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Unambiguous indexing of trigonal crystals from white-beam Laue diffraction patterns: application to Dauphiné twinning and lattice stress mapping in deformed quartz

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Synchrotron X-ray Laue microdiffraction is used to investigate the microstructure of deformed quartz, which has trigonal symmetry. The unambiguous indexing of a Laue diffraction pattern can only be achieved by taking the intensities of the diffraction peaks into account. The intensities are compared with theoretical structure factors after correction for the incident X-ray beam flux, X-ray beam polarization, air absorption, detector response and Lorentz factor. This allows mapping of not only the grain orientation but also the stress tensor. The method is applicable for correct orientation determination of all crystals with trigonal symmetry and is indispensable for structure refinements of such materials from Laue diffraction data.

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1. Introduction

By using a submicrometre/micrometre-sized high-brilliance polychromatic X-ray beam at synchrotron facilities, with the help of focusing optics such as Kirkpatrick-Baez mirrors (Kirkpatrick & Baez, 1948), it has become possible to measure grain orientations and local lattice strain with high precision (Tamura et al., 2003). With this polychromatic X-ray microdiffraction (µXRD) technique, data are rapidly recorded as a raster scan array of Laue diffraction patterns (LPs) on surfaces, using a large two-dimensional area detector. Data are analyzed to obtain macroscopic orientation and strain maps of the sample, with spatial resolution only limited by the size of the focused X-ray beam and scanning step size. This tool can be applied not only to simple metals (Valek et al., 2002; Spolenak et al., 2003; Budiman et al., 2006; Chen et al., 2010) but also to complicated geological crystal structures such as calcite and plagioclase (Chen et al., 2011a; Wenk, Chen & Smith, 2011). The penetration power of hard X-rays into the samples opens up the application of three-dimensional scanning methods (Larson et al., 2002) to map the orientation distribution in three dimensions, a capability unique to µXRD.

However, the indexing of LPs of primitive trigonal lattices using the conventional LP indexing algorithms has been problematic. A primitive trigonal lattice is metrically identical to a primitive hexagonal unit cell with the same c/a value and same orientation. Corresponding positions of Laue reflections are thus indistinguishable, but there are differences in diffraction intensities. Such intensity differences in Laue diffraction patterns were first used by Schubnikow & Zinserling (1932, Fig. 19) to identify Dauphiné twins in quartz that are related by a 180° rotation around the c axis. Unlike twinning laws in many other materials, the diffraction pattern does not change in terms of reflection positions and reflection wavelengths but only changes in reflection intensities. Conventional LP indexing software identifies crystal orientation by fitting only diffraction peak positions but ignoring peak intensities (Jacobson, 1986; Chung & Ice, 1999; Tamura et al., 2003). It is therefore not possible to unambiguously index an LP of primitive trigonal crystals. Instead, resulting orientation matrices contain a 60° (or 180°) orientation ambiguity around the c axis. In previous μ XRD Laue studies of quartz, the trigonal mineral was treated as hexagonal (Kunz, Chen et al., 2009; Chen et al., 2011b). It is worth noting that this problem does not exist for trigonal Bravais lattices with a rhombohedral centering symmetry such as calcite, because of the special extinction rules imposed by this specific Bravais symmetry.

Recent developments in the Laue diffraction data analysis method allow diffraction peak intensities to be taken into account (Dejoie *et al.*, 2011). In this report we demonstrate the application of this method to the unambiguous indexing of LPs from primitive trigonal crystals. In order to allow for the automatic treatment of thousands of LPs as typically obtained during a μ XRD scan, the algorithm is implemented in a computer program to realize maps of the two-dimensional microstructure (orientation, strain, stress *etc.*) distribution in a sample. To illustrate our new method, we mapped the orientation and the residual stress in a deformed quartz grain from a moderately deformed granite using Laue μ XRD.

The structure of α -quartz has trigonal symmetry (space group $P3_121/P3_221$), but is described in a primitive hexagonal-symmetry unit cell (a = b = 4.921, c = 5.4163 Å; $\alpha = \beta = 90$, $\gamma = 120^\circ$; *e.g.* Glinnemann *et al.*, 1992).

2. Materials and experimental details

The sample is a moderately deformed granite from the Santa Rosa mylonite zone in southern California (PC 89). A quartz crystal with undulatory extinction was previously analyzed with the Laue method for residual strain (Kunz, Chen *et al.*, 2009). Here we use the same sample to determine the unambiguous orientation of trigonal quartz. An uncovered thin section of the sample PC 89 was prepared in a standard way: first glued on a glass slide with epoxy, then cut and ground down to approximately 30 μ m thickness, and subsequently polished with SiC and Al₂O₃. A quartz grain surrounded by biotite, muscovite and plagioclase was selected in this study. Optical microscopy revealed undulatory extinction, indicating some degree of elastic and plastic deformation. In addition, a 30 μ m thin section of a synthetic quartz single crystal was analyzed to test the reliability of the method.

The μ XRD experiment was conducted on Beamline 12.3.2 at the Advanced Light Source of the Lawrence Berkeley National Laboratory. A polychromatic (5 < *E* < 24 keV) X-ray beam (white beam) was focused with a set of Kirkpatrick–Baez mirrors to a 1 × 1 μ m spot (Kunz, Tamura *et al.*, 2009). The sample, mounted on a high-precision stage, was placed at

45° relative to the incident X-ray beam at its focal point with the help of a Kevence laser triangulation setup. The sample was scanned through the X-ray beam at the focus spot. At each step a Laue diffraction pattern was collected in reflection geometry, using a two-dimensional DECTRIS Pilatus 1M X-ray detector mounted at 90° with respect to the incident beam. As a result of the diffraction geometry in reflection, mainly high-order reflections are measured. The distance from the sample to the center of the detector was about 140 mm. The diffraction geometry, including the exact sample-todetector distance, the center channel of the detector and the tilt of the detector relative to the sample surface, was calibrated with a strain-free Si single crystal mounted on the thin section next to the scanned area. More details of the experimental setup are described by Kunz, Chen et al. (2009). For the undeformed single crystal, a single LP was collected. On the deformed PC 89 quartz grain, a $150 \times 92 \,\mu\text{m}$ area was scanned with 1 µm step size, resulting in 13 800 individual LPs. Typical LPs from the quartz standard and the deformed quartz are shown in Figs. 1(a) and 1(b), respectively. It should be noted that Fig. 1(b) shows one of the patterns that contains fairly sharp reflections, although they are much broader than those in Fig. 1(*a*).

3. Methods and analysis

All Laue patterns from the μ XRD experiment were automatically indexed using the software package *XMAS* (Tamura *et al.*, 2003); the indexing was based on peak positions and thus did not resolve the trigonality of quartz. The diffraction peaks were detected according to a user-defined threshold of the peak-to-background ratio. The peak position and width of each individual diffraction peak were determined by fitting



Figure 1

Laue diffraction images taken on (a) single-crystal quartz and (b) quartz from a deformed granite (PC 89) with selected Miller indices marked, as indexed by XMAS. The inset shows a zoom on the $4\overline{2}\overline{2}0$ reflection.

peak intensity (I) as a function of position on the Pilatus camera (x and y pixels) using a two-dimensional Lorentzian function (Noyan & Cohen, 1987, p. 168, equation 6.6). The raw integrated peak intensity was also recorded. Grain orientation was first indexed by fitting the positions of the Laue diffraction peaks based on the lattice parameters of unstrained quartz (Glinnemann *et al.*, 1992). We retained an LP only if more than 30 reflections were successfully fitted by *XMAS*. The Miller indices for the hexagonal lattice of some of the indexed reflections are displayed in the two patterns in Fig. 1.

The output results of each diffraction pattern containing peak position, integrated peak intensity, Miller indices and the wavelength of the diffracted X-ray beam were then processed in the following way. First, the number of harmonic reflections, *i.e.* the reflections from parallel sets of lattice planes (nh nk *nl*), where *n* is an integer, was determined using the method introduced by Dejoie et al. (2011). Only the reflections without harmonic overlap between 5 and 24 keV were considered. The structure factor, F_1 , of each selected reflection was calculated on the basis of the crystal structure (Glinnemann et al., 1992), using the Miller indices given by XMAS. The square of the modulus of the structure factor $|F_1|^2$ has to be corrected for polarization of the incident X-ray beam, the absorption of the diffracted beam by air, the incident X-ray beam flux as a function of wavelength, the detector response and the Lorentz factor (Dejoie et al., 2011, equations 1-10). This is necessary because, in Laue diffraction, diffraction from a lattice plane varies with orientation, wavelength and diffraction angle. The incident flux function was obtained by a 'reverse method' with Laue diffraction patterns taken on a thin sectioned calcite single crystal with the Pilatus camera (Dejoie et al., 2011). Since the orientation indexing given by XMAS has a 60° (180°) ambiguity, *i.e.* the peak indexed as *hkil* could actually be \overline{hkil} , the structure factors for both options were calculated and corrected. The new values were denoted as $|F_1|_{corr}^2$ and $|F_2|_{corr}^2$, respectively. The integrated intensity of each peak in a diffraction pattern should be linearly proportional to $|F|_{corr}^2$ if the indexing is correct. Therefore, the intensity of each selected reflection was plotted against the calculated $|F_1|^2_{corr}$ and $|F_2|^2_{\text{corr}}$ separately, and both $I - |F_1|^2_{\text{corr}}$ and $I - |F_2|^2_{\text{corr}}$ were fitted with a linear function $I = k_i |F_i|_{corr}^2$ (*i* = 1, 2). The slope was calculated by

$$k_i = \sum I / \sum |F_i|_{\rm corr}^2, \tag{1}$$

where $\sum I$ is the summation of the integrated intensity and $\sum |F_i|_{\text{corr}}^2$ is the summation of the square of the modulus of the structure factor of all the selected Laue reflections in a pattern. The quality of fitting was characterized by the deviation factor *R*, which was defined by

$$R = \sum |I - k_i|F_i|_{\text{corr}}^2 |\sum I.$$
 (2)

A smaller value of the deviation factor R indicates a better linear relation of the integrated intensity and the square of the modulus of the structure factor, indicating the correct choice of orientation.

Table 1

Comparison of the corrected structure factors of selected peaks under different indexing.

 $|F_1|_{corr}^2$, $|F_2|_{corr}^2$ and the integrated intensities have arbitrary units. The first indexing given by *XMAS* was not correct.

Integrated intensity	Miller index given by XMAS	$ F_1 ^2_{\rm corr}$	Alternative Miller index	$ F_2 ^2_{\rm corr}$
84124	4154	152.1	4154	9.1
19437	5146	133.8	5146	1.8
20971	5161	156.6	51 6 1	1.9
57666	3255	299.7	3255	10.6
3009	4156	133.7	4156	1.2
840586	4152	48.3	4152	117.0
653670	3145	40.9	3145	82.6

4. Results and discussion

4.1. Orientation determination

In the case of the thin sectioned strain-free synthetic quartz single crystal shown in Fig. 1(*a*), 134 reflections were indexed by *XMAS*; 101 of them were without harmonics within the energy range of the incident beam, and thus 101 points are plotted in Fig. 2 for the two trigonal orientation variants. The intensity *I* was rather poorly fitted to $|F_1|_{corr}^2$ (R = 0.96), as shown in Fig. 2(*a*), but shows a high degree of linear proportionality to $|F_2|_{corr}^2$ (R = 0.15, Fig. 2*b*), which indicates that the indexing given by *XMAS* (F_1) was wrong [*e.g.* in Fig. 1(*a*), 2022 was indexed as 0222]. A more detailed comparison of the corrected structure factor of some randomly selected peaks under the two different trigonal indexings is listed in Table 1, and it shows clearly that the corrected structure factor values change dramatically if the second indexing is used.

The need to correct structure factors is highlighted in Fig. 2(*e*), which is a plot of the intensity of the reflections for the diffraction pattern of Fig. 1(*a*) versus the square of the modulus of the corresponding structure factor before and after correction. It shows that the raw intensities are not proportional to the square of the modulus of the structure factors without correction, $|F|^2_{uncorr}$ (black squares), and the deviation factor *R* is calculated to be 0.73, rather than a lower deviation factor *R* of 0.15 for the structure factors after correction, $|F|^2_{corr}$ (shaded circles).

In order to investigate how the correct solution depends on the number of indexed peaks, various combinations were explored. When 77 peaks are indexed, 58 of them with no harmonics, the R factors are 0.94 and 0.15 when fitting I to $|F_1|^2_{\text{corr}}$ and $|F_2|^2_{\text{corr}}$, respectively; thus the results are essentially the same. The results remain the same when the number of indexed peaks is further reduced to about 30. This is, in our experience, the minimum number of peaks for a successful indexing even on a diffraction pattern with streaks. Therefore, from empirical evidence, it is concluded that as long as an LP is reasonably indexed using XMAS, the number of peaks is not a critical factor to determine the correct crystal orientation. Of course, a sufficient number of 'trigonal' reflections need to be included; 'hexagonal' reflections such as 1010 or 0110 are insensitive for the trigonal orientation determination (see e.g. Fig. 1*a*).

Similar to the case of the strain-free quartz single crystal, Figs. 2(c) and 2(d) display the $I - |F_i|^2_{corr}$ plots for the diffraction pattern of Fig. 1(b), taken on a quartz crystal in the deformed rock sample. For 51 reflections with no harmonics out of 68 indexed peaks output from XMAS, it is found that $|F_2|_{corr}^2 (R =$ 0.09) fits significantly better than $|F_1|^2_{\text{corr}}$ (R = 0.90), again suggesting that the quartz crystal has an orientation 60°



rotated relative to the one given by XMAS. It is noted that the integration box size for the peak fit was chosen to be 17×17 pixels wide when the diffraction patterns were indexed using XMAS. By checking all the diffraction patterns and the results from XMAS in this way, the correct orientation is obtained for each position, allowing the generation of a macroscopic orientation map. It turns out that the original orientation results chosen by XMAS for this scan have an equal chance of being right or wrong.

As demonstrated in Fig. 1(b), the diffraction peaks on this sample are smeared because of the plastic deformation induced by the accumulation of dislocations and subgrain boundaries (Cahn, 1949; Nye, 1953). It is thus a matter of concern how much the peak integration box size influences the determination of the integrated intensity and how much it will influence the crystal orientation indexing. If the integration box size is set to 25×25 pixels, which is the maximum that still allows the LPs to be indexed efficiently by XMAS on a personal computer within reasonable time, more than 90% of the recorded LP results remain unchanged, compared with the case of using a 17×17 pixel wide peak integration box size. The pixel deviation is set to be not greater than 0.08 when



Figure 2

(a), (c) The intensity fitting of the diffraction patterns shown in Figs. 1(a) and 1(b), respectively, using the orientation given by XMAS. (b), (d) The intensity fitting of the same diffraction patterns but setting the crystal orientation 60° rotated along the c axis with respect to the results given by XMAS. (e) The intensity fitting of the LP shown in Fig. 1(a). Circles and black squares represent the structure factors with and without corrections, respectively [compare with (b)].

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analyzing the LPs automatically with both integrated box sizes. In the other 10% of cases, not enough reflections could be indexed, because the box size was too large. The average R factor of the correctly indexed patterns taken on the deformed quartz sample is 0.22 (7) for a box size of 17×17 pixels and 0.24 (7) for a box size of 25×25 pixels. The R factor of the incorrectly indexed patterns was roughly 0.9 in both cases, which is much larger than the values of the correctly indexed patterns, and there is no ambiguity in identifying the correct orientation. Thus, the orientation determinations are not affected appreciably by the box size.

4.2. Dauphiné twinning and lattice strain mapping of deformed quartz

The distribution of crystal orientation is mapped and displayed by showing the Euler orientation angles in the

Bunge (1969) setting, φ_1 , Φ and φ_2 in Figs. 3(a)-3(c), respectively, based on the LP indexing by setting the box size to $17 \times$ 17 pixels and filtering out all the patterns in which fewer than 20 reflections can be found with no harmonic overlap. The gray color in these figures represents the failure of orientation indexing, either because the LPs failed to be indexed by XMAS because they may represent different phases or because too few reflections were available for linear fitting. It can be seen that many patterns near the bottom-right corner are not indexed, mainly because the crystal in this region is highly deformed, so the diffraction peaks are low in intensity. From the definition of the Bunge-Euler angles, we know that φ_1 and Φ define the orientation of the *c* axis of the crystal lattice, while φ_2 represents the orientation of the trigonal +a axis (rotation around the *c* axis). The distributions of φ_1 and Φ seem continuous in Figs. 3(a) and 3(b). This demonstrates that the orientation distribution of the c axis of the quartz crystal is



Figure 3

The distribution of the Euler angles (a) φ_1 , (b) Φ and (c) φ_2 in the Bunge setting. (d) A histogram showing the distribution of the φ_2 angle, demonstrating the existence of two discrete orientations separated by a 60° rotation around the c axis. (e), (f) The intragrain orientations for the parent and twin domains.

quite homogeneous in the scanned region, except for a small separate grain [circled in Fig. 3(a)] that exists at the top-right corner, indicated by the red color in Fig. 3(a).

The orientation distribution map of φ_2 in Fig. 3(c) shows that the grain with a single c-axis orientation (Figs. 3a and 3b) is divided into two domains with different a-axis orientations. One domain is indicated in yellow, while the other domain is displayed in blue. The φ_2 difference between the domains is 60° , identifying it as a Dauphiné twin. This was further quantified in a histogram displaying the distribution of φ_2 (Fig. 3d). The 90–105° region, which composes about 74% of the total indexed scanning steps, corresponds to the matrix grain, while the 30–45° range indicates the twin domain, which covers approximately 26% of the indexed area. The circled small grain, which makes up only 0.4% of the whole scanned region, can also be found in the range of 60–65° in the histogram. More detailed intragrain orientations for both the parent and the twin domains are shown in Figs. 3(e) and 3(f).

In theory, if an LP were taken on a Dauphiné twin boundary, the method introduced here would not work because the Laue reflections from the twin and host domains would superimpose. However, it is noticed that a pair of split peaks are observed in many of the diffraction patterns taken close to the twin boundaries as a result of the relative rotational distortion of the two domains after twinning. In these cases a sufficiently small box size needs to be selected for accurate integrated intensity measurement and thus unambiguous indexing of both sets of diffractions peaks. The method is not limited to a unique orientation on the diffraction pattern, but can also be applied when several grains/ domains are present.

In principle, once a Laue diffraction pattern has been correctly indexed, a second-rank deviatoric strain tensor can be obtained by measuring the deviation between the experimental diffraction peak position and the theoretically calculated results based on the unstrained lattice parameters (Pavese, 2005; Tamura et al., 2009), and then a second-rank stress tensor can be derived by applying Hooke's law, $\sigma_i = C_{ii}\varepsilon_i$, as long as the fourth-rank stiffness tensor C_{ii} is available. While the strain tensor measurement in sample coordinates is not affected by the 60° ambiguity of orientation determination, the stress tensor can only be computed from the strain tensor if the quartz crystal orientation is determined correctly. This is because the stiffness constants of trigonal quartz show a very strong anisotropy (Ogi et al., 2006). The stiffness tensor is illustrated in Fig. 4(a), where the stiffness constant C_{ij} ellipsoid is plotted relative to crystal coordinates in a spherical projection. The maxima and minima of the stiffness constants are roughly perpendicular to negative $\{01\overline{1}1\}$ and positive $\{10\overline{1}1\}$ unit rhombohedra, respectively. It is



Figure 4

(a) Stereographic projection of the stiffness of quartz. The stiffest and softest directions are roughly parallel to the $\{01\overline{1}1\}$ and $\{10\overline{1}1\}$ plane normals, respectively. (b)-(d) The macroscopic stress distribution in the scanned area σ_{xx} , σ_{yy} , σ_{zz} along the **x**, **y** and **z** directions of the sample coordinates, respectively. Yellow-red colors indicate tensile stress and blue colors compressive stress.

Table 3

Table 2	
Statistics of the strain and stress	s distribution in different grains in the
scanned area.	

	Parent domain	Twin domain	Average of all grains
ε_{rr} (10 ⁻³)	0.77 (1)	0.63 (2)	0.74 (1)
ε_{vv} (10 ⁻³)	-0.18(1)	-0.04(2)	-0.15(1)
ε_{77} (10 ⁻³)	-0.58(1)	-0.59(1)	-0.58(1)
σ_{xx} (MPa)	75 (1)	81 (2)	76 (1)
σ_{yy} (MPa)	-12(1)	-7(2)	-11 (1)
σ_{zz} (MPa)	-43 (1)	-71 (1)	-50 (1)

worthwhile to note that the strain measured from the Laue diffraction patterns is deviatoric strain instead of full strain; furthermore, the stress calculated by applying Hooke's law is not deviatoric stress because of the anisotropy of quartz (Chen *et al.*, 2011*a*). For the example of a deviatoric strain tensor (in the fixed sample coordinates),

$$\varepsilon = \begin{pmatrix} 0.5 & 0.3 & 0.2 \\ 0.3 & 0.4 & 0.6 \\ 0.2 & 0.6 & -0.9 \end{pmatrix} \times 10^{-3},$$

for a quartz single crystal that is oriented with its c axis parallel to the sample z axis and the a axis parallel to the x axis, the stress tensor acting on the crystal will be

$$\sigma = \begin{pmatrix} 14 & 17 & 12\\ 17 & 49 & 68\\ 12 & 68 & -84 \end{pmatrix} MPa$$

in the sample coordinates. However, if the orientation of the crystal is wrongly indexed because of the 60° ambiguity, an erroneous stress tensor,

$$\sigma = \begin{pmatrix} 57 & 31 & 34 \\ 31 & 6 & 72 \\ 34 & 72 & -84 \end{pmatrix} MPa$$

will be computed after conversion to the same sample coordinates (Nye, 1953). Comparing these two stress tensors, it is found that all components are dependent on the orientation, except that the normal stress along the z direction remains unchanged, because the two orientations share the same c axis. Two common methods can be applied in order to obtain the full stress tensor. One is to measure the energy/wavelength of a single reflection (Chung & Ice, 1999). The other is an estimation based on 'reasonable' assumptions; for example, in thin films sometimes the out-of-plane stress (σ_{zz}) is assumed to be zero (Choi *et al.*, 2003), while in bulk samples in some cases the stress is assumed to be hydrostatic (Chen *et al.*, 2009).

Figs. 4(b)-4(d) display the three normal components of the stress tensor in the sample coordinates. White curves represent grain boundaries or twin boundaries, while separated white spots could be caused by either incorrect indexing or very high stress. Tensile and compressive stresses are defined to be positive and negative, respectively. It is shown that the stress is mainly tensile along the **x** direction, and compressive along the **y** and **z** directions. These values are compatible with the reported fracture stresses in the single crystal, which were about 250 MPa in compression and 120–430 MPa in tension (Fitzgerald, 1960). No obvious relationship between the stress

distribution and Dauphiné twinning can be seen from the normal stress components in the x and y directions; however, the compressive normal stress in the z direction in the twin domain is significantly higher (more negative) than that in the parent domain. The statistics for each normal component of the strain and stress tensors are shown in Table 2. Our observation agrees with the observations that Dauphiné twinning can be imposed mechanically by high transient stresses (Schubnikow & Zinserling, 1932), such as during meteorite impact or seismic failure. For example, in shocked quartzite from Vredefort, twin boundaries identified by electron backscatter diffraction could be associated with deformation lamellae (Wenk, Janssen et al., 2011) and these deformation lamellae contained large residual strains (Chen et al., 2011b). The lattice strain study assumed hexagonal symmetry for quartz. It would be interesting to see if a trigonal identification would show a switch in stresses across twin boundaries.

5. Conclusions

An improved method based on diffraction intensities has been developed to unequivocally index the LPs of trigonal crystals with primitive Bravais lattices. The method has been applied to trigonal α -quartz. The crystal orientation distribution map of a deformed quartz crystal displays Dauphiné twin boundaries. Combining the unequivocal orientation and the deviatoric strain tensor, a correct stress tensor is obtained at each position, which has not been achieved before (Kunz, Chen *et al.*, 2009; Chen *et al.*, 2011*b*).

Besides quartz, the newly developed LP indexing method can be applied to other trigonal crystals and appears useful for any crystal structures displaying strong pseudosymmetries. Furthermore, our method of fitting the reflection intensity, which includes information on the atomic positions within the unit cell, is a first step towards crystal structure refinements using Laue diffraction. An analogous algorithm has been implemented in the current version of *XMAS* on Beamline 12.3.2 of the Advanced Light Source at Berkeley.

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