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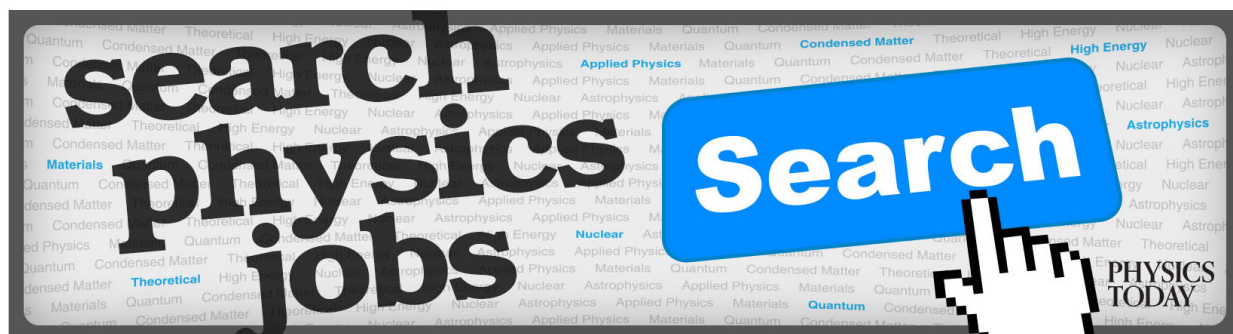
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Laboratory and Shop Notes

BRIEF contributions in any field of instrumentation or technique within the field of the journal can be accorded earlier publication if submitted for this section.

A Simplified Percent Transmission Computer

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July 20, 1950

THE use of any single beam spectrometer requires the transformation of the spectra measured by the instrument into the related and more useful percent transmission curves normally reported in the literature. This task can be lightened to some extent with a device reported by Gershinowitz and Wilson¹ but this conversion remains a tedious and time consuming portion of spectral analysis.

The electric analog computer first reported by Fuoss and Mead² and modified by Zerwekh³ can be further simplified to provide a rapid, reasonably accurate method of making these calculations. An alternative solution to the problem is the more arduous task of converting the single beam instrument into one using a split beam.

The basic changes reported here eliminate the need for a second recorder and avoid the use of specially wound coils. This allows construction of the computer as a compact unit attached directly to the door of the spectrometer recorder. The door is swung open to permit normal use of the recorder.

The electrical circuit is essentially the same as, and the component designation is identical, with that of Zerwekh. The values of the components used were $R_1 = RI_0 = 50,000\Omega$; $R_2 = RI = 100\Omega$; $R_3 = 500\Omega$; a 2 v battery. Beckmann Helipot, linear to 0.1 percent, were used for R_I and RI_0 as substitutes for the specially wound coils used by Zerwekh. Figure 1 shows a top view of the actual computer (not to scale).

Attached to each potentiometer is an extending shaft which is threaded in the center section and has a crank at the outer end. A flexible wire, fastened in the threads and over a pulley at the other end of the computer, is attached to a carriage sliding on a rail of rectangular cross section. As the crankshaft is turned, the wire moves the carriage. The diameter of the shaft in the threaded section is such that a pointer mounted on the sliding carriage moves over the entire range of the recorder scale as the resistance of the Helipot is varied from zero to maximum. The guide rail is rectangular to minimize vertical motion of the pointer. The other Helipot is connected, in a like fashion, at the opposite end of the computer to a second carriage on the same guide rail.

The same recording instrument used to register the spectra can be used to plot the I/I_0 curve. Jack connections must be placed in the input circuit of the recorder so that either the spectrometer signal or the computer signal can be received by the recorder.

The background and desired spectrum are superimposed as usual. The paper in the recorder is then turned back to the beginning of the two spectra. The computer output is substituted for the spectrometer signal by means of the appropriate jack connections and the power supply circuit closed. With the two Helipot set on zero resistance the zero adjustment clamps are loosened and

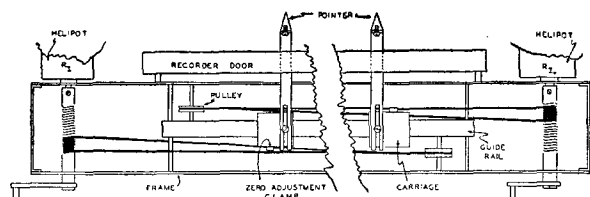


FIG. 1 Top view of computer.

the two carriages moved until both pointers are set on the no-signal reference line of the two spectra. Now the two pointers are placed together at the center of the scale and the value of R_3 is changed so that the pen records 100 percent transmission when the pointers are together. In the case that the two spectra do not have a common reference line, the independent zero-adjustments of R_I and RI_0 permit separate settings. The R_3 adjustment would then be made with the pointers not together but at equal distances from their respective reference lines.

The recorder is started and while the two cranks are manipulated so that the pointers follow the curves, the pen traces I/I_0 . This latter curve will necessarily be displaced from the spectra since there must be clearance between the pointers and the pen. Adjustment screws are provided on the pointers so that this offset can be made a convenient amount. If the background is not perfectly aligned with the sample spectra, correction can be made with these screws.

In some spectra it may be difficult to record the background and sample spectra with the same amplification (e.g. solid spectra in which reflection loss is significant). If a standard test signal is recorded at the time of the experiment, the amplification need not be identical for both runs. In this case R_3 is adjusted so that the pen records the ratio of the test signal deflections when the pointers are together.

The ease of operation and accuracy of this computer are the same as those of the instrument described by Zerwekh.

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¹ H. Gershinowitz and E. B. Wilson, J. Chem. Phys. 6, 200 (1938).

² R. M. Fuoss and D. J. Mead, Rev. Sci. Inst. 16, 223 (1945).

³ C. E. Zerwekh, Rev. Sci. Inst. 20, 371 (1949).

Note on Crystals Suitable for Double Crystal X-Ray Spectroscopy in the Region 3 to 15Å*

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July 26, 1950

THE problem of finding crystals of large enough grating space to be useful in the vacuum region above 3Å has been considered from time to time. Typical of studies on crystals suitable for single crystal x-ray use in vacuum spectrometers was that of Stenstrom¹ who found mica, gypsum, beryl, sugar, CaSO_4 and $\text{K}_4\text{Fe}(\text{CN})_6$ to be satisfactory. Double crystal work imposes more stringent requirements on the crystals than does single crystal work because the x-ray beam is of the order of a hundred times broader in the double crystal arrangement and correspondingly larger areas of the crystal faces are used. Thus one is limited to compounds for which large crystals are available and for which the crystal faces are relatively perfect. The test of perfection is the width of the parallel rocking curve obtained by rocking the second crystal about the position (1, -1) in which it is parallel to the first crystal, since the narrower the rocking curves, the higher the

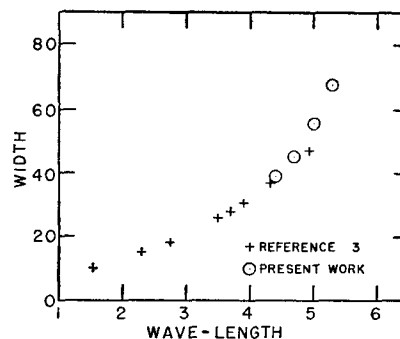


FIG. 1. Full width (in seconds) at half-maximum of the parallel rocking (1, -1) curve for calcite as a function of the wave-length in Angstroms.

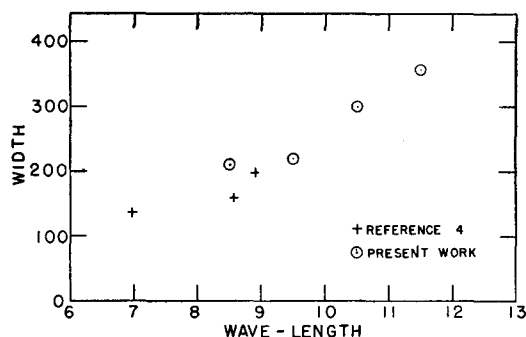


FIG. 2. Full width (in seconds) at half-maximum of the parallel rocking (1, -1) curve for beryl as a function of the wave-length in Angstroms.

resolution the crystals will achieve in the (1, +1) position. An additional requirement for the spectrometer used in this laboratory² is that the crystals must have properties suitable for use in a vacuum which is essentially as good as that in the x-ray tube itself, because there is no film covering the window between the x-ray tube and the spectrometer chamber.

As far as we are aware the only studies of the parallel rocking curves in this wave-length region are those of Parratt³ who obtained curves on calcite out to 4.94 Å and on quartz out to 7 Å, and those of Munier, Bearden, and Shaw⁴ who used beryl at wave-lengths up to 8.9 Å. It was the purpose of the present investigation to extend these results to longer wave-lengths and to determine if crystals of other compounds might be useful.

The results for calcite³ and beryl⁴ have been verified in the present work and have been extended to 5.3 Å and 11.5 Å, respectively. The data are presented in Figs. 1 and 2. It is evident that the resolution offered by calcite is excellent for use out to 5.3 Å which is about as far as one can go with the small grating space. Beryl offers satisfactory resolution out to 11.5 Å. Beryl had so low a reflecting power at wave-lengths shorter than the AlK edge as to be useless in this region. The calcite surfaces were cleaved and etched lightly with dilute HCl whereas the beryl surfaces were natural (10 $\bar{1}0$) faces. The radiation was the continuous spectrum from a tungsten target and the x-ray tube was operated at a voltage low enough so as not to produce second-order radiation for the particular glancing angle being studied.

An effort was made to find other crystals of large grating space which would be useful at these wave-lengths in the double crystal spectrometer. Other crystals investigated were mica, gypsum, quartz, and topaz. Mica and gypsum seemed particularly promising because of the large grating space for the natural cleavage faces and because these faces are easily available with large area. Many attempts were made to secure parallel rocking (1, -1) curves for two mica crystals, but without success. Thin crystals, thick crystals, and thin crystals pasted onto optical flats were all tried but no (1, -1) curves were obtained. Presumably this was because of macroscopic waviness of the natural mica crystal surfaces, which was reduced almost to microscopic waviness upon cementing to flat surfaces but was not eliminated; thus portions of the two crystal faces were parallel to each other over large rocking angles and no sharp (1, -1) peak was produced. Gypsum did not have suitable vacuum properties for use in an x-ray vacuum. The pumps did not achieve an operating vacuum even after prolonged pumping and the crystals gradually turned white. Presumably water of crystallization was slowly given off and plaster of Paris was formed. Coating the crystals with glyptal proved ineffective. Quartz and topaz were found to have too low a coefficient of reflection to be useful. Sugar and $K_4Fe(CN)_6$ were not tested because they were not available in large enough size. Gratings built up by successive dipping in a proper organic liquid have not as yet been tried nor would they appear to offer much promise because of the stringent

requirements which double crystal work makes on the perfection of the grating.

* Supported in part by the ONR.

¹ W. Stenstrom, *Ann. d. Physik* **57**, 347 (1918).

² S. T. Stephenson and F. D. Mason, *Phys. Rev.* **75**, 1711 (1949).

³ L. G. Parratt, *Phys. Rev.* **41**, 561 (1936); *Rev. Sci. Inst.* **5**, 395 (1934); *Rev. Sci. Inst.* **6**, 387 (1935).

⁴ Munier, Bearden, and Shaw, *Phys. Rev.* **58**, 537 (1940).

A Greaseless Gas Flow Valve

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August 25, 1950

IN experiments where appreciable pressures (1 mm Hg < p < 1 atmos.) of highest purity samples are to be introduced into a high vacuum system, the following greaseless valve has been found very useful. In essence, it consists of a small volume that terminates in three sintered glass disks (Corning grade F) and is partially filled with Hg (see Fig. 1). Disk A connects to the gas sample, disk B to the vacuum system, and disk C to a manipulating vacuum-pressure line. The valve is closed when C is at atmospheric pressure (Fig. 1a). Gas will flow from the sample bulb through disks A and B into the vacuum system as soon as the pressure above disk C is reduced sufficiently to pull the mercury up (Fig. 1b). It is readily seen that this occurs when

$$p_c \leq p - \Delta h.$$

This valve will operate satisfactorily under all conditions if the following requirements are met:

1. When the valve is closed, the Hg level in the manipulating arm should lie above a (Fig. 1a), thereby keeping the pressure to the right of disk B somewhat above atmospheric. This will prevent air from bubbling from the system through disk B, the mercury, and disk A into the sample bulb at times when the system is let up to air and the sample is partially depleted. This requirement is met by arranging the three disks as shown in Fig. 1a and filling the tube to the level of disk A.

2. Disk C should be mounted in such a way that disk B is completely bared when the valve is open. At the same time, the bend of the U-tube should lie below level b to prevent part of the sample from bubbling through into the manipulating arm.

The valve may be thoroughly degassed at elevated temperatures before the mercury is distilled into it. In this respect it is more versatile than the simpler gas introduction system described by Taylor and Young¹ who obtain flow by making contact between two movable sintered disks in a pool of Hg. Because the gas flow rate through fine sintered disks is relatively slow, it is easy to time the valve manipulation so as to admit any desired pressure to the vacuum system.

In a simpler version of this valve, the mercury is moved mechanically by a stainless steel bellows (see Fig. 2) which is attached to a manipulating screw. While this method is not amenable to

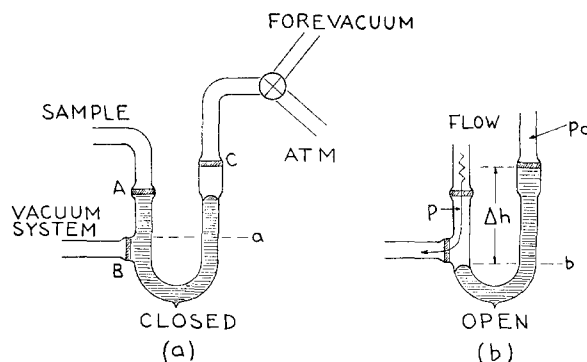


FIG. 1. Essential parts of sintered glass valve.