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High Pressure Optical Absorption Cell for Reactive Liquids

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When using optical absorption measurements to follow the progress of chemical reactions at pressures up to a few kilobars, or when working with corrosive liquids one requires an absorption cell to separate the liquid which is being examined from the pressure transmitting medium. The cell must be chemically inert and it should be possible to fill it without the inclusion of air and to assemble it quickly into the high pressure bomb.

In the visible part of the spectrum this can be achieved by using a syringe-like glass cell which at its end has two opposing sides flattened to provide optical windows. If these flats are roughly ground and flame polished the scattering by such a cell is usually quite low when it is immersed in the pressure medium. The pressure is transmitted to the liquid in the cell by the movement of the glass plunger.[1]

To make such a cell out of silica for measurement in the uv region is difficult and we therefore developed a small stainless steel cell which can be used inside a 10 kbar bomb fitted with 12.7 mm thick, 6 mm aperture sapphire windows.[2] Details of the cell are shown in Fig. 1. None of the dimensions are critical, but they are chosen so that the cell fills practically all the space in the high pressure bomb. The internal volume of the cell is kept as small as possible (approx. 1 cc) so that only a very small volume of liquid (approx. 3 cc) is compressed in the high pressure bomb and compression heating is thereby reduced to a minimum.

The distance between the windows of the cell is 8 mm, but the optical path can readily be reduced to 0.2 mm by inserting polished silica disks. The windows consist of 2 mm thick fused silica plates and are sealed to the cell by soft O-rings. The pressure is transmitted to the inside by a slightly tapered plug machined out of polyethylene as shown. This plug has a slight groove cut into it which almost reaches to the top edge and which allows air to escape as the plug is inserted.

When the cell is used without spacers it is filled and cleaned from the top by means of a syringe, but when spacers are used it is found necessary to remove one window in order to clean the cell properly. When several disks are used to obtain very short optical paths it is often necessary to apply about 10 bar to the cell before all the spaces between the disks are filled with liquid.

Since there is no pressure difference between the inside and the outside of the cell the optical pathlength will change with pressure only to the extent of the compression of the stainless steel and this change will be quite negligible in the context of ordinary spectrophotometric measurements. It is of course necessary to correct measurements for absorption by the pressure medium and for lens effects in the pressure windows. This correction is found from blank measurements on pure solvent at the same wavelength and pressure.

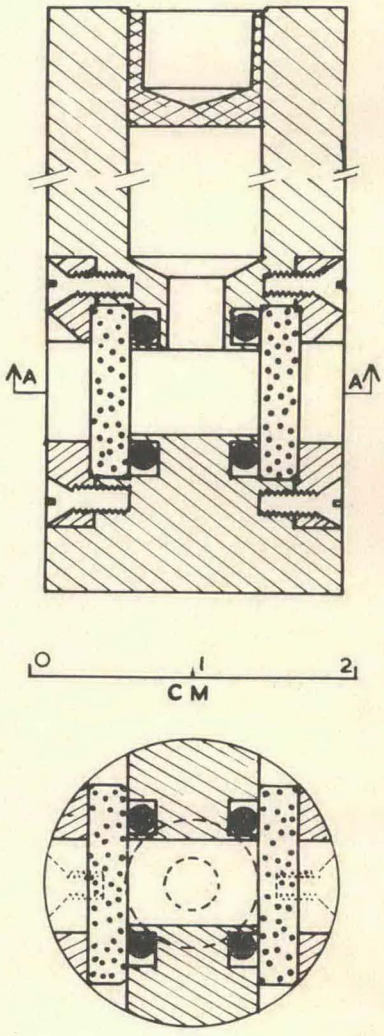

FIG. 1. High pressure optical absorption cell.

[1] A. H. Ewald and S. D. Hamann, Australian J. Chem. **9**, 54 (1956).
[2] D. Langer and D. M. Warschauer, Rev. Sci. Instr. **32**, 32 (1951).