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Chemical Equilibrium and Kinetics in the Self-Catalytic Reactions of 2,2,2-Trifluoroacetic Acid with Alcohols †

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Abstract

This study presents the first experimental data on the chemical equilibrium and kinetics of two self-catalytic reactions of trifluoroacetic acid (TFA) with 2,2,2-trifluoroethanol (TFE-ol) and TFA with propan-1-ol (P-ol), in which TFA acts simultaneously as a reactant and a catalyst. The results show that at an initial molar ratio of TFA/P-ol = 1/9 in the temperature range from 30 to 80 °C and the reaction rate coefficient k_1 changes over the course of the reaction. Notably, for the TFA/TFE-ol system (at an initial reactant ratio of TFA/TFE-ol = 1/9 and 5/5 and a temperature of 50 °C), the reaction rate coefficient k_1 remains constant. A new model describing the kinetics of the reaction with a variable rate coefficient is proposed, which accurately fits the experimental data for the TFA/P-ol reaction.

Keywords: chemical kinetic; self-catalytic reactions; esterification; trifluoroacetic acid; 2,2,2-trifluoroethanol; propan-1-ol

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1. Introduction

Fluorinated organic esters are of particular interest in modern industry due to their thermal and oxidative stability, low surface energy, high fluidity, density, and significant pharmacological effects [1]. Their production and consumption have been growing rapidly for several decades. Among those compounds, trifluoroacetic acid esters are currently utilized as reagents and solvents in fine organic synthesis, pharmaceutical chemistry and the production of other industrial goods.

The most common and widely applied method for ester synthesis is the esterification reaction between alcohols and acids. This reaction is catalyzed by Brønsted–Lowry or Lewis acids and the reaction mechanism is well-known and widely accepted [2]. In some cases, when one of the reactants is a strong acid, it acts as a catalyst within the system. In this scenario, the esterification reaction is self-catalytic and can proceed without the addition of an external catalyst.

Although such reactions have been investigated in various systems, experimental data on chemical equilibrium and kinetics remain limited [3–6]. Furthermore, the theoretical foundations of self-catalytic esterification kinetics and the existing models for such systems are not universally applicable [5–9]. Therefore, in order to develop technologies for producing organofluorine esters of strong acids, it is essential to conduct both an experi-

mental study of the chemical kinetics of these reaction systems and the development of mathematical models for their description.

The object of this study is the reactions of trifluoroacetic acid (TFA) with 2,2,2-trifluoroethanol (TFE-ol) and TFA with propan-1-ol (P-ol). In addition to scientific interest, the commercial relevance of the products of these reactions should be noted. For example, n-propyl 2,2,2-trifluoroacetate (P-TFA) is used as a component of non-aqueous electrolytes in secondary lithium-ion batteries [10], in various syntheses [11,12] including pesticides [13], semiconductor thin films [14] and ionic liquids [15], and also in perfumery and pharmaceuticals [16]. 2,2,2-Trifluoroethyl trifluoroacetate (TFE-TFA) is used as a highly efficient inhibitor against growth of Li dendrites [17].

Thus, the aim of the present work is to investigate the chemical equilibrium and kinetics of the esterification reaction between TFA with TFE-ol and P-ol without the addition of an external catalyst.

2. Materials and Methods

The study investigated the esterification reactions of TFA with TFE-ol (1) and TFA with P-ol (2).

$$CF_3COOH + CF_3CH_2OH \leftrightarrow CF_3COOCH_2CF_3 + H_2O \quad (1)$$

$$CF_3COOH + CH_3CH_2CH_2OH \leftrightarrow CF_3COOCH_2CH_2CH_3 + H_2O \quad (2)$$

The compounds used in this work, along with their purity, are presented in the Supplementary Materials File (Table S1).

The equilibrium constant (K_{eq}—Equation (3)) of the esterification reactions was determined by the continuous thermostatic of samples at atmospheric pressure starting with a known initial molar ratio of reagents. The samples were thermostated in the thermocabinet TS-1/20 SPU (manufactured "MedComplete A.V.K.", Moscow, Russia) and analyzed periodically. The samples were thermostated until the mixture composition was no longer changed and the fluctuations in the reaction mixture composition over time reached constant values within the experiment's uncertainty. The equilibrium constant is calculated as the average value from the three parallel experiments. A detailed description of the methodology for determining K_{eq} and uncertainty analysis is given in [18].

$$K_{eq} = \frac{x_{est} x_{water}}{x_{alc} x_{TFA}} \quad (3)$$

where x_i is the mole fraction of component i (*est*—ester, *alc*—alcohol).

The dependence of the K_{eq} on temperature T is described by the Van't Hoff Equation (4). The dependence $\ln K_{eq} = (1/T)$ allows us to determine the standard enthalpy and entropy of the reaction.

$$lnK_{eq} = -\frac{\Delta_r H}{RT} + \frac{\Delta_r S}{R} \quad (4)$$

where $\Delta_r H$ is standard enthalpy of the reaction, J·mol^{-1}; $\Delta_r S$ is standard entropy of the reaction, J·mol^{-1}·K^{-1}; R is gas constant, 8.314 J·mol^{-1}·K^{-1}; T is temperature in K.

The study of the kinetics of Reactions (1) and (2) is carried out in a reactor with a stirrer (Figure S1). All the experiments are performed at atmospheric pressure. One of the reactants is loaded into the reactor and heated to working temperature with constant stirring. The second reactant is then added to the system; this point is considered the starting point of the experiment, $\tau = 0$. Working temperature is reached in less than 30 s. Further, the reaction mixture is sampled at specified intervals.

Quantitative analysis of the samples were performed using nuclear magnetic resonance (NMR). Examples of NMR spectra (Figures S2–S4) and a detailed description of the analytical procedure are given in the Supplementary Materials File.

The kinetics of Reactions (1) and (2) are described by Equation (5).

$$v = \frac{dx}{d\tau} = k_1 x_{alc} x_{TFA} - k_2 x_{est} x_{H_2O} \quad (5)$$

where v is the reaction rate in mole fr.·min^{-1}, τ is the reaction time in min, k_1 and k_2 are the rate coefficients of the forward and reverse reaction in mole fr.$^{-1}$·min^{-1}, respectively.

3. Results and Discussion

3.1. Chemical Equilibrium

The investigation of the chemical equilibrium of Reaction (1) at various initial reagent ratios across the temperature range T from 30 to 50 °C revealed that the chemical equilibrium constants K_{eq} is independent on temperature within this range. The experimental values of K_{eq} for Reaction (1) are presented in Table 1.

Table 1. Chemical equilibrium constants K_{eq} of reaction TFA + TFE-ol ↔ TFE-TFA + water in the temperature T range from 30 to 50 °C.

x^0_{TFA}/x^0_{TFE-ol}	9/1	8/2	5/5	2/8	1/9
K_{eq}	0.140	0.124	0.0428	0.00968	0.00164
$\sigma(K_{eq})$	0.022	0.019	0.0026	0.00092	0.00029

It is noteworthy that no splitting of the liquid phase was observed for the equilibrium compositions across all temperatures and mixtures studied.

The chemical equilibrium for Reaction (2) was investigated within the region, where no liquid phase splitting occurs at equilibrium. The experimentally determined equilibrium constants for Reaction (2), along with the standard enthalpy and entropy values derived for the studied initial reagent ratios, are listed in Table 2.

Table 2. Chemical equilibrium constants K_{eq}, standard enthalpy $\Delta_r H$ and entropy $\Delta_r S$ for the reaction TFA + P-ol ↔ P-TFA + water.

x^0_{TFA}/x^0_{P-ol}	$K_{eq} \pm \sigma\ (K_{eq})$			$\Delta_r H$/J·mol^{-1}	$\Delta_r S$/J·mol^{-1}K^{-1}
	30 °C	50 °C	70 °C		
1/9	0.122 ± 0.003	0.151 ± 0.007	0.177 ± 0.010	7998.733	8.951
2/8	0.249 ± 0.006	0.283 ± 0.007	0.322 ± 0.004	5529.725	6.661
3/7	0.545 ± 0.044	0.615 ± 0.045	0.612 ± 0.032	2539.428	3.485

The Van't Hoff equation parameters (Table 2) indicate that Reaction (2) is endothermic under the investigated conditions.

3.2. Chemical Kinetic

The kinetics of the esterification reaction between TFA and TFE-ol were studied at a temperature of 50 °C using initial molar ratios of reactants TFA/TFE-ol of 1/9 and 5/5. The experimental datasets showing the time dependence of the composition of the reaction mixture are provided in the Supplementary Materials File (Tables S2 and S3). The model

used to describe the reaction kinetics is based on Equation (5), taking into account that $K_{eq} = k_1/k_2$ can be expressed as:

$$k_1 d\tau = \frac{dx}{x_{alc}x_{TFA} - \frac{1}{K_{eq}}x_{est}x_{H_2O}} \quad (6)$$

Integrating the left side of Equation (6) with respect to τ and the right side with respect to x yields an equation for calculating the reaction rate coefficient k_1 based on the dependence of the reaction mixture composition x_i on time τ. For the scenario, when no products are present in the system at the initial reaction time τ = 0, at any time τ $x_{est} = x_{water}$, $x_{alc} = x^0_{alc} - x_{est}$ and $x_{TFA} = x^0_{TFA} - x_{est}$, where x^0_i is the mole fraction of component i at τ = 0; the following solution was received in [19]:

$$k_1\tau = \frac{1}{\sqrt{C}} ln\left[\frac{2x^0_{TFA}x^0_{alc} - x_{est}\left(x^0_{TFA} + x^0_{alc} - \sqrt{C}\right)}{2x^0_{TFA}x^0_{alc} - x_{est}\left(x^0_{TFA} + x^0_{alc} + \sqrt{C}\right)}\right] = Y \quad (7)$$

where

$$C = \left(x^0_{TFA} + x^0_{alc}\right)^2 - 4x^0_{TFA}x^0_{alc}\left(1 - {}^1/_{K_{eq}}\right) \quad (8)$$

Figure 1 illustrates the dependences Y, along with the experimental concentration of TFE-TFA over time, along with the calculated values using Equation (7). These calculations utilized the experimental data from Tables S2 and S3 and the chemical equilibrium constants (K_{eq}) from Table 1.

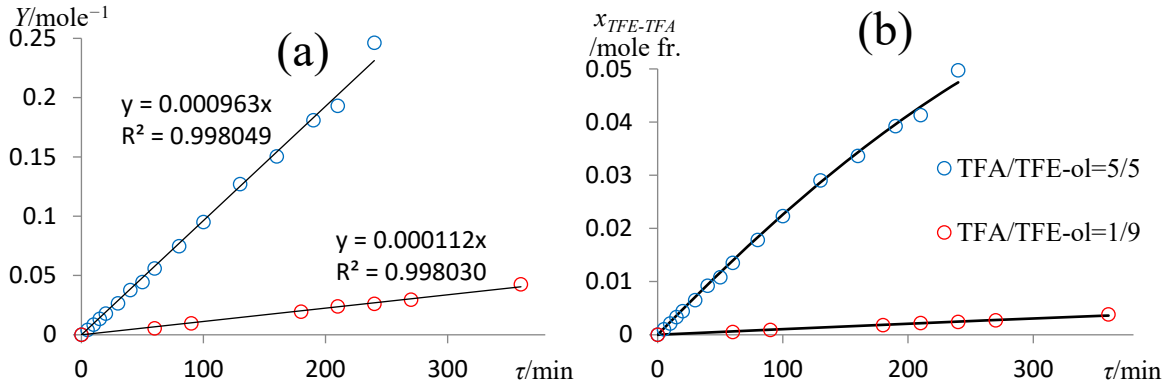

Figure 1. Dependencies of Y (**a**) and TFE-TFA concentration $x_{TFE\text{-}TFA}$ (**b**) on the experiment time τ. Points—experimental data; lines represent the model.

Figure 1a shows that the dependencies $Y = f(\tau)$ are linear. Consequently, according to Equation (7), the reaction rate coefficients k_1 for both considered reagent ratios investigated remain constant during the experiments, corresponding to the slope of the respective line. This model, assuming a constant value for k_1, provides an accurate description of the experimental data (Figure 1b). Although reaction (1) is self-catalytic, no change in reaction rate coefficients k_1 was observed during the experiment, in contrast to the findings reported in [5] for the reaction of TFA with methanol. This is likely attributed to the fact that the change in TFA concentration during the experiment is insufficient to induce a noticeable deviation in the reaction rate coefficients k_1.

The kinetics of the esterification reaction between TFA and P-ol were investigated at an initial molar ratio of the TFA/P-ol = 1/9 and at temperatures of 30, 70 and 80 °C. The experimental dataset illustrating the time τ dependence of the composition x_i of the reaction mixture at different temperatures is in the Supplementary Materials File (Tables S4–S9).

For Reaction (2), the dependence $Y = f(\tau)$ is non-linear (Figure 2), which indicates that the reaction rate coefficient k_1 changes over time τ. This non-linearity is likely attributed to a significant change in the concentration of TFA, which also functions as a catalyst in the reaction process. A decrease in TFA concentration leads directly to a reduction in the reaction rate coefficient k_1. This effect is characteristic of self-catalytic reactions and has been previously observed in TFA esterification reactions with alcohols [5].

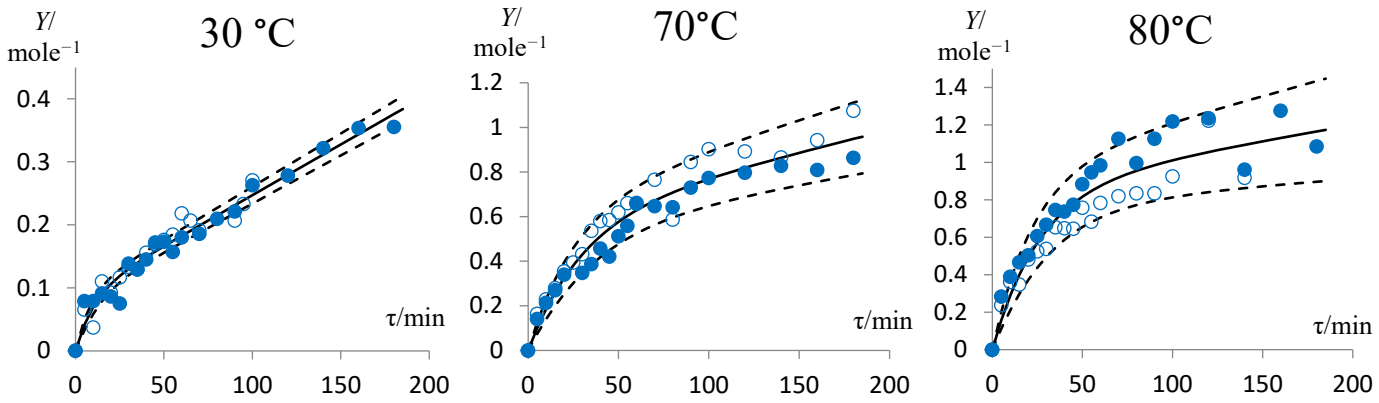

Figure 2. Dependence of Y on time τ. Points: Values calculated by Equation (7) based on experimental data from Tables S4–S9. Empty points: Sets 1; Filled points: Sets 2. Black line: Calculation by Equation (9) using coefficients from Table 2. Dotted lines: Confidence interval $\pm$ $\sigma(Y)$ calculated based on the standard deviation of parameters a, b, and c.

Various methods for describing the kinetics of self-catalytic reactions have been proposed in [5–9]. The models used in [5–7] utilize the activities of the reaction mixture components and treat the reaction process as an interaction between three components: two reagents and a catalyst. Consequently, the rate coefficient k_1 becomes a function of various parameters, such as the activity of the mixture components and the degree of acid dissociation. Furthermore, there are examples of self-catalytic reactions being described as analytical concentrations of substances [8,9]. However, these proposed models also require information about the properties of substances and systems, specifically the dissociation constants of the acids under the studied conditions. For system (2) such data are currently unavailable in the scientific literature.

This work proposes a different approach to describing the kinetics of self-catalytic reactions, based on estimating the values of the rate coefficient k_1 exclusively from kinetic data (the dependence $x = f(\tau)$). According to Equation (6), k_1 can be viewed as the derivative of the right-hand side of the equation with respect to the reaction time τ. or taking into account Equation (7), $k_1 = dY/dt$.

The analytical form of the function describing the dependence $Y = f(\tau)$ can be determined by analyzing the change in the value of k_1 during the reaction. The reaction rate coefficient k_1 is maximum at the initial moment of the reaction τ and decreases over time as a result of the decrease in TFA concentration during the reaction. It asymptotically approaches a minimum value under chemical equilibrium conditions, where the TFA concentration is also minimal. In this scenario, the function $Y = f(\tau)$ starts from the point (0;0) and increases monotonically, asymptotically approaching the line $Y = a\tau + b$, where parameter "a" represents the reaction rate coefficient k_1^{eq} under chemical equilibrium conditions. Thus, the following requirements apply to the function $Y = f(\tau)$: originates from the point (0;0), since when $x_{est} = 0$ $Y = 0$; it is defined and differentiable with respect to τ when $\tau \geq 0$; increases monotonically, asymptotically approaching the line $Y = a\tau + b$;

the derivative of the function with respect to τ decreases asymptotically, tending towards $a = k_1^{eq}$. The following function meets these requirements:

$$Y = a\tau + b(1 - \exp(-c\,\tau)) \quad (9)$$

and its derivative with respect to τ is:

$$dY/d\tau = a + bc \cdot \exp(-c\,\tau) = k_1 \quad (10)$$

It follows that $a + bc$ is the value of the reaction rate coefficient k_1^0 when there are no reaction products in the system, i.e., when $x_{est} = x_{water} = 0$.

The parameters of Equation (9) were estimated using the Levenberg–Marquardt method, which was implemented in Python 3.11. The code used for the fitting procedure is provided in the Supplementary Materials File. The service https://chat.deepseek.com/ (accessed on 20 August 2025) was used in the process of creating the code. The determined parameters of Equation (9) are listed in Table 3.

Table 3. Parameters a, b, and c of Equation (9) for different temperatures T.

T/°C	$a \pm \sigma(a) = k_1^{eq} \pm \sigma(k_1^{eq})$	$b \pm \sigma(b)$	$c \pm \sigma(c)$	$a + bc = k_1^0 \pm \sigma(k_1^0)$
30	0.00161 ± 0.00010	0.0860 ± 0.0089	0.097 ± 0.030	0.0100 ± 0.0027
70	0.00200 ± 0.00071	0.59 ± 0.10	0.0329 ± 0.0081	0.02141 ± 0.00085
80	0.0017 ± 0.0012	0.86 ± 0.16	0.038 ± 0.010	0.0344 ± 0.0016

The fit of dependencies $Y = f(\tau)$ by Equation (9) using the parameters from Table 3 is shown in Figure 2.

The dependence of component concentrations x_i on time τ was calculated using the proposed model by combining Equations (7) and (9). The calculations were performed in Python 3.11; the code used is provided in the Supplementary Materials File. The service https://chat.deepseek.com/ (accessed on 20 August 2025) was used in the process of creating the code. A comparison of experimental data and simulation results is shown in Figure 3.

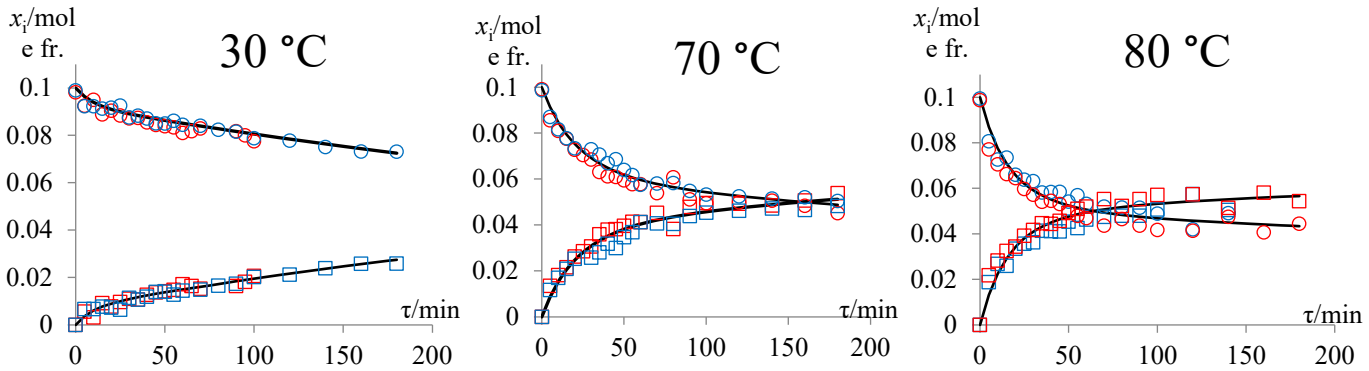

Figure 3. Dependence of the reaction mixture composition x_i on time τ at an initial molar ratio of TFA/P-ol = 1/9 for different temperatures. Dots: Experimental data according to Tables S4–S9. Circles: TFA; Squares: P-TFA. Red: Sets 1; Blue: Sets 2. Lines: Model calculated by Equations (7) and (9).

As seen in Figure 3, the proposed model accurately describes the experimental data. The difference in the behavior of the reaction rate coefficient k_1 between Reactions (1) and (2) is likely due to the difference in the extent of reaction. During Reaction (2), the TFA concentration decreases by 25–55% relative to the initial amount, whereas in Reaction (1), the decrease in TFA concentration does not exceed 5%. Since TFA acts as a catalyst in the studied systems, a significant change in its amount affects the value of the reaction rate coefficient k_1.

4. Conclusions

This study presents, for the first time, a set of original experimental data on the chemical equilibrium and kinetics of the self-catalytic reactions of TFA with P-ol and TFA with TFE-ol. The results demonstrate that the kinetics of the reaction between TFA and TFE-ol are characterized by a constant rate coefficient (k_1) under the studied conditions. In contrast, for the reaction between TFA and P-ol, the rate coefficient (k_1) varies during the reaction. Consequently, a new kinetic model incorporating a variable rate coefficient (k_1) is proposed for the TFA/P-ol system, which provides an accurate description of the experimental data. The obtained experimental data and the proposed model are valuable for the development of production processes for 2,2,2-trifluoroethyl trifluoroacetate and n-propyl 2,2,2-trifluoroacetate. Future research will focus on expanding the experimental dataset and testing the proposed model on other self-catalytic systems.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/engproc2025117065/s1, Table S1: Specifications of the compounds used; Figure S1: Reactor with a stirrer. 1—reaction mixture; 2—jacketed; 3—condenser; 4—thermometer; 5—stirrer bar; Figure S2. ^{19}F NMR spectrum of the reaction mixture CF_3COOH + $CF_3CH_2OH \leftrightarrow CF_3COOCH_2CF_3 + H_2O$ in Acetone; Figure S3. ^{19}F NMR spectrum of the reaction mixture $CF_3COOH + CH_3CH_2CH_2OH \leftrightarrow CF_3COOCH_2CH_2CH_3 + H_2O$ in d-DMSO; Figure S4. ^{1}H NMR spectrum of the reaction mixture $CF_3COOH + CH_3CH_2CH_2OH \leftrightarrow CF_3COOCH_2CH_2CH_3 + H_2O$ in d-DMSO; Table S2: Experimental data of the composition x_i of the reaction mixture over time τ at initial molar ratio TFA/TFE-ol = 1/9 and temperature 50 °C; Table S3: Experimental data of the composition x_i of the reaction mixture over time τ at initial molar ratio TFA/TFE-ol = 5/5, and temperature 50 °C; Table S4: Experimental data of the composition x_i of the reaction mixture over time τ at initial molar ratio TFA/Pol = 1/9 and temperature 30 °C; set 1; Table S5: Experimental data of the composition x_i of the reaction mixture over time τ at initial molar ratio TFA/Pol = 1/9 and temperature 30 °C; set 2; Table S6: Experimental data of the composition x_i of the reaction mixture over time τ at initial molar ratio TFA/Pol = 1/9 and temperature 70 °C; set 1; Table S7: Experimental data of the composition x_i of the reaction mixture over time τ at initial molar ratio TFA/Pol = 1/9 and temperature 70 °C; set 2; Table S8: Experimental data of the composition x_i of the reaction mixture over time τ at initial molar ratio TFA/Pol = 1/9 and temperature 80 °C; set 1; Table S9: Experimental data of the composition x_i of the reaction mixture over time τ at initial molar ratio TFA/Pol = 1/9 and temperature 80 °C; set 2.

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Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

a, *b*, *c*	coefficients of Equations (9) and (10)
D-DMSO	dimethyl sulfoxide-d6
$\Delta_r H$	standard enthalpy of the reaction, J·mol^{-1}
K_{eq}	chemical equilibrium constant
k_1, k_2	rate coefficients of the forward and reverse reaction respectively in mole fr.$^{-1}$·min^{-1}
NMR	nuclear magnetic resonance
P-ol	propan-1-ol
P-TFA	propyl 2,2,2-trifluoroacetate
R	gas constant, 8.314 J·mol^{-1}·K^{-1}
$\Delta_r S$	standard entropy of the reaction, J·mol^{-1}·K^{-1}
TFA	trifluoroacetic acid
TFE-ol	2,2,2-trifluoroethanol
T	temperature in °C or K
σ	standard deviation
τ	reaction time in min
v	reaction rate in mole fr·min^{-1}
x_i	mole fraction of component i.
i	component index
alc	alcohol
est	ester
eq	equilibrium
0	initial condition when $\tau = 0$

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