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High Pressure Optical Absorption Cell for Reactive Liquids

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(Received 21 January 1965)

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.

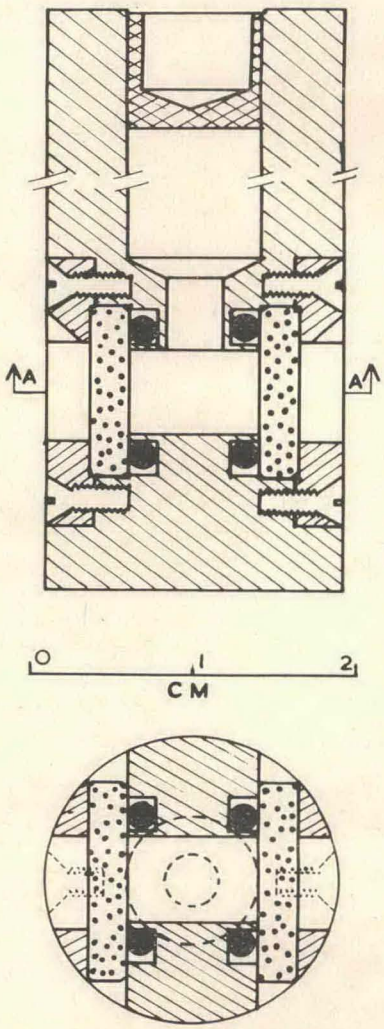

FIG. 1. High pressure optical absorption cell.

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.

[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).

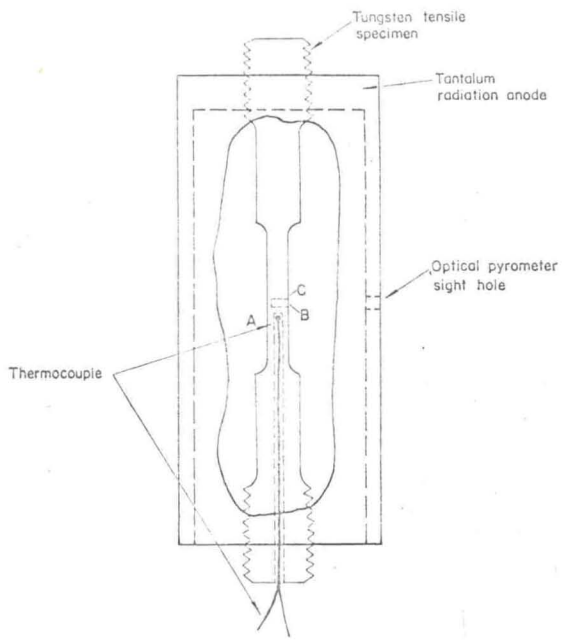

FIG. 2. Schematic drawing of arrangement used for temperature calibration.

During a tensile test the specimen temperature is measured with a calibrated Leeds and Northrup optical pyrometer by sighting on the specimen gauge length through the hole in the tantalum radiation anode. The specimen surface brightness readings were calibrated against true temperature in the following way. A 1.6 mm diam hole was spark machined axially in an electropolished tungsten tensile specimen (12.7 mm gauge length, 3.2 mm gauge diam) to the center of the gauge length and fitted with an insulated Pt/Pt-10 Rh thermocouple which is in contact with the specimen (Fig. 2). Another hole, 0.8 mm diam (length-to-radius ratio=6) was spark machined in the gauge length perpendicular to the specimen axis for blackbody pyrometer readings.

Table I lists the results from a typical temperature calibration, comparing the thermocouple and blackbody temperatures with optical pyrometer readings on the specimen surface. At temperatures $\lesssim$1200°C there is excellent agreement between blackbody and thermocouple readings. For temperatures below 1200°C, however, blackbody readings deviated from the thermocouple readings and reliance was placed on the latter. At temperatures >1400°C it was necessary to remove the thermocouple because evaporation from the alumina insulator coated the specimen surface, thereby changing the emissivity, and it was necessary to check the accuracy of the blackbody readings by a melting point determination. A wire of "A" nickel (liquidus temperature=1446°C) was wound tightly around the specimen directly below the blackbody hole. On heating, the nickel was observed to melt at a temperature of 1446°C as measured by the optical pyrometer blackbody reading. Thus, the true temperature (indicated by c in Table I) was taken to be that of the thermocouple readings below 1400°C, and as given by blackbody readings above 1400°C.

TABLE I. Results from typical temperature calibration (refer to Fig. 2).

Pt/Pt-10 Rh thermocouple at A (specimen center)	Optical pyrometer[a] at B (blackbody)	Optical pyrometer[a] at C (specimen surface)[b]
800°C	904°C	1104°C
972[c]	1021	1240
1090[c]	1113	1328
1191[c]	1194[c]	1395
1315[c]	1317[c]	1486
1398[c]	1400[c]	1554
...	1471[c]	1597
...	1580[c]	1692
...	1729[c]	1815
...	1785[c]	1859
...	1942[c]	2004
...	2085[c]	2139

[a] Optical pyrometer readings are corrected for sight glass losses.
[b] Specimen surface was electropolished as in tensile tests.
[c] "True temperature."

This technique has proved successful in elevated temperature tensile testing of tungsten over the temperature range 1000–2000°C.

The authors are grateful to A. R. Fink and J. F. Schofield for assistance, and to the Air Force Materials Laboratory for financial support under Contract No. AF 33(615)-1727.

[1] R. F. Brodrick, "Development of an Electron Beam Heating Facility and its Use in Mechanical Testing of Tungsten to 6000°F," ASD-TDR-63-484 (July 1963).
[2] H. Doering and P. Shahinian, "Brightness and Two-Color Pyrometry Applied to the Electron Beam Furnance," NRL Rept. No. 6062 (December 1963).

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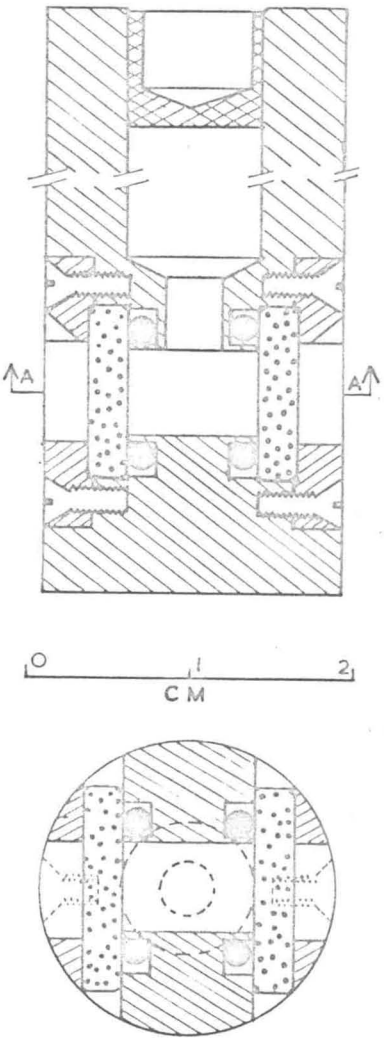

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Two Simple Methods of Making Grainless Fluorescent Screens*

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VIEWING screens for electron beams are usually made of fluorescent powders. Due to the particle size, the resolution is seldom better than 25 μ. With grainless fluorescent screens, the resolution could be the same as that of the optical microscope. In order to be viewed under highest light-optical magnification, the fluorescent layer should be only a few tenths of a micron thick. Otherwise, some of the electrons, penetrating deeper, will cause fluorescence at distances for which the light microscope is not focused. However, the evaporation of thin layers of ordinary fluorescent materials[1] is difficult because of decomposition.

For light-optical reasons, the thin, grainless fluorescent layer should be attached to a transparent carrier of the thickness of a regular cover-glass used in light microscopy. An optimum thickness would be about 0.18 mm for which most of the light microscope objectives have been designed. This could be achieved by chemically treating a piece of cover-glass so that a thin fluorescent layer would form on its surface. Cover-glasses may be made of quartz and thus quartz could be used just as well as a substrate.

(1) A suspension of equal weights of ZnO and water containing a few parts per thousand of wetting agent Tween 20 and 1–2% of $MnCl_2$ is painted on a quartz sur-