

Product Information

LOW TEMPERATURE ATTACHMENT (COOLING GAS NOZZLE FOR PROTEIN MEASUREMENT) FOR AUTOMATIC FOUR-CIRCLE X-RAY DIFFRACTOMETER AFC

1. Introduction

Low temperature attachment is available as one of the attachments for the Rigaku AFC (single crystal X-ray diffractometer system for structure analysis). This attachment has so far been used to suppress thermal vibrations of specimen, to examine structural changes, such as phase transition, of crystals due to temperature, to conduct structure analysis of crystals which are unstable at room temperature, and so on. Recently, however, with an increase in structure analysis of protein crystals and the like substances, more emphasis is being laid on the reduction of radiation damage to the sample in the use of this type of attachment. To cope with the trend, we at Rigaku have newly developed a "coaxial type low temperature nozzle" suitable for protein crystal measurement. The outline of the device is described below.

2. Outline of Device

Fig. 1 through Fig. 3 show photos of the "mov-able type low temperature nozzle" mounted on the AFC goniometer. The photos illustrate the position of the nozzle, as it moves, at $\chi=0^\circ$, $\chi=+90^\circ$, and $\chi=-90^\circ$ respectively. The nozzle is fixed to the inner side of the χ -circle of the goniometer so that it

revolves according to the movement of the χ -circle. The nozzle is so designed that a cooling gas will always flow in the ϕ axial direction, allowing operation over the temperature range from -30°C to the ambient temperature. In the case of the Rigaku's existing low temperature attachment Model A-3 (Fig. 6), its major portions including the transfer tube are made of stainless steel, and this attachment is usable over the range from -160°C (min. -178°C) to ambient.

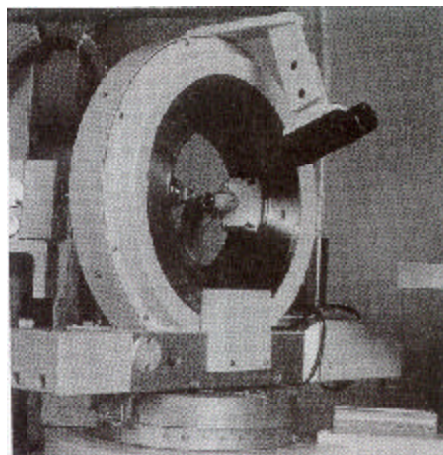


Fig. 2. Position of the Movable Type Low Temperature Nozzle at $\chi=90^\circ$.

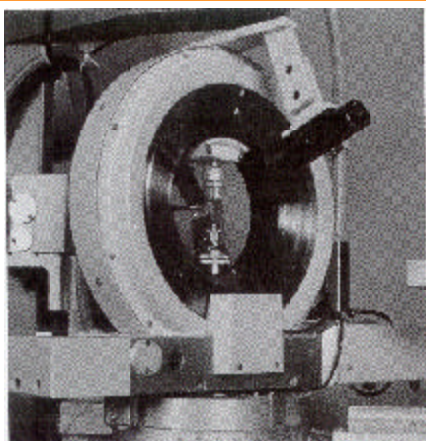


Fig. 1. Position of the Movable Type Low Temperature Nozzle at $\chi=0^\circ$.

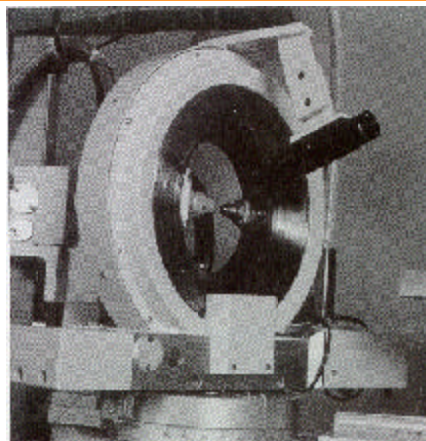


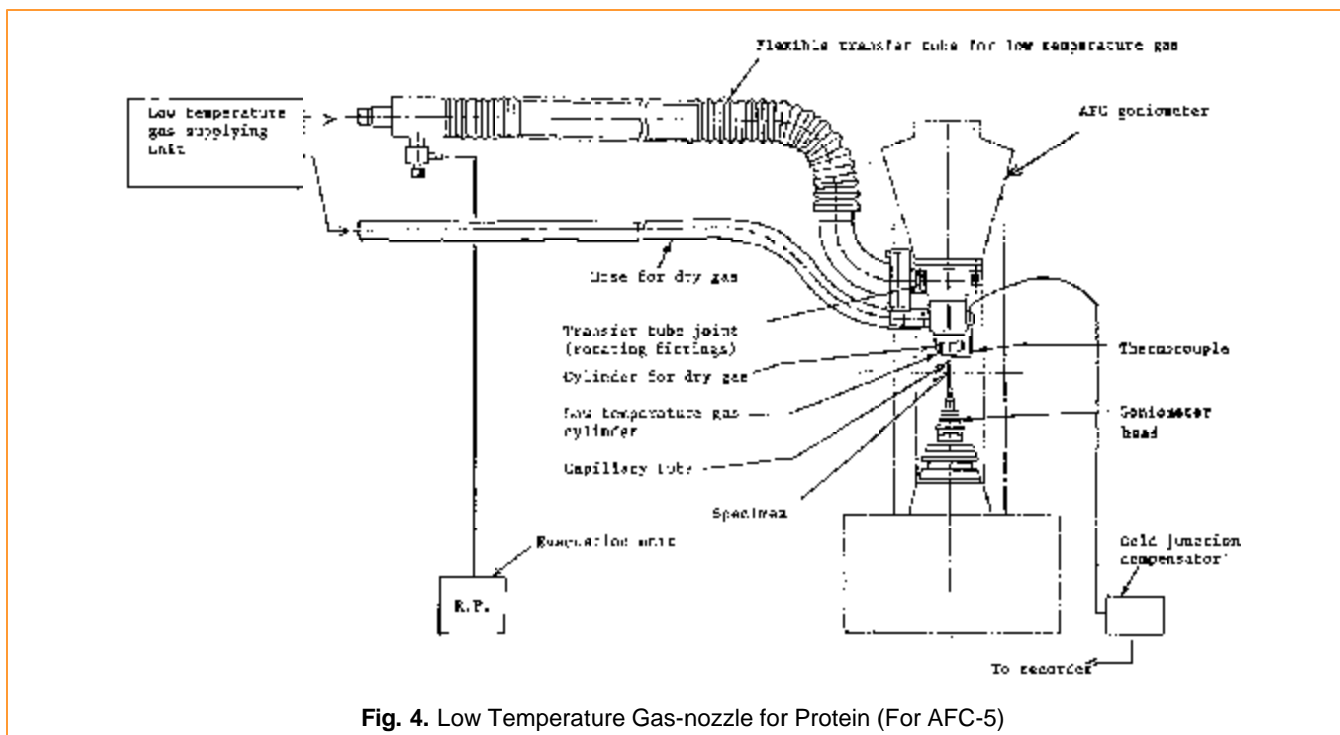
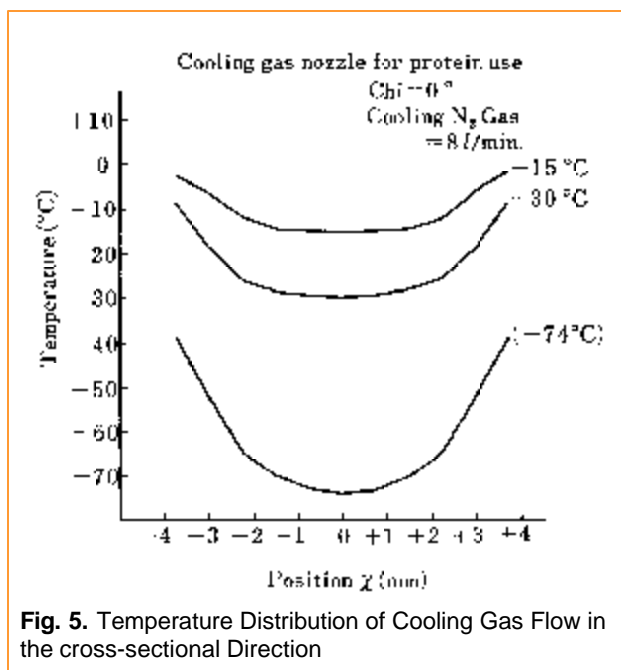
Fig. 2. Position of the Movable Type Low Temperature Nozzle at $\chi=-90^\circ$.

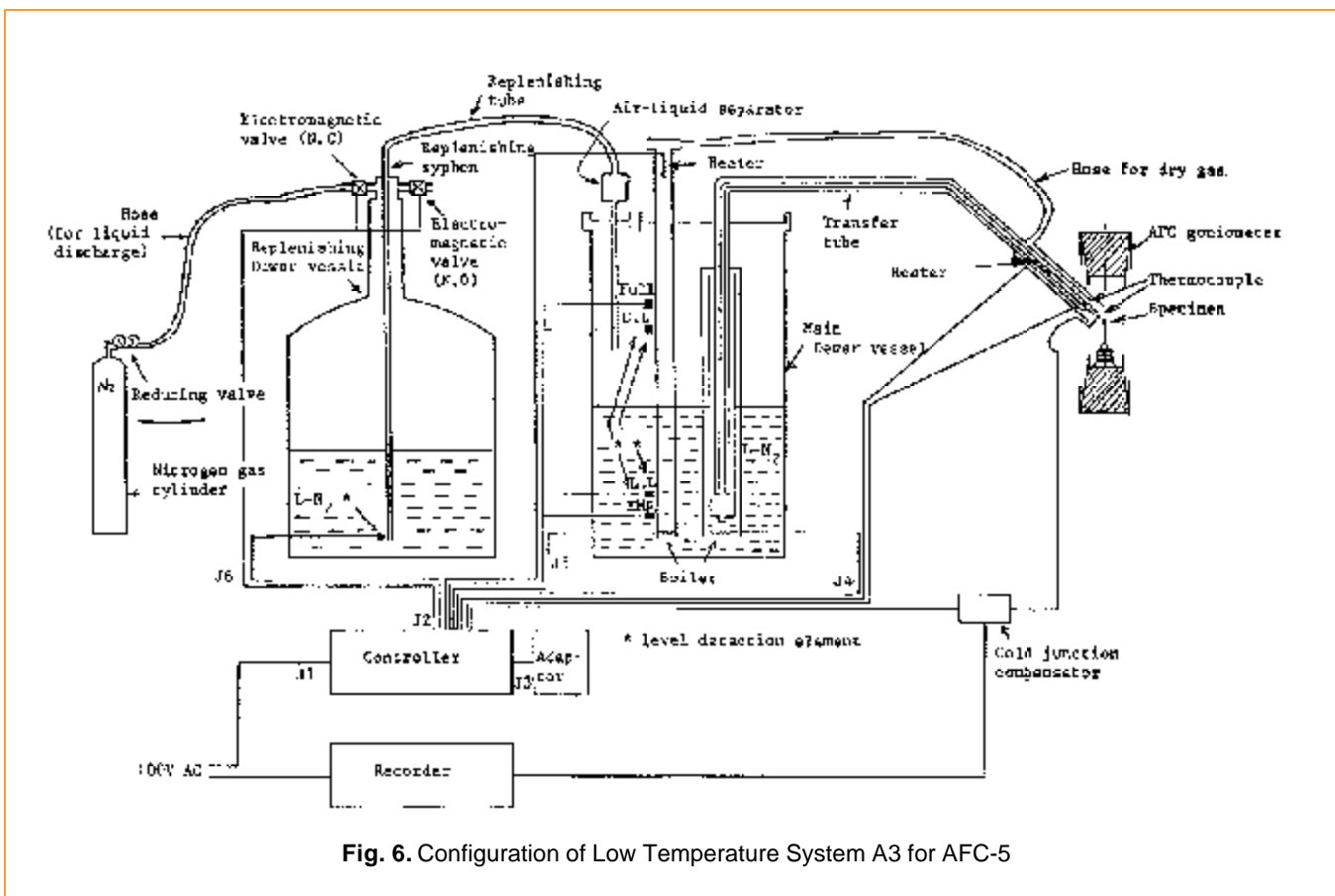
But it is of a fixed nozzle type and as such, while it is well effective for the conventional measurement technique where the sample is fixed to the tip of a glass filament, it is inadequate for those crystals like a protein crystal to be positioned together with a mother liquor at the center inside a capillary tube. The "movable type low temperature nozzle", on the other hand, can also cope with such a crystal mounting method without causing a frost problem.

As shown in Fig. 4, "the movable low temperature nozzle" has a double cylinder construction in which the inner small cylinder is used for a low-temperature cooling gas stream. It is 7 mm in inside diameter and its front end is positioned at about 12 mm away from the X-ray irradiating position of the crystal. The outer large cylinder is used for a dry gas stream. It is 16 mm in inside diameter and is used at -5°C or less temperatures. The tip of the nozzle is equipped with a thermocouple for temperature monitoring to enable monitoring of its temperature at all times on the chart recorder.

As may be seen in Fig. 5, an exceedingly small thermal gradient can be obtained in an area of approx. 3mm dia. with the "cooling gas nozzle" even at around -30°C . The thermal stability depends also on that of the cooling gas source, being roughly 1/2 of the latter (e.g. $\pm 2^{\circ}\text{C}$ when a cooling gas source of $\pm 1^{\circ}\text{C}$ stability is employed).

Further, a pipe which connects the "cooling gas source" and the "low temperature nozzle" with each other is a "flexible transfer tube" of a vacuum double piping system. It is 1500mm long and is made of stainless steel. This flexible transfer tube can freely bend according to the motion of the goniometer to follow its moving position during measurement to feed a cooling gas to the "low temperature nozzle".





3. Outline of Specifications

[cooling gas nozzle for protein use]

Temperature:

-30°C-ambient

Thermal stability:

±2°C (depending on the stability of the cooling gas source)

I.D. of low temp. nozzle:

7 mm dia.

Gas flow direction:

Movable in the ϕ axial direction

Angular limitation:

In conjunction use with the model AFC-5R (χ -circle dia. 190 mm):

2 θ axis: -30° ~ +161°

ω axis: -15° ~ +39°

χ axis: -90° ~ +90°

ϕ axis: No limitation

4. Concluding Remarks

As explained, the system configuration of this low temperature attachment is such that the "low temperature nozzle", "the pipe section" and the "cooling gas source" are respectively separable from one another quite independently. Accordingly, it is possible to operate this system by utilizing the existing cooling gas source and piping on the user's hand and only by newly adding the low temperature nozzle. The cooling gas source referred to here does not necessarily need to be a low temperature device using liquid nitrogen. We presume a dry gas refrigeration system combined with an adiabatic hose is also operable. We expect this low temperature nozzle may find a wide range of application for the time ahead.