

Article

Classification of 2D-GIWAXS Images of Highly Textured Molecular Thin Films Towards Indexation: Triclinic Lattices

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Abstract

Low-symmetry triclinic lattices, as formed by molecular thin films, are conveniently characterized by grazing-incidence wide-angle X-ray scattering (GIWAXS). These films are often highly textured with a specific crystallographic plane (*hkl*) parallel to the surface and thus form uniaxial powders. A classification scheme is presented that lays the groundwork for determination of the surface unit cell and indexing such complex scattering images. After identifying a first surface unit cell, methods are discussed for normalizing the lattice constants and further reduction of the unit cell.

Keywords: molecular thin films; triclinic structures; uniaxial powders; surface unit cell; normalized unit cell; reduced unit cell; grazing-incidence wide-angle X-ray scattering

1. Introduction

Grazing-incidence wide-angle X-ray scattering (GIWAXS) is a powerful method to characterize the structure of molecular thin films [1–3]. Such films typically have low-symmetry lattices—triclinic or monoclinic—and are highly textured, i.e., there is a well-defined close-packed molecular plane (*hkl*) that forms parallel to the substrate. For isotropic substrates such as glass slides or silicon wafers covered with native oxide, there is no preferential in-plane orientation—crystalline grains form with a random in-plane orientation. Such uniaxial powders or 2D powders have characteristic scattering patterns that contain full information about the lateral packing and the perpendicular stacking of the molecular planes in a single GIWAXS image. Such scattering patterns can be collected on modern lab-based X-ray apparatus with a suitable 2D detector [2,3]. At a high-flux synchrotron radiation facility, crystallization processes during film formation and morphological transitions can even be studied in situ and in real time [4–9].

Molecular thin films of organic semiconductors are important for sensors [10–12], light-emitting devices [13–15], photovoltaics [16–18], and water-splitting [19,20]. They are prepared by thermal evaporation in vacuum or solution processes such as spin coating or blade coating, followed by a thermal annealing step [21–24]. Typically, such molecules crystallize in low-symmetry crystal lattices, in particular triclinic and monoclinic. Such low-symmetry lattices feature pronounced polymorphism—molecular materials often have several bulk polymorphs and can also form specific thin film polymorphs and metastable polymorphs as a function of the specific sample preparation conditions [25–27]. This behavior does not only have important implications for the charge transport in thin films of conjugated molecules [27–33] but also for solubility of the films, an important aspect in pharmaceutical molecules [34].



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The general goal of such structural studies is to investigate the processing–structure–performance relationship, with GIWAXS covering the first two aspects [21,22]. For a quick check on what kind of film has formed (preferential molecular plane, polymorph), it is important to index the thin film diffraction pattern efficiently. A lot of information can already be obtained by determining the surface unit cell [3]. Successful indexing is also an essential first step towards a complete thin film structure determination [35–39].

After a review of the methodology for finding the surface unit cell, an application example will be discussed in detail, including basic crystallographic principles for finding the normalized primitive unit cell as well as methods to check whether this unit cell can be further reduced.

2. Methodology

When molecules form a well-defined close-packed layer parallel to the substrate, this layer will feature a surface unit cell with in-plane lattice vectors $\vec{a}$ and $\vec{b}$. The usual construction of the reciprocal lattice

$$\vec{c}^* = 2\pi \frac{\vec{a} \times \vec{b}}{(\vec{a} \times \vec{b}) \cdot \vec{c}} \quad (1)$$

implies that the $\vec{c}^*$ vector of the reciprocal surface unit cell is perpendicular to the plane of the film and has no in-plane components. Hence, the reciprocal surface unit cell can be parametrized in a convenient way by a set of lattice vectors $\vec{a}^*$, $\vec{b}^*$, $\vec{c}^*$ with [3]:

$$\vec{a}^* = \begin{pmatrix} a_{\parallel}^* \\ 0 \\ a_{\perp}^* \end{pmatrix}, \quad \vec{b}^* = \begin{pmatrix} b_{\parallel}^* \cos(\gamma_{\parallel}^*) \\ b_{\parallel}^* \sin(\gamma_{\parallel}^*) \\ b_{\perp}^* \end{pmatrix}, \quad \vec{c}^* = \begin{pmatrix} 0 \\ 0 \\ c_{\perp}^* \end{pmatrix} \quad (2)$$

This parametrization splits the problem of finding the six reciprocal lattice parameters into two steps: The first task is to determine the in-plane lattice with components $a_{\parallel}^*$, $b_{\parallel}^*$, $\gamma_{\parallel}^*$ [40]. Then, the perpendicular components can be determined in a straightforward way: $c_{\perp}^*$ is associated with the spacing of the (00 l) reflections and $a_{\perp}^*$ and $b_{\perp}^*$ can be identified as the lowest $q_{\perp}$ values for the (10 l) and (01 l) rods, which were identified in the first step. This parametrization also motivates the introduction of *scattering rods*, which are series of diffraction spots with fixed h and k and varying l values. Scattering rods are a useful concept in grazing-incidence scattering and are also essential for retrieving a complete set of in-plane q -values, when diffraction spots with low $q_{\perp}$ values are missing or too weak to be observed.

2.1. The In-Plane Reciprocal Surface Unit Cell

The in-plane reciprocal lattice vector components are determined from the experimentally observed scattering rods, which yield a sorted sequence:

$$q_1 < q_2 < q_3 < \dots \quad (3)$$

These q -values correspond to a set of lengths of reciprocal lattice vectors forming a 2D lattice with unit cell parameters $a_{\parallel}^*$, $b_{\parallel}^*$, and $\gamma_{\parallel}^*$. The determination of these lattice parameters is usually the most difficult part of indexing a GIWAXS pattern. However, Lagrange–Gauss (LG) reduction imposes strict limits on the reduced unit cell in 2D [40]. Reduction methods were originally developed by Lagrange [41] and Gauss [42] for their

mathematical work on quadratic forms. It was then actually Gauss himself who pointed out, in a little sidenote to a book review [43], that positive quadratic forms had a deep connection with distances in point lattices and he speculated that this connection would be useful for the emerging science of crystallography.

Such a reduced unit cell features the two shortest possible lattice vectors $\vec{a}_{\parallel}^*$ and $\vec{b}_{\parallel}^*$ with an enclosed angle $\gamma_{\parallel}^*$ close to 90° . It is useful to also introduce the short diagonal $d_{\parallel}^*$, which equally defines an optimum triangle within the reduced reciprocal surface unit cell (see Figure 1). $d_{\parallel}^*$ is given by

$$d_{\parallel}^{*2} = a_{\parallel}^{*2} + b_{\parallel}^{*2} - 2 a_{\parallel}^* b_{\parallel}^* \cos(\gamma_{\parallel}^*) \quad (4)$$

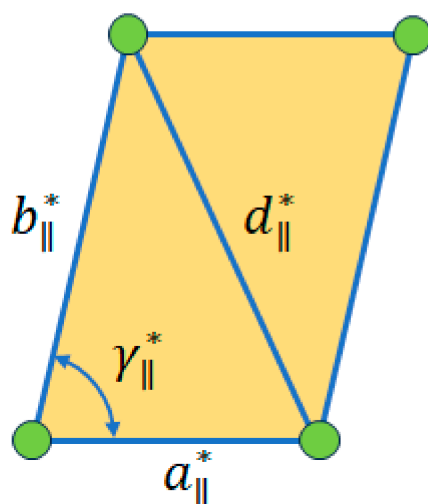


Figure 1. Two equivalent ways of describing the Lagrange–Gauss-reduced unit cell. Instead of using $a_{\parallel}^*$, $b_{\parallel}^*$ and $\gamma_{\parallel}^*$, it is convenient to include the diagonal $d_{\parallel}^*$, which can be easily identified with a suitable q_i . Reproduced from [40] (D. M. Smilgies, 2026).

Conveniently, $d_{\parallel}^*$ can be directly assigned to one of the observed q_i values, as will be detailed in Section 2.3, with LG reduction imposing

$$a_{\parallel}^* \leq b_{\parallel}^* \leq d_{\parallel}^* \quad (5)$$

2.2. Narrowing Down the $\gamma_{\parallel}^*$ Range

Before tackling the actual indexing problem, some other property of LG-reduced unit cells shall be explained, as is relevant here. $\gamma_{\parallel}^*$ offers a simple geometric way to quickly distinguish which assignments correspond to proper LG-reduced lattices. The angle is related to $a_{\parallel}^*$, $b_{\parallel}^*$, $d_{\parallel}^*$ by

$$\cos(\gamma_{\parallel}^*) = \frac{a_{\parallel}^{*2} + b_{\parallel}^{*2} - d_{\parallel}^{*2}}{2 a_{\parallel}^* b_{\parallel}^*} \quad (6)$$

From the triangle formed by $a_{\parallel}^*$, $b_{\parallel}^*$, $d_{\parallel}^*$ and the LG-condition, $a_{\parallel}^* \leq b_{\parallel}^* \leq d_{\parallel}^*$ follows that the smallest possible $\gamma_{\parallel}^*$ is obtained for a given $a_{\parallel}^*$ and $b_{\parallel}^*$ when an isosceles triangle can be formed with $b_{\parallel}^* = d_{\parallel}^*$; otherwise, the unit cell is not LG-reduced. This implies

$$\gamma_{\parallel}^* \geq \gamma_{\parallel,lim}^* = \arccos\left(\frac{a_{\parallel}^*}{2 b_{\parallel}^*}\right) \quad (7)$$

This condition narrows the possible range of $\gamma_{\parallel}^*$ down considerably more for high aspect ratios of $b_{\parallel}^*/a_{\parallel}^*$, as shown in Figure 2.

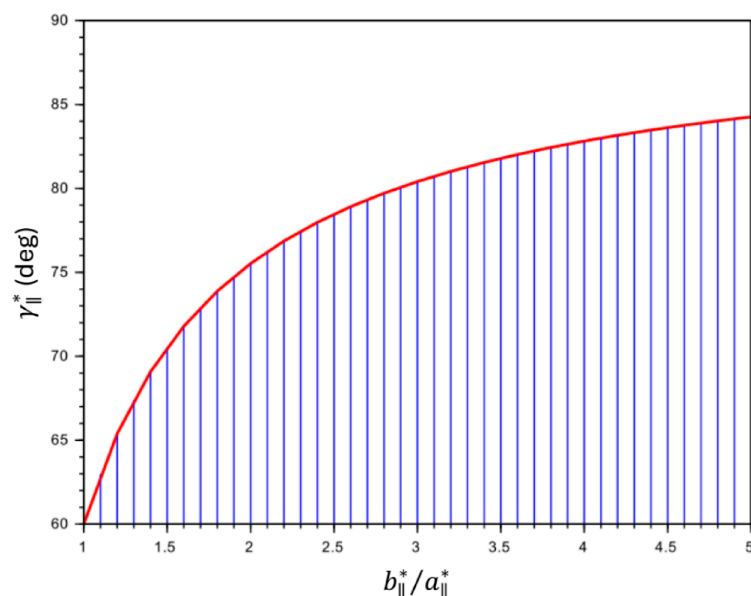


Figure 2. $\gamma_{\parallel}^*$ range compatible with LG reduction as a function of the aspect ratio $b_{\parallel}^*/a_{\parallel}^*$. The area above the red solid curve is available and the hatched area corresponds to $a_{\parallel}^*, b_{\parallel}^*, \gamma_{\parallel}^*$ combinations that can be further reduced. Note how the available $\gamma_{\parallel}^*$ range becomes smaller as the aspect ratio increases.

We will also explain our choice of reducing the $\gamma_{\parallel}^*$ range further to $\gamma_{\parallel}^* \leq 90^\circ$ in more detail. In our previous publication, we motivated this condition by noting that in our case, we were only interested in the length of q -vectors rather than the full lattice symmetry. In particular, Figure 3 shows that lattices with $\gamma_{\parallel}^* = 90^\circ + \theta$ are mirror images of lattices with $\gamma_{\parallel}^* = 90^\circ - \theta$ and thus corresponding lattice vectors have the same lengths. This allowed us to restrain the $\gamma_{\parallel}^*$ range further to

$$\gamma_{\parallel,lim}^* \leq \gamma_{\parallel}^* \leq 90^\circ \quad (8)$$

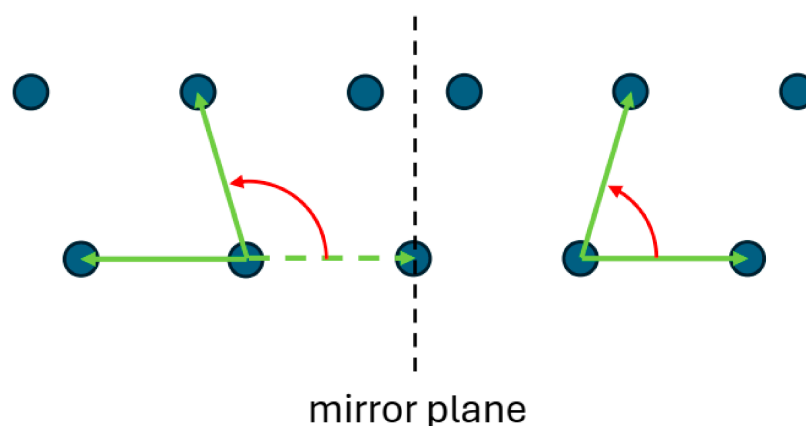


Figure 3. Equivalence of lattices with $\gamma_{\parallel}^* = 90^\circ - \theta$ and $\gamma_{\parallel}^* = 90^\circ + \theta$. The two lattices are shown to be related by a mirror plane (dashed line) and hence generate the same length sequences of scattering vectors q_i .

For all other $\gamma_{\parallel}^*$ values, a further reduced cell can be constructed with the LG algorithm. This reduced range is also necessary for consistency and to guarantee that the chosen diagonal (see Figure 1) remains shorter than the other diagonal.

2.3. Classification of GIWAXS Images of Triclinic Structures

The first task of indexing the in-plane reciprocal surface unit cell is to identify $a_{\parallel}^*$, $b_{\parallel}^*$, and $d_{\parallel}^*$ with the appropriate q_i of the above sequence. Here, we will consider triclinic lattices, while we will leave the case of monoclinic lattices to the second part of this study. A similar classification was made in our previous paper [40]; however, here, we will refine the analysis further and include a more detailed analysis of special cases. The classification scheme lends itself to automation with software and the Scilab scripts used for the application example below will be available on GitHub.

The last three cases deal with the complications imposed when there are multiples of $a_{\parallel}^*$ in the q -sequence, i.e., for $b_{\parallel}^*/a_{\parallel}^* > 2$. First of all, the multiples of $a_{\parallel}^*$ need to be identified and disregarded from the q_i sequence. For $\gamma \sim \gamma_{lim}$, the difference between $b_{\parallel}^*$ and $d_{\parallel}^*$ may become too small to be resolved, in which case $d_{\parallel}^*$ should be set equal to $b_{\parallel}^*$. The special case when $b_{\parallel}^*$ happens to be equal or very close to $2a_{\parallel}^*$ in length is already covered in the previous trial assignments. For $n > 2$, other coincidences with $b_{\parallel}^*$ may have to be additionally considered. A quick comparison of the measured scattering images with the predicted spot positions, for instance, using the *indexGIXS* program in reciprocal space mode [44], can help to quickly identify such a situation.

2.4. A Simple Measure of Success

After a suitable candidate for $a_{\parallel}^*$, $b_{\parallel}^*$, $d_{\parallel}^*$ is found, the corresponding theoretical sequence can be generated by sorting the calculated vectors $q(h, k)$ by their lengths.

$$q(h, k) = \sqrt{h^2 a_{\parallel}^{*2} + k^2 b_{\parallel}^{*2} + 2hk a_{\parallel}^* b_{\parallel}^* \cos(\gamma_{\parallel}^*)} \quad (9)$$

The corresponding scattering rods can be overlaid onto the scattering pattern using a program such as *indexGIXS* [44] for visual inspection. This step not only matches the identified case to the data but also provides now a definite set of indices (hk) to associate with each of the scattering rods.

A simple numerical measure of success for the assignments is the total deviation Δ from the experimental q_i sequence. In the spirit of putting the focus on the experimental data, only the deviation of q_i from the closest calculated q -vector $q(h, k)$ will be considered and then all partial deviations will be summed by their absolute value. This also makes Δ robust with regard to missing reflections in the q_i sequence. In order to normalize the total deviation with regard to the length N of the experimental sequence and put it into a convenient range, Δ is divided by N and multiplied by 10. With typical sequence lengths q_i of 5 to 20 values, this provides a useful metric against which the various assignments of $a_{\parallel}^*$, $b_{\parallel}^*$, and $d_{\parallel}^*$ can be tested. From previous examples [40], it was found that for a good match, the value of Δ lay below 1 or was at least close to 1.

3. Application Example

As a test case for the outlined methods, we use the complex GIWAXS pattern of zinc phthalocyanine (ZnPc) grown by thermal evaporation onto a graphene-covered substrate [45], as shown in Figure 4. Due to π - π interactions with the graphene layer, conjugated molecules tend to lie flat on graphene, which was also reported for ZnPc. Preventing the molecule from forming its stable β polymorph, this creates a metastable, albeit highly ordered, film that transforms back into the β polymorph upon heating to 400 °C [45]. The

pattern has already been successfully indexed by the original authors and serves here as a high-quality test object. Apart from sample quality, it is also important to choose good scattering parameters to achieve sharp reflections. As a rule of thumb, the width of the sample along the beam should be less than 5% of the sample to detector distance, in order to avoid radial streaking in the reflections [46].

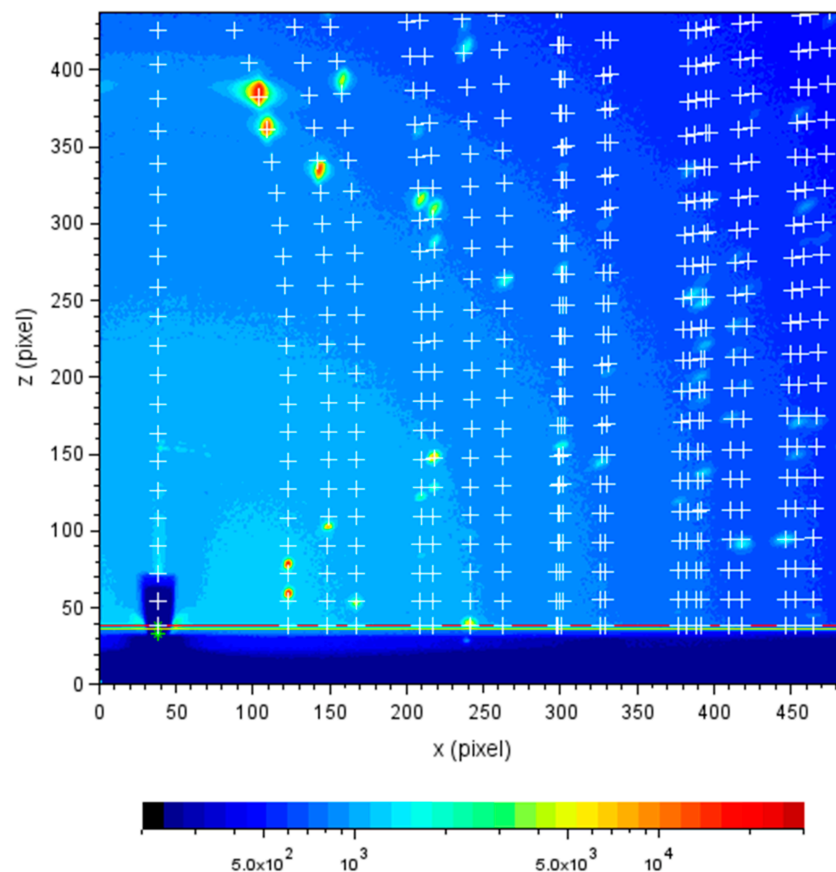


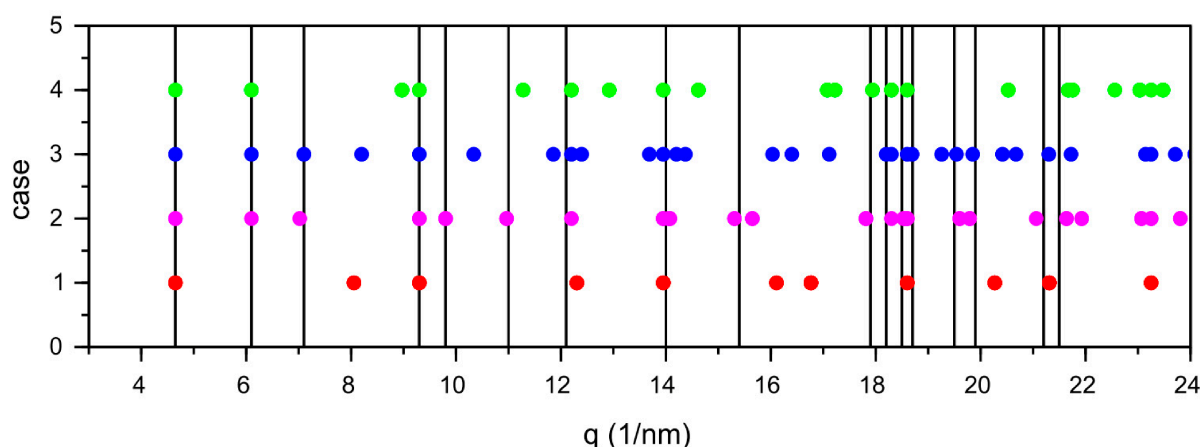
Figure 4. GIWAXS image of a ZnPc film thermally grown on a graphene-coated substrate. The best match sequence of $q(h, k)$ and associated scattering rods (crosses) are overlaid.

3.1. The In-Plane Components

A total of 17 in-plane q_i values were determined. The best match was found by assigning the first three values according to the second case in Table 1, $a_{\parallel}^* = b_{\parallel}^* < d_{\parallel}^*$. The associated GIWAXS image with the scattering rods overlaid is shown in Figure 4. In addition, a simple graphical tool is provided in Figure 5. Here, every trial calculated sequence of q -values is represented by a sequence of dots, while the experimental sequence of q_i -values is represented by vertical lines. This simple tool, shown here for the ZnPc pattern in Figure 4, provides a quick graphical overview of all trial assignments and their matches with the experiment. The procedures outlined here were cast into a computer program, *Classify*. The program is written in the free Scilab software [47] (version scilab-2026.1.0) and available on GitHub [48]. The best match was found for a rhombic lattice (case 2) with $a_{\parallel}^* = b_{\parallel}^* = 4.65 \text{ nm}^{-1}$ and $\gamma_{\parallel}^* = 82.0^\circ$, yielding a Δ of 0.14.

Table 1. Basic assignments of $a_{\parallel}^*, b_{\parallel}^*, d_{\parallel}^*$ to the q_i values.

Lattice Type	Condition	Assignment
hexagonal	$a_{\parallel}^* = b_{\parallel}^* = d_{\parallel}^*$	$a_{\parallel}^* = b_{\parallel}^* = d_{\parallel}^* = q_1$
square or rhombic	$a_{\parallel}^* = b_{\parallel}^* < d_{\parallel}^*$	$a_{\parallel}^* = b_{\parallel}^* = q_1, d_{\parallel}^* = q_2$
rectangular or oblique	$a_{\parallel}^* < b_{\parallel}^* < d_{\parallel}^*$	$a_{\parallel}^* = q_1, b_{\parallel}^* = q_2, d_{\parallel}^* = q_3$
oblique, special case	$a_{\parallel}^* < b_{\parallel}^* = d_{\parallel}^*$	$a_{\parallel}^* = q_1, b_{\parallel}^* = d_{\parallel}^* = q_2$
high aspect ratio, $n \times a_{\parallel}^* < b_{\parallel}^*$	$n \times a_{\parallel}^* < b_{\parallel}^* < d_{\parallel}^*$	$a_{\parallel}^* = q_1, b_{\parallel}^* = q_{n+1}, d_{\parallel}^* = q_{n+2}$
high aspect ratio, $\gamma \sim \gamma_{lim}$	$n \times a_{\parallel}^* < b_{\parallel}^* \sim d_{\parallel}^*$	$a_{\parallel}^* = q_1, b_{\parallel}^* = d_{\parallel}^* = q_{n+1}$
high aspect ratio, $b_{\parallel}^* < n \times a_{\parallel}^* < d_{\parallel}^*$	$a_{\parallel}^* < b_{\parallel}^* < n \times a_{\parallel}^* < d_{\parallel}^*$	$a_{\parallel}^* = q_1, b_{\parallel}^* = q_n, d_{\parallel}^* = q_{n+2}$

**Figure 5.** Tentative assignments based on the seven cases in Table 1. Observed q_i -values are shown as vertical lines. Colored dots are predictions based on the trial assignments of the q_i . Cases 5–7 do not occur since multiples of $a_{\parallel}^*$ were not found.

As a final step, the full set of measured q_i was used to perform a least-squares refinement of the in-plane reciprocal lattice parameters. By matching the experimental q_i values with the calculated $q(h, k)$ values and their indices from Equation (9), the in-plane surface unit cell parameters were refined as outlined previously [40]. The final set of parameters is $a_{\parallel}^* = b_{\parallel}^* = 4.657 \text{ nm}^{-1}$ and $\gamma_{\parallel}^* = 81.21^\circ$, yielding an improved Δ of 0.07 from an original value of 0.14.

3.2. The Out-of-Plane Components

In the previous section, it was shown how the in-plane lattice constants of the reciprocal surface unit cell can be found and the scattering rods can be indexed with their in-plane indices (hk). For the next step, the scattering rods ($00l$), ($10l$), and ($01l$) are used to directly identify $c_{\perp}^*$, $a_{\perp}^*$, and $b_{\perp}^*$, respectively, as the lowest $q_{\perp}$ values on these rods. As a trial assignment, all values are chosen as positive. This will ensure that the angular lattice parameters are all acute in reciprocal space and obtuse in real space. This step is often straightforward, since low-index reflections are usually observable. However, the example highlights some complications that can occur.

The first thing to notice was that there was no observed reflection on the ($00l$) scattering rod. ZnPc is a planar molecule that lies flat on a graphene-covered substrate [45] and thus, the d-spacing perpendicular to the surface plane is relatively short, yielding a large Bragg angle. The reflections on the ($00l$) rod under grazing incidence are diffuse, i.e., these are not proper Bragg reflections, but due to the diffuse halo around the actual Bragg reflections [49] which may become too weak to observe at high Bragg angles. Using Bragg–Brentano geometry, the ZnPc (001) reflection was indeed observed [50].

Next, we found that $a_{\parallel}^*$ and $b_{\parallel}^*$ were equal, i.e., the $(10l)$ and $(01l)$ rods coincided. However, this does not necessarily mean that $a_{\perp}^*$ and $b_{\perp}^*$ need to be equal as well. So $a_{\perp}^*$ was tentatively assigned the lowest and $b_{\perp}^*$ the second-lowest q_{zj} value. Then, $c_{\perp}^*$ was determined by fitting the next reflections on the overlapping $(10l)$ and $(01l)$ rods. A viewing program for the scattering pattern and the overlaid calculated diffraction spot positions such as *indexGIXS* [44] is very useful for this purpose.

However, the resulting fit turned out to be poor. In particular the $(1\bar{1}l)$ and the $(11l)$ rods did not come out right, as shown in the detailed view in Figure 6. Comparison with the published structure revealed that their in-plane reciprocal lattice vectors had very similar lengths, but their reciprocal surface unit cell had a $\gamma_{\parallel}^*$ angle larger than 90° , but with the same difference from 90° as ours. We chose to keep our value of $\gamma_{\parallel}^*$, since our methodology was built on the condition of $\gamma_{\parallel}^* \leq 90^\circ$ (see Figures 1–3). However, we found that if the value from $a_{\perp}^*$ was changed from positive to negative, a good fit resulted. Similarly, keeping $a_{\perp}^*$ positive but making $b_{\perp}^*$ negative also resulted in a good match, while having both parameters negative gave the same incorrect result as with both parameters positive. Interestingly, upon transforming back to direct space, these choices with either $a_{\perp}^* < 0$ or $b_{\perp}^* < 0$ resulted in different unit cell parameters, but all unit cells had the same vector lengths and volume.

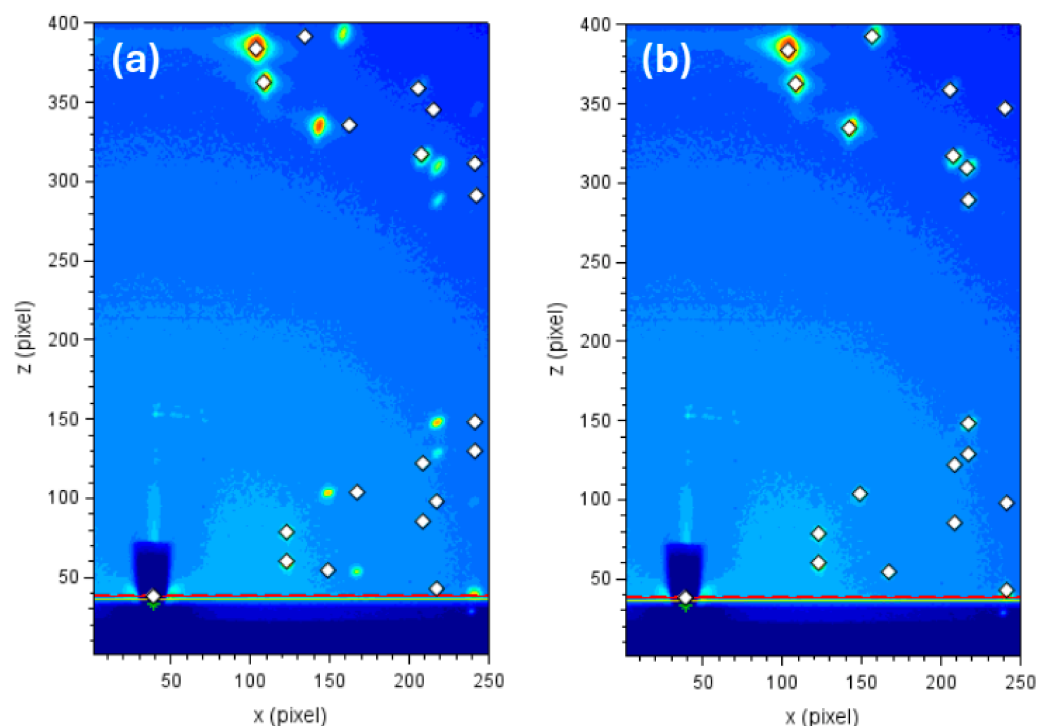


Figure 6. Proper choice of the sign of the out-of-plane components. $a_{\perp}^*$ and $b_{\perp}^*$ being both positive or both negative resulted in a poor match of the $(1\bar{1}l)$ and $(11l)$ scattering rods (a). A good fit resulted in the two cases where $a_{\perp}^*$ and $b_{\perp}^*$ had opposite signs (b). The diamonds indicate the calculated spot positions.

Before transforming back to direct space, the full set of spot positions, now that they have been indexed, is used to refine the out-of-plane parameters. In this case, the least-squares fitting problem is already linear:

$$h a_{\perp}^* + k b_{\perp}^* + l c_{\perp}^* = q_{zj} \quad (10)$$

where q_{zj} are measured out-of-plane position values along the (hk) scattering rods and (hkl) are determined with *indexGIXS*. Again, the least-square fitting solution for this overde-

terminated system of equations can be found by solving the normal equations [40]. The optimized parameters were $a_{\perp}^* = -1.349 \text{ nm}^{-1}$, $b_{\perp}^* = 2.347 \text{ nm}^{-1}$, and $c_{\perp}^* = 19.403 \text{ nm}^{-1}$. The corresponding Scilab script for this optimization can be found on Github as well [48].

3.3. The Normal Form of the Triclinic Unit Cell

At this point, all components of the vectors of the reciprocal surface unit cell have been found and Equation (1) can be used to transform the reciprocal unit cell back to the direct space surface unit cell by exchanging starred vectors with unstarred vectors and vice versa. However, we are left with three possible triclinic unit cells to describe the same lattice and a question arises of whether there is a common normal form of the unit cell. Such a normal form is important for different unit cells providing good indexation of the structure to not be considered as different structures.

First of all, we observe that all three unit cells feature a mix of acute and obtuse angles. Such unit cells can be further refined. Following Niggli [51] and Gruber [52], we find that the simple transformation

$$\vec{a} \rightarrow -\vec{a} \quad (11)$$

does not change the lengths of the unit cell vectors or the volume of the unit cell. However, it affects the angles of $\vec{b}$ and $\vec{c}$ with respect to $\vec{a}$ and turns them into the complementary angle:

$$\beta \rightarrow 180^\circ - \beta, \gamma \rightarrow 180^\circ - \gamma \quad (12)$$

Similar transformations can be found for $\vec{b}$ and $\vec{c}$. Out of the three unit cell angles, all three can either be acute or obtuse—in which case we are done—or two can be acute and one can be obtuse, or vice versa. In the latter case, those two angles define the lattice vector to be inverted. Hence, it is always possible to find a representation of the triclinic unit cell, where the three angles are either *all acute* or *all obtuse*.

It should be mentioned that, despite the fact that these two normal forms of the triclinic lattice unit cell have the same unit cell vector lengths, they are distinctly different. While this difference does not affect the lengths of the scattering vectors in an experiment, it can affect the location of the reflections, as can be seen in the example of ZnPc, i.e., even for 2D powder scattering. When we switch from a positive $a_{\perp}^*$ to a negative one, the angle of $\vec{a}$ with $\vec{c}$ along the surface normal switches from acute to obtuse, similar for $b_{\perp}^*$. So bringing the lattice parameters into the normal form using the simple transformation described above is indeed an essential step. Table 2 lists the different sets of lattice constants from the literature [45] and this work.

Table 2. Lattice constants derived for the ZnPc surface unit cell.

a (nm)	b (nm)	c (nm)	α	β	γ	Reference
1.36	1.36	0.38	118.8°	108.4°	81.1°	Ref. [45]
1.36	1.36	0.38	61.2°	71.6°	81.1°	Ref. [45]—normalized form ¹
1.362	1.365	0.383	118.30°	71.70°	98.77°	This work $a_{\perp}^* < 0$
1.362	1.365	0.383	61.70°	108.30°	98.77°	This work $b_{\perp}^* < 0$
1.362	1.365	0.383	61.70°	71.70°	81.23°	This work—normalized form ¹

¹ For comparison purposes, lattice parameters were not resorted in the standard order $a \leq b \leq c$.

In fact, the two parametrizations for the reciprocal surface unit cell for ZnPc yield the same reduced form of the direct lattice. As for the lattice determined by the original authors [45], the two normal forms of the lattice constants are in good agreement except for some small differences, which may be due to calibration or measurement errors.

3.4. The Reduced Primitive Unit Cell

The surface unit cell does not have to have the same size as the bulk unit cell, in particular, for the case in which the plane parallel to the substrate does not correspond to one of the faces of the bulk unit cell. A simple criterion to assess the size of the surface unit cell is to estimate the number of molecules per unit cell by estimating the density from the molecular mass and the volume of the unit cell.

$$\rho_{mol} = \frac{Z MW}{V_{uc} N_A} \quad (13)$$

Z is the number of molecules in the unit cell, MW is the molecular weight in (g/mol), V_{uc} is the volume of the unit cell and N_A is Avogadro's constant in (1/mol). For purely organic molecules, this density would be close to 1 g/cm³. If available, published density values can be used. Comparing the density values from microscopic estimates and macroscopic measurements, the number of molecules that would be contained in the unit cell, Z , can be estimated. For ZnPc, the published density value is around 1.6 g/cm³ [53], with the higher density being based on the heavy Zn atom in its center. For $Z = 1$, we essentially obtain the same value 1.62 g/cm³ from the unit cell. Hence, the unit cell identified above is primitive.

After Niggli, for any primitive unit cell, a unique reduced unit cell can be found [51]. This is particularly useful for the comparison of triclinic unit cells, which can assume many shapes, as seen in the previous section. Dirichlet already found the normalization conditions used in the previous section, and he additionally demanded that the face diagonals of the unit cell be longer than the largest edge of the unit cell face, resulting in three additional conditions for the reduced unit cell [54] (see also Equation (4)):

$$a^2 + b^2 - 2 a b |\cos(\gamma)| \geq b^2 \quad (14)$$

$$a^2 + c^2 - 2 a c |\cos(\beta)| \geq c^2 \quad (15)$$

$$b^2 + c^2 - 2 b c |\cos(\alpha)| \geq c^2 \quad (16)$$

The form of the conditions was chosen such that these conditions applied to both all acute and all obtuse angles [51,52]. In addition, the Dirichlet conditions were expressed directly in terms of the lattice constants. For the obtuse case, a condition for the body diagonal has to be added:

$$a^2 + b^2 + c^2 - 2 b c |\cos(\alpha)| - 2 c a |\cos(\beta)| - 2 a b |\cos(\gamma)| \geq c^2 \quad (17)$$

These conditions were further refined for special cases such as $a = b$ or angles equaling 90° by Eisenstein [55] and were finally cast into 44 crystallographic cases by Niggli [51]. For the surface unit cell found above, we found that it had already failed the Dirichlet conditions and thus could be reduced further.

Here, we applied the modern LLL algorithm to the surface unit cell [56]. This algorithm is based on the LG reduction in two dimensions [57]. In three dimensions, the second-shortest vector is first made almost perpendicular to the shortest lattice vector; then, the third vector is made almost perpendicular to both of the previous vectors, using a modification of the well-known Gram–Schmidt orthogonalization. If vectors have to be resorted by size, the procedure is repeated. LLL has become famed in the cryptography community where the algorithm is used to find reduced unit cells in high-dimensional lattices for encryption purposes. LLL has a parameter δ with $0.25 < \delta < 1$ that guarantees a finite number of iterations. For just three dimensions, LLL can be used in the most precise mode with $\delta = 1$ and almost always finds the unique Niggli cell. For comparing

two structures based on their primitive unit cells, it is recommended to use LLL on both sets of unit cells, since unit cells in the older literature may not be fully reduced.

Scilab scripts for LLL were prepared by Claude Sonnet 5 and Gemini 1.5 Pro. The algorithm contains less than 200 lines of code and converges instantaneously. The reduction in the refined surface unit cell was consistent between different versions of the code and yielded identical reduced unit cells to within six digits. Interestingly, we found that there was in fact a further reduced unit cell for ZnPc that closely resembled the structure of the triclinic α -phase of the related molecule copper phthalocyanine (CuPc) [58]. When properly normalized, as shown in Table 3, both unit cells appear almost identical.

Table 3. Comparison of the reduced ZnPc unit cell with the lattice constants of α -CuPc [58].

	<i>a</i> (nm)	<i>b</i> (nm)	<i>c</i> (nm)	α	β	γ
ZnPc	0.383	1.208	1.294	90.05°	91.98°	95.65°
α -CuPc	0.381	1.200	1.291	90.1°	91.0°	95.6°

The algorithm was extended to also provide the transformation matrix *S* to the new set of lattice vectors. This base transformation

$$\begin{pmatrix} \vec{a'} \\ \vec{b'} \\ \vec{c'} \end{pmatrix} = S \begin{pmatrix} \vec{a} \\ \vec{b} \\ \vec{c} \end{pmatrix} = \begin{pmatrix} 0 & 0 & 1 \\ 0 & 1 & -2 \\ 1 & 0 & 1 \end{pmatrix} \begin{pmatrix} \vec{a} \\ \vec{b} \\ \vec{c} \end{pmatrix} \quad (18)$$

yielded the new reduced unit cell. How does it relate to the surface unit cell? Specifically, what is the plane parallel to the substrate within the refined unit cell? By using the transposed matrix S^T from the equation above, we find [59]:

$$\begin{pmatrix} H' & K' & L' \end{pmatrix} = (HKL) S^T = (001) \begin{pmatrix} 0 & 0 & 1 \\ 0 & 1 & 0 \\ 1 & -2 & 1 \end{pmatrix} = \begin{pmatrix} 1 & -2 & 1 \end{pmatrix} \quad (19)$$

For the properly normalized unit cell, the $(\bar{1}21)$ plane was also found to be parallel to the substrate for α -CuPc [58].

The associated Scilab scripts, as well as the *indexGIXS* program, are provided on GitHub [47]. The reduced unit cell and its orientation were tested with *indexGIXS* and yielded a consistent match of the GIWAXS pattern with the refined surface unit cell, as shown in Figure 7. A similar reduction procedure including the transformation matrix is also provided by the Bilbao Crystallographic Server [60] and confirms the results presented here. However, the server does not provide information about the methods used.

These findings underline that for comparison of triclinic structures and polymorph determination, using the fully reduced unit cell is important, as we have seen in this study: we have found a variety of primitive unit cells that all index the GIWAXS pattern perfectly. Finding the proper reduced unit cell for a triclinic lattice is important since polymorphism is prevalent in this low-symmetry crystallographic system. In particular, care is indicated before claiming a new polymorph, if based on a unit cell that has not undergone proper scrutiny. For ZnPc, previously reported single crystal structures were monoclinic; however, Schünemann et al. found evidence for a triclinic α -ZnPc phase in thin films [61], in analogy to the α -phase of the closely related molecule CuPc [58,62]. The fully reduced unit cell of ZnPc films in this study has very similar lattice parameters, which supports that α -ZnPc was indeed formed [45].

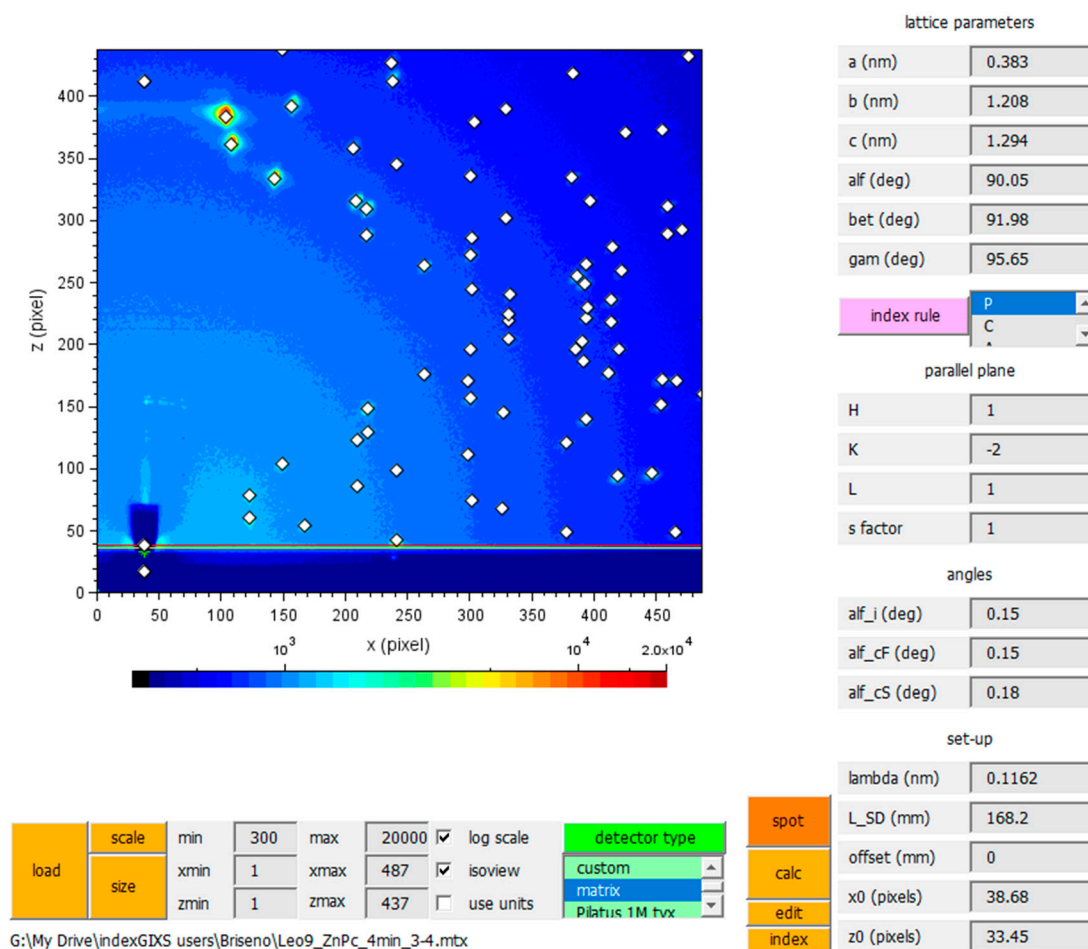


Figure 7. The ZnPc pattern with the fully reduced unit cell and a $(1\bar{2}1)$ orientation that closely resembles the α -phase of CuPc. The full *indexGIXS* output with all parameters is shown.

4. Conclusions

A method for determining the surface unit cell of low-symmetry molecular thin films from GIWAXS images is provided, refining our earlier reported method further. LG reduction seriously reduces the possible range of reciprocal surface unit cell parameters. While a finite number of assignments of $a_{\parallel}^*$, $b_{\parallel}^*$, and $d_{\parallel}^*$ are possible, these can be quickly accepted or rejected by matching the experimental q_i sequence with calculated sequences. A successful match will also reveal the corresponding in-plane Miller indices (hk) of the scattering rods. Using this information, the out-of-plane components can be identified conveniently. After each indexing step, a least-squares optimization should be performed. The current paper focuses on situations where all scattering rods are observable. In a second part, monoclinic structures will be discussed, including situations with missing scattering rods and parasitic reflections.

Even within the framework of LG-reduction, it was found that triclinic lattices could have a number of primitive unit cells. Basic crystallographic methods were used to show that these cells were equivalent and could always be normalized to a single choice with all angles acute or all angles obtuse. Finally, the standard surface unit cell thus identified was found to be further reducible, which resulted in a fully reduced bulk unit cell for this structure using the LLL algorithm. Such additional reduction steps are important for polymorph identification when comparing thin film data with previously known single crystal polymorphs.

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Data Availability Statement: The test data set and associated Scilab scripts are available on GitHub in the folder “classifyGIXS” [47].

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Abbreviations

The following abbreviations are used in this manuscript:

GIWAXS	Grazing-incidence wide-angle X-ray scattering
ZnPc	Zinc phthalocyanine
CuPc	Copper phthalocyanine

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