Rapid-scanning microphotometry

By A. W. Helz

Trace Elements Investigations Report 579

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RAPID-SCANNING MICROPHOTOMETRY

By A. W. Helz

ABSTRACT

A rapid-scanning microphotometer is described with which a 10-inch spectrum may be scanned in two minutes. The resulting chart may be 60, 300, or 1,500 cm long (wavelength scale) and 4 cm high (intensity scale). Commercially available components are used.

INTRODUCTION

Commercially available microphotometers for obtaining quantitative spectrographic data are needlessly slow for many applications in which the potential precision of the microphotometer exceeds errors elsewhere in the procedure. Such is the case in spectrochemical work when, because of the nature of the problem, no adjustment is made for variable matrices and no internal standard is used. A recording microphotometer using commercially available components is described below for such applications in which high precision is compromised in favor of scanning speed. The solution to this problem is very similar to that of Junkes and Salpeter 1/ noted at the time of writing this report.

A direct-reading microphotometer was converted to a rapid-scanning instrument by making the following changes. A Jarrell-Ash no. 200 projection comparator microphotometer was used because of its availability.

1/ Junkes, J., and Salpeter, E. W., Ein Schnellregistrierphotometer zur Auswertung von Spektralanfnahmen: Microchimica Acta, no. 2-3, 534 (1955).

This instrument is easily converted to this application, but there is nothing uniquely determining its choice. Coincidentally, this same instrument was used by Junkes and Salpeter. The changes made were: (1) a plate carriage drive incorporating an accurate screw and synchronous motor were installed, (2) a 929 phototube was installed, and (3) a recording system was attached consisting of a d-c amplifier and electromagnetic oscillograph (The Brush Development Co. BL-202 double-channel oscillograph and BL-932 d-c amplifier were used). The critical requirements are an oscillograph writing speed with a relative flat response from d-c to 100 cycles per second, a pen peak-to-peak amplitude of at least 4 cm, and accurate chart paper drive of several speeds up to at least 25 mm/sec (125 mm/sec chart speed is available on the instrument used). With this system, two minutes are sufficient time for scanning a 10-in. spectrum. The resulting record may be 60, 300, or 1,500 cm long (wavelength scale) and 4 cm high (intensity scale).

This work is part of a program conducted by the U. S. Geological Survey on behalf of the Division of Raw Materials of the U. S. Atomic Energy Commission.

DESCRIPTION OF THE MICROPHOTOMETER

A photograph of the microphotometer is shown in figure 1. The optical system is the type in which an intense narrow light beam is focused on the spectrogram which in turn is magnified ten times and focused on a slit directly over a photoelectric receiver. Considerations of speed of response in combination with amplifier gain, color response curve, stability, and battery requirements resulted in the choice of the vacuum-type photoelectric cell (929) for the receiver. This arrangement was sufficiently sensitive in combination with the amplifier to make it unnecessary to resort to a

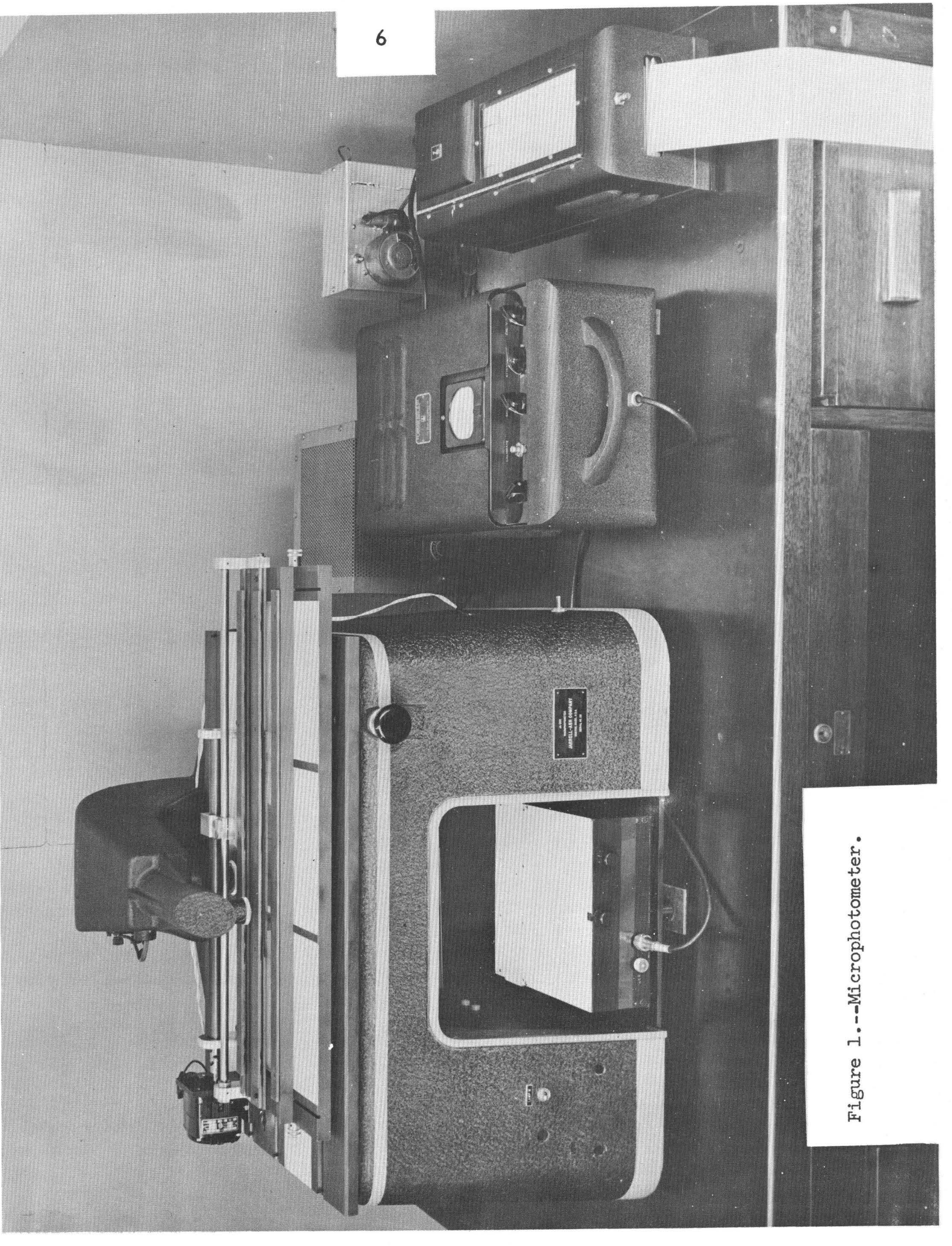

Figure 1.--Microphotometer.

multiplier phototube. The photocell is mounted directly behind the slit in a totally enclosed (for light and electrical shielding) brass box which also includes a 67-1/2 volt dry battery.

Attempts to use the steel cable drive on the instrument proved it to be inadequate for this use because (1) variable slippage of the friction drive made inaccurate the correspondence between line separation on the plate and that on the chart and (2) vibration of the plate carriage could cause false intensity indications through greatly altered scanning speed for an individual line. A direct drive screw was installed. The screw is 5/8 in. in diameter, 14 in. long, and is threaded 20 turns per inch. This is driven with a geared synchronous motor which turns the screw at a rate of 100 rpm to give the two-minute-per-10-in. scanning speed. A bar attached to the carriage and parallel to the screw passes through a split hole in the traveling nut as shown in figure 1. A thumb screw on the nut permits disengaging the plate carriage in order to facilitate alining the plate with rapid hand motion of the carriage. If the plate-driving motor and the oscillograph motor both are synchronous, the chief sources of error remaining in the wavelength scale are paper slippage and shrinkage. A second channel recording on the same paper may be used to obtain better wavelength precision by recording time markers in synchronism with the plate and chart motors.

The maximum acceptable scanning speed depends upon the pen amplitude loss with increased speed which is a function not only of the oscillograph response speed, sensitivity, and optics of the system, but also of the line width, sharpness, and blackness. Spectrograms taken with an 0.025-mm slit were scanned at various speeds. The diminution in pen amplitude became observable at a carriage speed of about 2 mm/sec, i.e., 2 minutes for a 10-in. spectrum. At a scanning speed of 6 mm/sec the pen amplitude is about 80 percent the maximum attained with sufficient time.

Good regulation of the power supplies for the scanning light and the d-c amplifier are important, but the requirements here are not as rigid as for the conventional microphotometers because of the reduction of scanning time by a factor of 25. The amplifier has balance and gain controls which become the maximum and minimum transmission (clear plate and total blackness) settings adjusted to write at the two extremes of the chart, 2 cm on each side of the mechanical zero position of the pen.

The sensitivity of the system is such that there is a considerable amount of gain to spare when using a slit 0.10 mm wide by 1 cm high over the phototube, the equivalent dimensions at the plate being one-tenth as large. This sensitivity is also a function of the light source and subsequent optics which were satisfactory on the original equipment without any changes.

EXAMPLES OF PERFORMANCE

Figures 2 and 3 illustrate charts produced by the instrument. They are all made from an iron spectrum, second order, approximately 2.8 A/mm on the plate. Figure 2 is a two-minute scan of the entire plate width for which the chart speed was 5 mm/sec. The optional 0.6 second timing marks for added wavelength precision as produced by the second channel are shown below. Two sections of the spectrum indicated in figure 2 A are shown in B and C. These were made with a chart speed five times greater or 25 mm/sec. Wavelengths of a number of iron lines are marked on all figures of charts as well as the wavelength values in angstroms of 1 cm chart length and of 1 unit timing mark.

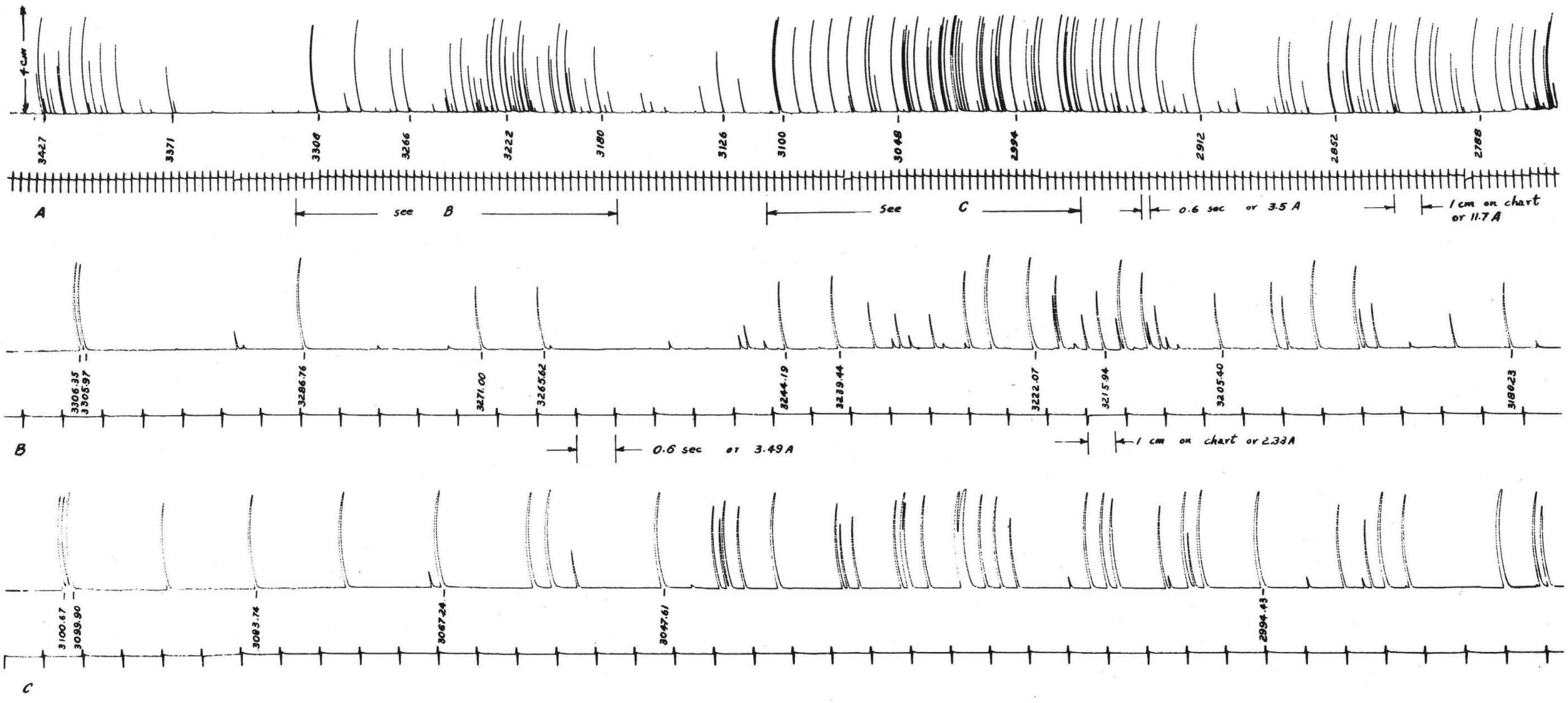

Figure 2.--Iron spectrum, second order, full 10-in. scanning in two minutes with a chart speed of 5 mm/sec. (The 0.6-second timing marks are shown below.) B and C are expanded parts of A.

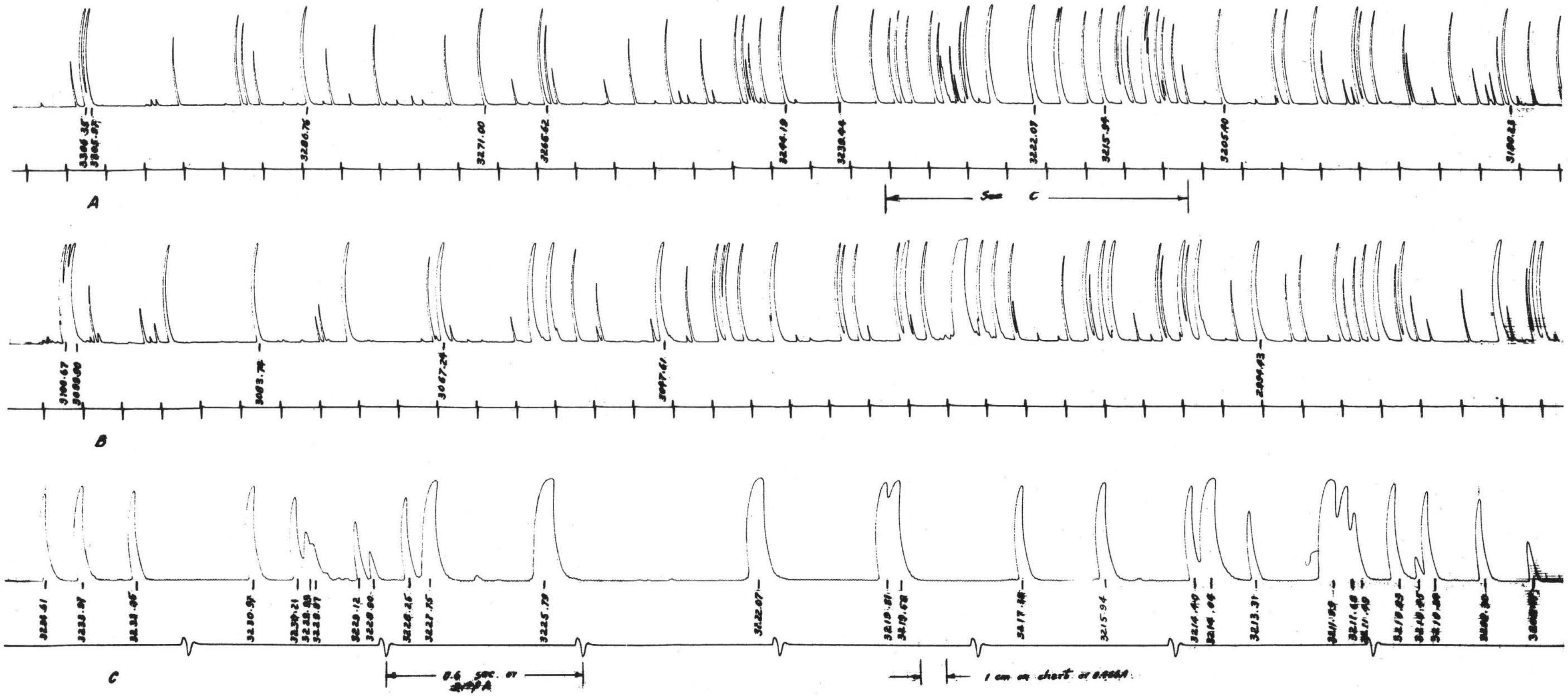

Figure 3.--Iron spectrum, same as figures 2A and B, with exposure increased 4 times. C is expanded part of A with a maximum chart speed of 125 mm/sec.

Figures 3A and B differ from 2B and C only in one respect; the spectrum scanned for figure 3 was made with an exposure 4 times that for figure 2. A section of the spectrum indicated on figure 3A is shown in figure 3C magnified in wavelength by running the chart paper at 125 mm/sec.

The charts referred to above were all run consecutively without any changes in the settings of the instrument. Therefore, an idea of the intensity reproducibility can be had by comparing lines of figures 2B and C with the corresponding sections of figure 2A. Similarly, the ordinates of the lines in figure 3C are comparable with those of the section marked in figure 3A. Measurements of the charts are facilitated with a 10X magnifier having in the focal plane a 2 cm scale divided to 0.1 mm. Four full-length charts run at the intermediate speed and taken at different times were measured to give an estimate of the kind of precision one may expect. Thirty-four lines about equally spaced throughout the spectrum with a variety of line intensities were measured on each of the charts. Reading the ordinates on a scale of 0 to 80 units, actually 0 to 40 mm, and comparing each of the four individual values with their average shows an average (4 values for each of 34 lines) reading precision of ± 0.4 unit, ± 0.2 mm, or ± 0.5 percent. The extreme departures from the average for all 136 readings were +1.0 and -1.2 units, 0.5 and -0.6 mm, or 1.3 and -1.5 percent.

The average error for measurements of distance between lines (for wavelength identification) is 0.2 percent on the equipment described. The maximum error found was 0.8 percent. These errors were not random, indicating that the paper was running slower or faster than for the reference during the entire run. An occasional run, not in the above test, deviated considerably more than shown above. Because of this uncertainty, the timing marker subsequently was added.

APPLICATION

An interesting application is to measure lines for identification as one might do with a measuring comparator but requiring only the 10X hand magnifier. Using the iron lines 3205.400, 3215.940, 3217.380, and 3244.192 A as reference points the wavelengths of 30 lines were calculated by linear interpolation from measurement of the fast chart (125 mm/sec). Comparison with the M. I. T. tables showed an average difference between calculated and table wavelengths of 0.041 A and a maximum difference of 0.090.

Probably more important is the application to spectrochemical work referred to in the introduction. A scanning-time reduction by a factor of 25 is obtained with little loss in resolution and a negligible loss, for many problems, in the precision of intensity measurements.

Endeavors to improve the use of photographic materials for spectroscopy in spite of the superiorities of direct-reading photoelectric devices are warranted because both the multiple channel and the rapid scanning direct reading instruments increase in complexity with the number of lines to be recorded. The photographic emulsion has at present no competition in regard to simplicity for recording the total radiation of hundreds of lines simultaneously and continuously during the entire exposure time.