

Polyethylene Microcells for Infrared Analysis

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Polyethylene film has been used recently for preparing cells for infrared analysis (1, 2). We have successfully adapted this technique for samples as small as three μl for liquids and three mg for solids. Also, we have found that compensation of spectral bands for the polyethylene may be achieved by use of an appropriately made blank, thus increasing the spectral usefulness of the technique.

For liquid samples, polyethylene microcells, approximately 3 x 13 mm, were made as follows: A piece of Handi-Wrap**, approximately 10 x 40 mm, was folded to give a double sheet 10 x 20 mm. The sheet was then clamped between the narrow faces of two brass plates, 3 x 15 x 15 mm, with the folded edge perpendicular to the broad face of the plates and about two mm from one end of the butted faces. Both sides of the folded sheet were heat sealed with a small electric soldering iron set to an appropriate temperature by a variable transformer. The cell was removed from between the brass plates and the sample was introduced into the open end by means of a ten μl Hamilton (The Hamilton Co., Inc., Cat. No. 701-N) syringe. Finally, the top of the cell was gently held between the smooth jaws of small cutting pliers and sealed with the hot soldering iron.

Spectra were run at the fast scan on the Perkin Elmer model 237 infrared spectrophotometer. The KBr disc holder supplied with the instrument was adapted to hold the microcell by placing a metal shield (0.7 mm thick and 1.35 cm diam. with a 1 x 8 mm slit) into the recessed end of the holder. The microcell was then centered over this opening, and the holder assembled. The metal shield permits about 40% of the radiation to be transmitted. Since the KBr disc holder is placed close to the focal point of the incident light, a beam condenser is not necessary. Placing a similar polyethylene cell in another KBr disc holder in the reference side of the instrument permits compensation for the few absorption bands resulting from the polyethylene. However, the I_0 line is somewhat irregular due (probably) to imperfections in the surface of the polyethylene film. All polyethylene used was taken from an adjacent area of a sheet of Handi-Wrap to obtain material of uniform thickness. It is advisable to run an I_0 line to insure that the polyethylene absorption bands are adequately compensated prior to filling the cell for analysis.

A second type of cell was used for solid and viscous liquid samples. A piece of polyethylene, approximately 4 x 4 cm, was stretched over the smoothly polished end of a brass pipe (7/16 in. o.d., 5/16 in. i.d., and one in. long)

and held in place by a rubber "O" ring. The sample dissolved in a suitable solvent was placed dropwise on the stretched polyethylene surface and gently heated with an

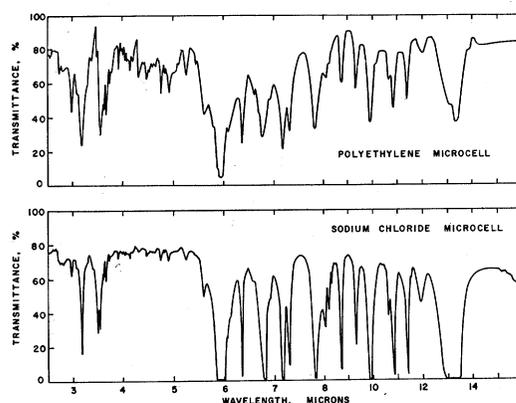


FIG. 1. INFRARED SPECTRUM OF FURFURAL IN POLYETHYLENE AND NaCl MICROCELLS

infrared lamp to remove the solvent. A second piece of polyethylene similarly mounted was butted against the film holding the sample and the edges of the two films were sealed with a soldering iron as described above. In attaching this cell to the KBr disc holder, a metal shield having a 5/16-in. diam. hole was used.

A spectrum of authentic furfural obtained on a three μl sample using a polyethylene reference cell is compared in Figure 1 with a spectrum using a conventional NaCl microcell.

Due to imperfect sealing or vapor permeability of the polyethylene, some loss of material may occur with liquid samples during spectral scanning. Except for relatively volatile material, the extent of the loss is small and major bands in a full 2.5 to 15.0 micron scan can be obtained.

The technique offers an inexpensive means of obtaining adequate spectra for qualitative purposes of samples as small as three μl of liquid or three mg of solid. In fact, recognizable spectra can sometimes be obtained with only one mg of sample.

Literature Cited

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- (2) E. F. Ferrand, Jr., APPLIED SPECTROSCOPY **16**, 22 (1962)

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