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Apparatus for the Spectrophotometric Study of Small Crystals*

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By means of the apparatus described in this paper it is possible to obtain absorption spectra of crystals, of appropriate thickness, whose diameter is 0.3 to 0.4 mm by means of Cary recording spectrophotometer, model 14M. It is also possible to obtain spectra of crystals of 0.1 mm diameter over the range of wavelength that can be used with the Cary high intensity source accessory (Cary model 1471200, available from Applied Physics Corporation, Monrovia, California). Ferguson, Wood, and Knox[1] have given a brief description of apparatus they used for obtaining spectra of small crystals through an orifice of 0.2 to 0.3 mm diameter. These authors, however, focused a beam of light on the sample and analyzed the transmitted beam with a Perkin-Elmer model 112 spectrometer. Concurrently with their work, we found that the spectra of similar small crystals could be obtained by means of existing commercial instrumentation without modification. The potential usefulness of a simple but nevertheless overlooked method for obtaining absorption spectra of small crystals has prompted the publication of this note.

The crystal holder which was developed is pictured in Fig. 1 and a detailed drawing of the apparatus is given in Fig. 2. These figures are self-explanatory. It is important, however, that the small aperture be aligned properly with respect to the light beam. If this is not done, spurious transmission effects are seen as the slit of the spectrophotometer opens up at either end of the three respective spectral ranges, ultraviolet, visible, or near infrared. Alignment is accomplished by adjustment of the two set screws in the casing and maintained by the spring shims in the side of the casing.

The procedure required for fastening a crystal to the holder involves placing a ring of adhesive, such as Apiezon Q (available from Apiezon Products Ltd., distributed by James G. Biddle Co., Plymouth Meeting, Pennsylvania) around the periphery of the beveled side of the orifice and carefully pressing the crystal onto the adhesive. This loading procedure is best performed with the aid of a magnifying glass or stereoscopic microscope. It is desirable to position the crystal so that its faces are normal to the direction of the light beam of the spectrophotometer; however, useful spectra can be obtained with crystal chips or poorly aligned crystals.

Compared to air in the reference beam, a properly aligned crystal holder with a 0.35 mm diam aperture has a measured absorbance of 1.7 which is independent of

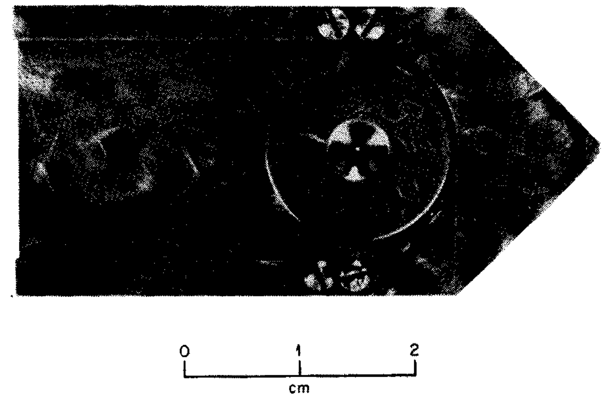

Fig. 1. Small crystal holder.

wavelength at slitwidths less than 0.4 mm. The absorbance of the aperture increases as a function of slitwidths greater than 0.4 mm. Absorption spectra of materials in the holder can be determined in a routine manner with the use of neutral density screens in the reference beam of the instrument so long as sensitivity controls can be adjusted to give slitwidths of less than 0.4 mm. With larger slitwidths, spectra must be corrected for the nonlinear baseline response. The resolution of the spectrophotometer is, of course, affected by any increase in slitwidth; this is the only compromise of the instrumental characteristics that

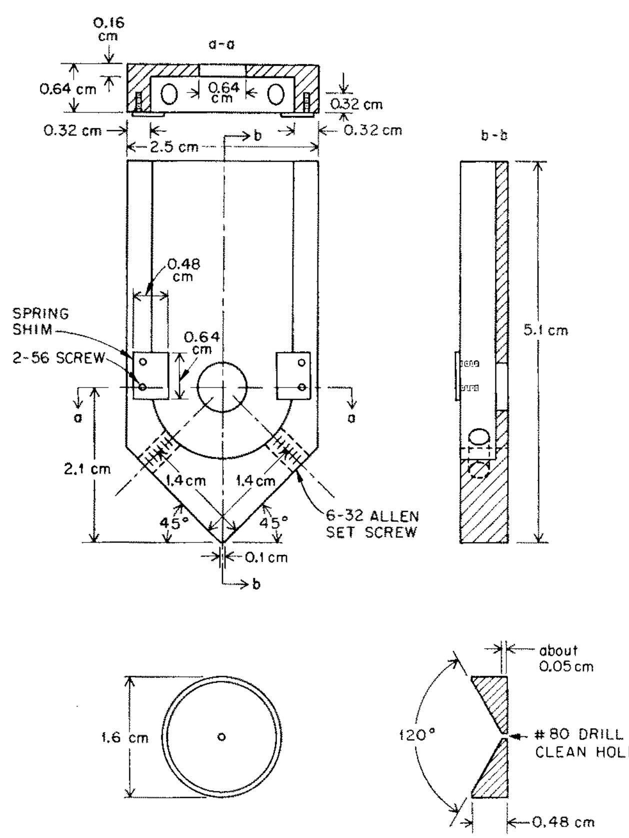

Fig. 2. Small crystal holder. Brass or nonsoft aluminum alloy.

has to be considered. For room temperature spectra of didymium glass standards, no loss of resolution was noted, however.

With the use of the Cary high intensity light source accessory, operated at line voltage, the absorbance of a holder with a 0.13 mm diam aperture is 2.7 vs an air reference at slitwidths less than 0.3 mm. Spectra can be determined through this aperture with the same limitations that were previously discussed. The accuracy of absorption spectra obtained with these crystal holders is quite satisfactory; didymium glass and ruby chips of known orientation were used in this study. The wavelength reproducibility was quite adequate. The reproducibility of peak intensities is at least a good approximation; some of our earlier data with ruby crystals yielded relative standard deviations of 10%, but it is believed that this demonstrates inhomogeneities of the Cr(III) concentration in the flame fusion grown crystals rather than faults in the technique.

* Research sponsored by the U. S. Atomic Energy Commission under contract with the Union Carbide Corporation.

[1] J. Ferguson, D. L. Wood, and K. Knox, J. Chem. Phys. **39**, 881 (1963).

Simple Device to Improve Airbrasive Cutting Capability

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An Airbrasive machine[1] has been found by us to be a useful means for cutting hard brittle material (glass, quartz, alumina, tungsten, etc.). One difficulty encountered is accurate cutting of lines, circles, spirals, etc. by hand. The simple device illustrated in Fig. 1 is a very good and inexpensive remedy. It has the additional advantage that the work can be left unattended while cutting proceeds. A single variable speed reversible motor drives the arrangement through a main rod and a bevel gear assembly. Linear motion is achieved from rotary in a manner obvious from the illustration; length of cut is controlled by the radius of the actuating circle. Either the piece to be cut or the nozzle may be moved while the other piece is held stationary. Circles are cut by attaching the material to be cut to one of the rotating holders or to a chuck (see Fig. 2). Centered and off-centered circles, ellipses, and semicircles can also be obtained. Spirals are cut by attaching the nozzle to the linear motion holder and rotating the piece to be cut at right angles to the nozzle. Parts are made of nylon and Teflon to reduce wear from the abrasive particles.

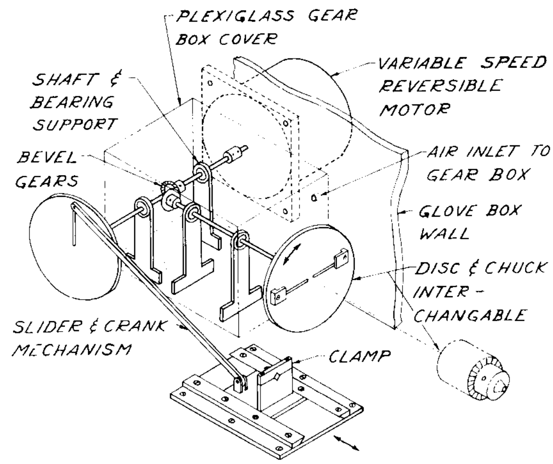

Fig. 1. Diagram of mechanism used to hold work during cutting operation.

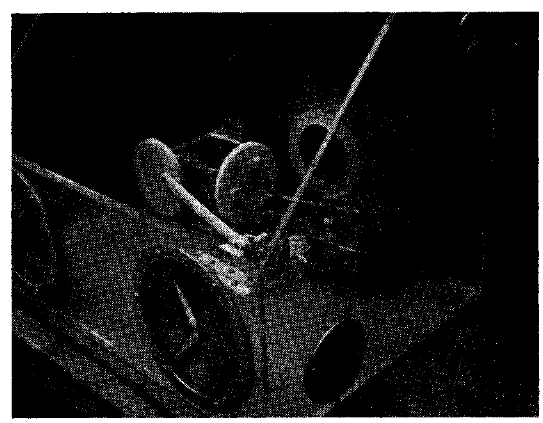

Fig. 2. Photograph of device during circle cutting operation.

Since a slight negative pressure exists in the box from the exhaust unit no tendency for particles to get into the motor or gear assembly has been found.

[1] Manufactured by the S. S. White Co., New York, New York.

Water-Cooled Wrede-Harteck Gauge Fitting*

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A WREDE-HARTECK gauge (WHG) fitting has been constructed that differs in four important respects from one described several years ago by Sharpless, Clark,