

# Multi-Analyser Spectrometers & Milli-Kelvin Crystal Temperature Stabilisation

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## Abstract

*ESRF beamlines ID16 & ID28 are dedicated to high and very high energy resolution inelastic X-ray scattering experiments. During the last 2 years an important upgrade has been made on the very high energy resolution horizontal spectrometers at both ID16 & ID28, and also the Raman spectrometer at ID16. All 3 of these spectrometers now operate routinely with 9 analyser crystals.*

*This paper gives an overview of the mechanical & optical design of these 3 spectrometers, and of the very high energy resolution analyser crystals with their milli-kelvin temperature stabilisation.*

## 1. Introduction

ESRF beamlines ID16 & ID28 are dedicated to inelastic scattering experiments, focusing on the study of collective ion excitations in condensed matter. ID16 also has an end station dedicated to inelastic x-ray scattering from electronic excitations (the Raman spectrometer). The general layout of ESRF Beamline ID16 is shown in figure 1, and the optical schematic is shown in figure 2. (note: ID28 is very similar optically)

There is a high heat load silicon 1,1,1 pre-monochromator at 51m from the source. The x-ray beam is further monochromatised by a backscattering monochromator at 72m from the source. This has a flat silicon crystal operating at a Bragg angle of  $89.98^\circ$  and utilising a high order energy reflection (e.g. 11,11,11). It produces a monochromatic beam with a relative energy resolution of  $2 \times 10^{-8}$ . The beam is then focussed by mirrors and Be lenses to the sample position at 42m from the source. Behind the sample there is the very high energy resolution horizontal spectrometer with a 7m arm and 9 analyser crystals.

Alternatively the Raman spectrometer can be installed in the last hutch in place of a long flight tube. In this case the backscattering monochromator is not used, but there is the possibility of using a post-monochromator located just after the pre-monochromator.

This paper will concentrate on these 2 spectrometers for ID16. The very high energy resolution horizontal spectrometer at ID28 is very similar to the one at ID16.

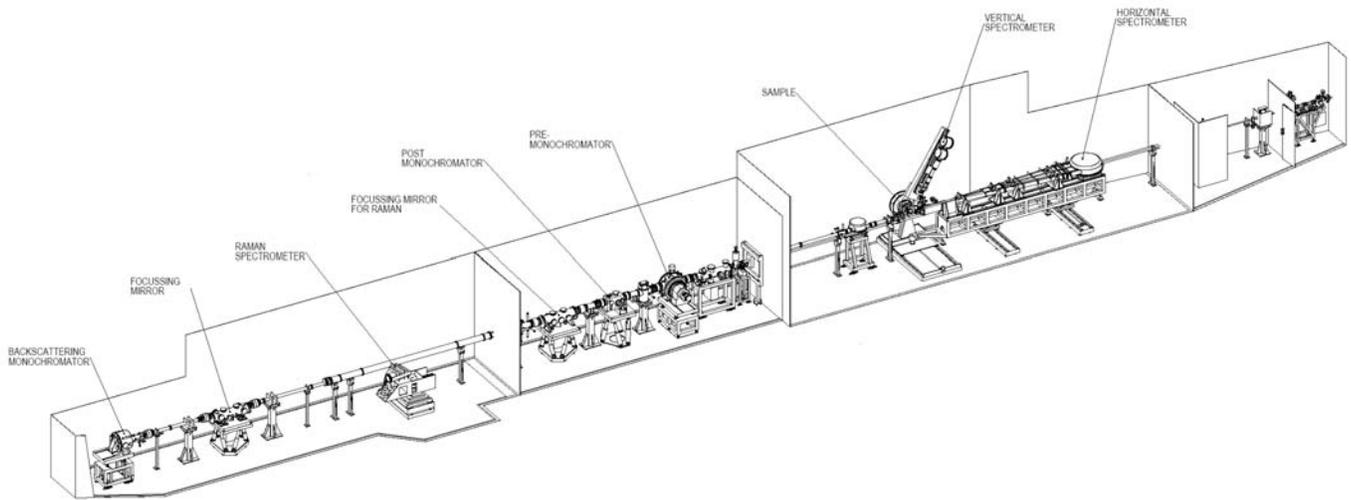


Figure 1: General Layout of ESRF Beamline ID16

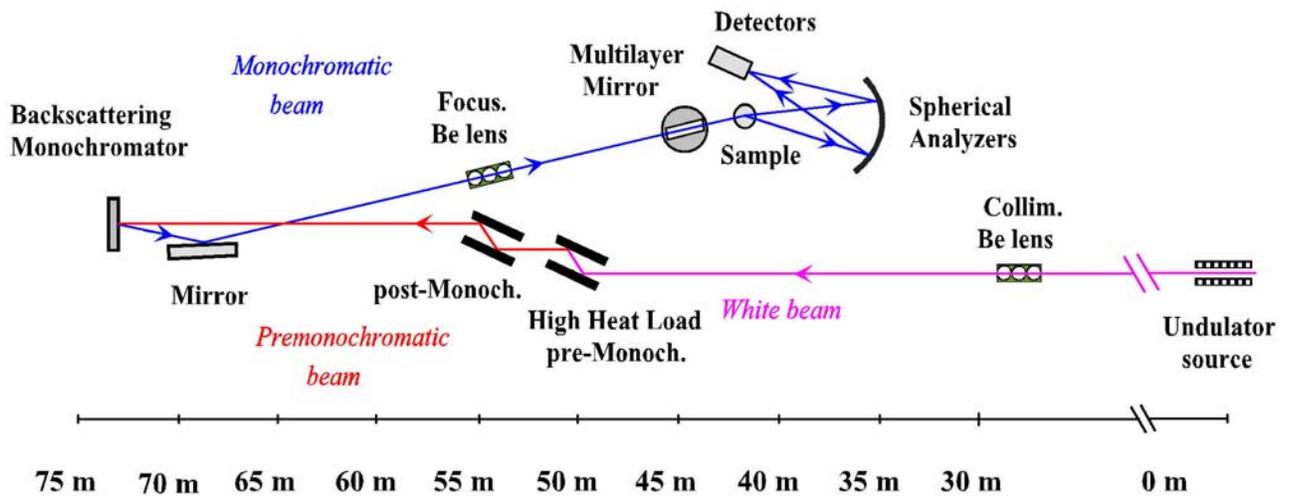


Figure 2: Optical Schematic of Beamline ID16

## 2. The Very High Energy Resolution Spectrometers

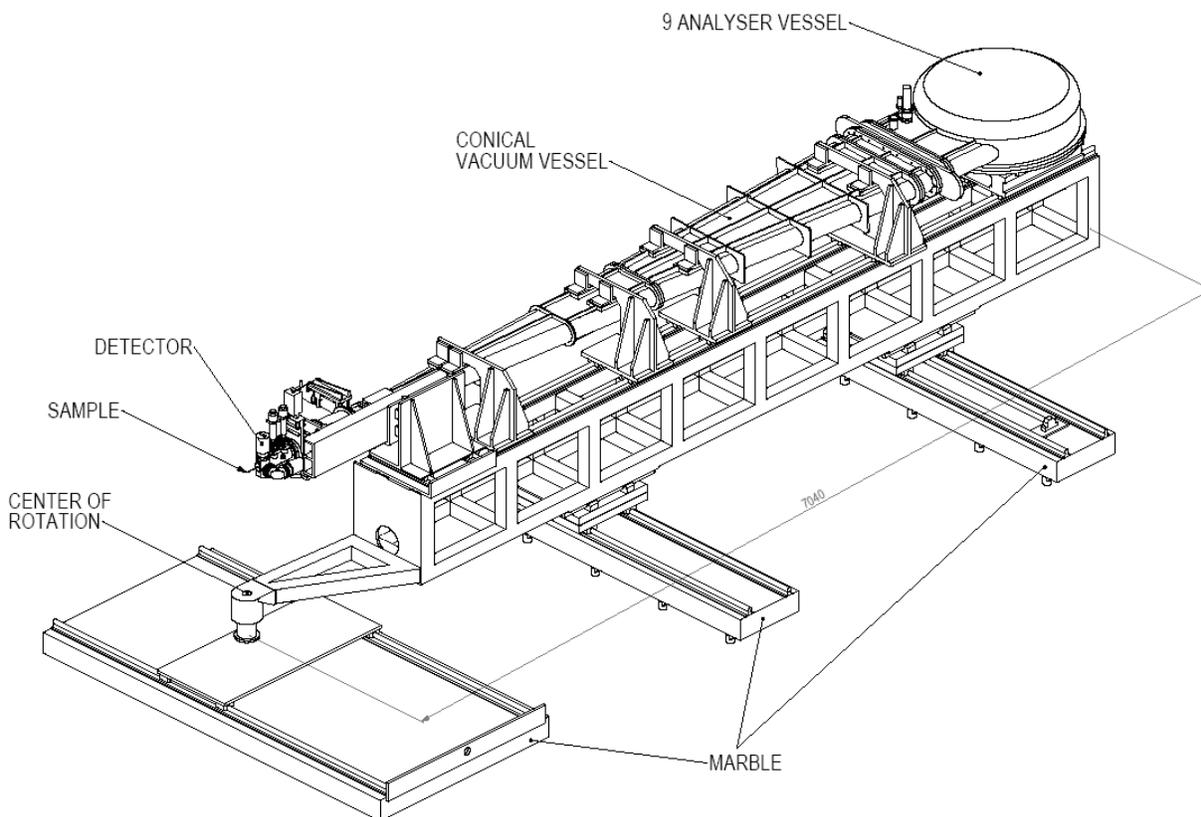


Figure 3 : The very high energy resolution spectrometer (ID16)

The ID16 spectrometer shown in figure 3 consists of a 7m long arm rotating in the horizontal plane through an angle of 13 degrees (50 degrees for the ID28 spectrometer where the experimental hutch is much larger). It is equipped with 9 spherical analyser crystals located inside a vacuum vessel at the end of the 7m arm, and two 5-element silicon diode detectors near the sample. Thus there is the capability to record 9 spectra simultaneously. A long conical vacuum vessel with bellows connects the analyser vessel to the detector vessel. All these components are supported by a rigid steel structure which is moved along rails on 3 marble slabs by a motorised leadscrew, 3 sets of rails and 3 large rotational bearings. The center of rotation is directly below the sample position. A turbo pump provides the vacuum of  $10^{-5}$  mbar inside the vessels.

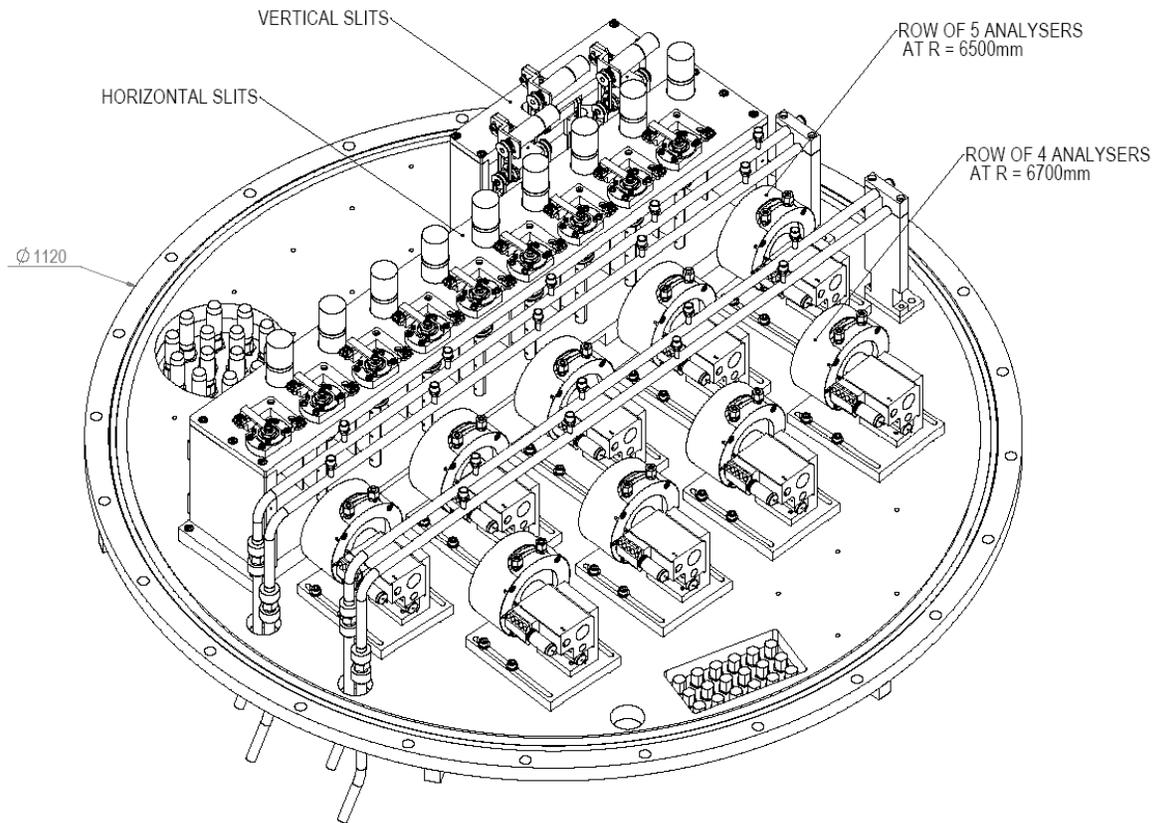


Figure 4 : Inside the ID16 9 Analyser Vessel

The 9 analysers are arranged in a first horizontal row of 5 analysers at a nominal radius of 6500mm from the sample, and a second row of 4 analysers at 6700mm as shown in figure 4. The angle between adjacent analysers is  $0.77^\circ$  and  $6.16^\circ$  between the 2 extreme analysers. The radial distance of each analyser assembly can be adjusted manually  $\pm 50$ mm to optimise the focal distance. The analyser assemblies are mounted onto the vessel base flange of 25mm thickness and 1120mm diameter. The top part of the vessel is easily removable to allow access for installation, alignment, and maintenance. Vacuum sealing is provided by a viton o-ring.

Just before the analysers there are motorised horizontal slits for all 9 analysers, and motorised vertical slits for only the first 3 analysers. Typical slit settings are  $20 \times 60 \text{ mm}^2$  or  $60 \times 60 \text{ mm}^2$  (horizontal x vertical). Electrical feedthroughs (Lemo with viton o-ring) for all the 30 stepper motors and for the temperature stabilisation of the 9 analyser crystals are located in the vessel base flange. Cooling water for the 9 analyser crystals is distributed via 2 manifolds located just above the analysers and flexible plastic tubing (not shown in figure 4) to/from each analyser assembly.

Each analyser crystal is mounted in an assembly as shown in figure 5. The silicon crystal, which is 100mm in diameter, is made up of some 10 000 individual cubes of  $0.8 \times 0.8 \text{ mm}$  section glued onto a spherical silicon

substrate. Each individual cube surface simply reflects the x-ray beam onto the detector giving a nominal beamlet size of 1.6 x 1.6 mm at the detector. The spherical surface of the crystal ensures that all the beamlets converge at the detector. The crystal works in a backscattering geometry with a high order reflection (e.g. 13,13,13) very close to 90° (actually 89.98°) giving an energy resolution of only a few meV. The temperature of the crystal is stabilised to within 1mK (see section 4) with a special thermal mount and control electronics.

The crystal & thermal mount are assembled onto a custom made compact 2 rotation goniometer, allowing the alignment of the x-ray beam onto the detector. (Commercially available goniometers have been used in the past but these are physically much larger & provide an angular movement far greater than required.) This goniometer is driven by 2 ESRF mini-jacks mounted on orthogonal axes, and it has a commercial spherical bearing acting as the center of rotation. The ESRF mini-jacks are stepper motor driven linear actuators with 10mm stroke and linear resolution 87 nm. The angular resolution of the goniometer is 1.8 µrad (half step of motor).

The detector vessel (figure 6) has two 5 element silicon diode detectors supplied by Canberra Eurisys Mesures (Strasbourg, France). Each detector element receives the focalised x-ray beam from one single analyser. Thus it is possible to record 9 spectra simultaneously. (The 10<sup>th</sup> detector element is not used.) Two arrays of pinholes with an opening of 3 x 3 mm located just before the detector prevent the cross over between x-ray beams from adjacent analysers. These pinholes have motorised yz movements for inserting in the x-ray beam and for alignment. Tantalum screens shield the detectors from the scattered beam from the sample.

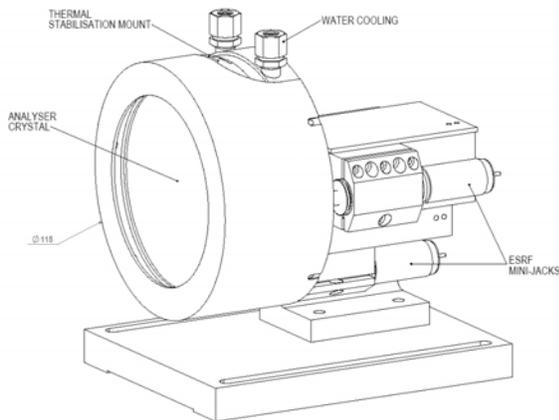


Figure 5 : Analyser Crystal Assembly

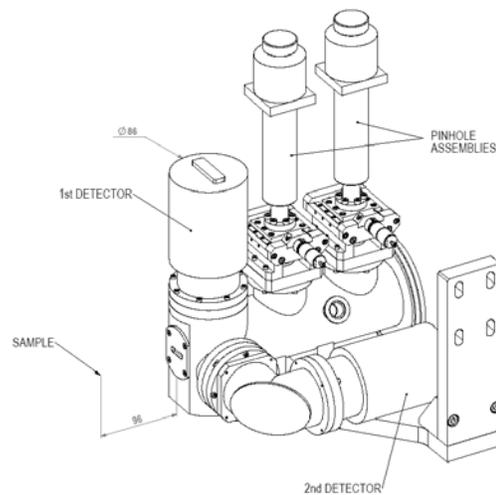


Figure 6 : Detector Vessel Assembly

### 3. The Raman Spectrometer

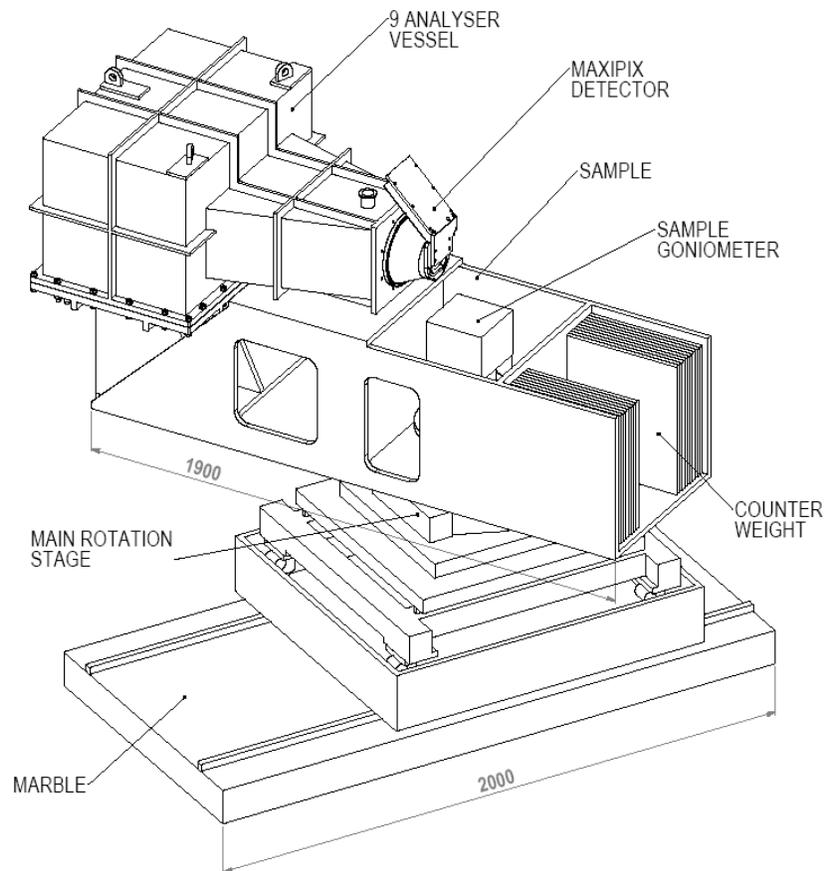


Figure 7 : The Raman Spectrometer

The raman spectrometer (figure 7) consists of a 1m arm rotating in the horizontal plane through a maximum angle of 150 degrees. The rotation is made by a stepper motor driven Huber 440 stage. Below this stage there is a custom made motorised YZ table for alignment, and rails on a marble table for inserting the spectrometer into the beam path. The 9 analyser vessel & its support are manufactured in aluminium to reduce the weight on the rotation stage. A substantial counter weight is never the less required. A turbo pump provides the vacuum of  $10^{-5}$  mbar. A sample goniometer provides independent rotations & translations for the sample.

The 9 analyser crystals are at a nominal radial distance of 1m from the sample and are arranged in a 4+4+1 array as shown in figure 8. The spherically shaped silicon crystals are 100mm in diameter and are also working in a backscattering geometry. Each crystal is mounted onto a similar 2 rotation goniometer to those of the horizontal spectrometer. An additional linear stage with +/- 50mm travel allows the sample to crystal distance to be adjusted to optimise the focus. However the temperature stabilisation is not required as the Raman experiments do not require very high energy resolution. Just before the analyser crystals there are motorised horizontal slits, and also a single motorised vertical slit for the single analyser on the right .

The MaxiPix detector, supplied by the ESRF detector group, is a multi-pixel silicon diode allowing spectra from all 9 analysers to be obtained simultaneously.

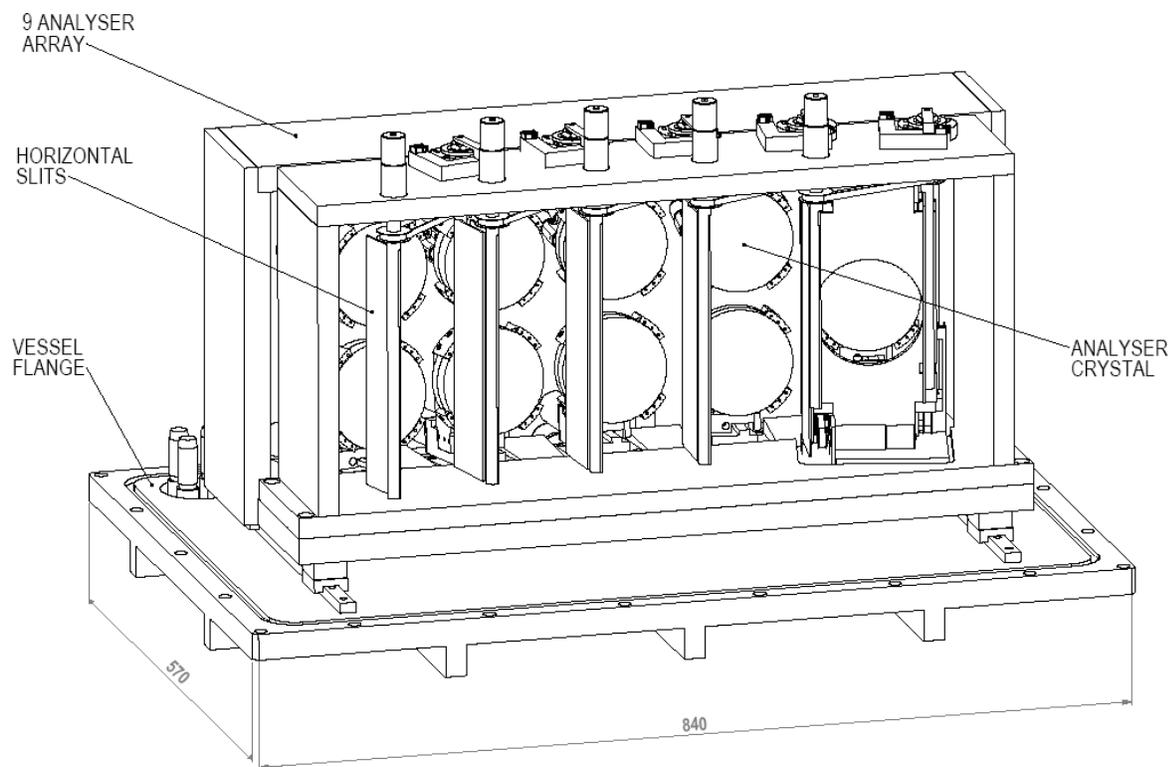


Figure 8 : Inside the Raman 9 Analyser Vessel

#### 4. The Very High Energy Resolution Analyser Crystal Temperature Stabilisation

In order to achieve a very high energy resolution of the order of  $\Delta E/E = 10^{-8}$ , the temperature of the analyser crystal has to be controlled to within 1mK to avoid catastrophic thermal dilitation of the crystal d-spacing. According to Bragg's law  $E=hc/2d \sin\theta_B$ , and therefore, at fixed  $\theta_B$ :

$$\Delta E/E = \Delta d/d = \alpha \Delta T ,$$

Considering the value of  $\alpha = 2.58 \times 10^{-6} / ^\circ\text{K}$  at ambient temperature, and the necessary resolving power  $\Delta E/E = 10^{-8}$ , the temperature of the monochromator and the analyzer crystal has to be controlled with mK precision.

The analyser crystal thermal mount & temperature control system is shown schematically in figure 9.

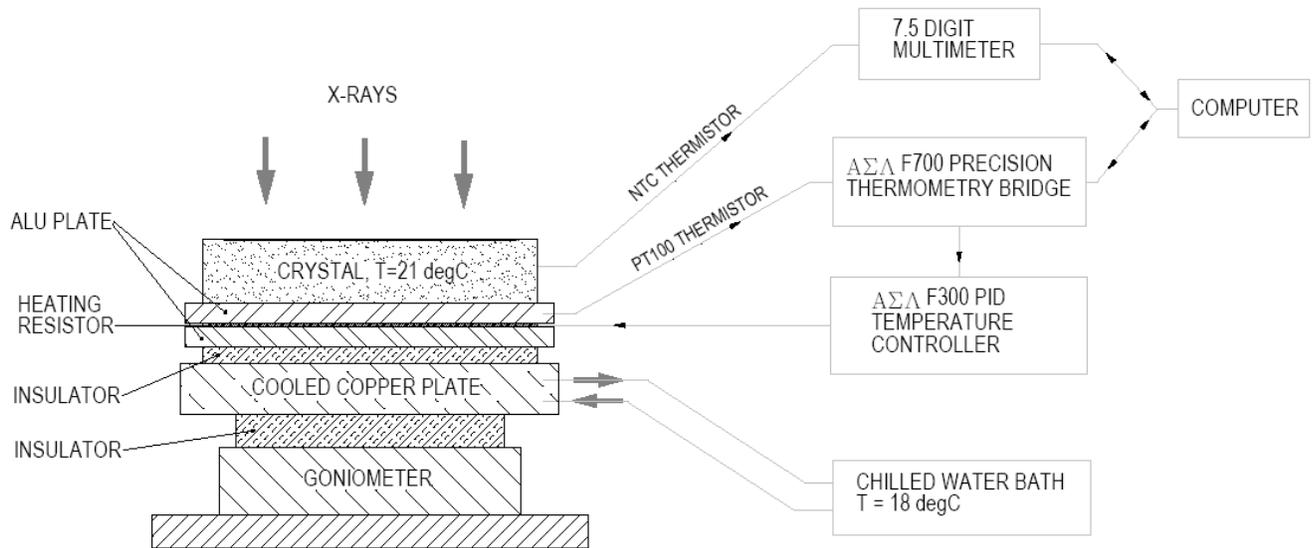


Figure 9 : Analyser Crystal Thermal Mount & Temperature Control System

The thermal mount assembly has a “cold” water cooled copper plate which serves as a reference base temperature at around 18°C, with a stability of 0.1°C or better. The crystal is “warmed” to around 21°C by a resistive heater sandwiched between 2 aluminium alloy plates. A thin thermal insulator between the copper plate & the aluminium plates reduces the leakage heat flow between these parts. Another thicker insulator isolates this thermal mount from the goniometer. This assembly sits in a vacuum vessel so there are no convection currents. Also as the crystal is at ambient temperature there is negligible thermal radiation. The experimental hutch is thermally stabilised to within 0.1°C.

The temperature control system consists of a feedback loop composed of a very high Precision Thermometry Bridge reading a 100 Ω platinum resistance (Pt100) and a (Proportional Integrative Derivative) PID controller connected to the heating resistor. The Thermometry Bridge measures the ratio of two, four terminal resistors,  $R_t$  and  $R_s$ . The internal reference resistor  $R_s$  is mounted in a temperature controlled oven and it has a value of 100 *ohms* ± 50 *ppm* with a short term stability better than ± 1 *ppm*. The Thermometry Bridge is computer controlled and, in its high-sensitivity mode, allows to control the temperature to a minimum step of 0.25 *mK*.

The feedback Pt100 resistance, connected to the Bridge, is located in a small hole on the upper aluminium plate not too far away from the heating resistor. The temperature variations in the crystal are continuously monitored with a 11 kΩ NTC (Negative Thermal Coefficient) thermistor.

The Pt100 and the NTC thermistors are particularly suited to our applications because they have small dimensions, react very fast to the temperature variations, can resolve temperature steps smaller than 1/1000 of degree, and have a good thermal stability around room temperature. The thermistors are monitored utilising a 7½ digit multimeter interfaced with the computer. The temperature stability of the crystal is better than ± 1 *mK* per hour.