

Comparative apparent kinetics of menthol and thymol O-vinylation with acetylene in KOH-DMSO

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Research Article

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Abstract

The temperature dependence of the apparent O-vinylation kinetics of menthol and thymol with acetylene was compared in a potassium hydroxide–dimethyl sulfoxide (KOH–DMSO) medium. Experiments were performed in independently operated 250 mL reactor cells at an acetylene pressure of 14 atm, a KOH/substrate molar ratio of 0.50, and 240 rpm. After the set temperature and pressure had stabilized, samples were collected over 0–120 min at 100–140°C. Substrate and target vinyl ether concentrations were determined by GC-FID in three reactor runs per condition. Integrated zero-, first-, and second-order expressions were fitted on the original concentration scale and compared using the Akaike information criterion. An apparent zero-order expression gave the lowest AIC for all substrate-temperature combinations. From 100 to 130°C, the apparent zero-order rate coefficient increased from 0.1079 to 0.2399 mol L⁻¹ h⁻¹ for menthol and from 0.1341 to 0.2958 mol L⁻¹ h⁻¹ for thymol. Arrhenius fits over this interval gave apparent activation energies of 34.9 kJ mol⁻¹ for menthol and 37.5 kJ mol⁻¹ for thymol; however, the wide, overlapping 95% confidence intervals do not establish different activation barriers. At 140°C, both target-product accumulation and the apparent substrate-depletion rate declined relative to 130°C, while byproduct signals and dry resinous residue increased. These observations identify 130°C as the most favorable tested temperature for target vinyl ether formation and show that the measured rate law is an operational, rather than elementary, kinetic description.

Article Highlights

- Thymol reacted faster than menthol from 100 to 130 °C
- An apparent zero-order model best described the 0–120 min data
- Above 130 °C, target-product formation declined as residues increased

1. Introduction

Vinyl ethers are versatile intermediates and monomers because the electron-rich C = C–O unit participates readily in addition, cycloaddition, and cationic polymerization chemistry [1, 11–14]. Direct addition of hydroxy compounds to acetylene is atom-economical and remains an important non-transition-metal route to O-vinyl derivatives [1, 2, 11–14]. In KOH–DMSO media, strong cation solvation and generation of highly reactive alkoxide or phenoxide species promote nucleophilic attack on acetylene; water content and the detailed solvation structure of the superbases can alter both vinylation and competing pathways [1, 3, 15].

Phenols and naphthols have been O-vinylated with acetylene in KOH–DMSO at approximately 120°C and 13–15 atm in yields up to 80% [2]. Calcium carbide provides a safer, in situ acetylene source for the vinylation of primary and secondary alcohols, phenols, thiols, and nitrogen nucleophiles [4, 5, 13]. Earlier investigations of menthol and thymol established technological yield trends, response-surface relationships, and a maximum target-product yield near 130°C [6, 9, 16]. Those studies did not discriminate integrated kinetic orders from replicated 0–120 min concentration profiles or report

uncertainty-qualified Arrhenius parameters under one common protocol. Relevant work further shows that apparent constants in base-catalyzed liquid-phase systems may combine equilibrium, reaction, and decomposition contributions; therefore, model validation and explicit consideration of secondary pathways are essential [18].

This study evaluates the 0–120 min kinetic window for acetylene O-vinylation of menthol and thymol at 100–140°C. It compares three integrated rate expressions on a common response scale, estimates apparent Arrhenius parameters over the monotonic 100–130°C interval, and assesses the departure observed at 140°C without assigning an unverified elementary mechanism.

2. Materials and methods

2.1. Reagents and reactor

Menthol (99%), thymol (99%), potassium hydroxide (KOH; 85% assay), dried dimethyl sulfoxide (DMSO; 99%), and acetylene (99.5%) were used. For each run, 18.9418 g menthol or 18.2085 g thymol, 120 mL DMSO, and 3.9604 g KOH were charged. After correction for KOH assay, the KOH/substrate molar ratio was approximately 0.50. Because reaction mixtures are non-ideal and their final liquid volume was not independently measured, the GC-FID concentration at the defined time zero, rather than a recipe-based nominal concentration, was used as C_0 in kinetic fitting. Experiments were run at 240 rpm in independently controlled 250 mL cells of a GPXF-4 × 250 mL reactor system.

2.2. Kinetic experiment and definition of time zero

Each reactor cell was charged separately with freshly weighed reagents, sealed, pressure-tested, purged, pressurized with acetylene to 14 atm, and heated to 100, 110, 120, 130, or 140°C. The corresponding heat-up times were 37, 42, 46, 51, and 55 min and were excluded from the isothermal kinetic time. Time zero was defined when the internal temperature reached $T_{\text{set}} \pm 1^\circ\text{C}$ and acetylene pressure stabilized at 14.0 ± 0.2 atm under continued mixing at 240 rpm. Samples were withdrawn at 0, 15, 30, 45, 60, 90, and 120 min. R1–R3 denote three independently prepared and independently operated reactor charges, not repeated GC injections or aliquots from one batch. Thus, each substrate contributed 5 temperatures × 7 times × 3 independent reactor runs = 105 concentration observations.

2.3. GC–FID quantification

Biphenyl (99.5%) was added to each quenched sample before filtration as an internal standard. Analyses were performed on a Chromatec-Crystal 9000 gas chromatograph equipped with an FID and a CR-5 capillary column (30 m × 0.25 mm × 0.25 μm). Seven-level response-factor calibrations, each based on three injections per level and 1/x weighting, were used. The substrate-class calibration was $y = A_{\text{sub}}/A_{\text{IS}}$ versus $x = C_{\text{sub}}/C_{\text{IS}}$, $y = 1.2463x + 0.0031$ ($R^2 = 0.9992$); the target-vinyl-ether-class calibration was $y = A_{\text{product}}/A_{\text{IS}}$ versus $x = C_{\text{product}}/C_{\text{IS}}$, $y = 1.0837x + 0.0026$ ($R^2 = 0.9988$). Concentration ratios were calculated on one consistent molar basis after applying the same sample-dilution scheme. The common

function within each paired class was therefore treated as a class-level response-factor calibration, rather than as two purportedly independent but numerically identical compound-specific calibrations. Additional peaks were semi-quantified as the corresponding target-vinyl-ether equivalents and were not assigned to individual structures.

2.4. Kinetic and statistical analysis

All calculations were independently repeated from the raw $C_{\text{sub}}(t)$ and $C_{\text{product}}(t)$ values. Integrated zero-, first-, and second-order models were expressed as follows:

$$C = C_0 - k_0 t; C = C_0 \exp(-k_1 t); C = C_0 / (1 + k_2 C_0 t).$$

Each model parameter was fitted to the 0–120 min data by nonlinear least squares, with residuals evaluated on the original concentration scale. Models were compared by the Akaike information criterion, $AIC = n \ln(\text{SSE}/n) + 2p$, with $p = 1$ for all models. $\Delta AIC = 0$ identifies the best-supported model, whereas values above 2 indicate reduced relative support [10]. After zero order was selected, k_0 for each independent charge was calculated as $\Sigma[t(C_0 - C)]/\Sigma t^2$ and reported as mean $\pm$ SD. Arrhenius analysis was restricted to the monotonic 100–130°C region using $\ln k_0 = \ln A - E_a/(RT)$. The 95% confidence interval for E_a used the linear-regression slope standard error and a t distribution with $n - 2$ degrees of freedom.

3. Results and discussion

3.1. Concentration–time profiles

For both substrates, raising the temperature from 100 to 130°C increased substrate depletion and vinyl ether accumulation over 120 min (Fig. 1). For menthol, the 120 min product concentration rose from $0.2801 \pm 0.0035 \text{ mol L}^{-1}$ at 100°C to $0.5020 \pm 0.0013 \text{ mol L}^{-1}$ at 130°C; for thymol, it rose from 0.3387 ± 0.0007 to $0.7026 \pm 0.0024 \text{ mol L}^{-1}$. Thymol showed especially rapid product formation at 120–130°C. At 140°C, however, both systems gave lower conversion and product concentration than at 130°C.

3.2. Reaction-order discrimination

AIC comparison on the original concentration scale selected the zero-order model for all ten substrate–temperature combinations (Table 1). Its advantage over first order was moderate at 100–110°C (ΔAIC 2.6–3.7) and generally strong at 120–140°C (ΔAIC 4.9–28.3). Thus, assuming pseudo-first-order behavior a priori is not supported over this experimental interval. The observed zero order does not imply a zero-molecularity elementary event; it may be an apparent regime governed by gas–liquid transfer, limitation of active acetylene concentration, or saturation of the basic phase.

Table 1
Model discrimination for the 0–120 min data. Zero order
has $\Delta AIC = 0$ in every row.

Substrate	T (°C)	R^2 , zero order	ΔAIC_1	ΔAIC_2
Menthol	100	0.789	2.6	4.7
Menthol	110	0.843	3.4	6.2
Menthol	120	0.962	11.1	19.0
Menthol	130	0.969	17.9	28.3
Menthol	140	0.948	12.6	20.7
Thymol	100	0.809	3.2	5.9
Thymol	110	0.809	3.7	6.6
Thymol	120	0.984	28.3	41.5
Thymol	130	0.887	11.4	17.8
Thymol	140	0.853	4.9	8.7

3.3. Temperature dependence of the apparent rate

Zero-order rate constants are summarized in Table 2. For menthol, k_0 increased 2.22-fold from 0.1079 ± 0.0027 to 0.2399 ± 0.0011 mol L⁻¹ h⁻¹ between 100 and 130°C. For thymol, it increased 2.21-fold from 0.1341 ± 0.0003 to 0.2958 ± 0.0006 mol L⁻¹ h⁻¹. At 130°C, thymol k_0 was 23% higher than menthol k_0 . Under the same condition, the analytical yield was $70.26 \pm 0.13\%$ for thymol and $50.20 \pm 0.03\%$ for menthol. This yield difference reflects the integrated concentration profile and product-directed selectivity, not the rate constant alone.

Table 2
Zero-order k_0 and 120 min outcomes (mean $\pm$ SD, $n = 3$).

Substrate	T ($^{\circ}\text{C}$)	k_0 ($\text{mol L}^{-1} \text{h}^{-1}$)	$C_{\text{product},120}$ (mol L^{-1})	Conversion (%)	Yield (%)
Menthol	100	0.1079 ± 0.0027	0.2801 ± 0.0035	29.39	28.01 ± 0.39
Thymol	100	0.1341 ± 0.0003	0.3387 ± 0.0007	35.50	33.87 ± 0.05
Menthol	110	0.1182 ± 0.0010	0.2902 ± 0.0012	30.45	29.02 ± 0.07
Thymol	110	0.1491 ± 0.0004	0.3775 ± 0.0007	39.57	37.75 ± 0.03
Menthol	120	0.1770 ± 0.0004	0.3751 ± 0.0013	39.36	37.51 ± 0.12
Thymol	120	0.2792 ± 0.0004	0.5691 ± 0.0010	59.65	56.91 ± 0.07
Menthol	130	0.2399 ± 0.0011	0.5020 ± 0.0013	52.68	50.20 ± 0.03
Thymol	130	0.2958 ± 0.0006	0.7026 ± 0.0024	73.65	70.26 ± 0.13
Menthol	140	0.2234 ± 0.0006	0.4815 ± 0.0008	50.52	48.15 ± 0.03
Thymol	140	0.1532 ± 0.0005	0.3767 ± 0.0021	39.48	37.67 ± 0.16

3.4. Arrhenius parameters

Over 100–130 $^{\circ}\text{C}$, $\ln k_0$ versus $1/T$ gave $R^2 = 0.944$ for menthol and $R^2 = 0.883$ for thymol (Fig. 3). The estimates were $E_{\text{a}} = 34.9 \text{ kJ mol}^{-1}$ (95% CI 8.9–60.8) and $A = 7.59 \times 10^3 \text{ mol L}^{-1} \text{h}^{-1}$ for menthol, and $E_{\text{a}} = 37.5 \text{ kJ mol}^{-1}$ (95% CI – 4.0–79.1) and $A = 2.29 \times 10^4 \text{ mol L}^{-1} \text{h}^{-1}$ for thymol. With only four temperatures, confidence intervals are wide and the two-sided slope test for thymol gives $p = 0.060$. These values should therefore be treated as apparent parameters, and no statistically established menthol–thymol difference in E_{a} is claimed. Nevertheless, observed kinetic rates were higher for thymol at every temperature from 100 to 130 $^{\circ}\text{C}$.

3.5. Departure from Arrhenius behavior at 140 $^{\circ}\text{C}$

At 140 $^{\circ}\text{C}$, the apparent substrate-depletion coefficient decreased by 6.9% for menthol and 48.2% for thymol relative to 130 $^{\circ}\text{C}$; the 120 min target-product concentration decreased by 4.1% and 46.4%, respectively. Over the same temperature change, the dry resinous residue at 120 min increased from 0.01869 ± 0.00067 to $0.02861 \pm 0.00085 \text{ g}$ for menthol and from 0.03224 ± 0.00068 to $0.04459 \pm 0.00085 \text{ g}$ for thymol. At 140 $^{\circ}\text{C}$, semi-quantified byproduct signals also rose with time. These observations demonstrate an association between the kinetic downturn and increased secondary-product formation, but they do not by themselves identify the cause of the lower substrate-depletion rate. Product polymerization can lower measured target-product accumulation, whereas the decline in substrate depletion may additionally reflect inhibition, catalyst/base deactivation, altered acetylene availability, or mass-transfer behavior. The 140 $^{\circ}\text{C}$ point was therefore excluded from the simple Arrhenius fit and interpreted as a change in the operational kinetic regime rather than as proof of a specific elementary

pathway. This interpretation agrees with studies showing that kinetic models may be misleading when secondary pathways are omitted [18] and that vigorous agitation or explicit transport tests are needed before gas-liquid concentration profiles are treated as intrinsic kinetics [19].

3.6. Novelty and distinction from previous work

Earlier menthol/thymol studies reported technological or empirical yield responses to temperature, pressure, KOH loading, and residence time, and identified a yield maximum near 130°C [6, 9, 16]. The present work explicitly treats those publications as prior comparators. Its distinct contribution is the AIC-based discrimination of integrated rate expressions from replicated concentration-time series, the between-reactor distribution of k_0 , and uncertainty-qualified Arrhenius parameters over 100–130°C. This distinction is important because endpoint-yield optimization and kinetic model discrimination answer different questions. Other studies by the authors addressed stability modeling in a naphthylacetic acid-poly(vinyl alcohol) system [7] and the environmental performance of an agrochemical formulation [8]; neither study investigated acetylene O-vinylation kinetics. The operational use of a zero-order fit is also methodologically consistent with a study in which zero-order constants were obtained by linear least-squares regression and alternative first-order behavior was evaluated separately [17]. The observed apparent zero order is further consistent with the broader understanding that KOH–DMSO acetylene chemistry is strongly conditioned by solvation and acetylene availability [1, 3, 12, 15], although the present data do not isolate those effects mechanistically.

3.7. Limitations

The Arrhenius fit contains only four temperatures, giving wide confidence intervals; for thymol, the two-sided slope test gives $p = 0.060$. The apparent zero-order law does not distinguish intrinsic chemical kinetics from gas-liquid mass transfer or acetylene availability. A mixing-rate series and pressure-variation study, analogous to the transport checks used in gas-liquid kinetic investigations [19], are therefore required. Additional GC peaks were only semi-quantified, and the dry residue was not structurally characterized. Reported standard deviations represent between-reactor variability across $R1$ – $R3$; repeated calibration injections were used only to establish analytical response and were not counted as independent kinetic replicates. Accordingly, the results support an operational kinetic comparison but not an elementary mechanism.

4. Conclusions

Over 0–120 min at 14 atm acetylene, a KOH/substrate molar ratio of 0.50, and 240 rpm, an apparent zero-order expression described the menthol and thymol substrate profiles better than the tested first- and second-order expressions. From 100 to 130°C, thymol showed higher apparent rate coefficients than menthol; at 130°C the values were 0.2958 ± 0.0006 and 0.2399 ± 0.0011 mol L⁻¹ h⁻¹, respectively. The apparent activation energies were 37.5 and 34.9 kJ mol⁻¹, but their overlapping confidence intervals preclude a statistically established difference. At 140°C, lower target-product accumulation coincided with increased byproduct signals and resinous residue, indicating a changed operational regime. Among

the tested temperatures, 130°C gave the most favorable target-product formation. Establishing intrinsic chemical kinetics will require additional variation of acetylene pressure and mixing rate.

Declarations

Data availability

The raw time-concentration data, calibration records, independent-run metadata, and analysis code are available from the corresponding author on reasonable request.

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Author contributions:

Mahammadjon I. Soliev developed the research concept and methodology, designed and conducted the experiments, analyzed and interpreted the results, and prepared the original draft of the manuscript. The co-authors contributed to the experimental work, acquisition of analytical data, and discussion of the results. All authors reviewed and edited the manuscript, approved its final version, and agreed to its submission for publication.

Competing interests:

The authors declare that they have no financial or non-financial competing interests that could have influenced the research reported in this article.

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Figures

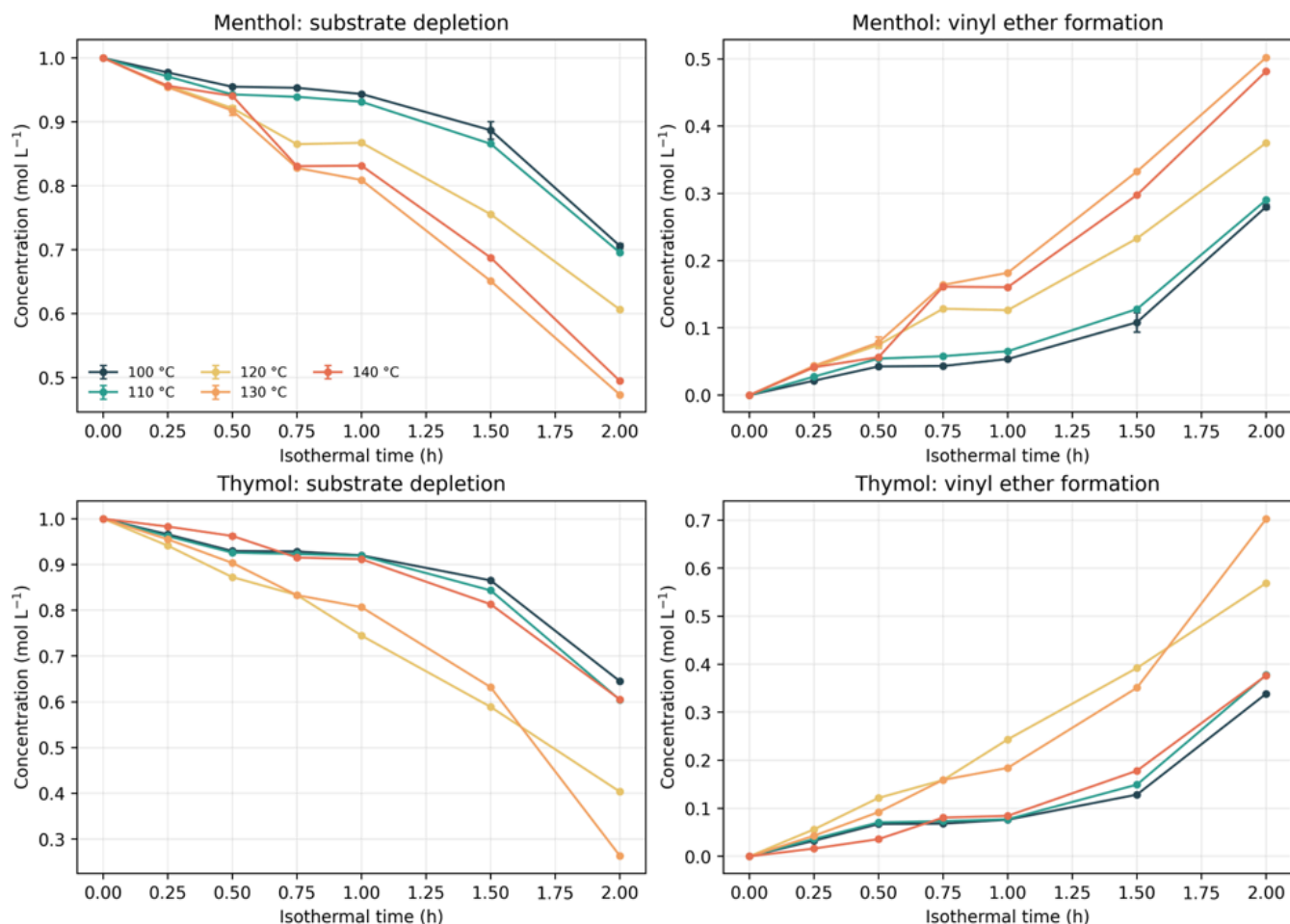


Figure 1

Concentration profiles over 0–120 min at 14 atm, 50 mol% KOH, and 240 rpm. Points are means of $n=3$; error bars are SD

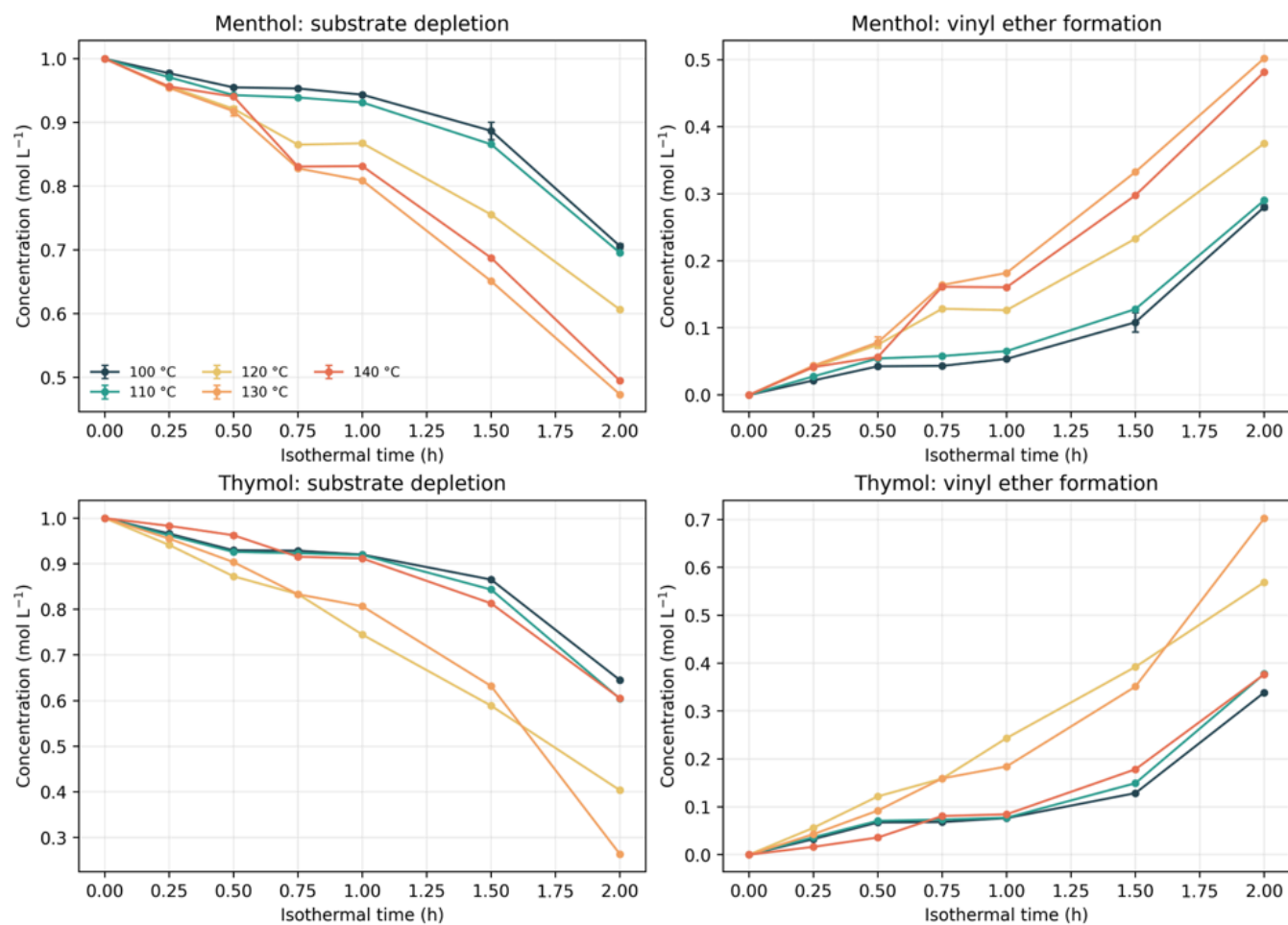


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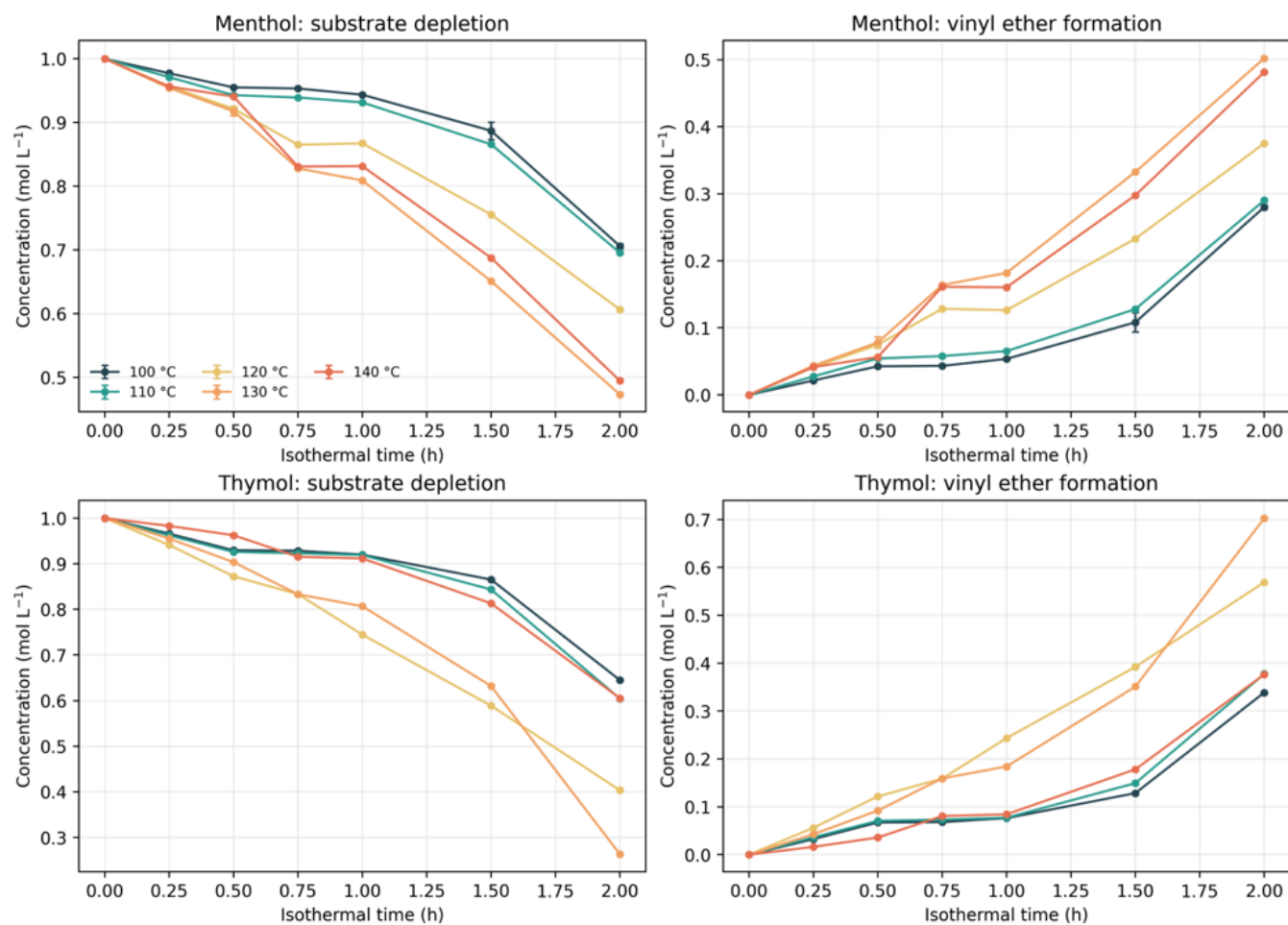


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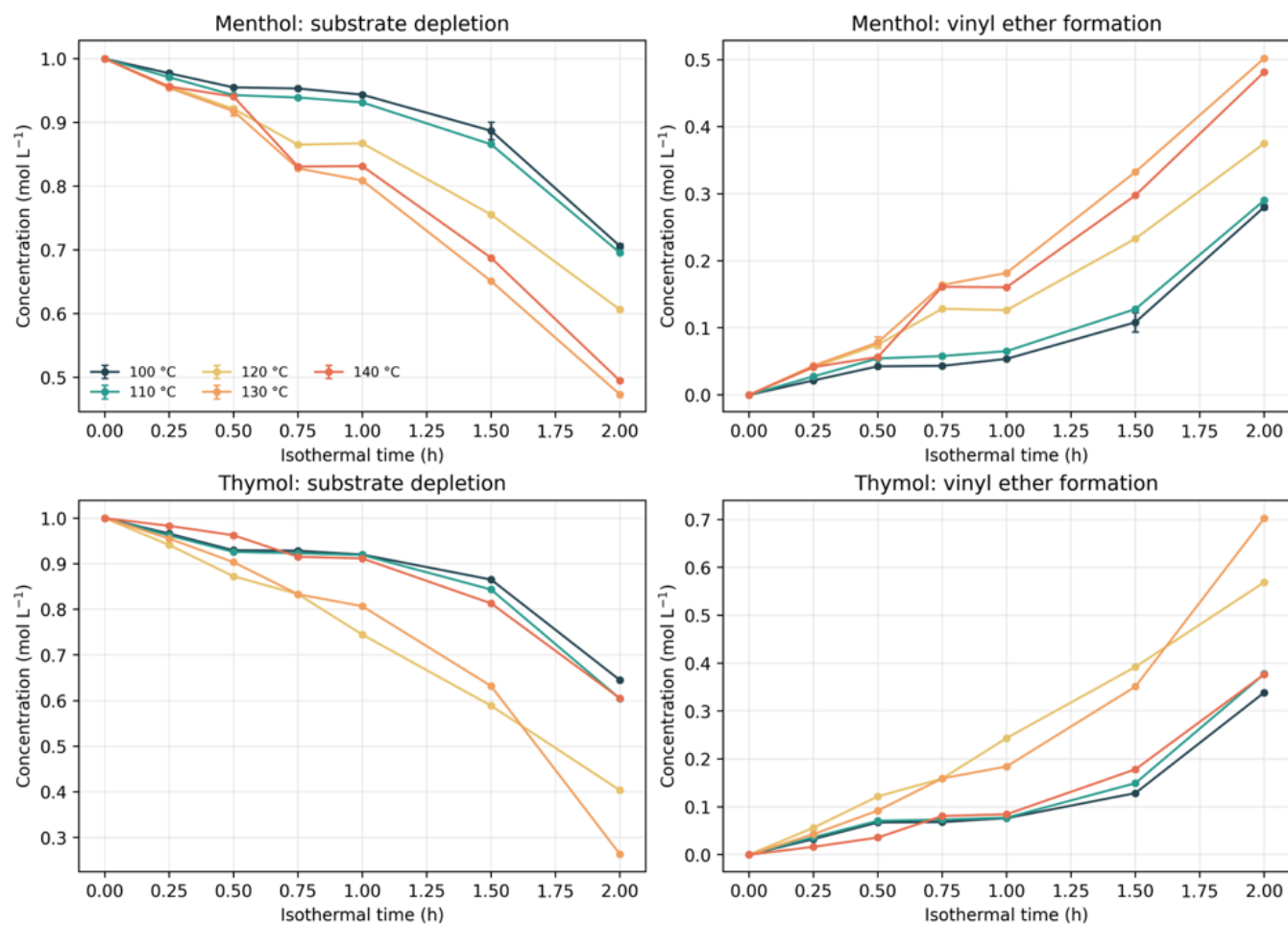


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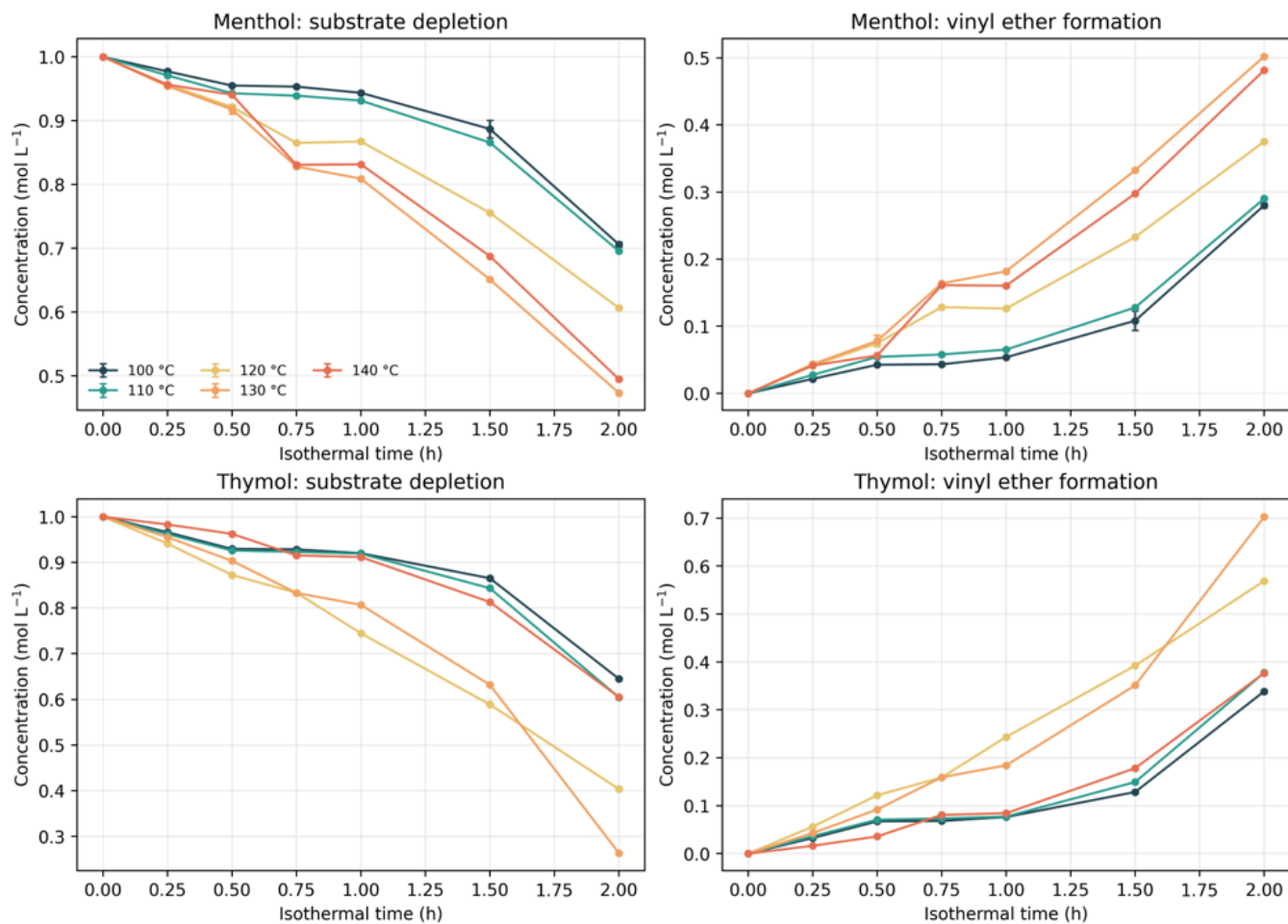


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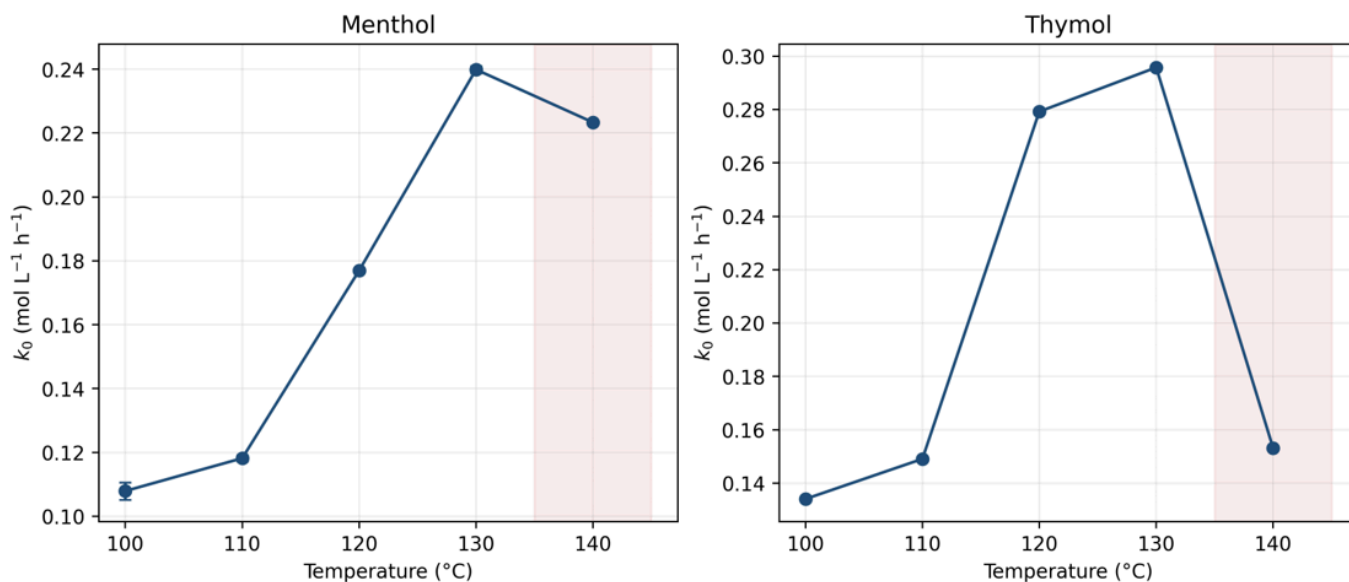


Figure 2

Effect of temperature on the apparent zero-order rate constant. The shaded region marks the 140 °C point excluded from the Arrhenius fit

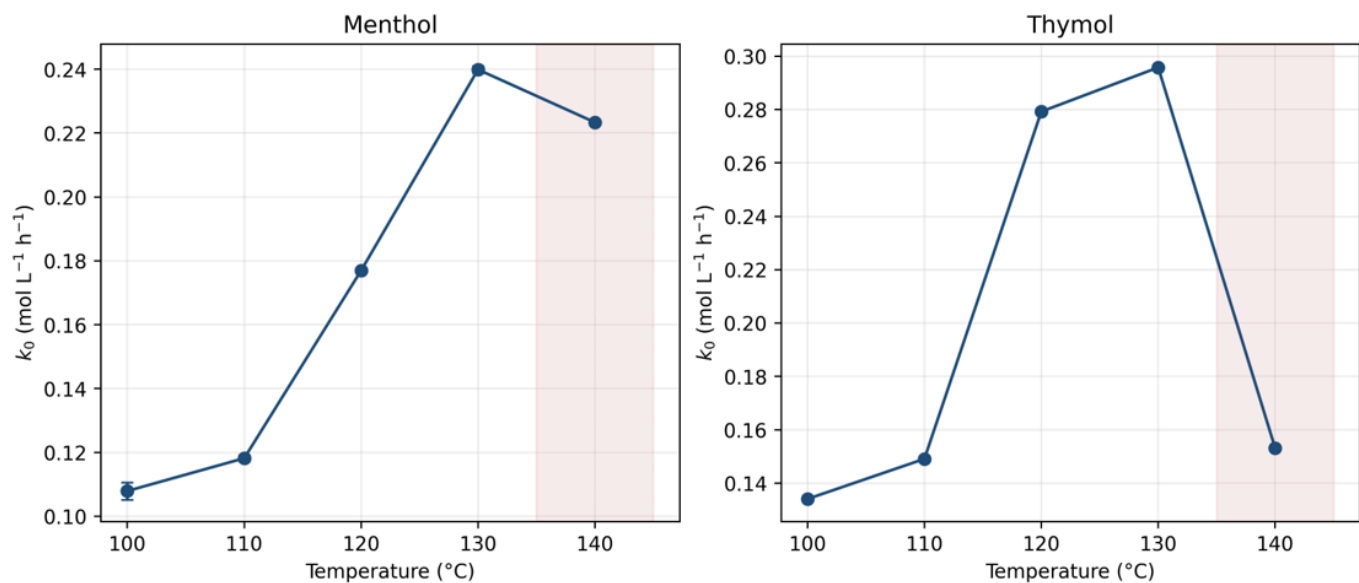


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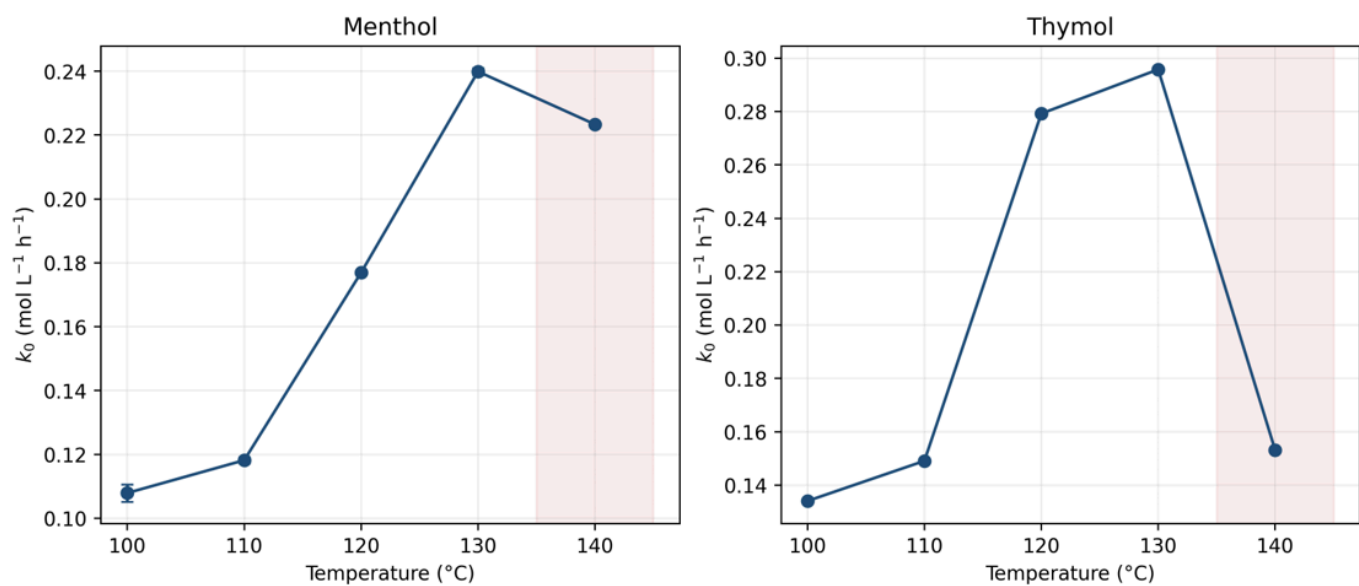


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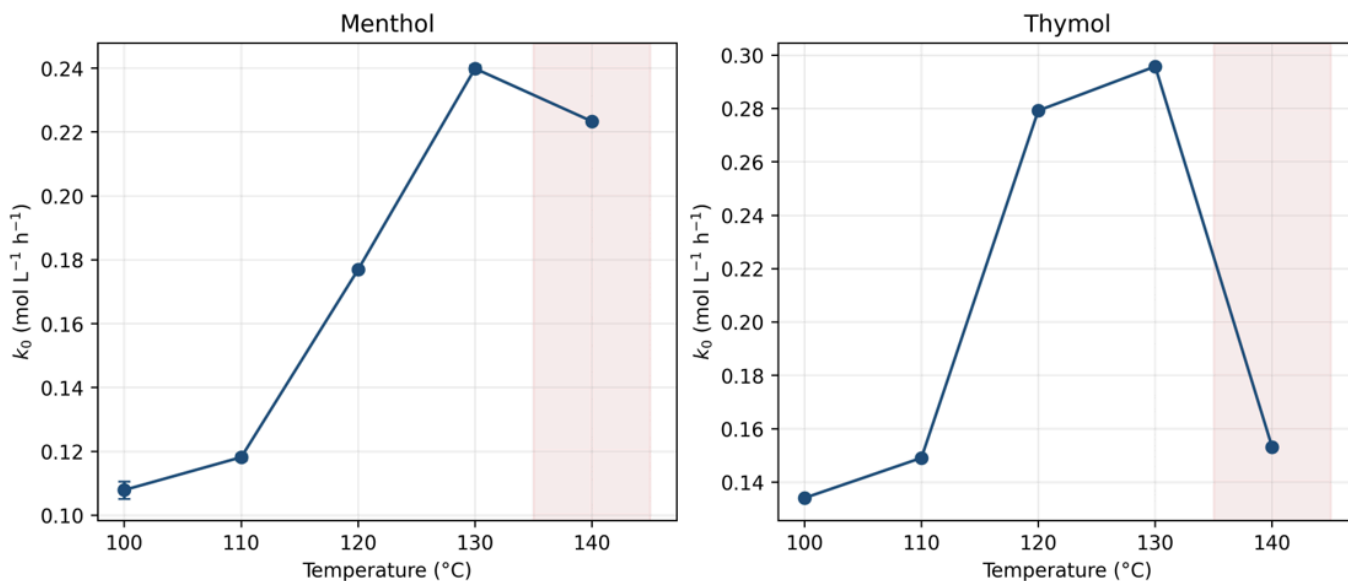


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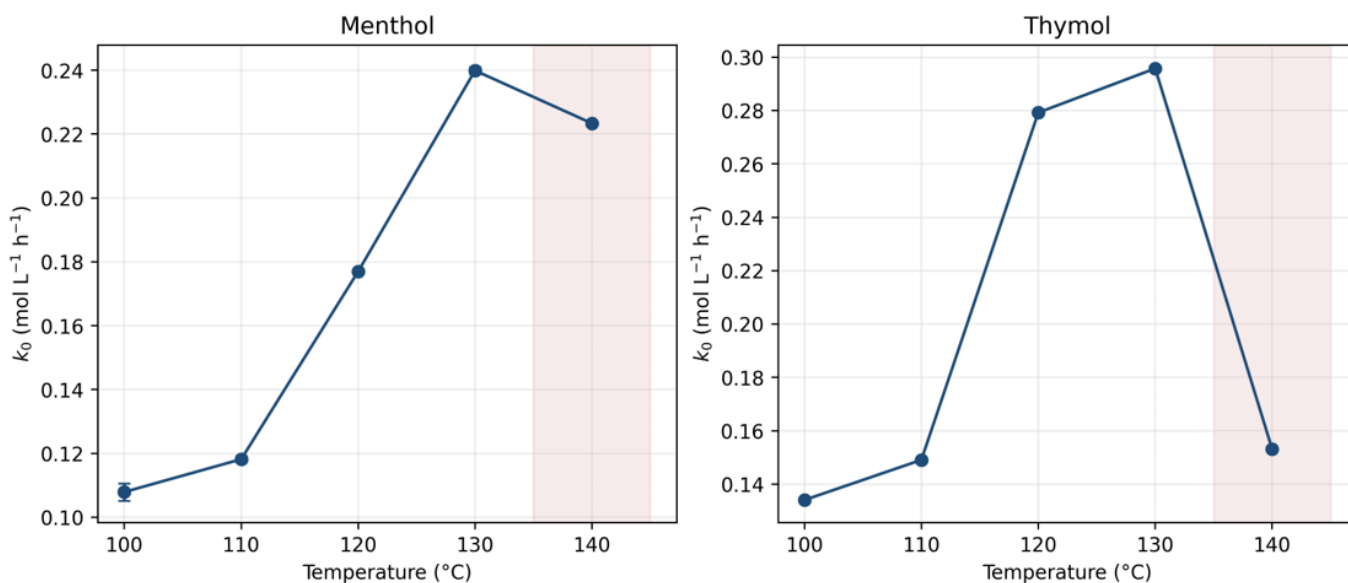


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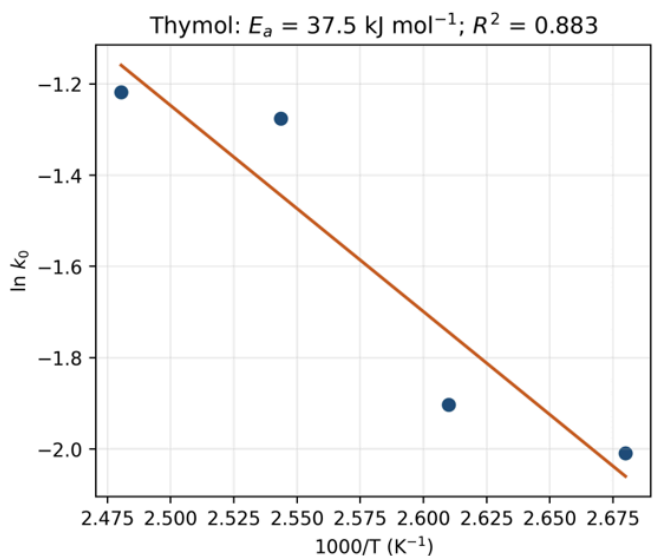
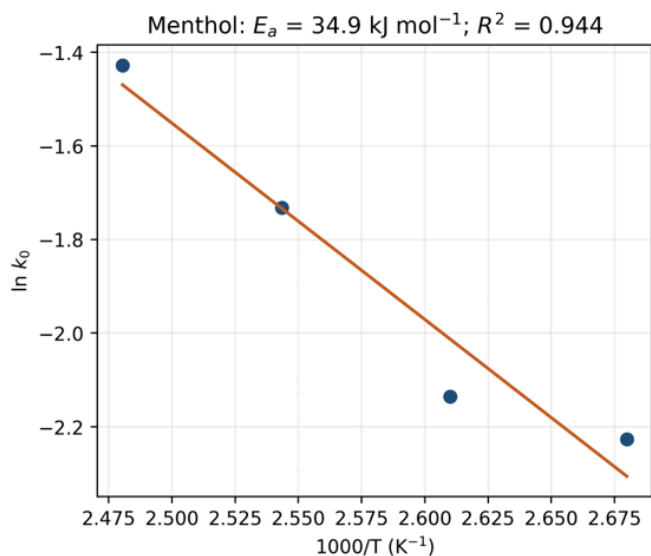


Figure 3

Arrhenius plots over 100–130 °C

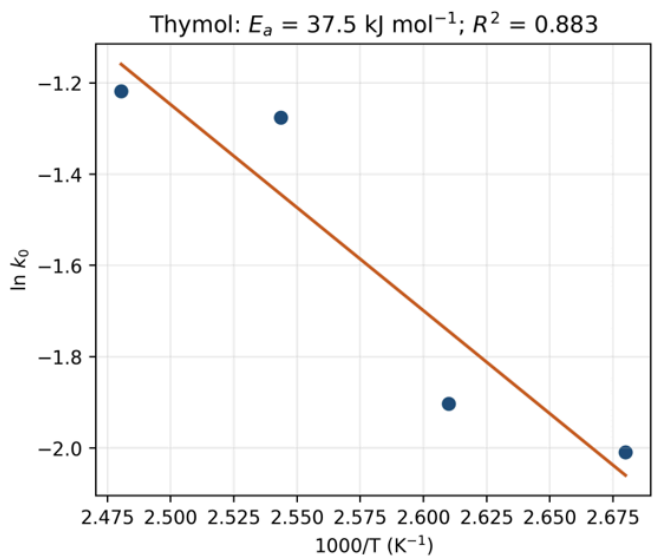
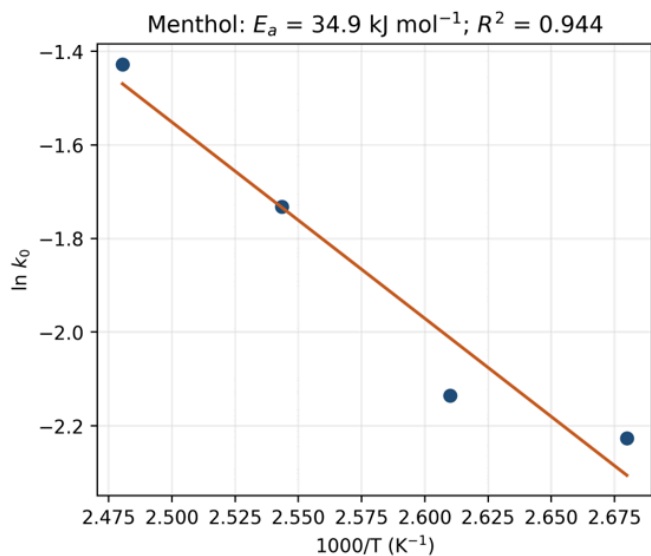


Figure 3

Arrhenius plots over 100–130 °C

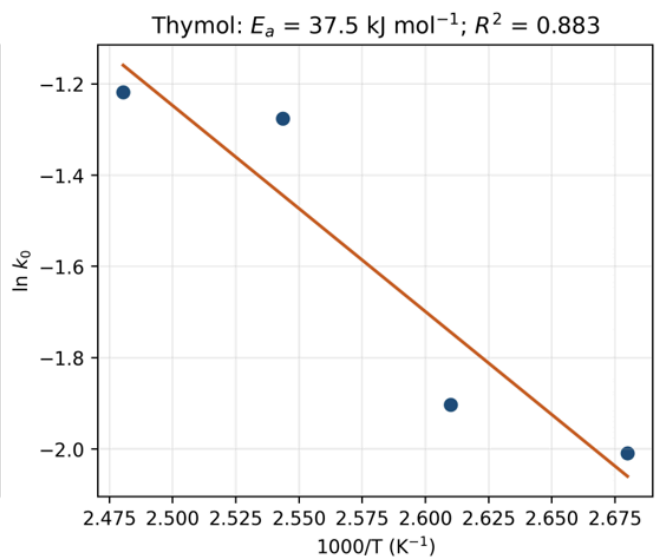
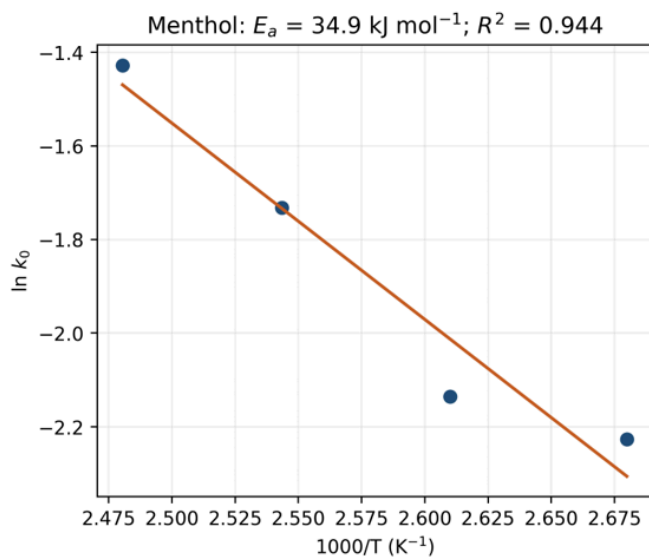


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Arrhenius plots over 100–130 °C

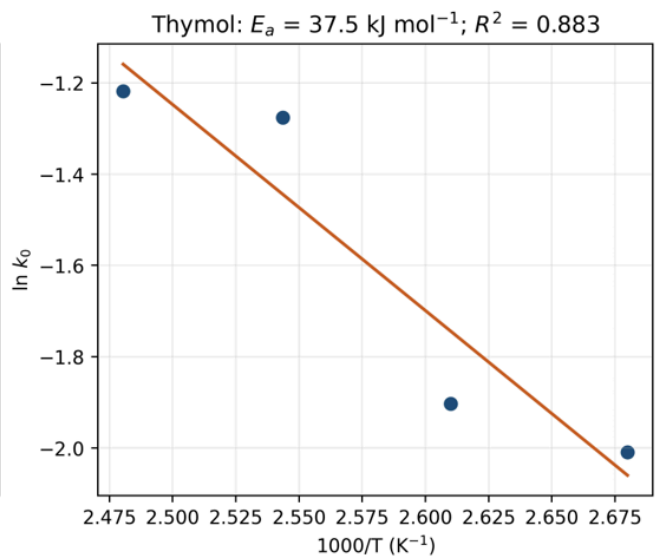
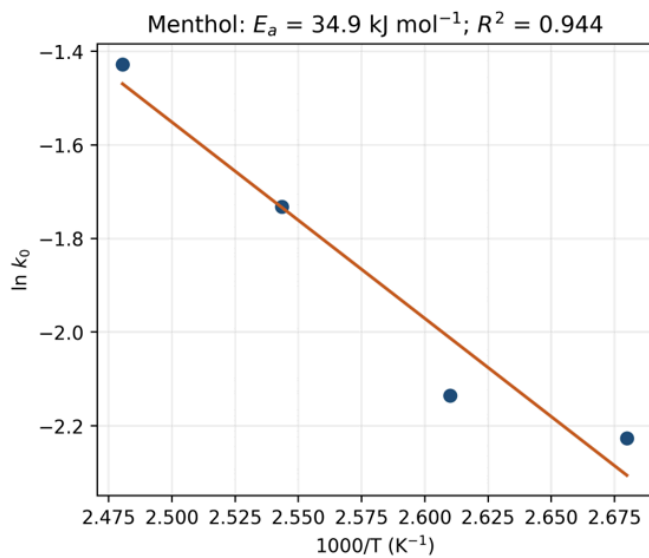


Figure 3

Arrhenius plots over 100–130 °C

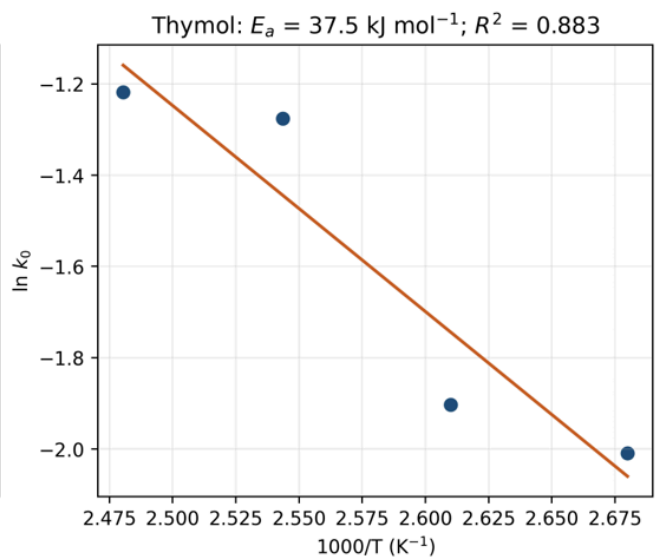
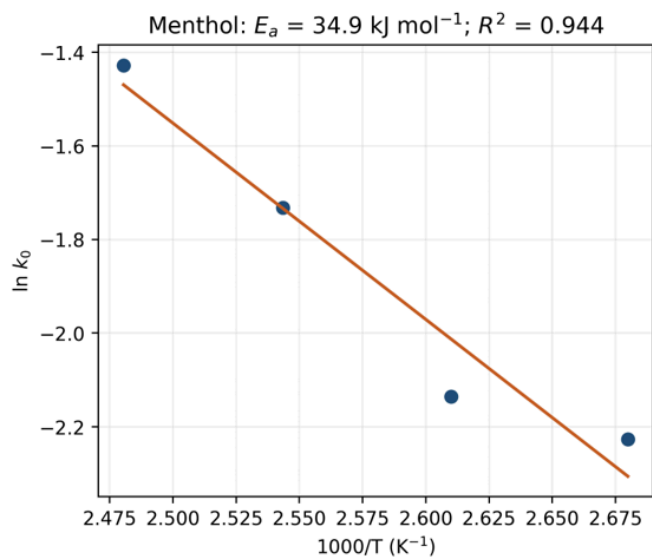


Figure 3

Arrhenius plots over 100–130 °C