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WAVELENGTH REPEATABILITY OF A CARY MODEL 14R SPECTROPHOTOMETER.(U)
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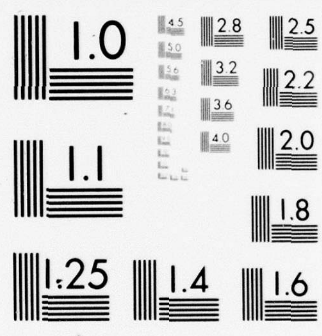

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NWC-TP-6109

WAVELENGTH REPEATABILITY OF A CARY MODEL 14R SPECTROPHOTOMETER

Research rept.

by
Marian E. Hills
Research Department

MAY 1979

20p.

NAVAL WEAPONS CENTER
CHINA LAKE, CALIFORNIA 93555

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FOREWORD

This report describes a study made to determine the precision of wavelength measurements made with a Cary Model 14R spectrophotometer.

This report was reviewed for technical accuracy by Allen L. Olsen and H. E. Bennett.

Approved by
E. B. ROYCE, *Head*
Research Department
31 May 1979

Under authority of
W. L. HARRIS
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Commander

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(U) *Wavelength Repeatability of a Cary Model 14R Spectrophotometer*, by Marian E. Hills. China Lake, Calif., Naval Weapons Center, May 1979. 14 pp. (NWC TP 6109, publication UNCLASSIFIED.)

(U) In our laboratory Cary spectrophotometers are used to measure the transmittance of optical components and materials. One instrument in particular, a Cary Model 14R spectrophotometer, is used to calibrate narrow-band interference filters. The precision of the wavelength measurements was determined using the D_{β} line of the deuterium source lamp as a wavelength standard.

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INTRODUCTION

In our laboratory Cary spectrophotometers are used to measure the transmittance of optical components and materials. One instrument in particular, a Cary Model 14R spectrophotometer, is used to calibrate narrow-band interference filters used in a rocketborne ozonesonde. Preliminary assessment of repetitive scans of a filter showed that the center of the bandpass appeared to shift to longer wavelengths in an orderly manner. This raised the question of whether the instrument was shifting its calibration (due perhaps to temperature changes), whether some temperature effect was shifting the bandpass of the interference filter, or whether some other effects or combination of effects was occurring.

This report deals with studies made to determine the precision of wavelength measurements made with the spectrophotometer and to show how the instrument might be shifting its calibration during operation.

EXPERIMENTAL

The 4860 Å line (D_β) of the deuterium source lamp was used as the wavelength standard. It was found that the lamp contained hydrogen as well as deuterium (Figure 1). Temperatures were measured with Type K thermocouples; the thermocouples were not calibrated because the change in temperature with time and instrument usage -- not temperature per se -- was sought.

The wavelength temperature coefficient of the spectrophotometer is stated to be 0.3 Å per degree Celsius.[1] However, no information is given about where the temperature changes should be measured if the entire monochromator is not at the same temperature. Since knowledge about specific locations at which thermocouples should be placed was not available, the decision was made not to open the monochromator

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[1] Varian Company. *Instructions for Cary Recording Spectrophotometer Model 14R*, Varian, Instrument Division, Palo Alto, Calif., Handbook (undated), p. 2.

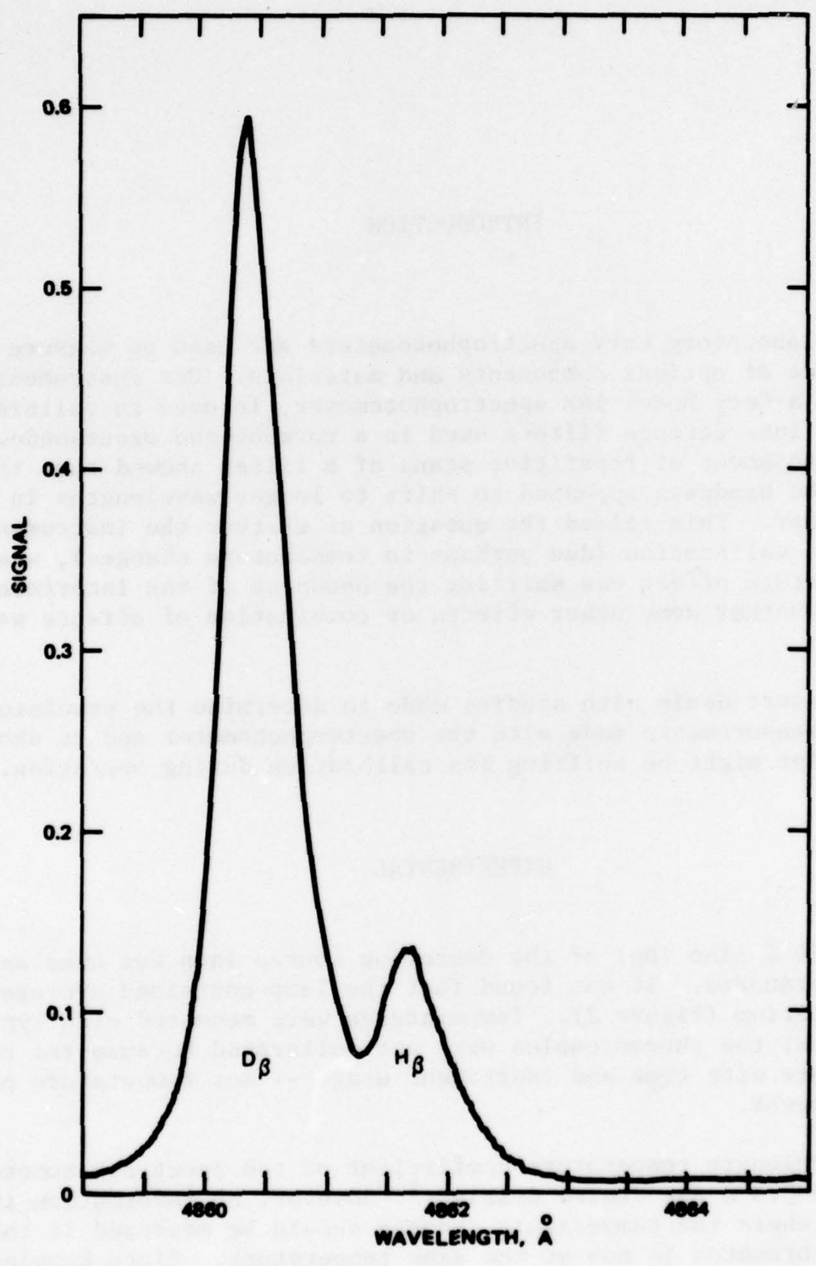

FIGURE 1. D_β and H_β Spectral Lines of Source Lamp.

housing and expose the elements to dust in order to place thermocouples inside of the monochromator housing, but rather to place thermocouples on the exterior of the housing. Thermocouples were placed on the lower left front wall of the housing and on the top of the housing at the right rear (under the heat shield); thermocouples were also placed in the sample compartment. One thermocouple was hung in air in the vicinity of the spectrophotometer.

The output from the thermocouples was read with a data logger which recorded the temperatures directly with a resolution of 0.1°C.

Two scanning speeds were employed: 1/4 Å/sec and 1/8 Å/sec; the chart was driven at 5 in./min. The standard chart paper for the instrument has three divisions per inch on the wavelength axis. Thus the combinations of scanning speeds with chart speed resulted in the analog record having 1 Å/barleycorn or 1/2 Å/barleycorn (a barleycorn is 1/3-inch). Since no readily available scales divide one-third of an inch into 5 or 10 parts, scales having 15 divisions/in. and 30 divisions/in. were made with a Gerber plotter. These scales are reproduced in Figure 2 for the convenience of others.

FIGURE 2. Scales of 30 Divisions Per Inch and 15 Divisions Per Inch.

In order to take the backlash out of the wavelength drive mechanism, the following regime was employed (the particular spectrophotometer used has a two-speed motor which gives one-half or one-fourth of the speed of the motor usually supplied with the instrument):

a. Scan from 5400 Å to 4900 Å at 50 Å/sec or 25 Å/sec.

b. Scan from 4900 Å to $\sim$4875 Å at 2 1/2 Å/sec or 1 1/4 Å/sec.

c. Scan from $\sim$4875 Å to 4859 Å at 1/4 Å/sec or 1/8 Å/sec.

d. Record analog data from 4865 Å to 4859 Å.

The instrument was equipped with a transmittance slidewire and operated in the reference mode. The reference beam was blocked with paper and tape; the reference compartment was kept closed as was the sample compartment. A piece of black tape was placed across the lower portion of the wavelength counter window in order to reduce the parallax error.

The wavelengths at which the line attained one-half of its peak value were read from the 4859 Å position marked on the chart. The line position, designated by λ_c, was then calculated (the average of the wavelengths at the two half-power points).

RESULTS AND DISCUSSION

After the spectrophotometer had been warmed up for one hour and a few preliminary scans had been made, scans were made at approximately 15-minute intervals for the next hour and at 30-minute intervals for the next 6 1/2 hours, at which time four scans were made in rapid succession. Table 1 gives the band position for the D_β line and the temperature recorded from the thermocouple attached to the front wall of the monochromator housing. For the twenty-two scans tabulated the mean and standard deviation of the wavelength, λ_c, are 4860.53 Å and 0.19 Å. The mean and standard deviation for the five scans numbered 18A through 22A are 4860.32 Å and 0.01 Å. The tabulation of wavelength to 0.01 Å resulted from interpolation of the chart and is not meant to imply an accuracy of 0.01 Å. The wavelength and temperature data are plotted as a function of time in Figure 3. Under the conditions at which the data shown in Figure 3 were collected the values of λ_c tend to decrease as the temperature of the front wall of the monochromator increases unless scans are repeated in rapid succession.

Another set of scans was made over a period of two days to confirm the previous results and to search for conditions under which scans made in quick succession would give the same position for the D_β line and not an increasing value. The data from the first day are tabulated in Table 2; they are plotted in Figure 4 except for scans made at 1/4 Å/sec. The first scan was made 16 minutes after the instrument was turned on. The mean value and standard deviation for the eighteen 1/8 Å/sec scans are 4860.59 Å and 0.16 Å. The data show the same general trends as did the earlier data.

The instrument was left on overnight. On the second day the instrument was kept scanning in wavelength (usually between ∿5400 Å and ∿4900 Å) during the time that scans were not being made until mid-afternoon. The data are tabulated in Table 3 and plotted in Figure 5. The temperature of the monochromator housing increased but seemed to level off near 34°C with continuous scanning. However, long before the temperature leveled

TABLE 1. D_β Line of Source and Temperature of Monochromator Housing. Scanning speed: 1/8 Å/sec.

Scan no.	Time,[a] t, min.	Wavelength, λ_c, Å	Temperature[b] °C
6A	59.95	4860.86	24.9
7A	75.27	4860.85	25.4
8A	89.97	4860.86	26.0
9A	106.25	4860.82	26.6
10A	120.05	4860.71	27.0
11A	150.05	4860.59	27.9
12A	182.82	4860.56	28.6
13A	210.23	4860.52	29.2
14A	239.90	4860.42	29.6
15A	244.47	4860.52	29.7
16A	270.00	4860.48	30.2
17A	299.87	4860.40	30.6
18A	330.08	4860.32	30.9
19A	359.93	4860.32	31.2
20A	390.07	4860.34	31.3
21A	420.60	4860.30	31.5
22A	450.22	4860.32	31.6
23A	480.72	4860.40	31.6
24A	509.87	4860.40	31.6
25A	513.13	4860.45	31.5
26A	515.93	4860.57	31.6
27A	518.80	4860.66	31.5

[a] Instrument turned on at t = 0.

[b] Measured on front wall of monochromator housing.

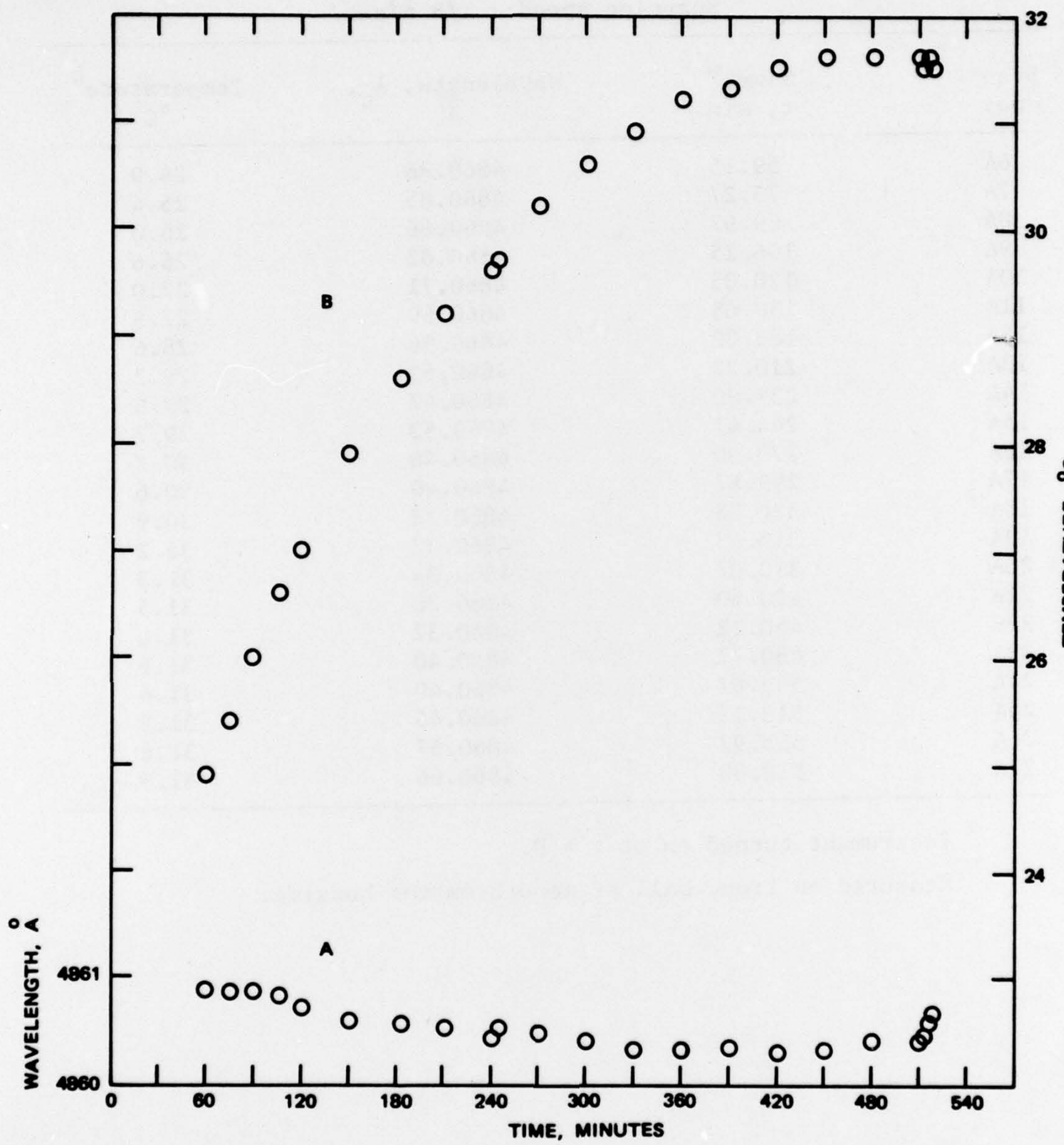

FIGURE 3. Plot of Data Tabulated in Table 1. A. Position of D_β line; B. Temperature at front wall of monochromator housing.

TABLE 2. D_β Line of Source and Temperature of Monochromator Housing.

Scan no.	Scan speed, Å/sec	Time,[a] t, min.	Wavelength, λ_c, Å	Temperature,[b] °C
1B	1/8	31.07	4860.70	23.7
2B	1/8	45.55	4860.77	24.1
3B	1/8	59.93	4860.80	24.6
4B	1/4	62.55	4860.79	24.7
5B	1/8	89.63	4860.64	25.7
6B	1/4	92.02	4860.67	25.8
7B	1/4	94.38	4860.78	25.9
8B	1/8	119.62	4860.80	26.8
9B	1/4	122.05	4860.79	26.8
10B	1/8	149.40	4860.75	27.6
11B	1/4	152.05	4860.70	27.7
12B	1/8	180.05	4860.62	28.3
13B	1/4	182.52	4860.57	28.3
14B	1/4	184.68	4860.70	28.4
15B	1/8	209.70	4860.58	29.0
16B	1/4	212.27	4860.62	29.1
17B	1/8	239.78	4860.48	29.5
18B	1/4	242.33	4860.58	29.5
19B	1/8	300.00	4860.40	30.0
20B	1/4	302.40	4860.50	30.1
21B	1/8	360.15	4860.35	30.5
22B	1/8	364.18	4860.40	30.5
23B	1/8	367.82	4860.52	30.5
24B	1/8	371.57	4860.59	30.6
25B	1/8	375.07	4860.68	30.7
26B	1/8	378.15	4860.74	30.8
27B	1/8	419.43	4860.48	31.0
28B	1/8	464.85	4860.35	31.0

[a] Instrument turned on at t = 15 min.

[b] Measured on front wall of monochromator housing.

TABLE 3. D_β Line of Source and Temperature of Monochromator Housing. Scanning speed: 1/8 Å/sec.

Scan no.	Time,[a] t, min.	Wavelength, λ_c, Å	Temperature,[b] °C
29B	47.88	4860.40	30.3
30B	51.88	4860.44	30.3
31B	55.18	4860.54	30.4
32B	59.48	4860.62	30.4
33B	63.03	4860.77	30.5
34B	66.48	4860.88	30.6
Instrument Scanning at 1/8 Å/sec Between 34B and 35B			
35B	120.08	4861.45	32.1
36B	123.83	4861.42	32.3
37B	127.32	4861.47	32.3
38B	130.75	4861.44	32.4
39B	134.27	4861.40	32.5
40B	137.78	4861.44	32.5
41B	141.37	4861.44	32.7
42B	144.78	4861.42	32.7
43B	148.30	4861.36	32.7
44B	151.80	4861.40	32.8
Instrument Scanning at 1/8 Å/sec Between 44B and 45B			
45B	213.57	4861.43	33.4
Instrument Scanning at 1/8 Å/sec Between 45B and 46B			
46B	300.98	4861.42	33.9
47B	304.52	4861.38	34.0
48B	307.90	4861.44	34.0
49B	311.43	4861.38	34.0
50B	314.70	4861.38	33.9
Instrument Scanning at 1/8 Å/sec Between 50B and 51B			
51B	358.83	4861.38	34.1
52B	362.23	4861.41	34.1
53B	365.65	4861.34	34.1
54B	369.07	4861.35	34.1
55B	372.52	4861.37	34.2

TABLE 3. (Contd.)

Scan no.	Time,[a] t, min.	Wavelength, λ_c, Å	Temperature,[b] °C
Instrument Not Scanning Between 55B and 56B			
56B	418.97	4860.41	33.1
57B	422.40	4860.42	33.0
58B	425.70	4860.50	33.0
59B	429.13	4860.62	32.9
60B	432.47	4860.65	33.0
Instrument Not Scanning Between 60B and 61B			
61B	453.15	4860.50	32.9
62B	456.58	4860.56	32.9
Instrument Not Scanning Between 62B and 63B			
63B	526.32	4860.22	31.8
64B	529.72	4860.30	31.8

[a] Instrument turned on 23.75 hr. before t = 0.

[b] Measured on front wall of monochromator housing.

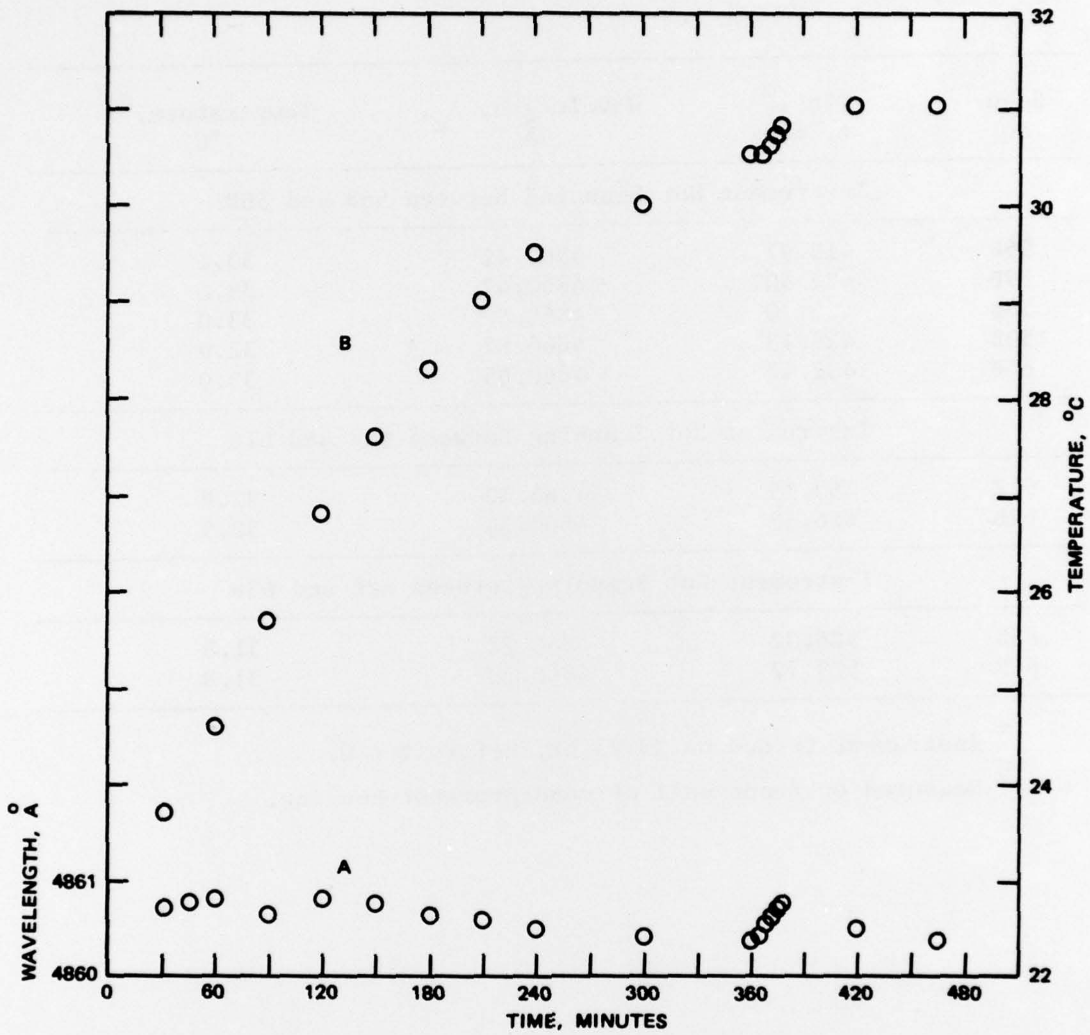

FIGURE 4. Plot of Some of the Data Tabulated in Table 2. A. Position of D_β line; B. Temperature at front wall of monochromator housing.

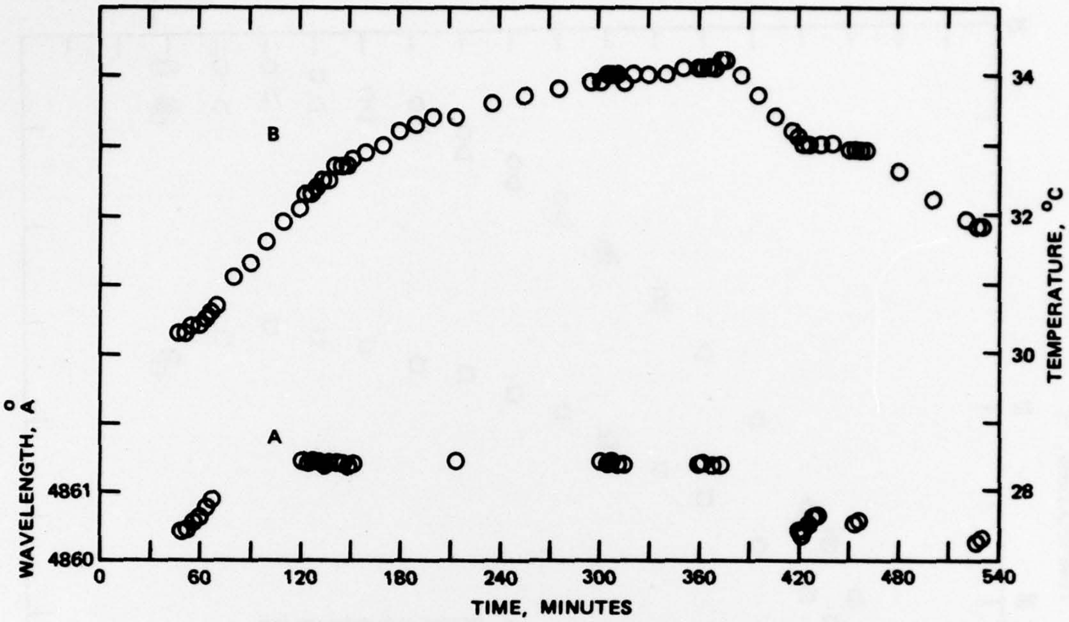

FIGURE 5. Plot of Data Tabulated in Table 3. A. Position of D_β line; B. Temperature at front wall of monochromator housing.

off the wavelength stabilized (see the period of 200-400 minutes, Figure 5). Once the scanning was stopped the temperature began to fall and the wavelength λ_c became smaller. Also the shift of λ_c to larger values for scans made in rapid succession resumed. The mean and standard deviation of the line position for scans numbered 35B through 55B are 4861.41 Å and 0.04 Å.

Since the temperature attained by a sample and the time to reach this temperature are of interest in the case of samples whose transmittance and/or spectral response are temperature dependent, the temperature of the sample compartment of the instrument is of interest. The various temperatures monitored are plotted in Figure 6. The data were taken at the same time as the optical measurements shown in Figure 3.

CONCLUSIONS

If excellent stability of the wavelength scale of the instrument is important, then the spectrophotometer must be used in a specified manner. A regime in which the instrument is kept scanning in wavelength at all times leads to stability of wavelength measurements. Another regime leads to nearly constant (very slowly decreasing) values of λ_c

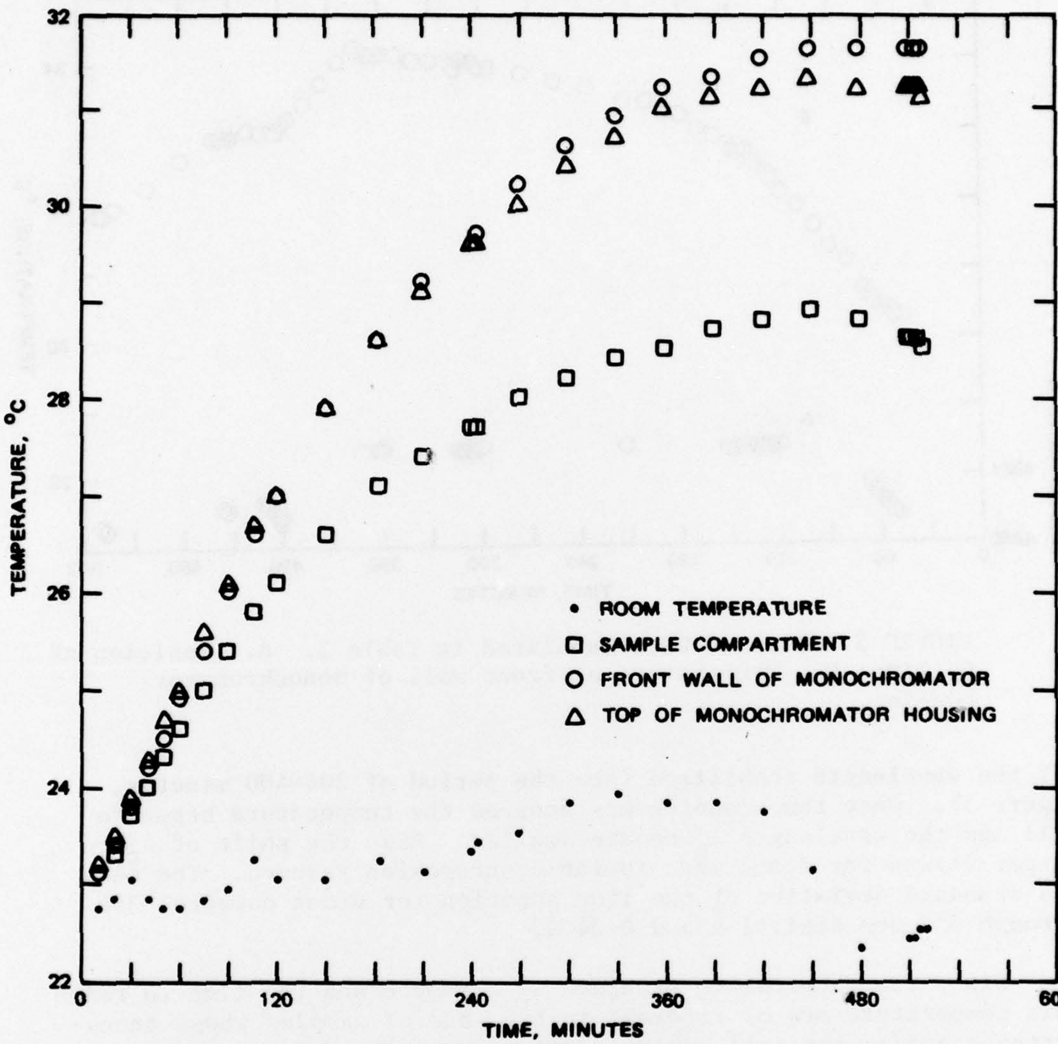

FIGURE 6. Temperature at Various Positions on Spectrophotometer During the Taking of Data Tabulated in Table 1.

during the time the temperature of the instrument is still increasing; this regime is a pattern of short scans (2-3 minutes duration) separated by non-scanning intervals of 30 minutes (Figure 3). It should be noted that the actual value of λ_c differs for the two regimes -- 4861.41 Å ± 0.04 Å for continuous scanning vs. 4860.32 Å ± 0.01 Å for individual short scans made at 30 minute intervals. The accepted value for the D_β line is 4859.99 Å.[2]

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[2] National Bureau of Standards. "Errors in Spectrophotometry and Calibration Procedures to Avoid Them," in *Standardization in Spectrophotometry and Luminescence Measurements*, National Bureau of Standards Special Publication 466, May 1977, p. 102.

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