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E9: The Fine Resolution Powder Diffractometer (FIREPOD) at BER II

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Abstract: The E9 (FIREPOD) is an upgraded fine resolution powder diffractometer for elastic neutron scattering, obtaining high quality data sets for Rietveld analysis, structure solution and phase analysis under ambient conditions as well as in situ at low / high temperatures, magnetic fields, gas pressure and various atmospheres.

1 Introduction

The Fine Resolution Powder Diffractometer E9 (FIREPOD) is an angle-dispersive powder diffractometer optimized for a flat resolution function with a minimum width of the reflections at the 2Θ-region with the highest density of reflections. The monochromator is placed at a distance of 11 m from the reactor core, which allows for a large take-off angle at the monochromator. An evacuated beam tube and a sapphire single crystal filter reduce air scattering and epithermal neutrons. Neutron flux at the sample is increased by an adjustable vertically focusing Ge-monochromator.

The detector consists of eight individual DENEX ^{3}He 2D detectors with 300 x 300 mm active area each and a common radial collimator to reduce background noise. The individual detectors are arranged in a novel setup, at optimized, non-constant distances from the sample. Five of the individual detectors can be placed close to the sample in a high intensity conformation. Data collection with fixed detector position measures parts of the 2Θ-range with increased intensity and without loss in quality.

Position-sensitive data integration of the Debye cones results in a strongly reduced asymmetry of the

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peaks. The 2D-data are directly accessible, allowing the early detection of preferred orientation or spottiness.

Depending on sample scattering power and volume and the resolution settings of the instrument, full powder diffraction patterns of a quality suitable for Rietveld refinement can be collected as fast as 30 minutes. With small 1 cm^3 samples and high resolution, between 1 and 6 hours should be estimated, depending on the scattering power of the sample. Scans measuring only a selected angular with fixed detector position can be as fast as 10 minutes per step for good scatterers.

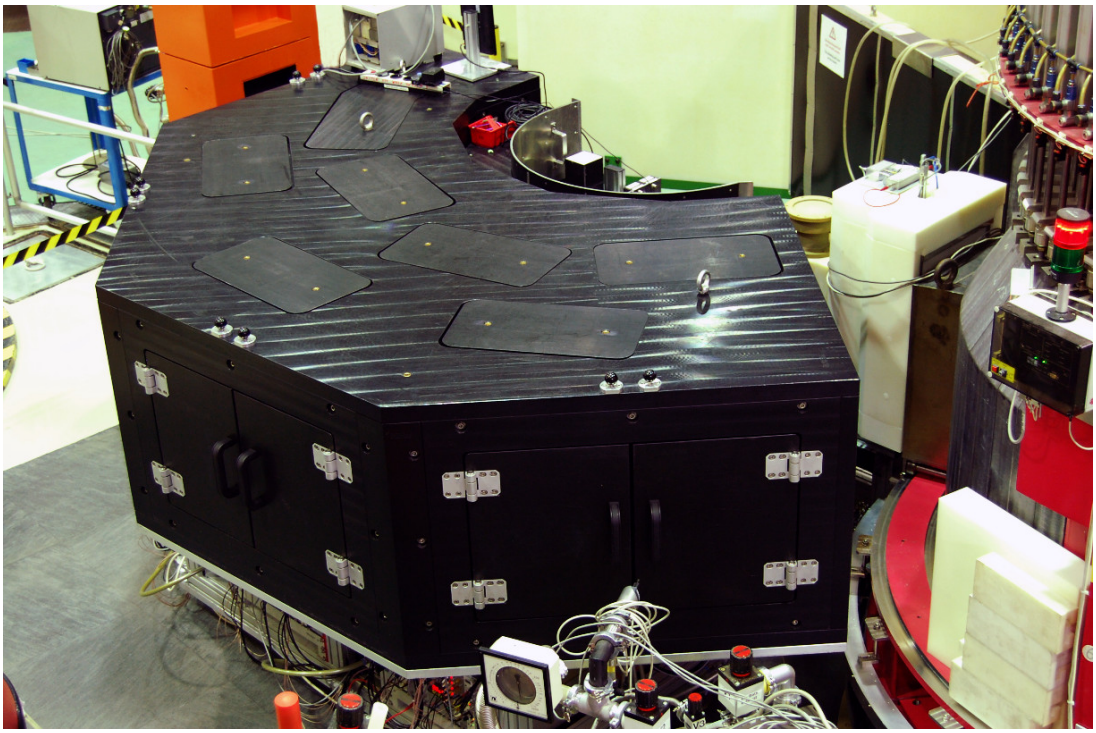

Figure 1: View of E9.

2 Instrument application

Typical applications are:

- Crystal structure determination
- Rietveld refinement
- Site occupation factors, e.g. of isoelectronic elements
- Determination of light atoms (e.g. H, Li)
- Rapid parameterized scans of selected angular regions of the diffraction pattern, e.g. temperature or magnetic field strength
- Non-destructive bulk phase analysis

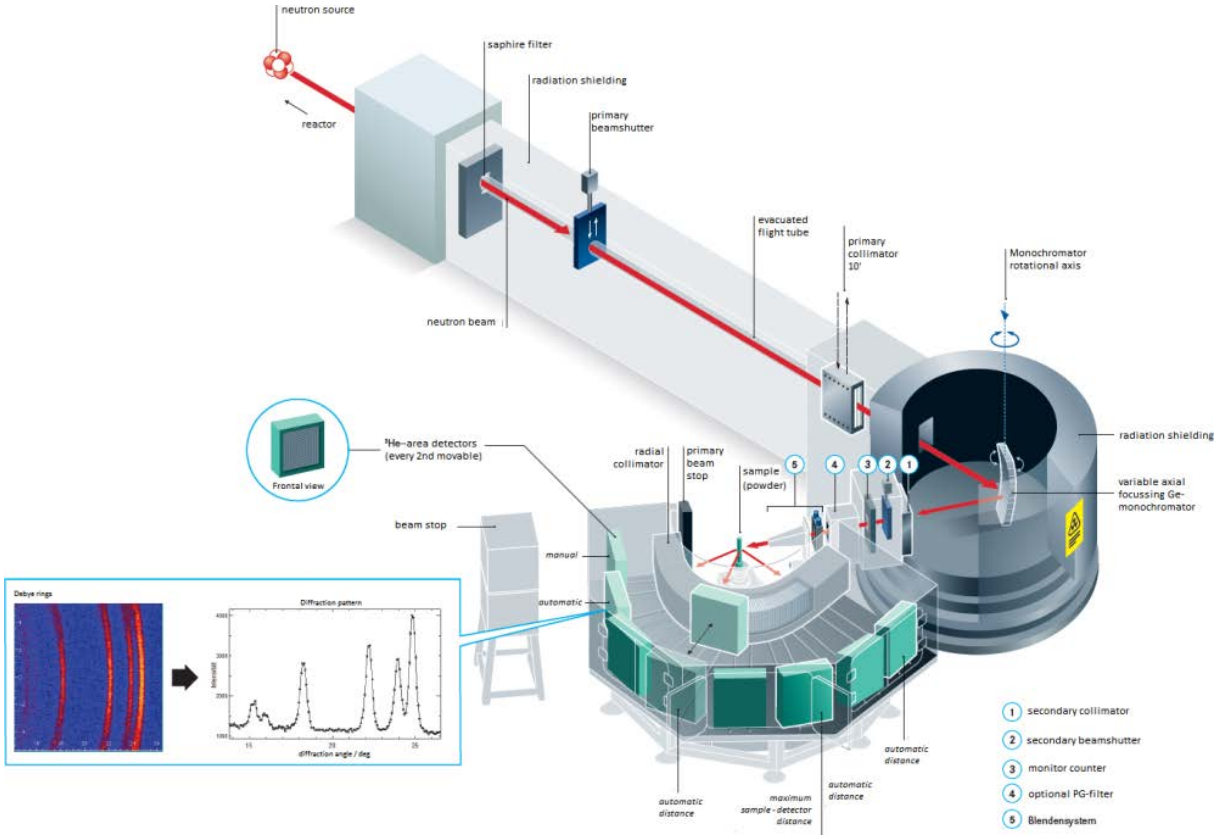

Figure 2: Schematic drawing of E9.

3 Technical Data

Beam tube	T5
Collimation	α1: 10' or 18' α2: 20'
Monochromator	Axially focusing, Risødesign, 300 mm height 19 Germanium composite plates with reflecting planes (311), (511), and (711) (511) normal to crystal surface Mosaicity FWHM = 17
Take off angle of monochromator	$50° \leq 2\Theta \leq 130°$ 111.7(1)° is used by default
Wave length	λ = 1.3084(2) Å from Ge(711) λ = 1.7982(1) Å from Ge(511) λ = 2.8172(2) Å from Ge(311) & PG filter
Flux	$\approx 10^5$ n/cm^{2}s
Range of scattering angles	$3° < 2\Theta < 142°$
Angle resolution	0.33°
Range of lattice spacing	25 Å< d < 0.7 Å from Ge(711) 35 Å< d < 1.0 Å from Ge(511) 55 Å< d < 1.5 Å from Ge(311)
d resolution	$\approx 2{\cdot}10^{-3}$
Sample size	1 cm^3 - 5 cm^3
Detector	Eight area detectors (300 mm x 300 mm) Oscillating radial collimator for background reduction
Polarized neutrons	No
Instrument options	Variable sample – detector distance for five of the individual area detectors
Sample environment	OS, OF, HTF, VM3, VM5, DEGAS, RTSC
Software	CARESS, BEAN

Table 1: Technical parameters of E9.

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