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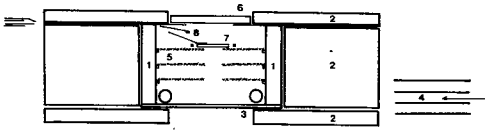
A simple microscope freezing stage

A SIMPLE and highly versatile microscope freezing stage has been developed for studies of fluid inclusions at temperatures down to −100 °C. The stage, utilizing cold gas as the heat exchange medium, has been in use for some time in the laboratories of the University of Durham and has proved to be both reliable and inexpensive to build, maintain, and use. It can be used in any working place with a supply of compressed air or inert gas.

Construction. The stage itself (fig. 1) is composed of a strong Perspex cylinder (1), encased in asbestos insulation (2) and sealed off at the bottom by a clear Perspex plate (3). Dry nitrogen, cooled by passing through a copper coil immersed in liquid nitrogen, is introduced to the chamber through a lagged stiff polythene tube (4). The tube is coiled around the base of the chamber and perforated by a series of upward directed pinpricks. Three copper gauzes (5) supported within the chamber serve to thoroughly mix the gas as it vents upwards. The top gauze also supports the specimen holder—generally a small aluminium wire cage. Used gas escapes around the glass lid (6) which simply rests upon a flat card gasket. The lid thus forms a valve to prevent moist air from entering the chamber and causing frosting when the cold gas flow is reduced. A small jet of dry air directed on to the lid outer surface is sufficient to prevent external frosting. Nitrogen flow is controlled by a very fine needle-valve which can be adjusted to yield cooling rates up to about 25 °C/min. The chamber temperature can be stabilized and maintained at any temperature down to about −100 °C.



This limitation is imposed solely by size of cooling coil and gas pressure. Subsequent to inclusion fluid freezing, the temperature may be raised at any suitable rate by decreasing the cold gas flow.

A copper-constantan thermocouple (7) attached to the specimen (cleavage flake or polished 1 mm mineral wafer) reads directly on to a chart recorder adapted to give maximum sensitivity in the required mV range. Another thermocouple (8) is generally kept in the cell to monitor gas temperature and keep a check for possible thermal gradients. The system is calibrated regularly using standard freezing point capillaries containing the following substances: benzene, +5 °C; butyric acid, −6·5 °C; bromine, −7·2 °C; methyl benzoate, −12·3 °C; benzonitrile, −13 °C; quinoline, −15·9 °C; decane, −19·7 °C. Overall precision on calibration and duplicate runs is generally better than ±0·5 °C.

The system has great advantages over other types of freezing stage some of which (including Peltier effect thermomodules, acetone–dry-ice mixtures, and adiabatic expansion of CO_2) have been experimented with here. The extremely rapid cooling rate, for example, far surpasses that attainable using other methods and has proved very useful, not only in speeding up the entire experimental process, but also for shock freezing fluid inclusions, some of which can persist as metastable liquids for long periods at sub-freezing point temperatures.

Low-temperature studies of fluid inclusions in minerals generally involves the measurement of eutectic temperatures and depression of freezing temperatures, and the examination of the behaviour of daughter minerals and liquid gases already present or newly formed at such temperatures (Roedder, 1963, 1972; Bazaroff and Motorina, 1968). They thus provided much information on the nature and composition of mineral-forming fluids.

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