Table 2.1.1
Types of beam-path components available in laboratory X-ray powder diffraction

The column numbering corresponds to the positions indicated in Fig. 2.1.2 at which individual components can be mounted.

Position 1	Positions 2 and 4	Position 3		Position 5
X-ray sources	X-ray optics	Goniometer base	Specimen stages	Detectors
Fixed target Moving target (rotating anodes, liquid-metal jets)	Absorptive (apertures, metal filters) Diffractive (monochromators, analysers) Reflective (multilayer mirrors, capillary optics)	Vertical [ω–θ (θ–θ), ω–2θ (θ–2θ)] Horizontal [ω–θ (θ–θ), ω–2θ (θ–2θ)]	Fixed, rotating Specimen changer Eulerian cradles Kappa stages Tilt/fixed χ stages *XYZ* stages Flow-through cells Non-ambient (low temperature, high temperature, humidity, high pressure)	Scintillation Gas ionization (metal wire, micro-gap) Semiconductor (SiLi, strip/pixel, CCD/CMOS)

sible angular range may be limited for large components owing to collision issues, while heavy loads on vertical goniometers may impede alignment and lead to early wear and tear. Restrictions will be discussed in Sections 2.1.5 to 2.1.7 for the individual components.

These days, the exchange of lighter components, such as most X-ray optics, specimen stages and detectors, does not require any tools at all (such as when a snap-lock mechanism is employed) or more than a few screws for fixing. Alignment is normally not required when components are factory pre-aligned and handled with care, and when mounts are manufactured with good quality. Intrinsic changes of the beam direction (*e.g.* focusing crystal monochromators or X-ray mirrors) or beam offsets (*e.g.* two-bounce channel-cut monochromators) need compensating translation and/or rotation of the components involved.

The exchange of large, heavy components, or complicated rebuildings such as the conversion of a goniometer (vertical $\leftrightarrow$ horizontal, θ–$\theta \leftrightarrow \theta$–$2\theta$ *etc.*), may be still possible for technically skilled users. However, special tools may be necessary, requiring shipment of the component(s), or even the instrument, back into the factory. In addition, X-ray, machine and electrical safety directives by the local authorities have to be obeyed, and conversions may require updating approval to use the instrument. In such cases it may be more economic to operate two dedicated instruments instead.

The instrument control software plays a particularly important role in the context of instrument configuration and automated instrument conversion. In modern instruments, each beam-path component is equipped with an identification chip or hole masks read out by light barriers, which uniquely identify the respective component and link it with all its individual stored or coded properties. This information may range from part numbers, usage history or alignment information such as beam offsets, through to a virtually unlimited wealth of any physical data required to configure and operate that particular component. This 'component recognition' feature provides for completely new and important capabilities of laboratory powder diffractometers, the most important of which are:

(*a*) Any beam-path components, and each change of status, can be automatically detected, validated and configured, allowing true 'plug & play' operation.

(*b*) Real-time conflict detection: detection of incompatible, incorrectly mounted or missing instrument components. This feature can also help the user in choosing compatible instrument components, as already discussed above.

(*c*) Automatic, motorized adjustments of beam direction or beam-offset changes, based on the information stored in the related components' ID chips, as individually determined at the factory *via* pre-alignment.

(*d*) Every instrument detail can be saved together with the measurement data, providing for a complete and accurate documentation of the experiment. In principle, every measurement can be exactly reproduced even years later.

(*e*) Measurement instructions can include instrument information. For example, manufacturers or users can configure the measurement software to propose instrument configurations deemed best for particular applications. A user with appropriate rights can choose to enforce a certain instrument configuration so that measurements will not start unless the instrument has detected the required configuration.

Both the platform concept and the huge advances in instrumentation and instrument control software have dramatically changed the laboratory X-ray instrumentation landscape in the past few years. The ease with which an instrument configuration can be changed is not only useful for less-skilled users. Probably even more importantly, it allows the use of the same instrument, in different configurations, for different X-ray application areas. It can generally be said that laboratory X-ray instrumentation has overcome the (mostly historical) dividing lines between different applications, which were mostly between single-crystal diffraction, powder diffraction and thin-film analysis. As far as differences still remain, these are usually solely the consequence of dedicated instrument components for meeting specific application requirements, resulting in specialized measurement and data-evaluation software, which is rarely included with each instrument.

2.1.4.3. *Range of applications*

It is the flexibility of today's X-ray diffractometers that leads to their usefulness for a wide range of X-ray scattering techniques beyond traditional X-ray powder 'Bragg diffraction'. Table 2.1.2 provides an overview.

X-ray scattering techniques represent the vast majority of techniques that X-ray diffractometers are used for. Properly configured, however, the same instrument can also be used to collect X-ray absorption (X-ray radiography) or X-ray emission (X-ray fluorescence) data, even if the achievable data quality cannot compete with dedicated instruments.

For X-ray radiography, an instrument will be configured in transmission geometry with the X-rays projected towards a

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