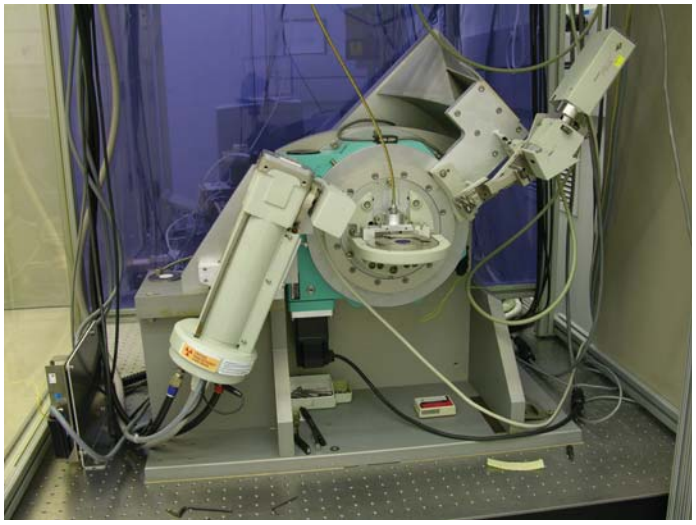

Figure 3.1.23
The X-ray powder diffractometer designed and fabricated at NIST, in conventional divergent-beam format.

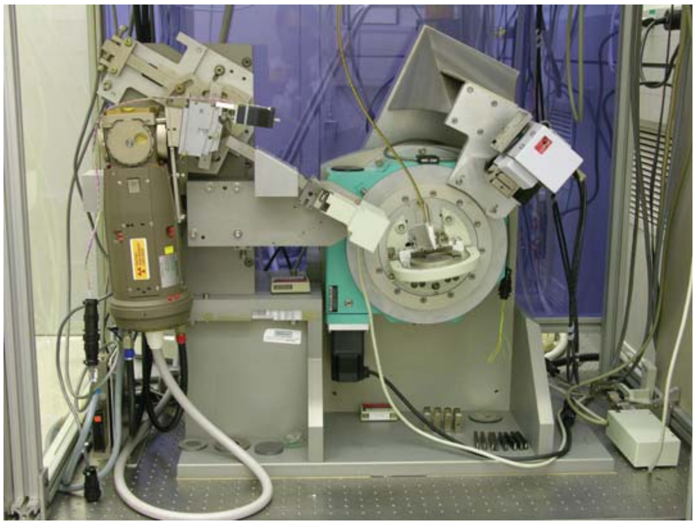

Figure 3.1.24
The NIST-built powder diffractometer configured with the Johansson incident-beam monochromator and a position-sensitive detector.

the instrument set up in conventional geometry with a post-monochromator and point detector, while Fig. 3.1.24 shows the setup with a Johansson IBM and a PSD. Data from these two configurations are discussed below.

The goniometer assembly, which is of θ–2θ geometry, uses a pair of Huber 420 rotation stages mounted concentrically with the rotation axes horizontal. The stage that provides the θ motion faces forward while the 2θ stage faces rearward; they are both mounted on a common aluminium monolith, visible in Figs. 3.1.23 and 3.1.24, which forms the basis of the chassis for the instrument. Both stages incorporate Heidenhain 800 series optical encoders mounted so as to measure the angle of the ring gear. With 4096-fold interpolation provided by IK220 electronics, an angle measurement to within $\pm 0.00028^\circ$ (1 arc second) was realized for both axes. The stages are driven by five-phase stepper motors that incorporate gear reducers of 10:1 for the θ stage and 5:1 for the 2θ stage, yielding step sizes of 0.0002° and 0.0004°, respectively. The manufacturer's specifications for the Huber 420 rotation stage claim an eccentricity of less than 3 µm and a wobble of less than 0.0008° (3 arc seconds). The construction of the goniometer assembly necessitated the development of a specialized jig to align the two 420 rotation stages with regard to both the concentricity (eccentricity) and parallelism (wobble) of their rotation axes. The result was that the overall eccentricity and wobble of the assembly met the specifications cited for the individual stages. The flexing of the detector arm, attached to the rearward-facing 2θ stage, was minimized by fabricating a honeycombed aluminium structure, 7.6 cm deep, which maximized stiffness while minimizing weight. Furthermore, the entire detector-arm assembly, including the various detectors, was balanced on three axes to minimize off-axis stress on the 2θ rotation stage (Black *et al.*, 2011). Thus, the goniometer assembly is exceedingly stiff and offers high-accuracy measurement and control of both the θ and 2θ angles.

The optics, graphite post-monochromator, sample spinner, X-ray generator and tube shield of the machine were originally components of a Siemens D5000 diffractometer, *circa* 1992. As previously discussed, the parts for the IBM configuration were obtained primarily from a Siemens D500, *circa* 1987. Both configurations include a variable-divergence incident-beam slit from a D5000. The PSD used in this work was a Bruker LynxEye XE. The cable attached to the sample spinner (as seen in Figs. 3.1.23 and 3.1.24) is a flexible drive for the spinner itself; the remote location of the drive motor (not shown) isolates the sample and machinery from the thermal influence of the motor. The machine was positioned on an optical table within a temperature-controlled (± 0.1 K) space. The temperature of the water used for cooling the X-ray tube and generator was regulated to within ± 0.01 K. Operation of the machine was provided through control software written in *LabVIEW*. Data were recorded in true x–y format using the angular measurement data from the optical encoders.

In conventional configuration, the 2.2 kW copper tube of long fine-focus geometry was operated at a power of 1.8 kW. This tube gives a source size of nominally 12×0.04 mm, while the goniometer radius is 217.5 mm. The variable-divergence slit was set to $\sim 0.9^\circ$ for the collection of the data discussed here. This results in a beam width, or footprint at the lowest θ angle, on the sample of about 20 mm, conservatively smaller than the sample size of 25 mm. A Soller slit with a divergence of 4.4° defined the axial divergence of the incident beam. A 2 mm anti-scatter slit was placed approximately 113 mm in front of the 0.2 mm (0.05°) receiving slit. The total path length of the scattered radiation (the goniometer radius plus the traverse through the post-monochromator) was approximately 330 mm. This setup reflects what is thought to be a medium-resolution diffractometer that would be suitable for a fairly broad range of applications and is therefore a reasonable starting point for a study of instrument calibration. With the IBM, the 1.5 kW copper tube of fine-focus geometry was operated at a power of 1.2 kW. This tube had a source size of nominally 8×0.04 mm. The variable-divergence incident slit was also set to 0.9° with a 0.2 mm (0.05°) receiving slit. The receiving optics were fitted with a 4.4° Soller slit. The total beam-path length was about 480 mm.

With the scintillation detector, data were collected using two methods, both of which encompassed the full 2θ range available with these instruments and for which the SRMs show Bragg reflections. The first involves data collection in peak regions only, as illustrated in Table 3.1.2 for SRM 660b. The run-time parameters listed in Table 3.1.2 reflect the fact that the data-collection efficiency can be optimized by collecting data in several regions, as both the intensity and breadth vary systematically with respect to 2θ. This was the manner in which data were collected for the certification measurements of SRMs 660c, 640e and 1976b. The