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The role of single-crystal X-ray diffraction in the description of new mineral species

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Abstract. The discovery and formal approval of a new mineral species require the integration of chemical, crystallographic, and structural information. Among the available analytical techniques, single-crystal X-ray diffraction (hereafter SCXRD) remains the most powerful method for determining the crystal structure of crystalline materials and, in turn, of minerals. In recent decades, advances in instrumentation and analytical methods have greatly expanded the capability of SCXRD, allowing reliable structure determination even from extremely small crystals and this is impacting the number of new mineral species that every year the mineralogical community is able to discover. Here, after an experience of around 20 years with SCXRD focused on new mineral discoveries and with about 80 new minerals collaboration, I will shortly describe how we apply SCXRD technique within the complex scientific procedure of the new mineral discovery and description.

Keywords: single-crystal X-ray diffraction, Commission on New Minerals, Nomenclature and Classification of the International Mineralogical Association, new mineral species, crystal structure, structure refinement, crystallographic data.

Conflict of interest. The author declares that he has no conflicts of interest.

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Роль монокристальной рентгеновской дифракции в описании новых минеральных видов

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Аннотация. Открытие и официальное утверждение нового минерального вида требует объединения химической, кристаллографической и структурной информации. Среди доступных аналитических методов монокристальная рентгеновская дифрактометрия остается наиболее мощным методом определения структуры кристаллических материалов, к которым относятся и минералы. В последние десятилетия достижения в области приборов и аналитических методов значительно расширили возможности монокристальной рентгеновской дифрактометрии, позволяя надежно определять структуру даже чрезвычайно мелких кристаллов, что влияет на количество ежегодно открываемых новых минеральных видов. В настоящей статье на основе примерно 20-летнего опыта работы с монокристальной рентгеновской дифрактометрией, в результате чего было утверждено около 80 новых минералов, кратко описано применение метода в рамках сложной процедуры открытия и описания новых минеральных видов.

Ключевые слова: рентгеновская дифракция на монокристаллах, Комиссия по новым минералам, номенклатуре и классификации Международной минералогической ассоциации, новые виды минералов, кристаллическая структура, уточнение структуры, кристаллографические данные.

Конфликт интересов. Автор заявляет об отсутствии конфликта интересов, связанных с рукописью.

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INTRODUCTION

The importance of crystallographic information in mineral characterization has increased significantly in recent years. The Commission on New Minerals, Nomenclature and Classification (CNMNC) of the International Mineralogical Association applies strict requirements for the approval of new mineral species, and in most cases a solved crystal structure is considered essential (Bosi et al. 2025). Consequently, SCXRD has become a fundamental step in the study and description of potential new minerals. Following, I will report all main steps that are usually followed to apply SCXRD for new mineral discover and description.

Extraction of grains and sample preparation

The first step in the crystallographic investigation of a potential new mineral consists of extracting suitable grains from polished sections or from mineral separates or directly from a massive matrix. It is crucial that these grains are typically identified on the basis of chemical data obtained previously through electron microprobe analysis or other microanalytical techniques. The best approach is to study by SCXRD exactly the same grain that was analysed previously through electron microprobe analysis.

The grain extraction is performed in most of the cases manually under a binocular microscope using fine tools such as tungsten needles and requires a significant experience especially when the grains to be studied are really rare crystals. The aim is to isolate a grain that can be mounted on a glass fiber (Fig. 1) or micromount and subsequently analyzed by SCXRD.

The size of the crystal is not necessarily the most critical factor for successful structure determination. Although larger crystals are easier to manipulate and generally produce stronger diffraction signals, crystal quality and structural order are much more important than size. Well-ordered crystals only a few tens of micrometers in size can result in high-quality diffraction data when analyzed using modern instrumentation. In practice, crystals in the range of 10–50 μm are often sufficient for single-crystal diffraction experiments but in general we try to collect crystals with size between 100 and 200 μm.

Preliminary diffraction screening

Once a grain has been extracted and mounted, a preliminary diffraction screening is performed. This step allows rapid evaluation of the diffraction quality and determination of whether the sample behaves as a single crystal or as a polycrystalline aggregate. This procedure, with modern instrumentation, can take no more than a few minutes.

During the screening, a small number of diffraction frames are collected to examine the reflection pattern. If the reflections are sharp and well separated, the grain can be considered a suitable single crystal (Fig. 2). In contrast, if the diffraction pattern consists of rings or diffuse reflections, the sample is likely to be polycrystalline or poorly crystalline (Fig. 3).

Even if this preliminary step is very quick test, however it is essential because it determines the most appropriate strategy for subsequent data collection, e.g., powder diffraction versus single crystal diffraction data collection.

Powder diffraction and Rietveld refinement

If the extracted grain does not behave as a single crystal but rather as a powder or microcrystalline aggregate, single-crystal structure determination is not possible. However, structural information can still be obtained through X-ray powder diffraction. In such cases, a high-quality powder dataset can be collected and analyzed using the Rietveld refinement method. This approach is particularly effective when the structure type of the mineral is already known or can be inferred from chemically similar compounds or synthetic analogues. Although powder diffraction "generally" provides less detailed structural information than single-crystal diffraction, it can nevertheless confirm structural relationships and refine crystallographic parameters. This method can therefore still contribute significantly to the characterization of a new mineral species when suitable single crystals are unavailable.

Single-crystal data collection

If the screening confirms that the grain is an actually a single crystal with adequate diffraction quality, a complete diffraction dataset is collected. The duration of data collection depends on several factors, including the size and quality of the crystal, the symmetry of the structure, the desired resolution and completeness of the dataset, the type of instrumentation used. In practice, collection times may range from a few hours to a couple of days to more than one week in very rare cases, depending on experimental conditions

Fig. 1. Typical gem-quality single crystal mounted through super glue on a borosilicate glass fiber for single-crystal X-ray diffraction data collection.

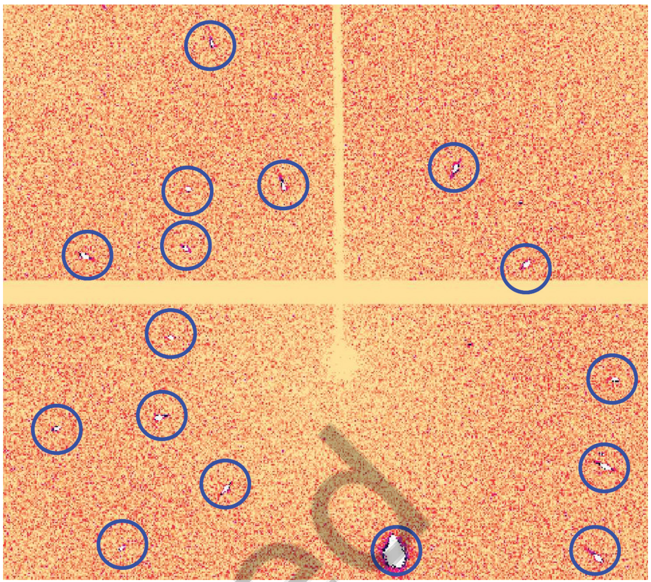

Fig. 2. Typical image collected at a single-crystal diffractometer and showing within the blue circles a series of diffraction spots (in this case the crystal is an olivine) indicating that the sample analysed is a single crystal.

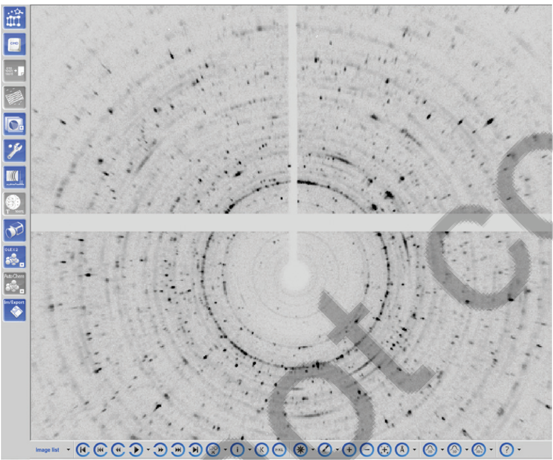

Fig. 3. Typical image collected at a single-crystal diffractometer and showing that the sample analysed is a polycrystalline material and cannot be studied by single-crystal X-ray diffraction.

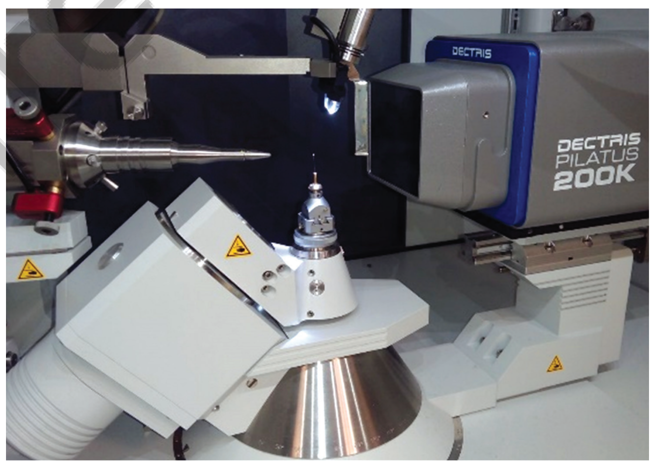

Fig. 4. Rigaku Oxford Diffraction Supernova equipped with a Dectris Pilatus 200K detector at the Department of Geosciences, University of Padova.

and the complexity of the structure. Parameters such as exposure time, detector distance, and rotation strategy are optimized to obtain the best possible dataset. Higher redundancy and completeness generally improve the statistical quality of the data and facilitate a reliable structure refinement.

Advantages of modern instrumentation

The crystallographic investigations described here that I carry out in my laboratory is thanks to a Rigaku Oxford Diffraction SuperNova diffractometer equipped with a Dectris Pilatus 200K detector (Fig. 4). This type of instrument resulted to be one of the best instruments internationally and offers several advantages compared with traditional laboratory diffractometers.

One of the main advantages is the high sensitivity and rapid readout of the hybrid pixel detector, which enables efficient data collection from very small crystals. This capability is particularly important right in mineralogical research, where potential new minerals

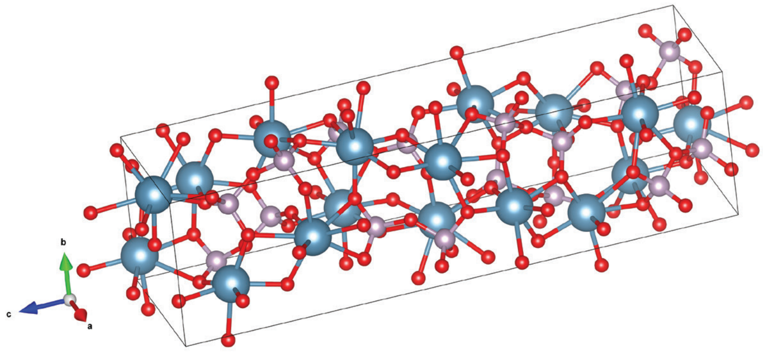

Fig. 5. Typical example of a figure representing the crystal structure of a mineral. In this case, I have reported the crystal structure of the recent discovered mineral grahampearsonite, Ca2P2O7 (Wang et al. 2026). The structure is plotted along a perspective view with the three axes reported on the left side. The red spheres are oxygens, whereas the violet spheres are phosphorus and the bluish ones are calcium. The solid lines represent the unit cell of the mineral. The structure was plotted using Vesta software (Momma and Izumi, 2011).

often occur as microscopic grains within complex mineral assemblages. I usually have no problems in measuring crystals of 10-15 μm in size using this instrumentation.

Another significant advantage is the ability to collect diffraction data from crystals enclosed within host minerals, including transparent phases such as diamond (see Angel et al. 2022 for an extensive review). This capability allows the study of mineral inclusions without the need to destroy the host crystal, which is particularly valuable in the investigation of high-pressure mineral assemblages and meteoritic materials. The instrument is also highly versatile, allowing both single-crystal and powder diffraction experiments to be performed. This flexibility is extremely useful when dealing with mineral samples that may exhibit variable crystallinity.

Structure solution and refinement

Once a satisfactory dataset has been obtained, the diffraction data are processed and the crystal structure is solved. Structure solution is typically followed by iterative refinement, during which the structural model is optimized to achieve the best agreement between observed and calculated diffraction intensities. One of the most important indicators of refinement quality is what in crystallography is called R factor, which quantifies the difference between observed and calculated structure factors. For a well-refined and publishable structure, the R factor should be reasonably low (e.g., < 5% for a typical X-ray diffraction measurement on a good quality crystal), and all refinement parameters should be physically meaningful. Additional indicators, such as displacement parameters, residual electron density, and the goodness-of-fit index, are also evaluated to ensure that the final structural model accurately represents the crystal structure. The quality of the structure refinement however does not depend only on the crystal quality, instrumentation, and data collection procedure. In order to get the best possible data, the crystallographer must face also problems with the site occupancies, which in turn are strongly related to the displacement parameters and possible cation and anion vacancies; Angel and Nestola (2016) wrote a review article dedicated to about 100 years of crystal structure data focusing on important aspects that should be considering when a crystal structure is refined.

Preparation of crystallographic data

After the refinement has been completed, the crystallographic results must be organized and documented for publication and for inclusion in the mineral proposal that must be submitted to the CNMNC. This documentation typically includes structure tables, such as unit-cell parameters, atomic coordinates, equivalent displacement parameters, and selected bond lengths and bond angles.

In addition, a CIF (Crystallographic Information File) is prepared. The CIF is a standardized file format that contains all crystallographic data required to reproduce and validate the structure determination. A checkCIF report is also generated. This automated validation procedure analyzes the CIF file and evaluates the consistency and quality of the crystallographic data, identifying potential problems or unusual features in the dataset. The CIF file and the checkCIF report must be included within the proposal and submitted to the CNMNC. To be remarked that the final CIF file is also required by all scientific magazine that will accept the publication of the new mineral. During the validation process, the checkCIF report may generate "alerts", which indicate potential issues in the crystallographic dataset or refinement. Alerts are classified according to their severity. The most important ones, known as "A-level alerts", correspond to issues that may significantly affect the reliability of the structure determination. If such alerts occur, they must be carefully examined and properly explained. In mineral descriptions, these alerts are usually addressed either directly in the CIF file or within the mineral proposal submitted to the CNMNC.

In addition to all information reported above, structure figures—such as polyhedral representations or projections of the atomic arrangement—are also prepared to illustrate the main structural characteristics of the mineral (Fig. 5).

Cases where a structure cannot be directly determined

In some cases, it may not be possible to determine a crystal structure directly from an extracted grain. This may occur when crystals are extremely small, poorly crystalline, or affected by structural disorder that prevents reliable diffraction measurements. Nevertheless, a new mineral proposal may still be submitted if it can be demonstrated convincingly that the phase has the same crystal structure as an already known mineral or synthetic compound. Structural equivalence can be established through powder diffraction data, chemical composition, micro-Raman spectroscopy, and comparison with known structural models. This situation is relatively common for minerals occurring as extremely small inclusions in meteorites or in complex mineral assemblages, where suitable single crystals may not be available.

CONCLUSIONS

Single-crystal X-ray diffraction is one of the most powerful tools available for the characterization of new mineral species. The technique provides direct information about atomic arrangement and allows precise determination of structural parameters. Thanks to modern instrumentation and improved analytical methods, it is now possible to solve crystal structures from extremely small crystals, greatly expanding the range of mineral phases that can be studied. As the requirements for mineral approval continue to evolve, the ability to obtain reliable structural data will remain central to the discovery and description of new minerals.

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