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PYROLYSIS OF 1-IODOPROPANE BY THE VARIABLE ENCOUNTER METHOD.(U)
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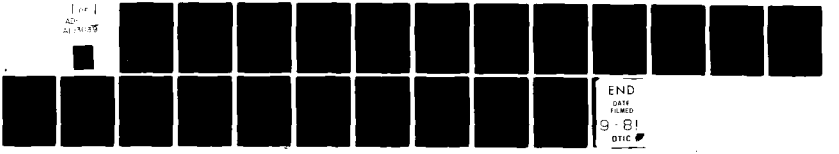

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20 ABSTRACT (Continue on reverse side if necessary and identify by block number)

The pyrolysis of 1-iodopropane has been studied by the Variable Encounter Method (VEM) at temperatures from 625 K to 840 K. Deactivating wall collisions are found to be stronger for this molecule than for the non-polar hydrocarbons previously studied in the same temperature range. Energy transfer collision efficiency declines with rise of wall temperature. The results of this study are compared with earlier steady-state work.

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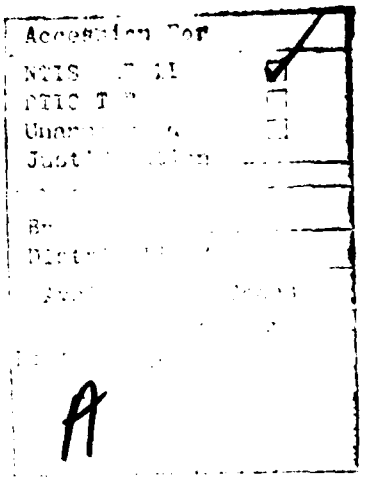
Abstract

The pyrolysis of 1-iodopropane has been studied by the Variable Encounter Method (VEM) at temperatures from 625 K to 840 K. Deactivating wall collisions are found to be stronger for this molecule than for the hydrocarbons previously studied in this temperature range. The results of this study are compared with earlier steady-state work.



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Introduction

The pyrolysis of 1-iodopropane has recently been the subject of a number of VLPP studies.[1-3] The mechanism of the decomposition has been discussed in detail by these earlier workers. The molecule decomposes by two channels whose critical reaction thresholds, E_o , differ by 4.3 kcal mole^{-1}:

$$C_3H_7I \xrightarrow{k_1} C_3H_6 + HI \qquad (E_o = 48.5 \text{ kcal mole}^{-1}) \qquad (1)$$

$$C_3H_7I \xrightarrow{k_2} \cdot C_3H_7 + I \longrightarrow C_2H_4 + \cdot CH_3 + I \qquad (E_o = 52.8 \text{ kcal mole}^{-1}) \qquad (2)$$

In reaction (2), the first step is rate determining.

This chemical system is an attractive one for study by the Variable Encounter Method (VEM) because it affords an opportunity for comparison of our results in the transient region with those obtained in steady state VLPP studies. Moreover, this molecule is the first polar one to be studied by the VEM technique. Information is desirable concerning the dependence of the energy transfer process between molecules and the wall upon this parameter.

In this paper we report the study of the pyrolysis of 1-iodopropane in a VEM system at temperatures from 625 K to 840 K. Unfortunately, the system seems to be not as clean as we had hoped originally.

Experimental

The experimental apparatus was similar to that used in previous VEM studies.[4,5] Reactor fingers with mean numbers of collisions, m , of 5.9 and 17.5 were used; these were described in ref. 5. Reproducible results (within the precision of the experiments) were obtained after "seasoning" the reactors for several hours. The resulting seasoned surface deteriorated with time when standing under vacuum (unlike our experience with hydrocarbon substrates[4,5]), or when the system was pumped for long periods, suggesting that the seasoning film itself was either being slowly pyrolyzed or had an appreciable vapor pressure.

Matheson-Coleman 1-iodopropane containing no detectible impurities was thoroughly degassed prior to use. Experiments varied in duration from one minute to several hours, depending on experimental conditions. The reaction was allowed to proceed usually from 1 to 30 per cent completion, and an aliquot was then taken. The products were analyzed by gas chromatography with fid detection. Separation of 1-iodopropane from its hydrocarbon reaction products was made on a 12 inch x 3/16 inch packed column consisting of 15% squalane on 45-60 mesh Chromosorb P. The reaction products were separated on a 6.5 foot x 3/16 inch column having the same packing.

In order to check for possible secondary reactions involving the radicals produced by reaction (2), a series of experiments was performed with trans-2-betene added as a getter. The ratio of iodopropane to butene was varied between 0.1 to 0.2.

Results and Calculations

Experimentally determined values of the mean probability of reaction per collision, $\bar{P}_c(m)$, by channel (1) are plotted versus temperature in Fig. 1 for the two reactors. These results are similar in behavior to those obtained in previous VEM studies. However, the rate data display somewhat more scatter (Fig. 2) than those obtained in previous studies and showed greater sensitivity to the seasoning procedure and history. Systematic fluctuations of $\sim$ 50% occur. Moreover, small amounts of C_2 impurities, presumably arising from the decomposition of the seasoning film, were always present and made it unattractive to expend much effort on the measurement of reaction rates for reaction (2) which produces ethylene. Decomposition by this latter channel is accompanied by the production of radicals and traces of propane were observed in some of the high temperature experiments. Some differences (10-30%) in the apparent rate constants for propylene formation were observed between the experiments with butene getter and those using neat 1-iodopropane (Fig. 2). However, both negative and positive deviations occur and reflect mainly data scatter.

We note that earlier experimental data obtained in VLPP studies of 1-iodopropane also indicates some apparent experimental complications.[2,3] In that work, apart from scatter in at least some of the data, comparable to that shown here, the observed k_1/k_2 ratio decreased (rather than the expected constancy) as the number of collisions in the reactor increased; also, rates in a 2140-collision reactor showed a larger temperature dependence than did a 19,950-collision reactor. Additionally, reaction (2), unlike reaction (1), was found to show no dependence on the pressure of added inert bath gas. Not much weight was placed on these complications by the earlier workers. We believe that although experimental complications do affect the quantitative precision of the results, that the general conclusions derived below and based on reaction (1) are

substantially valid.

The RRKM models employed for reactions (1) and (2) were based on those reported by Gaynor et al. (2). If molecules were effectively in the second order region, the only parameter of critical importance would be E_o, the critical energy for reaction, together with the molecular parameters. However, 1-iodopropane only approaches the second order region under the conditions used in this study. The fraction of molecules activated above E_o which react between collisions is $\sim$ 14% at 691 K and increases to $\sim$ 19% at 804 K. The molecular and activated complex parameters and vibrational frequencies used are listed in Table I. We have altered Gaynor's frequency factor and reaction path degeneracy for reaction (1), taking log A_∞ to be 13.0 and $L^\ddagger$ to be 1.5 1-Iodopropane has roughly equal probabilities of being in the trans and gauche forms (6,7); these have reaction path degeneracies for reaction (1) of 2 and 1, respectively. Hence, an overall reaction path degeneracy of 1.5 seems to us more realistic than the reported value of 0.33. Calculated values of the microscopic rate constants k_E are plotted versus energy in Fig. 3.

The computer simulation of the encounter process has been described elsewhere.[4] Four models have been used to characterize the probability, p_{ij}, of a down-transition by the molecule, from energy E_j to energy E_i:[5,8]

Model FE (flat exponential, i.e., $\langle\Delta E\rangle$ independent of E_j):

$$p_{ij} = A_1 \exp(-\Delta E/\langle\Delta E\rangle); \quad \Delta E = E_i - E_i$$

Model FG (flat gaussian, i.e., ΔE_{mp} independent of E_j) :

$$p_{ij} = A_2 \exp(-(\Delta E-\Delta E_{mp})^2/2\sigma^2);$$

Model EB (exponentially weighted Boltzmann):

$$p_{ij} = A_3 N(E_i) \exp(E_i/RT) \exp(-\Delta E/\langle\Delta E\rangle);$$

Model GB (gaussian weighted Boltzmann):

$$p_{ij} = A_4 N(E_i) \exp(-E_i/RT) \exp(-(\Delta E-\Delta E_m)^2/2\sigma^2);$$

The A's are normalization constants, $N(E_i)$ is the density of quantum states at energy E_i, and $\langle\Delta E\rangle$, ΔE_{mp} and σ are parameters of the models. In this calculation, σ was given the value of 0.7 ΔE_{mp}. For Models FE and FG, a few transitions can occur in principle to energy levels $E_i < 0$, and these were simply treated as elastic. Alternative treatments were explored and do not affect present conclusions. Of these models, FE has been shown to be inappropriate for surfaces that behave as near-strong colliders, as is the case here at the lower temperatures; results for this model will not be shown. Truncation of the transition probabilities for ΔE greater than 18,000 cm^{-1} (i.e. $p_{ij} = 0$ for $\Delta E \geq 18{,}000$) was employed for most of the computations but gave results essentially identical to those obtained in calculations where no truncation was employed.

Representative parameters of each of the models which produce a fit to the 5.9 reactor data are listed in Table 2, and the calculated probabilities of reactions per collision in a given reactor, $\bar{P}_c(m)$, are listed in Table 2 and presented in Fig. 1. Also shown is a calculated curve for an ideal strong collider. The parameters derived from the m = 5.9 fit were then applied to the calculation of curves for case m = 17 and comparison of these with experiment is also shown in Fig. 1.

The effective average energy of down-transitions, $\langle\Delta E'\rangle$ for Models EB and GB is energy dependent; therefore, in order to characterize these models, the average energy of down-transitions is presented for the case $E_j = \frac{2}{3} E_o$ (E_o for reaction (1)).

Discussion

At lower temperatures (Fig. 1), the $P_c(m)$ values for the reactor m = 17.5 appear within experimental uncertainty to be indistinguishable from those for a true strong collider and correspond to the steady state, i.e., to $\bar{P}_c(\infty)$. The values for the m = 5.9 reactor are a little lower than the strong collider values and show more unequivocally that the surface behaves as a rather efficient collider but only approaches near-strong behavior at temperatures below 700 K. Models EB and GB, which effectively reproduce strong collider behavior in the limit of very large values of the relevant parameters ($\langle\Delta E\rangle$ and ΔE_{mp}) appear to give a better fit to the data (Fig. 1) at low temperatures than does FG, although the scatter in the data is large and this prohibits a decisive conclusion.

Despite the experimental uncertainties, it is possible to draw some important qualitative conclusions. Once again the trend observed in previous VEM studies of decreasing step size with increasing temperature is borne out. More importantly, in its interaction with the wall, this molecule seems to be a much stronger collider than the hydrocarbon molecules previously studied by VEM in this temperature range.[4,5,8] We presume that this results from the larger polarity and stronger attractive molecular potential of the molecule.

We may make a limited comparison of our results with those reported in VLPP studies of 1-iodopropane.[1,3] The highest temperature at which we were able to obtain reliable data (840 K) is a little lower than the lowest temperature (850 K) at which the VLPP results were reported. But, while Gaynor, et al. report that the rates of reactions (1) and (2) are about equal, our calculated steady state k_2/k_1 ratio (at 805 K) based on the preferred energy transfer models is much lower, 0.29 or less, as shown in Table 3. In fact, based on our experiments, 0.29 is also a maximum in our observed experimental ratios. We are uncertain as to the origin and significance of this difference.

Table I. Molecular Parameters for RRKM Calculations[a]

	1-iodopropane	Complex (1)	Complex (2)
frequencies/cm^{-1}	3100(7)	3100(6)	3100(7)
	1450(5)	2200	1450(5)
	1150(6)	1450(4)	1150(4)
	1000(2)	1300	1000(2)
	700	1150(7)	700(2)
	600	1000	400
	425(2)	700(2)	200
	300	300	130(3)
	200	219(3)	70
	70		
$(I_A I_B I_C)^+/(I_A I_B I_C)$	--	2.83	3.09
$L^{\ddagger}$	--	1.5	1.0

	E_o/kcal $mole^{-1}$	$E_{a\infty}$/kcal $mole^{-1}$	$\log_{10}(A_\infty/s^{-1})$
Reaction (1)	48.5	49.5	13.0
Reaction (2)	52.8	55.7	15.5

a) Based on refs. 2 and 3, except as in text

Table 2. Calculated Values of $\bar{P}_c(m)$ and Parameters of the Energy Transfer Models

T/K	Model	$\langle\Delta E\rangle$ or ΔE_{mp}	$\langle\Delta E'\rangle$	$\bar{P}_c(5.9)$[a]	$P_c(17.5)$
690	FG	3740	4136	5.69-8[c]	1.19-7
	EB	1370	4113[b]		1.27-7
	GB	2165	3884[b]		1.27-7
805	FG	2730	3012	2.62-6	6.38-6
	EB	1335	2664[b]		7.07-6
	GB	1895	2888[b]		7.08-6

a) Models fitted to the experimental values for m = 5.9.

b) This quantity is energy-level dependent and was calculated for a representative value of $E = \frac{2}{3} E_o$; the quantity increases with increase of E.

c) Signifies 5.69×10^{-8}

Table 3. Calculated Values of k_2/k_1

T/K	Model FE(m = 17.5)	Model EB(m = 17.5)
691	0.14	0.17
805	0.21	0.29

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Figure Captions

Figure 1. Plot of experimental and calculated values of $P_c(m)$ for reaction (1) vs temperature (K) for 1-iodopropane. Experimental results: ○, m = 5.9 ; □, m = 17.5. Calculated results: curve 1, strong collider; curve 2, Models EB and GB for m = 17.5; curve 3, Model FG for m = 17.5; curve 4, calculated and experimental values for m = 5.9. Error bars show 95% confidence level.

Figure 2. Plots of ln $A/\mathring{A}$ vs time for several runs, with and without added butene. The ratio of rate constants in the two cases is given in the figure; ○, 626 K; △, 694 K; □, 828 K. Filled symbols refer to butene runs. Time is in minutes at 626 K and 694 K and in seconds at 828 K.

Figure 3. Plot of calculated values of $\log_{10}(k_E/s^{-1})$ vs E/cm^{-1} for reaction (1), (●), and reaction (2), (x).

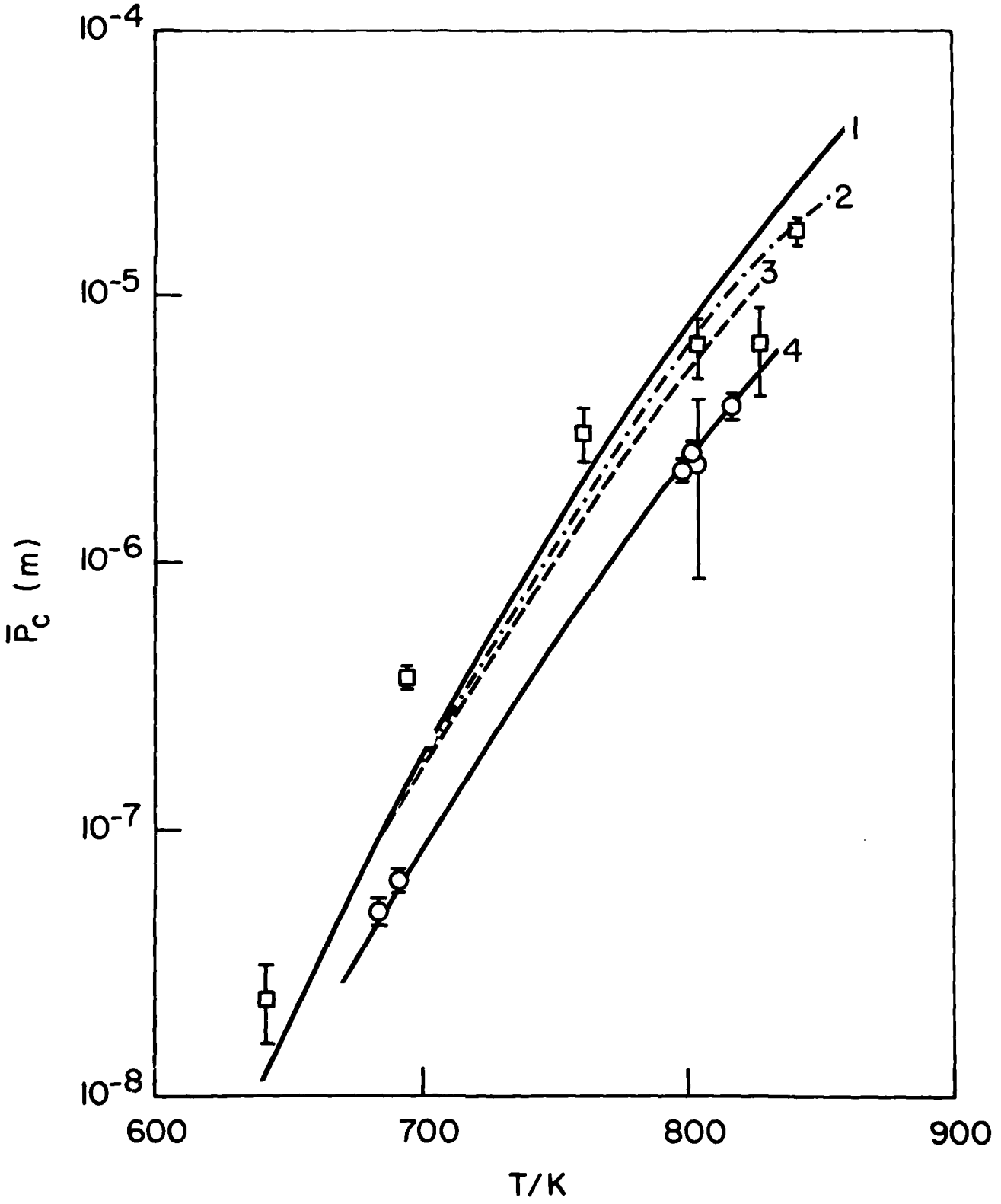


Fig 1 Wolfers et al

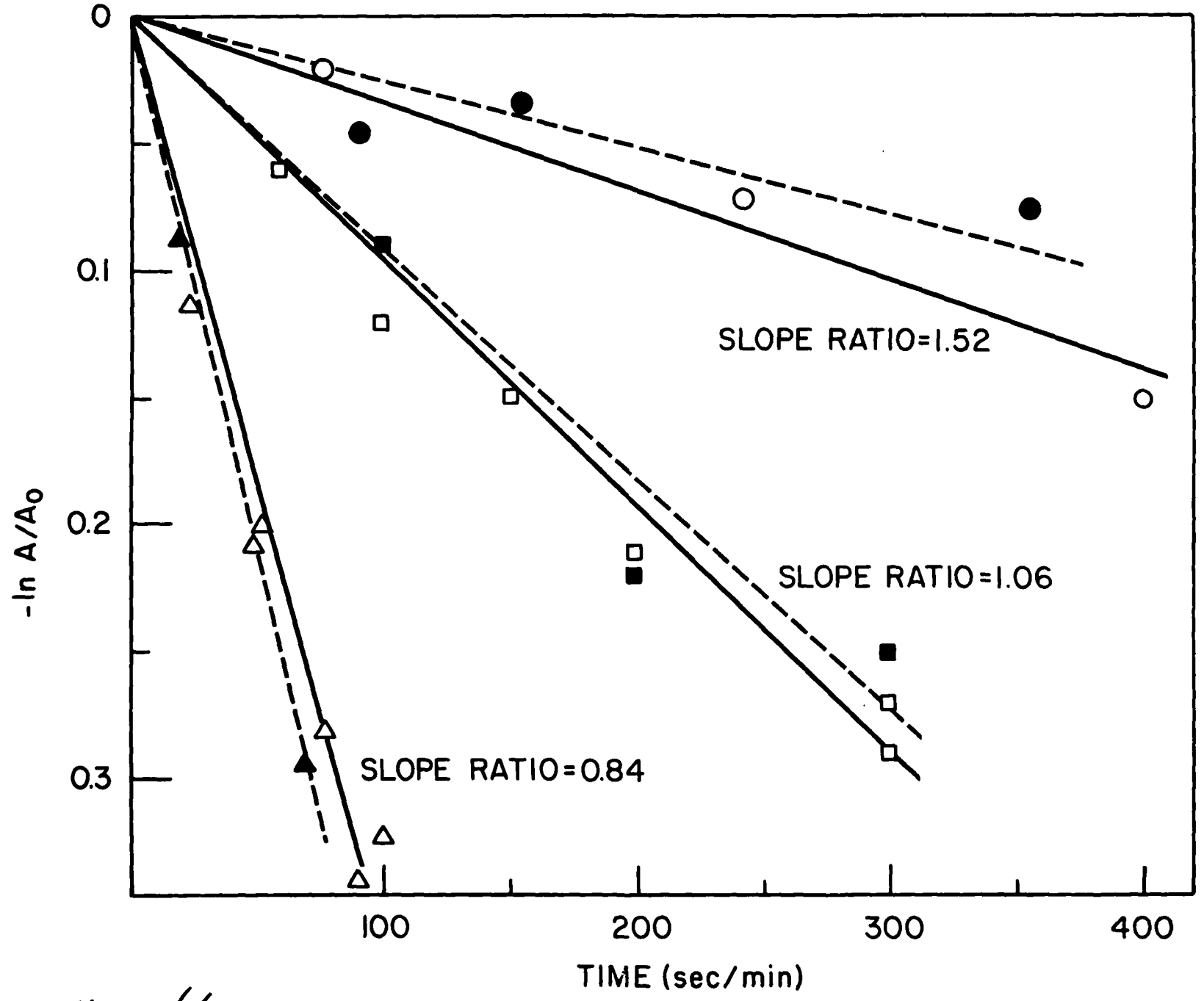
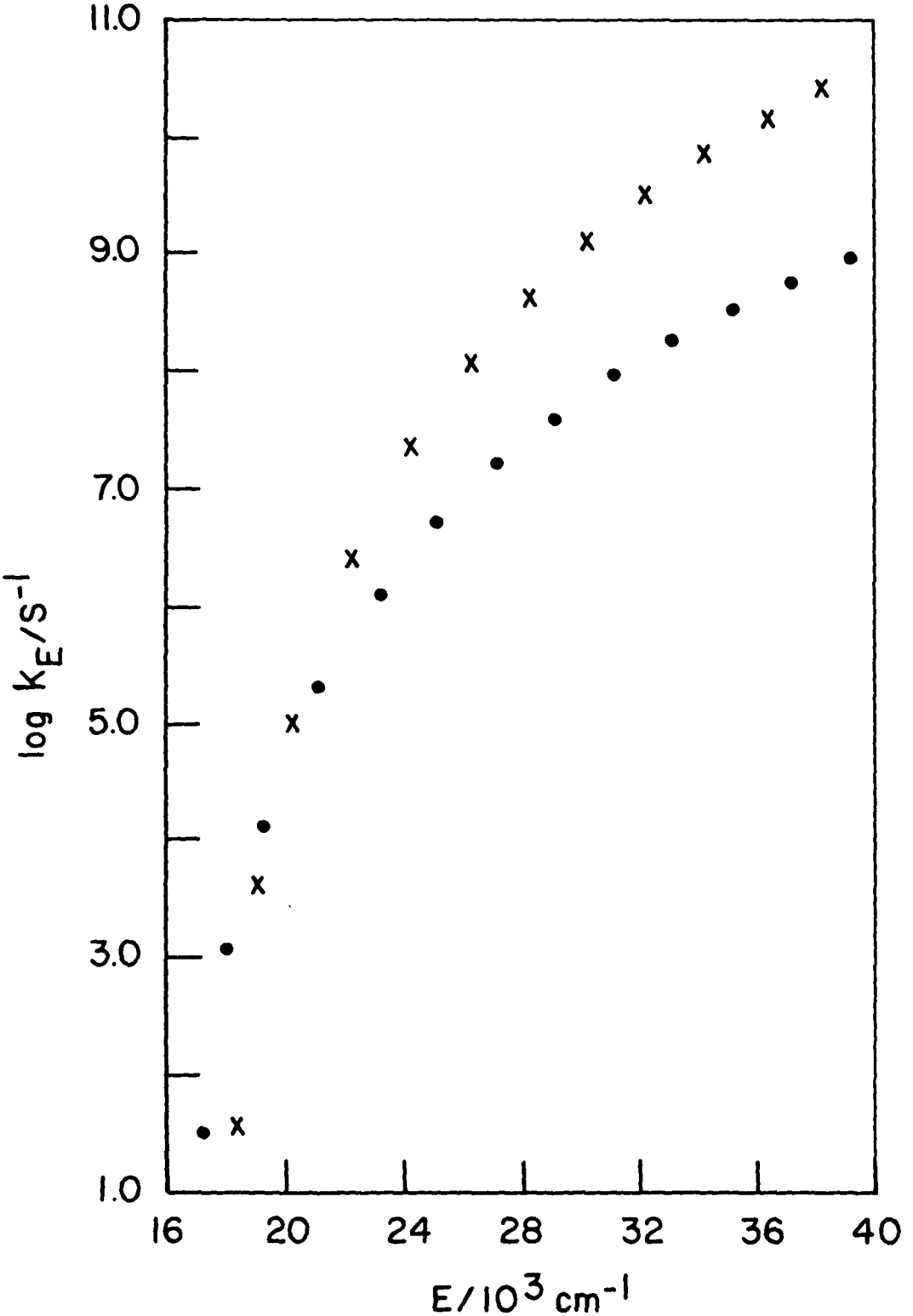


Fig 2 Walters et al



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