

Novel Micro and Semimicro Pelleting Technique for Infrared Spectroscopy

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SINCE the development of the potassium bromide pelleting technique in infrared spectroscopy, a variety of dies, each of which produces one specific size of potassium bromide pellet, have become commercially available or been described in the literature. Various reports have established the usefulness of these pellets in sizes varying from micro through macro [White, J. U., Weiner, S., Alpert, N. L., Ward, W. M., *ANAL. CHEM.* 30, 1694 (1958)].

With the technique and the simple accessories described one die is used (Perkin-Elmer Part No. 021-0106) [Hausdorff, H., *Appl. Spectroscopy* 8, 131 (1954)], which is designed to form a pellet 13 mm. in diameter, to produce vacuum-formed samples in various shapes and sizes. The technique consists essentially in using a matrix of potassium bromide to confine a dispersion of the sample, whose spectrum is sought, to a small aperture in a metal disk embedded in the matrix. Especially noteworthy is the preparation of vacuum-formed microsamples, 0.5 mm. in diameter, which have previously required separate expensive precision dies. The technique can be used with most commercial or "homemade" dies.

To produce microsamples with a diameter of 0.5 mm., the following procedure is used.

The disk holder is a circular piece of steel, 6 mm. in diameter by 0.5 mm. thick, with a 0.5-mm. hole (No. 76 drill, 0.5080 mm.) drilled in its center. In preparing the pellets, the disk holder is placed on the lower plunger of the die assembly. The sample, which is dispersed in potassium bromide by conventional grinding in a micromortar,

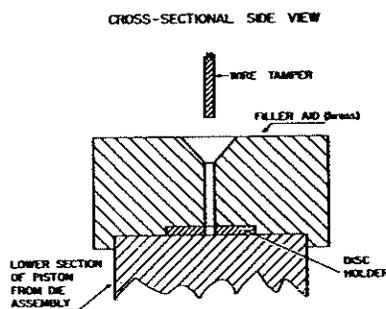


Figure 1. Filler aid in use

or by a lyophilization technique, is transferred to the hole with the aid of the accessory shown in Figure 1.

This filling aid fits over the disk holder and plunger. The potassium bromide mixture is placed in the funnel-like opening and tamped into the disk holder with a flat-ended wire, and the filler aid is lifted off. The hole in the disk holder need not be completely filled. The disk holder containing the sample is now incorporated into a conventional pellet by spreading about 250 mg. of potassium bromide over the disk holder with its sample, and pressing in a vacuum as usual. This technique yields a potassium bromide pellet 13 mm. in diameter and ca. 0.75 mm. thick, in which the steel disk holder is embedded. The sample fused in potassium bromide is concentrated into the 0.5-mm. hole (Figure 2). Optically clear specimens which can be easily aligned for microscopic measurement are obtained. The disk holder can be cleaned and re-used. One microgram of sample dispersed in 300 γ of potassium bromide will generally yield a satisfactory spectrum. A sample of this size has been used in conjunction with a Perkin-Elmer Model

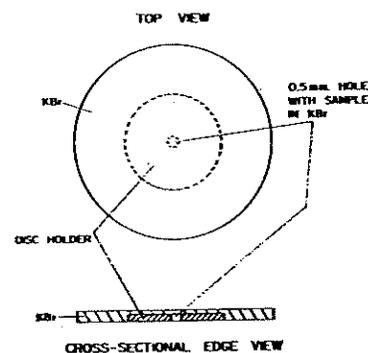


Figure 2. Potassium bromide pellet with disk holder insert and sample

85 reflecting microscope and the Perkin-Elmer Model 112 spectrometer.

Sample apertures of other sizes and shapes are obtained by using disk holders with different sized and shaped openings. With a beam-condensing attachment a disk holder with a hole $1/16$ to $1/8$ inch in diameter is desirable. For semimicro work, without any beam-condensing accessory, a disk holder with a rectangular aperture, 1×6 mm., gives excellent spectra with 75 γ of sample when the pellet is placed directly before the entrance slit of the spectrometer. Much less material (ca. 20 γ) will give a fair spectrum with disk holder of this size.

ACKNOWLEDGMENT

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