Laboratory Note

Mechanically refrigerated gas streams for studies to −70°C

Woodard & Straumanis (1971) have recently described an X-ray powder camera which uses a mechanical closed-cycle refrigerator for cooling samples to 25°K by means of conduction. The use of a flowing gas stream utilizing a copper-coil heat exchanger immersed in a cooling medium with the cold gas being transported to the sample *via* a glass Dewar tube is more versatile and is especially useful for single-crystal studies (Post, Schwartz & Fankuchen, 1951; Young, 1966; Rudman, 1967; Silver & Rudman, 1971). However, such systems suffer from the inconvenience that one must replenish the coolant on a regular basis.

The use of a mechanically refrigerated cooling-probe immersed in a bath containing the heat-exchanger would obviate the need for dry ice or liquefied gases. Until recently, the available apparatus was designed for use in stationary systems (*e.g.* vacuum traps) and could not cope with the cooling requirements demanded by a flowing-gas stream. Recently, a number of devices with larger cooling capacities have been developed.* We have successfully used a probe with a cooling capacity equivalent to the consumption of 0.6 kg of dry ice per hour, immersed in an alcohol bath. With an air flow suitable for cooling single-crystal or powder specimens, the bath stabilizes at about −85°C. Inefficiency in the heat exchanger and normal heat leaks in the transfer line result in minimum temperatures at the crystal of about −70°C. When such a probe-cooled bath is used in conjunction with dry compressed gas, a stable system requiring little or no attention over hundreds of hours is easily arranged.

* For example, Model CC100F, Neslabs Instruments, Inc., 871 Islington St., Portsmouth, New Hampshire, 03801, U.S.A.

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Letter to the Editor

Sensitive Test for Acentric Point Groups

Sir,

The purpose of this Letter is to draw attention to a recently available physical test for the absence of inversion centers that is both significantly higher in sensitivity than previous methods and is also rapid and simple. The method depends upon detection of second harmonics generated (SHG) in microcrystalline samples by interaction with a laser beam, as first described by Kurtz & Perry (1968).

The customary tests for determining the presence of inversion centers use either the intensities of the diffraction data or a characteristic physical property. Intensity statistical tests (*e.g.* Wilson, 1950) assume a random atomic distribution, and Patterson methods (*e.g.* Abrahams, 1967) require an accurately measured set of full three-dimensional intensity data: the validity of the former assumption, and the accuracy of the latter measurements, may be in doubt. The physical tests generally search for a piezoelectric or a pyroelectric response: the sensitivity of these tests, for crystals of small size, is often low. In consequence, the current literature continues to abound in examples of undetermined choice of point group.

In the new method, a *Q*-switched Nd:YAG laser source operated at a wavelength of 1.06 μm, or a ruby laser at 0.694 μm, with a peak pulse power in the kW range is used to irradiate the sample. The particle size is not important. The fundamental beam, after passing through the sample, is eliminated by appropriate filters and any second harmonics (0.53 or 0.347 μm) generated are then detected with a photomultiplier.

The principal limitations of the SHG method are the requirement of moderate transparency at the fundamental and second-harmonic wavelengths, and the lack of second harmonic generation in the acentric point groups 432, 422, and 622. Within these limitations, the SHG method provides a highly sensitive and definitive test for the absence of inversion centers.

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