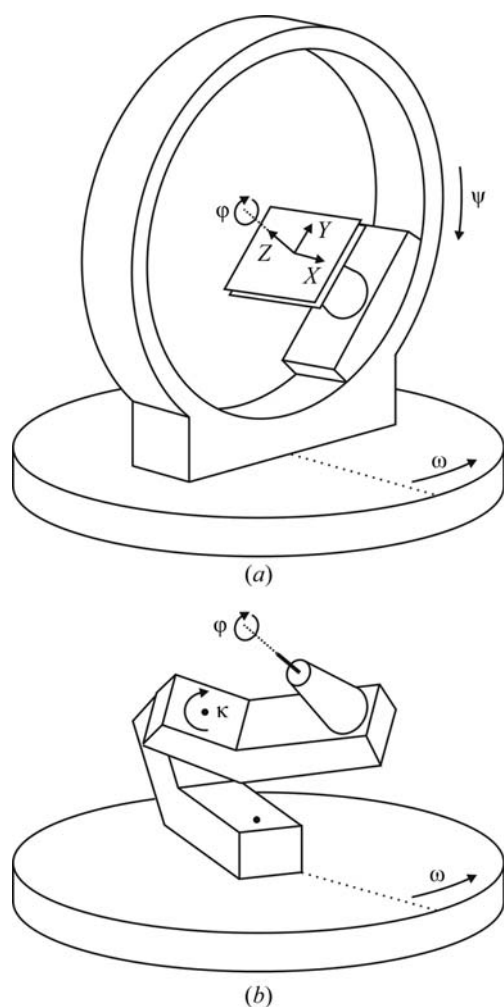


2. INSTRUMENTATION AND SAMPLE PREPARATION

**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 $\omega-2\theta$ as well as $\omega-\theta$, 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 $+100^\circ$ 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:

- mounting of heavy and bulky beam-path components and specimens;
- variable goniometer radii, typically ranging from about 15 to 60 cm; and
- 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.