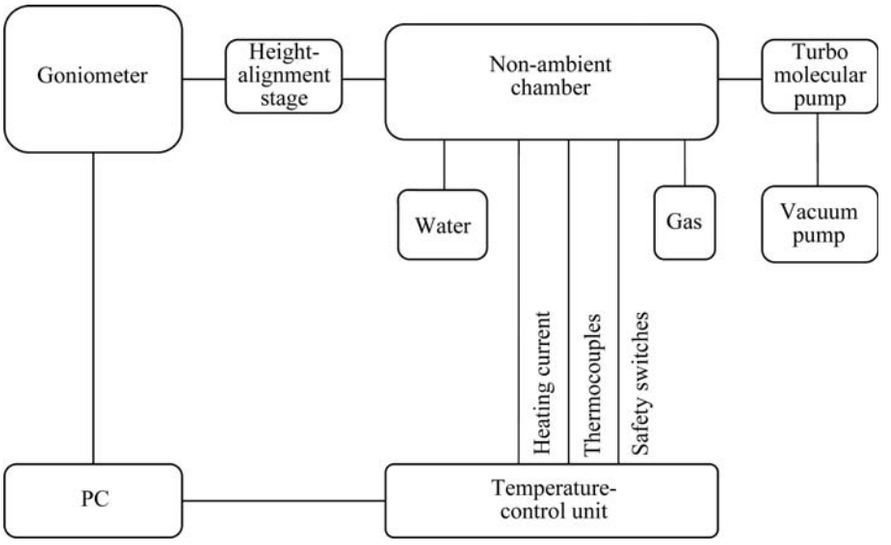

Figure 2.6.1
Typical hardware setup for a non-ambient X-ray diffraction experiment as described in Section 2.6.4; non-ambient chamber, temperature/process-control unit, vacuum/gas equipment, cooling water and goniometer with height-alignment stage connected to a PC.

the value, to send it to the control PC and to control the power for heating/cooling. In addition to controlling the sample conditions, the temperature-control unit (TCU) usually monitors other instrument components such as the cooling of the sample-stage housing and safety devices.

2.6.4.3. *Vacuum equipment, gas supply*

High-temperature X-ray diffraction measurements are often performed in vacuum or in an inert-gas atmosphere to avoid oxidation of the specimen or the sample support. Systems with a rotary pump typically achieve a vacuum of 10^{-2} mbar (where 1 mbar = 100 Pa); when adding a turbo molecular pump to the rotary pump, a vacuum of about 10^{-4} mbar can be reached. A low vacuum or a completely dry gas atmosphere, *e.g.* pure nitrogen (or helium, which has the advantage of a lower background in the diffraction patterns), is also needed for low-temperature experiments to avoid icing problems. Best practice is not to vent the flow of inert gas into the diffractometer enclosure or the laboratory atmosphere, but into the ventilation system (fume hood). Some local safety authorities may require such venting.

2.6.4.4. *Water cooling*

The housing of the sample stage must be kept close to room temperature to avoid heat transfer to the diffractometer and to ensure user safety. In most cases, water is used for this purpose, and the cooling water can be shared with the diffractometer.

2.6.4.5. *Diffractometer and height-compensation mechanism*

The non-ambient chamber has to be interfaced to the goniometer. Interfaces are available without and with a height-compensation mechanism; the latter can be manual or motorized.

When heating/cooling a specimen in an environmental heater, sample displacement is virtually unavoidable, mainly owing to the thermal expansion/contraction of the sample holder. It is possible to correct the temperature-dependent change of the sample position with a height-compensation mechanism (motorized z stage) or to model the displacement in the refinement software. When using a z stage that is controlled *via* software, the shifts in peak positions are only caused by the thermal lattice expansion/contraction of the sample under study. If no such mechanism is available, a parallel X-ray beam (which is not sensitive to sample displacement) can be used, but the resolution may be worse compared with measurements in para-focusing Bragg–Brentano geometry, and granularity may be significant. For strip heaters the displacement of the sample due to the strip is not so pronounced. If a peak of the material of the strip is visible in the diffractogram this can be used as a reference for height compensation if the thermal expansion of the strip material is also taken into account.

2.6.5. Specimen properties

In designing a non-ambient experiment the specimen properties must be taken into account; the holder material should not react with the sample. For flat sample geometry it is preferable that the specimen completely absorbs the X-ray beam. If the specimen is highly transparent, one can either use a thin specimen on a zero-background sample holder or use a capillary. For capillary measurements the X-ray beam must penetrate the capillary completely; if this is not the case, higher energy X-rays (such as Mo or Ag) can be used (Section 2.6.7.2). Every sample is unique, and a suitable solution must be devised.

2.6.6. High-temperature sample stages

A typical laboratory non-ambient setup consists of a non-ambient sample stage, often called a temperature chamber. The sample stage is mounted on a goniometer, preferably in a θ–θ configuration (Fig. 2.6.2). In this case the sample stays horizontal and there is no need to fear melting of the sample with the possibility of it dripping off/out of the sample holder.

A temperature-control unit, vacuum equipment, gas supply and water cooling have to be added to the system before it can be operational.

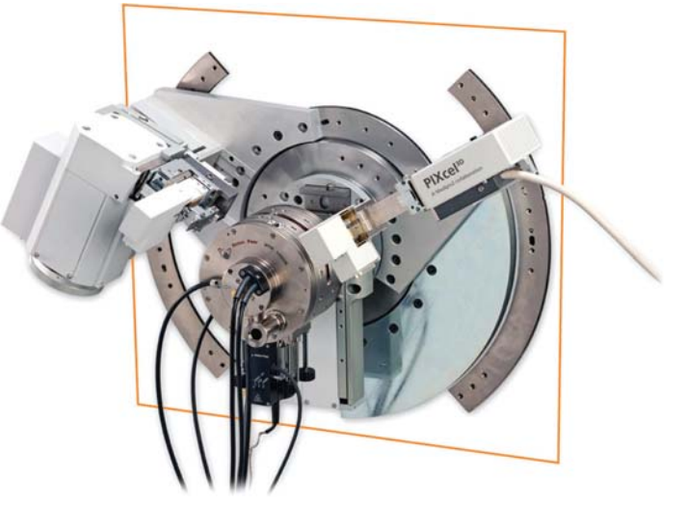

Figure 2.6.2
An Anton Paar HTK 1200N high-temperature oven chamber on a PANalytical Empyrean system equipped with a PIXcel3D detector.