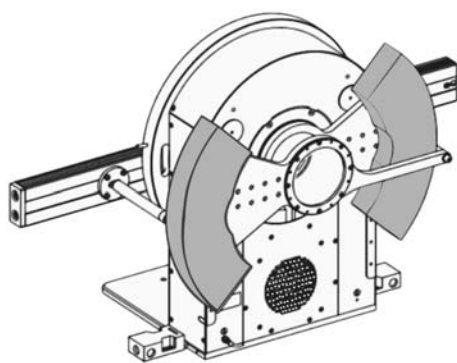


## 2.1. LABORATORY X-RAY SCATTERING

**Figure 2.1.10**

Example of counterbalancing of a vertical  $\theta$ - $\theta$  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  $\omega$ - $\theta$  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  $\omega$ - $2\theta$  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 ( $\omega$ ,  $2\theta$ ) 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  $\psi$  and  $\varphi$  axes of the specimen stage are mostly used for specimen orientation; the typical angular accuracy and precision are in the range of about  $0.01^\circ$ .

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  $10\ \mu\text{m}$ , and for a four-axis goniometer with a Eulerian cradle less than  $50\ \mu\text{m}$ ; 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  $0.01^\circ 2\theta$ , 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  $\alpha_I$ , 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

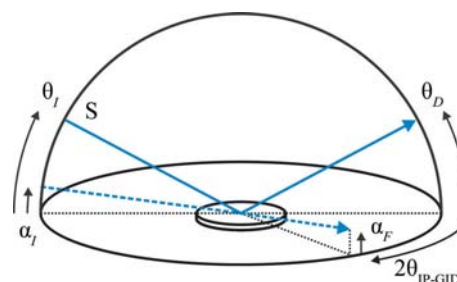
**Figure 2.1.11**

Illustration of coplanar and in-plane diffraction. S: X-ray source.  $\theta_I$ ,  $\theta_D$ : incident and diffracted beams for coplanar diffraction.  $\alpha_I$ ,  $\alpha_F$ ,  $2\theta_{\text{IP-GID}}$ : incident-beam angle, exit angle and diffracted-beam angle, respectively, for in-plane grazing-incidence diffraction.