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Automatic Device for Shell Freezing of Liquids



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(continued overleaf)

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The problem:

Many solutions especially those containing bio-organic compounds deteriorate during extended storage at both room temperature and subzero temperatures. One common method of preventing this deterioration is by freeze drying (lyophilization). Lyophilization is best accomplished by first freezing the liquid at liquid nitrogen temperature in a thin layer (shell freezing) on the interior surface of the lyophilization vessel. Typically, the liquids are poured into vessels which are then placed on a rack composed of parallel rotatable stainless steel rods. The rack is positioned inside a tank which is filled with liquid nitrogen. These rods are manually rotated to achieve uniform freezing. Manual rotation, however, has three disadvantages: (1) it is slow, (2) it is nonuniform, and (3) it is hazardous because the operator is working in close proximity to the liquid nitrogen.

The solution:

A unit was developed which automatically shell freezes the liquids without the manual operation.

How it's done:

The unit is an insulated enclosure (see figure) designed to contain liquid nitrogen. It also includes a set of stainless steel rotating rods for holding the vessels containing the liquids to be frozen, and an electric drive mechanism for rotating these rods.

The main container in the unit for holding the liquid nitrogen is constructed from 1.6-mm (0.062-inch) stainless steel and is bound by an inner and outer chamber, separated by 1.9 cm (0.75 inch) of insulation.

The rotary rods are made from 1.3-cm (0.5-inch) diameter, 1.7-mm (0.065-inch) wall stainless steel tubing. The portions of the rods that are in liquid nitrogen are insulated from the sprocket end of the tubes. The

sprockets are chain-driven and revolved at 45 rpm by a motor. A Teflon guide is used to hold the drive chain in contact with the sprockets. To prevent the drive mechanism from freezing, a blower unit (not shown) is included, to maintain the flow of room temperature air across the rotary sprockets.

In use, vessels containing the liquid to be frozen are placed between the rods. The rods can accept vessels up to 500 ml in size, and with bushings placed around each rod, will accept vessels as small as 3 mm(0.125 inch) in diameter. After the vessels are installed, the blower and the motor are turned on, and liquid nitrogen is introduced into the main chamber to the insulation point of the rotating rods. The rods are then rotated at a constant speed until the liquids are frozen, which takes about 30 seconds. At this point, the vessels are removed and a new batch of vessels can be introduced. The present device will accept 10 vessels at a time.

Note:

No further documentation is available. Specific questions, however, may be directed to:

> Technology Utilization Officer Goddard Space Flight Center Code 207.1 Greenbelt, Maryland 20771 Reference: B73-10253

Patent status:

NASA has decided not to apply for a patent.

Source: Burton Kelbaugh, Clifton Owen, and Grace Lee Picciolo Goddard Space Flight Center (GSC-11737)