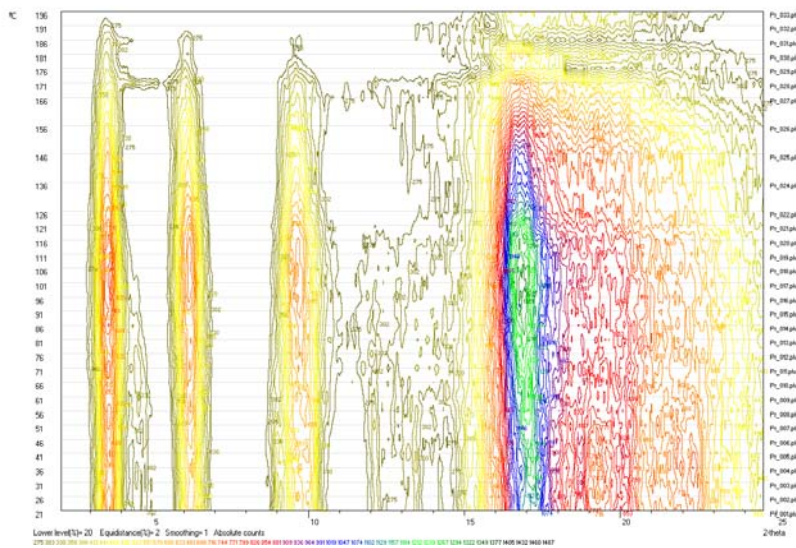
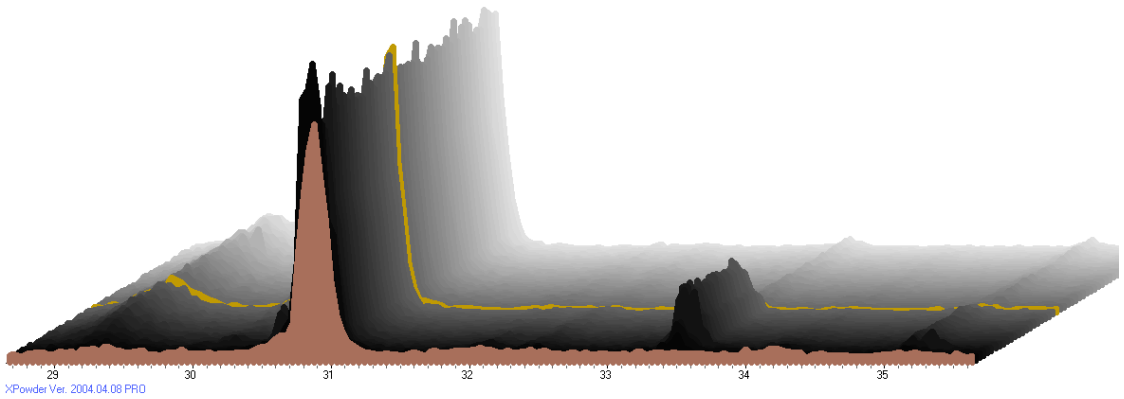
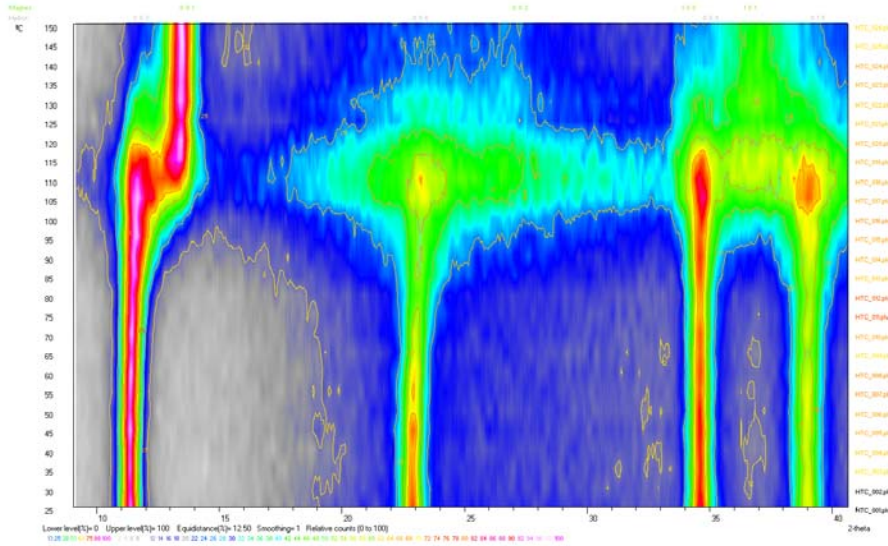


Mapping and thermodiffraction with X Powder

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Mapping and thermodiffraction with XPowder

<http://www.xpowder.com>

Next page

New routines for 2D representation (mapping) have been developed to evaluate systematic evolutions of physicochemical processes sensitive to monitoring by X-ray diffraction, such as cementations, solid-state chemical reactions, regional mineral crystallinity in geologic studies, etc. (see examples in the figures below).

The only requirement to exploit the potential of this application is that diffraction data must have been collected with identical λ , $\Delta\theta$, initial 2θ and final 2θ . In order to minimize instrumental errors it is advisable to collect the data as correlative scans although it is not a constraint. X powder can load as many as 50 diffractograms.

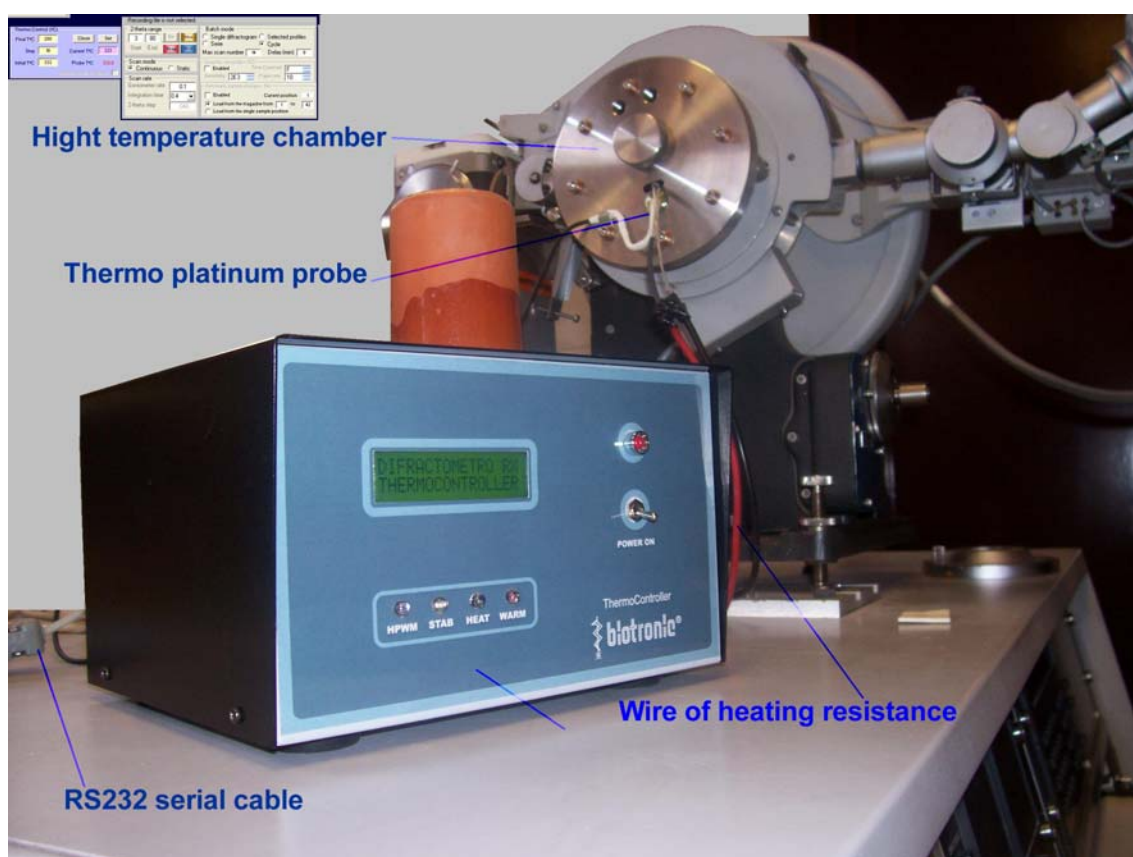
The transformations linked to changes in temperature are interesting processes than can be represented in 2D maps. XPowder reads the temperature directly from the PLV format (version 3) data files. In case of the data formats are not PLV, the graphical representations 2D and 3D uses fictitious and correlative values of this parameter (For example 1, 2, 3, etc.). An alternative is to acquire the data via XPowder PLUS from a PC connected to a diffractometer with temperature control (to see below).

However a simpler solution consists of changing the data to PLV format (from the main menu 'Main> File> Save as...') The PLV format is ANSI code and hence it can be edited with 'Notepad' program. The temperature of the registry can be introduced later on in any of the free lines of the data file (free lines are shown as 'Line' plus a number, for example, "Line32" in the example below) and XPowder will take this value of temperatures in the calculations (for example to obtain the thermal expansion tensor on automatic form). The example below shows a data file in PLV format where the temperature is in line 35:

```
XPowder diffraction software. PLV file           Line 28
format Ver. 3.0                                 Line 29
Sample= Vin.PLV                                  Line 30
Site= Belmez (Spain)                            Line 31
User= Clinker SA                                 Line 32
Date= 24/04/2006                                 Line 33
Time= 47109                                       Line 34
Start 2-theta scan= 3.000                        Temperature (°C)= 40
End 2-theta scan= 60.000                         Line 36
Step size= 0.040                                  Line 37
Scan mode= Continuous                             Line 38
Integration time(sec)= 0.4                        Line 39
Anode= Cu                                          Line 40
Filter= None                                       Line 41
Monochromator= Graphite 2°                       Line 42
K-Alpha 1= 1.540598                              Line 43
K-Alpha 2= 1.54433                               Line 44
Ka2/Ka1 Ratio= 0.5                               Line 45
K-Beta= 1.5406                                    Line 46
Automatic sampler changer= No                    Line 47
Single Gobel mirror= NO                          Line 48
Divergence slit= AUTOMATIC                       Line 49
Receiving slit= 1/0.1/1                          Data
Generator voltage(Kv)= 40.00                     3.000      51
Tube current(mA)= 40.00                          3.040      47
Maximun counts= 2843                             3.080      22
Line 25                                           .....
Spinner = No                                       .....
Line 27
```

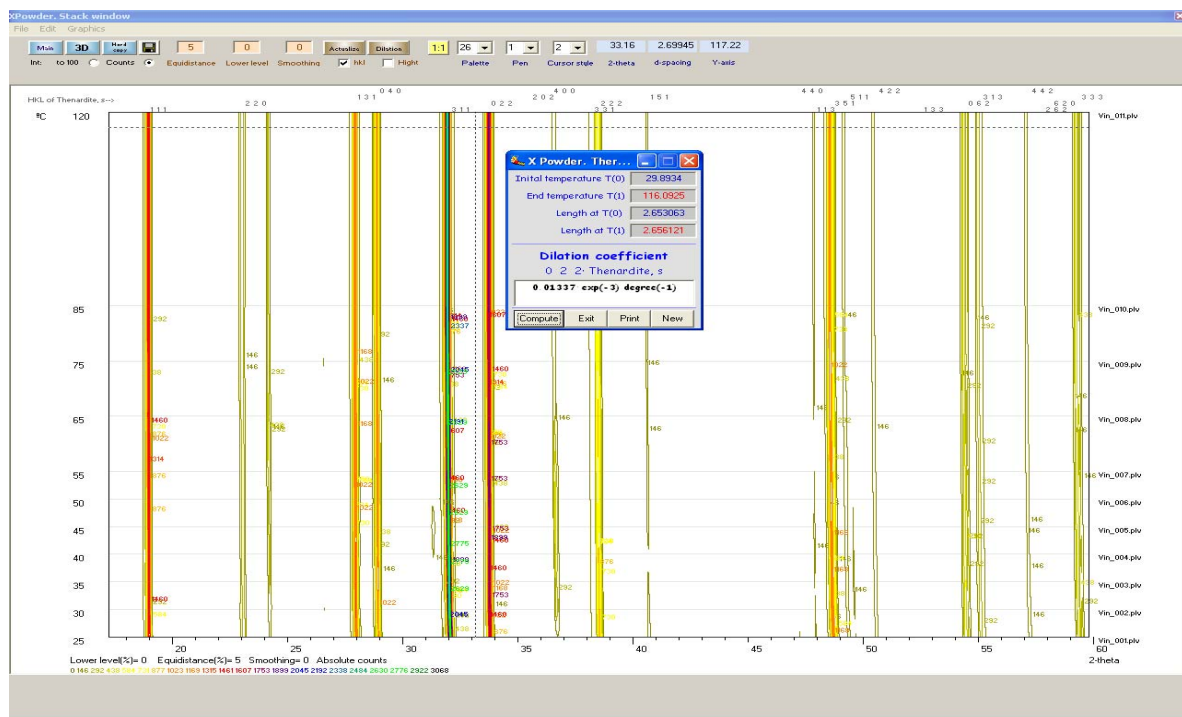
XPowder PLUS can be used for full-duplex control of the X-ray diffractometer Pw1710/00 with a PW1712 communication card via RS232 serial port. Additionally, we have developed an automatic temperature control loop consisting of a precision platinum probe, a specific temperature standard devices with auto-tuning for heating resistance checking and a electronic circuit for remote control via RS232 serial port (see figure). No modifications of the diffractometer are required. An upper limit of 200°C has been imposed to protect the X-ray device from damages. This upper limit is reasonable and suitable for many crystals (hydrated minerals, organics compounds, etc) that show detectable processes under 200°C such as dehydration, first dehydroxylations, phase transitions, crystallinity changes, thermal expansion, ionic selfdiffusion, etc. The program monitorises initial and upper temperature limits temperature, temperature step and delay between sequent scans.

The complete device (control unit, heating chamber, cables and 2nd PCI communication card) is delivered within a month and the purchase price is € 7000. To enable the thermal control from XPowder Plus, insert 'ThermoController= Comm3:9600,N,8,1', or the appropriate communication parameters in the 'Default.cnf' file. For further enquiries contact support@xpowder.com.



PRICE €7000.00

[Next Page: Dilation coefficient](#)



Once the qualitative composition is known and the hkl box is checked, by pressing the [Alt] key and clicking the mouse left button about the centre of the trace of hkl reflections at two different temperatures in the 2D map, the dilation coefficient in the hkl reciprocal direction can be calculated by [Compute] button. The results are saved in the log.txt file (Main-> File-> Save Log File as TXT).

Data output for dilation analysis

```

2 2 0· Thenardite, s
Initial temperature 29.64245
Final temperature 119.3547
Initial d-spacing 3.852078
Final d-spacing 3.868141
Dilation coefficient 0.04648·exp(-3)
degree(-1)

1 3 1· Thenardite, s
Initial temperature 29.8934
Final temperature 119.3547
Initial d-spacing 3.190553
Final d-spacing 3.195997
Dilation coefficient 0.01907·exp(-3)
degree(-1)

0 4 0· Thenardite, s
Initial temperature 29.8934
Final temperature 119.4802
Initial d-spacing 3.087255
Final d-spacing 3.09234
Dilation coefficient 0.01839·exp(-3)
degree(-1)

3 1 1· Thenardite, s
Initial temperature 29.8934
Final temperature 119.1038
Initial d-spacing 2.792854
Final d-spacing 2.803897
Dilation coefficient 0.04432·exp(-3)
degree(-1)

0 2 2· Thenardite, s
Initial temperature 29.8934
Final temperature 119.6057
Initial d-spacing 2.652177
Final d-spacing 2.654648
Dilation coefficient 0.01039·exp(-3)
degree(-1)

4 0 0· Thenardite, s
Initial temperature 30.01887
Final temperature 119.1038
Initial d-spacing 2.447279
Final d-spacing 2.460767

```

```

Dilation coefficient 0.06187·exp(-3)
degree(-1)

1 5 1· Thenardite, s
Initial temperature 30.26981
Final temperature 93.50755
Initial d-spacing 2.215348
Final d-spacing 2.220184
Dilation coefficient 0.03452·exp(-3)
degree(-1)

3 5 1· Thenardite, s
Initial temperature 29.8934
Final temperature 119.4802
Initial d-spacing 1.869949
Final d-spacing 1.874974
Dilation coefficient 0.03000·exp(-3)
degree(-1)

4 2 2· Thenardite, s
Initial temperature 29.76793
Final temperature 119.6057
Initial d-spacing 1.803361
Final d-spacing 1.810327
Dilation coefficient 0.04300·exp(-3)
degree(-1)

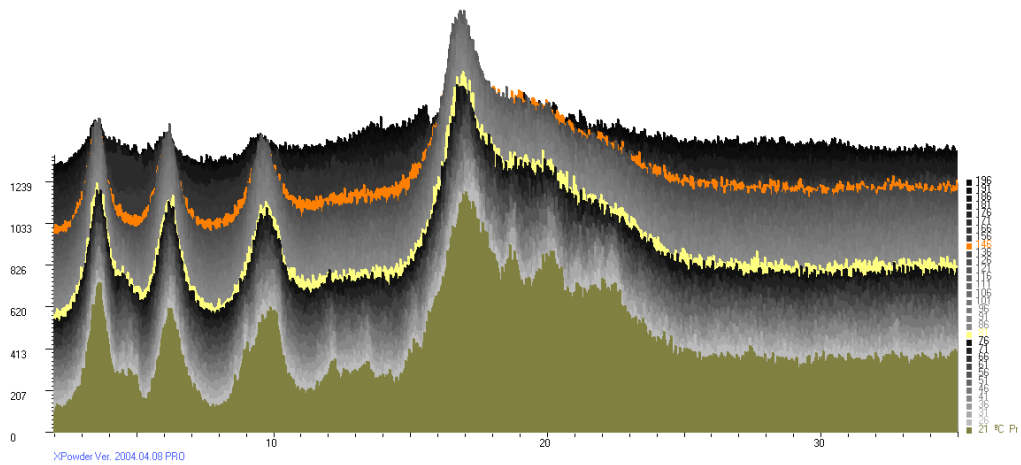
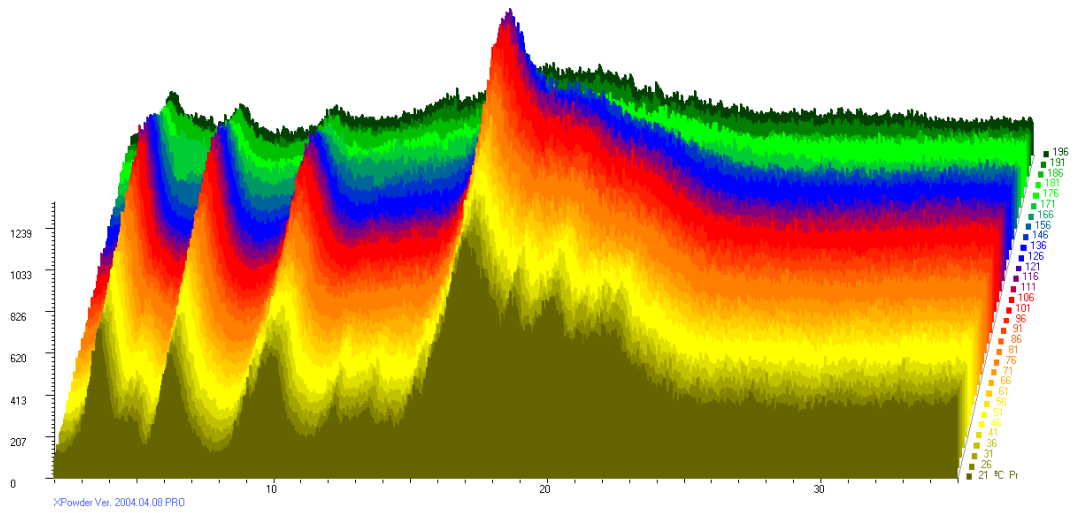
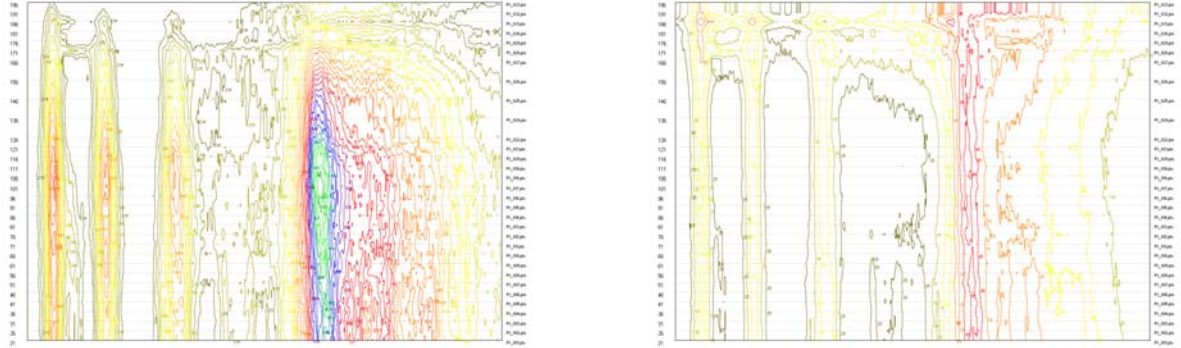
4 4 2· Thenardite, s
Initial temperature 30.01887
Final temperature 119.4802
Initial d-spacing 1.608248
Final d-spacing 1.614224
Dilation coefficient 0.04154·exp(-3)
degree(-1)

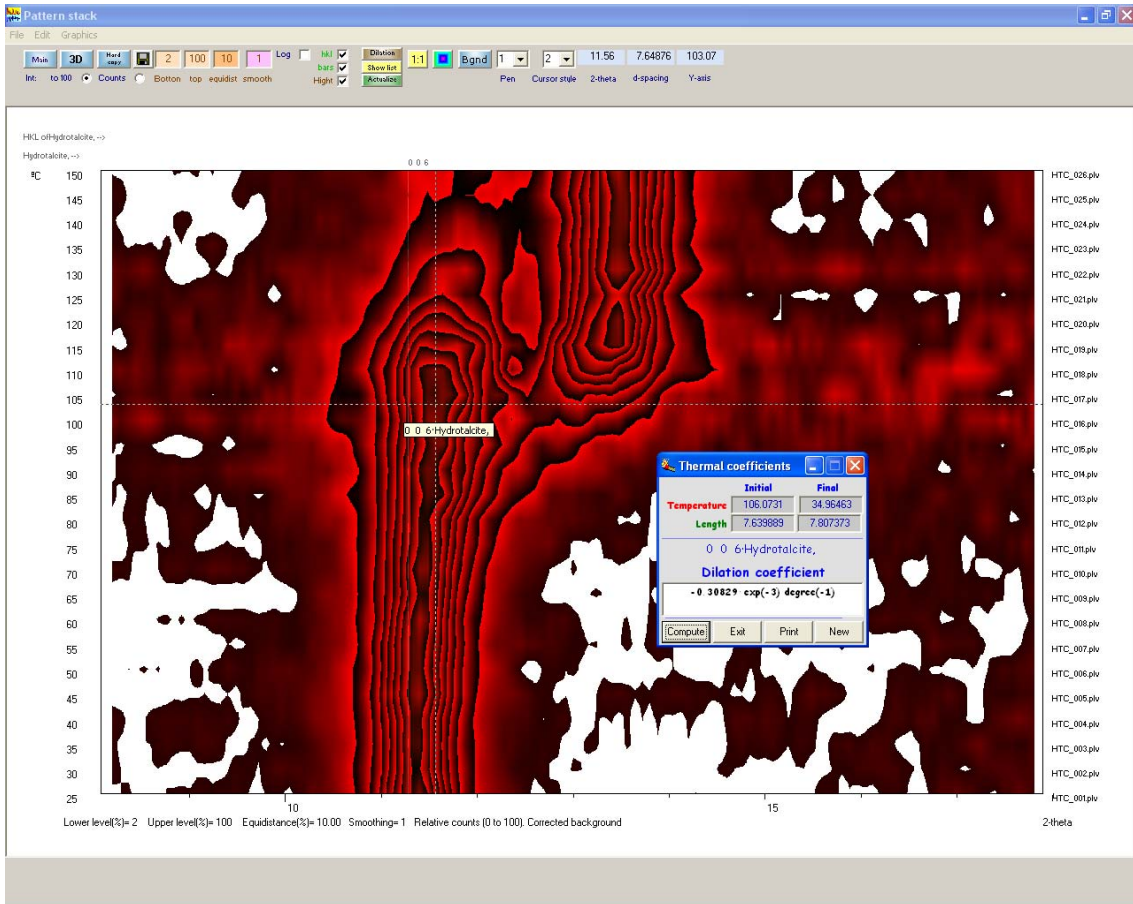
3 3 3· Thenardite, s
Initial temperature 29.76793
Final temperature 119.1038
Initial d-spacing 1.555015
Final d-spacing 1.559434
Dilation coefficient 0.03181·exp(-3)
degree(-1)
-- End thermal dilation coefficient --

```

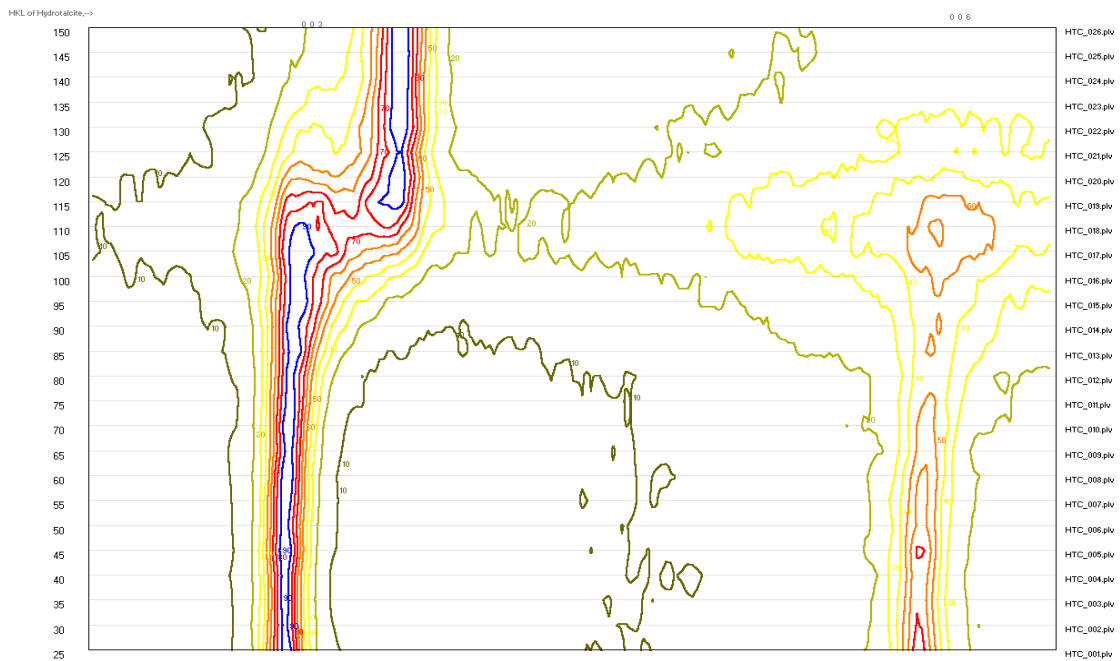
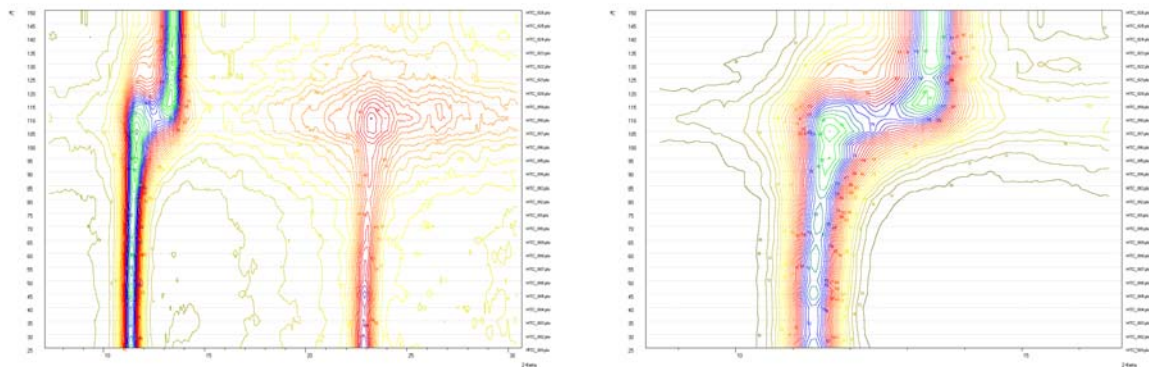
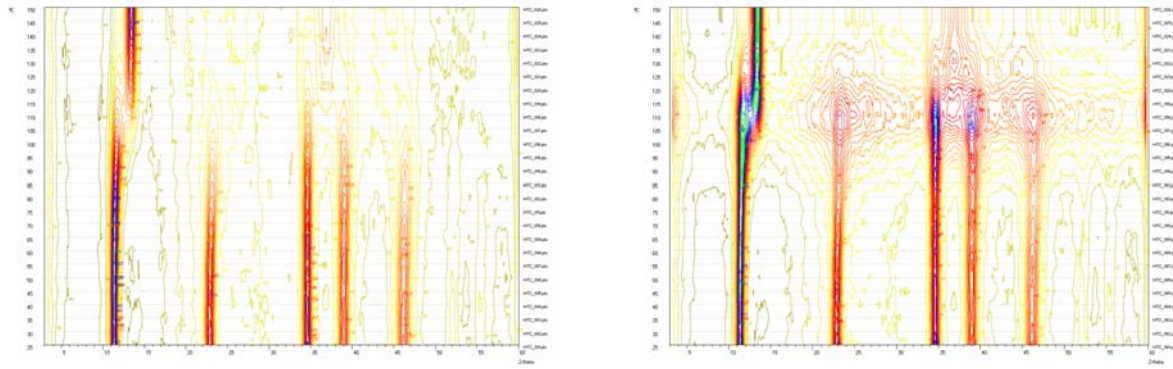
Next page: Phase transition

2D and 3D thermodiffratogram of a low crystallinity organic compound showing a phase transition at 81 °C and melting point close to 160 °C. A) 2D map of absolute counts. B) Idem with relative counts. C) 3D images of diffractograms D) 3D images showing phase transition (yellow) and melting point (orange).

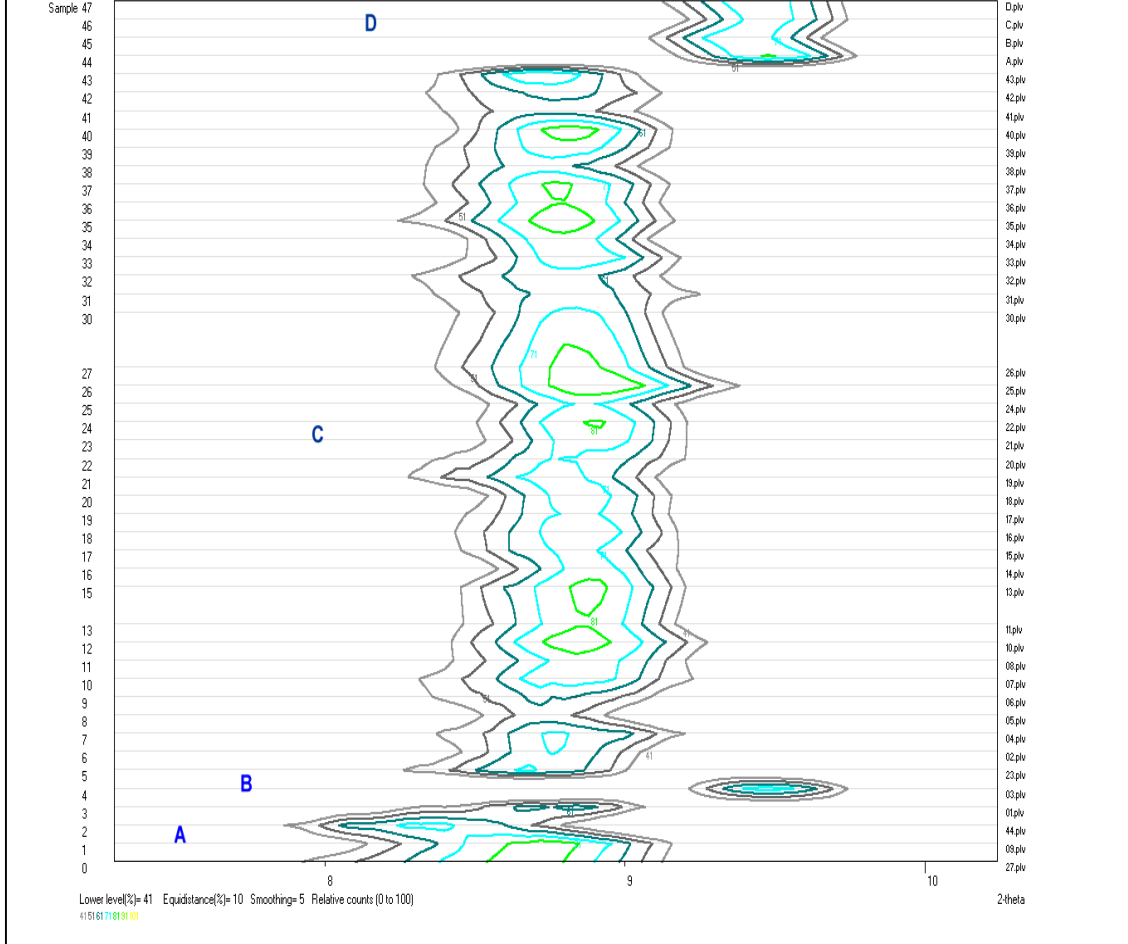




The 2D maps show a phase transition by partial dehydration of hydrotalcite at temperature close to 115 °C. A) Absolute counts. B) Relative counts. C) and D) Details of the map for $10 > 2\theta < 20^\circ$. E) See the degradation and crystallinity reduction from 80°C to the 115 °C, close to phase transition.

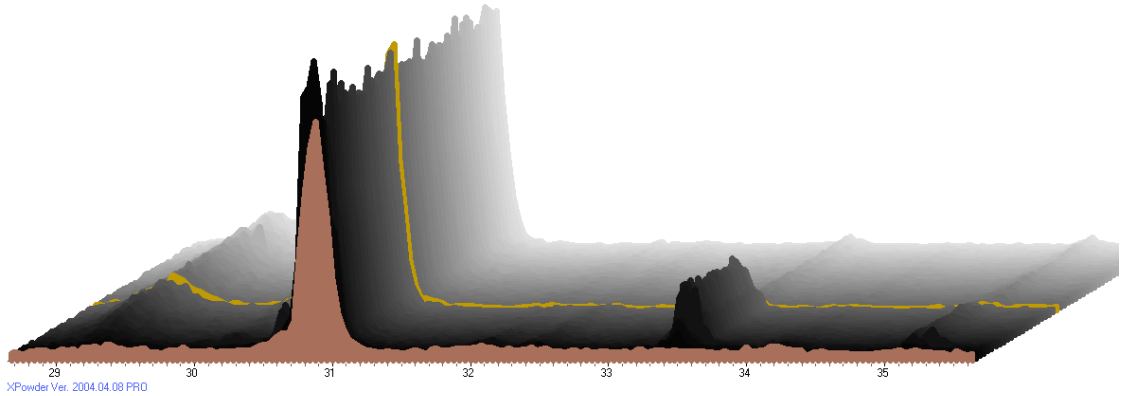


Mapping of 002 reflections of illite ($7 > 2\theta < 10^\circ$). UZone AU: Pelitic sediments. UZone BU: Sands with detritic micas. UZone CU: Phyllites of low metamorphic grade. UZone DU: Micaschists with garnet. The reflections are sharpened in the high-crystallinity zones (B and D), and broadened in low crystallinity zone (A, sedimentary clays). The wide zone C corresponding to a intermediate crystallinity zone.

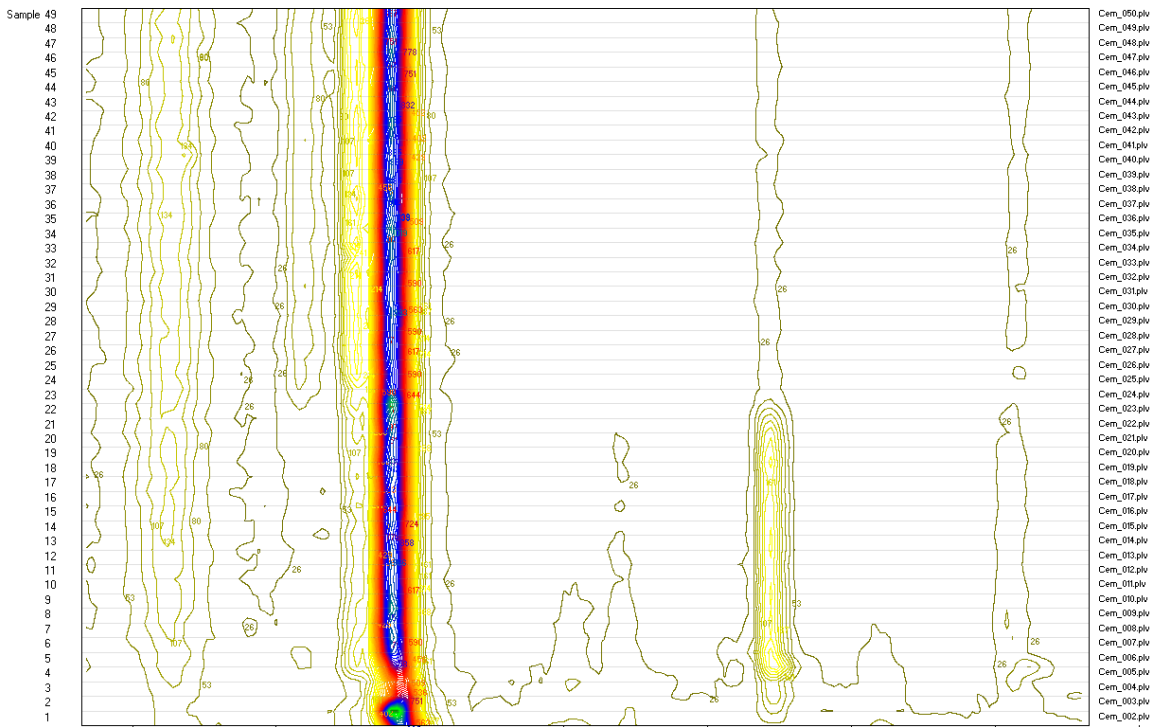


[Next page: Cementation](#)

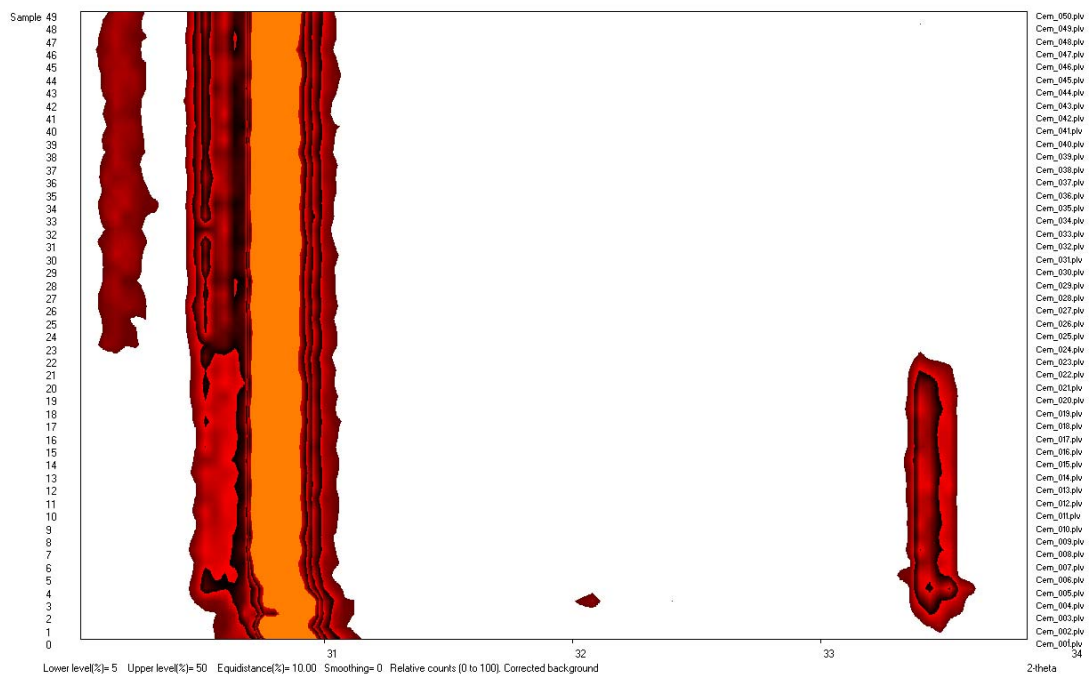
Crystallization of an artificial cement with Calcium silicate $\text{CaB}_{3\text{B}}\text{SiOB}_{5\text{B}}$ 5%, Dolomite $(\text{COB}_{3\text{B}})\text{E}_{2\text{B}}\text{CaMg}$ 88% and indeterminate amorphous components (7%) . Delay time= 60 minutes. 3D image shows in colours the initial stage and the main changes. Notice the agreement between 3D and 2D figures.



XPowder Ver. 2004.04.08 PRO



Cem_050.plv
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[TopT](#)