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Cell Compartment for Use at Liquid Nitrogen Temperature in a Double-Beam Spectrophotometer

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A thermally insulated cell compartment accomodating two identical quartz cells, path length less than or equal to 20 mm, has been constructed with which it is possible to work uninterruptedly (without refilling) at liquid nitrogen temperature for at least three hours. The compartment has been used together with a Unicam model SP 700 UV to near IR spectrophotometer.

In the past a considerable number of devices has been designed for spectral analysis of liquid and/or solid samples at moderately low temperatures, *e.g.* that of liquid nitrogen [1-12]. Mostly, a cell compartment has been constructed as a Dewar-vessel, made of quartz or metal, accomodating a single sample cell [1-8]. Thus, with a single-beam instrument, two consecutive spectra have to be taken, one giving the absorption of an appropriate reference system. With a double-beam instrument two spectra still have to be recorded, partial compensation being obtained by introducing a second cell kept at room temperature into the path of the reference beam.

A few authors have described the use of two parallel cells, mounted either in a common double-walled evacuated metal container or in two separate Dewar vessels [9-11].

Nelson [12] describes a single cell construction of considerable interest where he has wholly substituted an insulation of the expanded plastic type (styrofoam) for the Dewar vessel. He glued together sheets of prefabricated styrofoam to a rectangular or cylindrical container for liquid nitrogen. Evacuated quartz cylinders were inserted into the walls of the container and glued in place by use of a synthetic rubber composition (EC 801, Minnesota Mining & Manufacturing Co.). Due to its simplicity this design was very attractive and it appeared that it could easily be used for the manufacture of a two-cell com-

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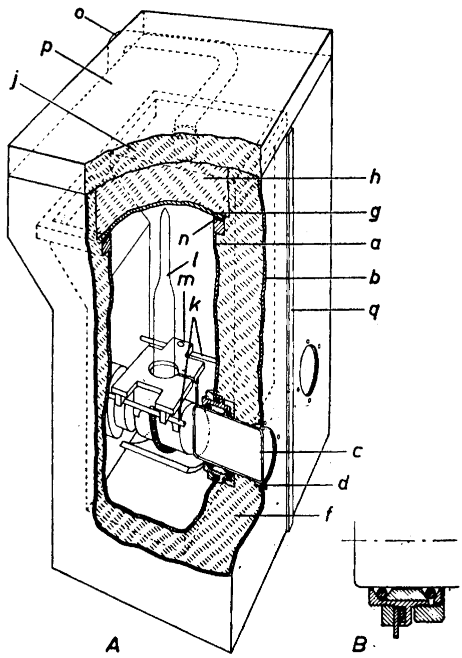

Fig. 1. Cell compartment.

partment. However, in repeating Nelson's work, we encountered a number of difficulties which partly had to do with lack of rigidity of the compartment and partly with difficulties in obtaining a satisfactory seal between the quartz cylinders and the walls made of "Styropor" (Badische Anilin- and Soda-Fabrik AG) *.

These difficulties were overcome by the following changes: In the first place stainless steel was introduced as a constructional material to assure rigidity and a perfectly reproducible mounting of the cells within the compartment. Secondly, the quartz cylinders were fastened using O-rings made of silicon rubber and, thirdly, a foamed-in-place type of polyurethane was used as insulating material.

DESIGN OF THE CELL COMPARTMENT

The cell compartment (Fig. 1 A) consists of an inner can, *a*, made of 1 mm stainless steel and equipped to accomodate two quartz cells, one sample cell and one reference cell, and an outer can, *b*, of the same shape, made of 1,5 mm aluminum plate. The minimum distance between the two cans is 52 mm at the bottom and 38 mm at the walls. The size and shape of the cans were determined from the available space between the monochromator and the detector

* The glue EC 801 with proper accelerator, EC 1 063, was in fact found to be so good as to tear out large pieces of quartz from the cylinder walls thus ruining the high vacuum inside them.

units of the spectrophotometer (Unicam SP 700) as well as from consideration of the storage capacity for liquid nitrogen.

The evacuated cylinders, *c*, with Ultrasil optically polished windows, inner diam. 22 mm, length 50 mm, were mounted in the side walls of the inner can using brass fittings and O-rings (Fig. 1 B). The horizontal distance between the centers of the cylinders was 102 mm and was determined by the beam distance of the spectrophotometer. The cylinders extruded from the inner can so that their outer windows reached the outer can. The distance between the inner windows was 22 mm.

The outer part of each quartz cylinder was equipped with a thin-walled brassflange, *d*, with which the cylinder was secured to the aluminum can, by means of screws. Each flange was equipped with an electrical heater to prevent condensation of moisture on the window *.

The space between the two cans was completely filled with foamed plastic, *f*, in the following manner. The inner can was supported on a prefabricated sheet and an appropriate amount of the foaming composition (Nopco Lockfoam E-302) was introduced and allowed to rise and cure. In order to obtain a uniform structure of the foam we found it advisable to fill the space in two stages.

The inner can was equipped with a lid, *g*, which could be screwed to the can. It was made of a 3 mm brass plate with 3 cm of high density expanded polyurethane insulation on top of it, *h*. In order to assure a close fit between the lid and the Lockfoam plastic during processing, the lid was wrapped in a thin PVC-film and kept in place until the curing of the foam had finished. Afterwards, excess Lockfoam was simply cut away and the surface was smoothened.

The interior of the steel can was divided into two halves by inserting a thin steel plate, *j*, which fitted into light traps in the bottom and in the lid of the can and, in addition, was equipped with two sprung phosphor-bronze blades along each vertical edge giving a complete light seal between the two compartments.

To support the cell holders, the steel can was equipped with two pairs of round brass bars, *k*, screwed to the walls of the can and carefully positioned above and parallel to the light paths. The cells used, *l*, were made of quartz with optically polished Ultrasil windows. The dimensions hitherto used have been o.d. 22 mm, cell length 17 mm. The cells were rigidly placed in brass holders, *m*, machined to allow a kinematic mounting to the brass bars.

As mentioned above, the lid of the inner can was screwed to the rim of the can, a teflon gasket, *n*, giving an almost gas-tight seal.

The inner can held *ca.* 2 litres of liquid nitrogen which was filled in through a short piece of glass tubing carried through the lid by an arrangement with two O-ring gaskets. The tube which had an upper standard joint also served as an escape for the gaseous nitrogen throughout all operations. In order to prevent condensation of moisture on the escape tube, *o*, during experiments, an extra 3 cm of Styropor insulation was placed on top of the lid, *p*.

The assembled cell compartment was inserted into a special housing attached to the Unicam instrument. A track and rail arrangement, *q*, served the

* In a later design, the brass-flanges with heaters were inserted from the outside of the aluminum can, thus facilitating the replacement of a faulty heating wire or of a quartz cylinder with only partial replacement of the plastic foam insulator.

double purpose of keeping the compartment in a fixed position relative to the housing and also acting as a light trap. The escaping nitrogen was carried to the outside of the housing through a tube bent so as to prevent extraneous light from entering the housing. The housing was closed by a tightly fitting metal lid.

Performance. The described cell compartment has now been used in more than 300 runs and proved to be reliable and easy to operate. One filling of liquid nitrogen will last for three to three and a half hours before the surface of the liquid reaches the cell windows.

The fact that the optical path has to pass two one millimeter layers of liquid nitrogen has never caused any trouble even though the recording of spectra down to 200 mμ has become routine [13]. On the contrary, the advantage of having the cell cooled on all surfaces is obvious, particularly in photolysis work using high energy light sources. Gas bubbles can easily be prevented from crossing the optical path by inserting glass troughs beneath the quartz cylinders on both sides of each sample cell.

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