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TECHNICAL REPORT
ARCSL-TR-83058

**EFFECT OF VISIBLE LIGHT ON THE
DIMERIZATION OF COBALT (II)
TETRASULFONATED PHTHALOCYANINE IN
WATER**

Prepared by
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Research Division

June 1983



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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Beelen and coworkers in the Netherlands recently reported that visible light enhanced the rate of selective oxidation of mercaptoethanol by dioxygen, catalyzed by cobalt (II) tetrasulfonated phthalocyanine (CoTSPC). The Dutch workers speculated that visible light promoted disaggregation of CoTSPC, producing more of the catalytically active monomer. If Beelen and associates are correct, then it is possible that the values for		

20. ABSTRACT (continued)

the dimerization constants of CoTSPC are in error, since the values were measured spectrophotometrically in the region of the spectrum where Beelen contends visible light promotes disaggregation. To test whether the dimerization constants are altered by the spectrophotometer, the absorbance of a CoTSPC was measured with an HP8450A diode-array UV-VIS spectrophotometer. The HP8450A measures the spectrum from 200 to 800 nm continuously, so by comparing the spectrum of CoTSPC in the UV region with the tungsten lamp active and then inactive, one can see whether the visible light affects aggregation. It turned out that no change was observed in the spectrum of CoTSPC when the tungsten lamp was turned off, so the dimerization constants measured in the visible region of the spectrum are valid and not affected by the spectrophotometer.

PREFACE

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EFFECT OF VISIBLE LIGHT ON THE DIMERIZATION OF COBALT (II) TETRASULPHONATED PHTHALOCYANINE IN WATER

1. INTRODUCTION

Beelen, daCosta Gomez, and Kuijer¹ recently reported that the rate of selective oxidation of mercaptoethanol by dioxygen, catalyzed by cobalt (II) tetrasulfonated phthalocyanine, is enhanced by a factor of two to four by light in the range 600 to 700 nm. Beelen and coworkers speculated that visible light promotes disaggregation of CoTSPC to form more of the catalytically active monomer. If Beelen and associates are correct, then it is possible that past measurements²⁻⁷ of the dimerization constant of CoTSPC are in error, since the dimerization constants were determined from absorbances measured near 660 nm, the region of the spectrum where light promotes disaggregation.

To test the hypothesis that visible light affects aggregation of CoTSPC, we measured the absorbance of CoTSPC with the HP 8450A diode-array spectrophotometer, which continuously measures the spectrum of a sample from 200 to 800 nm. One can monitor the spectrum of CoTSPC in the UV and see whether the UV spectrum changes when only the deuterium lamp is used.

2. EXPERIMENTAL

The CoTSPC was prepared by modifying a published procedure⁸ as reported by Ward and Seiders.^{6,7} Solutions of CoTSPC were prepared with distilled, deionized water.

Spectrophotometric measurements were made in 1-cm cells in a Hewlett-Packard HP 8450A UV-VIS spectrophotometer. The HP 8450A is a general-purpose, rapid-scanning instrument with a microcomputer controlling a photodiode array such that full spectra from 200 to 800 nm can be taken in one second. The sample is continuously illuminated by deuterium (UV) and tungsten lamps (visible); thus, one can see whether visible light affects the CoTSPC aggregation by comparing the spectrum in the UV with the tungsten lamp active and then turned off. A recent article discusses the advantages of diode-array spectrometry,⁹ while Thomas¹⁰ gives a detailed analysis of the theory and operation of the HP 8450A.

The solutions of CoTSPC were thermostatted with an HP 89100A temperature controller. The spectra were recorded on an HP 7470A plotter while absorbances at particular wavelengths were printed on an HP 2126A1P interactive terminal.

3. RESULTS AND DISCUSSION

In order to see whether visible light affects CoTSPC aggregation by monitoring a change in the UV spectrum, one must first establish that the UV spectrum is sensitive to changes in aggregation. To this end, the first experiment was done with a 1.97×10^{-5} M solution of CoTSPC that was

thermostatted at various temperatures from 20° C to 84° C to see whether the UV spectrum changed as more monomer formed as the temperature increased. Figure 1 illustrates the results of this experiment, while table 1 summarizes the temperatures and the peak absorbances. One can see that the peak absorbance near 320 nm increases with monomer concentration, and an isosbestic point appears at 308 nm. It is also clear why past workers preferred to monitor at 660 nm; the change in the visible region is twice as great as in the UV portion.

Table 1. Peak Absorbances Measured for a $1.97 \times 10^{-5}M$ Solution of CoTSPC at Various Temperatures

Temp °C	λ_{max} nm	Absorbance	λ_{max} nm	Absorbance
20.1	320	1.046	660	0.952
30.0	320	1.060	660	1.020
40.0	322	1.080	660	1.104
50.0	323	1.105	662	1.202
60.0	325	1.130	662	1.306
70.0	324	1.159	662	1.405
79.9	325	1.192	662	1.486
84.8	325	1.215	664	1.516

The next experiments compared the UV spectrum with the visible lamp active and then turned off. These experiments were also run at various temperatures in order to monitor the absorbance at the isosbestic point. If visible light only affects the aggregation of CoTSPC, the isosbestic should not change when the tungsten lamp is turned off; thus the isosbestic acts as a check on the precision of the experiments.

Figure 2 illustrates the spectra of CoTSPC at 20, 30, and 40° C with both lamps active and with only the deuterium lamp on. Table 2 lists the measured peak absorbances along with the absorbances at the isosbestic point. One can see that the peak absorbance is 0.002 or 0.003 absorbance unit greater with the tungsten lamp; however, the isosbestic point is 0.003 absorbance unit greater as well. If the peak absorbances are corrected to a common value for the isosbestic point, the peak absorbances are within 0.001 absorbance unit.

6

ABSORBANCE

COTSPC

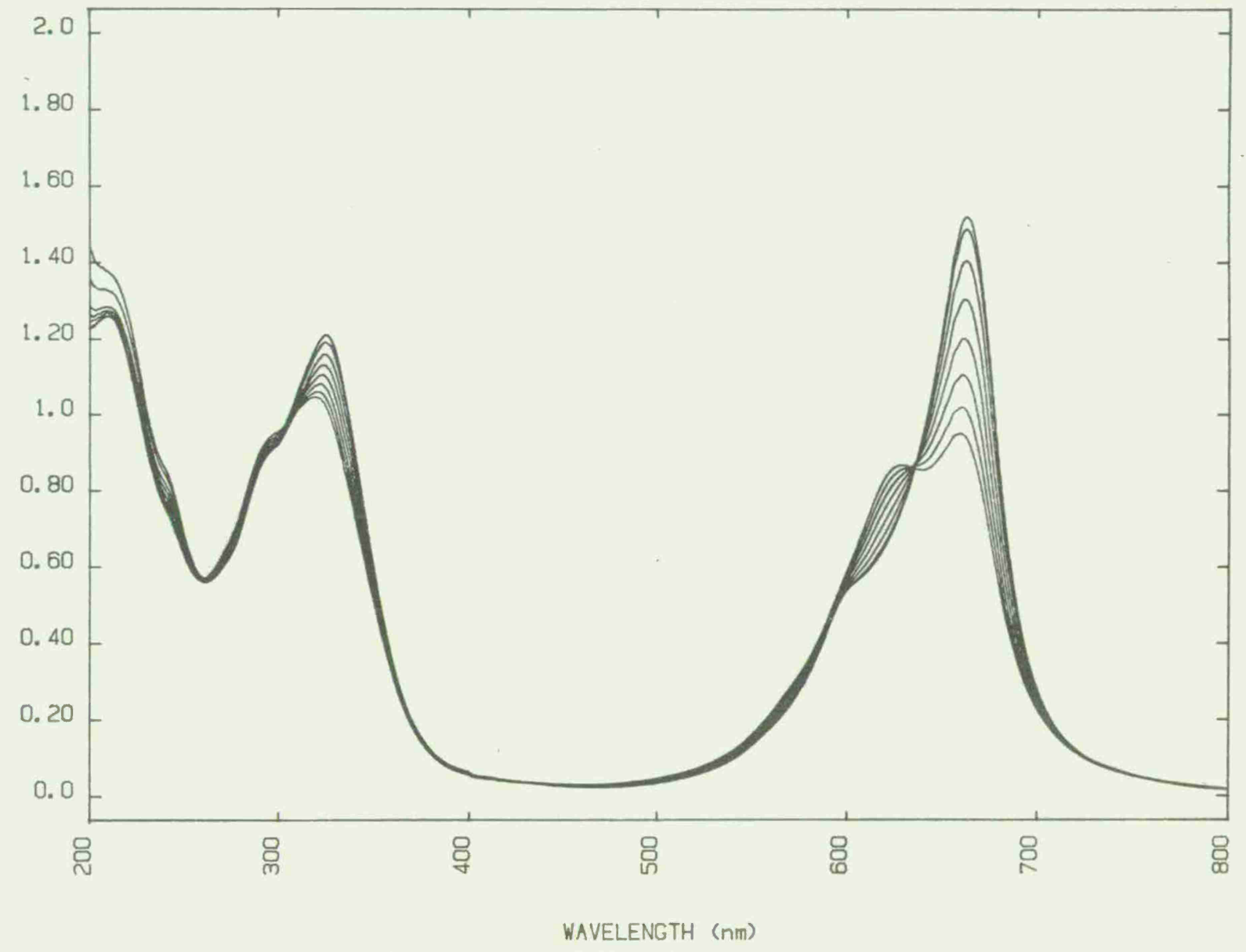


Figure 1. Absorption Spectrum of CoTSPC at Various Temperatures

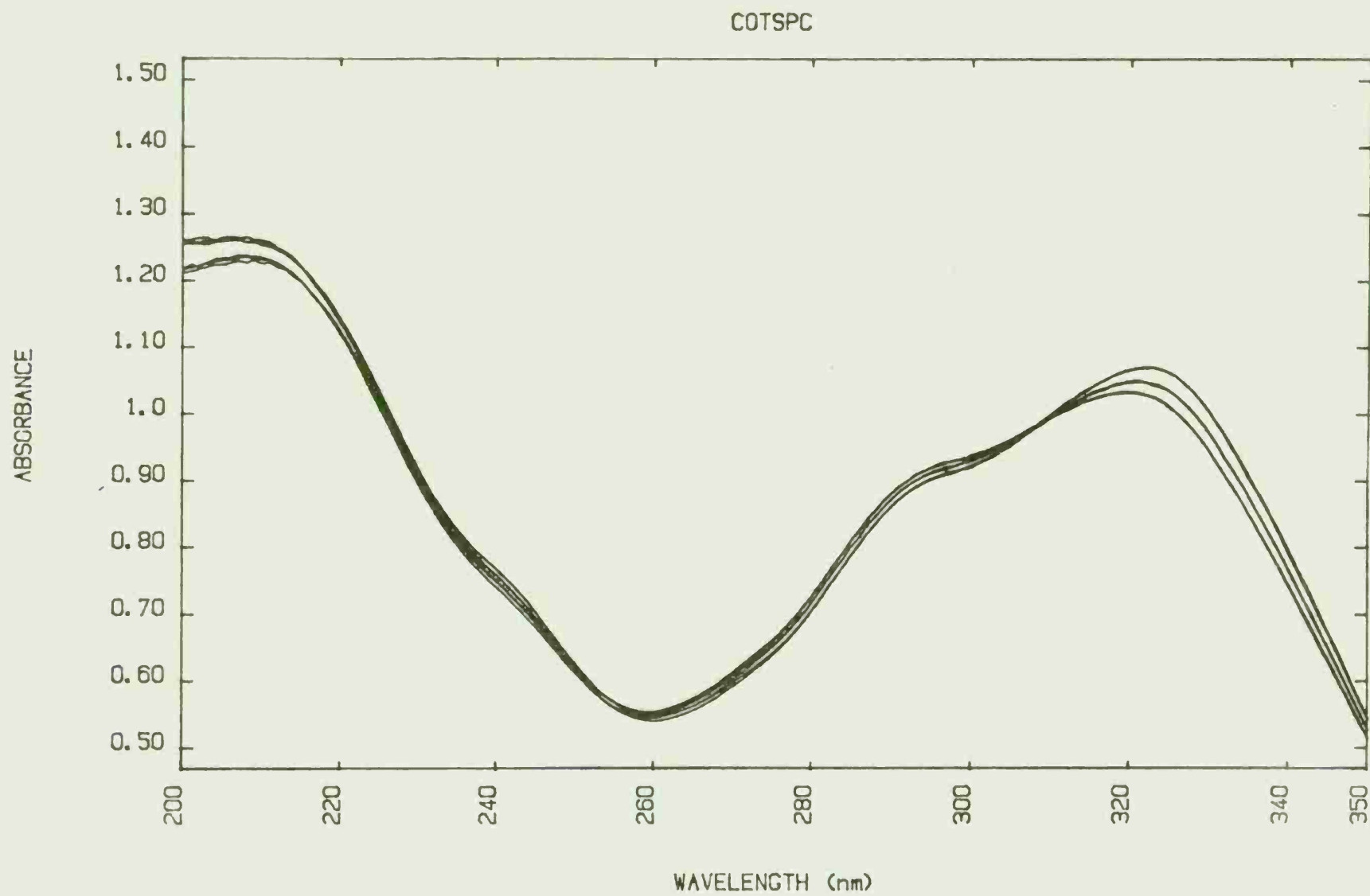


Figure 2. Comparison of UV Absorption Spectrum Run With the Deuterium Lamp On and With Both the Deuterium and Tungsten Lamps Active

Table 2. Effect of Tungsten Lamp on Spectrum of $2.05 \times 10^{-5}M$ CoTSPC Solution

Temp °C	λ nm	Deuterium Lamp		Deuterium and Tungsten Lamps		
		Absorbance	Absorbance, ^a Isosbestic	λ nm	Absorbance	Absorbance, ^a Isosbestic
20.0	320	1.033	0.980	319	1.035	0.983
30.0	321	1.049	0.980	321	1.052	0.983
40.0	323	1.071	0.979	322	1.071	0.983

^aIsosbestic point at 308 nm.

The next stage of our analysis attempts to estimate how much change in monomer and dimer concentration accompanies a change in absorbance of 0.001 or 0.002 absorbance unit.

The absorbance of a solution in a 1-cm cell containing only monomer and dimer is

$$A = \epsilon_M[M] + \epsilon_D[D] \quad (1)$$

where

A = absorbance

M = molar absorption coefficient of monomer, $M^{-1}cm^{-1}$,

D = molar absorption coefficient of dimer, $M^{-1}cm^{-1}$,

M = monomer concentration, M, and

D = dimer concentration, M.

The equilibrium between monomer and dimer is

$$K = [D]/[M]^2 \quad (2)$$

where K = dimerization constant, M^{-1} .

The total concentration of CoTSPC, [Co], is

$$[Co] = [M] + 2[D] \quad (3)$$

Equations (2) and (3) can be combined to give the monomer concentrations in terms of total CoTSPC as

$$[M] = \frac{-1 + \sqrt{1 + 8K [Co]}}{4K} \quad (4)$$

With equations (2) and (4), one can rewrite equation (1) in terms of the experimentally accessible [Co] as

$$A = \epsilon_M - \left(\frac{-1 + \sqrt{1 + 8K [Co]}}{4K} + \frac{\epsilon_D}{2} \right) \left[[Co] - \left(\frac{-1 + \sqrt{1 + 8K [Co]}}{4K} \right) \right] \quad (5)$$

A nonlinear, least-squares program¹¹ was used to find best-fit values of ϵ_M and ϵ_D at $\lambda = 320$ nm using absorbances measured at 320 nm at various temperatures. The independent variable in this calculation was the dimerization constant, K. Values of K at the measured temperatures were determined with values of ΔH° and ΔS° measured earlier.⁶ Table 3 summarized these calculations. Measurements were made at 49.9 and 68.8° C with the 2.05×10^{-5} M CoTSPC solution in order to have additional points for fitting equation (5). As table 3 shows, the best-fit values of ϵ_M and ϵ_D at 320 nm are $58.4 \pm 0.3 \times 10^3 \text{M}^{-1} \text{cm}^{-1}$ and $85.3 \pm 0.4 \times 10^3 \text{M}^{-1} \text{cm}^{-1}$, respectively, with error given as the standard deviation of the mean which is an output of the nonlinear, least-squares program. The right hand column in table 3 compares the absorbances calculated with the measured absorbances, which are in reasonable agreement.

Using the best-fit values of ϵ_M and ϵ_D and a CoTSPC concentration of 2.05×10^{-5} M, one can calculate how K, M, and D would change with a change in 0.002 absorbance unit from the measured value of 1.034 at 20° C. The results of these calculations are shown in table 4, where one can see that a change in absorbance of 0.002 absorbance unit produces less than 5 percent change in monomer concentration. Thus, one sees that if the visible light made a significant change in the dimerization of CoTSPC, one would have detected a measureable change in absorbance at 320 nm with a 2.05×10^{-5} M CoTSPC solution. At minimum, these results show that earlier determinations were unaffected by visible-light-enhanced disaggregation of CoTSPC.

4. CONCLUSION

There is no evidence that visible light from a spectrophotometer alters the dimerization of CoTSPC. This means that previous determinations of the dimerization constant were unaffected by light from the spectrophotometer, and it raises doubt as to whether visible light enhances disaggregation of CoTSPC as claimed by Dutch workers.

Table 3. Best-Fit Values of the Molar Absorbances of Monomeric and Dimeric CoTSPC at 320 nm

Temp °C	$K, M^{-1}, \times 10^{-5}{}^a$	A, exp't	A, cal'd ^b
20.0	3.123	1.034	1.033
30.0	1.579	1.051	1.051
49.9	0.461	1.088	1.092
68.8	0.163	1.133	1.131

^aCalculated with $\Delta H^\circ = -12.044$ cal/mole and $\Delta S = -15.943$ cal/mole $\cdot K$.

^bCalculated with best-fit values of $58.4 \pm 0.3 \times 10^3 M^{-1} \text{ cm}$ for ϵ_M and $95.3 \pm 0.4 \times 10^3 M^{-1} \text{ cm}^{-1}$ for ϵ_D .

Table 4. Computation of Monomer and Dimer Concentration at 20.0° C, Assuming Absorbance Ranges from 1.036 to 1.032

A, $\lambda = 320$ nm	$K, M^{-1}, \times 10^{-5}{}^a$	$C_M, M, \times 10^6$	$C_D, M, \times 10^6$
1.036	2.73	5.29	7.62
1.034 ^b	3.12	4.99	7.78
1.032	3.23	4.92	7.81

^aCalculated with $\epsilon_M = 5.84 \cdot 10^4 M^{-1} \text{ cm}^{-1}$, $\epsilon_D = 95.3 \cdot 10^4 M^{-1} \text{ cm}^{-1}$, and $[Co]_T = 2.05 \cdot 10^{-5} M$.

^bExperimental value at 20.0° C

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