positioning of the sample and for positioning the monochromator slit image with respect to the sample. If petrographic or dichroic observations are to be made on a material, a polarizing binocular tube is used along with a polarizing condenser.

The camera may be used in the position indicated or may be attached to the microsectioning ocular to obtain a photograph of the area of the specimen selected for a spectral determination. Photographs of this type are particularly useful in the interpretation of spectra. Stage photographs of substances in the diamond high pressure cell are valuable to high pressure phase studies.[3] Using monochromatic radiation, such photographs have been used to determine pressure gradients in the high pressure cell.[7]

The photometer tube may be mounted on a base with the microscope or on a wall bracket as we have done (see Fig. 1). The bracket is made in such a way as to permit three-dimensional travel of the photometer tube. This provides for a simple alignment of the phototube unit, microscope, and monochromator.

A $\frac{1}{3}$ reducing field lens may be used to project the sample and slit image into the plane of the centerable iris diaphragm to help reduce the amount of stray radiation. If stray radiation is no serious problem, the reducing field lens may be removed and the image projected into the phototube or upper ocular after passing through the fixed aperture slide (containing aperture diameters of 1 through 5 mm) and a 6.5-cm Milar field lens. The area of the microsection selected is controlled by the size of the fixed aperture, the magnification of the objective, and the use of the reducing field lens. With the range of objectives described earlier, one can obtain microsections of almost any diameter between 10 μ and 3 mm.

The fixed aperture slide is also centerable, a feature which is generally used to control the position of the radiation striking the photodetector. Since all areas of the photocell do not give equal photoresponse, in order to compare spectral intensities, it is necessary to keep the size and position of the fixed aperture constant. In these cases the positioning of the sample image with respect to the fixed aperture is accomplished by means of the centering screws of the objective mount, or by a fine adjustment of the sample on the stage. The final selection of the microsection is observed through the microsectioning ocular and a photographic record of the section may be made at this point, as discussed earlier, if desired.

The standard photodetectors for the model 350 spectrophotometer are used, viz. EMI type 9529B end-on photomultiplier for the uv and visible regions and a lead sulfide cell for the near-infrared region. The detectors are taken from the spectrophotometer and mounted on the photometer tube after lengthening and properly shielding the leads. With the addition of a switch and another detector, it is possible to leave the photodetector in its original position. This permits the instrument to be used as a conventional spectrophotometer by rotating the sample compartment mirror. The reversion of the instrument to its original form is particularly helpful in making service checks and adjustments.

In the microspectrophotometry of specimens mounted in a cuvette or on a microscope slide, the coniscopic optics are replaced by achromatic or apochromatic objectives of greater magnification and with a greater resolving power. The coniscopic and achromatic objectives enable us to obtain spectra from 0.36 to 2.3 μ.

Presently, the range of the instrument is being extended into the ultraviolet region by the use of Bausch & Lomb "Grey-Polaroid" 20× long working-distance reflection objectives in place of the coniscopic objective and condenser. After substitution of a fused silica upper field lens, it should be possible to get spectral determinations from 0.2 to 2.7 μ. Reflection objectives of higher magnification (up to 300×) are now available for conventional microscope investigations throughout the same spectral range.

PERFORMANCE

Since many of the performance characteristics (such as stability, photometric accuracy, and photometric reproducibility) are virtually the same as they are for the model 350 spectrophotometer, only those which are grossly altered will be discussed here.

A demonstration of the use of the I_0 compensator is given in Fig. 3. The absorption due to the optical system and a typical diamond cell is given along with the adjustment of the I_0 line to correct for these. The further expansion of the usable range of the instrument by use of reflection objectives has been mentioned earlier.

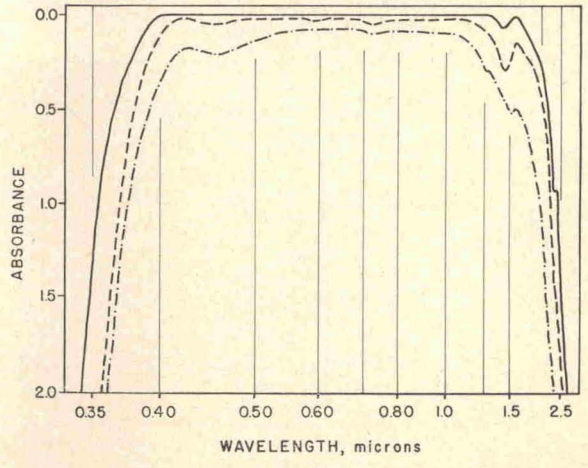

FIG. 3. Typical I_0 compensation curves; – – –, spectrum of optical system; – · –, spectrum of diamond cell in optical system; ——, I_0 correction. Absorbance is defined as log I_0/I.

[7] E. R. Lippincott and H. C. Duecker, Science 144, 1119 (1964).

The effectiveness of the magnification of the monochromator beam intensity mentioned earlier is partially reduced by the absorption and reflection losses within the optics. Thus, the total amount of light striking the photocell is less than when the spectrophotometer is used in a conventional manner. However, since we are able to project the energy onto a more sensitive part of the photocell, we get an increase in photoresponse. Photographic comparison of the light intensity at the photocell ports indicates an effective increase in spectral response of about 20%. However, this is affected considerably by the alignment of the instrument and the photocell, so that one might generally be concerned with a loss rather than a gain in photoresponse if the proper care is not taken. It appears that the same range of optical densities can be covered as with the conventional spectrophotometer.

The effect of the size of the area selected on the resolution of the spectrum is almost negligible for areas of 10 μ^2 or greater. There is some loss of resolution however, if the area is reduced to 1 μ^2. Figure 4 gives a comparison of spectra taken of the holmium oxide glass wavelength standard using scan areas of 1 and 10 μ^2 as indicated. Otherwise, the resolution appears to be about the same as that of the model 350 spectrophotometer alone, viz. 1.0 mμ or less in the visible region.

APPLICATIONS

The instrument has been used in the investigation of the effects of pressure on the spectra of a number of substances. Some materials, e.g., nickel dimethylglyoxime, have an absorption band in the visible region which shifts with pressure to a longer wavelength (see Fig. 5). Another group of materials, such as the thallous halides, has an extremely strong absorption band in the ultraviolet which shifts into and through the visible region as pressure is applied. Some substances have sharp phase transitions, e.g. mercuric iodide, with perhaps some other spectral

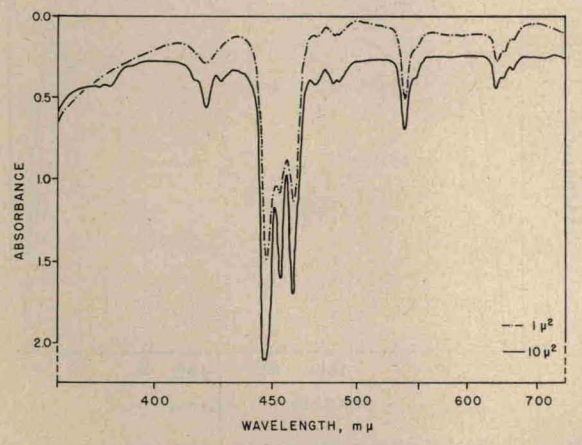

FIG. 4. Effect of scan area on resolution using areas indicated. Lower curve is shifted to make a comparison possible.

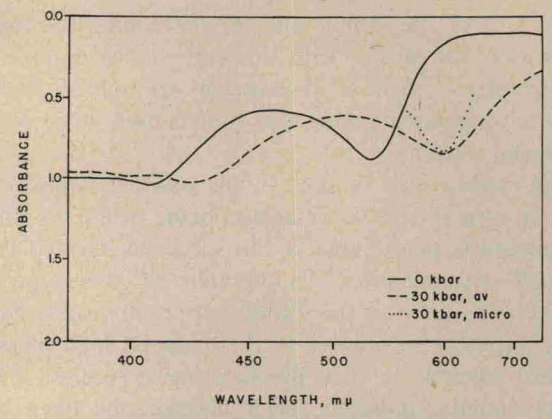

FIG. 5. Spectra of nickel dimethylglyoxime in diamond cell; ——, entire sample at 0 kbar; – – –, entire sample at 30 kbar; · · ·, 10-μ-diam microsection at 30 kbar.

effect superimposed. In Fig. 6 the absorption edge of the red and yellow (high pressure) form of mercuric iodide is shown along with a subsequent shift of the edge with pressure. An absorption spectra of the entire sample is a mixture of the spectra of the red and yellow forms and the component spectra cannot be determined without the use of the microscope spectrophotometer. Figure 6 gives the spectra for the red and yellow components at an applied pressure of 12 kbar, as well as for the brownish region beginning to form in the highest pressure zone.

The two other types of spectra may be complicated in a similar manner. However, for the first type, i.e., with nickel dimethylglyoxime, the average spectrum (taken from the entire sample) is apparently not too objectionable, since

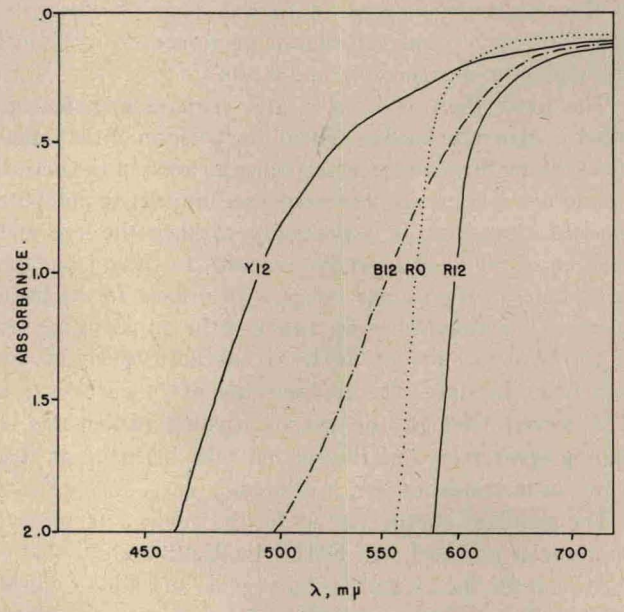

FIG. 6. RO spectra from red mercuric iodide at 0 kbar. Spectra of different areas of mercuric iodide in diamond cell at applied pressure of 12 kbar as follows: R12 from red region near phase boundary; Y12 from yellow region near phase boundary; B12 from brownish high pressure region forming at the center of the sample.