

OrientExpress

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Goals of OrientExpress

The program **OrientExpress** makes it possible to orient a single crystal of known unit cell dimensions from a single Laue pattern. This pattern can be registered on a flat or cylindrical detector : X Rays film, two-dimensional sensitive detector, bitmap picture. The program was succesfully applied to X rays (normal wavelength and hard X rays) and neutrons experiments, as well as to every sort (mineral, organic and protein) of crystals.

Given a single Laue pattern taken from a crystal of unknown orientation attached to a sample holder, the problem is first, to find the sample orientation (**Orientation Matrix**) and second to compute the rotation angles which will set the crystal to any desired orientation. To do that the program requires (**Input Data**) the following :

- The crystal system and the lattice mode.
- The cell parameters
- The detector to sample distance D and the δ angle between the normal to the detector and the direct beam.
- The wavelength range
- The dimensions of the detector
- The coordinates on the detector plane of a small number of diffraction spots selected by the user on the basis of simple criteria.

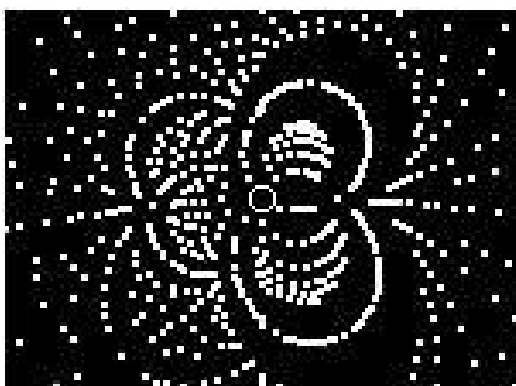
The Laue pattern indexing functionality

The method used, based on the indexing of a small set of selected reflections, proposes one or a small number of solutions. The program computes and displays the corresponding simulated Laue patterns or set of patterns. The « right » solution is unambiguously and easily obtained throuh the visual comparison of the experimental pattern with the set of simulated ones. Once the correct solution is found, the program makes it possible to compute the values of the rotations which, applied to the sample holder axes, will set the crystal to any desired new orientation. The simulated Laue diagram corresponding to the latter can be displayed.

The Laue pattern simulation functionality

When used independently, the Laue pattern simulation function of **OrientExpress** may also be useful. For example, when the data are insufficiently accurate the indexing procedure is generally unusuable. Nevertheless the sample orientation may be found with the help of the simulation of Laue pattern. Starting from an estimated orientation, it is possible to generate a continuous rotation of the crystal (and of the diagram) until the simulated diagram looks like the experimental one. This operation can be very accurate if the experimental diagram is a bitmap picture, because the calculated diffraction spots are superimposed on the picture.

OrientExpress Version 3.4



-This is a « beta » version for Windows95 or Windows NT system. Minimum Pentium 133MHz recommended.

-Picture images of experimental Laue diagrams obtained by scanner and stored under bitmap files (*.bmp) can be processed. In that case, the coordinates of the selected reflections can be measured in line. Then, when the orientation matrix is determined, the simulated diagram can be superimposed on the bitmap image on the screen.

-In line coordinates measurement with digitising tablet (Kontron Tablet).

-The user can simulate a rotation of the crystal leading a synchronous rotation of the simulated pattern on the screen, by continuously moving the mouse. This possibility makes it possible to orient a crystal from a bad experimental pattern when the user knows an approximative setting of the crystal.

Main menu

Data :

- File** : Load an existing data file (.dat)
- Keyboard** : Load the data by the keyboard
- Save** : Save the data file
- Edit** : Edit data or coordinates file with NotePad

Coordinates :

- File** : Load an existing coordinates file (.coo)
- Keyboard** : Load the coordinates by keyboard.
- Image** : Load a Laue image (*.bmp, *.jpg)
- Digitiser** : Acquisition of the coordinates by digitizing tablet (not in V3.2)
- Modification** : Modify either the Coordinate List or the Image Constants

Indexation Search :

- Search** : Indexes the loaded coordinates.
- Display solutions** : Display the list of solutions found by indexation.
- Choose a solution** : Load one of the found solutions.

Matrix :

- Acquisition** : Acquisition of the orientation matrix if it is known.
- Display** : Display the current orientation matrix.
- Refinement** : Refinement of the orientation matrix when determined by

Indexation.

- Rotation** : Discreet rotation of the sample around the experimental axes.
- Restoration** : Back to the original matrix after rotation.
- Print** : Printing of the current orientation matrix.

Simulation :

- L**ae : Simulation of a Laue pattern.
- S**tereo : Simulation of a stereo pattern.

Reorientation :

Calculation of angles to set the crystal to a desired orientation.

About :

Jean Laughier, B. Bochu and A. Filhol (Alan Hewat updated Help)

Data

See also **Input Data example**

The program reads the required data from two files prepared by means of a text editor or interactively from the Keyboard:

-Data file with the suffix «.dat». The contains of its lines must be sucessively:

- 1-A title
- 2-The keyword of the crystal system (CUBI, TETR, ORTH, HEXA, RHOM, MONO, TRIC)
- 3-The keyword of the lattice mode (Bravais lattices modes : P, I, F, A, B, C, plus : R (Hexagonal system, Rhomboedral lattice mode), D (diamond))
- 4-The symbol of the **Space group** (if it is unknown leave blank or P-1)
- 5-The cell parameters (a, b, c in Å, alpha, beta, gamma in degrees)
- 6-The crystal to detector distance D (in Cm) and the angle δ of the beam with the perpendicular to the detector (in degrees)
- 7-The wavelength range (mini, maxi) in Å
- 8-The dimensions of the detector (width, height) in Cm
- 9-FLAT for a flat detector or CYL for a cylindrical detector.

Note: The lattice mode and space group symbols are taken into account only for pattern simulation and are not essential

-Coordinates file *.coo. It contains:

- 1-The list of the coordinates (in Cm) of the N reflexions ($3 < N < 11$) measured with respect to the detector reference frame (Fd) (**Orientation matrix definition**), one pair of coordinates per line. The input stops at the end of file or if a pair 0,0 is found.
- 2-(Optional) A list of the coordinates (in Cm) of an extra set of M reflexions ($M < 30$) which will be used by the orientation matrix procedure only, one pair of coordinates per line. The input automatically stops at the end of file or if a pair 0,0 is found.

Space group

.

The Space group is taken into account only for the simulation generation. The program uses the file **Orientexpress.grp** containing the list of the space groups owning extinction for the general position and the corresponding rules under coded form. This list is not exhaustive. For a given group only one setting is present. This file can be easily implemented by adding other settings. Note that the keywords have capital characters and that the lattice symbol is missing. The extinction rules code is similar to these used by the Enraf-Nonius CAD4 software.

Input data example

-Data file (examp2.dat):

Triclinic crystal. Transmission Laue	(Title)
TRIC	(Crystal System keyword)
P	(Lattice mode keyword)
P-1	(Space group symbol)
9.010 12.890 18.180 121.80 90.58 97.30	(Cell parameters)
4.50 0.000	(Distance in Cm, angle in °)
0.4000 2.0000	(wavelength range in Å)
17.00 13.00	(detector dimensions in Cm)
FLAT	(geometry : FLAT or CYLN)

Coordinates file (examp2.coo):

-3.59 -30	(Coordinates of the N=6 measured reflections)
2.44 -03	
-3.96 2.61	
-2.69 3.40	
-.39 2.62	
1.62 3.80	
0 0	(End of sequence)
3.08 3.04	(Optional: Coordinates of 4 additional reflections for refinement)
-.94 -2.25	
.44 -4.31	
2.73 -4.39	
0 0	

Coordinates

To prevent the failure of the indexing procedure and to obtain an adequate orientation precision, the spot coordinates must be accurately measured. Since the coordinates are measured with respect to the reference frame (Rf), the origin of the latter must be also known accurately.

Measurement of an X Ray film.

The reading may be done with the aid of a light box and a transparent millimetric ruled sheet superimposed on the Laue pattern.

Measurement of a digital image.

The image must be stored as a *.bmp or *.jpg file. The option **Coordinates** of the main menu allows to load the image, determine the scale and locate the origin of the pattern. See **Bitmap picture menu** .

The scale (Nb of pixels/Cm) may be

1-Entered directly if it is known.

2-Measured by clicking two points of known distance. The image included in the

file **examp1.bmp** supplied with the program was obtained by the superimposition of a Laue pattern on a transparent horizontal ruler.

The origin is defined as the centre of the Laue pattern. It can be determined by:

1-Transmission geometry ($\delta=0^\circ$) : The user marks the centre of the direct beam on the image (while the pattern is being recorded, he briefly removes the beam stop to mark the film with the direct beam).

2-Backscattering geometry ($\delta=180^\circ$) : The origin position may be located from the cast shadow of an opaque circular part placed on the film plate.

3-Or the user drags a circle to define the origin, then clicks the mouse button.

After the scale and the origin are determined, the user clicks on up to 10 Laue spots (see **Bragg reflection indexing process**) and saves this set of measured coordinates to a coordinates file *.coo.

Note: A magnified image of the pointer area appears, and the user must point precisely at the diffraction spot.

Measurement with a digitising tablet

Note for the use of the digitiser DIGIKON :

Use the « F » key of the cursor box to end the measures, « D » key to delete the current point and « 1 » key to enter a measure.

For the coordinates measurement using a digitiser tablet you must :

1-First configure the connection port. This configuration is saved in the file named « Orientexpress.cfg » and will load automatically.

2-Determine the origin of the Laue diagram : Click on one or several points around the origin : One point if the beam path is visible, or more if the image has a shadow on the origin. The origin will be taken as the centroid of the measured points.

3-Click on the selected reflections (10 maximum. 5 to 6 recommended).

This set of measured coordinates will be saved in a coordinates file (.coo).

Indexing process

See also **Orientation Matrix definition**

Whatever the instrument geometry, the Laue pattern processing requires the accurate knowledge of the location of the origin O of the reference frame (Rf) attached to the detector.

The basic data set of the indexing process is N spots ($3 < N < 11$) **with small Miller indices**, selected by the user from the observed Laue pattern. The coordinates X_f , Y_f of the latter are measured relative to the reference frame (Rf). Reflections with small Miller indices are recognizable in that they lie at the crossing of **zone lines**, i.e. of lines going through Bragg spots corresponding to families of lattice planes which have an axis in common, the so-called **zone axis**. The latter lines are ellipses if $\delta=0^\circ$ (transmission geometry), hyperbolas if $\delta=180^\circ$ (backscattering geometry) and parabolas if $\delta=90^\circ$.

Among the above set of N spots, two are further selected to be named « fundamental spots » and whose Miller indices (h,k,l) must satisfy the following relationships :

$$\begin{aligned} H_{\min} &\leq h \leq H_{\max} \\ K_{\min} &\leq k \leq K_{\max} \\ L_{\min} &\leq l \leq L_{\max} \end{aligned}$$

Where H_{min} , K_{min} , L_{min} , H_{max} , K_{max} , L_{max} are user defined bounds of the part of reciprocal space where the end of the diffraction vectors are assumed to lie. From the coordinates X_{fi} , Y_{fi} and X_{fj} , Y_{fj} of the fundamental reflections i and j , the program then computes the scattering vector coordinates X_{3i} , Y_{3i} , Z_{3i} with respect to the reference frame (R_3) and the angle μ between them. The algorithm of the program is inspired by that of Riquet and Bonnet (1979, J.Appl.Cryst., 12, 39-41) :

1-The angle μ is compared with the angles ν_{nm} of all pairs n, m of reciprocal lattice vectors which satisfy the upper relationships. When $\mu = \nu_{nm}$ to within ε_a (ε_a being a tolerance level fixed in the program), it is assumed that a likely indexing of the two fundamental spots is obtained i.e. that their coordinates (X_1, Y_1, Z_1) with respect to the reciprocal axes have been found.

2-The equation $\mathbf{X1} = [\mathbf{R13}].\mathbf{X3}$ applied to the two scattering vectors of the fundamental spots and their vector product, gives an approximate orientation matrix $[\mathbf{R13}]$.

3-If the Miller indices, computed from $[\mathbf{R13}]$, of the remaining $N-2$ selected spots are integer numbers to within a tolerance ε_b the corresponding solution is retained and stored in memory.

4-If no solution has been retained once all the angular comparisons have been performed, the user is invited to either widen the intervals $[H_{mi}, H_{max}]$, $[K_{min}, K_{max}]$, $[L_{min}, L_{max}]$, or to select a new pair of fundamental reflections.

5-Going through all the possibilities normally leads to one or a small number of solutions.

The tolerances ε_a and ε_b allow for the experimental errors; They are set to 2° and 0.1 respectively in the program, values which have been found to provide a reasonable number of proposed solutions for usual experimental conditions. Smaller values would reject too many solutions (and perhaps even the right one) ; larger values would imply an unacceptably low experimental accuracy.

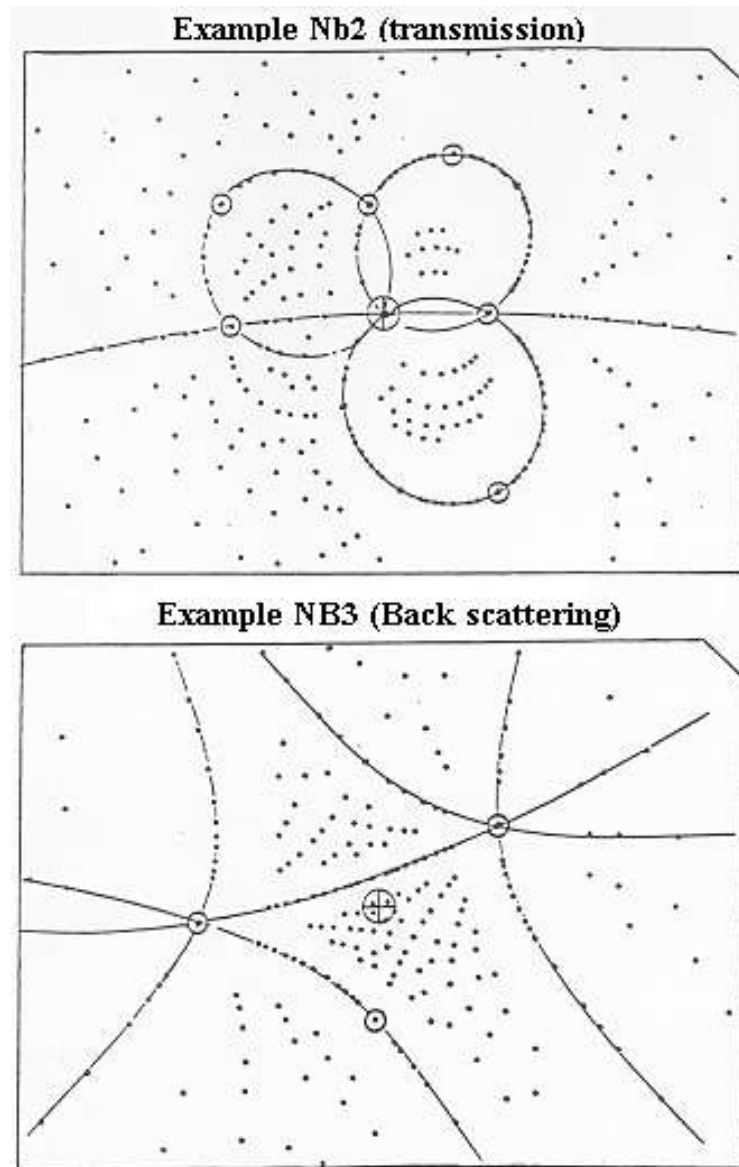
See **Selection of the right solution**

Selection of the right solution

Because of the tolerance levels ε_a and ε_b (**Bragg reflections indexing process**) multiple solutions are often proposed by the indexing algorithm. The version 3.2 selects the most likely solution (the one which has the smallest mean Miller indices). But the best selection is done by the visual comparison of the observed Laue pattern with the simulated ones corresponding to each computed solution. This comparison is particularly easy when the diagram is a bitmap image because the simulated Laue pattern is superimposed on the image. The program is also able to output the simulated patterns at scale 1 to the Windows printer. The direct matching of the observed pattern onto hard copies of the simulated ones provides a much higher accuracy which, generally, will eliminate ambiguity.

Zone lines

Lines going through Bragg spots corresponding to families of lattice planes which have an axis in common, the so-called *zone axis*. The latter lines are ellipses if $\delta = 0^\circ$ (transmission geometry), hyperbolas if $\delta = 180^\circ$ (backscattering geometry) and parabolas if $\delta = 90^\circ$.

Examples of zone lines :

Simulation menu

Picture Option:

-File: To superimpose the picture (*.bmp file) obtained by scanner on the image of the simulation. You have first to determine the scale of the picture and the center of the observed Laue diagram.

-Save: to save the simulated and observed image in a new bitmap (*.bmp) file to be treated by an appropriated software (brush for example).

Print Option:

To print the drawing on a Window printer or edit (or printing) the list of Miller indices and coordinates of the computed Laue diagram.

Options Option:

-RotationYZ: To simulate the rotation of the crystal by mouse motion. In this case, the crystal (and the simulation) can turn around horizontal (Y) and vertical axis (Z) of the plane of the image. Move the mouse while pressing a button. With this option, it is possible to determine the orientation of a crystal by starting from a supposed close orientation and moving the mouse until the simulated diagram becomes similar to the experimental one. If you displayed a scanner picture, the superposition will prove the validity of the solution.

Carefull! Move the mouse slowly (especially if the number of spots is high) to allow your computer to follow the movement (unless it is very powerful). If the number of spots is too high, you can decrease it by choosing the "manual" method of spots generation. See **Generation of the reflections**

-RotationX: To simulate the rotation of the crystal by mouse motion around an axis perpendicular to the plane of the image (X). Move the mouse around the center of the diagram while pressing a button.

-RotationY: To simulate the rotation of the crystal by mouse motion around a horizontal axis (Y). Move the mouse vertically while pressing a button.

-RotationZ: To simulate the rotation of the crystal by mouse motion around a vertical axis (Z). Move the mouse horizontally while pressing a button.

-Indexation: Click on a spot or a stereo pole. The fundamental Miller indices of the reflection or of the pole are displayed on the screen.

Note: The stereographic projection displays the computed poles corresponding to the possible Laue spots and the computed poles corresponding to the other directions (in the whole space) defined by the maximum index limit fixed by the user (spots generation).

Bitmap picture menu

File option:

Give the name of the bitmap picture file. The picture is loaded. Now, The scale and the origin of the picture must be defined.

Origin option:

Point option : For the determination of the origin when the image of the direct beam is visible (transmission geometry for example). Click the center of the diagram.

Circle option : If the mark of the direct beam is not apparent and if a circular shadow has the direct beam as center, you can drag a circle (variable radius) to determine the center of the diagram : Move the cursor near the center, press the left mouse button and **do not release it**. A circle appears and now you can move it to center the shadow by (slowly) moving the mouse. When you release the button, the origin is determined (see the lower panel). The circle radius can be adjusted by clicking in the + or - buttons.

Scale Option:

The acquisition of the scale (number of pixels per Cm) can be achieved by two ways:

1-Measure option: Click at two known points and give the distance in cm of these points. For example, You can superimpose a transparent ruler on the Laue diagram before scanning.

2-Enter option: You must give the scale directly in pixels per Cm (for example 1.bmp, the scale=29.5). The value of the scale is displayed on the back red panel (in pixels per cm).

Measure Coord.Option:

Only for the option **Coordinates** of the main menu. For measuring coordinates of the reflections, click first near the spot and then click again in the appearing zoom image. When the measurements are finished, click the option **Save coord**.

Save coord. Option:

Give the file output name. The program then returns to the simulation.

Digitiser menu

Note for the use of the digitiser DIGIKON :

Use the « F » key of the cursor box to end the measures, « D » key to delete the current point and « 1 » key to enter a measure.

Port configuration option :

Configure the port for the communication with the digitising tablet. (Com port number, baud rate, data bits, parity, handshaking mode) at the first use.

Origin Determination option :

Click on one or several point to determine the diagram origin which will be defined as the centroid of these points.

Coordinates Measure option :

Click on the selected reflections for indexing.

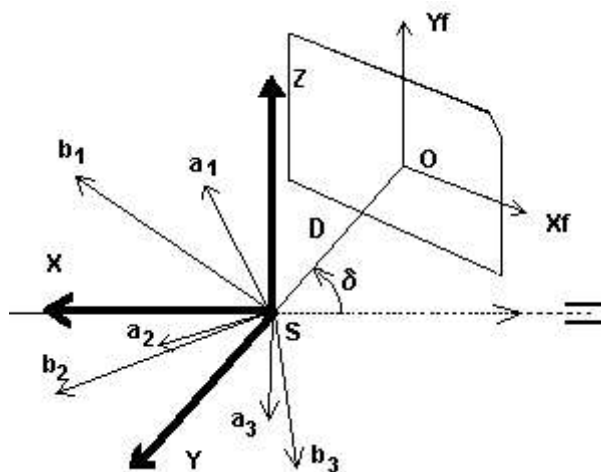
See **Measurement of the spot coordinates.**

Orientation Matrix

The **Orientation Matrix** describes the orientation of the crystal

Orientation matrix definition

See also **Orientation matrix meaning**



-**a₁, a₂, a₃**: basis vectors of the direct unit cell.

-**b₁, b₂, b₃**: basis vectors of the reciprocal unit cell.

Used frames in OrientExpress:

-(F0) (**a₁, a₂, a₃**): reference frame of the direct lattice.

-(F1) (**b₁, b₂, b₃**): reference frame of the reciprocal lattice.

-(F2) : orthogonal reference frame of the reciprocal lattice.

-(F3) (**X, Y, Z**): orthogonal reference frame attached to the measurement (**X** // to the beam, **Z** // to the film plane).

-(Fd) (**Xf, Yf**) Orthogonal two dimensions frame attached to the detector (**Yf** // to **Z**)

If **X3** and **X1** point out the same crystal vector, respectively, in the orthogonal frame (F3) attached to the Instrument and in the oblique frame (F1) attached to the crystal in the reciprocal space, the orientation matrix [R31] and the reverse matrix [R13] are defined by:

$$\mathbf{X3}=[\mathbf{R31}].\mathbf{X1} \text{ and } \mathbf{X1}=[\mathbf{R13}].\mathbf{X3}$$

with:

[R13]=	a1x	a1y	a1z	Direct matrix
	a2x	a2y	a2z	
	a3x	a3y	a3z	
[R31]=	b1x	b2x	b3x	Reciprocal matrix
	b1y	b2y	b3y	
	b1z	b2z	b3z	

aix, aiy, aiz are the projections of the **ai** vector and bix, biy, biz are the projections of the **bi** vector on the axis **Y**, **Y**, **Z** respectively.

Orientation matrix meaning

See also **Orientation matrix definition**

[R13] and [R31] tell you where are the crystal basis vectors in the reference instrument frame and what are the axis of this frame in the crystal:

- 1-The [R13] rows are the projections of the direct basis vectors **a1**, **a2**, **a3** onto **X**, **Y**, **Z**.
- 2-The [R31] columns are the projections of the reciprocal basic vectors **b1**, **b2**, **b3** onto **X**, **Y**, **Z**.
- 3-The [R13] columns are the projections of the unit vectors // **X**, **Y**, **Z** of the instrument frame onto **b1**, **b2**, **b3** expressed in reciprocal units b1, b2, b3.
- 4-The [R31] rows are the projections of the unit vectors // **X**, **Y**, **Z** of the instrument frame onto **a1**, **a2**, **a3** expressed in direct units a1, a2, a3.

Orientation matrix acquisition

See also **Orientation matrix meaning**

Orientation matrix definition

The aim of OrientExpress is to determine the orientation matrix of the crystal. But you can introduce this matrix by the option **Matrix/Acquisition**. In this case you must give one or two or three directions of the instrumental reference frame (X, Y, Z) expressed in direct or reciprocal units. If you give one or two directions the program determines itself the other(s). If you give two or three directions, they must be nearly perpendicular. For example :

0.2	1.5	0.0	direct space [u,v,w]
0.0	0.0	1.2	reciprocal space (h,k,l)
0.0	0.0	0.0	

These vectors are perpendicular because the product $hu+kv+lw=0$.

If the given vectors are not exactly perpendicular (within 0.5° error), the program makes a correction. The first direction is retained, the third is perpendicular to the first and second direction. Only the second direction is change

Orientation matrix refinement

See also **Orientation matrix meaning**

When the orientation matrix is determined from the observed coordinates, it is refined by a least square method. This matrix can be refined more accurately by another method using a bigger number (≤ 30) of measured coordinates (option **Matrix/Refinement**). This method also refines the crystal to detector distance which is usually measured approximately. The additional coordinates can be introduced by :

- Reading in the coordinates file (*.coo). In this case, the set of new coordinates to be used for the refinement must follow the set of coordinates used for the determination of the orientation. The two sets of coordinates are separated by a 0,0 line.

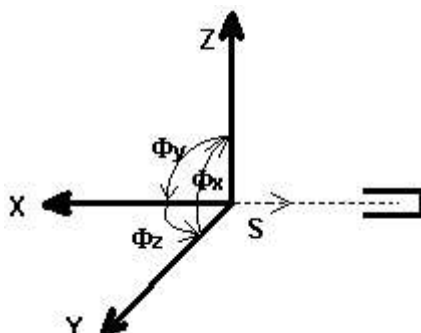
- Acquisition by keyboard in a special window.

- Acquisition on a bitmap image of the experimental diagram.

A report of the refinement can be found in a file named **report.txt**.

Crystal reorientation

See **Orientation matrix definition**



Once the crystal orientation matrix [R13] has been found, the program can compute the pairs of rotation angles which will give any desired new orientation when applied to the sample holder. In fact they will set either a direct lattice (D) or a reciprocal lattice @ axis of the sample either along the direct beam axis **X** or along the camera axis **Z**. In the following the rotations around the instruments axes are labelled (Φ_x , Φ_y , Φ_z) and their positive directions are shown in the figure.

Example : if $\Phi_z = 36^\circ$, $\Phi_y = 5^\circ$ are a pair of rotations computed by the program this means that the sample must be rotated first around the **Z** axis by 36° (**Y** thus goes to **Y'**) and then around **Y'** by -5° degrees.

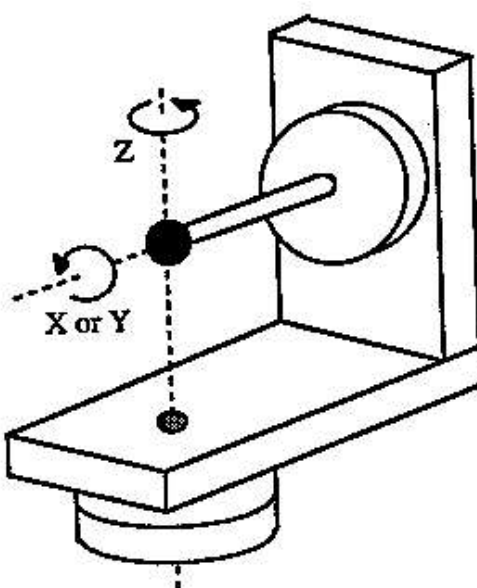
The rotation angles are computed with the assumption that the two rotation axes of the sample holder are initially set parallel to any of the three axes of the Cartesian frame (R_3). It is possible to use a standard goniometer head, however the angular range of the axes are in general too limited. Therefore we suggest the use of a device with two orthogonal 360° cradles.

Warning : Between sample reorientations the sample holder must be reset to its initial setting.

Note :

The type of rotation called **ZY'** means that the crystal must be turned **first** around the **Z** and **then** around the **Y** axis of the sample holder. It is equivalent to consider that the crystal will be turned first around the **Y** then around the room's **Z** axis.

Proposed design for the sample holder:



Indexing strategy

The indexing process is a critical one. It will be only successful if, on the one hand, the data are free of severe errors (tolerances : cell parameters $< 0.2\%$; distance $D < 0.5$ mm, Bragg spots coordinates < 0.3 mm) and if, on the other hand, the fundamental reflections have been sensibly selected (**Bragg reflections indexing process**).

1-Use a small number of reflections. The use of a large indexing data set (N observed reflections) to obtain an accurate orientation matrix is not advisable. In fact this will make the success of the indexing too much dependant of the measurement precision. In most cases values of N of 5 or 6 will fit. On the other hand, when the right solution is found, it is always a good idea to perform the final matrix refinement on a much larger set of extra reflections (≤ 30).

2-Select a pair of fundamental reflections from the set of N measured spots lying at the crossing of **zone lines** (small Miller indices) and use **small** h, k and l intervals. The magnitude of the computing time and the number of bad solutions rapidly increases with the magnitude of the selected bonds. On the other hand the $N-2$ remaining selected reflections must not have a Miller indice > 10 .

3-In case of failure widen the h, k and l intervals or select a new pair of fundamental reflections. If no pair is succesful, carefully check the input data (especially cell parameters, **distance D** and réflexion coordinates). See also **Special Cases**.

Special cases

1-Crystal in special orientation. When the crystal is initially set with a symetry axis nearly parallel to the beam axis, it is often difficult to find « good » fundamental reflections on the Laue pattern, i.e reflections with low Miller indices. This is because, firstly, the vectors with simple indices make a large angle with this axis and the corresponding spots are out of the detector area and secondly because the crossing points of **zone lines** are rare. In such a case two actions are possible :

-For example if the beam is near a (001) crystal axis, all the reflections (except the (001) one) have a **high** I Miller indice. Thus try to index with **normal h and k indices range, and wide l range**.

-Slightly rotate the sample and take a new Laue pattern which will exhibit measurable fundamental reflections.

2-Rhombohedral crystals. It is not advisable to use the hexagonal representation since the Miller indices of the Bragg spots would be much larger and thus the indexing process would be often much more arduous or even impossible.

Supplied examples

Five examples are supplied with the program :

1-File **examp1.dat** concerns a classical **transmission** Laue diagram of an alumin (Al_2O_3) crystal which is **rhomboedral** but is described here on a **hexagonal** cell. It is obtained with a flat x-ray film. The wavelength range is about **0.3-3 Å**. The coordinates, measured by transparent millimetric sheet, of six diffraction spots are included in the file **examp1.coo**. The file **examp1.bmp** contains a scanned image of the original diagram. (see **First example of indexation** and **Second example of indexation**)

2-Files **examp2.dat** and **examp2.coo** concern a classical **transmission** diagram with the same experimental conditions as above, obtained from an organic **triclinic** crystal.

3-Files **examp3.dat** and **examp3.coo** concern a classical **back-reflexion** diagram obtained from a **cubic** mineral crystal.

4-Files **examp4.dat** and **examp4.coo** concern a Laue experiment obtained in the ILL Institute in Grenoble (M. Lehmann) : Neutrons source (**2-6 Å**), **cylindrical camera** ($r=6.5$ Cm) with imaging plates, protein crystal (Lysozyme, **triclinic** structure, **$V_c=26242$ Å³**). (See **Example of Simulation**)

5- Files **examp5.dat** and **examp5.coo** concern a Laue diagram coming from a special experiment (ILL Institute, B. Hamelin) : **Transmission** geometry, hard X rays source (**0.05 to 0.15 Å**), long distance (100 cm) crystal to detector, two dimensional detector, and particular orientation of the crystal. (See: **Example of Simulation**).

First example

A transmission Laue pattern has been achieved on a crystal of rhombohedral aluminium oxide (Al_2O_3) and the corresponding data are contained in the file named **examp1.dat**. The coordinates measured with a transparent millimetric sheet, of 6 selected spots are stored in the file named **examp1.coo**.

Determination of the orientation of the sample

1-Click **data/file** (see **Main Menu**) and choose the file named **examp1.dat**. A window named **Data** appears in the left part of the main window. Note that Aluminum oxyde is rhomboedral and has been here described on an hexagonal cell with crystal system keyword **HEXA** and the lattice mode keyword **R**.

2-Click **Coordinates/File** and choose the file named **examp1.coo**. A window named **Coordinates** appears in the right part of the main window.

3-Click **Indexation/Search**. Default values are proposed for the choice of the two fundamental reflections and for bounds of possible Miller limits (see **Bragg Reflexions Indexing Process**). Click **OK**. The program finds two solutions with different indexations and loads the most likely solution. Note that this selected solution has the smallest Miller indices (on average). Click **OK**.

Verification of the selected solution (simulation)

4-Click **Simulation**. A window named **Spots generation** appears (see **Generation of the Reflections**). Choose the "automatic" method. Click **OK** and the simulated pattern appears. You cannot conclude if the simulated pattern is similar to the experimental one because you do not have the original pattern displayed. But a file named **examp1.bmp** is provided and contains the scanned picture of the experimental pattern. Now we are going to load this picture in order to compare it with the simulated one:

5-Click **Picture** option of the simulation menu (see **Bitmap Picture Option** and **Measurement of the Spot Coordinates**)

In the bitmap picture menu :

1-Select **File** option and load the file **examp1.bmp**. The image of the experimental pattern appears. Now it is necessary to determine the scale and the origin of the pattern.

2-Select **Scale/Enter**. Give the scale of the scanner image: 29.5. (It would be measured using the centimeter scale imposed on the image). The scale is written in the red window.

3-Select **Origin**. Move the cross cursor near the beam image and click. A zoom image of the central area appears.

4-Click exactly the middle of the image of the direct beam. Thus the origin is determined. The pixels coordinates of this point are written in the red window.

5-Select the option **OK**. The program returns to the simulation and the image of the pattern appears with the superimposed simulated one. You can conclude that the selected solution is a good one because of the good coincidence of the two images.

Test the other options of the simulation menu :

6-Select **Option/RotationXY**. Now, move slowly the mouse while pressing a button. This action simulates a rotation of the sample around the horizontal and vertical axes of the screen and causes the synchronous modification of the simulated pattern. To restore the initial matrix, return to the main menu, select **Matrix/Restoration** then **Simulation/Laue**. The initial Laue diagram is again displayed. Likewise test the option **Option/RotationZ** which makes a rotation around the axe perpendicular to the image.

7-Select **Option/Indexation**. Move the cursor exactly on a spot and click. The Miller indices of the Bragg fundamental reflection of the spot are displayed on the screen.

8-Select **Print/Results Edition** option. The list of the calculated Bragg reflections id can be edited by the NotePad windows program. Of course you can print this list.

9-Select the option **Diagram/Print**. The simulated Laue pattern is drawn with scale 1.

Second example

Here we shall use the data of the first example of Alumina (**examp1.dat**), but we are going to measure the coordinates of the diffraction spots directly on the scanner picture (**examp1.bmp**).

Indexation and simulation.

- 1-Click **Data/File** and choose the file **examp1.dat**.
- 2-Click **Coordinates/Scanner Picture**. The **Bitmap Picture Window** appears:
- 3-Determine the scale and the origin of the diagram (**Measurement of the Spot Coordinates**)
- 4-Click **Measure coord.** and point at five or six chosen diffraction spots (See **Bragg reflections indexing process**).
- 5-Click **Save/coord.** and give an output name for the coordinates file. You return to the main menu.
- 6-Click **Indexation/Search**. In the indexation window confirm the default values for the numbers of fundamental spots and the Miller indices bounds, or try other values. If the indexation process succeeds, and if multiple solutions were found, the most likely solution is loaded.
- 7-Click **Simulation/Laue**. Choose the "automatic" method of indices generation. The scanner image and the simulated pattern computed from the current orientation matrix appear.

Orientation matrix refinement.

- 8-If the indexation process succeeded, click the option **Matrix/Refinement** of the main menu. A window named **Acquisition of additional coordinates** appears. Choose the option **Acquisition of the additional coordinates on the bitmap picture**. The scanner picture appears again. Point at the diffraction spot you want to use for the refinement (max. number =30). Every time you click, the computed corresponding Miller indices are written in the right window. If the program cannot index the réflexion, a "???" string is written. This indexation failure may due to the fact that one of the Miller indices is greater than 10, or that the orientation matrix is too rough. When the acquisition is finished, click the **OK** button. A short report of the refinement is displayed in a window name **Matrix refinement**. You may edit (and print) a complete report with the option **Data/Edit** of the main menu and enter the file name **report.txt**.
- 9-**Simulate the Laue pattern again**. You can see the additional diffraction spots as red coloured small crosses.

Simulation

If you can't Index the patter, you can Simulate a pattern with a given orientation

Simulation of a Laue pattern

From the orientation matrix and the experimental data, the program computes the coordinates of the possible diffraction spots having integer Miller indices h, k, l and satisfying the following conditions :

- 1-The lattice spacing D_{hkl} must be within the interval $[\lambda_1/2\sin(\theta_1), \lambda_2/2\sin(\theta_2)]$

θ_1, θ_2 : The Bragg angle limits imposed by the instrument geometry;

λ_1, λ_2 : The wavelength limits of the radiation spectrum.

2-The reflection must be allowed by the Lattice mode given in the crystal data (i.e. Bravais lattices plus rhomboedral-hexagonal and diamond) and by the space group.

3-The diffraction spot must be located within the bounds of the sensitive surface of the detector.

Notes :

-The Bragg spot intensities, which depend on the structure, are not calculated by the simulation process.

-A spot may be the result of the overlap of several harmonics. Indeed for a given Bragg θ and a same lattice spacing D_{hkl} we may observe the first-order reflection h,k,l at wavelength λ , the second order reflection $2h,2k,2l$ at wavelength $\lambda/2$, etc.. provided that $\lambda, \lambda/2, \dots$ are within the range λ_1, λ_2 .

-if the unit cell volume is large, the number of possible reflections may be huge. To keep it at a reasonable value, when necessary, the program automatically performs a homothetic rescaling of the unit cell which has a little incidence on the aspect of the simulated pattern.

See **Generation of the Reflections**

-A list of the simulated spots (i.e. Miller indices, coordinates in the detector reference (Rf), fundamental wavelength, orders of the allowed harmonics) can be edited or printed during the simulation.

Generation of the reflections

For the simulation of the Laue patterns, OrientExpress takes into account the extinctions of the Bravais lattice and of the space group of the crystal, but does not compute the intensities of the reflections. Thus a very big number of Laue spots can be calculated depending on the wavelength range and of the volume of the unit cell of the crystal. In a real Laue pattern the most part of the weak reflections are not observed. In order to limit the number of simulated reflections, you can choose between two methods:

1-"**Automatic Method**". The program automatically performs an homothetic rescaling of the unit cell which has a little incidence on the aspect of the simulated pattern

2-"**Manual Method**". The number of reflections is limited by introduction of a D_{hkl} limit bigger than the D_{hkl} minimum corresponding to the wavelength minimum. You must adjust this value until the simulated Laue pattern looks like the experimental one.

For stereographic projection, you also must give the maximum Miller indice corresponding to the smallest direct axis.

Simulation example

Generally, the **"automatic"** method of spots generation (see **Generation of the Reflections**) gives a satisfactory simulation of the Laue pattern as, for example, in the case of Alumina crystal (**First example of Indexing**). However in particular cases (protein crystals with big cell volume which can give a very important number of Bragg reflections, or hard X rays experiment which will give a very small number of diffraction spots) the user may wish a better pattern simulation. You can test the two methods with the extreme examples supplied with the program:

Examp4.dat and **examp4.coo** contain the data of a neutron Laue experiment (cylindrical camera) using a Lysozyme crystal with a rather big cell ($V_c=26242 \text{ \AA}^3$). In this kind of experiment, the number of apparent diffraction spots depends on the sample quality and the user will desire a Laue pattern simulation adapted to his experimental image. The **"manual"** method of generating spots allows the user to introduce a minimum lattice spacing D_{hkl} below which the reflections are not calculated. Thus the best value of D_{hkl} can be determined by "trial and error".

Examp5.dat and **examp5.coo** files concern a Laue diagram from a silicon crystal and a hard X ray source (from 0.05 to 0.15 Å). Because of the long sample to film distance ($>100 \text{ Cm}$) and the particular orientation of the crystal (the beam is nearly parallel to an A_4 symmetry axis), only five diffraction spots, belonging to the same *zone line*, are visible on the film. Load the data and coordinates files and index the diagram with $[-1, 1]$ as h , k and l limits. Four solutions are found. The first one is automatically loaded, but it is not the "true" solution, which is the fourth one. This result can be proved only by simulation of a Laue pattern for each solution with the "manual" method of generation and the value of 0.417 \AA for the D_{hkl} limit. Indeed with these conditions, the fourth solution exhibits five and only five diffraction spots corresponding with the experimental ones.