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# A Liquid Nitrogen Cryostat for Optical and X-Band Electron Spin Resonance Measurements

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A sirnple and versatile liquid nitrogen cryostat is described. The characteristics of the systern are presented, and the operation of the systern for optical and electron spin resonance (ESR) measurements, with the sample in vacuurn, is described.

Apresenta-se criostato, simples e versatil, para nitrogênio líquido . Discutem-se suas características e descreve-se a operação do sistema para medidas ópticas e de ressonância eletrônica de spin em amostras em vácuo.

In solid state investigations, especially in color centers research, it is often useful to make optical and ESR rneasurernents of an irradiated sample,, at low temperature, without warming it up. We report here on a liquid nitrogen temperature cryostat designed for such measurernents in solid samples. Other advantages of this system are its great versatility and simple execution.

The dewar is conceived to fit either a Varian V 4533 X-band rotating cavity or a Varian V 4531 multi purpose cavity. It also fits almost all optical absorption spectrophotometers.

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A detailed drawing of the dewar is shown in Fig.1. The vertical dimension of the dewar is limited by the distance between the wave guide flange and the top of the rotating cavity. Its diameter is such that it allows an easy handling in a 3" gap magnet. In order to fit the rotating cavity, and have the maximum available volume, the cold finger is placed off center.

The inner and outer vessels are made of copper, and the top cover and insulating tube are made of stainless steel. The insulating tube has to be thin enough in order to minimize thermal conduction through it, but not so thin as to loose mechanical rigidity. The thickness used was between .15 mm and .20 mm. The vacuum port may be of brass or other nonmagnetic material.

In order to minimize radiation losses, the inner walls must be clean and polished. Also, to avoid oxidation of the walls, the dewar must be stored always in vacuum or, still better, silver or gold plating of the inner walls can be made.

The volume of the $LN_2$ vessel is 80 $cm^3$, and the precooling volume is about 150 $cm^3$. When the dewar is pumped to a vacuum of the order of $10^{-5}$ torr, the liquid nitrogen consumption is of 10 $cm^3$ per hour. When the vacuum valve is closed, the $LN_2$ consurnption increases, as the inner vacuum degenerates with time. The total evaporation of the liquid nitrogen, in this condition, takes about 2 1/2 hours. By using about 2 gr of zeolite absorber in the inner vessel, and baking the system, this time can be extended up to 4 hours or more. This duration time is usually enough for optical and ESR measurements, so that the vacuum pump may be disconnected during the measurements. This brings a great simplification in the sample orientation procedure, and an improvement of the signal to noise ratio in the ESR measurements, as the vacuum pump always introduces sample vibration in the cavity.

The dewar can be fitted to the different tails with the sample in vacuum by means of the "tail exchange chamber" shown in Fig.2. The dewar under vacuum is introduced through the top of the chamber, and the sealing o-rings are then fitted to the tail. When the chamber pres-

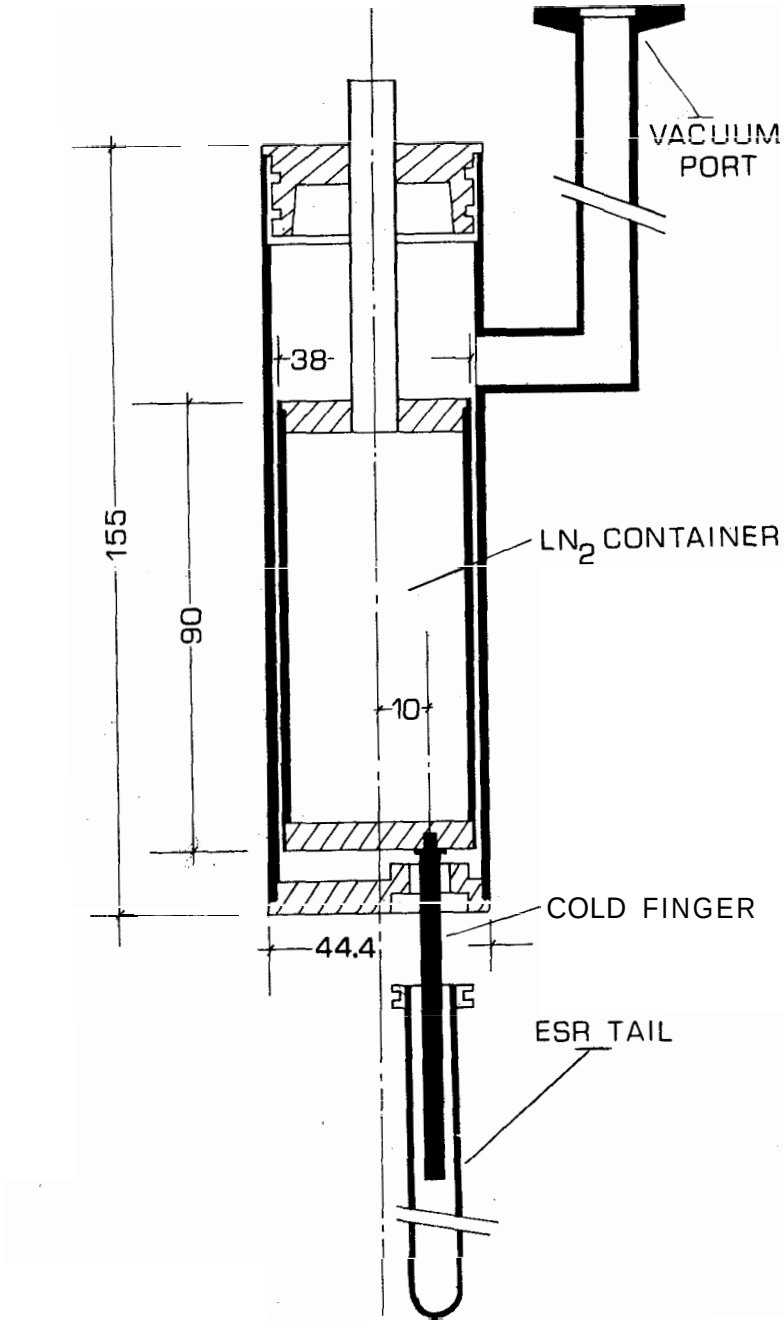


Fig.1 - Detailed drawing of the dewar. All dimensions are in mm.

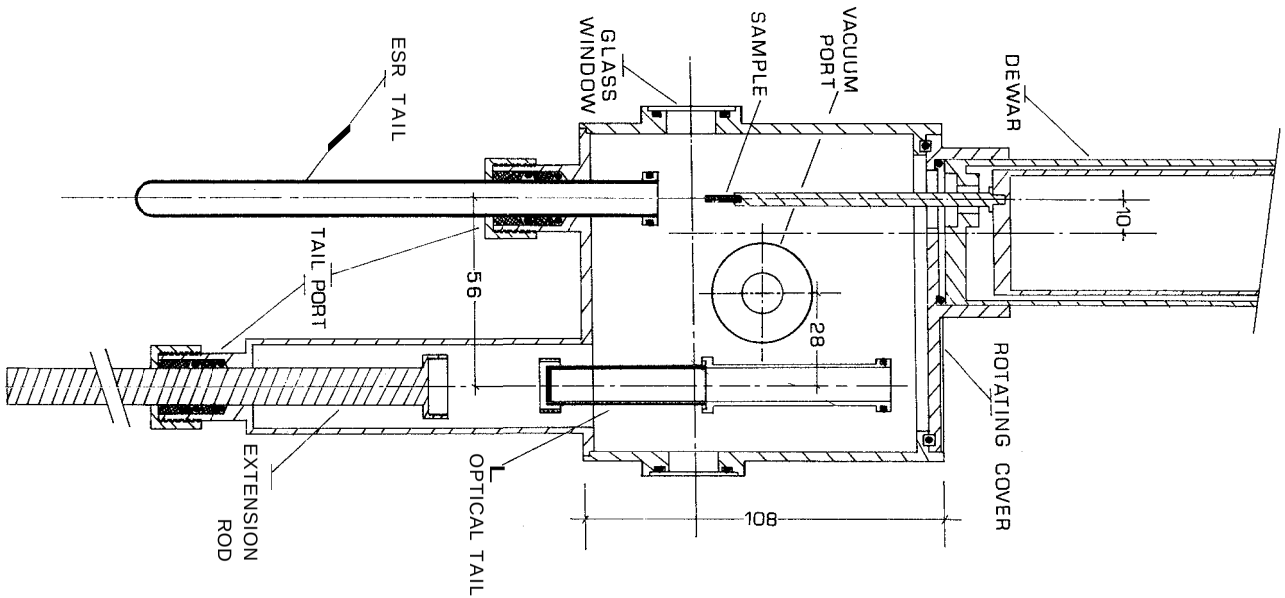


Fig.2 - The dewar fitted to the "tail exchange chamber". The extension rod, and the optical tail, are shown.

sure is low enough, the tail is pulled downwards. The top cover of the chamber is rotated to a new position, where other tail may be pushed upwards, and fitted to the dewar. When the operation is over, the vacuum in the chamber is broken, and the dewar may be brought to the measurement system. All the exchange operation can be controlled by means of the glass windows in the chamber wall.

As the optical tail is not cylindrically symmetrical, an extension rod must be used in order to maintain the vacuum during the vertical motion. This is the reason for the different design of one of the tail ports. When the exchange operation is completed, the extension rod may be taken away.

Severa1 kinds of tails may be conceived for using with this equipment. The optical one is made with a suprasyl quartz cell glued with epoxi to an aluminum piece which is fitted to the dewar. A thin aluminum tail or one of glass may be used for electron irradiation experiences. For X-radiation experiments, a Mylar window can be adapted to a tail or to the chamber itself, instead of one of the glass windows.

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