International Tables for Crystallography (2018). Vol. H, Section 2.1.5.2, pp. 34–35.

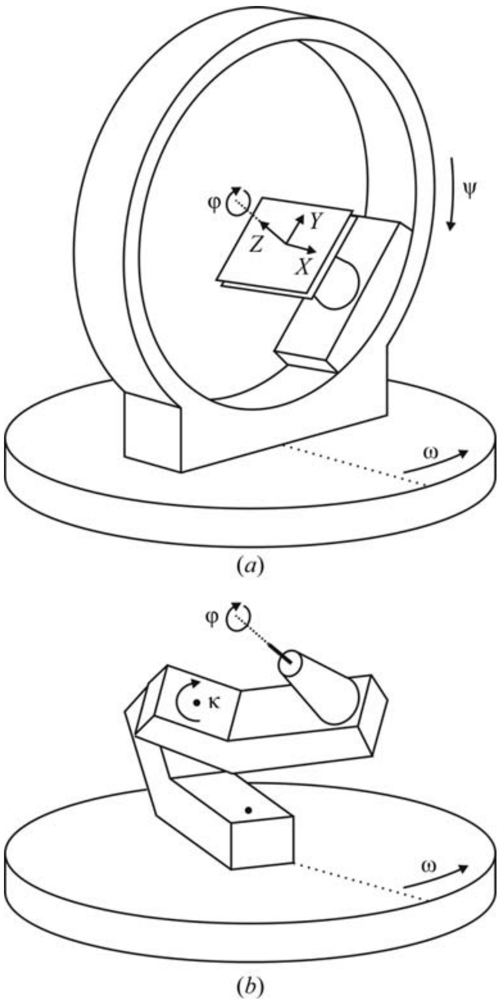

Figure 2.1.9
Geometric definition of the Eulerian and kappa geometries with identical specimen orientation in space. (*a*) Specimen rotation and translation in a Eulerian cradle equipped with an *XYZ* stage, (*b*) specimen rotation on a kappa stage.

improve particle statistics ('wobbling'). Obviously, all the setups shown in Fig. 2.1.8 will work for the full range of X-ray scattering and absorption techniques as discussed in Section 2.1.4.3, leading to the renaissance of the Debye–Scherrer geometry within the past 20 years.

2.1.5.1.2. *Specimen stage*

Depending on the requirements of the application, the specimen stage may offer additional degrees of freedom for specimen rotation as well as X, Y, Z translation. The goniometer base may be configured as ω–2θ as well as ω–θ, and may be oriented vertically as well as horizontally.

To orient a specimen in all possible orientations in space, the specimen stage will offer two more rotational degrees of freedom in addition to the ω and 2θ axes provided by the goniometer base. Such goniometers are known as four-axis diffractometers, with two basic geometries in common use for specimen orientation: Eulerian geometry and kappa geometry.

In the Eulerian geometry the specimen is oriented through the three Euler angles ω (defined by the ω axis of the goniometer base), ψ (psi), and φ (phi). The relationship between the laboratory and rotation axes is shown in Fig. 2.1.9(*a*) for a typical Eulerian cradle. The ω angle is defined as a right-handed rotation about the ω (or Z_L) axis. The ψ angle is a right-hand rotation about the ψ axis, which lies in the diffraction plane and runs parallel to the bisectrix between the incident and diffracted beams. The φ angle defines a left-handed rotation about an axis on the specimen, typically the normal to a flat specimen surface. In some texts the angle χ (chi) is used instead of ψ, with the relationship between the two angles defined as $\psi = 90 - \chi$. Eulerian cradles have the advantage of high mechanical stability and are often integrated with XYZ stages to handle bulky specimens. The geometrical definitions of specimen X, Y, Z translations are also shown in Fig. 2.1.9(*a*).

The kappa (κ) geometry shown in Fig. 2.1.9(*b*) represents an alternative way to orient a specimen in space. The ψ axis of the Eulerian geometry is replaced by the κ axis, which is tilted at 50° relative to the diffraction plane. It supports an arm carrying the specimen, with the φ axis tilted at 50° to κ. The role of the Eulerian ψ rotation is fulfilled by means of combined rotation along κ and φ, which allows Eulerian ψ angles in the range -100 to +100° to be obtained. The absence of the (bulky) ψ circle of Eulerian cradles allows an unobstructed view of the specimen and unhindered access from 'above', for example to mount a cooling device without risk of collision. These two advantages made the kappa geometry popular in single-crystal work. On the other hand, it is not possible to move the specimen to an 'upside-down' position, *i.e.* equivalent to Eulerian ψ angles less than $-100°$ or greater than 100°.

Most goniometers do not offer all six rotational and translational degrees of freedom. The majority of these are actually three-axis goniometers, where the specimen stage offers one additional axis for specimen rotation.

A comprehensive overview of commercially available specimen stages is beyond the scope of this chapter owing to the huge number of dedicated specimen stages available for different kinds of specimen types, levels of automation and non-ambient analyses. The most complete and most current information will be found in manufacturers' product information.

2.1.5.2. *Accuracy and precision*

Particularly high demands are made on goniometer accuracy and precision in Bragg-angle positioning (goniometer base) and specimen orientation (specimen stage). These are usually expressed by the angular accuracy and precision of the goniometer-base axes (ω, 2θ) and the sphere of confusion of specimen positioning in space. A detailed discussion is given by He (2009).

Depending on the application and the actual instrument configuration, additional requirements may be imposed on goniometers, and may limit the maximum accuracy and precision that are achievable. Typical requirements, often not compatible with each other, are:

(*a*) mounting of heavy and bulky beam-path components and specimens;
(*b*) variable goniometer radii, typically ranging from about 15 to 60 cm; and
(*c*) vertical goniometer operation to prevent specimens from falling off the holder.

Each of these requirements may have an impact on goniometer accuracy and precision, and potential early wear and tear. Typical loads range from several kg for fixed-target X-ray sources up to 50 kg and more for moving-target X-ray sources. Small detectors such as point and one-dimensional detectors range from less than 1 kg up to a few kg, while large two-dimensional detectors may weigh up to 50 kg and sometimes even more.

For vertical goniometers, the loads on the main axis bearings can be effectively reduced by counterbalances, as shown in Fig.

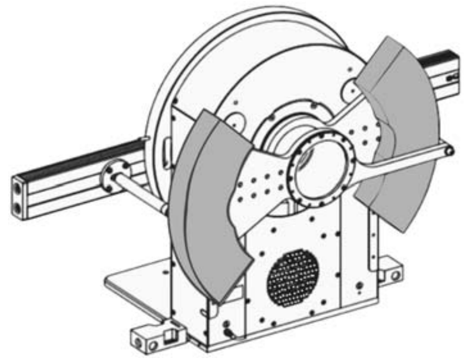

Figure 2.1.10
Example of counterbalancing of a vertical θ–θ goniometer. The counterweights (grey parts) are located at positions matching the weights and locations of the X-ray source and detector. Mounting of different beam-path components with significantly different weight or moving of, for example, the X-ray source and/or the detector to change the respective radii may require repositioning of the counterweights to maintain goniometer accuracy and instrument alignment.

2.1.10 for a goniometer in the ω–θ configuration. Heavy specimen stages may also be supported from below or mounted directly on the bench, disconnected from the goniometer base. However, for heavy beam-path components and larger goniometer radii there is the additional issue of high torques on the incident- and/or the diffracted-beam X-ray optical benches, leading to torsions along the benches. These may significantly deteriorate both the angular accuracy of a goniometer and instrument alignment. For heavy incident-beam-path components such as moving-target X-ray sources, a vertical goniometer base in the ω–2θ configuration is commonly used, as the incident-beam optical X-ray bench is mechanically fixed. For heavy incident- and diffracted-beam-path components a horizontal goniometer base is preferred.

Modern goniometers are equipped with stepping motors and optical encoders, and feature life-span lubrication for maintenance-free operation. The typical accuracy of the two goniometer base axes (ω, 2θ) is of the order of a few thousandths of a degree, with a precision of the order of a few tens of thousandths of a degree. The ψ and φ axes of the specimen stage are mostly used for specimen orientation; the typical angular accuracy and precision are in the range of about 0.01°.

The sphere of confusion of a goniometer is the result of a superposition of all axes and represents the minimum spherical volume covering all possible locations of an infinitely small specimen at all possible orientations. The size of the sphere of confusion depends on issues such as individual axis accuracy and precision, mechanical tolerances, thermal-expansion mismatches, and the weights of the specimen and beam-path components. The sphere of confusion for a two-axis goniometer or a four-axis goniometer with a kappa stage is typically less than 10 µm, and for a four-axis goniometer with a Eulerian cradle less than 50 µm; both values are without a specimen loaded.

Note that the final accuracy of the Bragg angles of the measurement data is mostly determined by instrument alignment, and not by the accuracy specifically of the two goniometer base axes. Optical encoders can measure and control axis positions, but they cannot detect any misaligned or even loose beam-path components. The final data accuracy is determined by the adjustability of an X-ray diffractometer with all its beam-path components. A modern X-ray diffractometer can be aligned to an angular accuracy of equal or better than 0.01° 2θ, which can be checked using suitable standard reference materials (see Chapter 3.1).

2.1.5.3. *Hybrid beam-path systems*

The trend towards multipurpose instrumentation as well as specific application requirements has led to a few specialized goniometer designs. Two major representatives of such designs are (1) multiple-beam-path systems and (2) systems with additional rotational degrees of freedom of beam-path components, such as is required for non-coplanar grazing-incidence diffraction (GID).

2.1.5.3.1. *Multiple-beam-path systems*

Multiple-beam-path systems are usually characterized by integrating more than one beam path on a single goniometer, employing different, complementary beam-path components to meet different application and specimen-property requirements. Mounting two different fixed-target X-ray sources (usually microsources) with different wavelengths (Cu, Mo) is very popular in single-crystal crystallography. Double detector arms are used to mount different types of detectors, most frequently one-dimensional detectors in combination with point detectors. Different X-ray optics can be used to implement different instrument geometries.

A significant driving force behind such multipurpose instrumentation is convenience, *i.e.* to serve a maximum range of applications and specimen types, ideally without the need to manually change the instrument configuration. Indeed, switching between different, preconfigured beam paths may often only require the push of a single software button. However, parallel mounting of different beam-path components raises issues related to the goniometer load and to limitations of angular scan ranges owing to collision issues.

In more recent designs, different X-ray optics have been combined into single motorized modules, allowing switching between different beam paths. Such 'combi-optics' are described in Section 2.1.6.3.4.

2.1.5.3.2. *Non-coplanar beam-path systems*

Non-coplanar (or 'in-plane') grazing-incidence diffraction is a technique for investigating the near-surface region of specimens (ten or fewer nanometres beneath the air–specimen interface). It exploits the high intensity of the total external reflection condition while simultaneously involving Bragg diffraction from planes that are nearly perpendicular to the specimen surface.

As illustrated in Fig. 2.1.11, the incident beam is set at an angle α_I, enabling total external reflection in the coplanar direction (that is coplanar to the diffraction plane); related applications include reflectometry and grazing-incidence small-angle X-ray scattering (GISAXS). 'In-plane' grazing-incidence diffraction

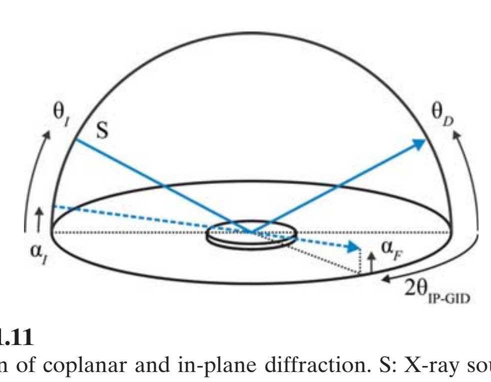

Figure 2.1.11
Illustration of coplanar and in-plane diffraction. S: X-ray source. θ_I, θ_D: incident and diffracted beams for coplanar diffraction. α_I, α_F, $2\theta_{\text{IP-GID}}$: incident-beam angle, exit angle and diffracted-beam angle, respectively, for in-plane grazing-incidence diffraction.