Preparation of Fine-Particles at Cryogenic Temperatures

The problem:
To prepare uniformly small-sized solid particles of a low-temperature volatile material for use as a gelling agent at cryogenic temperatures.

The solution:
A process of flash freezing, in which vaporized material, diluted with an inert gas, is injected below the surface of an agitated cryogenic liquid. Although the concept of production of very small particles by rapid condensation is widely known, the techniques that have been used are not effective at cryogenic temperatures.

How it's done:
A cryogenic liquid is used which is chemically inert with respect to both the injected material to be frozen and the diluent gas. The temperature of the liquid is held below the melting point of the injected material, but above that of the diluent.

The illustration shows a cross section of the injector which carries the vapor and diluent gas below the surface of the cryogenic liquid. It incorporates an electric heater to prevent the vapor from freezing in the inlet tube, and an orifice through which the pressurized vapor and gas mixture are injected into the agitated cryogenic liquid. The inlet tube is insulated from the heater by a glass tube, around which the heater wire is wound. This assembly is further insulated from the cryogenic liquid by polytetrafluoroethylene spacers and an outer glass tube. An asbestos layer and an outer sheath of aluminum foil may be wrapped around the outer tube to provide extra insulation. However, these layers should not be used in the presence of fluorine-containing oxidizers, because of the hazard of fire or explosion.

Notes:
1. Particles of chlorine trifluoride averaging 0.9 micron in diameter have been dispersed by this technique in liquid oxygen difluoride to form a gel.

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