

final etch mask was then formed by depositing aluminum over the developed resist and lifting off the excess metalization. Rasters finer than the 3000 Å shown in Fig. 3 are possible, with the final resolution being limited to 1000 Å by the oscilloscope CRT spot size, and by proximity effects in the electron sensitive resist.

The speed at which the pattern can be written with the scanner is dependent on the number of raster lines in the pattern, the slew rate of the internal SEM circuitry, and the decay time of the CRT phosphor. The P31 phosphor used here is one of the brightest available phosphors and has a relatively short decay time (to 1% of the original intensity) of 250 μs. With the present configuration, a pattern 25 μ on a side and with 200 line

resolution will take a minimum time of 25 s for complete exposure on PMMA resist using a charge density of  $5 \times 10^{-5}$  C/cm<sup>2</sup>. Though not suitable for large scale arrays and circuits, such a system can be used to convert a low cost SEM without beam blanking capabilities into a research tool for fabricating submicron structures and devices.

I would like to thank R. E. Howard, E. L. Hu, and L. D. Jackel for helpful discussions in the course of this work.

<sup>1</sup> International Scientific Instruments, Inc., Santa Clara, California.

<sup>2</sup> PMMA, 950000 molecular wt. Supplied by KTI Chemical Corp., Sunnyvale, California. Made from PMMA supplied to KTI by Esschem Chemical Corp., Essington, Pennsylvania.

## Capacitance cell for liquids

D. T. Jacobs

*Physics Department, The College of Wooster, Wooster, Ohio 44691*

S. C. Greer

*Department of Chemistry, University of Maryland, College Park, Maryland 20742*

(Received 26 December 1979; accepted for publication 15 March 1980)

We present a simple and chemically inert, guarded cylindrical capacitance cell for liquids in the temperature range 170 to 370 K and at pressures near atmospheric. A precision in the dielectric constant of 2 ppm is realizable with this cell.

PACS numbers: 06.30.Lz, 07.50.+f

A recent resurgence of theoretical interest in dielectric properties of liquids<sup>1</sup> and liquid mixtures, especially near phase transitions,<sup>2</sup> has led to renewed experimental effort in the precise and accurate measurement of the dielectric constant of liquids.<sup>3</sup> While dielectric cells for liquids have been described in the literature,<sup>4-7</sup> we found no design which met all the following needs:

1. The capacitor plates should be guarded, to eliminate edge effects on the accuracy.<sup>8</sup>
2. The capacitor should be amenable to three-terminal bridge measurements, to eliminate lead and grounding effects.<sup>9</sup>
3. The cell should be as chemically inert as possible; thus only stainless steel and Teflon should be in contact with the liquid sample.
4. The cell should be as mechanically stable as possible, for reproducibility on temperature cycling.
5. The plate alignment and positions should be reproducible on reassembly.
6. The cell should be useful over a temperature range of 170–370 K.
7. The materials and symmetry of the cell should be such as to allow temperature gradients to be kept to less than 1 mk, with proper thermostatting.
8. A measurement precision of 2 ppm should be

realizable, in order to test predicted functional forms for the data.<sup>2</sup>

We chose a cylindrical capacitor design because its symmetry makes the satisfaction of requirements 1, 4, 5, and 7 easier. Our approach follows that of Mopsik,<sup>4</sup> with further attention being given to inertness and simplicity. Shown in Fig. 1, the cell is made entirely of stainless steel (type 304) and Teflon. The cell consists of a "can" which constitutes the outer electrode, O, of the capacitor, and the "lid" to the can, which seals the cell and supports the inner assembly. This assembly is made of three steel parts separated by Teflon O rings and clamped by bolt D. These three segments are the inner electrode, I, and its two guards, G. The electrical lead soldered to I is brought out of the cell through the hollow center of the inner assembly to the connector C. The "lid" is sealed to the "can" by a Teflon O ring. Thus three Teflon O rings serve both to seal the fluid into the cell and to insulate I from G and G from O. A cylindrical Teflon sleeve, A, insulates I from G on the inner assembly. Nylon sleeves, S, are used to insulate the lid bolts from G. The electrical connection to O is easily made at B. Following accepted practice,<sup>8</sup> the length of G is three times the separation between I and the outer electrode, O.

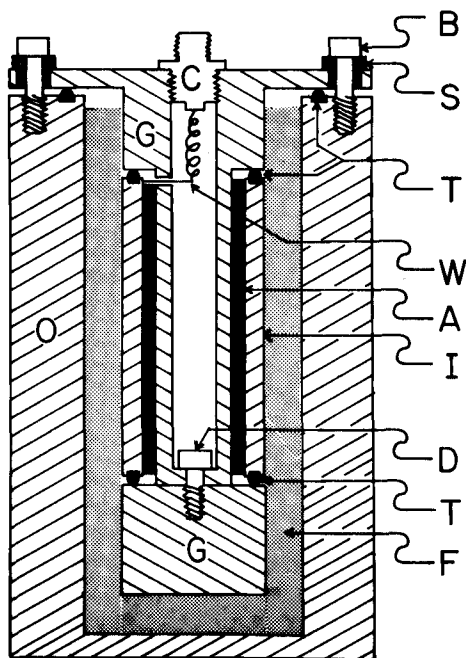


FIG. 1. Guarded cylindrical capacitance cell: O, stainless steel outer electrode; I, stainless steel inner electrode; G, stainless steel guard electrodes; C, SMA connector; S, nylon sleeves; T, Teflon O rings; B, stainless steel bolts; W, wire connecting C to I; A, Teflon sleeve; D, stainless steel bolt; F, fluid. The cell is shown to scale; the outer diameter of I is 2.5 cm and the inner diameter of G is 3.8 cm. The volume of fluid is about 60 cm<sup>3</sup>.

The reproducibility of the position of the inner assembly when the sample is changed is assured as follows: Two positioning holes (not shown in the figure) are drilled through the lid into the top edge of the can; two steel positioning rods fit into these holes, through the lid and into the can, thus aligning the lid with the can to 0.0001 in ( $3.9 \times 10^{-5}$  cm). After the can and lid are aligned in this way, the lid screws can be (symmetrically and systematically) tightened to seal the lid on the O ring. Then the positioning rods are removed, to regain electrical insulation of the lid from the can.

In order to measure liquid dielectric constants to 2 ppm, the temperature must be controlled to about 1 mk. We achieve this control with a vacuum thermostat.<sup>10</sup> The capacitance measurements are made with a General Radio 1615-A capacitance bridge, using a Princeton Applied Research 5204 lock-in amplifier, and a Hewlett-Packard 3311A function generator.

The capacitance of the cell when filled with air,  $C_0$ , agrees with that estimated from the cell geometry (6.7 df). The temperature dependence of  $C_0$ ,  $(dC_0/dT)/C_0$ , is  $17.5 \times 10^{-6} \text{ K}^{-1}$ , which is consistent with the thermal expansion of stainless steel,<sup>11</sup>  $(18 \pm 1) \times 10^{-6} \text{ K}^{-1}$ . As a test of the cell accuracy we measured the dielectric constant of carbon tetrachloride<sup>12</sup> and found, at 293.60 K and 10 kHz, a value of 2.2375. Literature values are 2.2370,<sup>13</sup> 2.237,<sup>14</sup> and 2.2378.<sup>15</sup> Thus the accuracy of measurements with our cell can be expected to be no worse than about 100 ppm.

In our experience using the cell over 6 months, cycling over 15 K near room temperature and at atmospheric pressure, we have observed a precision in our measurements of 2–3 ppm.

We thank G. H. Ahrens and G. E. Thompson of the University of Maryland Space Sciences Shop for their kind help. We are grateful to R. Gammon, C. T. Van Degrift, and R. Hocken for the loan of equipment and to R. B. Strem for technical assistance. Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this work.

<sup>1</sup> See for example, D. Chandler, *J. Chem. Phys.* **67**, 1113 (1977), and references therein.

<sup>2</sup> See for example, J. Goulon, J.-L. Greffe, and D. W. Oxtoby, *J. Chem. Phys.* **70**, 4742 (1979).

<sup>3</sup> See M. Hollecker, J. Goulon, J. M. Thiebaut, and J.-L. Rivail, *Chem. Phys.* **11**, 99 (1975), for an example of work on critical phenomena. See I. M. Hodge and C. A. Angell, *J. Chem. Phys.* **68**, 1363 (1978), for work on supercooled water. See also M. Chan, M. Ryschkewitsch, and H. Meyer, *J. Low Temp. Phys.* **26**, 211 (1977).

<sup>4</sup> F. I. Mopsik, *J. Res. Natl. Bur. Stand. Sect. A* **71A**, 287 (1967).

<sup>5</sup> B. A. Younglove and G. C. Straty, *Rev. Sci. Instrum.* **41**, 1087 (1970).

<sup>6</sup> G. C. Straty and B. A. Younglove, *Rev. Sci. Instrum.* **50**, 1309 (1979).

<sup>7</sup> H. B. Thompson and J. A. Walmsley, *J. Chem. Ed.* **56**, 837 (1979).

<sup>8</sup> M. Ference, Jr., and L. J. Giacometto in *Methods of Experimental Physics*, edited by I. Estermann (Academic, New York, 1959), Vol. 1.

<sup>9</sup> Manual to GR1615-A capacitance bridge (General Radio Co., Concord, Mass.).

<sup>10</sup> D. T. Jacobs and S. C. Greer, to be published.

<sup>11</sup> R. C. Weast, ed., *Handbook of Chemistry and Physics*, 52nd edition, (Chemical Rubber Co., Cleveland, 1972).

<sup>12</sup> Mallinckrodt spectrophotometric grade.

<sup>13</sup> R. H. Stokes, *J. Chem. Thermodynamics* **5**, 379 (1973).

<sup>14</sup> A. A. Maryott and E. R. Smith, NBS Circular 514 (1951).

<sup>15</sup> F. I. Mopsik, *J. Chem. Phys.* **50**, 2559 (1969).