Table 2.1.2
X-ray applications for with modern X-ray diffractometers

X-ray scattering
Powder diffraction
Qualitative (phase identification) and quantitative phase analysis
Indexing, structure determination and structure refinement from powder data
Microstructure analysis (texture, size, strain, microstrain, disorder and other defects)
Pair distribution function analysis ('total scattering')
Thin-film analysis
Grazing incidence X-ray diffraction (GIXRD)
X-ray reflectometry
Stress and texture
High-resolution X-ray diffraction
Reciprocal-space mapping
In-plane GIXRD
Single-crystal diffraction
Chemical crystallography
Protein crystallography
Small-angle X-ray scattering
X-ray topography
X-ray absorption
X-ray radiography (X-ray-absorption-based imaging)
X-ray emission
X-ray fluorescence

specimen. X-rays that pass through the specimen can be detected to give a two-dimensional representation of the absorption contrast within the specimen. For tomography, the X-ray source and detector will be moved to blur out structures not in the focal plane. Multiple images can be used to generate a three-dimensional representation of the specimen by means of computed tomography. Obvious disadvantages are the large effective focal spot size of the X-ray sources and the relatively low resolution of the detectors that are typically used for powder diffraction, which, in combination with a limited adjustability of both the X-ray-source-to-specimen and specimen-to-detector distances, lead to substantial unsharpness issues and poor resolution. High-quality images can be achieved when using micro-focus X-ray sources and charge-coupled device (CCD) detectors with focus and pixel sizes smaller than 10 µm, respectively, but such an instrument configuration is not suitable for applications requiring ideal powders (see also Sections 2.1.6 and 2.1.7).

Collecting X-ray fluorescence data is comparatively straightforward. Data can be collected simultaneously to X-ray scattering data when employing a suitable detector, such as an energy-dispersive detector (Section 2.1.7.2.3). There are a couple of disadvantages to be considered, such as absorption issues (the specimen will be normally measured in air rather than in vacuum, hampering the analysis of light elements) and the inefficiency of excitation by the characteristic line energies of the X-ray source anode materials typically used for diffraction (hampering the analysis of elements with higher atomic numbers than that of the anode material).

2.1.5. Goniometer designs

A goniometer, by definition, is an instrument that either measures an angle or allows an object to be rotated to a precise angular position. In an X-ray diffractometer the purpose of the goniometer is to move the X-ray source, specimen and detector in relation to each other. Goniometers are usually categorized by the number of axes available for X-ray source, specimen and detector rotation, and are thus called one-, two-, three-, ..., n-axis (or -circle) goniometers.

Because of practical reasons, most goniometers consist of two distinct components, a goniometer base and a specimen stage, with the specimen stage mounted on the goniometer base.

The goniometer base typically offers two axes, one axis to rotate the X-ray source or the specimen stage, the other axis to rotate the detector. In some designs goniometer bases are omitted, specifically if there is no need to move the X-ray source and the detector, such as in Debye–Scherrer-type diffractometers with large detectors. Such machines are usually dedicated to a particular application without the need for high flexibility.

Depending on the requirements of the application, additional rotational and translational degrees of freedom may be needed to rotate and translate a specimen in space; these are usually implemented in the specimen stage. More rotational degrees of freedom may include the rotation of the X-ray source line focus or a rotation of the detector out of the diffraction plane to measure diffraction by lattice planes (nearly) perpendicular to the specimen surface, so-called non-coplanar diffraction.

2.1.5.1. *Geometrical conventions and scan modes*

In the literature there is some inconsistency related to the naming of axes and the choice of signs for angles (left- *versus* right-handed). A comprehensive treatment of geometrical conventions has recently been given by He (2009); in the following these conventions will be adhered to.

In many texts the notations θ–2θ and θ–θ rather than ω–2θ and ω–θ are used, mostly because of historical reasons. The first diffractometers operated in Bragg–Brentano geometry (see Section 2.1.3.1.2) and were equipped with single-axis goniometers. In such a goniometer the single axis drives two shafts which are mechanically coupled 1:2 or 1:1; thus the notations θ–2θ and θ–θ were coined. Today, the majority of all goniometer bases allow coupled as well as uncoupled rotation of the ω and θ axes. Therefore the ω–2θ and ω–θ notations should be generally preferred, as they represent the more general notations.

2.1.5.1.1. *Goniometer base*

A typical goniometer base provides two coaxial and independently driven axes, ω and 2θ, mounted perpendicular to the diffraction plane. These two axes are the main axes of a goniometer, since they have the most effect on the accuracy and precision of measured Bragg angles. The diffraction plane and the axes are generally described by a right-handed Cartesian coordinate system, as illustrated in Fig. 2.1.6, where the direct X-ray beam propagates along the X_L axis. Z_L is up and coincident with the ω and 2θ axes, and X_L–Y_L define the diffraction plane with the detector circle coplanar to it. Since X_L is coincident with the incident X-ray beam, it is also the axis of the Debye cones. The semi-apex angles of the cones are determined by the 2θ values given by the Bragg equation. The angles 2θ and γ describe the direction of scattering vectors in space (compare Fig. 2.1.1), where γ is defined as the azimuthal angle from the origin at $-Z_L$ with a right-hand rotation axis along the opposite direction of the incident beam ($-X_L$ direction).

The ω and 2θ axes are mechanically arranged as the inner circle and outer circle, respectively. The inner circle usually carries either the specimen stage or the X-ray source, while the detector is mounted on the outer circle. As a consequence, there are two common base goniometer configurations in use: In the ω–2θ (or θ–2θ with $\omega = \theta$) configuration, the incident-beam direction is