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SPECTRAL PROPERTIES OF PASSIVELY Q-SPOILED LASERS

SemiAnnual Technical Summary Report

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ABSTRACT

A description of experiments which bear on the problem of the spectral behavior of passively Q-switched lasers and of the physico-chemico behavior of Q-spoiling dyes is given. The results of experiments of spectral bleaching and of thermal and photochemical responses of dyes are described. In particular, a number of the characteristics of two bleachable dyes applicable to the neodymium laser system (1.06μ) are discussed.

FOREWORD

This report was prepared jointly by the Optical Physics and Chemical Physics Divisions of Korad Corporation, Santa Monica, California, under Contract Nonr-5100(00) entitled "Spectral Properties of Passively Q-Spoiled Lasers". The study was conducted under the project leadership of B. H. Soffer, with R. C. Pastor, H. Kimura, and B. B. McFarland participating.

SPECTRAL PROPERTIES OF PASSIVELY Q-SPOILED LASERS

1. INTRODUCTION

The objective of this program is an experimental research study of the spectral properties of passively Q-spoiled lasers. The study is directed toward an understanding of the spectral diffusion or relaxation in laser materials and the physical behavior of passive Q-spoiling materials. In this study, the properties of interest in these Q-spoiling materials are primarily those which may influence the spectral behavior of lasers.

We reported in the previous Semi-Annual Technical Summary Report¹ on experimental evidence in support of the existence of significant spectral hole-burning in certain dyes used as passive bleachable Q-spoiling elements. The experiments were performed utilizing the narrow spectral output of one passively Q-spoiled laser to bleach the passive cell of another laser. The frequency locking of the two lasers that ensued served to demonstrate the spectral hole-burning phenomenon. One of the problems investigated during the current report period² was a study of the spectral cross-relaxation behavior of bleachable dyes. The results directed our attention to certain pertinent photo and thermo-chemical responses of the dyes. The preliminary results of these studies are reported in the following section.

2. TECHNICAL DISCUSSION

A. Spectral Cross-Relaxation

An experiment was performed to study the spectral cross-relaxation behavior of a polymethine dye used as a saturable absorber for Q-spoiling. The problem considered was the comparative efficacy of a single spectral laser line and several spectrally-separated

¹ Korad Corporation Semi-Annual Technical Summary Report on Contract Nonr-5150(00) dated 28 April 1966. See also B. H. Soffer and B. B. McFarland, Appl. Phys. Ltrs., 8, 166 (1966).

² Korad Corporation Third Quarterly Report on Contract Nonr-5150(00) dated 6 July 1966.

lines of the same total power in bleaching the dye. If the dye solution has, for example, a very long spectral relaxation time, referred to a particular spectral separation (greater than the separation of the inhomogeneous components), then the several separated weak lines would not aid each other in the bleaching process and would not have the over-all bleaching effect of a single more powerful line operating in the more saturated region of the dye transmission characteristic. Thus, by varying the number of lines and their spectral separation, one might map the details of the spectral relaxation properties of the dye.

The dye studied was the Kodak Q-spoiling dye #9740 (EKD), which is undoubtedly* a polymethine dye in the carbocyanine dye family, dissolved in chlorobenzene. An Nd^{+3} glass laser was employed in order to obtain large variations in both the number and separation of spectral components, as desired by the use of resonant reflectors. A schematic representation of the experimental apparatus used is given in Figure 1. The Nd^{+3} glass laser, Q-spoiled with EKD, was used as a source to bleach another cell of EKD. Two photodiodes sampled the signals both before and after the test cell to measure a bleaching or saturation characteristic curve (i.e., peak power transmission vs peak power input). The signals from both diodes were displayed on a traveling wave oscilloscope trace, one signal being delayed some 50 nsec by a length of cable. Spurious reflections engendered by a non-matching T-connector were eliminated by terminating the cables with matching impedances at the photodiodes. The system was also operated with a cell of solvent alone, replacing the dye cell, as an aid to calibration and a check on solvent nonlinear behavior. The beam was finally directed to a 3.4 meter spectrograph where the laser spectrum was analyzed. The spectral plates were not photometrized -- all photometric data being recorded by the photodiodes.

A typical bleaching curve is shown in Figure 2. Single spectral component data points are distinguished from the multi-

* At this writing, Eastman Kodak will not reveal to us the chemical nature of the dye. (Private communication from K. S. French.)

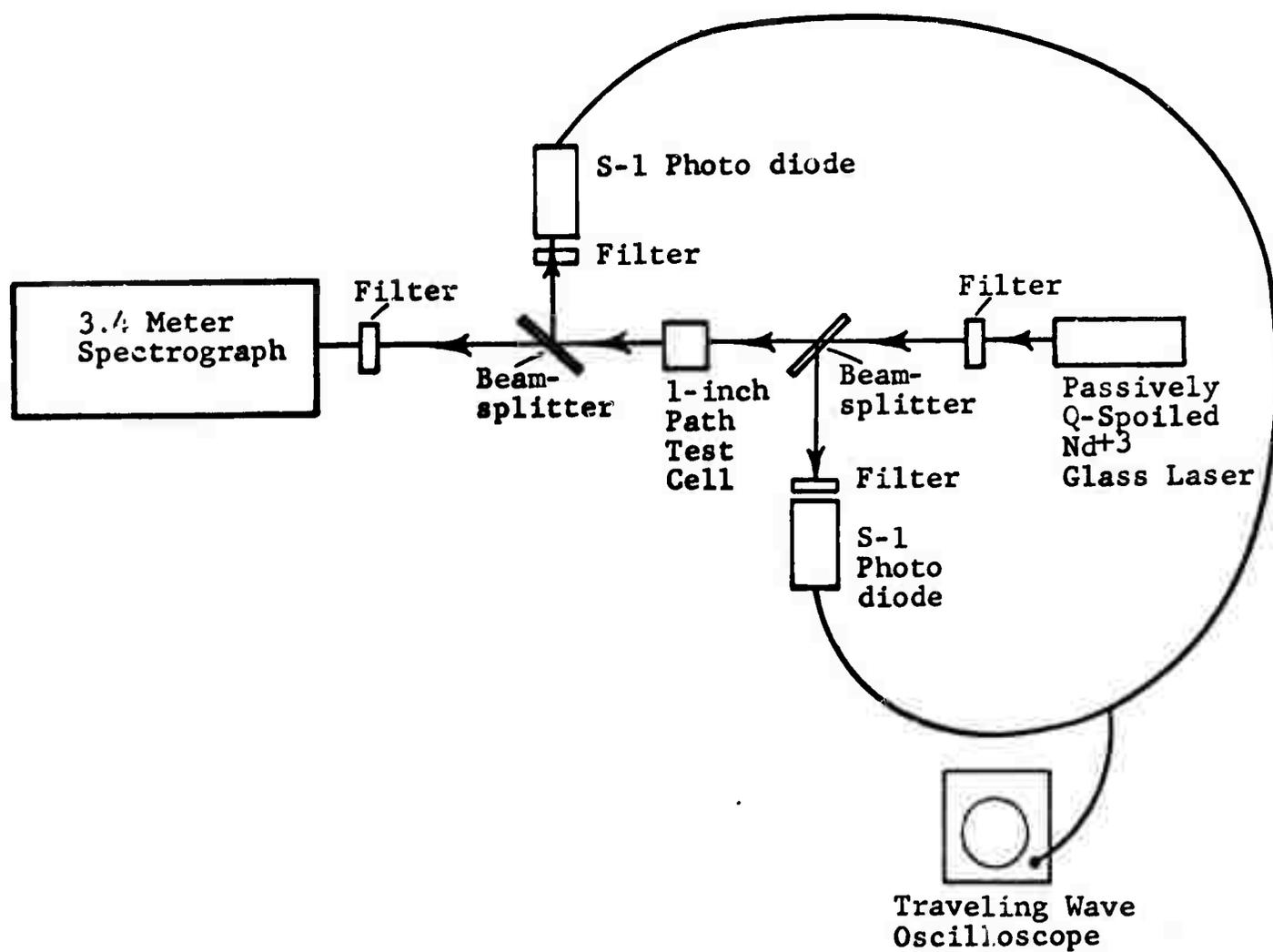


Figure 1

Schematic of Experimental Arrangement

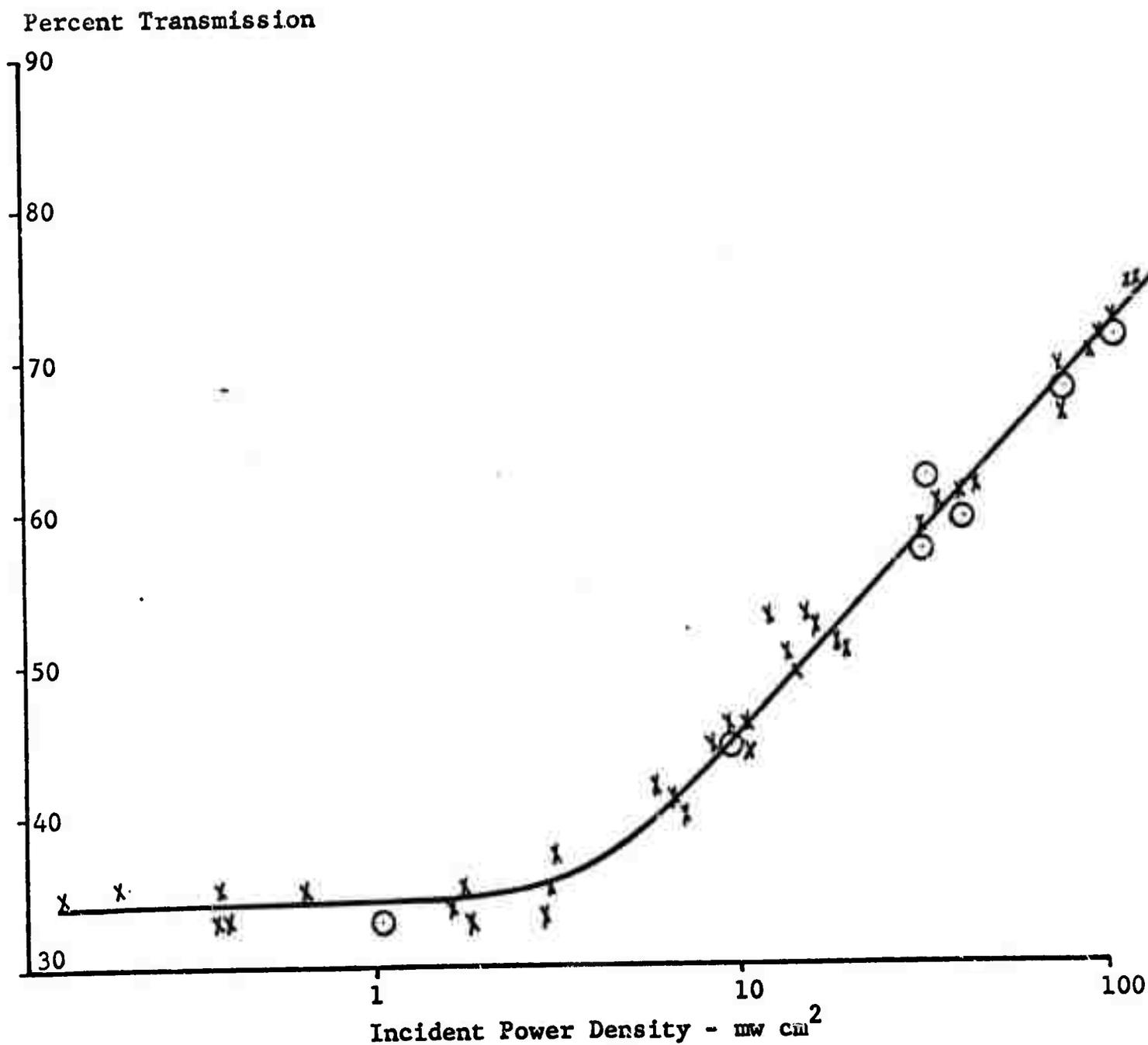


Figure 2

Saturation Behavior of Kodak Q-spoiling Dye #9740 (EKD)

- X Single Spectral Component Pulse
 O Multi Spectral Component Pulse

spectral component data points. In this set of data, the spectral separation of the components of the multi-lined channeled spectra was one wavenumber. No significant variation, outside of the experimental errors, can be noted even when as many as six spectral components were employed in a pulse. An analysis of the data shows that if any spectral "hole-burning" occurred within the observable time resolution defined by the giant pulse experiment, then it was at least five times smaller an effect than the maximum effect expected for the case of very long spectral relaxation times -- the bound being set by the average deviation of the errors in the measurements. Therefore, either the individual homogeneously broadened components of the dye absorption spectrum are wide, compared to about 6 cm^{-1} , or cross-relaxation times within this spectral range are too rapid to have consequences observable by this technique.

An incidental systematic effect observed in collecting this data is noteworthy. At any arbitrary positioning of the beam splitters (wedged to obviate interference effects) and the photodiodes, generally greater disparate skewed and structured laser power pulse shapes would be recorded, even with no dye or control cell interposed in the beam. This was carefully demonstrated not to be an instrumental problem by permuting the delay lines, the photodiodes, and other portions of the apparatus. Only by the judicious adjustment of the angular and spatial sample of the laser beam directed into each diode could the pulses be made congruent, the pulse shape depending on the particular choice made. We conclude from this, therefore, that each giant laser pulse is spatially and angularly quite variable in its temporal character and only the average over space and angle has the smooth "bell" shape character usually reported.

In the course of the spectral hole-burning studies described above, it was observed that the Nd^{+3} glass laser passively Q-spoiled with bleachable dyes exhibited a certain erratic spectral behavior from pulse to pulse. The laser would usually emit a single narrow line, and could easily be constrained to do so with

resonant reflectors. But, even so constrained, the wavelength of the emission would vary from pulse to pulse. Occasionally, a burst of several lines would appear -- channeled at the inter-order spacing of the resonant reflector. Furthermore, this effect was observed to some degree with both the Korad³ and the Kodak Q-spoiling dyes.

Preliminary theoretical and experimental studies of models which have a bearing on this problem have been performed during this report period.

Chemical Kinetics Pertinent to the B. Performance of Dyes Used for Passive Q-spoiling

The following discussion has, for its object, the correlation of the kinetics of various processes which are relevant to certain spectral properties of dye employed for passive Q-spoiling. In this exposition we set up a model which suggests that a local heating effect may be responsible for the seemingly erratic spectral behavior described previously in passively Q-spoiled Nd⁺³ glass lasers. The roles of geometric cis-trans isomerism and the more important role of the triplet state are discussed.

(1) Photochemical Response

The scheme which describes the spectral behavior of dyes in liquid solutions at ordinary temperatures is shown by the Jablonski diagram in Figure 3. The pertinent transitions between the various levels are given.

The biradical nature associated with the triplet makes this state chemically reactive. As a rule, the singlet state is less reactive, and the excited singlet provides a lower barrier for cis-trans isomerization. If we represent the geometrically-favored molecular species A, the spectrally-distinct isomer as A', and use subscripts to identify the

³ B. H. Soffer and R. H. Hoskins, *Nature*, 204, 276 (1964). See also "Eastman Q-Switch Solution 9740", a circular released by Distillation Products Industries (October 1965).

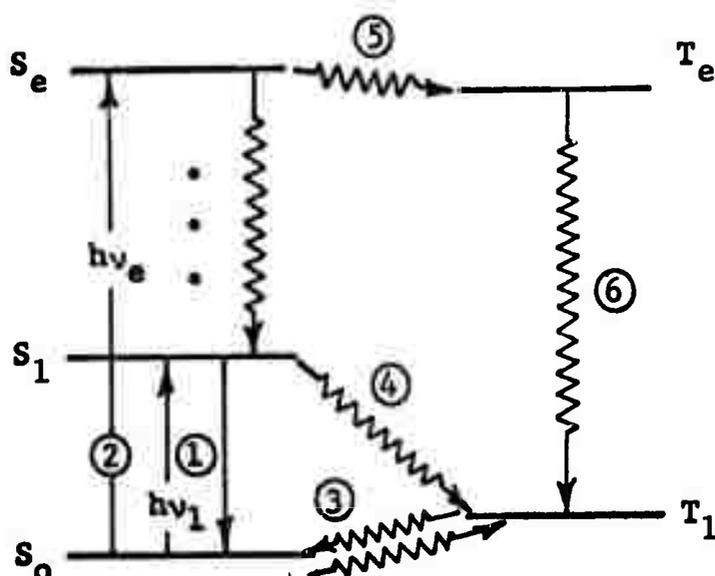


Figure 3

Jablonski Diagram

The levels on the left (labeled S) are singlet states. Those on the right (labeled T) are triplet states. The three dots stand for an unspecified number of levels intervening. The solid-line arrows are radiative transitions and the wavy-line arrows are non-radiative. The vibrational structure of each electronic state is not shown in order to maintain simplicity.

electronic states, we can summarize the preceding information in the kinetic scheme shown in Figure 4.

The species A_0 , which is important to Q-spoiling (i.e., path (1)), occupies a central position. The concentration of A_0 may change reversibly by means of a physical process -- say, an unfavorable kinetics leading to trapping in a metastable form, geometrically and electronically (and therefore spectrally) different. On the other hand, the change may be irreversible through a chemical process.

The isomerization path (8) shows two alternatives. There is a similar Jablonski diagram (see Figure 3) for A' . Because of steric hindrance, the cis forms have their main electronic absorption occurring at shorter wavelengths than the stretched-out trans species, i.e., $\nu_1' > \nu_1$. However, the position of S_0' with respect to S_0 in the energy (vertical) scale depends on ΔH (enthalpy change)*. Hence, depending on $\nu_1' - \nu_1$ and ΔH , isomerization may proceed by $S_1 \rightsquigarrow S_0'$, or $S_e \rightsquigarrow S_1'$. The former alternative is not seen in the polymethine Korad dye solution irradiated by $h\nu_1$. Here, we have somewhat modified and generalized the diagram presented in the Fourth Quarterly Report, favoring the role of the triplet state.

Our preliminary observations favor the path $S_e \rightsquigarrow S_1'$ as a more likely one for isomerization. When the same solution is irradiated at room temperature with the same photon flux, but with photon energy $h\nu_e$, the concentration of A_0 diminishes. As the peak absorption at ν_1' diminishes, the ratio of the peak absorption at ν_1' to ν_1 increases. (No shift in the absorption is noted and it may be assumed that the ratio of linewidths remains constant.) When the radiation is turned off, it takes hours for recovery of the absorption at ν_1 and the ratio of

* The enthalpy change determines the Boltzman distribution between A_0 and A' as a function of T . If A and A' can be prepared pure, ΔH is also obtained from the difference of their respective heat of combustion.

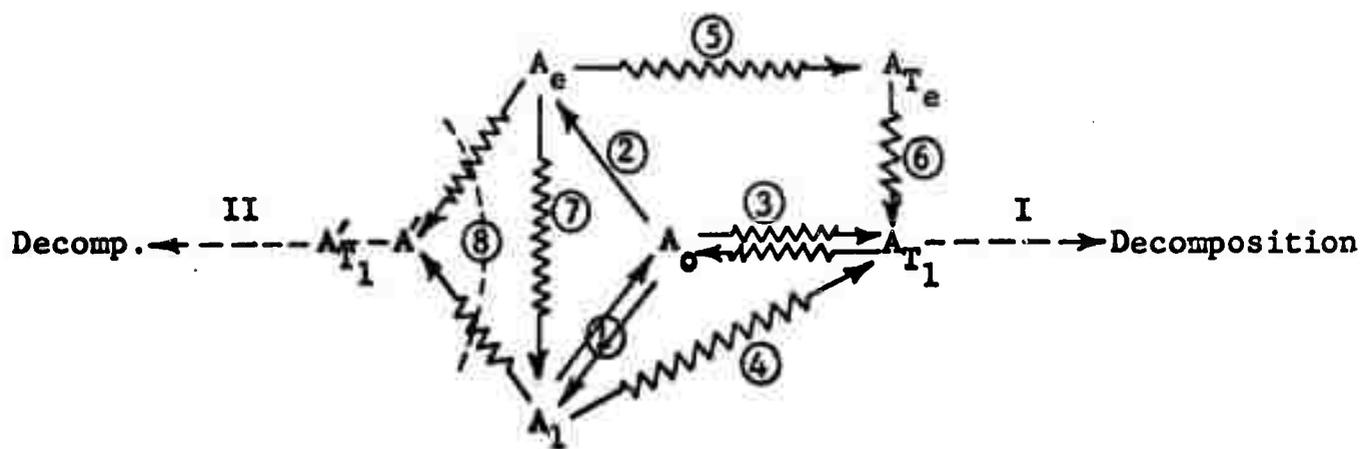


Figure 4

Schematic Diagram of Kinetic Processes

peak absorption at ν_1' to ν_1 returns to its original value. The long recovery time involved rules out all other excited electronic states of species A.

The preceding results were obtained by irradiating Korad dye solution at $\lambda_e = 0.365 \mu$. Details of transitions to the lower excited electronic states of A, which are higher than S_1 , followed by a crossover to A' and subsequent electronic and vibrational transitions, have not been studied. These uncertainties are embodied in the scheme $S_e \rightsquigarrow S_1'$. However, from the known values of ΔH in cis trans transitions, $\sim 5 \text{ kcal mol}^{-1}$, we can say that $\Delta H < N_0 h\nu_1$, where the Einstein unit, $N_0 h\nu_1$, is equivalent to 28 kcal mol^{-1} . Since $N_0 h\nu_1$ is 32 kcal mol^{-1} , one can provide a justification for the failure to observe a crossover at $S_1 \rightsquigarrow S_0'$. In addition, these values favor a return crossover, i.e., recovery of A from A' by means of $S_0' \rightsquigarrow S_0$.*

- In line with the scheme $S_e \rightsquigarrow S_1'$ is the observation of an irreversible contribution to the decay of A_0 if irradiation at $h\nu_e$ is carried out for longer periods. Although the branching ratio at S_1' may not be very favorable to A_{T_1}' , longer irradiation times favor the population of the latter level. And the same reason given previously for the chemical reactivity of A_{T_1} holds for A_{T_1}' (see decomposition paths I and II in Figure 4).¹ It can therefore be appreciated that the kinetics of paths (3) and I, which is relevant to both A and A' , deserve a separate study.

(2) Thermal Response

Figures 5A and 5B show the fundamental absorption at $\sim 1 \mu$ of the two dyes which are in use for Q-spoiling at 1.06μ . Both are polymethine dyes. The less-pronounced geometric isomerism in the case of the Korad dye is attributed to an

* A triplet-triplet crossover, i.e., $A_{T_1}' \rightsquigarrow A_{T_1}$, cannot be ruled out as another possibility.

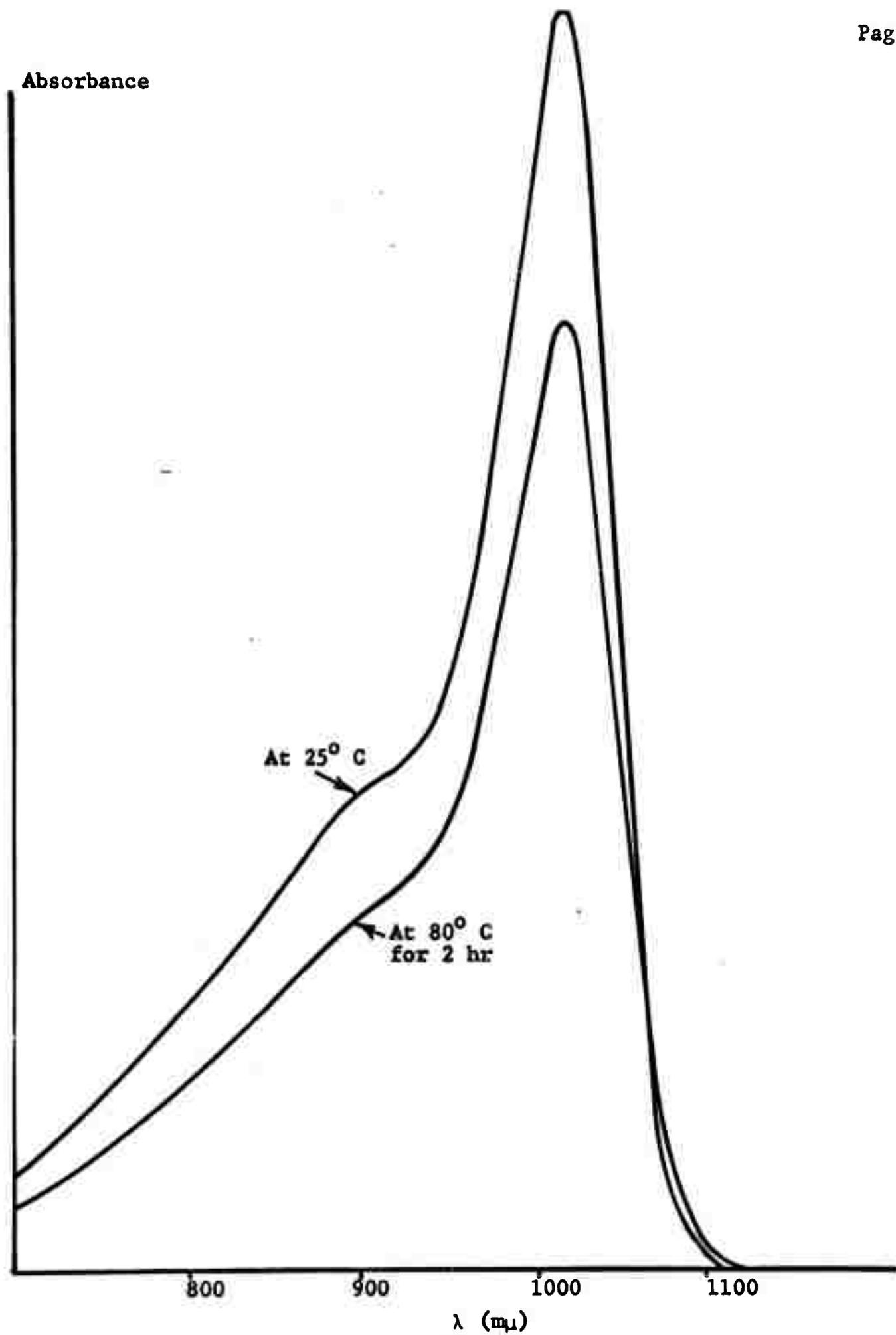


Figure 5A
Absorption Spectrum of Korad Dye

Absorbance

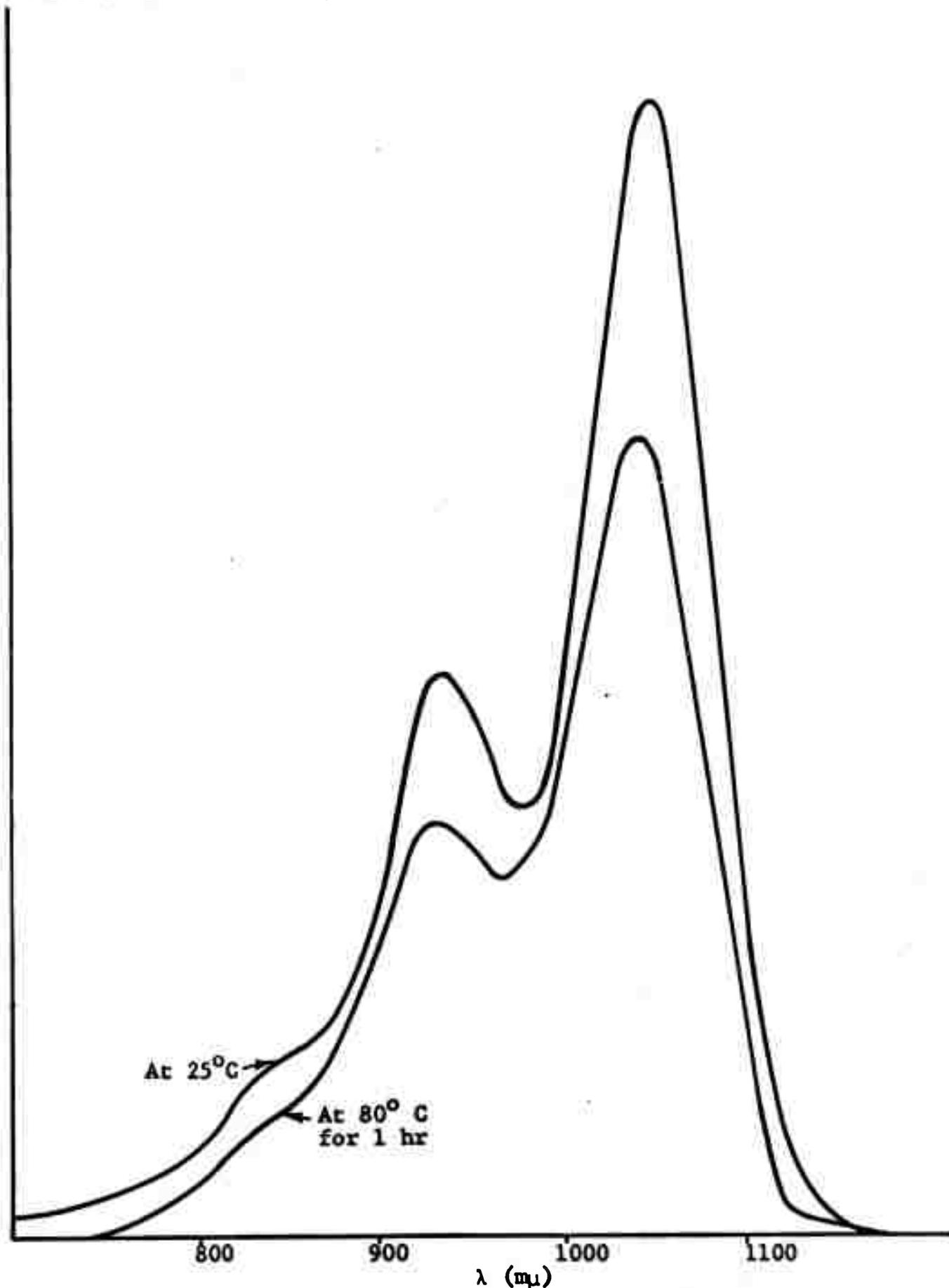


Figure 5B

Absorption Spectrum of Kodak Dye

increased rigidity in the polymethine chain through the intervention of an isophorone residue.⁴ See Figure 6.

As shown in Figure 5, there is a decay in the concentration of A_0 which is effected thermally. Cycling at temperatures not too far above room temperature for relatively short periods results essentially in a complete recovery. The only radiation involved in the present study is a weak probing at ν_1 to monitor the concentration of A_0 vs time. Hence, we are confining ourselves to the portion of the Jablonski diagram shown in Figure 7.

Further examination of the optical absorption spectrum in the temperature range investigated showed no isobestic point. Furthermore, the entire absorption in the fundamental region decays with no region of buildup. This allows us to eliminate from consideration the complication of cis trans isomerism (see path (8) of Figure 7).^{*} Consequently, the model for the kinetics of decay of A_0 is shown in Figure 8. Since the study involves no irradiation, paths (1) and (4) are not involved. They have been included only to call attention to the fact that in Q-switching (i.e., path (1)), the thermal behavior is complicated (local heating) by path (4). The branching ratio, though not very favorable to path (4), may not be insignificant when weighted by the energy difference. Hence, if the energy separation of T_1 from S_0 is small, the population of level T_1 from S_1 is augmented thermally from level S_0 . If T_1 is indeed a chemically reactive state, the mechanisms cited pose a threat to the conservation of species A.

⁴ "Recent Progress in the Chemistry of Natural and Synthetic Colour-Matters and Related Fields", edited by T. S. Gore, B. S. Joshi, S. V. Sunthakar, and B. D. Tilak (Academic Press, 1962). Also see "The Search for Longer Conjugated Chains in Cyanine Dyes", by L. G. S. Brooker, Page 573.

^{*} The result indicates that the activation energy of path (8) is greater than that of path (3).

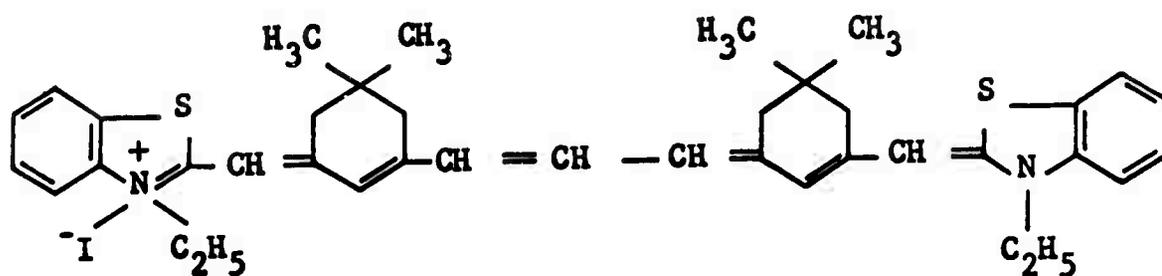


Figure 6

Korad Dye 3, 3' - diethyl - 9, 11; 15, 17 -
dineopentylene-thia-pentacarbocyanine iodide

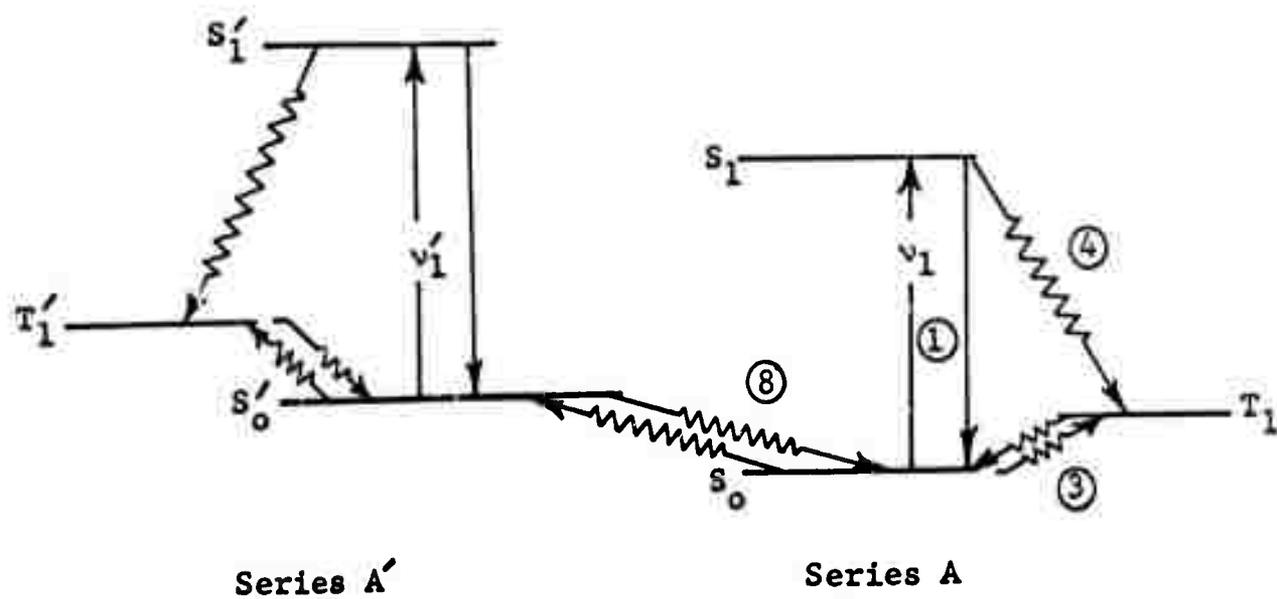


Figure 7

Energy Levels and Nonradiative Transitions
Involved in a Low Thermal Excitation

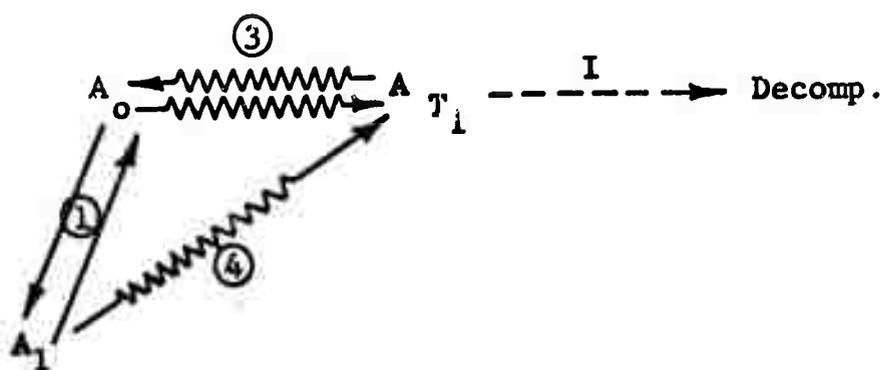


Figure 8
Kinetic Scheme for Thermal Response

Figures 9 and 10 show the normalized concentration of species A_0 vs time for Korad and Kodak dye respectively. The concentration of A_0 is assayed by means of the strength of the absorption at ν_1 and a value of unity is assigned to the concentration at time zero. Note that at the lower temperatures only the reversible path (3) is in evidence. At higher temperatures, the contribution of the irreversible path I becomes dominant.

The specific reaction rates for paths (3) and I were derived from these curves, assuming first-order processes.* Figure 11 shows the good curve fit for the value of the equilibrium constant K for path (3), and the specific reaction rates at the various temperatures. The specific reaction rate for the forward direction of path (3) is k_f and, for path I, is k_i .

Since the slope in Figure 11 of $\ln K$ vs $1/T$ equals that of $\ln k_f$ vs $1/T$ (Korad dye), it follows that no activation energy is required to relax from level T_1 to S_0 . The activation energy for the forward path of (3) is 7 kcal mol^{-1} , which is probably the equivalent of one or two vibrational levels of S_0 . It is of interest that the same activation energy (see Figure 11) is obtained for the forward path (3) for Kodak dye (EQS 9740). This is probably not unexpected, since the same chain length in the polymethine conjugation appears to be involved in the two cases.

In comparing Figures 9 and 10, it is seen that the Kodak dye is more unstable thermally. As pointed out earlier, thermal stability is linked to the performance stability (local heating) under Q-switching. This difference in stability is indeed seen in comparative runs. At a power density of $\sim 3 \times 10^6 \text{ w cm}^{-2}$ to each laser pulse (1.06μ), at a rep rate of 10 pps, EQS 9740 is irreversibly deteriorated

* This assumption is justified for path (3). For path I, which may involve a complicated sequence, all that one requires is that the rate-determining step is first-order in A_{T_1} .

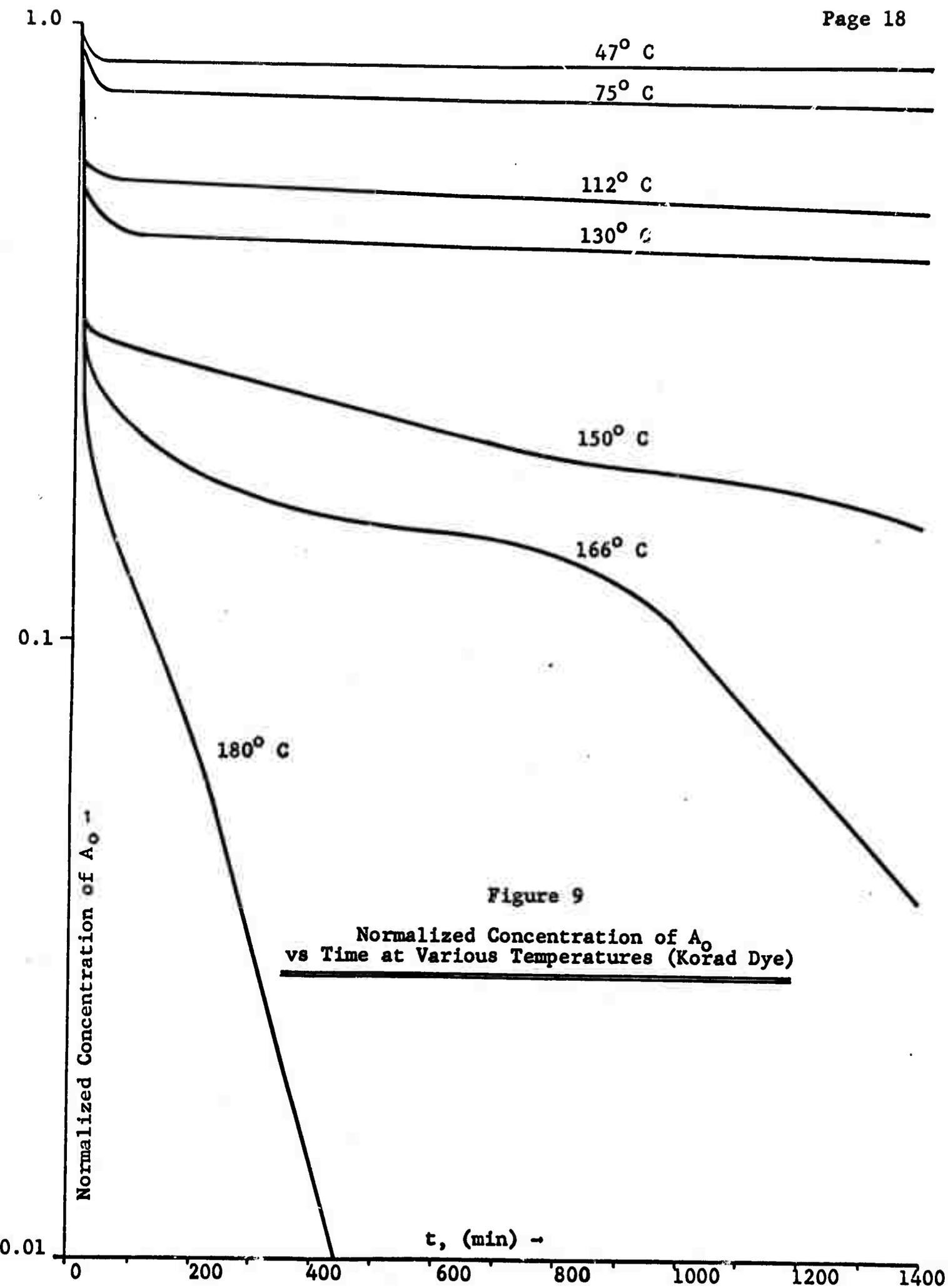


Figure 9

Normalized Concentration of A_0
vs Time at Various Temperatures (Korad Dye)

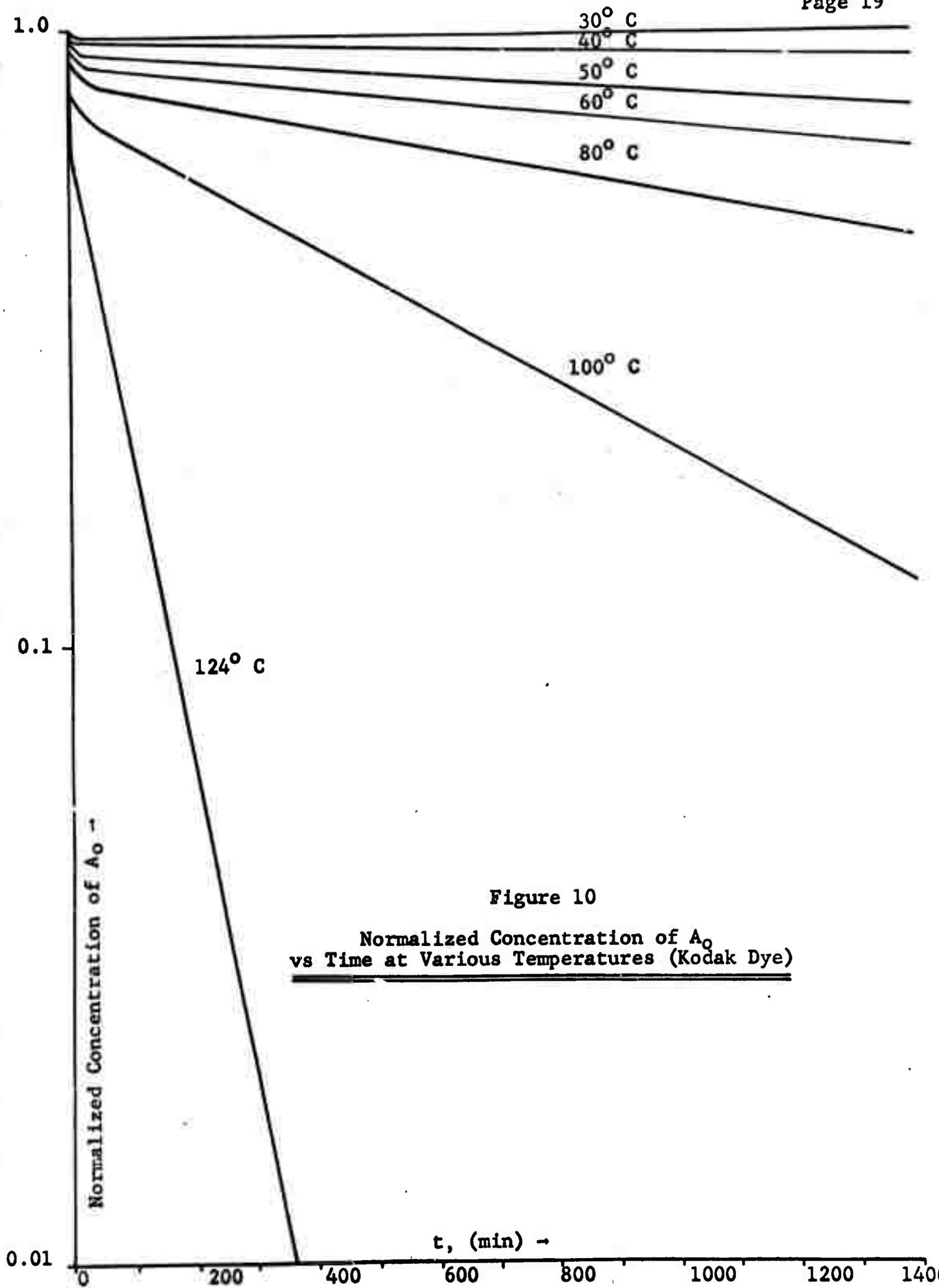


Figure 10

Normalized Concentration of A_0
vs Time at Various Temperatures (Kodak Dye)

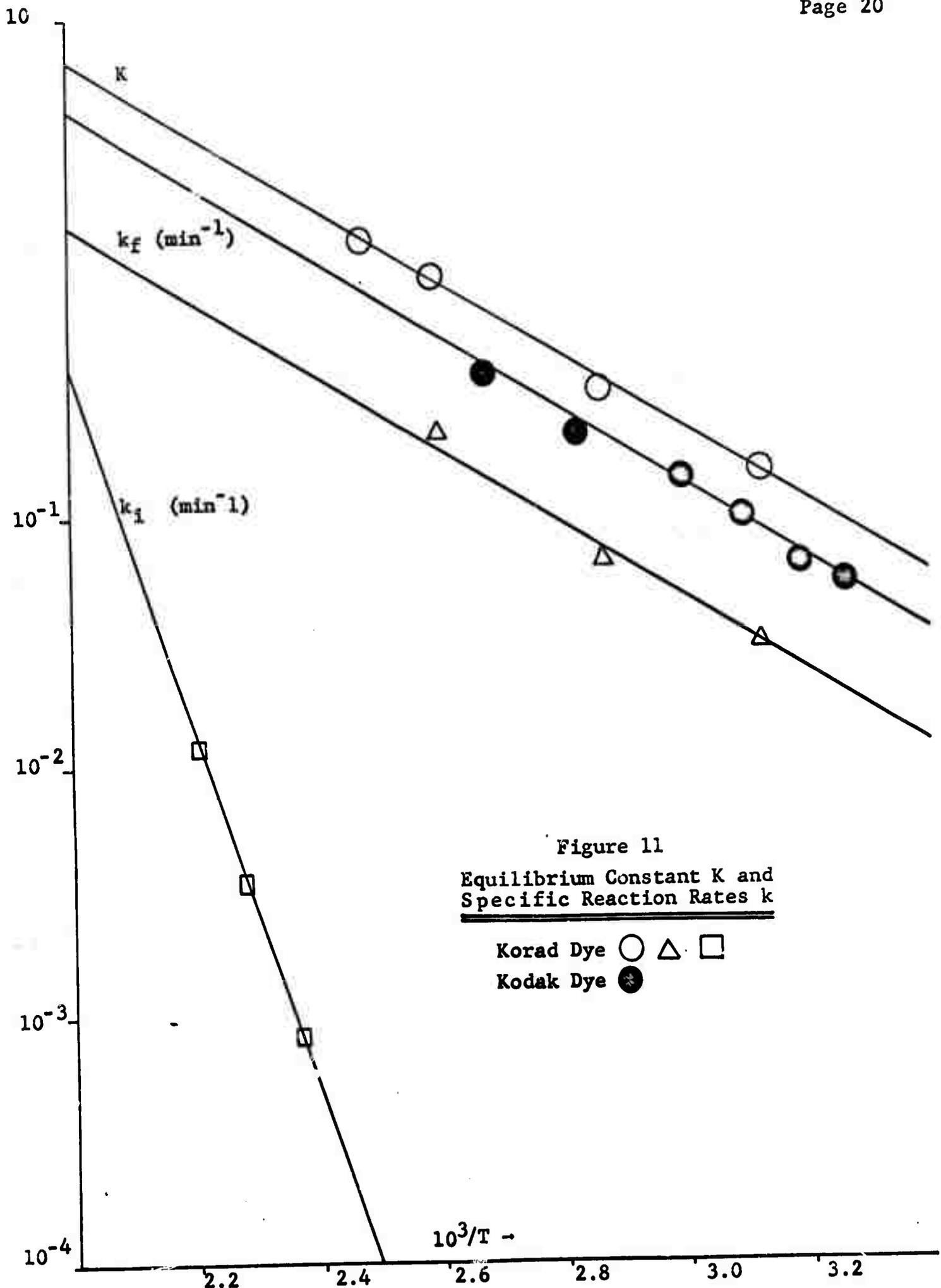


Figure 11
Equilibrium Constant K and
Specific Reaction Rates k

Korad Dye \circ \triangle \square
 Kodak Dye \bullet

at $< 1 \times 10^3$ pulses. Under the same conditions, the Korad dye solution begins to show only a slight damage at $\sim 3 \times 10^4$ pulses.

An additional test on the values of specific reaction rates is afforded by recovery experiments. EQS 9740, the dye solution with the more sensitive (irreversible) thermal response, was employed. The recovery, through reversible path (3), was obtained by measuring the A_0 regenerated at room temperature after a 20-hr exposure at various temperatures. The comparison of experiment and the prediction curve based on the rates obtained from curve fitting (see Figure 10) is shown in Figure 12.

It follows from the model employed (see Figure 5) that τ (the half-life of species A_0 at a given temperature) is given by

$$\tau = \left(1 + \frac{1}{K}\right) \frac{1}{k_i} .$$

From the constants obtained, taking a fiducial temperature of 125°C , it follows that

$$\tau \text{ (Korad)} \geq 2 \times 10^4 \text{ min} ,$$

and

$$\tau \text{ (EQS 9740)} \leq 1 \times 10^2 \text{ min} .$$

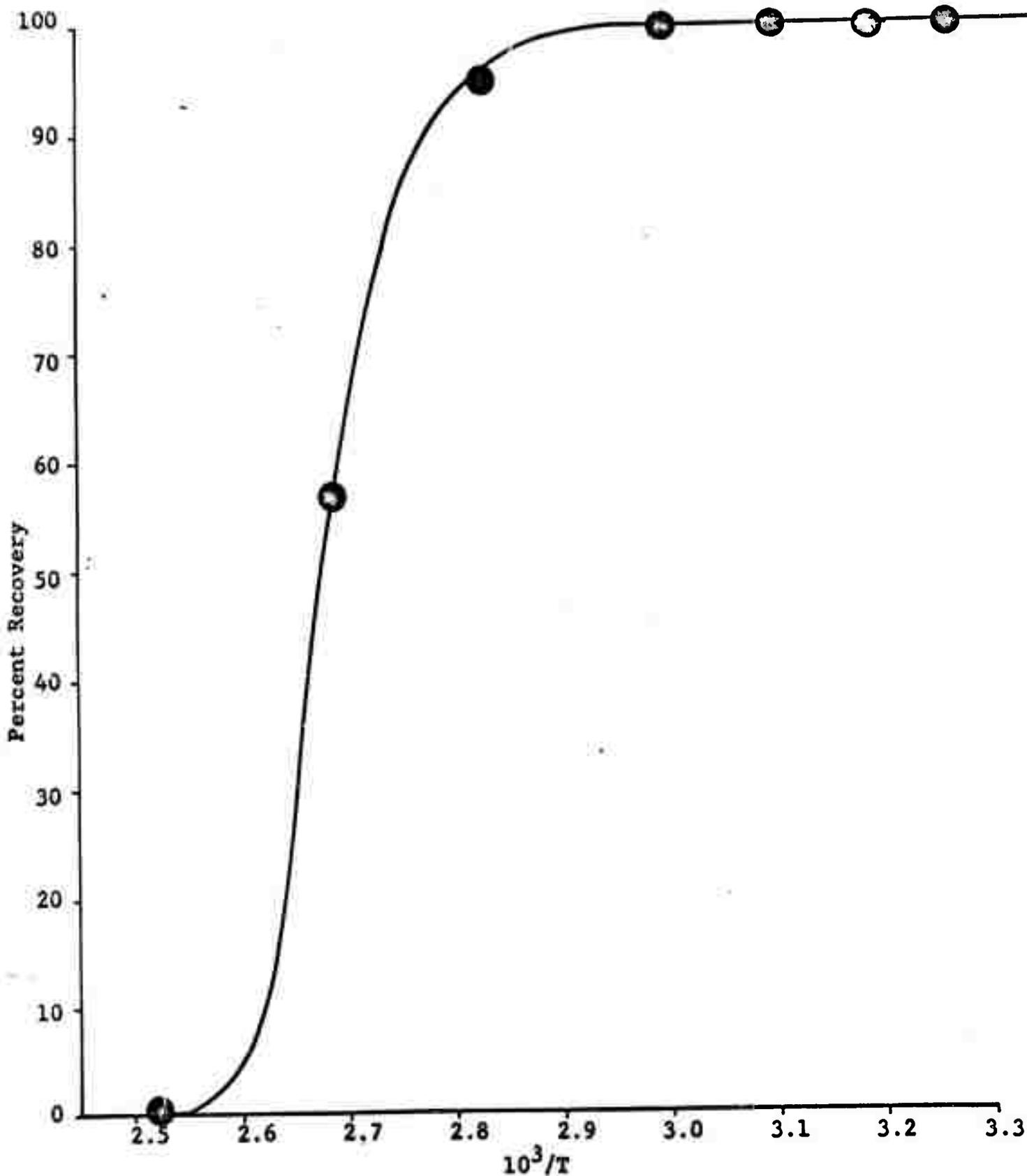


Figure 12

Recovery of Kodak EQS 9740 (chlorobenzene solution) at Room Temperature After 1200 min Exposure at Various Temperatures

(Curve is calculated from K and k_1 . Circles stand for observations.)

3. CONCLUSIONS

Local thermal redistribution of the spectral optical cross-section of the dye solution is believed to be the cause of the erratic laser behavior noted in Q-spoiled Nd⁺³ glass lasers. A preliminary study of kinetic models and mechanisms has revealed the roles of cis trans isomer partitioning and especially the role of the triplet state to these processes.

4. PLANS FOR NEXT PERIOD

Further work will be performed during the next report period to elucidate the kinetics and mechanisms of the thermal and photo-processes described in this report.

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There is no limitation on the length of the abstract. However, the suggested length is from 150 to 225 words.

14. **KEY WORDS:** Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, roles, and weights is optional.