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NOTES

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A simple liquid nitrogen gas-flow cryostat for variable temperature laser luminescence studies

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A liquid nitrogen gas-flow cryostat is described which provides steady temperatures between 77 K and room temperature for variable temperature laser luminescence studies. The cryostat is constructed from common laboratory apparatus and requires no quartz glassware. Liquid nitrogen is consumed by the cryostat at a rate of 1 ℓ per hour.

The temperature dependence of luminescence decay dynamics can provide important information about excitation quenching mechanisms ranging from phonon-assisted energy transfer processes[1–3] to energy migration by diffusion or hopping.[4–6] However, cryogenic systems for variable temperature laser luminescence studies are generally closed cycle refrigerators which employ expensive liquid helium as the coolant and costly high vacuum pumps. Although cheaper cryogenic systems based on liquid nitrogen are commercially available for variable temperature luminescence studies above 77 K, these too constitute a significant financial investment. We herein describe a simple but efficient variable temperature gas flow cryostat for laser luminescence studies which utilizes readily available laboratory apparatus and liquid nitrogen.

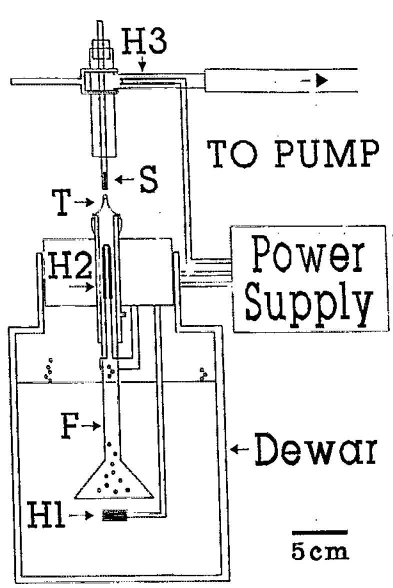

FIG. 1. Schematic of gas flow cryostat for variable temperature laser luminescence studies. H1 is 15 Ω constantan wire heater, H2 and H3 are 5 cm pencil graphite heaters, S is sample in sealed 5 mm glass tube, T is the thermocouple, and F is vacuum-jacketed filter funnel.

The salient features of the variable temperature gas flow cryostat (Fig. 1) are that it obviates the need for large quartz dewars or evacuated cryogenic glassware because the sample(S) is not enclosed. Instead, a laminar flow of nitrogen gas is maintained around the sample by boiling liquid nitrogen from the reservoir. Laser light ranging from ultraviolet to infrared frequencies may therefore be used as excitation sources without attenuation or scattering by glass or quartz enclosures.

The primary heater (H1) consists of three meters of insulated constantan wire (15 Ω) wrapped in alternate loops to eliminate magnetic fields while affording a large surface area for boiling liquid nitrogen without bumping. The secondary (H2) and tertiary (H3) heaters consist of two 5 cm pieces of graphite obtained from hard pencil lead

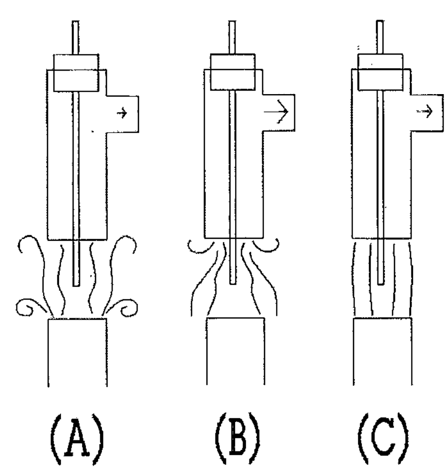

FIG. 2. Visual adjustment of suction to ensure laminar flow: (a) suction too low, (b) suction too high, (c) ideal suction.

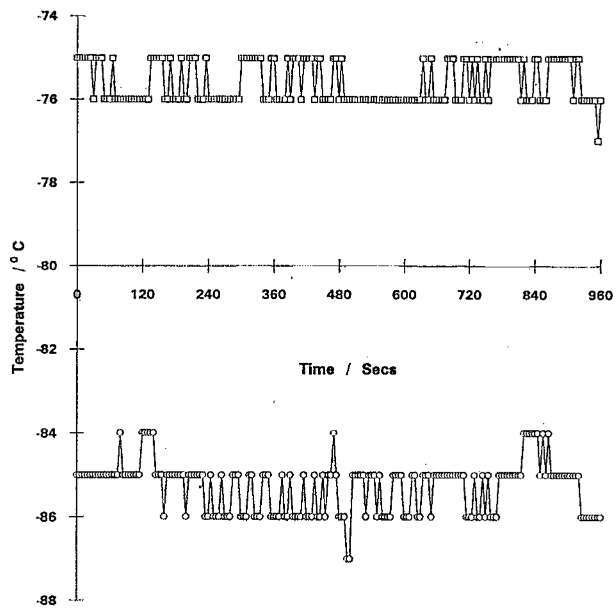

FIG. 3. Temperature variation of gas flow cryostat with time: (a) −75 °C, (b) −85 °C.

($\approx$15 Ω). The secondary heater raises the temperature of the nitrogen gas to the desired temperature without disrupting the laminar flow of gas through the modified inverted funnel (F). The tertiary heater prevents the condensation of water and formation of ice which may block the sample tube and disrupt the laminar flow of gas over the sample. The small diameter and good thermal/electrical conductivity of the pencil graphite are exploited in these configurations.

The suction provided by the pump line or an aspirator is matched to the gas flow to reduce turbulence (Fig. 2) and thereby ensure uniform cooling of the sample and steady temperatures. The suction also minimizes fluctuations in luminescence intensity which would occur if the cold air was allowed to mix with the humid air in the vicinity of the sample and form mist.

The short neck protruding from the dewar is double jacketed to reduce fluctuations caused by ambient temperature. Similarly, the mouth of the dewar is plugged tightly with styrofoam. If the diameter of the tube from which the gas exits the dewar is small (1 cm), the cryostat may be operated continuously for 6 h using a 5 ℓ capacity dewar. However, occasional adjustment of the power supplied to the various heaters (maximum 60 W) is presently required as liquid nitrogen is depleted from the dewar. An electronic temperature controller would eliminate these adjustments.

The thermocouple (T) is placed as close as possible to the sample, although we have found that the temperature measured from within the sample tube and 1 cm below are identical. A Cu-CuNi thermocouple with a useful range of 3–575 K (Type T) and an OMEGA 2168A digital thermometer were used to monitor the temperature. Typical plots of temperature versus time at 5 s intervals are shown in Fig. 3 for two temperature settings.

[1] T. Holstein, S. K. Lyo, and R. Orbach, *Laser Spectroscopy of Solids*, Vol. 49, edited by W. M. Yen and P. M. Seltzer (Springer, Berlin, 1981).

[2] W. M. Yen, *Modern Problems in Condensed Matter Science*, Vol. 21, *Spectroscopy of Solids Containing Rare Earth Ions*, edited by A. A. Kaplyanskii and R. M. Macfarlane (North-Holland, Amsterdam, 1988).

[3] T. Miyakawa and D. L. Dexter, Phys. Rev. B **1**, 2961 (1970).

[4] D. L. Dexter, J. Chem. Phys. **21**, 836 (1953).

[5] M. Inokuti and F. Hirayama, J. Chem. Phys. **43**, 1978 (1965).

[6] M. J. Weber, Phys. Rev. B **4**, 2932 (1971).