analysis, Isolines, Automation, side loading

key words: Cluster Analysis, Isolines, Automation, side loading

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COLLECTING DATA WITHOUT A DIFFRACTOMETER

A new tool is added to the HighScore suite: the Bitmap-to-Scan converter. This tool generates an XRD scan from a graphics file or picture, as in the example of Fig. 4, which shows the original graphic of the X-Ray diffraction pattern of a rust sample. Fig. 5 shows the Rietveld refinement performed on the converted scan.

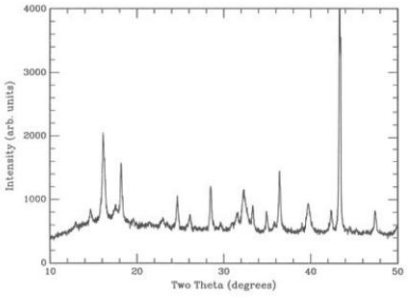

fig 4. *Original graphic of the X-Ray diffraction pattern of a rust sample published in Powder Diffraction 1, 299 (1986).*

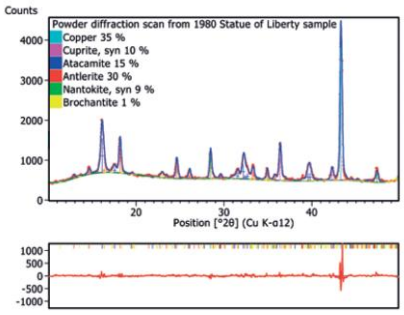

fig 5. *Rietveld refinement performed on the converted scan.*

SAMPLE PREPARATION

Geological samples can often be challenging to present to the diffractometer in a uniform and randomly oriented fashion. Good sample preparation is critical to the results. For example, at the U.S. Geological Survey in Denver, CO (USA) nearly all samples are prepared for XRD using the classic side loading or side-drifting method. This technique has been around for many years, used by National Institute for Science and Technology (NIST) for the preparation of standard reference patterns

Other materials well suited to this sample preparation method include coal, clays, micas, feldspars, pyroxenes, amphiboles, carbonates, and gypsum. E.g. high organic content coal (90%) is difficult to pack, tends to heave out of or settle in a back loading holder, but is well suited for preparation by side loading. Many pulverized materials with rodo r flake shaped crystals/particles due to cleavage, can also benefit from this sample preparation method.

fig 6. *Example of side loading holders*

New sample preparation holders (Fig. 6) have been added for quick and easy preparation of samples with side loading technique.

COMPACT DIFFRACTOMETERS

Technology developments allow manufactures to convert typical floor-standing size of X-Ray diffractometers to more compact instruments, able to settle on a bench. Fig. 7 shows an example of the dimensions of a recently presented compact diffractometer, which equiped with a high performance X-Ray linear or area detector is able to collect data of quality as presented in Fig. 8, in twenty minutes measuring time. This is, no difference in acquisition time with large floor-standing systems, and with much less cost of ownership.

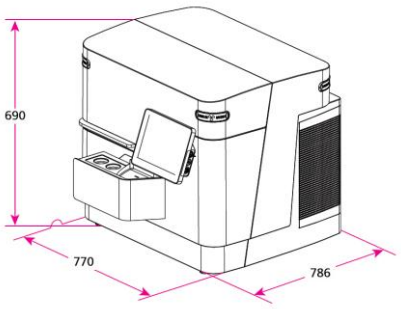

fig 7. *Compact diffractometer dimensions*

CONCLUSIONS

New software and hardware developments improve the sample preparation and data acquisition, analysis and presentation tools available.
Advances has been shown with focus of the efficient processing large amounts of diffraction data.

REFERENCES

Degen, T., Sadki, M., Bron, E., König, U., Nénert, G. (2014): The HighScore suite. Powder Diffr., Vol. 29, No S2.

Jenkins, Ron, and Snyder, Robert L., (1996), Introduction to X-Ray Powder Diffractometry, Volume 138 in Chemical Analysis, 1996,John Wiley & Sons, Inc., New York, NY, pages 247-249

Victor E., Jenkins, Ron, and Smith, Deane K., edited by Buhrke, 1998, John Wiley & Sons, Inc., New York, NY, page 148

König, U, and Gobbo, L (2009): Modern X-ray diffraction techniques as a fast industrial analysis method for iron ores-from exploration to process control. Australian Institute of Mining and Metallurgy Publication Series (No 7): 121–128.

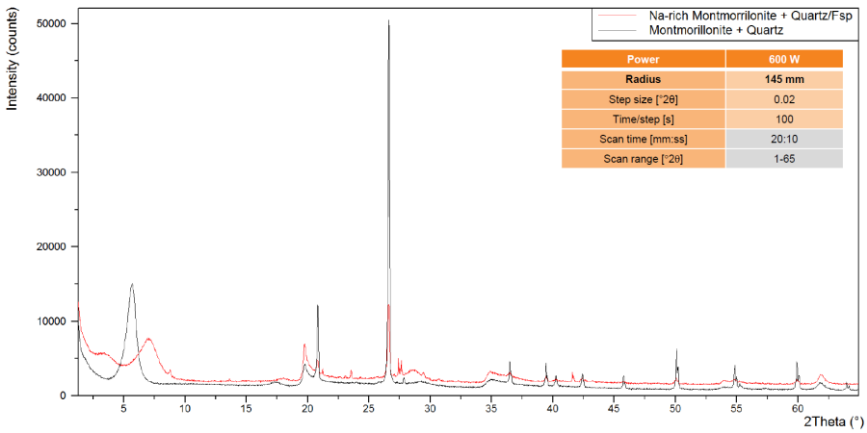

fig 8. *Clay mineral scans obtained with compact diffractometer in 20 minutes measuring time.*