# Infrared Measurements With a Small Grating From 100 to 300 Microns

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Good resolution has been obtained with a small grating in the region from 100 to 300microns. A high-pressure mercury lamp, having a quartz inner envelope, was used as a source and a Golay cell as a detector. The radiation was reflected from three filtering plates before passing through the entrance slit. This filtering served to reduce the stray radiation to about 5 percent.

### 1. Introduction

In the past several years small gratings have been used in spectrometers for the near-infrared region. It has also been shown that a small prism spectrometer could be converted, with only minor changes, into a far-infrared instrument.<sup>2</sup> Good resolution has been obtained in the region from 25 to  $125 \ \mu \text{ using a thermocouple as the detector.}$  Further work has been done on the instrument, which increased the resolution and extended the wavelength range.

### 2. Experimental Procedure

In order to extend the measurements beyond 125  $\mu$ three changes were made in the instrument. A Golay cell with a crystal quartz window replaced the thermocouple, a high-pressure mercury lamp was substituted for the globar source, and a different arrangment of the filters replaced the previous threefilter arrangement described in footnote 2.

The Golay cell was found to have a greater sensitivity in the detection of the long-wavelength radiation than the thermocouple, making it possible to use narrower slits and to obtain better resolution.

High-pressure mercury lamps have much greater intensity in the far-infrared region than the globar source. It was found that an AH4 mercury lamp was adequate for the measurements made in the region from 100 to 300  $\mu$ . A section 1 by  $\frac{1}{2}$  in. was removed from the outer glass envelope which enclosed the quartz lamp. A small glass tube was introduced through the opening and nitrogen was circulated about the electrodes, which greatly added to the life of the lamp.

Ultraviolet and visible radiation are intense from the lamp, and the observers' eyes should be shielded. At first the lamp was housed in a black box with an aperture that permitted the radiation to fall upon the slit of the spectrometer. Later the lamp was coated with a layer of magnesium oxide. The MgO film absorbed only a small fraction of the radiation at 100  $\mu$ , but it was effective in scattering the shortwavelength region. A crystal of magnesium oxide 1-mm thick absorbed about 80 percent of the radiation at 100  $\mu$ , but with thinner layers the energy loss with the coating became negligible.

As measurements are carried out to longer wavelengths, increased filtering is required for the removal of the stray radiation. A new arrangement of the optical system has been made so that three reflection filters are employed in front of the entrance slit of the spectrometer. Figure 1 represents the system that was used. The filters that were placed in positions A, B, and C were reststrahlen plates of CsBr and CsI, gratings, and roughened silver scatter plates. In the measurements shown in figures 2 and 3, one filter of each type was used. For the region from 70 to 100  $\rm cm^{-1}$  the external filters as shown in figure 1 were a CsBr reststrahlen plate and two roughened silver plates, one of which was blackened. For the spectral region from 53 to 70  $\rm cm^{-1}$  a CsI reststrahlen plate was substituted for the CsBr plate. In some of the measurements a grating with 1,000 lines/in. was substituted for one of the silver plates.

Some spectra have been obtained in the region from 25 to 50  $\rm cm^{-1}$  by the use of small gratings with 60 lines/in. and 100 lines/in. In the measurements in this region it was necessary to use wider slits, and in the region of 400  $\mu$  the slitwidth was 4 mm. In order to obtain the wide slits, the regular slit jaws were removed and replaced by two metal jaws so that there was a separation of 2 mm when the slit drum read zero. This change enabled the observer to set the slits between 2 and 4.5 mm.

The strong water-vapor line at  $26 \text{ cm}^{-1}$  was observed, but it is estimated that the spectrum con-

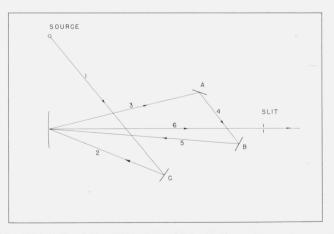


FIGURE 1. Optical arrangement for the filters. The reflection filters are placed in positions A, B, and C.

<sup>&</sup>lt;sup>1</sup> This work was supported by the U. S. Atomic Energy Commission. <sup>2</sup> Earle K. Plyler and Nicolo Acquista, J. Research NBS **56**, 149 (1956) RP2660.

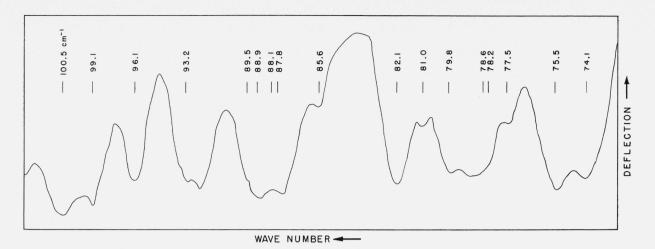


FIGURE 2. Absorption spectrum of water vapor from 75 to 100 cm<sup>-1</sup>.

The slitwidths were  $1.5 \ \mathrm{mm}$  and a 180-lines/in, grating was used.

tained about 40 percent of stray radiation in this region. Before quantitative measurements can be made between 300 and 400  $\mu$  on the instrument a more efficient filtering system will be required.

#### 3. Results

As the present investigation has been primarily concerned with the methods of measurements, only three examples of the results will be given. The absorption spectra of several molecules have been reported in other publications.<sup>3</sup>

Figure 2 represents the absorption spectrum of water from 73 to 100 cm<sup>-1</sup> as measured with a 180lines/in. grating. This region was included in the previous publication (see footnote 2), but now the stray radiation has been almost completely removed and the absorption lines of high intensity cause the deflection to approach the zero line. The wave numbers marked on the lines are the calculated values determined by Benedict.<sup>4</sup>

<sup>3</sup> D. E. Mann, J. H. Meal, and Earle K. Plyler, J. Chem. Phys. 24, 1018–1022 (1956).

The amount of water vapor in the path wa<sup>S</sup> sufficient to produce nearly 100-percent absorption for the lines of moderate and high intensity; thus some of the weak lines could be observed. The observed wave numbers of some of the lines of low intensity agree closely with the calculated values. The wavelengths were determined experimentally by the measurement of the angle of the grating with reference to the central image.

Figure 3 represents the region from 53 to 70  $\mu$ . The more intense lines of the spectra have been identified and the wave numbers are marked on the chart. The two lines near 60 cm<sup>-1</sup> are slightly distorted by second-order lines. The linewidths are about 0.6 cm<sup>-1</sup>, which is a measure of the resolution obtained. The spectrum of water vapor recorded in figures 2 and 13 is for a path length of 2 m and relative humidity of 40 percent. The slitwidths were 1.5 mm for the region from 75 to 100 cm<sup>-1</sup>, and 2.5 mm from 53 to 72 cm<sup>-1</sup>.

<sup>4</sup> W. S. Benedict, private communication.

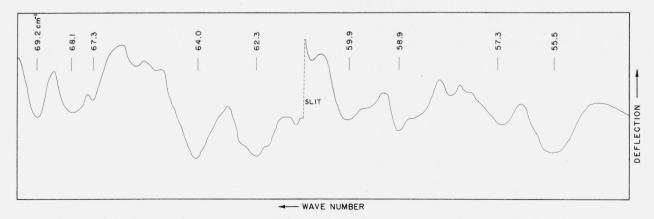
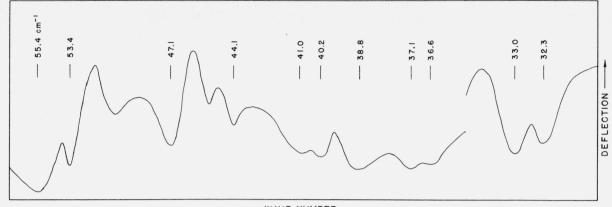


FIGURE 3. Absorption spectrum of water from 55 to 70 cm<sup>-1</sup> as measured with a 180-lines/in. grating. The slitwidths were 1.5 mm to 62 cm<sup>-1</sup> and 2.5 mm from 55 to 62 cm<sup>-1</sup>.



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FIGURE 4. Absorption spectrum of water vapor from 36 to 55 cm<sup>-1</sup> as measured with a 100-lines/in. grating and from 30 to 36 cm<sup>-1</sup> with a 60-lines/in. grating.

The slitwidth was 4 mm for the entire region.

Figure 4 shows the absorption spectrum of water vapor from 30 to 55 cm<sup>-1</sup>. The stray radiation was reduced to a small percentage by using a 0.2-mm window of LiF as a transmission filter. The slitwidths were 4 mm, and a 100-lines/in. grating was used from 36 to 55 cm<sup>-1</sup> and a 60-lines/in. grating was used from 30 to 36 cm<sup>-1</sup>. In observing the two lines at 32.3 and 33 cm<sup>-1</sup> a window of KBr, 0.4-mm thick, was found effective in eliminating higher orders from the grating. The line at 47 cm<sup>-1</sup> could

be observed through the KBr window, but the energy in the higher wavelength region (47 to 100  $\text{cm}^{-1}$ ) was almost zero.

In conclusion it can be stated that a small grating is adequate for measuring spectra from 25 to 300  $\mu$ , and with further improvements in the filter system the measurements can be extended to 400  $\mu$ .

WASHINGTON, July 18, 1957.