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A helium-flow cryostat (3 to 300 K) for neutron four-circle spectrometry

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Résumé. — On décrit un cryostat à circulation continue d'hélium possédant une géométrie sphérique et permettant une rotation complète de 360° des 3 angles d'Euler. Le cryostat n'augmente pas l'étendue des zones inaccessibles dues au berceau d'Euler. On peut atteindre et maintenir constante, sans gradient, une température entre 3 K et 300 K, à l'aide d'une régulation contrôlée par ordinateur. Le temps de réponse de l'ensemble est de quelques minutes même pour les températures les plus hautes. La consommation d'hélium liquide reste acceptable, grâce à l'emploi d'une ligne de transfert à faibles pertes thermiques, qui utilise des conduites d'hélium suspendues magnétiquement. L'ensemble est utilisé à l'ILL depuis 5 ans pour des nombreuses études de physique du solide.

Abstract. — We describe a continuous-flow cryostat system with spherical geometry and full 360° freedom of motion for all three Euler angles. The cryostat does not increase the dead angular zones of the Euler-cradle itself. Any sample temperature between 3 K and 300 K can be set and kept gradient-free and constant by a computer controlled temperature regulation. The time constant of the device is in the range of a few minutes, even at higher temperatures. These performances are obtained with a reasonable helium consumption owing to a special low-loss transfer line with magnetically suspended helium ducts. The device has been operated routinely at the ILL for five years for a variety of solid state studies.

1. Introduction.

The problem of having variable low temperatures down to 4 K for samples mounted on Eulerian cradles arises mainly from the fact that the required cryostats have to be both very small and mobile around three independent axes of rotation. Standard helium bath-type cryostats are therefore useless and one has to consider continuous-flow-cooling. This implies the development of both low-loss cryostats operating in any orientation and of flexible siphons which are able to bring the liquid helium from the storage Dewar to the cryostat with a minimum of losses. A similar cryostat has already been in use at the ILL and has been described before [1]. Its He-transfer line had two flexible joints which allowed for limited cryostat motion only and required a moving helium vessel. The minimum temperature was 8 K at a helium consumption of 2 l/hour.

A movable cryostat able to reach a minimum temperature of 3.9 K at a consumption of 3.6 l/hour has also been described [2]. It is adapted for offset χ -circles only which present the disadvantage of having considerable dead angular zones. This cryostat

also has a cylindrical sample environment further restricting the possible angular settings.

Cryorefrigerators represent a very elegant alternative and are widely used both for X-ray and neutron diffractometers. But the compact single stage machines which can easily be adapted to a 4-circle geometry only reach temperatures down to 45 K. Until now commercial multi-stage refrigerators reaching 10 K are generally too bulky to be mounted inside standard neutron Eulerian cradles except offset γ -circles thus creating cumbersome blind angular areas which are particularly felt when azimuthal scans are to be performed.

Below 10 K the only non helium consuming method would be a double stage refrigerator coupled to a Joule-Thomson cooler. Although such devices are commercially available they are still too bulky to be envisaged for four-circle uses. The cryostat described here has been operated at the ILL since 1978 mainly for temperatures below 10 K and certain magnetic phase transitions could be studied down to 2.6 K. It features further advantages over the above quoted devices which makes it competitive even for certain experiments at higher temperatures. These properties

are extremely good temperature homogeneity within the sample chamber ; very low helium consumption above 45 K (≤ 5 l/day) ; quick response-time especially useful when temperature steps are computer programmed and finally, using a spherical construction and an articulated siphon with two rotating joints allowing a complete 360° rotation for all angles (ϕ moves inside) and no relevant extra blind zones than those inherent to the circle. The siphon is in the incident beam for $-5 < \omega < 5^\circ$ but this angular region is practically unused because of neutrons scattered by the air molecules. If required small azimuthal rotations may be used to keep ω always above 5 degrees.

2. The articulated siphon.

Particular attention has been paid to minimize the losses of the siphon. This siphon of a total length of nearly 4 m must be able to transfer liquid helium to a cryostat which executes full 360° rotations about a vertical axis (ω) and a horizontal axis (χ) (see Fig. 1). Both rotations are achieved using cooled Johnston [3] joints. They can be made practically loss free by cooling them with a very small amount of He-gas extracted from the main flow. The helium capillaries and the inside walls of the vacuum tubes are gold

Fig. 1. — The cryostat with its special He-siphon installed on the D10 Eulerian cradle. The arrows indicate the two rotating Johnston's, the circles the corners with magnetic spacers.

plated and we have used no mechanical spacers to maintain the capillaries in position. Instead two corners of the siphon are equipped with magnetic spacers using small SmCo_4 permanent magnets [4]. This way the losses are due to radiation only. They could in principle be reduced by cooled intermediate shields all along the siphon but this was judged too delicate for the present cryostat to be operated on a user basis.

3. The cryostat (Fig. 2).

The basic idea is to reduce the motion of the body of the cryostat itself to two axes of rotation only by having the sample-holding ϕ -shaft rotating inside the cryostat with the sample end at low temperature and the motor driven end at room temperature. A hollow Invar shaft with inside a small heat exchanger for the outlet helium gas can easily take such a temperature difference without significant losses. The maximum thermal contraction of this shaft is of the order of 0.01 mm and can be neglected.

Fig. 2. — Schematic diagrams of the cryostat/siphon system showing the various components explained in the text.

The helium flow entering the cryostat is separated into two branches (Fig. 2). The main flow (He gas) traverses a heat exchanger and leaves the cryostat. The second branch of flow is through the inner sphere of the cryostat containing the sample. For the lowest temperatures a cylindrical phase separation box with the cylinder axis parallel to the χ rotation axis and a flow constrictor guarantee helium gas at 4.2 K (or less if we pump on this circuit) at the entrance of the sample chamber independently of the angular position of the cryostat. This solution works well in practice since the cryostat can be turned upside down without variation of the sample temperature. Slight

temperature excursions are observed during the motion itself. We also observe a slight variation of the helium consumption depending on the position of the cryostat.

The response time of the cryostat is rather fast and of the order of a few minutes for a temperature change of 10 K. Therefore and in conjunction with the programmable temperature setting the cryostat is well suited for quick measurement of temperature dependent properties.

The sample size can be from less than 1 mm³ to a few cm³. The inner sample sphere ($\varnothing$ 6 cm) the cold shield ($\varnothing$ 8 cm) and the outer vacuum sphere ($\varnothing$ 12 cm) are made of thin walled (0.8 mm) aluminium. Using diaphragms before detector or analyser one can easily avoid any scattering from the Al-spheres even for the largest samples.

4. Temperature regulation and control.

A resistive 290 Ω CLTS⁽¹⁾ sensor placed on the main heat exchanger in the vicinity of a wound thermo-coax heater is used for temperature regulation. CLTS was chosen for its extremely small size and very fast thermal response well adapted to the fast response of the cryostat. Its main drawback is the poor reproducibility which is compensated here by a very stable 25 Ω RhFe sample thermometer and a computer controlled regulation which continuously rescales the CLTS setting using the RhFe reading as a reference. Both sensors, wired in the 4-wire mode, have enough sensitivity to be used over the total temperature range of 2.6 to 300 K. The PID regulator used here uses a PROM memory to linearize the CLTS response; PID parameters are set manually. The temperature set-point can be given manually (in K) or via the computer terminal, the regulator giving an interrupt signal when equilibrium is reached. The PDP 11/34 computer of the spectrometer is used for this purpose as it is essentially inactive during the temperature setting sequences. The RhFe resistor value is transformed into temperature with a Chebycheff polynomial routine⁽²⁾. The overall accuracy of the reading

is better than 0.05 K. The stability is 0.02 K above and 0.1 K below 10 K. During the years a number of known phase transformations with well characterized critical temperatures have always been found at the correct temperatures.

5. Operation and performance.

The cryostat is permanently used at the ILL on the D10 thermal Triple-Axis Four-Circle spectrometer. Since 1978 around 70 [5] different experiments have been performed. The only difficulty encountered using the cryostat was the tightness of the indium seal for the inner Al-sphere. Due to thermal cycling this seal sometimes leaks which requires opening-up the cryostat and changing it. To speed up this operation a special fast clamp has been developed to replace the early 12-screw seal.

Although a small adjustable head with three orthogonal adjustments to centre the sample is available, users find it usually simpler to precentre their sample onto standard holder-pins. No decentring as a function of temperature is observed (Invar ϕ -shaft).

Under laboratory conditions the liquid helium consumption for a sample temperature of 4.2 K is 2 l/hour. The mean real-life consumption turned out to be 2.5 l/hour for the sample at 4 K. He-vase exchange is easy and takes a few minutes. Sample heating-up during this procedure is of the order of 10 K, nominal sample temperature is recovered within minutes.

The type of experiments performed include standard low temperature structure determinations including the collection of large data sets on small samples (for example, three weeks at 3.9 K for an organic conductor), investigations of phase transition problems with good temperature resolution. Also more delicate experiments on diffuse scattering or weak satellite patterns have been successfully performed owing to the absence of parasitic cryostat scattering.

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- [5] See for example Experimental reports in ILL Annual Reports.

⁽¹⁾ CLTS : Compensated Linear Temperature Sensor supplied by : Micro Measurements, Romulus, Michigan, USA.

⁽²⁾ Calibrated by Cryogenic Consultants LTS, Pitchkott, Nr. Aylesbury, Bucks., HP22 4HT England, tel. 029-664-259.